BS EN 15605:2010

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Copper and copper alloys — Inductively coupled plasma optical emission spectrometry

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National foreword

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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.

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ISBN 978 0 580 66406 9

ICS 77.120.30

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 August 2010

Amendments issued since publication

Date	Text affected

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 15605

July 2010

ICS 77.120.30

Supersedes CEN/TS 15605:2007

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

This European Standard was approved by CEN on 12 June 2010.

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Ref. No. EN 15605:2010: E

Contents

Page

Foreword

This document (EN 15605:2010) 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of an identical text or by endorsement, at the latest by January 2011, and conflicting national standards shall be withdrawn at the latest by January 2011.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.

This document supersedes CEN/TS 15605:2007.

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

CEN/TS 15605, *Copper and copper alloys — Inductively coupled plasma optical emission spectrometry*

In comparison with the first edition of CEN/TS 15605:2007, the following significant technical changes were made:

- Revision from a Technical Specification to a European Standard;
- Method G (specifying the analysis of Copper-tin-lead alloys) is edited under an informative basis (see Annex B), taking into account the mediocrity of the precision criteria related to this method;
- Precision criteria for methods A and E were added;
- Precision criteria for methods B, C, D and F were improved and up-dated.

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 United Kingdom.

1 Scope

This European Standard specifies six inductively coupled plasma emission spectrometry methods (A to F) for the determination of alloying elements and impurities in copper and copper alloys in the form of unwrought, wrought and cast products.

A complementary method, for the analysis of Copper-tin-lead alloys, is described in Annex B (informative). The precision criteria concerning this method do not reach the suitable level, for all the elements specified (zinc and phosphorus, namely).

These methods are applicable to the elements listed in Tables 1 to 6 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

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*

3 Principle

Dissolution of a test portion with hydrochloric and nitric acids. 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

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

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

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

4.4 **Nitric acid,** solution 1 + 1

Add 500 ml of nitric acid (4.3) to 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 aluminium (Al ≥ 99,99 %) and transfer into a 600 ml beaker. Add 250 ml of hydrochloric acid solution (4.2) and cover with a watch glass. After cooling to room temperature, 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 aluminium (Al ≥ 99,99 %) and transfer into a 400 ml beaker. Add 50 ml of hydrochloric acid solution (4.2) and cover with a watch glass. After cooling to room temperature, 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 antimony (Sb ≥ 99,99 %) and transfer into a 250 ml beaker. Add 50 ml of hydrochloric acid (4.1) and 25 ml of nitric acid solution (4.4) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling to room temperature, 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 arsenic (As ≥ 99,99 %) and transfer into a 250 ml beaker. Add 20 ml of nitric acid solution (4.4) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling to room temperature, 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 beryllium (Be ≥ 99,99 %) and transfer into a 250 ml beaker. Add 40 ml of hydrochloric acid (4.1) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling to room temperature, 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 beryllium (Be ≥ 99,99 %) and transfer into a 250 ml beaker. Add 20 ml of hydrochloric acid (4.1) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling to room temperature, 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 cadmium (Cd ≥ 99,99 %) and transfer into a 400 ml beaker. Add 10 ml of nitric acid (4.3) and cover with a watch glass. Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling to room temperature, 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 cobalt (Co ≥ 99,99 %) and transfer into a 250 ml beaker. Add 10 ml of hydrochloric acid (4.1) and 10 ml of nitric acid (4.3) and cover with a watch glass. Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling to room temperature, 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 cobalt (Co ≥ 99,99 %) and transfer into a 250 ml beaker. Add 5 ml of hydrochloric acid (4.1) and 5 ml of nitric acid (4.3) and cover with a watch glass. Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling to room temperature, 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 iron (Fe ≥ 99,99 %) and transfer into a 400 ml beaker. Add 100 ml of hydrochloric acid (4.1) and 50 ml of nitric acid solution (4.4) and cover with a watch glass. Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling to room temperature, 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 iron (Fe ≥ 99,99 %) and transfer into a 250 ml beaker. Add 20 ml of hydrochloric acid (4.1) and 10 ml of nitric acid solution (4.4) and cover with a watch glass. Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling to room temperature, 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 lead (Pb ≥ 99,99 %) and transfer into a 400 ml beaker. Add 50 ml of nitric acid solution (4.4) and cover with a watch glass. Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling to room temperature, 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 lead (Pb ≥ 99,99 %) and transfer into a 250 ml beaker. Add 20 ml of nitric acid solution (4.4) and cover with a watch glass. Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling to room temperature, 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 magnesium (Mg ≥ 99,99 %) and transfer into a 250 ml beaker. Add 50 ml of nitric acid solution (4.4) and cover with a watch glass. Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling to room temperature, 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 (4.6) 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 desiccator.

Weigh (5 ± 0,001) g of this purified manganese and transfer into a 400 ml beaker. Add 50 ml of hydrochloric acid (4.1) and 125 ml of nitric acid solution (4.4) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling to room temperature, 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 and transfer into a 250 ml beaker. Add 10 ml of hydrochloric acid (4.1) and 25 ml of nitric acid solution (4.4) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling to room temperature, 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 nickel (Ni ≥ 99,99 %) and transfer into a 400 ml beaker. Add 125 ml of nitric acid solution (4.4) and cover with a watch glass. Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling to room temperature, 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 nickel (Ni ≥ 99,99 %) and transfer into a 400 ml beaker. Add 125 ml of nitric acid solution (4.4) and cover with a watch glass. Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling to room temperature, 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 nickel (Ni ≥ 99,99 %) and transfer into a 250 ml beaker. Add 25 ml of nitric acid solution (4.4) and cover with a watch glass. Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling to room temperature, 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 dihydrogen phosphate into a 1 000 ml one-mark volumetric flask and dissolve it with water. 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 tin (Sn ≥ 99,99 %) and transfer into a 400 ml beaker. Add 50 ml of hydrochloric acid (4.1) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling to room temperature, 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 tin (Sn ≥ 99,99 %) and transfer into a 400 ml beaker. Add in 50 ml of hydrochloric acid (4.1) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling to room temperature, 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 tin (Sn ≥ 99,99 %) and transfer into a 250 ml beaker. Add 50 ml of hydrochloric acid (4.1) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling to room temperature, 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 zinc (Zn ≥ 99,99 %) and transfer into a 400 ml beaker. Add 250 ml of hydrochloric acid solution (4.2) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling to room temperature, 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 zinc (Zn ≥ 99,99 %) and transfer into a 400 ml beaker. Add 50 ml of hydrochloric acid solution (4.2) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling to room temperature, 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 mol/l nitric acid.

5 Apparatus

Optical emission spectrometer (OES), equipped with inductively coupled plasma (ICP) and nebulization 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.

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 chips, drillings or millings, e.g. with a maximum thickness of 0,5 mm.

7 Procedure

7.1 Method A: Coppers

7.1.1 Preparation of the test portion solution

7.1.1.1 Test portion

Weigh (1 ± 0,001) g of the test sample.

7.1.1.2 Test portion solution 5 g/l

Transfer the test portion (7.1.1.1) into a 250 ml beaker.

Add 20 ml of hydrochloric acid solution (4.2) and 5 ml of nitric acid (4.3). Cover with a watch glass and heat gently until the sample is completely dissolved, then heat to boiling. Allow to cool. Rinse the cover and the walls of the beaker with water.

Transfer the dissolved test portion into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix well.

7.1.1.3 Test portion solution 1 g/l

In order to obtain solutions with 1 g/l concentration, pipette exactly 20 ml of the test portion solution (7.1.1.2) into a 100 ml one-mark volumetric flask containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

7.1.2 Check test

Make a preliminary check of the apparatus by preparing a solution of a copper reference material or a synthetic sample containing known amounts of the elements listed in Table 1 and carrying out the procedure specified in 7.1.4.

7.1.3 Establishment of the calibration curves

7.1.3.1 Preparation of the calibration solutions

7.1.3.1.1 General

In all cases the acidity of the calibration solutions shall be similar to that of the test portion solutions.

If, for measurement purposes, a blank is required, prepare a solution containing only the same amounts of acids as in the test solution.

7.1.3.1.2 Preparation of the 5 g/l calibration solutions, using mono-elemental solutions

Weigh the quantities of copper (4.7) shown in Table 7 and introduce them into each of a series of 250 ml beakers.

Add 20 ml of hydrochloric acid solution (4.2) and 5 ml of nitric acid (4.3) to each beaker. Cover with a watch glass and heat gently in order to dissolve the copper. Rinse the cover and the walls of the beaker with water and heat to boiling. Allow to cool and transfer each copper solution into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1).

Transfer the volumes of the elemental solutions of Sn, Pb, Zn, Fe, Ni, Mn, P, Al, Be, Co and Cd shown in Table 7 to each volumetric flask. Dilute to the mark with water and mix well.

7.1.3.1.3 Preparation of the 1 g/l calibration solutions

Dilute accurately the 5 g/l calibration solutions (7.1.3.1.2) in order to obtain 1 g/l calibration solutions.

