PD CEN/TS 15656:2015

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Copper and copper alloys — Determination of phosphorus content — Spectrophotometric method

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National foreword

This Published Document is the UK implementation of CEN/TS 15656:2015. It supersedes DD CEN/TS 15656:2009 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee NFE/34/1, Wrought and unwrought copper and copper alloys.

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

Published by BSI Standards Limited 2015

ISBN 978 0 580 87644 8

ICS 77.120.30

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 30 April 2015.

Amendments/corrigenda issued since publication

Date	Text affected

TECHNICAL SPECIFICATION
SPÉCIFICATION TECHNIQUE
TECHNISCHE SPEZIFIKATION

CEN/TS 15656

April 2015

ICS 77.120.30

Supersedes CEN/TS 15656:2009

English Version

Copper and copper alloys - Determination of phosphorus content - Spectrophotometric method

Cuivre et alliages de cuivre - Détermination du phosphore - Méthode spectrophotométrique

Kupfer und Kupferlegierungen - Bestimmung des Phosphorgehaltes - Spektrophotometrisches Verfahren

This Technical Specification (CEN/TS) was approved by CEN on 24 February 2015 for provisional application.

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

Ref. No. CEN/TS 15656:2015 E

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Foreword

This document (CEN/TS 15656:2015) has been prepared by Technical Committee CEN/TC 133 “Copper and copper alloys”, the secretariat of which is held by DIN.

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This document supersedes CEN/TS 15656:2009.

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

CEN/TS 15656:2009, *Copper and copper alloys — Determination of phosphorus content — Spectrophotometric method*.

In comparison with CEN/TS 15656:2009 only editorial modifications have been made.

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, 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.

1 Scope

This Technical Specification specifies a molybdovanadate spectrophotometric method for the determination of phosphorus in copper and copper alloys in the form of castings or unwrought or wrought products.

The method is applicable to products having phosphorus mass fractions between 0,001 % and 0,5 %.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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. Elimination of interfering elements by fuming with perchloric, hydrofluoric and hydrobromic acids. Decomposition of insoluble phosphates by fusion with sodium carbonate. For contents below 0,01 % mass fraction, extraction of phosphorus as phosphomolybdic acid and spectrophotometric determination as molybdenum blue; for contents between 0,005 % and 0,05 % mass fraction, extraction and spectrophotometric determination as phosphovanadomolybdic acid.

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 (ρ = 1,40 g/ml)

4.2 Nitric acid solution, 1 + 1

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

4.3 Hydrofluoric acid, HF (ρ = 1,13 g/ml)

4.4 Perchloric acid, $HClO_4$ (ρ = 1,67 g/ml)

4.5 Hydrobromic acid, HBr (ρ = 1,50 g/ml)

4.6 Isobutanol

4.7 Sodium carbonate, Na_2CO_3

4.8 Methanol

4.9 Methyl isobutyl ketone

4.10 Ammonium molybdate solution, 50 g/l

Dissolve 50 g of ammonium molybdate tetrahydrate $[(NH_4)_6Mo_7O_{24} \cdot 4H_2O]$ in 250 ml of water. Add a solution of 115 ml of the perchloric acid (4.4) and 500 ml of water at room temperature. Dilute to 1 000 ml with water.

After prolonged storage, a white precipitate may form. While this residue will not affect the analysis, care should be taken to prevent its contamination of the aliquot used in the analysis.

Immediately before use, the aliquot used in the analysis should be purified by shaking with 10 ml of the isobutanol (4.6).

4.11 Ammonium molybdate solution, 150 g/l

Dissolve 150 g of ammonium molybdate tetrahydrate $[(NH_4)_6Mo_7O_{24} \cdot 4H_2O]$ in 1 000 ml of water.

4.12 Hydrochloric acid, HCl (ρ = 1,19 g/l)

4.13 Tin(II) chloride solution, 400 g/l

Dissolve 10 g of tin(II) chloride dihydrate ($SnCl_2 \cdot 2H_2O$) in 25 ml of hydrochloric acid (4.12). Prepare this solution fresh before use.

