
Zinc and zinc alloys —

Part 1:

**Analysis of solid samples by optical
emission spectrometry**

Zinc et alliages de zinc —

*Partie 1: Analyse d'échantillons massifs par spectrométrie d'émission
optique*



Reference number
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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3815-1 was prepared by Technical Committee ISO/TC 18, *Zinc and zinc alloys*, Subcommittee SC 1, *Methods of sampling and analysis of zinc and zinc alloys*.

This first edition of ISO 3815-1 cancels and replaces ISO 3815:1976, which has been technically revised.

ISO 3815 consists of the following parts, under the general title *Zinc and zinc alloys*:

- *Part 1: Analysis of solid samples by optical emission spectrometry*
- *Part 2: Analysis by inductively coupled plasma optical emission spectrometry*

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Zinc and zinc alloys —

Part 1: Analysis of solid samples by optical emission spectrometry

1 Scope

This part of ISO 3815 specifies analytical methods for determining the chemical composition of zinc and zinc alloys in accordance with ISO 301 and ISO 752 by optical emission spectrometry.

This part of ISO 3815 includes recommendations for preparation of test pieces from zinc and zinc alloys.

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 301, *Zinc alloy ingots intended for casting*

ISO 752:2004, *Zinc ingots*

ISO 20081:—¹⁾, *Zinc and zinc alloys — Method of sampling — Specifications*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 301, ISO 752, ISO 20081 and the following apply.

3.1

optical emission spectrometry

measurement of the intensity of electromagnetic radiation emitted by the components of a sample when excited

NOTE Each element emits radiation of well defined and specific wavelength, whose intensity is linked to its concentration.

4 Sampling

Sampling shall be in accordance with ISO 20081:—¹⁾, 7.1.

1) To be published.

5 Preparation of test pieces

Test pieces shall be prepared so that they are suitable for optical emission spectrometry. The shape and size of test pieces are dependent upon the sample holder and apparatus used. The surface of test pieces shall be prepared according to requirements given in the operation manual of the apparatus used.

6 Test procedure

6.1 General

Analysis shall be carried out by optical emission spectrometry using solid test pieces prepared in accordance with Clause 5. For analysis of the impurities and alloying elements in zinc and zinc alloys as defined in ISO 301 and ISO 752 by optical emission spectrometry, different analytical lines can be used. A list of appropriate wavelengths of analytical lines is given in Annex A.

Which of the different analytical lines is used depends on the analytical programme and the type of spectrometer.

The reproducibility of the apparatus used shall be in accordance with the values given in Annex B.

6.2 Calibration

The apparatus used shall be suitable for the detection and determination of all elements specified in the relevant product standard (see 6.1).

For the calibration of the spectrometer, certified reference materials (CRM) should be used primarily, reference materials (RM) secondarily and internal reference materials (IRM) last of all.

If IRMs are used, their chemical composition shall be determined by the methods of analysis as specified in 8.1 of ISO 752:2004.

The apparatus shall be re-calibrated within a reasonable time. The reference samples used for re-calibrating the apparatus shall have physical and chemical properties similar to the sample to be analysed.

6.3 Method of testing

In general, each test piece shall be tested at least two times. If heterogeneity or malfunction of the spectrometer is suspected, additional tests shall be performed on the same sample using a new surface.

NOTE In the specific case of producer labs, if sampling frequency is high enough and historical data supports it, each test piece need only be tested once, provided that the result for the lot to be analysed is the average of at least two tests.

6.4 Expression of results

Test results shall be expressed as mass fraction, calculated as the arithmetic mean of all valid single results of the test sequence according to 6.3, excluding failing single test.

Results shall be expressed as defined in ISO 301 and ISO 752.

6.5 Test report

The test report shall include the following items:

- a) identification of sample;
- b) test result of each test sequence according to 6.4;
- c) name of laboratory or testing organization;
- d) date of the test report;
- e) reference to this part of ISO 3815 (ISO 3815-1:2005);
- f) signature of the laboratory manager or other responsible person.

Annex A (informative)

List of analytical lines

Table A.1 provides a list of analytical lines, in nanometres, commonly used for analysis of zinc and zinc alloys by optical emission spectrometry.

Table A.1

Element	Analytical lines nm
Zn	481,0
Pb	405,7 ^a ; 368,3
Cd	228,8; 361,0
Fe	371,9
Cu	327,4; 324,7; 510,5 ^b
Sn	317,5
Al	396,1; 394,4; 305,2 ^c
Mg	285,20; 279,08; 382,93
^a	Pb 405,7 is used for Pb concentrations up to 1,5 %.
^b	Cu 510,5 is used for Cu concentrations from 0,5 % to 5 %.
^c	Al 305,2 is used for Al concentrations from 4 % to 30 %.

Annex B (informative)

Reproducibility

Table B.1 provides an example of coefficients of variation characterizing optical emission spectrometers, representing the average of 10 flashings per test piece.

Table B.1

Element	Content % (mass fraction)	Coefficient of variation %
Pb	0,01	2
Cd	0,01	2
Sn	0,01	2
Fe	0,01	3
Cu	0,001	1
Cu	1	1
Al	0,001	3
Al	4,0	1
Mg	0,1	2

Annex C (informative)

Limits of detection

Table C.1 provides data on best achievable limits of detection (LOD) for spark optical emission spectrometers (OES).

Table C.1

Element	Line nm	LOD ^a µg/g
Pb	405,7	< 1
Cd	228,8	0,1
Fe	371,8	1
Cu	324,7	< 1
Sn	317,5	1
Al	396,1	0,2
Mg	279,0	< 1

^a LOD = 3 s_0 , where s_0 = standard deviation at zero concentration.

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