Pipette exactly 20 ml of the 5 g/l calibration solutions (7.1.3.1.2) into 100 ml one-mark volumetric flasks each containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

Table 7 — Method A — Composition of the 5 g/l calibration solutions

Element	Label			Drift survey solution (see 7.1.3.3)
	P1	P2	P3	
Cu (4.7)	976,3 mg 97,63 %	973,3 mg 97,33 %	979,4 mg 97,94 %	988,0 mg 98,80 %
Sn (4.31)	0,2 mg [a] 0,02 %	0,5mg [b] 0,05 %	6,0 mg 6 ml 0,60 %	1,0 mg 1 ml 0,10 %
Pb (4.21)	6,0 mg 6 ml 0,60 %	0,5mg [c] 0,05 %	0,2mg [d] 0,02 %	1,0 mg 1 ml 0,10 %
Zn (4.33)	0,2mg [e] 0,02 %	0,5 mg [f] 0,05 %	6,0 mg 6 ml 0,60 %	1,0 mg 1 ml 0,10 %
Fe (4.19)	0,5 mg [g] 0,05 %	6,0 mg 6 ml 0,60 %	0,1 mg [h] 0,01 %	1,0 mg 1 ml 0,10 %
Ni (4.27)	0,1 mg [i] 0,01 %	6,0 mg 6 ml 0,60 %	0,5 mg [j] 0,05 %	1,0 mg 1 ml 0,10 %
Mn (4.24)	6,0 mg 6 ml 0,60 %	0,1 mg [k] 0,01 %	0,5 mg [l] 0,05 %	1,0 mg 1 ml 0,10 %
P (4.28)	4,0 mg 4 ml 0,40 %	0,1 mg [m] 0,01 %	0,5 mg [n] 0,05 %	2,0 mg 2 ml 0,20 %
Al (4.10)	6,0 mg 6 ml 0,60 %	0,5 mg [o] 0,05 %	0,2mg [p] 0,02 %	1,0 mg 1 ml 0,10 %
Be (4.14)	0,5 mg [q] 0,05 %	6,0 mg 6 ml 0,60 %	0,1 mg [r] 0,01 %	1,0 mg 1 ml 0,10 %
Co (4.17)	0,1 mg [s] 0,01 %	6,0 mg 6 ml 0,60 %	0,5 mg [t] 0,05 %	1,0 mg 1 ml 0,10 %
Cd (4.15)	0,1 mg [u] 0,01 %	0,5 mg [v] 0,05 %	6,0 mg 6 ml 0,60 %	1,0 mg 1 ml 0,10 %

[a] 2 ml of a 0,1 g/l tin stock solution.
[b] 5 ml of a 0,1 g/l tin stock solution.
[c] 5 ml of a 0,1 g/l lead stock solution.
[d] 2 ml of a 0,1 g/l lead stock solution.
[e] 2 ml of a 0,1 g/l zinc stock solution.
[f] 5 ml of a 0,1 g/l zinc stock solution.
[g] 5 ml of a 0,1 g/l iron stock solution.
[h] 1 ml of a 0,1 g/l iron stock solution.
[i] 1 ml of a 0,1 g/l nickel stock solution.
[j] 5 ml of a 0,1 g/l nickel stock solution.
[k] 1 ml of a 0,1 g/l manganese stock solution.
[l] 5 ml of a 0,1 g/l manganese stock solution.
[m] 1 ml of a 0,1 g/l phosphorus stock solution.
[n] 5 ml of a 0,1 g/l phosphorus stock solution.
[o] 5 ml of a 0,1 g/l aluminium solution.
[p] 2 ml of a 0,1 g/l aluminium stock solution.
[q] 5 ml of a 0,1 g/l beryllium stock solution.
[r] 1 ml of a 0,1 g/l beryllium stock solution.
[s] 1 ml of a 0,1 g/l cobalt stock solution.
[t] 5 ml of a 0,1 g/l cobalt stock solution.
[u] 1 ml of a 0,1 g/l cadmium stock solution.
[v] 5 ml of a 0,1 g/l cadmium stock solution.

7.1.3.2 Adjustment of the apparatus

Start the inductively coupled plasma optical emission spectrometer and let it run for at least half an hour before performing any measurements.

Adjust all the instrumental parameters, each gas (outer, intermediate or central) flow rate, torch position, entrance slits, exit slits, photomultiplier tubes voltages (when appropriate), wavelengths of the analytical lines specified in Table 8, pre-spraying time and integrating time, according to the manufacturer's instructions, while aspirating (for each element) the highest concentration calibration solution.

Prepare the software for the measurement of the intensity of each analytical line and for the calculation of the corresponding mean value and relative standard deviation.

Each time the internal standard is used (Y line 371,03 nm), prepare the software to calculate the ratio between each analyte intensity and internal standard intensity.

Table 8 — Wavelengths for determinations in coppers

Element	**Wavelength** nm
Sn	189,90
Pb	220,30
Zn	334,55 [a]
Fe	238,20
Ni	231,60
Mn	257,60
Al	308,20
P	178,23
Be	313,00
Co	228,60
Cd	226,50

[a] 206,20 nm and 213,80 nm wavelengths may be used to determine zinc, provided that interference from copper has been investigated.

7.1.3.3 Spectrometric measurements of the calibration solutions

Carry out the spectrometric measurements of the 1 g/l solutions identified P1, P2, P3 (see Table 7 and 7.1.3.1.3). For phosphorus use the 5 g/l calibration solutions (7.1.3.1.2). A simultaneous spectrometer shall be available for measurements using yttrium as "internal standard" (ratio mode).

For phosphorus determination use "absolute intensity" taking into account that "Gaussian" measuring mode may also be recommended.

For each calibration solution (including the "drift survey" solution) at least three integrations are necessary in order to calculate the mean ratioed intensities (mean absolute intensity for phosphorus).

In order to control the drift of the spectrometer, analyse the "drift survey" solution (see Table 7 and 7.1.3.1.3) at least once for every five test portion solutions. Maximum drift allowed is given in Table 9.

Table 9 — Drift survey for coppers

Element	**Concentration range** %	**Maximum drift** %
all	0,1 to 0,5	0,02

7.1.3.4 Calibration curves

Establish the calibration curves using measured ratioed intensities (absolute intensity for phosphorus) and corresponding analyte amounts.

Use appropriate spectrometer software for regression calculations and calculate the corresponding correlation coefficients: these coefficients should be, at least, 0,999.

7.1.4 Determination

7.1.4.1 General

The analyses shall be carried out independently, in duplicate.

7.1.4.2 Spectrometric measurement

Carry out measurements on the test solution (7.1.1.3) (7.1.1.2 for phosphorus).

Determine the contents directly using the calibration curves (7.1.3.4).

7.2 Method B: Copper-zinc alloys

7.2.1 Preparation of the test portion solution

7.2.1.1 Test portion

Weigh (1 ± 0,001) g of the test sample.

7.2.1.2 Test portion solution 5 g/l

Transfer the test portion (7.2.1.1) into a 250 ml beaker.

Add 20 ml of hydrochloric acid solution (4.2) and 5 ml of nitric acid (4.3). Cover with a watch glass and heat gently until the sample is completely dissolved, then heat to boiling point. Allow to cool.

Rinse the cover and the walls of the beaker with water.

Transfer the dissolved test portion into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1).

Dilute to the mark with water and mix well.

7.2.1.3 Test portion solution 1 g/l

In order to obtain solutions with 1 g/l concentration, pipette exactly 20 ml of the test portion solution (7.2.1.2) into a 100 ml one-mark volumetric flask containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

7.2.2 Check test

Make a preliminary check of the apparatus by preparing a solution of a copper-zinc reference material or a synthetic sample containing known amounts of the elements listed in Table 2 and carrying out the procedure specified in 7.2.4.

7.2.3 Establishment of the calibration curves

7.2.3.1 Preparation of the calibration solutions

7.2.3.1.1 General

In all cases the acidity in the calibration solutions shall be similar to that of the test portion solutions.

If, for measurement purposes, a blank is required, prepare a solution containing only the same amounts of acids as in the test solution.

7.2.3.1.2 Preparation of the 5 g/l calibration solutions, using mono-elemental solutions

Weigh the quantities of copper (4.7) and zinc (4.8) shown in Table 10 and introduce them into each of a series of 250 ml beakers.

Add 20 ml of hydrochloric acid solution (4.2) and 5 ml of nitric acid (4.3) to each beaker. Cover with a watch glass and heat gently in order to dissolve the metals. Rinse the cover and the walls of the beaker with water and heat to boiling. Allow to cool and transfer each copper-zinc solution into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1).

Transfer the volumes of the elemental solutions of Sn, Pb, Fe, Ni, Mn, P, Al and As shown in Table 10 to each volumetric flask. Dilute to the mark with water and mix well.

7.2.3.1.3 Preparation of the 1 g/l calibration solutions

Dilute accurately the 5 g/l calibration solutions (7.2.3.1.2) in order to obtain 1 g/l calibration solutions.

Pipette exactly 20 ml of the 5 g/l calibration solutions (7.2.3.1.2) into 100 ml one-mark volumetric flasks each containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

Table 10 — Method B — Composition of the 5 g/l calibration solutions

Element	**Label**				**Drift survey solution** (see 7.2.3.3)
	L1	L4	L6	L7	
Cu (4.7)	519,8 mg 51,98 %	517,3 mg 51,73 %	768,1 mg 76,81%	883,5 mg 88,35 %	551,0 mg 55,10 %
Sn (4.31)	0,5 mg [a] 0,05 %	1,0 mg 1 ml 0,10 %	20,0 mg 20 ml 2,00 %	5,0 mg 5 ml 0,50 %	10,0 mg 10 ml 1,00 %
Pb (4.20) and (4.21)	0,3 mg [b] 0,03 %	40,0 mg 8 ml (4.20) 4,00 %	5,0 mg 5 ml (4.21) 0,50 %	1,0 mg 1 ml (4.21) 0,10 %	20,0 mg 4 ml (4.20) 2,00 %
Zn (4.8)	420,0 mg 42,00 %	250,0 mg 25,00 %	200,0 mg 20,00 %	100,0 mg 10,00 %	320,0 mg 32,00 %
Fe (4.18) and (4.19)	50,0 mg 10 ml (4.18) 5,00 %	1,0 mg 1 ml (4.19) 0,10 %	0,1 mg [c] 0,01 %	5,0 mg 5 ml (4.19) 0,50 %	25,0 mg 5 ml (4.18) 2,50 %
Ni (4.26) and (4.27)	0,2 mg [d] 0,02 %	40,0 mg 8 ml (4.26) 4,00 %	5,0 mg 5 ml (4.27) 0,50 %	1,0 mg 1 ml (4.27) 0,10 %	20,0 mg 4 ml (4.26) 2,00 %
Mn (4.23) and (4.24)	0,1 mg [e] 0,01 %	60,0 mg 12 ml (4.23) 6,00 %	1 mg 1 ml (4.24) 0,10 %	2,0 mg 2 ml (4.24) 0,20 %	20,0 mg 4 ml (4.23) 2,00 %
P (4.28)	4,0 mg 4 ml 0,40 %	0,5 mg [f] 0,05 %	0,1 mg [g] 0,01 %	1,0 mg 1 ml 0,10 %	2,0 mg 2 ml 0,20 %
Al (4.9) and (4.10)	5,0 mg 5 ml (4.10) 0,50 %	90,0 mg 9 ml (4.9) 9,00 %	0,2 mg [h] 0,02 %	0,5 mg [i] 0,05 %	30,0 mg 3 ml (4.9) 3,00 %
As (4.12)	0,1 mg [j] 0,01 %	0,2 mg [k] 0,02 %	0,5 mg [l] 0,05 %	1,0 mg 1 ml 0,10 %	2,0 mg 2 ml 0,20 %

[a] 5 ml of a 0,1 g/l tin stock solution.
[b] 3 ml of a 0,1 g/l lead stock solution.
[c] 1 ml of a 0,1 g/l iron stock solution.
[d] 2 ml of a 0,1 g/l nickel stock solution.
[e] 1 ml of a 0,1 g/l manganese stock solution.
[f] 5 ml of a 0,1 g/l phosphorus stock solution.
[g] 1 ml of a 0,1 g/l phosphorus stock solution.
[h] 2 ml of a 0,1 g/l aluminium stock solution.
[i] 1 ml of a 0,5 mg/l aluminium stock solution.
[j] 1 ml of a 0,1 g/l arsenic stock solution.
[k] 2 ml of a 0,1 g/l arsenic stock solution.
[l] 5 ml of a 0,1 g/l arsenic stock solution.

