



BSI Standards Publication

Copper and copper alloys — Determination of tellurium content

Part 2: Medium tellurium content — Flame
atomic absorption spectrometric method
(FAAS)

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

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

Cuivre et alliages de cuivre - Détermination du tellure -
Partie 2: Tellure en moyenne teneur - Méthode par
spectrométrie d'absorption atomique dans la flamme
(SAAF)

Kupfer und Kupferlegierungen - Bestimmung von
Tellurgehalten - Teil 2: Mittlerer Tellurgehalt -
Flammenatomabsorptionsspektrometrisches Verfahren
(FAAS)

This European Standard was approved by CEN on 19 June 2010.

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Foreword

This document (EN 15916-2:2010) 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of an identical text or by endorsement, at the latest by January 2011, and conflicting national standards shall be withdrawn at the latest by January 2011.

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 document:

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

This is one of two parts of the standard for the determination of tellurium content in copper and copper alloys. The other standard is:

EN 15916-1, *Copper and copper alloys — Determination of tellurium content — Part 1: Low 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 implement this European Standard: 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.

1 Scope

This European Standard 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 0,20 % and 1,00 %.

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 nitric acid followed, after suitable dilution, by aspiration 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.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Nitric acid, HNO_3 ($\rho = 1,40$ g/ml).

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

4.3 Nitric acid solution, 2 + 1.

Add 200 ml of nitric acid (4.1) to 100 ml of water.

4.4 Hydrochloric acid solution, HCl (2 mol/l).

In a 1 000 ml one-mark volumetric flask containing about 500 ml of water, add 165 ml of hydrochloric acid (4.2). Dilute to the mark with water and mix well.

4.5 Tellurium stock solution, 1 g/l Te.

Weigh ($0,5 \pm 0,001$) g of tellurium (Te > 99,9 %) and transfer it into a 250 ml beaker. Add 10 ml of nitric acid solution (4.3) and dissolve on a water bath. Evaporate to a wet residue, and then dissolve in 10 ml of hydrochloric acid solution (4.4). Transfer to a 500 ml one-mark volumetric flask, dilute to the mark with hydrochloric acid solution (4.4) and mix well.

1 ml of this solution contains 1,0 mg of Te.

4.6 Copper base solution, 10 g/l Cu.

Weigh 10,0 g of pure electrolytic copper (Cu > 99,95 %) and transfer it into a 600 ml beaker. Add 200 ml of nitric acid (4.1). Heat gently until copper is dissolved, then heat until all nitrous fumes are expelled. Dilute with hydrochloric acid (4.2). Cool to room temperature. Transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with hydrochloric acid (4.2) and mix well.

5 Apparatus

5.1 Atomic absorption spectrometer, fitted with an air/acetylene burner.

5.2 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 ($0,5 \pm 0,001$) g of the test sample.

7.1.2 Test portion solution

Transfer the test portion (7.1.1) into a 250 ml beaker. Add 10 ml of nitric acid (4.1). Cover with a watch glass and heat gently until the test portion is completely dissolved and then heat until all nitrous fumes have been expelled. Add 40 ml of hydrochloric acid (4.2) and transfer the test solution quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

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 omitting 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

7.4.1.1 General

In all cases, copper, chloride and nitrate concentrations, and acidity in the calibration solutions shall be similar to those of the test portion solutions.

The presence of copper in the calibration solutions compensates for chemical interaction effects of copper in the test solution. Normally no similar additions are required to compensate for the effect of alloying elements. If an alloying element is present in the material to be analysed in mass fraction > 10 %, an appropriate mass of this element shall be added to the calibration solutions. The volumes of copper base solution added (4.6) have been calculated to compensate for chemical interaction effects of copper in test solutions of copper or high-copper alloys. Overcompensation may occur if the same volumes are added when the test samples are copper-based alloys where the percentage of copper is lower. In these cases the volumes of copper base solution shall be decreased to match the copper content of the test sample in solution.

The tellurium concentration of the calibration solutions shall be adjusted to suit the sensitivity of the spectrometer used, so that the curve of absorbance as a function of concentration is a straight line.

7.4.1.2 Tellurium mass fraction between 0,20 % and 1,0 %

Into each of a series of six 100 ml one-mark volumetric flasks, introduce the volumes of tellurium stock solution (4.5) and copper base solution (4.6) shown in Table 1. Dilute to the mark with water and mix well.

