SLOVENSKI STANDARD

SIST-TS CEN/TS 15605:2008

01-januar-2008

Baker in bakrove zlitine - Analiza s spektrometrijo optiļ ne emisije z induktivno sklopljeno plazmo

Copper and copper alloys - Inductively coupled plasma optical emission spectral analysis

Kupfer und Kupferlegierungen - Optische Emissionsspektralanalyse mit induktiv gekoppelter Plasmaanregung

Cuivre et alliages de cuivre - Analyse par spectrométrie d'émission optique avec source a plasma induit par haute fréquence

Ta slovenski standard je istoveten z: CEN/TS 15605:2007

ICS:

77.120.30 Baker in bakrove zlitine Copper and copper alloys

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TECHNICAL SPECIFICATION

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CEN/TS 15605

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English Version

Copper and copper alloys - Inductively coupled plasma optical emission spectrometry

Cuivre et alliages de cuivre - Analyse par spectrométrie d'émission optique avec source à plasma induit par haute fréquence

Kupfer und Kupferlegierungen - Optische Emissionsspektrometrie mit induktiv gekoppelter Plasmaanregung

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (CEN/TS 15605:2007) 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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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 15605, *Copper and copper alloys — Inductively coupled plasma optical emission spectrometry*

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, 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 document specifies seven inductively coupled plasma emission spectrometry methods (A to G) for the determination of alloying elements and impurities in copper and copper alloys in the form of unwrought, wrought and cast products.

These methods are applicable to the elements listed in Tables 1 to 7 within the composition ranges shown:

Table 1 — Coppers

Element	Mass fraction %	
	min.	max.
Sn	0,02	0,60
Pb	0,02	0,60
Zn	0,02	0,60
Fe	0,01	0,60
Ni	0,01	0,60
Mn	0,01	0,60
Al	0,02	0,60
P	0,01	0,40
Be	0,01	0,60
Co	0,01	0,60
Cd	0,01	0,60

Table 2 — Copper-zinc alloys

Element	Mass fraction %	
	min.	max.
Sn	0,05	2,00
Pb	0,03	4,00
Zn	10,00	42,00
Fe	0,01	5,00
Ni	0,02	4,00
Mn	0,01	6,00
P	0,01	0,40
Al	0,02	9,00
As	0,01	0,20

Table 3 — Copper-tin alloys

Element	Mass fraction %	
	min.	max.
Sn	3,00	16,00
Pb	0,01	9,00
Zn	0,03	6,00
Fe	0,01	1,00
Ni	0,05	7,00
Mn	0,01	0,40
P	0,01	0,60
Al	0,01	0,50
Sb	0,02	1,60
As	0,02	0,25

Table 4 — Copper-aluminium alloys

Element	Mass fraction %	
	min.	max.
Sn	0,02	0,50
Pb	0,03	0,50
Zn	0,03	1,00
Fe	0,05	7,00
Ni	0,10	8,00
Mn	0,01	5,00
Al	6,00	14,00
Cd	0,01	0,50
Mg	0,002	0,15

Table 5 — Copper-beryllium alloys

Element	Mass fraction %	
	min.	max.
Sn	0,02	0,20
Pb	0,01	0,20
Zn	0,03	0,20
Fe	0,03	0,30
Ni	0,04	2,50
Mn	0,006	0,15
Al	0,03	0,20
Be	0,08	4,00
Co	0,03	4,00

Table 6 — Copper-nickel alloys

Element	Mass fraction %	
	min.	max.
Sn	0,10	0,50
Pb	0,03	0,50
Zn	0,04	2,00
Fe	0,10	4,00
Ni	7,00	35,00
Mn	0,02	3,00
Al	0,02	0,50

Table 7 — Copper-tin-lead alloys

Element	Mass fraction %	
	min.	max.
Sn	3,00	12,00
Pb	8,00	25,00
Zn	0,05	3,00
Fe	0,01	0,50
Ni	0,05	3,00
Mn	0,01	0,30
P	0,02	0,40
Al	0,01	0,40
Sb	0,02	0,80

NOTE 1 The ranges specified for each method can be extended or adapted, for the determination of lower mass fractions.

NOTE 2 Other elements may be included. However such elements and their mass fractions should be carefully checked, taking into account interference, sensitivity, resolution and linearity criteria for each instrument and each wavelength.

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*

NOTE Informative references to documents used in the preparation of this Technical Specification, and cited at the appropriate places in the text, are listed in the Bibliography.