4.14 Sulphuric acid, H_2SO_4 (ρ = 1,84 g/l)

4.15 Sulphuric acid solution, 10 mol/l

To 100 ml of water add 56 ml of sulphuric acid (4.14) while cooling.

4.16 Tin(II) chloride solution, 2 g/l

Dilute 1 ml of the tin(II) chloride stock solution (4.13) with 10 ml of sulphuric acid solution (4.15) and make up to 200 ml with water.

Prepare this solution fresh before use.

4.17 Ammonium vanadate solution, 2,5 g/l

Dissolve 2,5 g of ammonium vanadate (NH_4VO_3) in 1 000 ml of water.

4.18 Citric acid solution, 500 g/l

Dissolve 500 g of citric acid ($C_6H_8O_7$) in 1 000 ml of water.

4.19 Phosphorus stock solution, 100 mg/l

Dissolve 0,439 3 g of potassium dihydrogen orthophosphate (KH_2PO_4), freshly dried at 105 °C, with water into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this stock solution contains 0,1 mg of phosphorus.

4.20 Phosphorus standard solution, 10 mg/l

Transfer 20 ml of the phosphorus stock solution (4.19) into a 200 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this standard solution contains 0,01 mg of phosphorus.

5 Apparatus

All vessels shall be free of contamination by phosphorus. Cleaning with hot hydrochloric acid (4.12), is recommended.

5.1 PTFE beakers, capacity 100 ml

5.2 Spectrophotometer, fitted with cells of optical path lengths 1 cm and 4 cm

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 (1 ± 0,001) g of the test sample.

7.1.2 Test portion solution

7.1.2.1 Dissolve the test portion (7.1.1) in a PTFE beaker (5.1) with 10,0 ml of nitric acid solution (4.2). Heat gently, if necessary. To eliminate silicon, add 0,50 ml of hydrofluoric acid (4.3) and 10,0 ml of perchloric acid (4.4) and heat until fuming begins.

7.1.2.2 Dilute the solution with 10 ml of water and add 10,0 ml of hydrobromic acid (4.5). To eliminate interference from arsenic, antimony and tin, heat gently until fuming begins again. If tin content is > 1 % (mass fraction), repeat the fuming step with 10,0 ml of hydrobromic acid (4.5).

7.1.2.3 Dissolve the copper bromide formed during the fuming steps by adding several millilitres of nitric acid solution (4.2) and bring to fuming. Dilute with 30 ml of water. Heat to boiling for 10 min, then cool to room temperature. Filter the solution through a fine pored filter. Wash the filter with hot water until it is free of acid, then dry and ignite the filter in a small platinum crucible covered with a platinum cover. The temperature has to be increased slowly. Mix the residue with about 0,3 g of sodium carbonate and fuse.

NOTE If the test sample contains zirconium, titanium, niobium and/or tantalum, phosphorus can be found totally or partially as insoluble phosphates. The procedure for dissolving these residues is described below.

After cooling, dissolve the melt with a small amount of water. Filter off any insoluble residue and wash with hot water, adding the washings to the filtrate. Neutralize the combined filtrate and washings with perchloric acid (4.4). Add the neutralized solution of the original copper-containing filtrate. The total volume should not exceed 50 ml; if necessary, the volume should be reduced by evaporating.

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 and of pure copper as used for the determination, but omitting the test portion. Correct the result obtained from the determination in accordance with the result for the blank.

7.3 Check test

Make a preliminary check of the apparatus by preparing a solution of reference material or a synthetic sample containing a known amount of phosphorus and of composition similar to the material to be analysed. Carry out the procedure specified in 7.1 and 7.5.

