INTERNATIONAL STANDARD

ISO 17925

First edition
2004-11-01

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

Reference number
ISO 17925:2004(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

© ISO 2004

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS

Not for Resale

Contents

Page

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*.

INTERNATIONAL STANDARD **ISO 17925:2004(E)**

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.

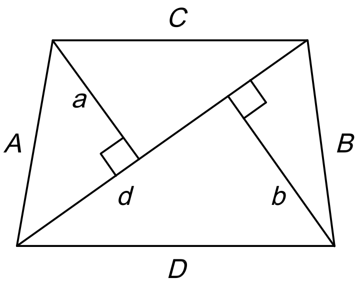


Figure 1 — Dimensions of square test specimens

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.

Filter the sample solution through a medium filter paper, wash using hydrochloric acid (1+10) (4.2) a few times with warm water. Transfer all filtrated solution to a 100 ml volumetric flask, dilute to the mark and mix (test solution No. 1).

Place the residue together with the filter paper in a platinum crucible (5.4). After drying, incinerate at a temperature of 800 °C and cool the resultant fly ash in air. Fuse with 2 g of sodium carbonate (4.6) at 1 050 °C and cool in air. Dissolve by 50 ml of mixed acid (4.5). Transfer to a 100 ml volumetric flask, dilute to the mark and mix (test solution No. 2).

7.3.3 Optimization of spectrometer

Start the ICP-AES or FAAS. Allow enough time for the apparatus to become stable before taking any measurements.

Optimize the instrument in accordance with the manufacturer's instructions.

Prepare the software to measure the intensity, mean value and relative standard deviation of the analytical lines.

If an internal standard is used, prepare the software to calculate the ratio between analyte intensity and internal standard intensity. The intensity of the internal standard shall be measured simultaneously with the analyte intensity.

Check the instrument performance requirements given in 5.2.2 to 5.2.4 for ICP-AES and 5.3.2 to 5.3.5 for FAAS.

7.3.4 Blank test

7.3.4.1 General

In parallel with each determination of test solutions, carry out a blank test with the same procedure and apparatus using a blank test solution.

7.3.4.2 Blank test solution

Put 30 ml of stripping solution (4.8) into a beaker. Add an adequate amount of the zinc standard solution for matrix matching (4.13) and/or aluminium standard solution (4.17) so that zinc and/or aluminium content is equivalent to those in the test solutions.

Transfer to a 100 ml volumetric flask and dilute to the mark with water and mix.

If the test solution is to be diluted, the blank test solution shall also be diluted.

7.3.5 Establishment of the ICP calibration graph

7.3.5.1 Calibration series

Some examples of calibration series are described below as series C-1 to C-4. However, each laboratory may establish its own calibration series combining the series C-1 to C-4 (see Annex A).

7.3.5.2 ICP Calibration series C-1

To each of the 100 ml volumetric flasks, pipette the stock standard solution of each analyte as indicated in Table 3. Add 30 ml of stripping solution (4.8). Then add an adequate amount of zinc standard solution for matrix matching (4.13) and/or aluminium standard solution (4.17) so that zinc and/or aluminium content is equivalent to those in the test solutions. To determine the silicon content of test solution No. 2, add an equivalent amount of sodium carbonate (4.6) and mixed acid (4.5). Dilute to the mark and mix.

Table 3 — Calibration series C-1

Calibration solution C-1	Volume of stock standard solution[a] added ml	Mass of analyte in 100 ml of standard solution mg
C-1-1	0	0
C-1-2	5	5
C-1-3	10	10
C-1-4	15	15
C-1-5	20	20
C-1-6	25	25

[a] 4.10, 4.14, 4.18, 4.21, 4.24 or 4.27.

Subtract the mean absolute intensity, I_0, of the zero member from the mean absolute intensity, I_i, of each solution giving the net absolute intensity, I_N.

$$I_N = I_i - I_0$$

Prepare a calibration graph by making a linear regression through the points with the net intensities on the Y-axis and the concentration, expressed as micrograms per millilitre, of each element to be determined in the calibration solution on the X-axis.

7.3.5.3 ICP Calibration series C-2

To each 100 ml volumetric flask, pipette the stock standard solution of each analyte as indicated in Table 4. Add 30 ml of stripping solution (4.8). Then add an adequate amount of zinc standard solution for matrix matching (4.13) and/or aluminium standard solution (4.17) so that zinc and/or aluminium content is equivalent to those in the test solutions. To determine the silicon content of test solution No.2, add an equivalent amount of sodium carbonate (4.6) and mixed acid (4.5). Dilute to the mark and mix.

