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Zinc and/or aluminium based coatings on steel — Determination of coating mass per unit area and chemical composition — Gravimetry, inductively coupled plasma atomic emission spectrometry and flame atomic absorption spectrometry

Revêtements à base de zinc et/ou d'aluminium sur acier — Détermination de la masse surfacique et de la composition chimique du revêtement — Gravimétrie, spectrométrie d'émission atomique avec plasma induit par haute fréquence et spectrométrie d'absorption atomique dans la flamme

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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 17925 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

Zinc and/or aluminium based coatings on steel — Determination of coating mass per unit area and chemical composition — Gravimetry, inductively coupled plasma atomic emission spectrometry and flame atomic absorption spectrometry

1 Scope

This International Standard specifies methods of determining the coating mass per unit area by gravimetry and chemical composition on one side-surface of zinc- and/or aluminium-based coatings on steel by means of inductively coupled plasma atomic emission spectrometric or flame atomic absorption spectrometry. For example, this test method applies for zinc and/or aluminium based coatings on steel such as galvanize (hot dip and electrolytic), galvaneal (hot-dip), zinc-nickel electrolytic, zinc-5 % aluminium coating (hot-dip) and zinc-55 % aluminium coating (hot-dip). Galvanizing gives a pure zinc coating. Galvanealling gives a zinc-iron alloyed coating. Zinc-nickel electrolytic methods give zinc-nickel alloyed coatings.

This method is applicable to zinc contents between 40 % (mass fraction) and 100 % (mass fraction); aluminium contents between 0,02 % (mass fraction) and 60 % (mass fraction); nickel contents between 7 % (mass fraction) and 20 % (mass fraction); iron contents between 0,2 % (mass fraction) and 20 % (mass fraction); silicon contents between 0,2 % (mass fraction) and 10 % (mass fraction); lead contents between 0,005 % (mass fraction) and 2 % (mass fraction). For example, the applicable elements for these products are as follows: galvanizing is specified for iron and aluminium; galvanealling is specified for zinc, iron and aluminium; zinc-nickel electrolytic methods are specified for zinc, iron and nickel; zinc-5 % aluminium coating is specified for zinc, iron, aluminium and silicon; zinc-55 % aluminium is specified for zinc, iron, aluminium and silicon.

FAAS determination for the chemical composition of a coating layer is not applicable for zinc.

These test methods are intended as referee methods to test such materials for compliance with mass per unit area and compositional specifications of International Standards.

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 648:1977, *Laboratory glassware — One-mark pipettes*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

ISO 14284:1996, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

3 Principle

Stripping the coating on one side on the base steel in a mixture of hydrochloric acid solution containing an inhibitor to prevent the attack on the base steel. Determination of the mass per unit area of coating from the mass difference of the specimen before and after stripping. Calculating the coating mass as the mass difference divided by the specimen's surface area.

Dilution of the stripped solution of the coating on one side. Filtration and nebulization of the solution into an inductively coupled plasma atomic emission spectrometer (ICP-AES) or flame atomic absorption spectrometer (FAAS). Calculating the chemical compositions of coating layer as the content of the analytical element divided by the pre-measured coating mass.

Examples of the analytical lines are given in Table 1.

Table 1 — Examples of analytical lines together with interfering elements

Element	ICP-AES		FAAS	
	Analytical line nm	Interfering elements	Analytical line nm	Interfering elements
Zinc	481,0 206,19			
Aluminium	396,15		309,3 306,16	
Nickel	231,60		232,00 231,10 233,75	
Iron	271,44 259,94		248,33 252,29	
Silicon	251,61 288,16	Aluminium	251,61 288,16	Aluminium
Lead	220,35	Zinc Aluminium	217,00 283,31	

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only grade 2 water as specified in ISO 3696.

4.1 Hydrochloric acid, HCl, $\rho \approx$ 1,18 g/ml.

4.2 Hydrochloric acid, HCl, diluted 1+10.

4.3 Nitric acid, HNO_3, $\rho \approx$ 1,40 g/lml.

4.4 Nitric acid, HNO_3, diluted 1+1.

4.5 Mixed acid, prepared by adding 50 ml of nitric acid (4.3) and 10 ml of hydrochloric acid (4.1) to a flask containing 150 ml of water and mixing.

4.6 Sodium carbonate, Na_2CO_3.

4.7 Hexamethylenetetramine, $C_6H_{12}N_4$, capable of preventing acid attack of the base metal while stripping the coating from the base steel.

4.8 Stripping solution, prepared by transferring from 170 ml to 500 ml of hydrochloric acid (4.1) to a 1 l graduated cylinder containing from 450 ml to 820 ml of water and adding 3,5 g of hexamethylenetetramine (4.7) then diluting to the mark with water followed by mixing.

