



SLOVENSKI STANDARD
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Aluminium and aluminium alloys - Chemical analysis - Guideline for spark optical emission spectrometric analysis

Aluminium und Aluminiumlegierungen - Chemische Analyse - Leitfaden für die optische Funkenemissionsspektralanalyse

Aluminium et alliages d'aluminium - Analyse chimique - Directives pour l'analyse par spectrométrie d'émission optique à étincelles

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Aluminium and aluminium alloys - Chemical analysis - Guideline for spark optical emission spectrometric analysis

Aluminium et alliages d'aluminium - Analyse chimique -
Directives pour l'analyse par spectrométrie d'émission
optique à étincelles

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Analyse - Leitfaden für die optische
Funkenemissionsspektralanalyse

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Foreword

This European Standard (EN 14726:2005) 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of an identical text or by endorsement, at the latest by March 2006, and conflicting national standards shall be withdrawn at the latest by March 2006.

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

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EN 14726:2005 (E)**Introduction**

In optical atomic emission spectral analysis (OES) with excitation by electric spark discharges, (S-OES), a small portion of the sample is thermally atomized through the erosion of an electric spark. In the spark discharge, the aerosol is atomized, partially ionised and excited to emit optical radiation. The characteristic radiation for each element is used in OES for element detection and for the quantitative determination of the element contents.

The test result obtained on a small test portion (mostly less than one milligram per spark spot) of one or more laboratory samples is referred either to a laboratory sample or to many tons of a melt or a cast product of aluminium or aluminium alloys. Cast structure and segregation interfere with measurement.

S-OES is suitable for determining the chemical composition of metal samples as defined in technical specifications. S-OES serves as inspection, test and measuring equipment for alloy compositions to control and inspect the manufacturing and casting processes. For those purposes, laboratory samples are taken from the liquid metal after melting down of the alloying constituents and during casting in different process stages.

Besides process inspection, S-OES, within the framework of a chemical-analytical service, facilitates the preparation of a test report which refers to the laboratory sample or to an agreed sample area of that sample.

The testing equipment, including software used in the testing laboratories, is subject to change. Therefore, these guidelines describe the common principles.

It gives guidance to specific criteria which should be met and the detailed documentation for the laboratory procedures to obtain traceable test results with uncertainty intervals.

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1 Scope

This European Standard gives guidance on the criteria and the fundamental operation procedure governing the spark optical emission spectral analysis (S-OES) of metal samples, beginning with sampling, through sample preparation to reporting the test, as well as the requirements of the whole spark source spectrometric procedure which shall be fulfilled with respect to:

- observance of the agreed precision and trueness [ISO 3534-1, ISO 5725-1] of the analytical results with reference to the agreed sample or sample area;
- traceability of the analytical results to the international base units — mass (kg) and amount of substance (mol);
- proof of agreement between the spark spectrometric result and the average composition of the supplied metal sample within the given uncertainty [ISO 3534-1] of the analytical result;
- comparability of the test results between different laboratories;
- as far as possible, a trouble-free operation of the spark spectrometer.

These guidelines refer to computer-controlled simultaneously measuring spectrometers for the analysis of solid samples.

2 Normative references

The following referenced documents are indispensable for the application of this European Standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

- <https://standards.iteh.ai/catalog/standards/sist/c765a072-75bc-4cbc-9ea8-162ff751766d/sist-en-14726-2005>
- 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/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*
- ISO 3534-1, *Statistics — Vocabulary and symbols — Part 1: Probability and general statistical terms*
- ISO 3534-2, *Statistics — Vocabulary and symbols — Part 2: Statistical quality control*
- ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*
- ISO Guide 30, *Terms and definitions used in connection with reference materials*

3 Terms and definitions

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

4 Symbols and abbreviations

Symbols and formula signs are defined immediately by the relevant formulae. Abbreviations are put in brackets immediately after a term first appears in the text (e.g. S-OES).

EN 14726:2005 (E)**5 Review and principle of test method**

- a) Spark spectrometric measurement executed after mechanical preparation of the metal sample, which, if necessary, is taken from the metal melt.
- b) Evaluation of the measuring signal:
 - 1) alloy calibration and universal calibration: with stored evaluation functions, which are evaluated by calibration using reference material of a similar chemical composition;
 - 2) master curve calibration: with recalculated evaluation functions after measuring reference material of known chemical composition.
- c) Evaluation and correction of measuring results as determined in preliminary testing or in accordance with data supplied by the manufacturer of the spectrometer.
- d) Control, documentation and statistical evaluation of all parameters which codetermine the uncertainty (ISO 3534-1) of the analytical results with reference to the mean chemical composition of the supplied sample (see clause 9) or with reference to the agreed sample area.

