



SLOVENSKI STANDARD

SIST-TS CEN/TS 15916-1:2012

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Baker in bakrove zlitine - Določevanje telurja - 1. del: Majhna koncentracije telurja - Metoda z uporabo spektrometrije s plamensko atomsko absorpcijo (FAAS)

Copper and copper alloys - Determination of tellurium content - Part 1: Low tellurium content – Flame atomic absorption spectrometric method (FAAS)

Kupfer und Kupferlegierungen - Bestimmung des Tellurgehaltes - Teil 1: Niedriger Tellurgehalt - Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

Cuivre et alliages de cuivre - Dosage du tellure - Partie 1: Tellure en faible teneur - Méthode par spectrométrie d'absorption atomique dans la flamme

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ICS:

77.040.30	Kemijska analiza kovin	Chemical analysis of metals
77.120.30	Baker in bakrove zlitine	Copper and copper alloys

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TECHNICAL SPECIFICATION
SPÉCIFICATION TECHNIQUE
TECHNISCHE SPEZIFIKATION

CEN/TS 15916-1

February 2011

ICS 77.120.30

English Version

**Copper and copper alloys - Determination of tellurium content -
Part 1: Low tellurium content - Flame atomic absorption
spectrometric method (FAAS)**

Cuivre et alliages de cuivre - Détermination du tellure -
Partie 1: Tellure en faible teneur - Méthode par
spectrométrie d'absorption atomique dans la flamme
(SAAF)

Kupfer und Kupferlegierungen - Bestimmung des
Tellurgehaltes - Teil 1: Niedriger Tellurgehalt -
Flammenatomabsorptionsspektrometrisches Verfahren
(FAAS)

This Technical Specification (CEN/TS) was approved by CEN on 11 October 2010 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (CEN/TS 15916-1:2011) has been prepared by Technical Committee CEN/TC 133 "Copper and copper alloys", the secretariat of which is held by DIN.

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.

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

CEN/TS 15916-1, *Copper and copper alloys — Determination of tellurium content — Part 1: Low tellurium content — Flame atomic absorption spectrometric method (FAAS)*

This is one of two parts of the Technical Specification/European Standard for the determination of tellurium content in copper and copper alloys. The other part is:

EN 15916-2, *Copper and copper alloys — Determination of tellurium content — Part 2: Medium tellurium content — Flame atomic absorption spectrometric method (FAAS)*

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, 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 the United Kingdom.

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CEN/TS 15916-1:2011 (E)**1 Scope**

This Technical Specification specifies a flame atomic absorption spectrometric method (FAAS) for the determination of the tellurium content of copper and copper alloys in form of castings or unwrought or wrought products.

The method is applicable to products having tellurium mass fractions between 0,000 2 % and 0,020 %.

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*

3 Principle

- Dissolution of a test portion in hydrochloric acid and hydrogen peroxide.
- Precipitation of tellurium by hypophosphorous acid in the presence of arsenic.

After filtration, dissolution of the precipitate with hydrochloric acid - hydrogen peroxide mixture followed, after suitable dilution, by aspiration of the test solution into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 214,3 nm line emitted by a tellurium hollow-cathode lamp.

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4 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Hydrochloric acid, HCl ($\rho = 1,19$ g/ml)

4.2 Nitric acid, HNO₃ ($\rho = 1,40$ g/ml)

4.3 Hypophosphorous acid, H₃PO₂ ($\rho = 1,25$ g/ml)

4.4 Hydrochloric acid solution, 1 + 1

Add 500 ml of hydrochloric acid (4.1) to 500 ml of water.

4.5 Hydrogen peroxide, H₂O₂, 30 % solution ($\rho = 1,11$ g/ml)

4.6 Arsenic(III) oxide, 4,0 g/l solution

Dissolve 4,0 g of arsenic(III) oxide (As₂O₃) in 100 ml of water with the addition of 10 ml of hydrochloric acid solution (4.4), heat gently. Dilute with water to 1 000 ml and mix.

4.7 Arsenic(III) oxide, 0,8 g/l solution

Dilute 100 ml of arsenic(III) oxide solution (4.6) in water to 500 ml.

4.8 Tellurium stock solution, 0,1 g/l Te

Weigh $(0,1 \pm 0,001)$ g of tellurium ($\text{Te} > 99,9 \%$) and transfer it into a 250 ml beaker. Add 20 ml of nitric acid (4.2) and dissolve on a steam bath. Transfer to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this solution immediately prior to use.

1 ml of this solution contains 100 μg of Te.

4.9 Tellurium standard solution, 0,025 g/l Te

Transfer 25,0 ml of the tellurium stock solution (4.8) to a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this solution immediately prior to use.

1 ml of this solution contains 25 μg of Te.

4.10 Hydrochloric acid - hydrogen peroxide mixture

Mix 10 ml of hydrochloric acid (4.1) and 5 ml of hydrogen peroxide (4.5).

Prepare this solution immediately prior to use.

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5 Apparatus

5.1 Membrane filter, cellulose nitrate, 25 mm to 30 mm diameter, pore size 0,45 μm to 0,8 μm , fitted on appropriate filtration apparatus

5.2 Atomic absorption spectrometer, fitted with an air/acetylene burner

5.3 Tellurium hollow-cathode lamp

6 Sampling

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

Test samples shall be in the form of fine drillings, chips or millings with a maximum thickness of 0,5 mm.

