

BS EN 15079:2015



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Copper and copper alloys — Analysis by spark optical emission spectrometry (S-OES)

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National foreword

This British Standard is the UK implementation of EN 15079:2015. It supersedes BS EN 15079:2007 which is withdrawn.

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

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

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Copper and copper alloys - Analysis by spark optical emission spectrometry (S-OES)

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d'émission optique à étincelles (SEO-E)

Kupfer und Kupferlegierungen - Analyse durch optische
Emissionsspektrometrie mit Funkenanregung (F-OES)

This European Standard was approved by CEN on 24 April 2015.

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Foreword

This document (EN 15079:2015) 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of an identical text or by endorsement, at the latest by November 2015, and conflicting national standards shall be withdrawn at the latest by November 2015.

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 EN 15079:2007.

Within its programme of work, Technical Committee CEN/TC 133 requested CEN/TC 133/WG 10 "Methods of analysis" to revise this standard:

EN 15079:2007, *Copper and copper alloys — Analysis by spark source optical emission spectrometry (S-OES)*.

In comparison with EN 15079:2007, the following changes were made:

- a) Definitions 3.1 to 3.5 have been improved;
- b) 7.2.2 Calibration has been modified.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard specifies a routine method for the analysis of copper and copper alloys by spark optical emission spectrometry (S-OES). The method is applicable to all elements except copper commonly present in copper and copper alloys as impurities or minor or main constituents, which can be determined by S-OES.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1811-1, *Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 1: Sampling of cast unwrought products*

ISO 1811-2, *Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 2: Sampling of wrought products and castings*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 reference material

RM
material, sufficiently homogeneous and stable with respect to one or more specified properties which has been established to be fit for its intended use in a measurement process

[SOURCE: ISO GUIDE 30:1992/Amd.1:2008, definition 2.1]

3.2 certified reference material

CRM
reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate, that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

[SOURCE: ISO GUIDE 30:1992/Amd.1:2008, definition 2.2]

3.3 test sample

representative quantity of material for testing purposes

3.4 drift control samples

series of homogeneous materials that contain all the elements which have been calibrated and that cover the low, mid and high points of the calibration range for each element, used to detect variations over time in these points

Note 1 to entry: Drift control samples can also be used for statistical process control (SPC) of the instrument.

3.5

recalibration samples

samples at both low and high points of the calibration ranges used to recalibrate the spectrometer

Note 1 to entry: These samples are measured during the calibration procedure and the intensities obtained are stored in the computer according to the manufacturer's instructions.

Note 2 to entry: No chemical analyses are necessary, but the homogeneity of these samples should be carefully evaluated.

3.6

quality control sample

sample with known composition which is analysed in the same way as the test sample to check the trueness of the analytical results

4 Principle

Measurement of the intensity of the radiation, whose wavelength is characteristic of each element, generated by a spark resulting from the application of an electrical discharge between the sample, as one electrode, and an inert counter-electrode.

Concentrations of elements are determined by relating the measured intensities of test samples to calibration curves prepared from reference materials.

5 Apparatus

5.1 Optical emission spectrometer

Spectrometer with spark source capable of measuring the intensities of the optical radiation emitted at specific wavelengths by the elements present in the material.

The wavelengths generally used are given in Annex A.

5.2 Apparatus for sample surface preparation

The most common surface preparation techniques for copper and copper alloys are turning and milling or grinding for copper, provided that the composition of the surface shall not be influenced.

The lathe, milling machine or any other machines used for surface preparation shall be able to produce a surface that conforms to the requirements of 7.1.

WARNING — The appropriate safety recommendations for the use of mechanical apparatus shall be observed. These operations shall be carried out only by properly trained personnel wearing appropriate personal protective equipment.

6 Sampling

Sampling shall be carried out in accordance with ISO 1811-1 or ISO 1811-2, as appropriate.

The sample needs to be sufficiently homogeneous with regard to the spark impact. The measuring surface should be free of defects.

7 Procedure

7.1 Surface preparation

7.1.1 The surface of the sample shall be prepared to a finish that is sufficiently flat and smooth in order to tighten the sample chamber, and shall be reasonably free from contaminants, pores, cracks, inclusions and shrinkage cavities which might otherwise affect analytical results.

In order to avoid variation of results due to the influence of surface finish, the same method of preparation shall be used for all samples.

To avoid cross-contamination between different sample materials, for example pure copper and copper alloys, all relevant components of the machine shall be thoroughly cleaned before use or separate tools shall be used.

Once the surface has been prepared, avoid any contamination, for example fingerprints.