Table 4 — Calibration series C-2

Calibration solution C-2	Volume of stock standard solution[a] added ml	Mass of analyte in 100 ml of standard solution mg
C-2-1	0	0
C-2-2	1	1
C-2-3	2	2
C-2-4	3	3
C-2-5	4	4
C-2-6	5	5

[a] 4.10, 4.14, 4.18, 4.21, 4.24 or 4.27.

Subtract the mean absolute intensity, I_0, of the zero member from the mean absolute intensity, I_i, of each solution giving the net absolute intensity, I_N.

$$I_N = I_i - I_0$$

Prepare a calibration graph by making a linear regression through the points with the net intensities on the Y-axis and the concentration, expressed as micrograms per millilitre, of each element to be determined in the calibration solution on the X-axis.

7.3.5.4 ICP Calibration series C-3

To each 100 ml volumetric flask, pipette standard solution A of each analyte as indicated in Table 5. Add 30 ml of stripping solution (4.8). Then add an adequate amount of zinc standard solution for matrix matching (4.13) and/or aluminium standard solution (4.17) so that the zinc and/or aluminium content is equivalent to those in the test solutions. To determine the silicon content of test solution No. 2, add an equivalent amount of sodium carbonate (4.6) and mixed acid (4.5). Dilute to the mark and mix.

Table 5 — Calibration series C-3

Calibration solution C-3	Volume of standard solution A[a] added ml	Mass of analyte in 100 ml of standard solution mg
C-3-1	0	0
C-3-2	1	0,1
C-3-3	2	0,2
C-3-4	4	0,4
C-3-5	6	0,6
C-3-6	8	0,8
C-3-7	10	1

[a] 4.11, 4.15, 4.19, 4.22, 4.25 or 4.28.

Subtract the mean absolute intensity, I_0, of the zero member from the mean absolute intensity, I_i, of each solution giving the net absolute intensity, I_N.

$$I_N = I_i - I_0$$

Prepare a calibration graph by making a linear regression through the points with the net intensities on the Y-axis and the concentration, expressed as micrograms per millilitre, of each element to be determined in the calibration solution on the X-axis.

7.3.6 Establishment of the FAAS calibration graph

7.3.6.1 Calibration series

Some examples of calibration series are described below as series C-1 to C-4. However each laboratory may establish its own calibration series combining the series C-1 to C-4 (see Annex A).

7.3.6.2 FAAS Calibration series C-1

To each 100 ml volumetric flask, pipette the stock standard solution of each analyte as indicated in Table 6. Add 30 ml of stripping solution (4.8). Then add an adequate amount of zinc standard solution for matrix matching (4.13) and/or aluminium standard solution (4.17) so that the zinc and/or aluminium content is equivalent to those in the test solutions. To determine the silicon content of test solution No. 2, add an equivalent amount of sodium carbonate (4.6) and mixed acid (4.5). Dilute to the mark and mix.

Table 6 — Calibration series C-1

Calibration solution C-1	**Volume of stock standard solution[a] added** ml	**Mass of analyte in 100 ml of standard solution** mg
C-1-1	0	0
C-1-2	5	5
C-1-3	10	10
C-1-4	15	15
C-1-5	20	20
C-1-6	25	25

[a] 4.14, 4.18, 4.21, 4.24 or 4.27.

Subtract the mean absolute intensity, I_0, of the zero member from the mean absolute intensity, I_i, of each solution giving the net absolute intensity, I_N.

$$I_N = I_i - I_0$$

Prepare a calibration graph by making a linear regression through the points with the net intensities on the Y-axis and the concentration, expressed as micrograms per millilitre, of each element to be determined in the calibration solution on the X-axis.

7.3.6.3 FAAS Calibration series C-2

To each 100 ml volumetric flask, pipette the stock standard solution of each analyte as indicated in Table 7. Add 30 ml of stripping solution (4.8). Then add an adequate amount of zinc standard solution for matrix matching (4.13) and/or aluminium standard solution (4.17) so that the zinc and/or aluminium content is equivalent to those in the test solutions. To determine the silicon content of test solution No. 2, add equivalent amount of sodium carbonate (4.6) and mixed acid (4.5). Dilute to the mark and mix.

