SLOVENSKI STANDARD
SIST EN 14726:2019

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Nadomešča:
SIST EN 14726:2005

Aluminij in aluminijeve zlitine - Ugotavljanje kemijske sestave aluminija in aluminijevih zlitin z optično emisijsko spektrometrijo z iskro

Aluminium and aluminium alloys - Determination of the chemical composition of aluminium and aluminium alloys by spark optical emission spectrometry

Aluminium und Aluminiumlegierungen - Bestimmung der chemischen Zusammensetzung von Aluminium und Aluminiumlegierungen durch optische Funkenemissionsspektralanalyse

Aluminium et alliages d'aluminium - Détermination de la composition chimique de l'aluminium et des alliages d'aluminium par spectrométrie d'émission optique à étincelles

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Aluminium and aluminium alloys - Determination of the chemical composition of aluminium and aluminium alloys by spark optical emission spectrometry

Aluminium et alliages d'aluminium - Détermination de la composition chimique de l'aluminium et des alliages d'aluminium par spectrométrie d'émission optique à étincelles

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (EN 14726:2019) 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of an identical text or by endorsement, at the latest by September 2019, and conflicting national standards shall be withdrawn at the latest by September 2019.

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 14726:2005.

The following modifications were implemented in the present version of EN 14726:

- a new scope;
- updated Normative references;
- reorganization of the standard to respect the template used for the determination of chemical composition;
- new items added in the test report clause (Clause 13);
- new Annexes B, C, D and E.

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, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

In spark optical emission spectrometry (S-OES), a small portion of the sample is thermally vaporized through the erosion of an electric spark. In the spark discharge, the aerosol is vaporized, partially ionized and excited to emit optical radiation. The characteristic radiation of each element is used in spark optical emission spectrometry for its detection and for its quantitative determination.

Optical emission spectrometry (OES): A technique that measures the emission characteristic of a material in the ultraviolet, visible, or infrared wavelength regions of the electromagnetic spectrum. Atomised particles are excited, and each element emits a characteristic radiant energy. This characteristic radiation is detected using either a photomultiplier tube or a solid state detector; appropriate software is used to record the presence of elements and to quantitatively determine elemental content.

Spark optical emission spectrometry (S-OES): A technique that utilizes a high voltage capacitor discharge to ablate and atomise a section of the tested material in an inert atmosphere. The excited atoms and ions emit electromagnetic radiation, which is detected and analysed by an optical emission spectrometer.

Spark optical emission spectrometry is suitable for determining the chemical composition of alloys before the manufacturing and casting processes: in these cases, samples are taken from the liquid metal at different stages of the casting process. Spark optical emission spectrometry is also used to determine the chemical composition of final products.

The method covered by this document is primarily for the analysis of aluminium or aluminium alloy chill cast solid samples, as described in EN 14361, although other samples forms are acceptable.

1 Scope

This document describes the criteria and the procedure for analysing aluminium and aluminium alloys with spark optical emission spectrometry (S-OES). The scope of this document covers the following:

- sample preparation;
- operational guidelines for an optical emission spectrometer (including maintenance);
- traceability of the analytical results to the International System of units: mass (kg);
- assessing the uncertainty associated with each analytical result.

This document refers to simultaneous spark emission spectrometers for the analysis of solid samples.

It applies to the determination of silicon, iron, copper, manganese, magnesium, chromium, nickel, zinc, titanium, boron, gallium, vanadium, beryllium, bismuth, calcium, cadmium, cobalt, lithium, sodium, phosphorus, lead, antimony, tin, strontium and zirconium in aluminium and aluminium alloys.

Elements other than those listed above may be analysed on the condition that:

a) suitable reference materials are available; and

b) the instrument is suitably calibrated and equipped.

In the case of determining mercury, for compliance purposes an alternate method with a limit of quantification < 0,000 1 % is recommended as its detection is compromised by intense iron interference at 253,65 nm.