7.2.3.2 Adjustment of the apparatus

Start the inductively coupled plasma optical emission spectrometer and let it run for at least half an hour before performing any measurements.

Adjust all the instrumental parameters, each gas (outer, intermediate or central) flow rate, torch position, entrance slits, exit slits, photomultiplier tubes voltages (when appropriate), wavelengths of the analytical lines specified in Table 11, pre-spraying time and integrating time, according to the manufacturer's instructions, while aspirating (for each element) the highest concentration calibration solutions.

Prepare the software for the measurement of the intensity of each analytical line and for the calculation of the corresponding mean value and relative standard deviation.

Each time the internal standard is used (Y line 371,03 nm) prepare the software to calculate the ratio between each analyte intensity and internal standard intensity.

Table 11 — Wavelengths for determinations in copper-zinc alloys

Element	**Wavelength** nm
Sn	189,90
Pb	220,30
Zn	334,55 [a]
Fe	238,20
Ni	231,60
Mn	257,60
P	178,23
Al	308,20
As	228,82 (188,98 in presence of Cd)

[a] 213,80 nm wavelength may be used to determine zinc, provided that interference from copper has been investigated.

7.2.3.3 Spectrometric measurements of the calibration solutions

Carry out the spectrometric measurements of the 1 g/l solutions identified L1, L4, L6, L7 for all the elements except zinc (see Table 10 and 7.2.3.1.2). For phosphorus and arsenic use the 5 g/l calibration solutions (7.2.3.1.3). For zinc calibration follow, as indicated hereafter:

- Zinc contents between 10 % and 25 %: use L4, L6 and L7;
- Zinc contents between 25 % and 42 %: use L1, L4 and L6.

A simultaneous spectrometer shall be available for measurements using yttrium as "internal standard" (ratio mode).

For phosphorus and arsenic determination, use "absolute intensity" taking into account that "Gaussian" measuring mode may also be recommended.

For each calibration solution (including the "drift survey" solution) at least three integrations are necessary in order to calculate the mean ratioed intensities (mean absolute intensity for phosphorus and arsenic).

In order to control the drift of the spectrometer, analyse the "drift survey" solution (see Table 10 and 7.2.3.1.3) at least once for every five test portion solutions. Maximum drifts allowed are given in Table 12.

Table 12 — Drift survey for copper-zinc alloys

Element	**Concentration range** %	**Maximum drift** %
all	0,5 to 2,0	0,05
	2,0 to 10,0	0,1
	10,0 to 50,0	0,2

7.2.3.4 Calibration curves

Establish the calibration curves using measured ratioed intensities (absolute intensity for phosphorus and arsenic) and corresponding analyte amounts.

Use appropriate spectrometer software for regression calculations and calculate the corresponding correlation coefficients: these coefficients should be, at least, 0,999.

7.2.4 Determination

7.2.4.1 General

The analyses shall be carried out independently, in duplicate.

7.2.4.2 Spectrometric measurement

Carry out measurements on the test solution (7.2.1.3) (7.2.1.2 for phosphorus and arsenic).

Determine the contents directly using the calibration curves (7.2.3.4).

7.3 Method C: Copper-tin alloys

7.3.1 Preparation of the test portion solution

7.3.1.1 Test portion

Weigh (1 ± 0,001) g of the test sample.

7.3.1.2 Test portion solution 5 g/l

Transfer the test portion (7.3.1.1) into a 250 ml beaker.

Add 20 ml of hydrochloric acid solution (4.2) and 5 ml of nitric acid (4.3). Cover with a watch glass and heat gently until the sample is completely dissolved, then heat to boiling. Allow to cool. Rinse the cover and the walls of the beaker with water.

Transfer the dissolved test portion into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1).

Dilute to the mark with water and mix well.

7.3.1.3 Test portion solution 1 g/l

In order to obtain solutions with 1 g/l concentration, pipette exactly 20 ml of the test portion solution (7.3.1.2) into a 100 ml one-mark volumetric flask containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

7.3.2 Check test

Make a preliminary check of the apparatus by preparing a solution of a copper-tin reference material or a synthetic sample containing known amounts of the elements listed in Table 3 and carrying out the procedure specified in 7.3.4.

7.3.3 Establishment of the calibration curves

7.3.3.1 Preparation of the calibration solutions

7.3.3.1.1 General

In all cases the acidity of the calibration solutions shall be similar to that of the test portion solutions.

If, for measurement purposes, a blank is required, prepare a solution containing only the same amounts of acids as in the test solution.

7.3.3.1.2 Preparation of the 5 g/l calibration solutions, using mono-elemental solutions

Weigh the quantities of copper (4.7) shown in Table 13 and introduce them into each of a series of 250 ml beakers.

Add 20 ml of hydrochloric acid solution (4.2) and 5 ml of nitric acid (4.3) to each beaker. Cover with a watch glass and heat gently in order to dissolve the copper. Rinse the cover and the walls of the beaker with water and heat to boiling. Allow to cool and transfer each copper solution into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1).

Transfer the volumes of the elemental solutions of Sn, Pb, Zn, Fe, Ni, Mn, P, Sb, Al and As shown in Table 13 to each volumetric flask. Dilute to the mark with water and mix well.

7.3.3.1.3 Preparation of the 1 g/l calibration solutions

Dilute accurately the 5 g/l calibration solutions (7.3.3.1.2) in order to obtain 1 g/l calibration solutions.

Pipette exactly 20 ml of the 5 g/l calibration solutions (7.3.1.2) into a 100 ml one-mark volumetric flasks each containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

Table 13 — Method C — Composition of the 5 g/l calibration solutions

Element	Label			Drift survey solution (see 7.3.3.3)
	E1	E5	E8	
Cu (4.7)	739,9 mg 73,99 %	902,5 mg 90,25 %	804,9 mg 80,49 %	777,0 mg 77,70 %
Sn (4.29)	160,0 mg 16 ml 16,00 %	90,0 mg 9 ml 9,00 %	30,0 mg 3 ml 3,00 %	120,0 mg 12 ml 12,00 %
Pb (4.20) and (4.21)	0,1 mg [a] 0,01 %	1,0 mg 1 ml (4.21) 0,10 %	90,0 mg 18 ml (4.20) 9,00 %	40,0 mg 8 ml (4.20) 4,00 %
Zn (4.32) and (4.33)	0,3 mg [b] 0,03 %	1,0 mg 1 ml (4.33) 0,10 %	60,0 mg 6 ml (4.32) 6,00 %	20,0 mg 20 ml (4.33) 2,00 %
Fe (4.19)	0,1 mg [c] 0,01 %	1,0 mg 1 ml 0,10 %	10,0 mg 10 ml 1,00 %	2,0 mg 2 ml 0,20 %
Ni (4.26) and (4.27)	70,0 mg 14 ml (4.26) 7,00 %	1,0 mg 1 ml (4.27) 0,10 %	0,5 mg [d] 0,05 %	30,0 mg 6 ml (4.26) 3,00 %
P (4.28)	6,0 mg 6 ml 0,60 %	1,0 mg 1 ml 0,10 %	0,1 mg [e] 0,01 %	2,0 mg 2 ml 0,20 %
Sb (4.11)	16,0 mg 16 ml 1,60 %	1,0 mg 1 ml 0,10 %	0,2 mg [f] 0,02 %	5,0 mg 5 ml 0,50 %
Mn (4.24)	0,1 mg [g] 0,01 %	0,5 mg [h] 0,05 %	4,0 mg 4 ml 0,40 %	1,0 mg 1 ml 0,10 %
Al (4.10)	5,0 mg 5 ml 0,50 %	0,5 mg [i] 0,05 %	0,1 mg [j] 0,01 %	2,0 mg 2 ml 0,20 %
As (4.12)	2,5 mg 2,5 ml 0,25 %	0,5 mg [k] 0,05 %	0,2 mg [l] 0,02 %	1,0 mg 1 ml 0,10 %

[a] 1 ml of a 0,1 g/l lead stock solution.

[b] 3 ml of a 0,1 g/l zinc stock solution.

[c] 1 ml of a 0,1 g/l iron stock solution.

[d] 5 ml of a 0,1 g/l nickel stock solution.

[e] 1 ml of a 0,1 g/l phosphorus stock solution.

[f] 2 ml of a 0,1 g/l antimony stock solution.

[g] 1 ml of a 0,1 g/l manganese stock solution

[h] 5 ml of a 0,1 g/l manganese stock solution

[i] 5 ml of a 0,1 g/l aluminium stock solution

[j] 1 ml of a 0,1 g/l aluminium stock solution

[k] 5 ml of a 0,1 g/l arsenic stock solution

[l] 1 ml of a 0,1 g/l arsenic stock solution

7.3.3.2 Adjustment of the apparatus

Start the inductively coupled plasma optical emission spectrometer and let it run for at least half an hour before performing any measurements.

