INTERNATIONAL STANDARD

ISO 3815-2

First edition
2005-07-15

Zinc and zinc alloys —

Part 2: Analysis by inductively coupled plasma optical emission spectrometry

Zinc et alliages de zinc —

Partie 2: Analyse par spectrométrie d'émission optique avec source à plasma à couplage inductif

Reference number
ISO 3815-2:2005(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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 3815-2 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 3815-2 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 2:
Analysis by inductively coupled plasma 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 inductively coupled plasma optical emission spectrometry.

This part of ISO 3815 includes provisions for preparation of test solutions and calibration solutions for zinc and zinc alloys.

The ranges specified for each method can be extended and/or adapted for determinations of low concentrations.

This part of ISO 3815 can be applied to other elements (e.g. Ni, Cr and Ti). However, such results will need to be carefully checked by taking into account the interferences, the sensitivity, the resolution and the 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 301, *Zinc alloy ingots intended for casting*

ISO 752, *Zinc ingots*

ISO 1169, *Zinc alloys — Determination of aluminium content — Volumetric method*

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

EN 988, *Zinc and zinc alloys — Specifications for rolled flat products for building*

EN 12844, *Zinc and zinc alloys — Castings — Specifications*

1) To be published.

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
inductively coupled plasma optical emission spectrometry
measurement of the intensity of electromagnetic radiation emitted by the components of a sample when excited by a plasma

NOTE A plasma is a high-temperature emission source generated by an RF field consisting of a mixture of argon atoms, argon ions and electrons. Sample atoms entering the plasma emit light radiation, whose characteristic wavelengths and intensities are used to identify the elements and determine concentrations, respectively. Samples are usually presented to the plasma in solution form.

4 Principle

A sample of the metal or alloy is dissolved in a mixture of nitric and tartaric acid, and after adequate dilution and atomization of the solution in an inductively coupled plasma, the content of the particular elements is determined by optical emission spectrometry.

5 Reagents

During the test, use only reagents of known or analytical grade and distilled or demineralized water.

5.1 Hydrochloric acid, ρ = 1,19 g/ml.

5.2 Nitric acid, ρ = 1,4 g/ml.

5.3 Tartaric acid, solid.

5.4 Tartaric acid solution, 25 g/l.

5.5 Zinc, purity 99,999 % (mass fraction), free of lead, cadmium, iron, tin, copper, magnesium, nickel, chromium and titanium.

5.6 Aluminium nitrate nonahydrate, heavy metal impurities: lead max. 0,002 %, iron max. 0,001 %.

5.7 Acid mixture

Dissolve 1 g of tartaric acid (5.3) in about 500 ml of water in a 1 000 ml volumetric flask, add 200 ml of nitric acid (5.2), dilute to the mark with water and mix.

5.8 Zinc solution

Prepare a solution of 100 g/l by carefully dissolving 100 g of zinc (5.5), weighed to the nearest 0,1 g, with a minimum of nitric acid (5.2), added in small increments. Transfer to a 1 l volumetric flask, add 40 ml of tartaric acid solution (5.4) and, after cooling to room temperature, dilute to the mark with water and mix.

It is recommended to verify the purity of the zinc used (absence of analyte elements). If any analyte element is present, determine its concentration by the standard addition method and take this concentration into account in the calibration standards (see 8.2).

5.9 Mono-element standard solutions

Prepare solutions of 1 g/l of lead, cadmium, iron, copper, aluminium and magnesium, by dissolving 1 g of each of these metals, weighed to the nearest 0,001 g, in the acid mixture (5.7) and making up to volume with the acid mixture (5.7).

Prepare a solution of 1 g/l of tin, by carefully adding 100 ml of hydrochloric acid (5.1) to 1 g of pure tin. When dissolution is complete, cool and transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water and mix.

By dilution, prepare standard solutions of 100 mg/l and 10 mg/l, if necessary (see 8.2).

5.10 Aluminium standard solution, to be used for alloys with aluminium content $>$ 3 % (mass fraction).

Prepare a solution of 50 g/l by dissolving 695,2 g of aluminium nitrate nonahydrate (5.6), weighed to the nearest 0,1 g, in water. Transfer to a 1 l volumetric flask, add 40 ml of tartaric acid solution (5.4), dilute to the mark with water and mix.