The test result obtained from a spark optical emission spectrometer generally concerns an amount of less than one milligram per spark spot. The result can be used to refer to the laboratory test sample, to the aluminium or aluminium alloy melt or to the cast product.

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

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

3 Terms and definitions

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

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

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

4 Symbols and abbreviations

Symbols are defined for each formula. Abbreviations are put in brackets immediately after a term first appears in the text (e.g. S-OES).

5 Principle

Measurement of the intensity of the radiation, whose wavelength is characteristic of each element, generated by a spark resulting from the application of an electrical discharge between the sample, as one electrode, and an inert counter-electrode, after mechanical preparation of the surface of the sample, which is in general taken from the metal melt.

The content of each element is determined by relating the measured intensities of the samples to calibration curves.

Signals are evaluated using:

- alloy calibration and universal calibration: reference materials with similar chemical compositions are used to prepare evaluation functions;
- master curve calibration: reference materials with known chemical compositions are measured, and evaluation functions are recalculated.

Evaluation of the accuracy of the results, in accordance with quality assurance procedures, to be defined by each laboratory.

6 Apparatus

6.1 Spark optical emission spectrometer

The optical emission spectrometer shall utilize excitation by spark discharge and be suitable for the determination of the chemical composition of aluminium and aluminium alloys.

Spark optical emission spectrometers are composed of the following main functional devices:

a) system for atomization and excitation:

    1) spark generator (spark source);

    2) spark stand with counter electrode.

b) optical system (system for spectral radiation splitting);

c) system for radiation intensity measurement (radiation detectors);

d) system for acquisition of the measured values, data processing and evaluation.

The documentation of spark optical emission spectrometers should be in accordance with the requirements of EN ISO/IEC 17025.

6.2 Equipment for sample preparation

Lathes, milling machines, circular and band saws, grinders or any other suitable device can be used for the preparation of the samples. Equipment used for the surface final preparation stage shall be capable of machining both reference samples and test samples to the same condition.

NOTE Adjustable cutting speeds are advantageous for alloys of different hardnesses.

7 Consumables and reference materials

7.1 Consumables

Consumables are generally specified:

- in the laboratory analytical procedures,
- in the manufacturer equipment instructions, or
- according to preliminary tests.

Consumables include, but are not restricted to the following:

- feed gas of specified purity (argon for spectrometry, high purity; see instrument manufacturer recommendation);
- gas purification cartridge for the feed gas (if required to meet the instrument manufacturer specifications);
- cleaning brushes for the counter-electrode, if needed (the filaments should not contaminate the electrode);
- particle trap for filtering the metal condensate out of the waste-gas stream;
- spare and expendable parts for the spark optical emission spectrometer in accordance with the manufacturer's instructions (e.g. counter electrode, insert for sample table, etc.).

7.2 Reference materials and recalibration samples

The certified reference materials, the reference materials (see ISO Guide 30) and the recalibration samples should be listed and documented in a laboratory procedure:

- certified reference materials for calibration (see Clause 10);
- blank sample: high-purity aluminium or aluminium alloy prepared from high-purity constituents (e.g. Al Sn30) (see Clause 10);
- binary samples (if required e.g. for line interference correction (see Clause 10));
- control samples for checking the accuracy of the calibration; they shall not be included in the calibration functions (see Clauses 10 and 11);
- samples for the control of the spectrometer drift (see Clause 12);
- recalibration samples for drift correction (see Clause 10).

8 Samples

8.1 General case

Sampling plays an essential role in the accuracy of the analytical results. Sampling allows obtaining laboratory samples whose dimensions are suitable for the preparation of test samples for S-OES and whose chemical composition shall represent that of the material to be tested.

Sampling of molten metal shall be carried out according to EN 14361.

Test samples, shall present a defined area which represents their average chemical composition. This area shall be sufficiently homogeneous across the test section. 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.

NOTE 1 The test result only refers to the effective test area which is the vaporised fraction of the sample.