Adjust all the instrumental parameters, each gas (outer, intermediate or central) flow rate, torch position, entrance slits, exit slits, photomultiplier tubes voltages (when appropriate), wavelengths of the analytical lines specified in Table 14, pre-spraying time and integrating time, according to the manufacturer's instructions, while aspirating (for each element) the highest concentration calibration solutions.

Prepare the software for the measurement of the intensity of each analytical line and for the calculation of the corresponding mean value and relative standard deviation.

Each time the internal standard is used (Y line 371,03 nm) prepare the software to calculate the ratio between each analyte intensity and internal standard intensity.

Table 14 — Wavelengths for determinations in copper-tin alloys

Element	**Wavelength** nm
Sn	189,90
Pb	220,30
Zn	334,55 [a]
Fe	238,20
Ni	231,60
Mn	257,60
Al	308,20
P	178,23
Sb	206,80
As	228,82 (188,98 in presence of Cd)

[a] 213,80 nm wavelength may be used to determine zinc, provided that interference from copper has been investigated.

7.3.3.3 Spectrometric measurements of the calibration solutions

Carry out the spectrometric measurements of the 1 g/l solutions identified E1, E5, E8 (see Table 13 and 7.3.3.1.3). For phosphorus and arsenic use the 5 g/l calibration solutions (7.3.3.1.2).

A simultaneous spectrometer shall be available for measurements using yttrium as "internal standard" (ratio mode).

For phosphorus and arsenic determination use "absolute intensity" taking into account that "Gaussian" measuring mode may also be recommended.

For each calibration solution (including the "drift survey" solution) at least three integrations are necessary in order to calculate the mean ratioed intensities (mean absolute intensity for phosphorus and arsenic).

In order to control the drift of the spectrometer, analyse the "drift survey" solution (see Table 13 and 7.3.3.1.3) at least once for every five test portion solutions. Maximum drifts allowed are given in Table 15.

Table 15 — Drift survey for copper-tin alloys

Element	**Concentration range** %	**Maximum drift** %
all	0,1 to 0,5	0,02
	0,5 to 2,0	0,05
	2,0 to 10,0	0,1
	10,0 to 50,0	0,2

7.3.3.4 Calibration curves

Establish the calibration curves using measured ratioed intensities (absolute intensity for phosphorus and arsenic) and corresponding analyte amounts.

Use appropriate spectrometer software for regression calculations and calculate the corresponding correlation coefficients: these coefficients should be, at least, 0,999.

7.3.4 Determination

7.3.4.1 General

The analyses shall be carried out independently, in duplicate.

7.3.4.2 Spectrometric measurement

Carry out measurements on the test solution (7.3.1.3) (7.3.1.2 for phosphorus and arsenic).

Determine the contents directly using the calibration curves (7.3.3.4).

7.4 Method D: Copper-aluminium alloys

7.4.1 Preparation of the test portion solution

7.4.1.1 Test portion

Weigh (1 ± 0,001) g of the test sample.

7.4.1.2 Test portion solution 5 g/l

Transfer the test portion (7.4.1.1) into a 250 ml beaker.

Add 20 ml of hydrochloric acid solution (4.2) and 5 ml of nitric acid (4.3). Cover with a watch glass and heat gently until the sample is completely dissolved, then heat to boiling. Allow to cool.

Rinse the cover and the walls of the beaker with water.

Transfer the dissolved test portion into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1).

Dilute to the mark with water and mix well.

7.4.1.3 Test portion solution 1 g/l

In order to obtain solutions with 1 g/l concentration, pipette exactly 20 ml of the test portion solution (7.4.1.2) into a 100 ml one-mark volumetric flask containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

7.4.2 Check test

Make a preliminary check of the apparatus by preparing a solution of a copper-aluminium alloy reference material or a synthetic sample containing known amounts of the elements listed in Table 4 and carrying out the procedure specified in 7.4.4.

7.4.3 Establishment of the calibration curves

7.4.3.1 Preparation of the calibration solutions

7.4.3.1.1 General

In all cases the acidity of the calibration solutions shall be similar to that of the test portion solutions.

If, for measurement purposes, a blank is required, prepare a solution containing only the same amounts of acids as in the test solution.

7.4.3.1.2 Preparation of the 5 g/l calibration solutions, using mono-elemental solutions

Weigh the quantities of copper (4.7) shown in Table 16 and introduce them into each of a series of 250 ml beakers.

Add 20 ml of hydrochloric acid solution (4.2) and 5 ml of nitric acid (4.3) to each beaker. Cover with a watch glass and heat gently in order to dissolve the copper. Rinse the cover and the walls of the beaker with water and heat to boiling. Allow to cool and transfer each copper solution into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1).

Transfer the volumes of the elemental solutions of Sn, Pb, Zn, Fe, Ni, Mn, Al, Cd and Mg shown in Table 16 to each volumetric flask. Dilute to the mark with water and mix well.

7.4.3.1.3 Preparation of the 1 g/l calibration solutions

Dilute accurately the 5 g/l calibration solutions (7.4.3.1.2) in order to obtain 1 g/l calibration solutions.

Pipette exactly 20 ml of the 5 g/l calibration solutions (7.4.3.1.2) into 100 ml one-mark volumetric flasks each containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

Table 16 — Method D — Composition of the 5 g/l calibration solutions

Element	**Label**			**Drift survey solution** (see 7.4.3.3)
	C1	C3	C6	
Cu (4.7)	756,5 mg 75,65 %	787,4 mg 78,74 %	909,0 mg 90,90 %	805,5 mg 80,55 %
Sn (4.31)	0,5mg [a] 0,05 %	0,2 mg [b] 0,02 %	5,0 mg 5 ml 0,50 %	1,0 mg 1 ml 0,10 %
Pb (4.21)	5,0 mg 5 ml 0,50 %	0,3 mg [c] 0,03 %	0,5mg [d] 0,05 %	1,0 mg 1 ml 0,10 %
Zn (4.33)	10,0 mg 10 ml 1,00 %	0,5 mg [e] 0,05 %	0,3mg [f] 0,03 %	1,0 mg 1 ml 0,10 %
Fe (4.18) and (4.19)	0,5mg [g] 0,05 %	70,0 mg 14 ml (4.18) 7,00 %	5,0 mg 5 ml (4.19) 0,50 %	30,0 mg 6 ml (4.18) 3,00 %
Ni (4.25), (4.26) and (4.27)	80,0 mg 8 ml (4.25) 8,00 %	1,0 mg 1 ml (4.28) 0,10 %	20,0 mg 4 ml (4.26) 2,00 %	40,0 mg 8 ml (4.26) 4,00 %
Mn (4.23) and (4.24)	1,0 mg 1 ml (4.24) 0,10 %	50,0 mg 10 ml (4.23) 5,00 %	0,1 mg [h] 0,01 %	10,0 mg 10 ml (4.24) 1,00 %
Al (4.9)	140,0 mg 14 ml 14,00 %	90,0 mg 9 ml 9,00 %	60,0 mg 6 ml 6,00 %	110,0 mg 11 ml 11,00 %
Cd (4.15)	5,0 mg 5 ml 0,50 %	0,5 mg [i] 0,05 %	0,1 mg [j] 0,01 %	1,0 mg 1 ml 0,10 %
Mg (4.22)	1,5 mg [k] 0,15 %	0,1 mg [l] 0,01 %	0,02 mg [m] 0,002 %	0,5 mg [n] 0,05 %

[a] 5 ml of a 0,1 g/l tin stock solution.
[b] 2 ml of a 0,1 g/l tin stock solution.
[c] 3 ml of a 0,1 g/l lead stock solution.
[d] 5 ml of a 0,1 g/l lead stock solution.
[e] 5 ml of a 0,1 g/l zinc stock solution.
[f] 3 ml of a 0,1 g/l zinc stock solution.
[g] 5 ml of a 0,1 g/l iron stock solution.
[h] 1 ml of a 0,1 g/l manganese stock solution.
[i] 5 ml of a 0,1 g/l cadmium stock solution.
[j] 1 ml of a 0,1 g/l cadmium stock solution.
[k] 15 ml of a 0,1 g/l magnesium stock solution.
[l] 1 ml of a 0,1 g/l magnesium stock solution.
[m] 2 ml of a 0,01 g/l magnesium stock solution.
[n] 5 ml of a 0,1 g/l magnesium stock solution.

7.4.3.2 Adjustment of the apparatus

Start the inductively coupled plasma optical emission spectrometer and let it run for at least half an hour before performing any measurements.

Adjust all the instrumental parameters, each gas (outer, intermediate or central) flow rate, torch position, entrance slits, exit slits, photomultiplier tubes voltages (when appropriate), wavelengths of the analytical lines specified in Table 17, pre-spraying time and integrating time, according to the manufacturer's instructions, while aspirating (for each element) the highest concentration calibration solutions.

Prepare the software for the measurement of the intensity of each analytical line and for the calculation of the corresponding mean value and relative standard deviation.

Each time the internal standard is used (Y line 371,03 nm), prepare the software to calculate the ratio between each analyte intensity and internal standard intensity.

Table 17 — Wavelengths for determinations in copper-aluminium alloys

Element	**Wavelength** nm
Sn	189,90
Pb	220,30
Zn	334,55 [a]
Fe	238,20
Ni	231,60
Mn	257,60
Al	308,20
Cd	266,50
Mg	279,55

[a] 213,80 nm wavelength may be used to determine zinc, provided that interference from copper has been investigated.

7.4.3.3 Spectrometric measurements of the calibration solutions

Carry out the spectrometric measurements of the 1 g/l solutions identified C1, C3, C6 (see Table 16 and 7.4.3.1.3). For magnesium use the 5 g/l calibration solutions (7.4.3.1.2).

A simultaneous spectrometer shall be available for measurements using yttrium as "internal standard" (ratio mode).

For magnesium determination use "absolute intensity" taking into account that "Gaussian" measuring mode may also be recommended.