7.4 Establishment of the calibration curve

7.4.1 Preparation of the calibration solutions

7.4.1.1 General

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

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

7.4.1.2 Phosphorus mass fraction between 0,001 % and 0,005 %

Into each of a series of five PTFE beakers (5.1) introduce in each beaker (1 ± 0,001) g of electrolytic tough pitch copper (P < 0,000 1 %).

Follow exactly the procedure described in 7.1.2. Add the volumes of phosphorus standard solution (4.20) as shown in Table 1, just before heating for fuming with perchloric acid (4.4). Follow the procedure as described in 7.1.2.2, 7.1.2.3 and 7.5.2.1.

Table 1 — Calibration for phosphorus mass fractions between 0,001 % and 0,005 %

Phosphorus standard solution volume (4.20) ml	**Corresponding phosphorus mass** mg	**Corresponding phosphorus concentration after final dilution** mg/ml	**Corresponding phosphorus mass fraction of test sample** %
0[a]	0	0	0
1	0,01	0,000 2	0,001
2	0,02	0,000 4	0,002
4	0,04	0,000 8	0,004
5	0,05	0,001 0	0,005

[a] Blank test on reagents for calibration curve.

7.4.1.3 Phosphorus mass fraction between 0,005 % and 0,01 %

Into each of a series of seven PTFE beakers (5.1) introduce in each beaker (1 ± 0,001) g of electrolytic tough pitch copper (P < 0,000 1 %).

Follow exactly the procedure described in 7.1.2. Add the volumes of phosphorus standard solution (4.20) as shown in Table 2, just before heating for fuming with perchloric acid (4.4). Follow the procedure as described in 7.1.2.2, 7.1.2.3 and 7.5.2.2.

Table 2 — Calibration for phosphorus mass fractions between 0,005 % and 0,01 %

Phosphorus standard solution volume (4.20) ml	Corresponding phosphorus mass mg	Corresponding phosphorus concentration after final dilution mg/ml	Corresponding phosphorus mass fraction of test sample %
0[a]	0	0	0
5	0,05	0,000 5	0,005
6	0,06	0,000 6	0,006
7	0,07	0,000 7	0,007
8	0,08	0,000 8	0,008
9	0,09	0,000 9	0,009
10	0,10	0,001 0	0,010

[a] Blank test on reagents for calibration curve.

7.4.1.4 Phosphorus mass fraction between 0,01 % and 0,10 %

Into each of a series of six PTFE beakers (5.1) introduce in each beaker (1 ± 0,001) g of electrolytic tough pitch copper (P < 0,000 1 %).

Follow exactly the procedure described in 7.1.2. Add the volumes of phosphorus stock solution (4.19) as shown in Table 3, just before heating for fuming with perchloric acid (4.4). Follow the procedure as described in 7.1.2.2, 7.1.2.3 and 7.5.2.3.

Table 3 — Calibration for phosphorus mass fractions between 0,01 % and 0,10 %

Phosphorus stock solution volume (4.19) ml	Corresponding phosphorus mass mg	Corresponding phosphorus concentration after final dilution mg/ml	Corresponding phosphorus mass fraction of test sample %
0[a]	0	0	0
1	0,1	0,002	0,010
4	0,4	0,008	0,040
6	0,6	0,012	0,060
8	0,8	0,016	0,080
10	1,0	0,020	0,10

[a] Blank test on reagents for calibration curve.

7.4.1.5 Phosphorus mass fraction between 0,10 % and 0,50 %

Into each of a series of six PTFE beakers (5.1) introduce in each beaker (1 ± 0,001) g of electrolytic tough pitch copper (P < 0,000 1 %).

Follow exactly the procedure described in 7.1.2. Add the volumes of phosphorus stock solution (4.19) as shown in Table 4, just before heating for fuming with perchloric acid (4.4). Follow the procedure as described in 7.1.2.2, 7.1.2.3, 7.5.2.3 and 7.5.2.4.