7 Procedure

7.1 Preparation of the test portion solution

7.1.1 Test portion

Weigh $(10 \pm 0,001)$ g of the test sample.

7.1.2 Test portion solution

Transfer the test portion (7.1.1) into a 750 ml conical flask. Add 180 ml of hydrochloric acid (4.1) and 30 ml of water. With constant cooling and swirling, complete the dissolution by adding small portions of hydrogen peroxide (4.5). Boil the solution for approximately 5 min to destroy excess hydrogen peroxide, and then allow

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to cool. This operation shall be performed carefully, since the presence of hydrogen peroxide in the test solution will result in a violent reaction when the hypophosphorous acid is added. Add 5 ml of arsenic(III) oxide solution (4.7) and 30 ml of hypophosphorous acid (4.3) to the cooled solution, then boil for 5 min. Allow the precipitate to settle for 30 min before filtering through a membrane filter (5.1). Wash the filter 5 times with small amounts of hydrochloric acid solution (4.4). Transfer the membrane filter and any accessories into a 100 ml beaker. Add 5 ml of warm hydrochloric acid - hydrogen peroxide mixture (4.10). Remove the filter and any accessories and wash them with water. Gently evaporate to a small volume (about 3 ml).

7.1.3 Tellurium mass fraction between 0,000 2 % and 0,001 %

Transfer the solution (7.1.2) to a 5 ml one-mark volumetric flask, wash the beaker sides, dilute to the mark with water and mix. Proceed as indicated in 7.5.

7.1.4 Tellurium mass fraction between 0,001 % and 0,005 %

Transfer the solution (7.1.2) to a 25 ml one-mark volumetric flask. Add 13,5 ml of hydrochloric acid (4.1) and 4 ml of arsenic(III) oxide solution (4.6). Dilute to the mark with water and mix. Proceed as indicated in 7.5.

7.1.5 Tellurium mass fraction between 0,005 % and 0,02 %

Transfer the solution (7.1.2) to a 100 one-mark volumetric flask. Add 64 ml of hydrochloric acid (4.1) and 19 ml of arsenic(III) oxide (4.6). Dilute to the mark with water and mix. Proceed as indicated in 7.5.

7.2 Blank test

Carry out a blank test simultaneously with the determination, following the same procedure and using the same quantities of all reagents as used for the determination, but substituting pure copper for the test portion.

7.3 Check test

Make a preliminary check of the apparatus by preparing a solution of a reference material or a synthetic sample containing a known amount of tellurium and of composition similar to the material to be analysed. Carry out the procedure specified in 7.5.

7.4 Establishment of the calibration curve**7.4.1 Preparation of calibration solutions**

Into each of a series of eight 50 ml one-mark volumetric flasks, introduce 33,5 ml of hydrochloric acid (4.1) and 10,0 ml of arsenic(III) oxide solution (4.6). Add, the volumes of tellurium stock solution (4.8) or tellurium standard solution (4.9) shown in Table 1. Dilute to the mark with water and mix.

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Table 1 — Calibration for tellurium mass fractions between 0,000 2 % and 0,020 %

Tellurium standard solution volume (4.9) ml	Tellurium stock solution volume (4.8) ml	Corresponding tellurium mass mg	Corresponding tellurium concentration after final dilution µg/ml
0 ^a	—	0	0
2	—	0,05	1,0
4	—	0,10	2,0
10	—	0,25	5,0
—	5,0	0,50	10,0
—	7,5	0,75	15,0
—	10,0	1,00	20,0
—	12,5	1,25	25,0

^a Blank test on reagents for calibration curve.

7.4.2 Adjustment of the atomic absorption spectrometer

Fit the tellurium hollow-cathode lamp (5.3) into the atomic absorption spectrometer (5.2), switch on the current and allow it to stabilize. Adjust the wavelength in the region of 214,3 nm to minimum absorbance. Following manufacturer's instructions, fit the correct burner, light the flame and allow the burner temperature to stabilize. The flame shall be set-up on oxidising conditions. Taking careful note of the manufacturer's instructions regarding the minimum flow rate of acetylene, aspirate the calibration solution of highest concentration of analyte and adjust the burner configuration and gas flows to obtain maximum absorbance.

7.4.3 Spectrometric measurement

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Aspirate the series of calibration solutions (7.4.1) in succession into the flame and measure the absorbance for each solution. Take care to keep the of aspiration rate constant throughout the preparation of the calibration curve. Spray water through the burner after each measurement (see NOTE).

NOTE For certain types of spectrometer, instead of water it is preferable to use a solution containing the attack reagents, in the same concentrations as in the test portion solutions.

7.4.4 Calibration curve

Establish the calibration curve using measured absorbances and corresponding analyte amounts. Use appropriate spectrometer software or an off-line computer for regression calculations or prepare a graphical representation.

7.5 Determination

7.5.1 General

The analyses shall be carried out independently, in duplicate.

7.5.2 Preliminary spectrometric measurement

Carry out a preliminary measurement on the test portion solution (7.1.3 or 7.1.4 or 7.1.5) following the procedure specified in 7.4.3 at the same time as the spectrometric measurements are carried out on the standard calibration solutions (7.4.1). Estimate the preliminary analyte amount by using the calibration curve (7.4.4).