



SLOVENSKI STANDARD

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Copper and copper alloys - Determination of main constituents and impurities by wavelength dispersive X-ray fluorescence spectrometry (XRF) - Part 2: Routine method

Kupfer und Kupferlegierungen - Bestimmung von Hauptbestandteilen und Verunreinigungen durch wellenlängendispersive Röntgenfluoreszenzanalyse (RFA) - Teil 2: Routineverfahren

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Cuivre et alliages de cuivre - Détermination des éléments principaux et des impuretés par analyse spectrométrique de fluorescence X à dispersion en longueur d'onde (XRF) - Partie 2 : Méthode de routine

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ICS 77.120.30

English Version

Copper and copper alloys - Determination of main constituents
and impurities by wavelength dispersive X-ray fluorescence
spectrometry (XRF) - Part 2: Routine method

Cuivre et alliages de cuivre - Détermination des éléments
principaux et des impuretés par analyse spectrométrique
de fluorescence X à dispersion en longueur d'onde (XRF) -
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Kupfer und Kupferlegierungen - Bestimmung von
Hauptbestandteilen und Verunreinigungen durch
wellenlängendispersive Röntgenfluoreszenzanalyse (RFA) -
Teil 2: Routineverfahren

This European Standard was approved by CEN on 22 September 2006.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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 Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, 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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Foreword

This document (EN 15063-2:2006) 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 May 2007, and conflicting national standards shall be withdrawn at the latest by May 2007.

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

EN 15063-2, *Copper and copper alloys — Determination of main constituents and impurities by wavelength dispersive X-ray fluorescence spectrometry (XRF) — Part 2: Routine method*

This is one of two parts of the standard for the determination of main constituents and impurities in copper and copper alloys. The other part is:

EN 15063-1, *Copper and copper alloys — Determination of main constituents and impurities by wavelength dispersive X-ray fluorescence spectrometry (XRF) — Part 1: Guidelines to the routine method*

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, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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1 Scope

This part of this European Standard specifies a routine method for analysing copper and copper alloys by wavelength dispersive X-ray fluorescence spectrometry.

The method is applicable to:

- all elements detectable by XRF: impurities, minor and main constituents;
- analysis of either unwrought, including chill-cast or wrought products.

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.

EN 15063-1:2006, *Copper and copper alloys — Determination of main constituents and impurities by wavelength dispersive X-ray fluorescence spectrometry (XRF) — Part 1: Guidelines to the routine method*

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*

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3 Principle

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An appropriately cleaned test specimen is irradiated by an X-ray-beam of high energy. The secondary X-rays produced are dispersed by means of crystals and the intensities are measured by detectors, usually counters at selected characteristics wavelengths.

Concentration of the elements is determined by relating the measured intensities from the sample under test to analytical curves prepared from certified reference materials (CRMs) or reference materials (RMs). Fixed channels or a sequential system are used to provide simultaneous or sequential determinations of element concentrations.

4 Terms and definitions

For the purposes of this European Standard, the terms and definitions given in EN 15063-1:2006 apply.

5 Apparatus

5.1 Sample preparation equipment

Suitable machine; lathe, milling or grinding machine.

NOTE Surface contamination should be avoided.

5.2 X-ray fluorescence spectrometer

Simultaneous or sequential wavelength dispersive spectrometer.

NOTE This test method is written for use with commercially-available instruments.

5.3 X-ray tube

Tube with an anode made of a high-purity element.

NOTE 1 Rhodium is recommended as the material of the anode for analysis of copper and copper alloys.

NOTE 2 Considering a list of elements to be analysed in a copper matrix, it is advisable to check the tube power supply in order to set the right voltage in accordance with the manufacturer's instructions.

The Voltage shall be adjusted to produce secondary fluorescence, according to the following equation:

$$U_f = 12396/\lambda_{\text{abs}} \quad (1)$$

where

U_f is the minimum voltage required for exciting the line of interest;

λ_{abs} is the wavelength of the absorption edge of the fluorescent element.

If a K line is used, the K absorption edge is used.

If an L line is used, the L absorption edge is used.

Ideally, the operating voltage should approximate to or exceed $3 U_f$.

5.4 Analysing crystal

To cover all elements specified in this method, flat or curved crystals, made of LiF and PET (for elements with a low atomic number), are required. Crystals of other materials, optimized for individual elements, can also be used (see Table A.1).

5.5 Collimators

For sequential instruments, a two collimator system is necessary. A coarse collimator for elements with a low atomic number (atomic no. approx. < 22) and a fine collimator for elements with a high atomic number.