Table 4 — Calibration for phosphorus mass fractions between 0,10 % and 0,50 %

Phosphorus stock solution volume (4.19) ml	**Corresponding phosphorus mass** mg	**Corresponding phosphorus concentration after final dilution** mg/ml	**Corresponding phosphorus mass fraction of test sample** %
0[a]	0	0	0
10	1,0	0,004	0,10
20	2,0	0,008	0,20
30	3,0	0,012	0,30
40	4,0	0,016	0,40
50	5,0	0,020	0,50
[a] Blank test on reagents for calibration curve.			

7.4.2 Adjustment of the spectrophotometer

Taking carefully note of the manufacturer's instructions, switch on the current and allow the spectrophotometer (5.2) to stabilize. Set the wavelength at the values of 623 nm or 436 nm as appropriate.

7.4.3 Spectrophotometric measurement of the calibration solutions

Measure the absorbance immediately at the appropriate wavelength using cells of appropriate path length and reference solution as shown in Table 5.

7.4.4 Calibration curve

Establish the calibration curve using measured absorbencies and corresponding analyte amounts.

Use appropriate spectrometer software or off-line computer for regression calculations or prepare a graphical representation.

Table 5 — Wavelength, cell path length and reference solution

Clause Reference	**Phosphorus mass fraction** %	**Wavelength** nm	**Cell path length** cm	**Reference solution**
7.4.1.2	$0,001 \leq P < 0,005$	623	1	1 + 1 Isobutanol (4.6) Methanol (4.8)
7.4.1.3	$0,005 \leq P < 0,01$			
7.4.1.4	$0,01 \leq P < 0,10$	436	4	Methyl isobutyl ketone (4.9)
7.4.1.5	$0,10 \leq P < 0,50$		1	

7.5 Determination

7.5.1 General

The analyses shall be carried out independently, in duplicate.

7.5.2 Procedure

7.5.2.1 Phosphorus mass fraction between 0,001 % and 0,005 %

Transfer the solution from 7.1.2.3 to a 125 ml separating funnel and dilute to 50 ml with water.

Add 10 ml of the ammonium molybdate solution (4.10) and extract the phosphomolybdic acid with 15,0 ml of the isobutanol (4.6) by shaking for about 30 s. After separation of the two phases, transfer the aqueous phase to another separating funnel and repeat the extraction with 5,0 ml of the isobutanol (4.6). Repeat the extraction a third time with 5,0 ml of the isobutanol (4.6), then discard the aqueous phase.

Combine the three isobutanolic extracts in the first separating funnel and wash by shaking twice with 5 ml of water, discarding the wash water each time. To the organic phase, add 15 ml of the tin(II) chloride solution (4.16) and shake for about 30 s. Discard the aqueous layer after separation of the phases. Transfer the blue-coloured organic phase to a 50 ml one-mark volumetric flask and dilute with the methanol (4.8) to the mark.

7.5.2.2 Phosphorus mass fraction between 0,005 % and 0,01 %

Dilute the solution from 7.1.2.3 to the mark with water in a 100 ml one-mark volumetric flask and transfer a 50 ml aliquot portion to a 125 ml separating funnel.

Add 10 ml of the ammonium molybdate solution (4.10) and extract the phosphomolybdic acid with 15,0 ml of the isobutanol (4.6) by shaking for about 30 s. After separation of the two phases, transfer the aqueous phase to another separating funnel and repeat the extraction with 5,0 ml of the isobutanol (4.6). Repeat the extraction a third time with 5,0 ml of the isobutanol (4.6), then discard the aqueous phase.

Combine the three isobutanolic extracts in the first separating funnel and wash by shaking twice with 5 ml of water, discarding the wash water each time. To the organic phase, add 15 ml of the tin(II) chloride solution (4.16) and shake for about 30 s. Discard the aqueous layer after separation of the phases. Transfer the blue-coloured organic phase to a 50 ml one-mark volumetric flask and dilute with the methanol (4.8) to the mark.