Measurements shall be carried out soon after surface preparation. Samples should be stored, until the time of later measurement, in a desiccator.

7.1.2 The turning, milling or grinding shall be carried out at a suitable speed to avoid undue heating of the sample surface, which might otherwise lead to bias in analysis.

Any lubricants (e.g. propanol) used shall be selected to ensure that they do not affect the analytical result.

7.2 Calibration procedures

7.2.1 General

7.2.1.1 Calibration process

The calibration process is subdivided into calibration and drift compensation by recalibration.

7.2.1.2 Range of calibration

The range of calibration for an element shall extend well below the minimum content reported in the list of alloys composition and above the maximum content reported in the same list, taking into account that the lowest limit should be at least three times the detection limit.

7.2.1.3 Number of sparks on calibration samples

The number of sparks carried out on each reference material for calibration shall be not less than four. The spark areas shall be distributed over the prepared surface. Centre and border of the sample have to be avoided. All measurements shall be examined; if any measurement is obviously defective, further sparks shall be carried out to obtain the minimum four acceptable measurements. The average of the four acceptable measurements is used for calibration.

The influence of temperature is very important for direct measurement methods. The sample should be cooled to ambient temperature between each spark.

7.2.2 Calibration

The calibration of the spectrometer is carried out by using a series of certified reference materials which have the same or at least similar matrix and metallurgical structure as the samples to be analysed, in order to calculate the calibration functions from which the analysis of test samples can be obtained. The content range of the certified reference materials used shall cover that of all the samples to be analysed within each specific analytical program. For each element in each reference material the mean intensity is correlated to the corresponding certified content and a regression is calculated.

The calibration functions are usually stored within a computer, connected to the spectrometer. These calibration functions are 1st or 2nd degree mathematical equations.

The calibration is normally done when the apparatus is installed. The calibration shall be in accordance with the spectrometer manufacturer's instruction manual, using the appropriate certified reference materials, if available. If no certified reference materials are available, reference materials with an accurate analysis shall be used.

The trueness of the analytical procedure is checked by measuring a set of certified reference materials or — if not available — a set of reference materials not used in the calibration. These reference materials shall cover at least the low, mid and high points of the calibration range for each element.

7.2.3 Recalibration

Drifts of the spectrometer readings shall be corrected using a recalibration procedure as described in the manufacturer's instruction manual. Recalibrations can be done either for all analytical channels (global recalibration), or only for individual analytical channels (selective recalibration).

Recalibration can either be done periodically or due to a deviation from statistical process control (SPC) limits (see 7.3.4). When a periodical recalibration procedure is used, the period depends on the stability of the spectrometer and has to be established by a stability check of the spectrometer. The stability check shall be repeated at appropriate intervals.

NOTE The same set of check samples can be used both for drift control and for statistical process control of the spectrometer.

After recalibration, a set of reference materials not used in the recalibration procedure, shall be measured.

7.2.4 Type recalibration

Type recalibration offers a further possibility of correcting instrument drift and, in addition, for matrix influences. Here, one or two reference samples of the same material type and of comparable composition are analysed, together with the test sample. Subsequently, by means of linear correction, the contents of the test sample are related to the certified values of the reference sample(s).

NOTE This practice cannot be employed if the calibration function is a 2nd degree one, unless if the element to be calibrated has a content very close to this of the type calibration sample.

7.3 Analysis

7.3.1 Preparation of analytical programs

Due to the fact that programs shall be prepared matrix by matrix, the way in which measurements are taken into account shall be specified. Analytical programs can be prepared either by measuring only intensities of all the elements concerned, or by taking into account the ratios between the measured intensity for each element and the intensity corresponding to the "matrix element" taken as internal standard.

In principle, intensity measurements are used for trace analysis purposes, whilst rationed measurements are taken for the determination of other levels of content.

During the preparation of each analytical program, the possible optical interferences and/or the inter-elements effects shall also be carefully investigated and suitable corrections shall be made.

Normal mode and virtual mode represent the usual ways to treat the spectrometer outputs.

Normal mode: single comparison of the intensities or of the rationed intensities to the corresponding "calibration functions". This procedure is currently used in cases where the concentration of the element taken as "internal standard" remains constant for all the CRM used for calibration and for all the unknown samples.

Virtual mode: mathematical treatment of the results in order to take into account possible important variations of the concentration of the element chosen as "internal standard" (the copper content in brasses can vary from about 58 % to 85 %, for example). The intensity of the element taken as "internal standard" is measured but the corresponding concentration is calculated assuming that it is the difference from "100 % minus the sum of the concentrations of all the other elements present in the sample".