Table 7 — Calibration series C-2

Calibration solution C-2	**Volume of stock standard solution[a] added** ml	**Mass of analyte in 100 ml of standard solution** mg
C-2-1	0	0
C-2-2	1	1
C-2-3	2	2
C-2-4	3	3
C-2-5	4	4
C-2-6	5	5

[a] 4.15, 4.19, 4.22, 4.25 or 4.28.

Subtract the mean absolute intensity, I_0, of the zero member from the mean absolute intensity, I_i, of each solution giving the net absolute intensity, I_N.

$$I_N = I_i - I_0$$

Prepare a calibration graph by making a linear regression through the points with the net intensities on the Y-axis and the concentration, expressed as micrograms per millilitre, of each element to be determined in the calibration solution on the X-axis.

7.3.6.4 FAAS Calibration series C-3

To each 100 ml volumetric flask, pipette the standard solution A of each analyte as indicated in Table 8. Add 30 ml of stripping solution (4.8). Then add an adequate amount of zinc standard solution for matrix matching (4.13) and/or aluminium standard solution (4.17) so that the zinc and/or aluminium content is equivalent to those in the test solutions. To the determine the silicon content of test solution No. 2, add equivalent amount of sodium carbonate (4.6) and mixed acid (4.5). Dilute to the mark and mix.

Table 8 — Calibration series C-3

Calibration solution C-3	Volume of standard solution A[a] added ml	Mass of analyte in 100 ml of standard solution mg
C-3-1	0	0
C-3-2	1	0,1
C-3-3	2	0,2
C-3-4	4	0,4
C-3-5	6	0,6
C-3-6	8	0,8
C-3-7	10	1

[a] 4.15, 4.19, 4.22, 4.25 or 4.28.

Subtract the mean absolute intensity, I_0, of the zero member from the mean absolute intensity, I_i, of each solution giving the net absolute intensity, I_N.

$$I_N = I_i - I_0$$

Prepare a calibration graph by making a linear regression through the points with the net intensities on the Y-axis and the concentration, expressed as micrograms per millilitre, of each element to be determined in the calibration solution on the X-axis.

7.3.7 Determination of zinc, aluminium, nickel and iron contents in test solutions

After establishing the calibration graphs (7.3.5 or 7.3.6), examine the readings for the blank solution (7.3.4) and for the unknown test solution (7.3.2) followed by the calibration solutions (7.3.5 – Tables 3 to 5 or 7.3.6 – Tables 6 to 8) of each analyte.

If the readings of the calibration solutions are not within ± 2 % of the first value obtained when establishing the calibration graph, recalibrate the instrument and run the samples.

Obtain the content of each analyte in milligrams per litre in the blank solution and in the unknown test solution by plotting the signal of the blank solution and of the unknown test solution on the calibration graph.

7.3.8 Determination of silicon content in test solutions

After establishing the calibration graph (7.3.5 or 7.3.6), examine the readings for the unknown test solutions No. 1 and No. 2 (7.3.2), as in 7.3.6 followed by the calibration solutions (7.3.5 – Tables 3 to 5 or 7.3.6 – Tables 6 to 8) of each analyte.

If the readings of the calibration solutions are not within ± 2 % of the first value obtained when establishing the calibration graph, recalibrate the instrument and run the samples.

Obtain the content of each analyte in milligrams per litre in blank solution and unknown test solution by plotting the signal of blank solution and of the unknown test solution on the calibration graph.

7.4 Procedure of determination of chemical composition by flame atomic absorption method — zinc, aluminium, nickel and iron 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 than 25 mg (see Table 3) or is too high to apply the 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.

8 Expression of results

8.1 Expression of result in mass per unit area

8.1.1 Method of calculation

Calculate the mass per unit area, C, of the coating, expressed in grams per square meter, from the equation:

$$C = \frac{\left(m_1 - m_2\right) \times 10^6}{A}$$

where

m_1 is the mass, in grams, of the sample before stripping;

m_2 is the mass, in grams, of the sample after stripping;

A is the area, in square millimetres, of the exposed surface of the sample.

8.1.2 Precision

A planned trial of this method was carried out by 15 laboratories in 9 countries at 9 levels of mass per unit area, each laboratory making three determinations (see Notes 1 and 2).

The test samples used are listed in Table B.1.