5.6 Detectors

One scintillation counter (SC) for elements with a high atomic number and one gas-flow (FC) proportional elements with a low atomic number. Sealed proportional counters can also be used.

NOTE In case of use of gas flow detectors, P 10 gas (90 % argon mixed with 10 % methane) is needed.

5.7 Vacuum System

A vacuum system capable to keep the pressure at constant level of 13 Pa or less.

5.8 Data collection system

An electronic circuit capable of amplifying and integrating pulses received from the counters and a computer-system with a sufficient software package capable of calculating concentrations based on the measured intensities.

6 Sampling

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

7 Procedure

7.1 Surface preparation

7.1.1 A face of the sample shall be prepared by an appropriate method, e.g. turning, milling or grinding so that it is sufficiently flat and smooth, reasonably free from contaminants, pores, cracks, inclusions and shrinkage cavities.

In order to avoid variation in results due to the influence of the surface, the same method of preparation shall be used for all samples (test samples and reference material).

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

Once the surface has been prepared, it shall be kept clean to avoid any contamination, for example fingerprints. If necessary clean the surface, for example with pure ethanol, before measuring.

NOTE Measurements should be carried out as soon as possible after any surface preparation. Samples should be stored in a desiccator until the time of measurement.

7.1.2 Turning, milling or grinding shall be carried out at a suitable rate to avoid undue heating of the sample, which might otherwise cause changes in the sample and lead to variations in analysis.

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

7.2 Preparation of apparatus

Prepare the instrument for operation according to the manufacturer's instructions. Since most of the instruments are used for routine analysis it is assumed that they are operating, and in most cases, are already calibrated for copper and copper alloys.

Verify the calibration status using CRMs or, if not available, RMs.

If the instrument has been turned off for a long period (several hours), ensure that the conditions have stabilized before starting measuring, e.g. temperature and vacuum.

Safety precautions and national regulations for X-ray equipment shall be observed.

NOTE X-ray equipment should be used only under the guidance and supervision of a responsible, qualified person.

7.3 Measurements

Load and measure the samples in accordance with the manufacturer's instruction manual. Ensure that the sample is correctly fixed in the sample cup.

Different grain size dimensions of the samples analysed, compared with the calibration standards or different surface finishes, due to tool wear during preparation of the samples may cause different elemental responses, by which the total percentage of the elements analysed varies from 100 %. If that is the case, each element may be fully re-proportioned using suitable software.

To avoid machining striations influencing measurements, it is usual for instruments to be equipped with a sample spinner. If that is not the case, the sample should be orientated so that the relation between the X-ray beam and the striations is always the same from measurement to measurement.

7.4 Calibration

The instrument shall be calibrated upon installation, after major repairs, or as indicated by SPC-data. To compensate for the instrument's day to day drift, recalibration procedures shall be followed.

If a new calibration is performed, follow the calibration strategy according to the guideline, see EN 15063-1. Typical operating conditions for analysing copper and copper alloys are given in Table A.1 of Annex A of this standard.

Using the same sample preparation, measure the calibration samples together with the recalibration samples according to the manufacturer's instruction manual. Make regression calculations and store the coefficients.

Verify the calibration by measuring a set of CRMs or, if not available, RMs not used in the calibration and that cover at least the low, mid and high points of the calibration range for each element.

Matrix effects might exist between the various elements in the sample. To compensate for inter-element effects, mathematical corrections shall be applied. A variety of computer programmes for correction are commonly used and included in the software package from the manufactures.

7.5 Recalibration

To correct for day to day variations, a recalibration (reference measurement) procedure shall be used. Recalibration actions shall be based on the result of checking the instrument's performance (sample preparation eliminated) using drift control samples. The instrument's performance shall be checked at least every 6 h to 8 h or before and after the measuring a set of test samples.

After recalibration, a set of Drift Control Samples or RMs not used in the recalibration procedure shall be measured.

If the calibration is still outside the control limits, check the whole measurement system carefully (x-ray-tube, detectors, ...) and after a new standardization (reference measurement), a recalibration or calibration procedure shall be undertaken.

7.6 Control of the instrument status

Any change from the original instrument status (e.g. drifts) shall be checked at regular intervals by analysing samples for drift control or reference materials in accordance with the spectrometer manufacturer's instruction manual. It is common practice to document the status of the instrument using control charts. The difference between the results of these control measurements and their reference values are plotted continually on a control chart. If the values fall outside the control limits, then further actions (e.g. recalibration, further control measurements, new calibration) shall be taken.