7.3.2 Analysis method

The method used shall be in accordance with the spectrometer manufacturer's instruction manual.

7.3.3 Number of sparks on test samples

The number of sparks carried out on a test sample shall be at least two. The spark areas shall be distributed over the prepared surface and whenever possible approximately midway between the centre and the border, thereby avoiding the centre of the sample. Measurements shall be examined; if any measurement is obviously defective, further sparks shall be carried out to obtain the minimum of two acceptable measurements. The average of the two acceptable measurements is used.

NOTE If the sample has been sparked in the centre, then it is essential that the results obtained be treated with caution because of segregation.

7.3.4 Status check of the apparatus

Any change from the original status of the spectrometer (e.g. drifts) shall be checked at regular intervals by analysing drift control samples or reference materials in accordance with the spectrometer manufacturer's instruction manual. It is common practice to document the status of the spectrometer using control charts. The difference between the results of these checks and their reference values are plotted regularly on a control chart. If the values fall outside the control limits, then further actions (e.g. recalibration, type recalibration, further checks, new calibration) shall be taken.

The period between checking the spectrometer status depends on its stability and the analytical assignment and can be derived from the control charts.

8 Expression of results

For each element, the average of the acceptable measurements represents the final result. The acceptable limits should be fixed by each laboratory depending on the matrix, the content levels and the specific performance of the spectrometer used.

9 Precision

Calculate the standard deviation as a measure for the precision using a minimum of 10 values from drift control samples or reference materials obtained in accordance with 7.3, at least for the lowest and highest content of the calibration range of each element.

NOTE The precision depends on how the element concerned exists in the matrix (dissolved or separated) and on its content in the sample (1. near the detection limit (DL) or the background equivalent concentration (BEC) and 2. above twice the BEC). Typical values are:

- a) for dissolved elements
 - < 2 BEC: 3 % to 33 %
 - > 2 BEC: < 1 %
- b) for separated elements (depending on the dispersion)
 - < 2 BEC: 6 % to 33 %
 - > 2 BEC: < 5 %

10 Test report

The test report shall contain the following information:

- a) identification of the test sample;
- b) reference to this European Standard (EN 15079);
- c) test method used;
- d) results;
- e) unusual characteristics noted during the determination;
- f) 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)

Wavelengths for spectrometric analysis and typical calibration ranges for copper and copper alloys

The list of wavelengths in Tables A.1, A.2 and A.3 shows the values obtained from the 9 replies received to a questionnaire sent to spectrometer users in the European copper industry. The table is not exhaustive, the exclusion of a line does not have any significance.

The table gives:

- wavelengths of lines in use;
- matrix, in which the wavelengths are used, and;
- typical calibration ranges.

The lower limits quoted are normally attained using the maximum practical sensitivity available on a spectrometer. The ranges are limited mostly by the availability of certified reference materials or reference materials. There may be other lines than those given in Tables A.1, A.2 or A.3.

Table A.1 — Elemental wavelength and range of calibration for pure copper and low alloyed copper

Element	Wavelength nm	Typical range of calibration %	
		low	high
Ag	338,29	0,000 2	0,5
	328,07	0,000 2	0,5
	235,79	0,02	2
Al	396,15	0,000 1	3
	394,40	0,000 1	3
As	189,04	0,000 2	0,5
	193,75	0,001	0,5
Au	242,79	0,000 1	0,05
B	249,67	0,000 1	0,1
Be	298,61	0,01	3
	313,04	0,000 1	0,2
	457,26	0,03	3
Bi	306,77	0,000 5	0,3
Cd	228,80	0,000 1	0,3
	214,44	0,000 2	0,3
Co	345,35	0,000 3	0,5
	228,61	0,000 5	1
Cr	267,72	0,000 3	1
	357,86	0,000 3	1
	425,43	0,002	1
Cu	296,12	reference lines	
	521,82		
	453,08		
	510,55		
Fe	259,94	0,000 3	1
	371,99	0,000 3	1
	271,44	0,01	5
Hg	184,96	0,000 1	0,002
Ir	380,01	0,000 1	0,01
Mg	285,21	0,000 1	0,1
	279,55	0,000 1	0,1
	279,07	0,000 2	1
	382,93	0,01	1

Table A.1 (continued)