3 Principle

Dissolution of a test portion with hydrochloric and nitric acid. After suitable dilution and addition of an internal reference element, nebulization of the solution into an inductively coupled plasma emission spectrometer and measurement of the intensity of the emitted light, including that of the internal reference element.

4 Reagents and materials

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

The same reagents should be used for the preparation of calibration solutions and of sample solutions.

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

4.2 Hydrochloric acid, solution 1 + 1

Dilute 500 ml of hydrochloric acid (4.1) with 500 ml of water.

4.3 Nitric acid, HNO_3 (ρ = 1,40 g/ml).

4.4 Nitric acid, solution 1 + 1

Dilute 500 ml of nitric acid (4.3) with 500 ml of water.

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

4.6 Sulphuric acid, H_2SO_4 (4,5 mol/l).

4.7 Electrolytic copper.

4.8 Zinc granules of 99,999 % purity.

4.9 Aluminium stock solution, 10 g/l Al

Weigh (5 ± 0,001) g of pure aluminium (Al 99,99 % mass fraction), transfer into a 600 ml beaker and cover with a watch glass. Dissolve it in 250 ml hydrochloric acid solution (4.2). After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 10 mg of Al.

4.10 Aluminium stock solution, 1 g/l Al

Weigh (1 ± 0,001) g of pure aluminium (Al 99,99 % mass fraction), transfer into a 400 ml beaker and cover with a watch glass. Dissolve it in 50 ml hydrochloric acid solution (4.2). After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Al.

4.11 Antimony stock solution, 1 g/l Sb

Weigh (0,5 ± 0,001) g of pure antimony (Sb 99,99 % mass fraction), transfer into a 250 ml beaker and cover with a watch glass. Dissolve it in 50 ml hydrochloric acid (4.1) and 25 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, containing 100 ml of hydrochloric acid (4.1), dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Sb.

4.12 Arsenic stock solution, 1 g/l As

Weigh (0,5 ± 0,001) g of pure arsenic (As 99,99 % mass fraction), transfer into a 250 ml beaker and cover with a watch glass. Dissolve it in 20 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of As.

4.13 Beryllium stock solution, 5 g/l Be

Weigh (1 ± 0,001) g of pure beryllium (Be 99,99 % mass fraction), transfer into a 250 ml beaker and cover with a watch glass. Dissolve it in 40 ml hydrochloric acid (4.1). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 200 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 5 mg of Be.

4.14 Beryllium stock solution, 1 g/l Be

Weigh (0,5 ± 0,001) g of pure beryllium (Be 99,99 % mass fraction), transfer into a 250 ml beaker and cover with a watch glass. Dissolve it in 20 ml hydrochloric acid (4.1). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Be.

4.15 Cadmium stock solution, 1 g/l Cd

Weigh (1 ± 0,001) g of pure cadmium (Cd 99,99 % mass fraction), transfer into a 400 ml beaker and cover with a watch glass. Dissolve it in 10 ml nitric acid (4.3). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Cd.

4.16 Cobalt stock solution, 5 g/l Co

Weigh (1 ± 0,001) g of pure cobalt (Co 99,99 % mass fraction), transfer into a 250 ml beaker and cover with a watch glass. Dissolve it in 10 ml hydrochloric acid (4.1) and 10 ml of nitric acid (4.3). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 200 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 5 mg of Co.

4.17 Cobalt stock solution, 1 g/l Co

Weigh (0,5 ± 0,001) g of pure cobalt (Co 99,99 % mass fraction), transfer into a 250 ml beaker and cover with a watch glass. Dissolve it in 5 ml hydrochloric acid (4.1) and 5 ml of nitric acid (4.3). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Co.

4.18 Iron stock solution, 5 g/l Fe

Weigh (5 ± 0,001) g of pure iron (Fe 99,99 % mass fraction), transfer into a 400 ml beaker and cover with a watch glass. Dissolve it in 100 ml hydrochloric acid (4.1) and 50 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 5 mg of Fe.

4.19 Iron stock solution, 1 g/l Fe

Weigh (1 ± 0,001) g of pure iron (Fe 99,99 % mass fraction), transfer into a 250 ml beaker and cover with a watch glass. Dissolve it in 20 ml hydrochloric acid (4.1) and 10 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Fe.

4.20 Lead stock solution, 5 g/l Pb

Weigh (2,5 ± 0,001) g of pure lead (Pb 99,99 % mass fraction), transfer into a 400 ml beaker and cover with a watch glass. Dissolve it in 50 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 5 mg of Pb.