For each calibration solution (including the "drift survey" solution) at least three integrations are necessary in order to calculate the mean ratioed intensities (mean absolute intensity for magnesium).

In order to control the drift of the spectrometer, analyse the "drift survey" solution (see Table 16 and 7.4.3.1.3) at least once for every five test portion solutions. Maximum drifts allowed are given in Table 18.

Table 18 — Drift survey for copper–aluminium alloys

Element	Concentration range %	Maximum drift %
all	0,1 to 0,5	0,02
	0,5 to 2,0	0,05
	2,0 to 10,0	0,1

7.4.3.4 Calibration curves

Establish the calibration curves using measured ratioed intensities (absolute intensity for magnesium) and corresponding analyte amounts.

Use appropriate spectrometer software for regression calculations and calculate the corresponding correlation coefficients: these coefficients should be, at least, 0,999.

7.4.4 Determination

7.4.4.1 General

The analyses shall be carried out independently, in duplicate.

7.4.4.2 Spectrometric measurement

Carry out measurements on the test solution (7.4.1.3) (7.4.1.2 for magnesium).

Determine the contents directly using the calibration curves (7.4.3.4).

7.5 Method E: Copper-beryllium alloys

7.5.1 Preparation of the test portion solution

7.5.1.1 Test portion

Weigh (1 ± 0,001) g of the test sample.

7.5.1.2 Test portion solution 5 g/l

Transfer the test portion (7.5.1.1) into a 250 ml beaker.

Add 20 ml of hydrochloric acid solution (4.2) and 5 ml of nitric acid (4.3). Cover with a watch glass and heat gently until the sample is completely dissolved, then heat to boiling. Allow to cool. Rinse the cover and the walls of the beaker with water.

Transfer the dissolved test portion into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix well.

7.5.1.3 Test portion solution 1 g/l

In order to obtain solutions with 1 g/l concentration, pipette exactly 20 ml of the test portion solution (7.5.1.2) into a 100 ml one-mark volumetric flask containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

7.5.2 Check test

Make a preliminary check of the apparatus by preparing a solution of a copper-beryllium alloy reference material, or a synthetic sample containing known amounts of the elements listed in Table 5 and carrying out the procedure specified in 7.5.4.

7.5.3 Establishment of the calibration curves

7.5.3.1 Preparation of the calibration solutions

7.5.3.1.1 General

In all cases the acidity of the calibration solutions shall be similar to that of the test portion solutions.

If, for measurement purposes, a blank is required, prepare a solution containing only the same amounts of acids as in the test solution.

7.5.3.1.2 Preparation of the 5 g/l calibration solutions, using mono-elemental solutions

Weigh the quantities of copper (4.7) shown in Table 19 and introduce them into each of a series of 250 ml beakers.

Add 20 ml of hydrochloric acid solution (4.2) and 5 ml of nitric acid (4.3) to each beaker. Cover with a watch glass and heat gently in order to dissolve the copper. Rinse the cover and the walls of the beaker with water and heat to boiling. Allow to cool and transfer each copper solution into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1).

Transfer the volumes of the elemental solutions of Sn, Pb, Zn, Fe, Ni, Mn, Al, Be and Co shown in Table 19 to each volumetric flask. Dilute to the mark with water and mix well.

7.5.3.1.3 Preparation of the 1 g/l calibration solutions

Dilute accurately the 5 g/l calibration solutions (7.5.3.1.2) in order to obtain 1 g/l calibration solutions.

Pipette exactly 20 ml of the 5 g/l calibration solutions (7.5.3.1.2) into 100 ml one-mark volumetric flasks each containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

Table 19 — Method E — Composition of the 5 g/l calibration solutions

Element	**Label**			**Drift survey solution** (see 7.5.3.3)
	B1	B3	B5	
Cu (4.7)	951,3 mg 95,13 %	993,4 mg 99,34 %	928,5 mg 92,85 %	944,5 mg 94,45 %
Sn (4.31)	0,2 mg [a] 0,02 %	0,5 mg [b] 0,05 %	2,0 mg 2 ml 0,20 %	1,0 mg 1 ml 0,10 %
Pb (4.21)	2,0 mg 2 ml 0,20 %	0,4 mg [c] 0,04 %	0,1 mg [d] 0,01 %	1,0 mg 1 ml 0,10 %
Zn (4.33)	0,3 mg [e] 0,03 %	0,5 mg [f] 0,05 %	2,0 mg 2 ml 0,20 %	1,0 mg 1 ml 0,10 %
Fe (4.19)	3,0 mg 3 ml 0,30 %	0,6 mg [g] 0,06 %	0,3 mg [h] 0,03 %	1,0 mg 1 ml 0,10 %
Ni (4.27)	0,4 mg [i] 0,04 %	1,0 mg 1 ml 0,10 %	25,0 mg 25 ml 2,50 %	10,0 mg 10 ml 1,00 %
Mn (4.24)	0,06 mg [j] 0,006 %	0,1 mg [k] 0,01 %	1,5 mg [l] 0,15 %	0,5 mg [m] 0,05 %
Al (4.10)	2,0 mg 2 ml 0,20 %	0,5 mg [n] 0,05 %	0,3 mg [o] 0,03 %	1,0 mg 1 ml 0,10 %
Be (4.13) and (4.14)	0,8 mg [p] 0,08 %	2,0 mg 2 ml (4.14) 0,20 %	40,0 mg 8 ml (4.13) 4,00 %	20,0 mg 4 ml (4.13) 2,00 %
Co (4.16) and (4.17)	40,0 mg 8 ml (4.16) 4,00 %	1,0 mg 1 ml (4.17) 0,10 %	0,3 mg [q] 0,03 %	20,0 mg 4 ml (4.16) 2,00 %

[a] 2 ml of a 0,1 g/l tin stock solution.

[b] 5 ml of a 0,1 g/l tin stock solution.

[c] 4 ml of a 0,1 g/l lead stock solution.

[d] 1 ml of a 0,1 g/l lead stock solution.

[e] 3 ml of a 0,1 g/l zinc stock solution.

[f] 5 ml of a 0,1 g/l zinc stock solution.

[g] 6 ml of a 0,1 g/l iron stock solution.

[h] 3 ml of a 0,1 g/l iron stock solution.

[i] 4 ml of a 0,1 g/l nickel stock solution.

[j] 6 ml of a 0,01 g/l manganese stock solution.

[k] 1 ml of a 0,1 g/l manganese stock solution.

[l] 15 ml of a 0,1 g/l manganese stock solution.

[m] 5 ml of a 0,1 g/l manganese stock solution.

[n] 5 ml of a 0,1 g/l aluminium stock solution.

[o] 3 ml of a 0,1 g/l aluminium stock solution.

[p] 8 ml of a 0,1 g/l beryllium stock solution.

[q] 3 ml of a 0,1 g/l cobalt stock solution.

7.5.3.2 Adjustment of the apparatus

Start the inductively coupled plasma optical emission spectrometer and let it run for at least half an hour before performing any measurements.

Adjust all the instrumental parameters, each gas (outer, intermediate or central) flow rate, torch position, entrance slits, exit slits, photomultiplier tubes voltages (when appropriate), wavelengths of the analytical lines specified in Table 20, pre-spraying time and integrating time, according to the manufacturer's instructions, while aspirating (for each element) the highest concentration calibration solutions.

Prepare the software for the measurement of the intensity of each analytical line and for the calculation of the corresponding mean value and relative standard deviation.

Each time the internal standard is used (Y line 371,03 nm) prepare the software to calculate the ratio between each analyte intensity and internal standard intensity.

Table 20 — Wavelengths for determinations in copper-beryllium alloys

Element	Wavelength nm
Sn	189,90
Pb	220,30
Zn	334,55 [a]
Fe	238,20
Ni	231,60
Mn	257,60
Al	308,20
Be	313,00
Co	228,60

[a] 213,80 nm wavelength may be used to determine zinc, provided that interference from Copper has been investigated.

7.5.3.3 Spectrometric measurements of the calibration solutions

Carry out the spectrometric measurements of the 1 g/l solutions identified B1, B3, B5 (see Table 19 and 7.5.3.1.3). A simultaneous spectrometer shall be available for measurements using yttrium as "internal standard" (ratio mode).

For each calibration solution (including the "drift survey" solution) at least three integrations are necessary in order to calculate the mean ratioed intensities.

In order to control the drift of the spectrometer, analyse the "drift survey" solution (see Table 19 and 7.5.3.1.3) at least once for every five test portion solutions. Maximum drifts allowed are given in Table 21.

Table 21 — Drift survey for copper-beryllium alloys

Element	Concentration range %	Maximum drift %
all	0,1 to 0,5	0,02
	0,5 to 2,0	0,05
	2,0 to 10,0	0,1

7.5.3.4 Calibration curves

Establish the calibration curves using measured mean ratioed intensities and corresponding analyte amounts.

Use appropriate spectrometer software for regression calculations and calculate the corresponding correlation coefficients: these coefficients should be, at least, 0,999.

7.5.4 Determination

7.5.4.1 General

The analyses shall be carried out independently, in duplicate.

7.5.4.2 Spectrometric measurement

Carry out measurements on the test solution (7.5.1.3).

Determine the contents directly using the calibration curves (7.5.3.4).

7.6 Method F: Copper-nickel alloys

7.6.1 Preparation of the test portion solution

7.6.1.1 Test portion

Weigh (1 ± 0,001) g of the test sample.

7.6.1.2 Test portion solution 5 g/l

Transfer the test portion (7.6.1.1) into a 250 ml beaker.

Add 20 ml of hydrochloric acid solution (4.2) and 5 ml of nitric acid (4.3). Cover with a watch glass and heat gently.

In the cases where samples are completely dissolved (absence of free carbon, namely) add about 40 ml of water, then heat to boiling. Allow to cool. Rinse the cover and the walls of the beaker with water. Transfer the dissolved test portion into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix well.

In the cases where a residue is observed (namely, presence of free carbon) add about 40 ml of water, then heat to boiling. Allow to cool. Filter the solution through a dense-texture filter-paper. Transfer the filtrate to a 200 ml one-mark volumetric flask. Rinse the beaker cover, the walls of the beaker and the filter with water. Combine all the washings with the main solution.