The results obtained were treated statistically in accordance with ISO 5725-1, ISO 5725-2 and ISO 5725-3.

The data obtained show a logarithmic relationship between mass per unit area and repeatability limit, r, and reproducibility limits, R and R_w, as summarized in Table 9 (see Note 3). The graphical representation of the data is given in Annex C.

The precision obtained for hot-dip coated steel was found to be insufficient. This was due, not to the measurement method, but to low homogeneity of samples, which were prepared from commercial coating steel. Therefore the precision data obtained for hot-dip coated sample are merely informative.

NOTE 1 Two of the three determinations were carried out under repeatability conditions as defined in ISO 5725-1, i.e., one operator, same apparatus, identical operating conditions, same calibration, and a minimum period of time.

NOTE 2 The third determination was carried out at a different time (on a different day) by the same operator as in Note 1, using the same apparatus with a new calibration.

NOTE 3 From the results obtained on day 1, the repeatability limit, r, and reproducibility limit, R, were calculated using the procedure specified in ISO 5725-2. From the first result obtained on day 1 and the result obtained on day 2, the within-laboratory reproducibility limit, R_w, was calculated using the procedure given in ISO 5725-3.

Table 9 — Mass per unit area of repeatability limit and reproducibility limits

Mass per unit area	**Electrolytic coated steel** (normative)			**Hot-dip coated steel** (informative)		
	Repeatability limit	**Reproducibility limits**		**Repeatability limit**	**Reproducibility limits**	
g/m^2	r	R_w	R	r	R_w	R
15	1,26	1,66	3,28	—	—	—
25	1,26	1,66	3,28	—	—	—
50	—	—	—	6,76	8,92	13,98
100	—	—	—	6,76	8,92	13,98
150	—	—	—	6,76	8,92	13,98

8.2 Expression of result of chemical compositions

8.2.1 Method of calculation

Subtract the content of the analyte in the blank solution (see 7.3.4) from that in the test solution determined in 7.3.6 and 7.3.7.

The analyte content, w, expressed as a percentage by mass, is given by the equation:

$$w = \frac{(M_1 - M_0) \times D}{(m_1 - m_2) \times 100}$$

where

M_1 is the analyte concentration, in milligrams per litre, in the test solution;

M_0 is the analyte concentration, in milligrams per litre, in the blank test solution;

D is the dilution factor (see 7.3.1);

m_1 is the mass, in grams, of the sample before stripping (see 8.1.1);

m_2 is the mass, in grams, of the sample after stripping (see 8.1.1).

For silicon content, determine the sum of the values, w, obtained by test solutions No. 1 and No. 2.

8.2.2 Precision

A planned trial of this method was carried out by 15 laboratories in 9 countries, each laboratory making three determinations (see Notes 1 and 2 of 8.1.2).

The test samples used are listed in Table B.1.

The results obtained were treated statistically in accordance with ISO 5725-1, ISO 5725-2 and ISO 5725-3.

The data obtained show a logarithmic relationship between analyte content and repeatability limit, r, and reproducibility limits, R and R_w, as summarized in Tables 10 to 14 (see Note 3 of 8.1.2). The graphical representations of the data are given in Annex C.

Table 10 — Zinc content and repeatability limit and reproducibility limits (only for ICP)

Zinc content	**Repeatability limit**	**Reproducibility limits**	
% (mass fraction)	r	R_w	R
40,0	0,990	1,274	2,523
50,0	1,088	1,418	2,831
100,0	1,460	1,978	4,053

Table 11 — Aluminium content and repeatability limit and reproducibility limits

Aluminium content	**Repeatability limit**	**Reproducibility limits**	
% (mass fraction)	r	R_w	R
0,02	0,002	0,003	0,008
0,05	0,003	0,007	0,016
0,1	0,006	0,012	0,027
0,2	0,011	0,020	0,047
0,5	0,024	0,041	0,096
1,0	0,044	0,070	0,165
2,0	0,081	0,121	0,284
5,0	0,178	0,246	0,582
10,0	0,324	0,423	1,000
20,0	0,592	0,725	1,719
50,0	1,307	1,480	3,519
60,0	1,531	1,707	4,058