Mn	257,61	0,000 2	0,5
	403,45	0,000 2	2
Ni	231,60	0,000 2	0,1
	361,93	0,000 5	0,03
P	178,29	0,000 1	0,5
Pb	283,31	0,000 5	2
	405,78	0,000 5	5
Pt	299,79	0,000 2	0,005
	265,94	0,000 2	0,03
Pd	363,47	0,000 2	0,05
S	180,73	0,000 1	0,6
Sb	259,81	0,000 5	0,5
	206,83	0,000 5	0,5
Se	196,09	0,000 2	0,3
Si	288,15	0,000 2	5
Sn	175,79	0,000 3	1
	189,98	0,000 3	1
Te	185,72	0,000 3	0,5
	238,57	0,005	2
Ti	337,27	0,000 1	0,2
	324,20	0,000 5	0,2
Zn	334,50	0,000 5	1
	472,21	0,01	1
Zr	343,82	0,000 1	0,3
	349,62	0,000 1	0,3

Table A.2 — Elemental wavelength and range of calibration for copper-zinc alloys

Element	Wavelength nm	Typical range of calibration %	
		low	high
Ag	338,29	0,000 2	0,5
Al	394,40	0,001	5
	396,15	0,001	5
	266,04	0,1	9
As	189,04	0,001	0,2
B	249,67	0,000 2	0,02
Bi	306,77	0,001	0,05
Cd	228,80	0,001	0,1
Co	345,35	0,001	0,4
	228,61	0,001	0,4
Cr	425,43	0,000 5	0,1
Cu	296,12	reference lines	
	453,08		
	510,55		
Fe	371,99	0,001	2
	273,95	0,01	5
	273,07	0,02	5
Mg	285,21	0,000 5	0,02
Mn	403,45	0,001	6
	293,30	0,005	2
	263,82	0,01	6
Ni	341,48	0,001	7
	231,60	0,001	0,1
O	130,20	0,005	2
P	178,29	0,001	0,2
Pb	283,31	0,002	4
	405,78	0,002	4
	220,35	0,01	4
S	180,73	0,002	0,05
Sb	259,81	0,001 5	0,4
	206,83	0,001 5	0,4
Se	196,09	0,001	0,1

Table A.2 (continued)

Si	259,61	0,1	5
	288,15	0,002	6
Sn	175,79	0,002	2
	189,99	0,002	2
	317,50	0,02	5
Te	185,72	0,001	0,1
Ti	324,20	0,000 2	0,03
	337,27	0,000 2	0,03
Zn	334,50	0,002	50
	481,05	0,05	50
	307,20	1	50
	468,01	20	45

Table A.3 — Elemental wavelength and range of calibration for copper-nickel alloys, copper-tin alloys, copper-aluminium alloys and copper-nickel-zinc alloys

Element	Wavelength nm	Typical range of calibration %	
		low	high
Ag	338,29	0,000 2	0,1
Al	394,40	0,000 5	8
	396,15	0,000 5	8
	256,80	0,1	12
	266,04	0,1	12
As	189,04	0,000 5	0,4
	197,26	0,002	0,4
B	249,67	0,000 2	0,05
Be	313,04	0,000 1	0,3
Bi	306,77	0,000 5	0,2
	289,80	0,002	0,5
C	193,09	0,001	0,1
Cd	228,80	0,001	0,1
	214,44	0,001	0,1
Co	345,35	0,001	1
	228,61	0,001	2
Cr	357,87	0,001	0,1
	425,40	0,001	0,1
	267,72	0,001	3
Cu	296,12	reference lines	
	453,08		
	510,55		
Fe	259,94	0,001	0,2
	371,99	0,001	2
	273,95	0,001	10
	273,07	0,01	10
Mg	285,20	0,000 1	0,05
	279,55	0,000 1	0,2
	291,54	0,05	0,2
Mn	257,61	0,000 1	0,1
	293,30	0,001	2
	403,45	0,002	5
	263,82	0,005	5

Table A.3 (continued)

Ni	231,60	0,001	0,1
	241,61	0,001	0,2
	331,23	0,2	44
	218,55	1	44
	376,95	5	30
	341,48	0,01	7
P	178,29	0,000 2	1
Pb	283,31	0,001 5	3
	405,78	0,002	20
	220,35	0,01	10
S	180,73	0,000 5	0,2
Sb	206,83	0,002	2
Se	196,09	0,000 1	0,1
Si	288,15	0,001	1
	390,55	0,05	1
Sn	175,79	0,001	1
	189,99	0,002	2
	317,50	0,02	20
Te	185,72	0,005	0,1
	238,57	0,01	0,4
Ti	324,20	0,001	0,2
	337,28	0,01	0,5
Zn	334,50	0,002	20
	481,05	0,1	30
	307,20	0,5	30
Zr	349,62	0,000 5	0,3
	343,82	0,000 5	0,3

Annex B (informative)

Wavelengths, background equivalent concentrations (BEC) and detection limits (DL) for pure copper

Table B.1 shows the values obtained from a round robin test in which a high purity copper sample was analysed by eight laboratories. Spark-OES spectrometers of different types from two different manufacturers were used in these laboratories.