NOTE For an electrolytic zinc coated steel such as one with a lower coating mass, it would be better to use diluted hydrochloric acid for stripping in order to prevent the dissolution of base steel.

4.9 Stop-off materials, capable of protecting one side of a coated piece of steel sheet while the other side is being stripped in hydrochloric acid solution, without contaminating the acid solution and either gaining or losing mass thus avoiding interference with coating mass and chemical composition determination.

NOTE 1 Acid-resistant paints, lacquers or acid-resistant tapes are commonly used as stop-off materials.

NOTE 2 For this purpose mechanical devices may also be used, fastened to the test specimen instead of the above materials.

4.10 Zinc stock standard solution, 1 000 mg/l, prepared as follows:

weigh, to the nearest 0,000 5 g, 0,5 g of high purity zinc (minimum 99,99 % by mass) and dissolve in 25 ml hydrochloric acid (4.1). Cool and transfer the solution quantitatively to a calibrated 500 ml one-mark volumetric flask. Keep the flask at the same temperature as that at which the flask was calibrated. Dilute to the mark with water and mix.

4.11 Zinc standard solution A, 100 mg/l, prepared as follows:

using a calibrated pipette, transfer 100 ml of the zinc stock standard solution (4.10) into a calibrated 1 000 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix.

4.12 Zinc standard solution B, 10 mg/l, prepared as follows:

using a calibrated pipette, transfer 100 ml of the zinc standard solution A (4.11) into a calibrated 1 000 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix.

4.13 Zinc standard solution for matrix matching, 10 000 mg/l, prepared as follows:

weigh, to the nearest 0,01 g, 10 g of high purity zinc (minimum 99,99 % by mass) and dissolve in 200 ml hydrochloric acid (4.1), Cool and transfer the solution quantitatively to a calibrated 1 000 ml one-mark volumetric flask. Keep the flask at the same temperature as that at which the flask was calibrated. Dilute to the mark with water and mix.

4.14 Aluminium stock standard solution, 1 000 mg/l, prepared as follows:

weigh, to the nearest 0,000 5 g, 0,5 g of high purity aluminium (minimum 99,95 % by mass) and dissolve in a mixture of 25 ml hydrochloric acid (4.1) and 5 ml nitric acid (4.3). Cool and transfer the solution quantitatively to a calibrated 500 ml one-mark volumetric flask. Keep the flask at the same temperature as that at which the flask was calibrated. Dilute to the mark with water and mix.

4.15 Aluminium standard solution A, 100 mg/l, prepared as follows:

using a calibrated pipette, transfer 100 ml of the aluminium stock standard solution (4.14) into a calibrated 1 000 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix.

4.16 Aluminium standard solution B, 10 mg/l, prepared as follows:

using a calibrated pipette, transfer 100 ml of the aluminium standard solution A (4.15) into a calibrated 1 000 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix.

4.17 Aluminium standard solution for matrix matching, 10 000 mg/l, prepared as follows:

weigh, to the nearest 0,01 g, 10 g of high purity aluminium (minimum 99,99 % by mass) and dissolve in a mixture of 200 ml hydrochloric acid (4.1) and 5 ml nitric acid (4.3). Cool and transfer the solution quantitatively to a calibrated 1 000 ml one-mark volumetric flask. Keep the flask at the same temperature as that at which the flask was calibrated. Dilute to the mark with water and mix.

4.18 Nickel stock standard solution, 1 000 mg/l, prepared as follows:

weigh, to the nearest 0,000 5 g, 0,5 g of high purity nickel (minimum 99,95 % by mass) and dissolve in 30 ml nitric acid (1+1) (4.4). Cool and transfer the solution quantitatively to a calibrated 500 ml one-mark volumetric flask. Keep the flask at the same temperature as that at which the flask was calibrated. Dilute to the mark with water and mix.

4.19 Nickel standard solution A, 100 mg/l, prepared as follows:

using a calibrated pipette, transfer 100 ml of the nickel stock standard solution (4.18) into a calibrated 1 000 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix.

4.20 Nickel standard solution B, 10 mg/l, prepared as follows:

using a calibrated pipette, transfer 100 ml of the nickel standard solution A (4.19) into a calibrated 1 000 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix.

4.21 Iron stock standard solution, 1 000 mg/l, prepared as follows:

weigh, to the nearest 0,000 5 g, 0,5 g of high purity iron (minimum 99,95 % by mass) and dissolve in 25 ml hydrochloric acid (4.1). Cool and transfer the solution quantitatively to a calibrated 500 ml one-mark volumetric flask. Keep the flask at the same temperature as that at which the flask was calibrated. Dilute to the mark with water and mix.

4.22 Iron standard solution A, 100 mg/l, prepared as follows:

using a calibrated pipette, transfer 100 ml of the iron stock standard solution (4.21) into a calibrated 1 000 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix.