Table 12 — Iron content and repeatability limit and reproducibility limits

Iron content % (mass fraction)	**Repeatability limit** r	**Reproducibility limits** R_w	 R
0,2	0,063	0,076	0,185
0,5	0,129	0,150	0,375
1,0	0,221	0,250	0,640
2,0	0,380	0,418	1,093
5,0	0,776	0,824	2,216
10,0	1,333	1,377	3,783
20,0	2,290	2,300	6,458

Table 13 — Silicon content and repeatability limit and reproducibility limits

Silicon content % (mass fraction)	**Repeatability limit** r	**Reproducibility limits** R_w	 R
0,20	0,025	0,037	0,183
0,50	0,052	0,072	0,308
1,00	0,090	0,118	0,456
2,00	0,156	0,194	0,675
5,00	0,322	0,374	1,135
10,00	0,557	0,616	1,682

Table 14 — Nickel content and repeatability limit and reproducibility limits

Nickel content % (mass fraction)	**Repeatability limit** r	**Reproducibility limits** R_w	 R
13,12	0,37	0,46	1.41

9 Test report

The test report shall include the following information:

a) all information necessary for the identification of the sample, the laboratory and the date of analysis;

b) the method used by reference to this International Standard, i.e. ISO 17925;

c) the results and the form in which they are expressed;

d) the analytical line used;

e) any unusual features noted during the determination;

f) any operation not specified in this International Standard, or any optional operation, which may have influenced the results.

Annex A
(informative)

Analyte content

Analyte content in test solutions, relating to the coating mass and the analyte percent in coating, is shown in Table A.1. In this table, coating mass on samples means coating mass stripped from 50 mm square samples.

Table A.1 — Analyte content

Coating mass g/m^2	**Coating mass on samples** mg	**Analyte in coating** % (mass fraction)								
		0,01	0,02	0,1	0,2	1	2	10	20	100
		Analyte content in 50 mm square samples mg								
20	50	0,005	0,01	0,05	0,1	0,5	1	5	10	50
40	100	0,01	0,02	0,10	0,2	1,0	2	10	20	100
60	150	0,015	0,03	0,15	0,3	1,5	3	15	30	150
80	200	0,02	0,04	0,20	0,4	2,0	4	20	40	200
100	250	0,025	0,05	0,25	0,5	2,5	5	25	50	250
120	300	0,03	0,06	0,30	0,6	3,0	6	30	60	300
140	350	0,035	0,07	0,35	0,7	3,5	7	35	70	350
160	400	0,04	0,08	0,40	0,8	4,0	8	40	80	400
180	450	0,045	0,09	0,45	0,9	4,5	9	45	90	450
200	500	0,05	0,10	0,50	1,0	5,0	10	50	100	500

Annex B
(informative)

Additional information on the international cooperative tests

The repeatability and reproducibility data in Tables 9 to 14 were derived from the results of international analytical trials carried out in 1999 on 10 steel samples in 9 countries involving 15 laboratories.

The results of the trials were reported in document ISO/TC 17/SC 1 N 1274, August 10. The graphical representation of the precision data is given in Annex C.

The test samples used are listed in Table B.1. Precision data are listed in Tables B.2 to B.7.

Table B.1 — Test samples

Samples	**Coating mass** g/m^2	**Estimated chemical composition** % (mass fraction)				
		Zn	**Fe**	**Al**	**Ni**	**Si**
511 Zn (Hot-dip)	140	99	0,1	0,4		
512 Zn-Fe (Hot-dip)	60	88	12	0,27		
513 Zn (Electrolytic)	20	99	0,1			
514 Zn-Ni (Electrolytic)	20	86	0,4		12	
515 Zn-5%Al (Hot-dip)	130	95	0,2	4		0,06
516 Zn-55%Al (Hot-dip)	80	43	2,0	55		1,6
517 Al (Hot-dip)	32	not reported	0,5	89		10
518 Zn (Hot-dip)	110	99	less than 0,01	0,45		
519 Zn-55%Al (Hot-dip)	75	43,5	0,4	54		2

Table B.2 — Samples and precision data for coating mass

Samples	Mass per unit area g/m^2	Repeatability limit r	Reproducibility limits R_w	 R
513	17,86	1,35	1,84	3,03
514	20,46	1,17	1,47	3,53
517	37,92	3,91	23,72	22,48
512	57,15	5,93	6,37	8,32
519	75,90	4,50	6,58	12,72
516	80,82	5,10	6,98	9,23
518	116,86	5,65	7,46	14,34
515	131,20	9,66	11,09	22,22
511	139,31	9,70	15,09	17,05