6 Consumables and calibration materials**6.1 Consumables****6.1.1 General**

The consumables shall be specified in the analytical procedures or in the handbook of the equipment according to the instructions of the equipment manufacturer or according to preliminary tests.

6.1.2 Feed gas of specified purity (argon for spectrometry, high purity, see instrument manufacturer recommendation).

6.1.3 Gas purification cartridge for the feed gas (if required to meet the instrument manufacturer specifications).

6.1.4 Cleaning brushes for the counterelectrode, if needed (the filaments should not contaminate the electrode to any measurable extent).

6.1.5 Particle trap for filtering the metal condensate out of the waste-gas stream.

6.1.6 Spare and expendable parts for the spark spectrometer in accordance with the manufacturer's instructions (e.g. counter electrode, insert for sample table).

6.2 Reference material and recalibration samples**6.2.1 General**

The reference materials (ISO Guide 30) and recalibration samples shall be listed and documented in the testing procedure.

6.2.2 Recalibration samples for drift correction (see clause 12).

6.2.3 Blank sample, high-purity aluminium or aluminium alloy based on high-purity constituents (e.g. AlSn30) (see clause 12).

6.2.4 Binary samples (if required e.g. for line interference correction (see clause 12)).

6.2.5 Calibration samples (see clause 12).

6.2.6 Analytical control samples (see clauses 12 and 13).

6.2.7 Spectrometer control samples (see 14.2).

7 Testing equipment

7.1 Spark spectrometer

Optical atomic emission spectrometer with excitation by spark discharge, suitable for the chemical analysis of aluminium and aluminium alloys for major, minor and trace constituents.

Spark spectrometers comprise the following main functional groups:

- a) system for atomization and excitation:
 - 1) spark generator (spark source);
 - 2) spark stand with counter electrode.
- b) spectral apparatus (system for spectral isolation or radiation splitting).
- c) system designed for radiation measurement (radiation detector).
- d) system for acquisition of the measured values, data processing and evaluation.

NOTE In addition, there are units for power supply, gas supply, instrument control and supervision, for the further processing of analytical results, as well as input and output devices (keyboard, screen, printer, interface module).

In the documentation of spark spectrometer testing equipment, at least the following information shall be recorded (see also EN ISO/IEC 17025):

- designation of device (including internal identification number);
- manufacturer, type designation, serial number;
- procurement documents;
- condition when received (e.g. new, used, reconditioned);
- date placed in service (provisional taking over and taking over or acceptance of the equipment);
- description of the equipment.

The description of the equipment shall include the main properties and characteristic features of the equipment or give information about the relevant manufacturer's notes and instructions, as follows:

- type of spectrometer (e.g. computer-controlled simultaneous measuring spectrometer with 20 detector (measurement) channels for the analysis of);
- spectral apparatus and range of wavelengths (e.g. vacuum polychromator with diffraction grating lines/mm for wavelengths ofnm to nm);
- mounting (type) of spectral apparatus (e.g. 1 metre Paschen-Runge mounting);
- list of measuring channels (element, wavelength, calibration range);
- spark stands, feed gas (sample table, type of counter electrode);
- spark source;
- computer with peripherals;
- software.

EN 14726:2005 (E)**7.2 Equipment for sample preparation**

Lathes, milling machines, circular and band saws, a grinder or any suitable device can be used for the preparation of samples. For each machine, the following shall be documented: designation (if applicable, internal identification number), application range, manufacturer, type, serial number, date received and put into service, condition when received and equipment.

The machine or cutting tool used for the final preparation of the analytical surfaces shall be able to machine reference samples as well as laboratory samples to the same conditions. Adjustable cutting speeds are advantageous for alloys of different hardness.

8 Samples**8.1 Requirements on test samples**

In the test samples, a defined area shall exist which represents the average chemical composition¹⁾. This area shall be sufficiently homogeneous with regard to the test portions. The relation to the average composition of the sample shall be proved, particularly in the case of samples with content gradients (segregations) — (see 8.2.2 and clause 13).

An additional requirement for S-OES is, as far as possible, a similar cast structure in the reference sample and in the laboratory sample. Since reference samples are not available in any and every structural variation, an adapted structure shall be strived for during the production of the laboratory sample (for possible structural influences on the analytical result: see clause 13 and EN 14361).

8.2 Sampling**8.2.1 General**

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The sampling as a procedural step before the spectral analysis plays an essential role in the trueness and precision of the analytical result. By means of sampling, compact (solid) laboratory samples, whose dimensions are suitable for preparation test samples for S-OES and whose chemical composition agree as far as possible with that of the material to be tested, are prepared.