For the simultaneous multi-element analysis of different alloys, a mean analysis zone should be defined.

For S-OES, there is an additional requirement: as far as possible, the metallographic structure in the test sample and in reference materials should be similar.

NOTE 2 Cylindrical samples with Ø 40 mm × 30 mm (Ø 55 mm × 30 mm) and disc samples with a central sprue, e.g. Ø 50 mm × 10 mm or Ø 55 mm × 4,5 mm (also called plate or mushroom sample) are frequently used.

8.2 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 part to be analysed (see NOTE). Such piece shall have a minimum thickness of approximately 1 mm, cut in such a way that allows the plane surface to be machined or otherwise prepared. Additionally, it should be 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 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.

To check the homogeneity of a sample 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 therefore be given to areas susceptible to segregation.

Attention should be given to possible systematic deviations due to structural differences to the reference materials during evaluation.

A compromise for samples of sufficient size is to re-melt them in a suitable furnace under inert gas to produce a sample similar to those normally used for S-OES. However, volatile elements, such as sodium, magnesium can be partially lost during a re-melting operation.

8.3 Sample preparation

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 machining, 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 promotes the formation of electric sparks (see manufacturer's instructions). The machined surface shall not be touched directly with the fingers or otherwise contaminated, especially for the determination of sodium, calcium and phosphorous.

For the sample preparation of reference materials, check the homogeneity of a sample using the same technique with the same machining parameters shall be used so that a similar surface condition is achieved.

A compromise for samples of sufficient size is to re-melt them in a suitable furnace under inert gas to produce a sample similar to those normally.

9 Operating conditions of the spectrometer and measurements

The operating conditions of the spectrometer shall be optimized.

NOTE 1 For simultaneous spectrometers equipped with photomultipliers, the detector channels for the individual elements are pre-set on the base of the spectral line table, which is generally fixed by the apparatus manufacturer according to each task definition (system requirements/specifications). Changes and expansions are only possible by modifying the spectrometer. The space requirement for a detector channel does not allow the combination of just any lines in a spectrometer of a given design. As a result, several optical spectrometer units are sometimes used in one instrument.

NOTE 2 Information about possible interferences due to line overlap can be deemed from the spectral line reference table, whereby interferences as a result of lines being in a different order can also be considered. In general, the measuring signal of an analytical line is related to a line of the matrix element taken as a reference line. For trace analyses, sometimes the intensity of a background position is used as reference.

NOTE 3 Other measuring conditions, such as spark parameters for pre-sparking and measurement sparking, flush time, pre-spark time, delay time, measurement period, time-resolved intensity measurement, masking-out of the radiation from the plasma, high-voltage adjustment of the photomultiplier tubes, are as a rule adjusted by the manufacturer of the apparatus according to the measuring task or are optimized in accordance with the manufacturer's instructions.

As instrument and computer software design differ, information on measurements, spectrometer controls, the auxiliary equipment and maintenance operations shall be carried out in accordance with the manufacturer's instructions and other relevant documents.

These instructions should be transposed into laboratory procedures describing individual analytical programs and operational processes. To that, the following items should be taken into account:

a) start-up (restart):

   1) check before start-up (e.g. argon feed, exhaust-gas duct, cooling water, vacuum pump oil);

   2) switch on the spectrometer and all units generally in the following order: cooling water pump, vacuum pump (if any), instrument electronics and high voltage power supply;

   3) start computer and analytical program;

   4) check the instrument status stability (e.g. vacuum, temperature, instrument profile);

   5) check the analysis stability (e.g. measurement of suitable spectrometer control samples).

b) switching off:

   1) back-up of software;

   2) close vacuum valve or purge spectrometer with inert gas, if necessary;

   3) switch off the instrument units in the following order: high voltage power supply, instrument electronics, vacuum pump (if any), cooling water pump;

   4) switch off the instrument power supply;

   5) shut off gas feed.