

Determination of thickness and chemical composition of zinc- and aluminium-based metallic coatings — Routine method

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National foreword

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Determination of thickness and chemical composition of zinc- and aluminium-based metallic coatings - Routine method

Détermination de l'épaisseur et de la composition chimique
des revêtements en zinc et en alliage d'aluminium -
Méthode de routine

Bestimmung der Dicke und der chemischen
Zusammensetzung metallischer Überzüge auf Basis von
Zink und Aluminium - Standard-Verfahren

This European Standard was approved by CEN on 21 March 2005.

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Foreword

This European Standard (EN 10318:2005) has been prepared by Technical Committee ECISS/TC 20 “Methods of chemical analysis of ferrous products”, the secretariat of which is held by SIS.

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of an identical text or by endorsement, at the latest by November 2005, and conflicting national standards shall be withdrawn at the latest by November 2005.

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

1 Scope

This European Standard specifies a glow discharge optical emission spectrometric method for the determination of the thickness and chemical composition of metallic surface coatings consisting of zinc and aluminium based alloys. The alloying elements considered are aluminium, nickel, silicon and lead.

This method is applicable to zinc contents between 40 % (m/m) and 100 % (m/m); aluminium contents between 0,01 % (m/m) and 60 % (m/m); nickel contents between 0,01 % (m/m) and 15 % (m/m); silicon contents between 0,01 % (m/m) and 3 % (m/m); lead contents between 0,005 % (m/m) and 0,1 % (m/m).

2 Normative references

Not applicable.

3 Principle

The analytical method described here involves the following processes:

- a) Cathodic sputtering of the surface coating in a direct current glow discharge device;
- b) Optical excitation of the analyte atoms in the plasma formed in the glow discharge device;
- c) Spectrometric measurement of characteristic emission spectral lines of the analyte atoms as a function of sputtering time (depth profile); and
- d) Conversion of the depth profile in units of intensity versus time to mass fraction versus depth by means of calibration functions (quantification). Calibration of the system is achieved by measurements on calibration samples of known chemical composition and measured sputtering rate.

4 Apparatus

4.1 Glow discharge optical emission spectrometer

4.1.1 General

An optical emission spectrometer equipped with a Grimm type (1) or similar direct current glow discharge source and a simultaneous optical spectrometer, incorporating suitable spectral lines for the analyte elements (see Table 1 for recommended lines) shall be used.

The inner diameter of the hollow anode of the glow discharge shall be in the range 2 mm to 8 mm. A cooling device for thin samples, such as a metal block with circulating cooling liquid, is also recommended, but not strictly necessary for implementation of the method.

It is desirable for the instrument to conform to the performance specifications given in 4.1.2 and 4.1.3, to be evaluated in 6.2.6.

4.1.2 Minimum repeatability

Perform 10 measurements of the emission intensity on a homogeneous bulk sample with a content of the analyte exceeding 1 % (m/m). Allow the discharge at least 60 s stabilisation time (often referred to as *preburn*) before each intensity measurement. Each measurement shall be located on a newly prepared surface of the sample. Calculate the standard deviation of the 10 measurements. The standard deviation should not exceed 2 % of the mean intensity of the analyte. If this is the case, repeat the test two more times. If the high standard deviation is

repeatable, there is probably some malfunction in the instrument or the sample used is not homogeneous. Before proceeding, the cause of the problem should be investigated and rectified.

4.1.3 Limit of detection

Detection limits are instrument-dependent and matrix-dependent. Consequently, the detection limit for a given analyte cannot be uniquely determined for every available instrument or for the full range of Zn-based alloys considered here. For the purposes of this document, the detection limit for each analyte will be acceptable if it is equal to or less than one third of the lowest concentration to be determined in the intended applications. The detection limit is determined using the method explained below.

- a). Select a bulk sample to be used as a blank. The sample composition should be similar to the coatings to be analyzed in terms of the elemental composition of the matrix. Further, it shall be known to contain less than $0,1 \text{ mg kg}^{-1}$ of the analyte.
- b). Perform ten replicate burns on the blank. For each burn, acquire the emission intensity at the analytical wavelength for 10 s. These are the background emission intensity measurements. The glow discharge conditions used should be the same as those that will be used in the analysis of the coated samples. For each measurement, the blank should be preburned at these conditions for a sufficient length of time to achieve stable signals prior to the quantification of the emission intensity. An unsputtered area of the surface of the blank for each individual burn shall be used.
- c). Compute the detection limit using the following equation:

$$DL = \frac{3 \times S}{m}$$

where

DL is the detection limit;

S is the standard deviation of the ten background intensity measurements performed in step (2);

m is the analytical sensitivity derived from the instrument calibration expressed as the ratio of intensity to mass fraction.

If the detection limit calculated is greater than one third of the lowest concentration to be determined in the intended applications, then the test should be repeated. If the second value calculated is also greater than one third of the lowest concentration to be determined in the intended applications, then there may be an instrument malfunction. In such a case, the problem should be investigated prior to analyzing unknown samples.

4.2 Data acquisition

Since the principle of determination is based on continuous sputtering of the surface coating, the spectrometer shall be equipped with a digital readout system for time-resolved measurement of the emission intensities. A system with capability for data acquisition speed of at least 500 measurements/second per spectral channel is recommended, but for the applications within the scope of this standard a speed of 2 measurements/second per spectral channel may be acceptable.

5 Sampling

Carry out sampling in accordance with the recommendations of the manufacturer of the coated material. In general, the edges of a coated strip should be avoided. The size of the test samples should be suitable for the glow discharge source used. Typically, round or rectangular samples with a width of 20 mm to 100 mm are suitable.

6 Procedure

6.1 Selection of spectral lines

For each analyte to be determined there exists a number of spectral lines which can be used. Suitable lines shall be selected on the basis of several factors including the spectral range of the spectrometer used, analyte concentration range, sensitivity of the spectral lines and spectral interference from other elements present in the samples. In this type of application, where most of the analytes of interest are major elements in the samples, special attention shall be paid to the occurrence of self-absorption of certain highly sensitive spectral lines. Self-absorption may cause severe non-linearity of calibration curves at high analyte concentration levels, and such lines should therefore be avoided for the determination of majors. In Table 1, some suggestions concerning suitable spectral lines are given.

Table 1 — Suggested spectral lines for determination of given elements

Element	Wavelength (nm)	Estimated useful concentration range % (m/m)	Comments
Zn	330,26	0,001 to 100	
Zn	334,50	0,001 to 100	
Zn	481,053	0,001 to 100	
Al	172,50	0,1 to 100	
Al	396,15	0,001 to 100 ^a	Self-absorption
Ni	231,603	0,01 to 100	
Ni	341,78	0,001 to 100 ^a	Weak self-absorption
Ni	349,30	0,005 to 100 ^a	Weak self-absorption
Pb	202,20	0,001 to 10	
Pb	405,87	0,01 to 100	
Si	212,41	No data available	
Si	251,61	No data available	
Si	288,16	0,001 to 20	
Fe	249,318	0,01 to 100	
Fe	259,94	0,01 to 100	
Fe	271,44	0,1 to 100	
Fe	371,94	0,005 to 100 ^a	Weak self-absorption
Fe	379,50	0,01 to 100	
Cu	296,12	0,01 to 100	
Cu	327,40	0,001 to 5 ^a	Strong self-absorption

^a Use of non-linear calibration curve recommended.

6.2 Optimising the glow discharge spectrometer settings

6.2.1 General

Follow the manufacturer's instructions for preparing the instrument for use. In particular, check that the entrance slit to the spectrometer is correctly adjusted, following the procedure given by the instrument manufacturer. This