By taking an appropriate aliquot, determine the exact aluminium content in accordance with ISO 1169.

5.11 Internal standard solution

Weigh 6,25 g of Y_2O_3 (yttrium oxide p.a.), to the nearest 0,01 g, and transfer into a 5 l volumetric flask. Add about 2,5 l of water, 250 ml of nitric acid (5.2) and warm gently until dissolution. Cool to room temperature, dilute to the mark with water, and mix.

6 Sampling

Sampling shall be in accordance with ISO 20081.

7 Preparation of test solutions

7.1 Test portion

Weigh 10 g of the test sample to the nearest 0,001 g.

7.2 Preparation of the test solution

Introduce the test portion into a 500 ml beaker fitted with a watch-glass cover and dissolve by carefully adding 10 ml of tartaric acid solution (5.4) and 50 ml of nitric acid (5.2).

Alternatively, dissolve the test portion in 50 ml of hydrochloric acid-nitric acid mixture (45+1). In this case, zinc solution (5.8) and standard solutions (5.9), (5.10) are also prepared such that the resulting calibration solutions (8.2) have an acid composition identical to the sample solution.

After dissolution, transfer quantitatively to a 250 ml volumetric flask, add 10 ml of internal standard solution (5.11) and, after cooling to room temperature, make up to the mark with water and mix.

8 Test procedure

8.1 General

Carry out the test by optical emission spectrometry with plasma source, using test solutions prepared in accordance with Clause 7. For the 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 the wavelengths of appropriate analytical lines is given in Annex A.

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

8.2 Calibration

The apparatus used shall be capable and suitable for the detection and determination of all elements specified in the relevant product standards (ISO 301 and ISO 752).

Calibrate the apparatus within a reasonable time, in accordance with the procedures described in the instruction manual.

Prepare the calibration solutions taking into account the product specification on chemical composition, in accordance with Table 1 below.

Table 1 — Composition of spectrometer calibration solution

Calibration standard No.	1	2	3	4	5	6	7
Alloying element 1	0,8 m	m	(3m+M)/4	(m+M)/2	(m+3M)/4	M	1,2 M
Alloying element 2	1,2 M	M	(m+3M)/4	(m+M)/2	(3m+M)/4	m	0,8 m
Alloying element 3	(3m+M)/4	m	0,8 m	(m+M)/2	1,2 M	M	(m+3M)/4
Alloying element 4	(m+3M)/4	M	1,2 M	(m+M)/2	0,8 m	m	(3m+M)/4
Impurity element 1	blank	0,2 L	0,5 L	0,8 L	L	1,2 L	1,5 L
Impurity element 2	0,2 L	0,5 L	0,8 L	L	1,2 L	1,5 L	blank
Impurity element 3	0,5 L	0,8 L	L	1,2 L	1,5 L	blank	0,2 L
Impurity element 4	0,8 L	L	1,2 L	1,5 L	blank	0,2 L	0,5 L
Impurity element 5	L	1,2 L	1,5 L	blank	0,2 L	0,5 L	0,8 L
Impurity element 6	1,2 L	1,5 L	blank	0,2 L	0,5 L	0,8 L	L
Impurity element 7	1,5 L	blank	0,2 L	0,5 L	0,8 L	L	1,2 L
Remainder Zn	(100−sum)	(100−sum)	(100−sum)	(100−sum)	(100−sum)	(100−sum)	(100−sum)

m = minimum content specification for alloying element % (mass fraction)

M = maximum content specification for alloying element % (mass fraction)

L = maximum content specification for impurity element % (mass fraction)

Rounding rules:

— for contents > 1 % (mass fraction), 2 numbers behind the comma, the last number being 5 or 0;

— for contents < 1 % (mass fraction), round to 2 significant numbers, the last number being 5 or 0.

Do not apply rounding rules for contents < 0,001 5 % (mass fraction).

Into each of seven 250 ml volumetric flasks, transfer appropriate volumes (between 1 ml and 50 ml) of the relevant solutions (5.8, 5.9, 5.10). Calculate each volume to contain the exact mass of analyte such as to give a total mass of 10 g, in order to obtain direct reading in percent (mass fraction) in accordance with Table 1. Add 10 ml of internal standard solution (5.11), dilute to the mark with acid mixture (5.7), and mix.