7.5.2.3 Phosphorus mass fraction between 0,01 % and 0,10 %

Dilute the solution from 7.1.2.3 with 50 ml of water and 10 ml of nitric acid solution (4.2). Heat to boiling for 10 min, then cool to room temperature.

Add 10 ml of the ammonium vanadate solution (4.17) and 15 ml of the ammonium molybdate solution (4.11) while swirling.

After 10 min, transfer the solution to a separating funnel. Wash the beaker with water and add the washings to the separating funnel. Dilute with water to a total volume of about 100 ml.

Add 10 ml of the citric acid solution (4.18) and extract the phosphovanadomolybdic acid by shaking with 20 ml of the methyl isobutyl ketone (4.9) for 30 s. After separation of the phases, draw off the aqueous phase into the original PTFE beaker. Filter the organic phase through a cotton wool plug into a dry 50 ml one-mark volumetric flask.

Transfer the aqueous phase to the separating funnel and repeat the extraction with 20 ml of the methyl isobutyl ketone (4.9). Discard the aqueous phase and filter the organic phase into the same 50 ml one-mark volumetric flask through a cotton wool plug. Wash the separating funnel with 5 ml of the methyl isobutyl ketone (4.9), filter into the volumetric flask and dilute to the mark with the methyl isobutyl ketone (4.9).

7.5.2.4 Phosphorus mass fraction between 0,10 % and 0,50 %

Dilute the solution from 7.1.2.3 to the mark with water of a 100 ml one-mark volumetric flask. Transfer a 20 ml aliquot portion to a beaker, add 8 ml of the nitric acid solution (4.2) and 8 ml of the perchloric acid (4.4) and dilute with water to about 60 ml, then add 10 ml of the ammonium vanadate solution (4.17) and 15 ml of the ammonium molybdate solution (4.11), while swirling.

After 10 min, transfer the solution to a separating funnel. Wash the beaker with water and add the washings to the separating funnel. Dilute with water to a total volume of about 100 ml.

Add 10 ml of the citric acid solution (4.18) and extract the phosphovanadomolybdic acid by shaking with 20 ml of the methyl isobutyl ketone (4.9) for 30 s. After separation of the phases, draw off the aqueous phase into the original PTFE beaker. Filter the organic phase through a cotton wool plug into a dry 50 ml one-mark volumetric flask.

Transfer the aqueous phase to the separating funnel and repeat the extraction with 20 ml of the methyl isobutyl ketone (4.9). Discard the aqueous phase and filter the organic phase into the same 50 ml one-mark volumetric flask through a cotton wool plug. Wash the separating funnel with 5 ml of the methyl isobutyl ketone (4.9), filter into the volumetric flask and dilute to the mark with the methyl isobutyl ketone (4.9).

7.5.3 Spectrophotometric measurements

Measure in accordance with 7.4.3 and derive the concentration directly using the calibration curve (see 7.4.4).

8 Expression of results

By means of the calibration curve (7.4.1.2, 7.4.1.3, 7.4.1.4, 7.4.1.5) determine the mass of phosphorus, expressed in micrograms, of the test portion, corresponding to the absorbance obtained. The phosphorus content, expressed as a percentage by mass, is given by the formula:

$$\frac{m_1}{m_0 \times 10^6} \times 100 = \frac{m_1}{m_0 \times 10^4} \qquad (1)$$

where

m_0 is the mass of the test portion (7.1.1) or of the aliquot portion taken (7.5.2.2 and 7.5.2.4) in gram (g);

m_1 is the mass of phosphorus found in the test portion solution (7.5.3) in microgram (µg).

9 Precision

One laboratory tested this method and obtained the results summarized in Table 6.

Table 6 — Statistical information

Level	Found %	Repeatability r	Reproducibility R	Reference value %
1	0,004 37	0,000 39	n. a.	—
2	0,027 1	0,000 46	n. a.	—

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 15656);

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 4741, *Copper and copper alloys — Determination of phosphorus content — Molybdovanadate spectrometric method*

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389 Chiswick High Road London W4 4AL UK

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