NOTE Since the analytical result refers to the average content of the laboratory sample supplied, uncertainties which result from differences between the laboratory samples and the average composition of the shipment, the cast product, the contents of the casting furnace or from variations in the casting process should also be taken into account during the determination of the process variance or of systematic process deviations.

8.2.2 Sampling of molten metal

The sampling from metal melts shall be carried out according to EN 14361.

For the chemical analysis of molten metals, liquid samples shall be normally taken by means of a ladle. The metal is subsequently cast to produce solid laboratory samples. Alternatively, samples can also be taken with a dip or suction sampler.

The aim is to manufacture a laboratory sample with a known consistent cast structure and a good homogeneity and a distinct, definable test area which represents the respective average content of the complete sample for all analytes of the alloy types to be tested.

The position and size of the representative sample area varies with the sampling conditions, as well as with the type of alloy and the analytes. The contents gradients are evaluated in preliminary tests by means of S-OES and the average contents of the sample determined using independent analytical methods without structural influences. For the simultaneous multi-element analysis of different alloy types, a mean analysis zone is determined and, if required, systematic deviations for individual analytes or alloy types are corrected.

1) The test result refers only to the effective test portion which is known as the vaporized fraction of the sample material which gives the measured value.

NOTE 1 Cylindrical samples with $\varnothing 40 \text{ mm} \times 30 \text{ mm}$ ($\varnothing 55 \text{ mm} \times 30 \text{ mm}$) and disc samples with a central sprue, e.g. $\varnothing 50 \text{ mm} \times 10 \text{ mm}$ or $\varnothing 55 \text{ mm} \times 4,5 \text{ mm}$ (also called plate or mushroom sample) are frequently used.

NOTE 2 The design of the mould should be optimised for minimized, reproducible and defined segregation characteristics. In general this is achieved by fast cooling and directional solidification. Easy handling minimizes individual sampling errors.

8.2.3 Sampling of finished and semi-finished products

A piece, suitable for use on the spark stand, is mechanically separated (e.g. by sawing) from the metal part to be analysed (see NOTE 1). The piece thus cut off shall have a minimum thickness of approximately 1 mm with a plane surface or such that a plane surface can be machined or otherwise prepared, and large enough for a sealing edge to protrude over the opening of the sample table (exception: air stand). When using small samples, care should be taken to ensure that no overheating occurs due to sparking.

NOTE 1 To check the sample's homogeneity using spark spectrometry, the piece of metal or ingot can be analysed at various locations (e.g. along the diagonal through the part); special attention should thus be given to areas susceptible to segregation. A small piece of any finished or semi-finished product can never truly represent the whole and a sample of this type cannot be used to certify a cast. Lost or not properly cast samples means a lost cast, or one that can only be recovered by a concession analysis with the agreement of the customer.

NOTE 2 Possible systematic deviations due to structural differences to the reference material should be given attention during evaluation, or as uncertainty of the test results. A compromise with samples of sufficient size is to melt them in a small furnace under an inert gas to produce a laboratory sample similar to that normally used for S-OES. However, volatile elements, such as magnesium can be partially lost with this technique.

NOTE 3 Mobile spark spectrometers facilitate the semi-quantitative analysis of difficult-to-transport finished and semi-finished products without taking a test sample, but the residual spark crater (spark spot) should be allowed. As a rule, sample preparation is omitted which means that the uncertainty of the test results is greater. Consequently, experience dictates that only identity (confusion) checks or materials sorting can be carried out.

9 Sample preparation

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For spark optical emission spectral analysis, a plane, flat surface in the representative area is produced on the sample by machining. Lathes and milling machines are used.

During final fine machining, which is essential for the analysis, the cutting speed, cutting angle and cutting tool should be chosen in such a way that no sample material is raised above the machined surfaces and that no single hard grains are torn from a soft microstructure. A certain residual roughness (e.g. R_m 16 μm depth of roughness) promotes the formation of electric sparks (see manufacturer's instructions). The machined surface shall not be touched or otherwise contaminated.

For the sample preparation of calibration samples, certified control samples and laboratory samples, the same technique with the same machining parameters shall be used so that a similar surface condition is achieved, unless preliminary tests demonstrate that the influence of the different machining techniques can be neglected compared to the other measuring errors.

NOTE The influence of worn cutting tools can be examined by control samples during the inspection test.

Inspection after machining:

- analytical surface shall be free from pores, cracks, inclusions and shrinkage cavities;
- geometric position of the analytical surface in the defined area of the sample shall be checked regularly by measuring the thickness of the sample before and after machining.