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Copper and copper alloys - Analysis by spark source optical emission spectrometry (S-OES)

Kupfer und Kupferlegierungen - Analyse durch optische Emissionsspektrometrie mit Funkenanregung (F-OES)

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Cuivre et alliages de cuivre - Analyse par spectrométrie d'émission optique a étincelles (SEO-E)

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Copper and copper alloys

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Copper and copper alloys - Analysis by spark source optical emission spectrometry (S-OES)

Cuivre et alliages de cuivre - Analyse par spectrométrie d'émission optique à étincelles (SEO-E) Kupfer und Kupferlegierungen - Analyse durch optische Emissionsspektrometrie mit Funkenanregung (F-OES)

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

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

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Foreword

This document (EN 15079:2007) has been prepared by Technical Committee CEN/TC 133 "Copper and copper alloys", the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by November 2007, and conflicting national standards shall be withdrawn at the latest by November 2007.

Within its programme of work, Technical Committee CEN/TC 133 requested CEN/TC 133/WG 10 "Methods of analysis" to prepare this standard:

EN 15079, Copper and copper alloys — Analysis by spark source optical emission spectrometry (S-OES).

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

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1 Scope

This European Standard specifies a routine method for the analysis of copper and copper alloys by spark source optical emission spectrometry (S-OES). The method is applicable to all elements except copper commonly present in copper and copper alloys present as impurities or minor or main constituents, and detectable by S-OES.

2 Normative references

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

ISO 1811-1, Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 1: Sampling of cast unwrought products

ISO 1811-2, Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 2: Sampling of wrought products and castings

NOTE Informative reference to documents used in the preparation of this standard, and cited at the appropriate places in the text, are listed in the Bibliography.

3 Terms and definitions Teh STANDARD PREVIEW

For the purposes of this document, the following terms and definitions apply.

3.1

reference material

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material, one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a medsurement method, or for assigning values to materials

3.2

certified reference material

reference material, accompanied by a certificate, one or more of whose property values are certified by at least one procedure which establishes its traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence

3.3

test sample

representative quantity of material for testing purposes

3.4

drift control samples

spectrometer control samples

series of homogeneous specimens that contain all the elements which have been calibrated and that cover the low and high concentrations of the calibration range for each element, to be used in detecting variations over time

NOTE The same samples can also be used for statistical process control (SPC) of the instrument.

3.5

recalibration samples

very homogeneous samples at both low and high concentrations in the calibration ranges to be used for recalibration of the spectrometer NOTE 1 These samples are measured during the calibration procedure and the intensities obtained are stored in the computer as indicated in the manufacturer's instructions.

NOTE 2 No chemical analyses are necessary, but the homogeneity of the samples should be carefully tested.

3.6

quality control sample

sample with known composition which is analysed in the same way as the test sample to check the trueness of the analytical results

4 Principle

Measurement of the intensity of the radiation, whose wavelength is characteristic of each element, generated by the spark resulting from the application of an electrical discharge between the sample, as one electrode, and an inert counter-electrode.

The intensity measured is compared with the calibration of the element intensities and then converted to the element content.

5 Apparatus

5.1 Optical emission spectrometer

Spectrometer with spark source capable of measuring the intensities of the optical radiation emitted at specific wavelengths by the elements present in the material site h.ai)

The wavelengths generally used are given in Annex A (informative).

5.2 Appendix https://standards.iteh.ai/catalog/standards/sist/7dcfl fa1-147d-4a07-b2db-

5.2 Apparatus for sample surface preparation 15079-2007

The most common surface preparation techniques for copper and copper alloys are turning, milling or grinding, the last only for copper alloys, provided that the composition of the surface shall not be influenced.

The lathe, milling machine or any other machines used for surface preparation shall be able to produce a surface that conforms to the requirements of 7.1.

WARNING — The appropriate safety recommendations for the use of mechanical apparatus must be observed. These operations shall be carried out only by properly trained personnel wearing appropriate personal protective equipment.

6 Sampling

Sampling shall be carried out in accordance with ISO 1811-1 or ISO 1811-2, as appropriate.

The sample needs to be sufficiently homogeneous with regard to the spark impact. The analytical surface should be free of pores, cracks, sinkholes and slag inclusions.

7 Procedure

7.1 Surface preparation

7.1.1 The surface of the sample shall be prepared to a finish that is sufficiently flat and smooth in order to tighten the sample chamber, reasonably free from contaminants, pores, cracks, inclusions and shrinkage cavities which might otherwise affect analytical results.

In order to avoid variation of results due to the influence of surface finish, the same method of preparation shall be used for all samples.