Introduce the filter and its contents into a platinum crucible. Heat at 950 °C for 15 min.

Allow to cool, add 1 ml of nitric acid (4.3) and 1 ml of hydrofluoric acid (4.5) to the crucible, heat carefully to dryness. Add to the crucible 10 ml of hydrochloric acid (4.1) and heat very gently until a clear solution is obtained. Combine this solution with the main solution. Dilute to the mark with water and mix well.

7.6.1.3 Test portion solution 1 g/l

In order to obtain solutions with 1 g/l concentration, pipette exactly 20 ml of the test portion solution (7.6.1.2) into a 100 ml one-mark volumetric flask containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

7.6.2 Check test

Make a preliminary check of the apparatus by preparing a solution of a copper-nickel alloy reference material or a synthetic sample containing known amounts of the elements listed in Table 6 and carrying out the procedure specified in 7.6.4.

7.6.3 Establishment of the calibration curves

7.6.3.1 Preparation of the calibration solutions

7.6.3.1.1 General

In all cases the acidity of the calibration solutions shall be similar to that of the test portion solutions.

If, for measurement purposes, a blank is required, prepare a solution containing only the same amounts of acids as in the test solution.

7.6.3.1.2 Preparation of the 5 g/l calibration solutions, using mono-elemental solutions

Weigh the quantities of copper (4.7) shown in Table 22 and introduce them into each of a series of 250 ml beakers.

Add 20 ml of hydrochloric acid solution (4.2) and 5 ml of nitric acid (4.3) to each beaker. Cover with a watch glass and heat gently in order to dissolve the copper. Rinse the cover and the walls of the beaker with water and heat to boiling. Allow to cool and transfer each copper solution into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1).

Transfer the volumes of the elemental solutions of Sn, Pb, Zn, Fe, Ni, Mn and Al shown in Table 22 to each volumetric flask. Dilute to the mark with water and mix well.

7.6.3.1.3 Preparation of the 1 g/l calibration solutions

Dilute accurately the 5 g/l calibration solutions (7.6.3.1.2) in order to obtain 1 g/l calibration solutions.

Pipette exactly 20 ml of the 5 g/l calibration solutions (7.6.3.1.2) into 100 ml one-mark volumetric flasks each containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

Table 22 — Method F — Composition of the 5 g/l calibration solutions

Element	**Label**			**Drift survey solution** (see 7.6.3.3)
	N1	N3	N5	
Cu (4.7)	593,5 mg 59,35 %	792,3 mg 79,23 %	879,1 mg 87,91 %	694,0 mg 69,40 %
Sn (4.31)	5,0 mg 5 ml 0,50 %	0,6 mg [a] 0,06 %	0,3 mg [b] 0,03 %	2,0 mg 2 ml 0,20 %
Pb (4.21)	0,3mg [c] 0,03 %	0,6 mg [d] 0,06 %	5,0 mg 5 ml 0,50 %	2,0 mg 2 ml 0,20 %
Zn (4.33)	20,0 mg 20 ml 2,00 %	1,0 mg 1 ml 0,10 %	0,4 mg [e] 0,04 %	10,0 mg 10 ml 1,00 %
Fe (4.18) and (4.19)	1,0 mg 1 ml (4.19) 0,10 %	4,0 mg 4 ml (4.19) 0,40 %	40,0 mg 8 ml (4.18) 4,00 %	20,0 mg 4 ml (4.18) 2,00 %
Ni (4.25)	350,0 mg 35 ml 35,00 %	200,0 mg 20 ml 20,00 %	70,0 mg 7 ml 7,00 %	250,0 mg 25 ml 25,00 %
Mn (4.23) and (4.24)	30,0 mg 6 ml (4.23) 3,00 %	1,0 mg 1 ml (4.24) 0,10 %	0,2 mg [f] 0,02 %	20,0 mg 4 ml (4.23) 2,00 %
Al (4.10)	0,2 mg [g] 0,02 %	0,5 mg [h] 0,05 %	5,0 mg 5 ml 0,50 %	2,0 mg 2 ml 0,20 %

[a] 6 ml of a 0,1 g/l tin stock solution.

[b] 3 ml of a 0,1 g/l tin stock solution.

[c] 3 ml of a 0,1 g/l lead stock solution.

[d] 6 ml of a 0,1 g/l lead stock solution.

[e] 4 ml of a 0,1 g/l zinc stock solution.

[f] 2 ml of a 0,1 g/l manganese stock solution.

[g] 2 ml of a 0,1 g/l aluminium stock solution.

[h] 5 ml of a 0,1 g/l aluminium stock solution.

7.6.3.2 Adjustment of the apparatus

Start the inductively coupled plasma optical emission spectrometer and let it run for at least half an hour before performing any measurements.

Adjust all the instrumental parameters, each gas (outer, intermediate or central) flow rate, torch position, entrance slits, exit slits, photomultiplier tubes voltages (when appropriate), wavelengths of the analytical lines specified in Table 23, pre-spraying time and integrating time, according to the manufacturer's instructions, while aspirating (for each element) the highest concentration calibration solutions.

Prepare the software for the measurement of the intensity of each analytical line and for the calculation of the corresponding mean value and relative standard deviation.

Each time the internal standard is used (Y line 371,03 nm), prepare the software to calculate the ratio between each analyte intensity and internal standard intensity.

Table 23 — Wavelengths for determination in copper-nickel alloys

Element	Wavelength nm
Sn	189,90
Pb	220,30
Zn	334,55 [a]
Fe	238,20
Ni	231,60
Mn	257,60
Al	308,20

[a] 213,80 nm wavelength may be used to determine zinc, provided that interference from copper has been investigated.

7.6.3.3 Spectrometric measurements of the calibration solutions

Carry out the spectrometric measurements of the 1 g/l solutions identified N1, N3, N5 (see Table 22 and 7.6.3.1.3). A simultaneous spectrometer shall be available for measurements using yttrium as "internal standard" (ratio mode).

For each calibration solution (including the "drift survey" solution) at least three integrations are necessary in order to calculate the mean ratioed intensities.

In order to control the drift of the spectrometer, analyse the "drift survey" solution (see Table 22 and 7.6.3.1.3) at least once for every five test portions solutions. Maximum drifts allowed are given in Table 24.

Table 24 — Method F — Drift survey for copper-nickel alloys

Element	Concentration range %	Maximum drift %
all	0,1 to 0,5	0,02
	0,5 to 2,0	0,05
	2,0 to 10,0	0,1
	10,0 to 50,0	0,2

7.6.3.4 Calibration curves

Establish the calibration curves using measured ratioed intensities and corresponding analyte amounts.

Use appropriate spectrometer software for regression calculations and calculate the corresponding correlation coefficients: these coefficients should be, at least, 0,999.

7.6.4 Determination

7.6.4.1 General

The analyses shall be carried out independently, in duplicate.

7.6.4.2 Spectrometric measurement

Carry out measurements on the test solution (7.6.1.3).

Determine the contents directly using the calibration curves (7.6.3.4).

8 Expression of results

The calibration curves are drawn from the measurements carried out either in the "ratio" or in "intensity" mode according to the concentrations of the various elements. On the calibration curves, read the contents, expressed as mass fraction in percent of the elements determined in the test sample.

9 Precision

9.1 Method A — Coppers

Five laboratories co-operated in validating this method and obtained the results summarized in Table 25.

Table 25 — Method A — Statistical information

Element	Reference value %	Found %	Repeatability r	Reproducibility R
Sn	0,024 7	0,024 2	0,002 8	0,003 0
Pb	0,023 6	0,023 9	0,002 8	0,003 4
Zn	0,021 7	0,021 6	0,001 4	0,001 5
Fe	0,023 5	0,023 1	0,001 8	0,003 7
Ni	0,020 9	0,020 8	0,001 2	0,001 5
Mn	0,020 6	0,020 2	0,000 5	0,001 1
P	0,020 3	0,020 5	0,001 7	0,002 3
Co	0,020 8	0,020 8	0,001 0	0,001 3
Cd	0,018 6	0,018 6	0,000 8	0,001 3

9.2 Method B — Copper-zinc alloys

Eight laboratories co-operated in validating this method and obtained the results summarized in Table 26.

Table 26 — Method B — Statistical information

Element	Reference value %	Found %	Repeatability r	Reproducibility R
Sn	0,209	0,208 0	0,015 0	0,022 4
	0,27	0,254 0	0,014 2	0,016 7
	0,70	0,692 4	0,040 1	0,051 5
Pb	0,35	0,353 0	0,009 9	0,024 2
	2,24	2,253 1	0,055 0	0,154 8
	2,90	2,867 6	0,055 6	0,164 2
Zn	35,8	35,983 4	0,620 9	0,648 0
	38,02	38,440 0	0,676 6	1,031 9
Fe	0,15	0,146 0	0,011 6	0,025 6
	0,207	0,206 7	0,014 6	0,026 6
	1,02	1,013 0	0,038 7	0,100 2
Ni	0,105	0,102 3	0,003 9	0,010 3
	0,13	0,126 5	0,004 6	0,013 9
	0,56	0,554 5	0,019 6	0,043 9
Mn	0,022 2	0,020 3	0,002 2	0,006 6
	0,86	0,878 4	0,035 9	0,075 3
Al	0,027	0,026 7	0,004 8	0,007 3
	2,22	2,238 7	0,028 0	0,078 3
As	0,023 1	0,022 5	0,001 5	0,005 1
NOTE For Zinc, it was decided to remove the precision data corresponding to the sample BCS 385 (38,5 % content level), as it was assumed that this sample may not be enough homogeneous regarding to its Zinc content				

9.3 Method C — Copper-tin alloys

Seven laboratories co-operated in validating this method and obtained the results summarized in Table 27.