4.23 Iron standard solution B, 10 mg/l, prepared as follows:

using a calibrated pipette, transfer 100 ml of the iron standard solution A (4.22) into a calibrated 1 000 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix.

4.24 Silicon stock standard solution, 1 000 mg/l, prepared as follows:

weigh, to the nearest 0,000 1 g, 2,139 3 g of freshly calcined high purity silica (minimum 99,9 % by mass SiO_2) and transfer to a platinum crucible. The high-purity silica shall be calcined for 1 h at 1 100°C and cooled in a desiccator immediately before use. Mix thoroughly with 16 g of anhydrous sodium carbonate and fuse at 1 050°C for 30 min. Extract the fusion product with 100 ml of water in a polypropylene or polytetrafluoroethylene beaker (see Note below). Cool and transfer the extract, which should contain no trace of residue, to a 1 000 ml one-mark volumetric flask. Keep the flask at the same temperature as that at which the flask was calibrated. Dilute to the mark and mix, transfer immediately to a well-stoppered polytetrafluoroethylene bottle for storage. 1 ml of this stock solution contains 1 mg of silicon.

NOTE Extraction of the fusion product may require prolonged digestion in water followed by gentle heating.

4.25 Silicon standard solution A, 100 mg/l, prepared as follows:

using a calibrated pipette, transfer 100 ml of the silicon stock standard solution (4.24) into a calibrated 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

4.26 Silicon standard solution B, 10 mg/l, prepared as follows:

using a calibrated pipette, transfer 100 ml of the silicon standard solution A (4.25) into a calibrated 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

4.27 Lead stock standard solution, 1 000 mg/l, prepared as follows:

weigh, to the nearest 0,000 5 g, 0,5 g of high purity lead (min 99,95 % by mass) and dissolve in 30 ml nitric acid (1+1) (4.3). Cool and transfer the solution quantitatively to a calibrated 500 ml one-mark volumetric flask. Keep the flask at the same temperature as that at which the flask was calibrated. Dilute to the mark with water and mix.

4.28 Lead standard solution A, 100 mg/l, prepared as follows:

using a calibrated pipette, transfer 100 ml of the lead stock standard solution (4.27) into a calibrated 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

4.29 Lead standard solution B, 10 mg/l, prepared as follows:

using a calibrated pipette, transfer 100 ml of the lead stock standard solution (4.28) into a calibrated 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

4.30 Suitable solvent, appropriate for washing greasy or dirty test samples, e.g., acetone.

5 Apparatus

5.1 General

All volumetric glassware shall be class A and calibrated, in accordance with ISO 648 or ISO 1042 as appropriate.

Use ordinary laboratory apparatus other than volumetric glassware.

5.2 Inductively coupled plasma atomic emission spectrometer (ICP-AES)

5.2.1 General

The ICP-AES used shall be satisfactory only after optimizing according to the manufacture's instructions.

The spectrometer can be either the simultaneous or the sequential type. If a sequential spectrometer can be equipped with an extra arrangement for simultaneous measurement of the internal standard line, it can be used with the internal reference technique. If the sequential spectrometer is not equipped with this arrangement, an internal reference technique shall not be used.

5.2.2 Practical resolution of the spectrometer

Calculate the bandwidth (full width at half maximum) for the analytical line used including the line for internal reference. The bandwidth shall be less than 0,030 nm.

5.2.3 Short-term stability

Calculate the standard deviation of ten measurements of the absolute intensity or intensity ratio of the emitted light of the most concentrated calibration solution for analyte. The relative standard deviation shall not exceed 0,4 %.

5.2.4 Background equivalent concentration and detection limit

Calculate the background equivalent concentration (BEC) and detection limit (DL), for the analytical line in a solution containing only the analyte element. The values of BEC and DL shall be below the value listed in Table 2.

Table 2 — Background equivalent concentration, detection limit and characteristic mass

Element	Inductively coupled atomic emission spectrometry	
	Background equivalent concentration mg/l	Detection limit mg/l
Zinc	1,0	0,4
Aluminium	6,0	0,2
Iron	2,0	0,1
Silicon	3,0	0,1
Lead	2,5	0,1

5.3 Flame atomic absorption spectrometer (FAAS)

5.3.1 General

The FAAS used will be satisfactory only after optimizing according to the manufacturer's instructions.

5.3.2 Short-term stability

The standard deviation of ten measurements of the absorbance of the most concentrated calibration solution shall not exceed 1,5 % of the mean absorbance of the same solution.

The standard deviation of ten measurements of the absorbance of the least concentrated calibration solution shall not exceed 0,5 % of the mean absorbance of the same solution.

5.3.3 Detection limit

Calculate the detection limit (DL) for the analytical line in a solution containing only the analyte element. This is defined as three times the standard deviation of ten measurements of the absorbance of a solution containing the appropriate element at a concentration level selected to give an absorbance just above that of the zero member.

