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SWEDISH STANDARDS INSTITUTE

Koppar och kopparlegeringar – Bestämning av huvudbeståndsdelar och orenheter genom våglängdsskingrande röntgenfluorescencespektrometri (XRF) – Del 1: Riktlinjer för rutinmetod

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

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Europastandarden EN 15063-1:2014 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN 15063-1:2014.

Denna standard ersätter SS-EN 15063-1:2006, utgåva 1.

The European Standard EN 15063-1:2014 has the status of a Swedish Standard. This document contains the official version of EN 15063-1:2014.

This standard supersedes the Swedish Standard SS-EN 15063-1:2006, edition 1.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.

Denna standard är framtagen av kommittén för Koppar, SIS/TK 132.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 15063-1

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ICS 77.040.20; 77.120.30

Supersedes EN 15063-1:2006

English Version

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

Cuivre et alliages de cuivre - Détermination des éléments principaux et des impuretés par spectrométrie de fluorescence X à dispersion de longueur d'onde (FRX) - Partie 1 : Lignes directrices pour la méthode de routine

Kupfer und Kupferlegierungen - Bestimmung von Hauptbestandteilen und Verunreinigungen durch wellenlängendispersive Röntgenfluoreszenzanalyse (RFA) - Teil 1: Leitfaden für das Routineverfahren

This European Standard was approved by CEN on 8 November 2014.

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 CEN-CENELEC Management Centre 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 CEN-CENELEC 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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 15063-1:2014) 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 June 2015 and conflicting national standards shall be withdrawn at the latest by June 2015.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 15063-1:2006.

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

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*

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

In comparison with EN 15063-1:2006, the following changes have been made:

a) Definition 3.1 and 3.2 modified;

b) Clause 5 modified;

c) Editorial modifications have been made.

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, 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, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

Wavelength dispersive X-ray fluorescence spectrometry (XRF) has been used for several decades as an important analytical tool for production analysis. XRF is characterised by its speed and high precision over a wide concentration range and as the XRF-method in most cases is used as a relative method, the limitations are often connected to the quality of the calibration samples. The technique is well established and most of the physical fundamentals are well known.

This guideline is intended to be used for the analysis of copper and copper alloys but it may also be applied to other materials.

1 Scope

This European Standard provides guidance on the concepts and procedures for the calibration and analysis of copper and copper alloys by wavelength dispersive X-ray fluorescence spectrometry.

2 Principle

An appropriately prepared test sample 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 at selected characteristic wavelengths. Concentrations of elements are determined by relating the measured intensities of test samples to calibration curves prepared from reference materials.

3 Terms and definitions

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

3.1
reference material
RM
material, sufficiently homogeneous and stable with respect to one or more specified properties which has been established to be fit for its intended use in a measurement process

[SOURCE: ISO GUIDE 30:1992/Amd.1:2008, definition 2.1]

3.2
certified reference material
CRM
reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate, that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

[SOURCE: ISO GUIDE 30:1992/Amd.1:2008, definition 2.2]

3.3
test sample
representative quantity of material for testing purposes

3.4
calibration samples
series of certified reference materials or if not available, reference materials used for calibration

3.5
drift control samples
series of homogeneous materials that contain all the elements which have been calibrated and that cover the low, mid and high points of the calibration range for each element, used to detect variations over time in these points

Note 1 to entry: Drift control samples can also be used for statistical process control (SPC) of the instrument.

3.6
recalibration samples
samples at both low and high points in the calibration ranges used to recalibrate the spectrometer

Note 1 to entry: These samples are measured during the calibration procedure and the intensities obtained are stored in the computer according to the manufacturer's instructions.

Note 2 to entry: No chemical analyses are necessary, but the homogeneity of these samples should be carefully evaluated.

3.7
calibration
process to establish the curve(s) by measuring and calculating the best fit of net intensities for elemental concentrations of a number of calibration samples

3.8
recalibration
adjusting instrumental output to conform to the calibration

Note 1 to entry: To compensate for day to day instrumental variation, a set of recalibration samples are measured at the minimum low concentration and at a high concentration for each element (two-points recalibration). The measured intensities are compared to the initial measured intensities stored during the calibration procedure and the recalibration coefficients are calculated. Calibration constants are not changed.

3.9
reference measurements
measurements carried out to determine intensities for reference materials

Note 1 to entry: Initial intensities for the reference materials are stored during the calibration procedure and the intensities are updated to compensate for day to day variations.

3.10
spectral background
background caused by radiation energy of a wavelength corrected for its position in the spectrum, but not directly related to the desired observation

Note 1 to entry: For a spectral line, spectral background may consist of other lines, bands or continuous radiation.

3.11
background equivalent concentration
concentration of analyte, which, when it is excited, provides a net intensity equal to the spectral background

Note 1 to entry: See Annex A.

3.12
limit of detection
minimum concentration at which the signal generated by a given element can be positively recognised with a specified confidence level above any background signals

Note 1 to entry: See Annex A.

3.13
lower limit of detection
calculated minimum concentration based on counting statistical error at which the signal generated by a given element can be positively recognised, with a specified confidence level, above any background signals

Note 1 to entry: See Annex A.

3.14
limit of quantification
smallest concentration that can be determined with a specified confidence level related to the limit of detection by a factor dependent on the method

Note 1 to entry: See Annex A.

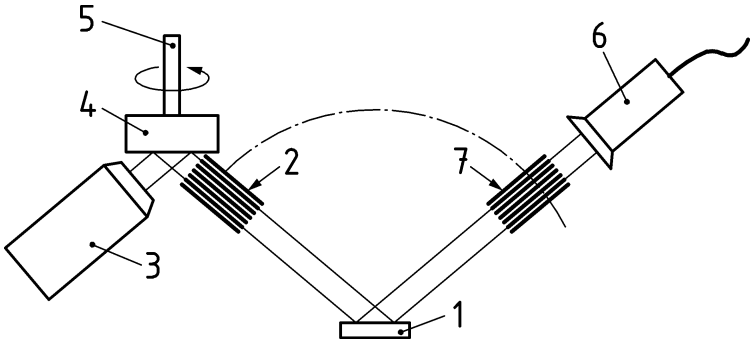
3.15
sensitivity
rate of change of signal with change in concentration

Note 1 to entry: See Annex A. Sensitivity is expressed as counts per second percent, and derived by difference in signals between a sample with a high concentration and one with a low concentration divided by the difference in concentrations.

4 Instrumentation

4.1 Principles of X-ray fluorescence spectrometers

The principles of two different X-ray fluorescence spectrometer concepts are shown in Figures 1 and 2. Each detail is described in the following sub-clauses.



Key

1	Crystal	5	Spinner
2	Primary collimator	6	Counter
3	X-ray tube	7	Secondary collimator
4	Test sample		

Figure 1 — Plane crystal spectrometer geometry, used in sequential instruments