Table 27 — Method C — Statistical information

Element	Reference value %	Found %	Repeatability *r*	Reproducibility *R*
Sn	5,21	5,211 1	0,059 0	0,081 1
	7,27	7,290 2	0,103 3	0,145 0
Pb	3,15	3,163 3	0,090 6	0,119 8
	5,11	5,210 8	0,062 6	0,131 4
Zn	3,47	3,528 3	0,135 4	$\leq r$
	5,07	5,156 7	0,094 3	0,124 5
Fe	0,056	0,051 7	0,002 0	0,006 4
Ni	1,10	1,100 1	0,012 7	0,030 6
	1,30	1,309 8	0,024 0	0,040 9
Mn	0,013 0	0,012 3	0,000 5	0,002 0
P	0,022	0,022 5	0,006 0	0,007 0
	0,090	0,089 2	0,009 0	0,010 6
Sb	0,23	0,236 6	0,014 6	0,018 1
As	0,130 0	0,137 3	0,011 0	0,015 5

9.4 Method D — Copper-aluminium alloys

Six laboratories co-operated in validating this method and obtained the results summarized in Table 28.

Table 28 — Method D — Statistical information

Element	Reference value %	Found %	Repeatability *r*	Reproducibility *R*
Sn	0,189	0,187 9	0,005 8	0,009 7
	0,199	0,200 3	0,011 2	0,016 9
	0,256	0,248 1	0,007 1	0,021 7
Pb	0,105	0,108 2	0,005 1	0,021 2
	0,149	0,153 0	0,007 7	0,013 4
	0,171	0,184 0	0,010 5	0,020 2
	0,229	0,230 2	0,011 6	0,012 7
Zn	0,314	0,327 6	0,012 1	0,027 9
	0,425	0,439 0	0,015 6	0,025 6
	0,501	0,527 3	0,023 6	0,038 1
	0,533	0,550 3	0,022 9	0,052 2
Fe	0,790	0,796 1	0,023 3	0,054 2
	0,806	0,813 2	0,017 3	0,061 6
	3,979	4,089 9	0,119 2	0,182 1
	6,085	6,074 6	0,098 6	0,338 0
Ni	0,819	0,824 1	0,017 5	0,041 1
	1,178	1,206 4	0,020 3	0,044 0
	3,865	3,864 9	0,083 1	0,202 1
	5,429	5,501 4	0,103 2	0,130 6
Mn	0,057	0,061 6	0,003 6	0,011 8
	1,84	1,864 9	0,019 2	0,093 1
Al	7,164	7,053 3	0,101 9	0,277 0
	10,956	10,946 4	0,147 2	0,199 2
	11,532	11,519 3	0,138 5	0,150 6
Cd	0,050 0	0,050 8	0,002 2	0,004 5
	0,069 5	0,070 2	0,002 8	0,005 8

NOTE 1 For Tin, it was decided to remove the precision data corresponding to a reference material labelled CA 35 (0,27 % content level), as it was assumed that this sample may be not enough homogeneous regarding to its Tin content.

NOTE 2 For Manganese, it was decided to remove the precision data corresponding to a reference material labelled CA 35 (1,56 % content level), as it was assumed that this sample may be not enough homogeneous regarding to its Manganese content.

NOTE 3 For Aluminium, it was decided to remove the precision data corresponding to a reference material labelled CA 20 (7,985 % content level), as it was assumed that this sample may be not enough homogeneous regarding to its Aluminium content.

9.5 Method E — Copper-beryllium alloys

Five laboratories co-operated in validating this method and obtained the results summarized in Table 29.

Table 29 — Method E — Statistical information

Element	Reference value %	Found %	Repeatability r	Reproducibility R
Sn	0,022	0,021 5	0,001 7	0,003 1
	0,100	0,097 4	0,003 5	0,009 3
Pb	0,023 4	0,023 2	0,002 5	0,002 6
	0,053	0,053 8	0,002 5	0,003 2
Zn	0,056	0,055 4	0,003 9	$\leq r$
	0,070	0,068 8	0,004 2	0,011 7
Fe	0,072	0,070 0	0,002 6	0,005 9
	0,165	0,163 4	0,004 1	0,011 7
	0,203	0,207 7	0,008 8	0,017 9
Ni	0,038 3	0,037 9	0,002 2	0,004 0
	0,049	0,048 4	0,002 0	0,003 9
	0,203	0,204 7	0,004 5	0,013 9
Mn	0,007 7	0,008 6	0,000 3	0,000 5
	0,019	0,019 7	0,000 3	0,000 6
Al	0,027	0,029 7	0,005 4	$\leq r$
	0,044 0	0,043 4	0,005 8	$\leq r$
	0,096	0,092 8	0,004 2	0,005 4
Be	0,18	0,159 3	0,003 6	0,003 9
	1,58	1,559 3	0,029 2	0,061 8
	2,92	2,873 2	0,060 8	0,127 3
Co	0,246	0,241 3	0,006 8	0,012 8
	0,64	0,635 7	0,009 8	0,024 0
	0,99	0,962 8	0,028 2	0,041 1

9.6 Method F — Copper-nickel alloys

Seven laboratories co-operated in validating this method and obtained the results summarized in Table 30.

Table 30 — Method F — Statistical information

Element	Reference value %	Found %	Repeatability *r*	Reproducibility *R*
Pb	0,055 3	0,056 7	0,006 5	0,008 5
Zn	0,474 7	0,474 4	0,011 3	0,040 5
Fe	1,532	1,519 4	0,043 8	0,072 9
Ni	7,775	7,871 7	0,144 4	0,183 1
	30,200	29,881 3	0,639 9	0,680 7
Mn	0,880	0,882 8	0,028 8	0,085 2
	2,7	2,822 3	0,078 9	0,192 9

NOTE For Iron, it was decided to remove the precision data corresponding to a reference material labelled NK (2,20 % content level), as it was assumed that this sample may be not enough homogeneous regarding to its Iron content.

10 Test report

The test report shall contain the following information:

a) identification of the test sample;

b) reference to this European Standard (EN 15605);

c) 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.

Annex A
(informative)

Optical emission spectrometer (OES) — Suggested performance criteria to be checked

A.1 Practical resolution of the sequential spectrometer

The resolution of a spectrometer can be defined as the wavelength difference, $\Delta\lambda$, between two lines which can still just be observed separately. In practice the parameter FWHM (Full Width at Half Maximum) is used as a resolution measure.

Ideally, the resolution should be of the same order as the physical line width in inductively coupled plasma optical emission spectrometer spectra, i.e. 2 pm to 5 pm (1 pm = 10^{-12} m). In practice, however, the observed width of the emission lines and, consequently, the resolution will often be determined by the bandwidth (r) of the spectrometer being used. As long as broadening resulting from aberrations can be neglected, this bandwidth is given by:

$$r = \text{FWHM} = (d\lambda/dx)\,(w_\text{i} + w_\text{u})/2 \tag{A.1}$$

where

w_i and w_u are the widths of the entrance slit and exit slit respectively;

$d\lambda/dx$ is the reciprocal linear dispersion which is given by:

$$d\lambda/dx = d(\cos \beta)/nL$$

where

L is the focal length of the spectrometer;

n is the order number;

d is the reciprocal of the groove density in the grating;

β is the diffraction angle.

Normally, commercial spectrometers present resolutions in the range of 4 pm to 30 pm. A good resolution is of great importance to cope with the frequent spectral interferences, which occur in inductively coupled plasma optical emission spectrometer. Since a line with a wavelength in the second order will have the same diffraction angle β as a line with a wavelength 2λ in the first order, a spectrometer should either have an order sorting possibility or an optical filter to avoid an order overlap.

A.2 Short and long term stability

The evaluation of the short-term stability consists on calculating the repeatability standard deviation of the inductively coupled plasma optical emission spectrometer.

A series of ten consecutive intensity measurements of the (high) multi-element calibration solution is carried out using the typical integration time for the system. The average I_avrg and standard deviation S_I of the ten measurements are calculated in addition to the relative standard deviation RSD$_\text{I}$, according to the following equation:

$$\mathrm{RSD_I} = (S_\mathrm{I}/I_\mathrm{avrg}) \times 100\ (\%) \qquad \text{(A.2)}$$

In inductively coupled plasma optical emission spectrometer for solutions with concentrations of at least twice the background-equivalent concentration (BEC), $\mathrm{RSD_I}$-values between 0,3 % and 1,0 % are generally accepted. Multi-element calibration solutions may be used for measurement at various analytical lines present in simultaneous optics.

Long term stability assessment is a measurement of the instrument drift. This is only required if the inductively coupled plasma optical emission spectrometer is set up to work for long intervals of time. It consists of carrying out the same short term stability tests at specific intervals of time, 15 min to 1 h, and plotting the deviation of the average found for every short term test against time. Deviations of more than 2 %/h should not be accepted. In case the instrument is not able to perform better, during analysis the control calibration solution should be measured more often and the mean results of the test sample solutions should be recalculated by interpolation between two consecutive control calibration solutions.

A.3 Evaluating the background equivalent concentration (BEC)

The BEC is used as a measure for instrument sensitivity. (Since the analyte signal has usually a relatively high background, its correction by the background intensity is recommended.). It is calculated using the following equation:

$$\mathrm{BEC} = \frac{I_{BG}}{I_{net}} \times C_{Analyte} \qquad \text{(A.3)}$$

where

I_BG is the intensity of the background;

I_net is the intensity of the analyte (overall intensity minus intensity of the background);

C_Analyte is the concentration of analyte that yields I_net.

The BEC values for the elements to be analysed can be found in wavelength tables (usually part of the instrument software). The BEC is the better the smaller its numerical value is.

A.4 Evaluating the limit of detection

To the blank test solution all the matrix determining components are added and respectively as much of the element concerned for solutions to be obtained with concentration levels of 0,5, 1,0, 1,5, 2,0, 3,0 and 5,0 times the estimated limit of detection.

All solutions are measured at least ten times (using the appropriate number of replicates).

The RSDs obtained are plotted against concentration and the graph obtained is used to read off the limit of detection.

This limit is the concentration at which the RSD is 33 %.

NOTE The limit of quantification is the concentration at which the RSD is 10 %.

Annex B
(informative)

Inductively coupled plasma emission spectrometry method for the analysis of Copper-tin-lead alloys

B.1 Introduction

This informative annex specifies an inductively coupled plasma emission spectrometry method for the determination of alloying elements and impurities in Copper-tin-lead alloys in the form of unwrought, wrought and cast products.