To avoid cross-contamination between different sample materials, for example pure copper and copper alloys, all relevant components of the machine shall be thoroughly cleaned before use or separate tools shall be used.

Once the surface has been prepared, avoid any contamination, for example fingerprints.

Measurements shall be carried out soon after surface preparation. Samples should be stored, until the time of later measurement, in a desiccator.

7.1.2 The turning, milling or grinding shall be carried out at a suitable speed to avoid undue heating of the sample surface, which might otherwise lead to bias in analysis.

Any lubricants (e.g. propanol) used shall be selected to ensure that they do not affect the analytical result.

7.2 Calibration procedures Teh STANDARD PREVIEW

7.2.1 General

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7.2.1.1 Calibration process

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The calibration process is subdivided into calibration and drift compensation by recalibration.

7.2.1.2 Range of calibration

The range of calibration for an element shall extend well below the minimum content reported in the list of alloys composition and above the maximum concentration reported in the same list, taking into account that the lowest limit should be at least three times the detection limit.

7.2.1.3 Number of sparks on calibration samples

The number of sparks carried out on a reference material for calibration shall be not less than four. The spark areas shall be distributed over the prepared surface. Centre and border of the sample have to be avoided. All measurements shall be examined; if any measurement is obviously defective, further sparks shall be carried out to obtain the minimum four acceptable measurements. The average of the four acceptable measurements is used for calibration.

NOTE The influence of temperature is very important for direct measurement methods. The sample should be cooled to ambient temperature between each spark.

7.2.2 Calibration

The calibration of the spectrometer is carried out using certified reference materials to calculate calibration functions from which the analysis of test samples can be obtained. The calibration functions are usually stored within a computer, integrated to the spectrometer. This calibration functions are 1st or 2nd degree mathematical equations.

The calibration is normally done when the apparatus is installed. The calibration shall be in accordance with the spectrometer manufacturer's instruction manual, using the appropriate certified reference materials, if available. If no certified reference materials are available, reference materials with an accurate analysis shall be used.

The trueness of the analytical procedure is checked by measuring a set of certified reference materials or — if not available — a set of reference materials not used in the calibration. These reference materials shall cover at least the low, mid and high points of the calibration range for each element.

7.2.3 Recalibration

Drifts of the spectrometer readings shall be corrected using a recalibration procedure as described in the manufacturer's instruction manual. Recalibrations can be done either for all analytical channels (global recalibration), or only for individual analytical channels (selective recalibration).

Recalibration can either be done periodically or due to a deviation from statistical process control (SPC) limits (see 7.3.4). When a periodical recalibration procedure is used, the period depends on the stability of the spectrometer and has to be established by a stability check of the spectrometer. The stability check shall be repeated at appropriate intervals.

NOTE The same set of check samples can be used both for drift control and for statistical process control of the spectrometer.

After recalibration, a set of reference materials not used in the recalibration procedure, shall be measured.

7.2.4 Type recalibration the STANDARD PREVIEW

Type recalibration offers a further possibility of correcting instrument drift and, in addition, for matrix influences. Here, one or two reference samples of the same material type and of comparable composition are analysed, together with the test sample. Subsequently, by means of linear correction, the contents of the test sample are related to the certified values of the reference sample(s)st/7dcf1fa1-147d-4a07-b2db-

a8c3a04b7ee6/sist-en-15079-2007This practice can't be employed if the calibration function is a 2nd degree one, unless if the element to be cali-NOTE brated has a very close level to this of the type calibration sample.

7.3 Analysis

7.3.1 Preparation of analytical programs

Due to the fact that programs shall be prepared matrix by matrix, the way in which measurements are taken into account shall be specified. Analytical programs can be prepared either by measuring only intensities of all the elements concerned, or by taking into account the ratios between the measured intensity for each element and the intensity corresponding to the "matrix element" taken as internal standard.

In principle, single intensity measurements are used for trace analysis purposes, whilst rationed measurements are taken for the determination of other levels of content.

During the preparation of each analytical program, the possible optical interferences and/or the inter-elements effects shall also be carefully investigated and suitable corrections shall be made.

Normal mode and virtual mode represent the usual ways to treat the spectrometer outputs.

Normal mode: single comparison of the intensities or of the rationed intensities to the corresponding "calibration functions". This procedure is currently used in cases where the concentration of the element taken as "internal standard" remains constant for all the CRM used for calibration and for all the unknown samples.

Virtual mode: mathematical treatment of the results in order to take into account possible important variations of the concentration of the element chosen as "internal standard" (the copper content in brasses can vary from