4.21 Lead stock solution, 1 g/l Pb

Weigh (1 ± 0,001) g of pure lead (Pb 99,99 % mass fraction), transfer into a 250 ml beaker and cover with a watch glass. Dissolve it in 20 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Pb.

4.22 Magnesium stock solution, 1 g/l Mg

Weigh (1 ± 0,001) g of pure magnesium (Mg 99,99 % mass fraction), transfer into a 250 ml beaker and cover with a watch glass. Dissolve it in 50 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Mg.

4.23 Manganese stock solution, 5 g/l Mn

The manganese used to prepare the solution is released from superficial oxide possibly present by introducing a few grams of metal in a 250 ml beaker containing 60 ml to 80 ml of sulphuric acid (4,5 mol/l) and approximately 100 ml of water. Shake and after a few seconds, allow the solution to settle and add water. Repeat the water cleaning several times. Remove the metallic manganese, introduce it into acetone, allow to settle and dry the metal in an oven at 100 °C for 2 min. Cool in a dessicator.

Weigh (5 ± 0,001) g of this purified manganese and transfer into a 400 ml beaker and cover with a watch glass. Dissolve it in 50 ml hydrochloric acid (4.1) and 125 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 5 mg of Mn.

4.24 Manganese stock solution, 1 g/l Mn

Weigh (1 ± 0,001) g of manganese, purified as described in 4.23, transfer into a 250 ml beaker and cover with a watch glass. Dissolve it in 10 ml hydrochloric acid (4.1) and 25 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Mn.

4.25 Nickel stock solution, 10 g/l Ni

Weigh (5 ± 0,001) g of pure nickel (Ni 99,99 % mass fraction), transfer into a 400 ml beaker and cover with a watch glass. Dissolve it in 125 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 10 mg of Ni.

4.26 Nickel stock solution, 5 g/l Ni

Weigh (5 ± 0,001) g of pure nickel (Ni 99,99 % mass fraction), transfer into a 400 ml beaker and cover with a watch glass. Dissolve it in 125 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 5 mg of Ni.

4.27 Nickel stock solution, 1 g/l Ni

Weigh (1 ± 0,001) g of pure nickel (Ni 99,99 % mass fraction), transfer into a 250 ml beaker and cover with a watch glass. Dissolve it in 25 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Ni.

4.28 Phosphorus stock solution, 1 g/l P

Weigh (4,394 ± 0,001) g of dried potassium dihydrogenphosphate, transfer into a 250 ml beaker and dissolve it with water. Transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of P.

4.29 Tin stock solution, 10 g/l Sn

Weigh (5 ± 0,001) g of pure tin (Sn 99,99 % mass fraction), transfer into a 400 ml beaker and cover with a watch glass. Dissolve it in 50 ml hydrochloric acid (4.1). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 10 mg of Sn.

4.30 Tin stock solution, 5 g/l Sn

Weigh (2,5 ± 0,001) g of pure tin (Sn 99,99 % mass fraction), transfer into a 400 ml beaker and cover with a watch glass. Dissolve it in 50 ml hydrochloric acid (4.1). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 5 mg of Sn.

4.31 Tin stock solution, 1 g/l Sn

Weigh (0,5 ± 0,001) g of pure tin (Sn 99,99 % mass fraction), transfer into a 250 ml beaker and cover with a watch glass. Dissolve it in 50 ml hydrochloric acid (4.1). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Sn.

4.32 Zinc stock solution, 10 g/l Zn

Weigh (5 ± 0,001) g of pure zinc (Zn 99,99 % mass fraction), transfer into a 400 ml beaker and cover with a watch glass. Dissolve it in 250 ml hydrochloric acid solution (4.2). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 10 mg of Zn.

4.33 Zinc stock solution, 1 g/l Zn

Weigh (1 ± 0,001) g of pure zinc (Zn 99,99 % mass fraction), transfer into a 400 ml beaker and cover with a watch glass. Dissolve it in 50 ml hydrochloric acid solution (4.2). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Zn.

4.34 Internal standard solution, 1g/l Yttrium in 0,5 M nitric acid.

5 Apparatus

5.1 Ordinary laboratory apparatus.

5.2 Optical emission spectrometer (OES), equipped with inductively coupled plasma (ICP) and nebulisation systems

The instrument used will be considered satisfactory if, after optimizing in accordance with the manufacturer's instructions, it meets the performance criteria given in Annex A.