Table B.3 — Samples and precision data for zinc

Samples	Zinc content % (mass fraction)	Repeatability limit r	Reproducibility limits R_w	 R
516	42,36	0,75	1,13	2,59
519	44,04	2,06	1,48	2,76
514	86,40	2,37	5,11	8,72
512	88,45	1,88	2,48	5,69
515	95,21	1,10	2,44	2,24
513	98,70	2,31	1,75	3,19
518	99,04	1,20	1,82	2,36
511	99,06	0,19	0,50	2,13

Table B.4 — Samples and precision data for aluminium

Samples	Aluminium content % (mass fraction)	Repeatability limit r	Reproducibility limits R_w	 R
512	0,267	0,023	0,035	0,143
518	0,543	0,024	0,029	0,064
515	4,06	0,14	0,20	0,32
519	52,82	1,15	1,29	3,75
516	54,72	1,12	1,08	3,51
517	81,73	3,03	3,97	7,14

Table B.5 — Samples and precision data for nickel

Samples	Nickel content % (mass fraction)	Repeatability limit r	Reproducibility limits	
			R_w	R
514	13,12	0,37	0,46	1,41

Table B.6 — Samples and precision data for iron

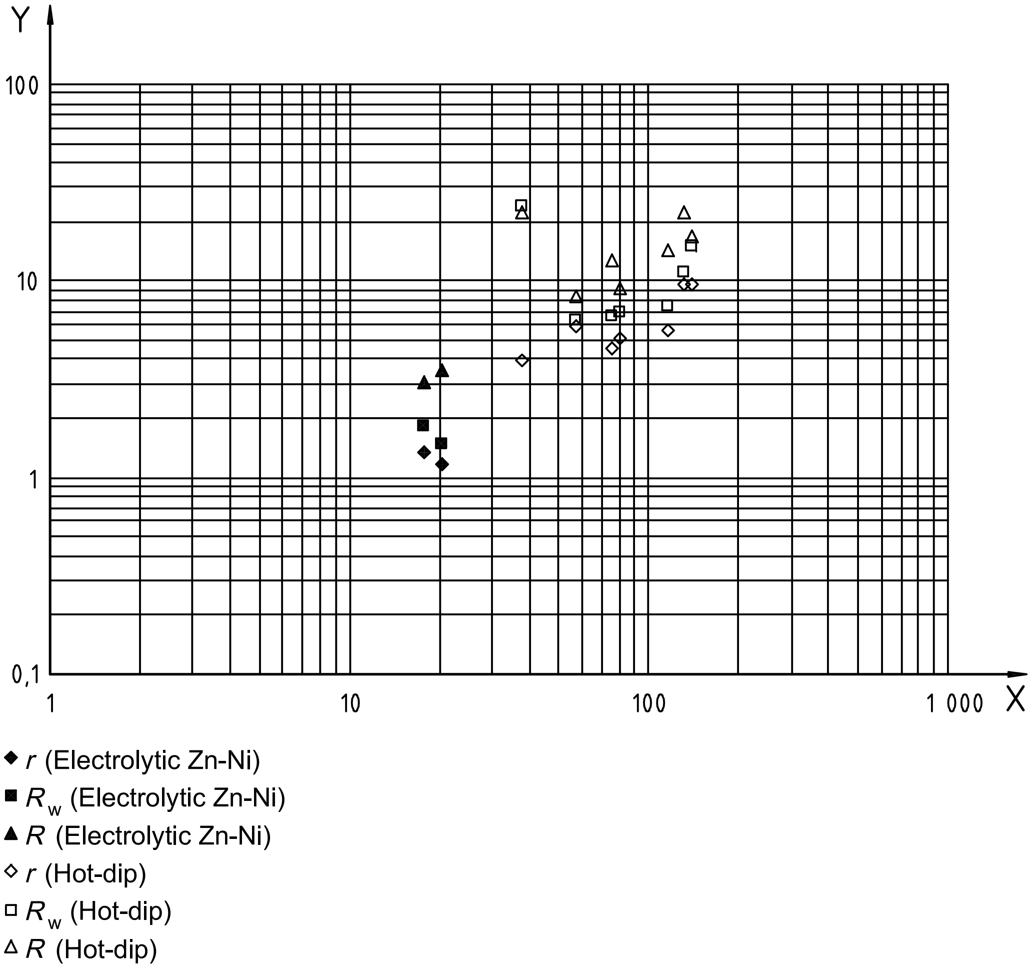
Samples	Iron content % (mass fraction)	Repeatability limit r	Reproducibility limits	
			R_w	R
515	0,073	0,016	0,023	0,096
518	0,100	0,026	0,024	0,056
511	0,121	0,045	0,049	0,063
513	0,325	0,146	0,239	0,488
514	0,347	0,168	0,170	0,806
519	1,04	0,10	0,15	0,41
516	1,28	0,10	0,13	0,32
517	7,71	1,32	1,48	7,25
512	11,93	1,13	0,85	1,84

