DD CEN/TS 15916-1:2011

BSI Standards Publication

Copper and copper alloys — Determination of tellurium content

Part 1: Low tellurium content — Flame atomic absorption spectrometric method (FAAS)

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National foreword

This Draft for Development is the UK implementation of CEN/TS 15916-1:2011.

This publication is not to be regarded as a British Standard.

It is being issued in the Draft for Development series of publications and is of a provisional nature. It should be applied on this provisional basis, so that information and experience of its practical application can be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the international organization responsible for its conversion to an international standard. A review of this publication will be initiated not later than 3 years after its publication by the international organization so that a decision can be taken on its status. Notification of the start of the review period will be made in an announcement in the appropriate issue of *Update Standards.*

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into an international Standard, to extend the life of the Technical Specification or to withdraw it. Comments should be sent to the Secretary of the responsible BSI Technical Committee at British Standards House, 389 Chiswick High Road, London W4 4AL.

The UK participation in its preparation was entrusted to Technical Committee NFE/34, Copper and copper alloys.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Compliance with a British Standard cannot confer immunity from legal obligations.

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Amendments issued since publication

Date	Text affected

TECHNICAL SPECIFICATION

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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.

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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.

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.

4 Reagents

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

4.1 **Hydrochloric acid,** HCl (ρ = 1,19 g/ml)

4.2 **Nitric acid,** HNO_3 (ρ = 1,40 g/ml)

4.3 **Hypophosphorous acid,** H_3PO_2 (ρ = 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,** H_2O_2, 30 % solution (ρ = 1,11 g/ml)

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

Dissolve 4,0 g of arsenic(III) oxide (As_2O_3) 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 ± 0,001) g of tellurium (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.

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 ± 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

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.

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

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).

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 (7.4.4).

7.5.3.2 Use of bracketing method

Carry out a second measurement on the test solution (7.1.3 or 7.1.4 or 7.1.5) following the procedure specified in 7.4.3, by bracketing between two calibration solutions with a composition similar to that of the calibration solutions (7.4.1), but having tellurium concentrations slightly higher and slightly lower (± 10 %) than the calculated 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 (4.8) or tellurium standard solution (4.9).

8 Expression of results

8.1 Use of calibration curve

Calculate the tellurium mass fraction, in per cent (%), using Equation (1):

$$w_{\mathrm{Te}} = \frac{c_1}{m} \times V_{\mathrm{f}} \times 100 \qquad (1)$$

where

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

c_1 is the tellurium concentration from the calibration curve, in microgram per millilitre (µg/ml) to be converted in milligram per millilitre (mg/ml);

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

V_{f} is the total volume of the test portion solution (7.1.3 or 7.1.4. or 7.1.5), in millilitre (ml).

8.2 Use of bracketing method

Calculate the tellurium mass fraction, in per cent (%), using Equation (2):

$$w_{\mathrm{Te}} = \frac{c_2}{m} \times V_{\mathrm{f}} \times 100 \qquad (2)$$

where

c_2 is the tellurium concentration, calculated using Equation (3), in microgram per millilitre (µg/ml) to be converted in milligram per millilitre (mg/ml);

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

V_{f} is the total volume of the test portion solution (7.1.2), in millilitre (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 microgram per millilitre (µg/ml), to be converted in milligram per millilitre (mg/ml);

C_2 is the higher tellurium concentration of the calibration solution used, in microgram per millilitre (µg/ml), to be converted in milligram per millilitre (mg/ml);

S_0 is the absorbance value of the test portion solution (7.1.3 or 7.1.4 or 7.1.5);

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

Five 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	0,001 01	0,001 00	0,000 049	0,000 209

10 Test report

The test report shall contain the following information:

a) identification of the test sample;

b) reference to this Technical Specification (CEN/TS 15916-1);

c) test method used;

d) results;

e) any unusual characteristics noted during the determination;

f) any operation not included in this Technical Specification 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

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

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*

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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