5.3.4 Calibration graph linearity

The slope of the calibration graph covering the top 20 % of concentration range (expressed as a change in absorbance) shall not be less than 0,7 times the value of the slope for the bottom 20 % of the concentration range determined in the same way. For instruments with automatic calibration using two or more standards, it shall be established prior to the analysis, by obtaining absorbance readings, that the above requirements for graph linearity are fulfilled.

5.3.5 Characteristics concentration

Calculate the characteristics concentration for the analyte in a matrix similar to the final test solution.

5.4 Platinum crucible

6 Sampling and samples

Sampling of test specimens for determining mass per unit area and chemical compositions of coating shall be carried out in accordance with ISO 14284 and products standards, if specified. Test specimens shall be of square, rectangular or round shape with a surface area of preferably 1 900 mm^2 to 3 500 mm^2. In case of dispute, test specimens shall be squares with sides of 50 mm ± 5 mm. One test specimen is required for each side to be determined.

7 Determination procedure

7.1 Sample preparation

Clean the specimens with suitable solvent (4.30) using a soft paper towel, then dry with oil-free compressed air.

Cover the side of the specimen from which the coating is not to be stripped with stop-off materials (4.9).

Use a roller, in the case of tape, to press the tape firmly against the sheet, making sure to remove all air bubbles or wrinkles. Trim off the excess tape.

Preferably protect the edges with stop-off materials (4.9).

7.2 Determination procedure for mass per unit area

Using a calliper traceable to national or International Standards, measure the size of sample to be stripped to the nearest 0,05 mm and calculate the area of the sample to the nearest 0,1 mm^2.

The area of the square test specimens shall be calculated as $(a + b) \times d/2$ when the test specimen is not really square and where d is the length of a diagonal line, and a and b are lengths of vertical lines from the corners to the diagonal line (see Figure 1). Dimensions a, b and d shall be measured to the nearest 0,05 mm using a device traceable to national or International Standards.

When the test specimen is not really square, if all corner angles are within a 4° deviation from 90°, the area of the square and rectangle test specimens may be calculated as $[(A + B) \times (C + D)]/4$, where A and B, C and D are lengths of opposite sides as shown in Figure 1.

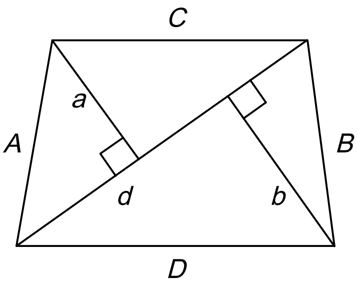

Figure 1 — Dimensions of square test specimens

Using a balance traceable to national or International Standards, weigh the prepared specimen to the nearest 0,1 mg and record the weight as the original weight of the specimen.

Place the sample in a sufficiently large beaker, e.g., 600 ml, with the protected coating side down.

Slowly add 30 ml of stripping solution (4.8) (see Note 2) at room temperature and leave until the coating has completely dissolved. The end of the dissolution process can be recognized by the cessation of the initially brisk evolution of hydrogen.

NOTE 1 The stripping time will depend on the chemical composition of the coating, its mass per unit area and the room temperature. Dilution of the stripping solution (4.8) may be acceptable in order to prolong the stripping time for thinner coatings.

NOTE 2 In the process of dissolving the coating layer, determination of the end point should be confirmed with the intent of optimizing the stripping time and hydrochloric acid concentration for each type of sample.

After the coating is stripped, remove the sample by an appropriate method. Holding it over the same beaker, rinse it carefully with water, and brush the stripped side to remove any loose substance which may be adhering to the surface and, if necessary, dip in alcohol.

Dry the stripped specimen with oil-free compressed air and weigh it to the nearest 0,1 mg using a balance traceable to national or International Standards.

Reserve this sample solution for the determination of chemical composition.

7.3 Procedure of determination of chemical composition by inductively coupled plasma atomic emission spectrometric method

7.3.1 Test solution for determination of zinc, aluminium, nickel, iron and lead contents

Use the sample solution reserved in 7.2 as test solution. If some undissolved substance remains in the solution, warm the beaker on a hot plate until all the stripped coating is dissolved. Transfer the solution to a 100 ml volumetric flask, dilute to the mark and mix thoroughly.

If the analyte content in the test solution is estimated to be greater 25 mg (see Table 3) or is too high to apply calibration series given in 7.3.5, pipette 10 ml of the sample solution into a volumetric flask, dilute to the mark and mix.

Record the dilution factor, D, as the volume of a volumetric flask divided by 10 ml.

7.3.2 Test solution for determination of zinc, aluminium, nickel, iron, lead and silicon contents

Use the sample solution reserved in 7.2 as test solution. If some undissolved substance remains in the solution, warm the beaker on a hot plate until all the stripped coating is dissolved.