Table 1 — Calibration for tellurium mass fractions between 0,20 % and 1,0 %

Tellurium standard solution volume (4.5)	Corresponding tellurium mass	Corresponding tellurium concentration after final dilution	Copper base solution volume (4.6)	Corresponding copper mass	Corresponding tellurium mass fraction of sample
ml	mg	µg/ml	ml	g	%
0 ^a	0	0	50	0,5	0
1	1,0	10	50	0,5	0,2
2	2,0	20	50	0,5	0,4
3	3,0	30	50	0,5	0,6
4	4,0	40	50	0,5	0,8
5	5,0	50	50	0,5	1,0

^a Blank test on reagents for calibration curve.

7.4.2 Adjustment of the atomic absorption spectrometer

Fit the tellurium hollow-cathode lamp (5.2) into the atomic absorption spectrometer (5.1), switch on the current and allow it to stabilize. Adjust the wavelength in the region of 214,3 nm to minimum absorbance. Following the 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

Aspirate the series of calibration solutions (7.4.1.2) in succession into the flame and measure the absorbance for each solution. Take care to keep the aspiration rate constant throughout the preparation of the calibration curve. Spray water through the burner after each measurement.

NOTE For certain types of apparatus, 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 (7.1.2) 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 according to 7.4.4.

NOTE In presence of silver a precipitate of AgCl will occur. Filter before any spectrometric measurement.

7.5.3 Spectrometric measurements

7.5.3.1 Use of the calibration curve

Repeat the measurements and calculate the concentration directly using the calibration curve.

7.5.3.2 Use of bracketing method

Carry out a second measurement on the test portion solution (7.1.2) following the procedure specified in 7.4.3, by bracketing between two calibration solutions of composition similar to that of the calibration solutions (7.4.1), but having tellurium concentrations slightly higher and slightly lower ($\pm 10\%$) than the estimated tellurium concentration of the test portion solution.

To prepare these calibration solutions, follow the procedure specified in 7.4.1 using, however, suitable quantities of tellurium stock solution (4.5).

8 Expression of results

8.1 Use of calibration curve

Calculate the tellurium mass fraction, in percent (%), as follows:

$$w_{\text{Te}} = \frac{c_1}{m} \times V_f \times 100 \quad (1)$$

where

w_{Te} is the tellurium mass fraction in percent (%);

c_1 is the tellurium concentration from the calibration curve, in micrograms per millilitre ($\mu\text{g/ml}$) to be converted in milligrams per millilitre (mg/ml);

m is the mass of the test portion, in milligrams (mg);

V_f is the total volume of the test portion solution (7.1.2), in millilitres (ml).

8.2 Use of bracketing method

Calculate the tellurium mass fraction, in percent (%), as follows:

$$w_{Te} = \frac{c_2}{m} \times V_f \times 100 \quad (2)$$

where

c_2 is the tellurium concentration, calculated using Equation (3), in micrograms per millilitre ($\mu\text{g/ml}$) to be converted in milligrams per millilitre (mg/ml);

m is the mass of the test portion, in milligrams (mg);

V_f is the total volume of the test portion solution (7.1.2), in millilitres (ml).

$$c_2 = C_1 + (C_2 - C_1) \times \frac{S_0 - S_1}{S_2 - S_1} \quad (3)$$

where

C_1 is the lower tellurium concentration of the calibration solution used, in micrograms per millilitre ($\mu\text{g/ml}$), to be converted in milligrams per millilitre (mg/ml);

C_2 is the higher tellurium concentration of the calibration solution used, in micrograms per millilitre ($\mu\text{g/ml}$), to be converted in milligrams per millilitre (mg/ml);

S_0 is the absorbance value of the test portion solution;

S_1 is the absorbance value of the calibration solution corresponding to the concentration C_1 ;

S_2 is the absorbance value of the calibration solution corresponding to the concentration C_2 .

9 Precision

Ten laboratories co-operated in validating this method and obtained the results summarized in Table 2.

Table 2 — Statistical information

Level	Reference value %	Found %	Repeatability r	Reproducibility R
1	0,61	0,605	0,012 0	0,022 0

10 Test report

The test report shall contain the following information:

- a) identification of the test sample;
- b) reference to this European Standard (EN 15916-2);
- c) test method used;
- d) results;
- e) any unusual characteristics noted during the determination;
- f) any operation not included in this European Standard or in the document to which reference is made or regarded as optional;
- g) date of the test and/or date of preparation or signature of the test report;
- h) signature of the responsible person.

Bibliography

- [1] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*
- [2] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*
- [3] ISO 5725-3, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

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