This method is applicable to the elements listed in Table B.1 within the composition ranges shown:

Table B.1 – 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 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.

B.2 Principle

Dissolution of a test portion with hydrochloric and nitric acids. 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.

B.3 Reagents

Refer to Clause 4 of this standard.

B.4 Apparatus

Refer to Clause 5 of this standard.

B.5 Sampling

Refer to Clause 6 of this standard.

B.6 Procedure

B.6.1 Preparation of the test portion solution

B.6.1.1 Test portion

Weigh (0,500 ± 0,001) g of the sample.

B.6.1.2 Test portion solution 2,5 g/l

Transfer the test portion (7.7.1.1) into a 250 ml beaker.

Add 10 ml of hydrochloric acid (4.2) and 2,5 ml of nitric acid (4.3). Cover with a watch glass and dissolve the sample at room temperature. When the sample is completely dissolved add about 40 ml of water and heat to boiling. If a $PbCl_2$ precipitate is present, heat the solution to just below the boiling point and maintain this temperature as long as necessary to dissolve the precipitate. Allow to cool.

Rinse the cover and the walls of the beaker with water.

Transfer the dissolved test portion into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1).

Dilute to the mark with water and mix well.

B.6.1.3 Test portion solution 0,5 g/l

In order to obtain solutions with 0,5 g/l concentration, pipette exactly 20 ml of the test portion solution (B.6.1.2) into a 100 ml one-mark volumetric flask containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

B.6.2 Check test

Make a preliminary check of the apparatus by preparing a solution of a leaded tin bronze reference material or a synthetic sample containing known amounts of the elements listed in Table B.1 and carrying out the procedure specified in B.6.4.

B.6.3 Establishment of the calibration curves

B.6.3.1 Preparation of the calibration solutions

B.6.3.1.1 General

In all cases the acidity of the calibration solutions shall be similar to those of the test portion solutions.

B.6.3.1.2 Preparation of the 2,5 g/l calibration solutions, using mono-elemental solutions

Weigh the quantities of copper (4.7) shown in Table B.2 and introduce them into each of a series of 250 ml beakers.

Add 10 ml of hydrochloric acid solution (4.2) and 2,5 ml of nitric acid (4.3) to each beaker. Cover with a watch glass and heat gently in order to dissolve the copper. Rinse the cover and the walls of the beaker with water and heat to boiling. Allow to cool and transfer each copper solution into a 200 ml one-mark volumetric flask containing 10 ml of hydrochloric acid (4.1).

Add about 40 ml of water and then transfer the volumes of the elemental solutions of Pb at first, Sn, Zn, Fe, Ni, Mn, P, Sb and Al, shown in Table B.2 to each volumetric flask. Dilute to the mark with water and mix well.

B.6.3.1.3 Preparation of the 0,5 g/l calibration solutions

Dilute accurately the 2,5 g/l calibration solutions (B.6.3.1.2) in order to obtain 0,5 g/l calibration solutions.

Pipette exactly 20 ml of the 2,5 g/l calibration solutions (B.6.3.1.2) into a 100 ml one-mark volumetric flask containing 5 ml of hydrochloric acid (4.1) and 5 ml of yttrium solution (4.34). Dilute to the mark with water and mix well.

B.6.3.2 Adjustment of the apparatus

Start the inductively coupled plasma optical emission spectrometer and let it run for at least half an hour before performing any measurements.

Adjust all the instrumental parameters, each gas flow rate (outer, intermediate or central), torch position, entrance slits, exit slits, photomultiplier tubes voltages (when appropriate), wavelengths of the analytical lines specified in Table B.3, pre-spraying time and integrating time, according to the manufacturer's instructions, while aspirating (for each element) the highest concentration calibration solutions.

Prepare the software for the measurement of the intensity of each analytical line and for the calculation of the corresponding mean value and relative standard deviation.

Each time the internal standard is used (Y line 371,03 nm) prepare the software to calculate the ratio between each analyte intensity and internal standard intensity.

Table B.2 — Composition of the 2,5 g/l calibration solutions

Element	Label			Drift survey solution (see B.6.3.3)
	E9	E10	E11	
Cu (4.7)	378,4 mg 75,68 %	357,7 mg 71,54 %	359,45 mg 71,89 %	356,5 mg 71,30 %
Sn (4.30)	45,0 mg 9 ml 9,00 %	15,0 mg 3 ml 3,00 %	60,0 mg 12 ml 12,00 %	30,0 mg 6 ml 6,00 %
Pb (4.20)	70,0 mg 14 ml 14,00 %	125,0 mg 25 ml 25,00 %	40,0 mg 8 ml 8,00 %	100,0 mg 20 ml 20,00 %
Zn (4.33)	2,50 mg 2,5 ml 0,50 %	0,25 mg [a] 0,05 %	15,0 mg 15 ml 3,00 %	5,0 mg 5 ml 1,00 %
Fe (4.19)	0,25 mg [b] 0,05 %	0,05 mg [c] 0,01 %	2,5 mg 2,5 ml 0,50 %	1,0 mg 1 ml 0,20 %
Ni (4.27)	2,50 mg 2,5 ml 0,50 %	0,25 mg [d] 0,05 %	15,0 mg 15 ml 3,00 %	5,0 mg 5 ml 1,00 %
Mn (4.24)	0,25 mg [e] 0,05 %	1,5 mg [f] 0,30 %	0,05 mg [g] 0,01 %	0,50 mg [h] 0,10 %
P (4.28)	0,30 mg [i] 0,06 %	0,1 mg [j] 0,02 %	2,0 mg 2 ml 0,40 %	0,50 mg [k] 0,10 %
Sb (4.11)	0,4 mg [l] 0,08 %	0,1 mg [m] 0,02 %	4,0 mg 4 ml 0,80 %	1,0 mg 1 ml 0,20 %
Al (4.10)	0,4 mg [n] 0,08 %	0,05 mg [o] 0,01 %	2,0 mg 2 ml 0,40 %	0,50 mg [p] 0,10 %

[a] 2,5 ml of a 0,1 g/l zinc stock solution.

[b] 5 ml of a 0,05 g/l iron stock solution.

[c] 1 ml of a 0,05 g/l iron stock solution.

[d] 2,5 ml of a 0,1 g/l nickel stock solution.

[e] 2,5 ml of a 0,1 g/l manganese stock solution.

[f] 15 ml of a 0,1 g/l manganese stock solution.

[g] 5 ml of a 0,01 g/l manganese stock solution.

[h] 5 ml of a 0,1 g/l manganese stock solution.

[i] 3 ml of a 0,1 g/l phosphorus stock solution.

[j] 1 ml of a 0,1 g/l phosphorus stock solution.

[k] 5 ml of a 0,1 g/l phosphorus stock solution.

[l] 4 ml of a 0,1 g/l antimony stock solution.

[m] 1 ml of a 0,1 g/l antimony stock solution.

[n] 4 ml of a 0,1 g/l aluminium stock solution.

[o] 5 ml of a 0,01 g/l aluminium stock solution.

[p] 5 ml of a 0,1 g/l aluminium stock solution.

Table B.3 — Wavelengths for determination in copper-tin-lead alloys

Element	Wavelength nm
Sn	189,90
Pb	220,30
Zn	334,55 [a]
Fe	238,20
Ni	231,60
Mn	257,60
P	178,23
Al	308,20
Sb	206,80

[a] 213,80 nm wavelength may be used to determine zinc, provided that interference from copper has been investigated.

B.6.3.3 Spectrometric measurements of the calibration solutions

Carry out the spectrometric measurements of the 0,5 g/l solutions identified E9, E10, E11 (see Table B.2 and B.6.3.1.3). For phosphorus use the 2,5 g/l calibration solutions (B.6.3.1.2). A simultaneous spectrometer shall be available for measurements using yttrium as "internal standard" (ratio mode).

For phosphorus determination use "absolute intensity" taking into account that "Gaussian" measuring mode may also be recommended.

For each calibration solution (including the "drift survey" solution) at least three integrations are necessary in order to calculate the mean ratioed intensities (mean absolute intensity for phosphorus).

In order to control the drift of the spectrometer, analyse the "drift survey" solution (see Table B.2 and B.6.3.1.3) at least once for every five test portion solutions. Maximum drifts allowed are given in Table B.4.

Table B.4 — Drift survey for copper-tin-lead alloys

Element	Concentration range %	Maximum drift %
all	0,1 to 0,5	0,02
	0,5 to 2,0	0,05
	2,0 to 10,0	0,1
	10,0 to 50,0	0,2

B.6.3.4 Calibration curves

Establish the calibration curves using measured ratioed intensities (absolute intensity for phosphorus) and corresponding analyte amounts.

Use appropriate spectrometer software for regression calculations and calculate the corresponding correlation coefficients: these coefficients should be, at least, 0,999.

B.6.4 Determination

B.6.4.1 General

The analyses shall be carried out independently, in duplicate.

B.6.4.2 Spectrometric measurement

Carry out measurements on the test solution (B.6.1.3) (B.6.1.2 for phosphorus).

Determine the contents directly using the calibration curves (B.6.3.4).

B.7 Expression of results

The calibration curves are drawn from the measurements carried out either in the "ratio" or in "intensity" mode according to the concentrations of the various elements. On the calibration curves, read the contents, expressed as mass fraction in percent of the elements determined in the test sample.

B.8 Precision

Six laboratories co-operated in validating this method and obtained the results summarized in Table B.5.

Table B.5 — Statistical information

Element	Reference value %	Found %	Repeatability r	Reproducibility R
Sn	9,35	9,316 9	0,227 7	0,332 5
Pb	9,25	9,093 5	0,157 0	0,296 4
Zn	0,13	0,138 8	0,011 0	0,044 4
Ni	0,28	0,276 8	0,012 2	0,013 8
P	0,056	0,053 8	0,012 5	0,017 2
Sb	0,18	0,187 4	0,016 3	0,019 9

NOTE As stated in the Scope, the precision criteria concerning this method do not reach the suitable level, namely for zinc and for phosphorus.

B.9 Test report

Refer to Clause 10 of this standard.

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