The table gives:

- element symbols;
- wavelengths of lines in use;
- background equivalent concentration (BEC) values taken from the calibration functions;
- detection limits (DL) calculated according to: $3 \cdot s \cdot \text{BEC}/100$, where s is the relative standard deviation of the measurements carried out on the pure copper sample analysed in the round robin test, in per cent (%).

The table gives an overview of the analytical capability of modern spectrometers used in pure copper analyses.

Table B.1 — Wavelengths, BEC-values and DL-ranges of spectrometers used in the round robin test of pure copper

1	2	3	4	5
Ag	Wavelength, nm	328,07		
	Range BEC value, µg/g	1 to 67		
	Range DL (calc), µg/g	0,01 to 0,5		
As	Wavelength, nm	189,04	193,75	
	Range BEC value, µg/g	4 to 12	4	
	Range DL (calc), µg/g	0,2 to 0,6	0,1	
Bi	Wavelength, nm	306,77		
	Range BEC value, µg/g	6 to 29		
	Range DL (calc), µg/g	0,2 to 0,5		
Cd	Wavelength, nm	214,44	228,80	
	Range BEC value, µg/g	6 to 9	2 to 5	
	Range DL (calc), µg/g	0,1 to 0,3	0,03	
Co	Wavelength, nm	228,62	345,35	
	Range BEC value, µg/g	35 to 56	4 to 45	
	Range DL (calc), µg/g	0,1 to 0,9	0,1 to 0,5	
Cr	Wavelength, nm	267,71	357,87	425,43
	Range BEC value, µg/g	30 to 35	0,2 to 0,6	2
	Range DL (calc), µg/g	0,2 to 0,8	0,01 to 0,1	0,05
Fe	Wavelength, nm	259,94	371,99	
	Range BEC value, µg/g	6 to 27	2 to 8	
	Range DL (calc), µg/g	0,1 to 0,9	0,1 to 0,7	
Mn	Wavelength, nm	257,61	403,45	
	Range BEC value, µg/g	2 to 4	2 to 3	
	Range DL (calc), µg/g	0,03 to 0,05	0,05 to 0,1	
Ni	Wavelength, nm	231,6	341,47	361,94
	Range BEC value, µg/g	15 to 26	90 to 200	3 to 6
	Range DL (calc), µg/g	0,2 to 0,3	0,3 to 2	0,1 to 0,4
P	Wavelength, nm	178,29		
	Range BEC value, µg/g	3 to 9		
	Range DL (calc), µg/g	0,08 to 0,3		
Pb	Wavelength, nm	283,31	405,78	
	Range BEC value, µg/g	12 to 95	140 to 170	
	Range DL (calc), µg/g	0,3 to 4	1 to 3	

Table B.1 (continued)

S	Wavelength, nm	180,73		
	Range BEC value, µg/g	3 to 12		
	Range DL (calc), µg/g	0,1 to 1		
Sb	Wavelength, nm	206,83	231,14	
	Range BEC value, µg/g	25 to 160	33	
	Range DL (calc), µg/g	0,6 to 3	1,2	
Se	Wavelength, nm	196,09		
	Range BEC value, µg/g	9 to 19		
	Range DL (calc), µg/g	0,3 to 1,5		
Si	Wavelength, nm	288,16		
	Range BEC value, µg/g	3 to 14		
	Range DL (calc), µg/g	0,1 to 1		
Sn	Wavelength, nm	175,79		
	Range BEC value, µg/g	12 to 19		
	Range DL (calc), µg/g	0,2 to 3		
Te	Wavelength, nm	170,00	185,73	
	Range BEC value, µg/g	15 to 25	51 to 110	
	Range DL (calc), µg/g	1 to 2	0,9 to 3	
Ti	Wavelength, nm	337,28		
	Range BEC value, µg/g	9 to 11		
	Range DL (calc), µg/g	0,03 to 0,06		
Zn	Wavelength, nm	206,19	334,5	
	Range BEC value, µg/g	4 to 14	104 to 111	
	Range DL (calc), µg/g	0,2 to 0,5	0,6 to 2	
Zr	Wavelength, nm	349,62		
	Range BEC value, µg/g	14 to 17		
	Range DL (calc), µg/g	0,04 to 0,6		
NOTE The lowest values for BEC and DL represent the state of the art of trace analysis in pure copper during the year 2000.				

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