Use internal standardization and background correction techniques, if these techniques optimize the calibration graphs. The correlation coefficient of each calibration graph should be at least 0,999.

In order to validate the calibration graphs, it is recommended to analyse a certified reference material (CRM), if available.

8.3 Method of testing

In general, each test solution shall be tested at least two times. If malfunction of the spectrometer is suspected, perform additional tests.

8.4 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 8.3, excluding failing single test.

Express results in accordance with ISO 301, ISO 752, EN 12844 and EN 988.

8.5 Test report

The test report shall include following items:

a) identification of sample;

b) test result of each test sequence according to 8.4;

c) name of laboratory or testing organization;

d) date of the test report;

e) reference to this part of ISO 3815 (ISO 3815-2);

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 inductively coupled plasma optical emission spectrometry.

Table A.1

Element	Analytical lines nm
Pb	283,306; 220,353
Cd	226,502
Fe	259,940
Cu	324,754
Sn	189,989; 303,412
Al	257,510
Mg	279,553
Y	321,669

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Price based on 6 pages

INTERNATIONAL STANDARD

ISO 5077

Second edition
2007-02-15

Textiles — Determination of dimensional change in washing and drying

Textiles — Détermination des variations dimensionnelles au lavage et au séchage domestiques

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ISO 5077:2007(E)

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Foreword

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ISO 5077 was prepared by Technical Committee ISO/TC 38, *Textiles*, Subcommittee SC 2, *Cleansing, finishing and water resistance tests*.

This second edition cancels and replaces the first edition (ISO 5077:1984), which has been technically revised.

Textiles — Determination of dimensional change in washing and drying

1 Scope

This International Standard specifies a method for the determination of the dimensional change of fabrics, garments or other textile articles when subjected to an appropriate combination of specified washing and drying procedures.

In the case of textile articles or deformable materials, it is necessary to exercise all possible caution in the interpretation of the results.

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 139, *Textiles — Standard atmospheres for conditioning and testing*

ISO 3759, *Textiles — Preparation, marking and measuring of fabric specimens and garments in tests for determination of dimensional change*

ISO 6330, *Textiles — Domestic washing and drying procedures for textile testing*

3 Principle

The specimen is conditioned in the specified standard atmosphere and measured before subjection to the appropriate washing and drying procedures. After drying, conditioning and remeasuring of the specimen, the changes in dimensions are calculated.

4 Apparatus and reagents

Use apparatus and reagents as specified in ISO 3759 and ISO 6330.

5 Atmospheric conditions

The atmospheric conditions required for conditioning and testing are specified in ISO 139.

6 Test specimens

6.1 The selection, dimensions, marking and measuring of test specimens are specified in ISO 3759.

6.2 When possible, three specimens from each sample should be used. One or two specimens may be used when insufficient sample is available.

7 Procedure

7.1 Determine the original length and width dimensions, as appropriate, after the specimens have been conditioned and measured according to the procedure specified in ISO 139 and ISO 3759.

7.2 Wash and dry the specimens according to one of the procedures specified in ISO 6330, as agreed between the interested parties.

7.3 After washing and drying, condition and measure the specimens and calculate the dimensional change of the specimens according to the procedure specified in ISO 3759.

8 Expression of results

8.1 Calculate the mean changes in dimensions in both the length and width directions in accordance with the arrangement in ISO 3759 as follows:

$$\frac{x_t - x_o}{x_o} \times 100$$

where

x_o is the original dimension;

x_t is the dimension measured after treatment.

Record the changes in measurement separately as a percentage of the corresponding original value.

8.2 Express the average dimensional changes to the nearest 0,5 %.

8.3 State whether the dimension has decreased (shrinkage) by means of a minus sign ($-$) or increased (extension) by means of a plus sign ($+$).

9 Test report

The test report shall specify the following:

a) the number and year of this International Standard;

b) the number of specimens washed and dried;

c) the procedure used for washing and drying from ISO 6330;

d) for fabric specimens, the average dimensional change in the length (warp or wale) and the average dimensional change in the width (weft or course) to the nearest 0,5 %;

e) for garments, the description, make and size of the garment tested;

f) for garments, an adequate description of each measuring position and the average dimensional change to the nearest 0,5 % at each position for each garment tested.