Table B.7 — Samples and precision data for silicon

Samples	Silicon content % (mass fraction)	Repeatability limit r	Reproducibility limits	
			R_w	R
515	0,028	0,005	0,009	0,074
516	1,31	0,08	0,15	0,43
519	1,61	0,18	0,15	0,34
517	7.81	0.45	0.54	2.49

Annex C
(informative)

Graphical representation of precision data

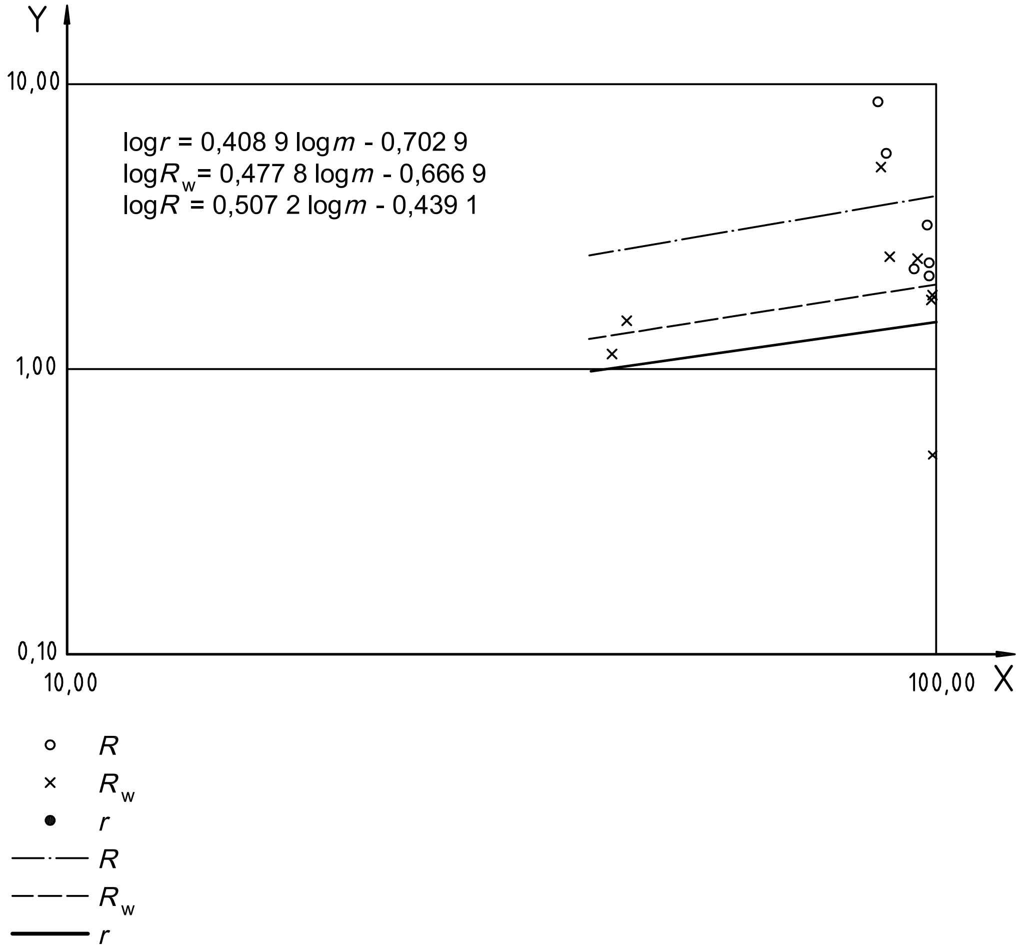


Key

X coating mass, g/m^2

Y precision, g/m^2

Figure C.1 — Precision data for coating mass

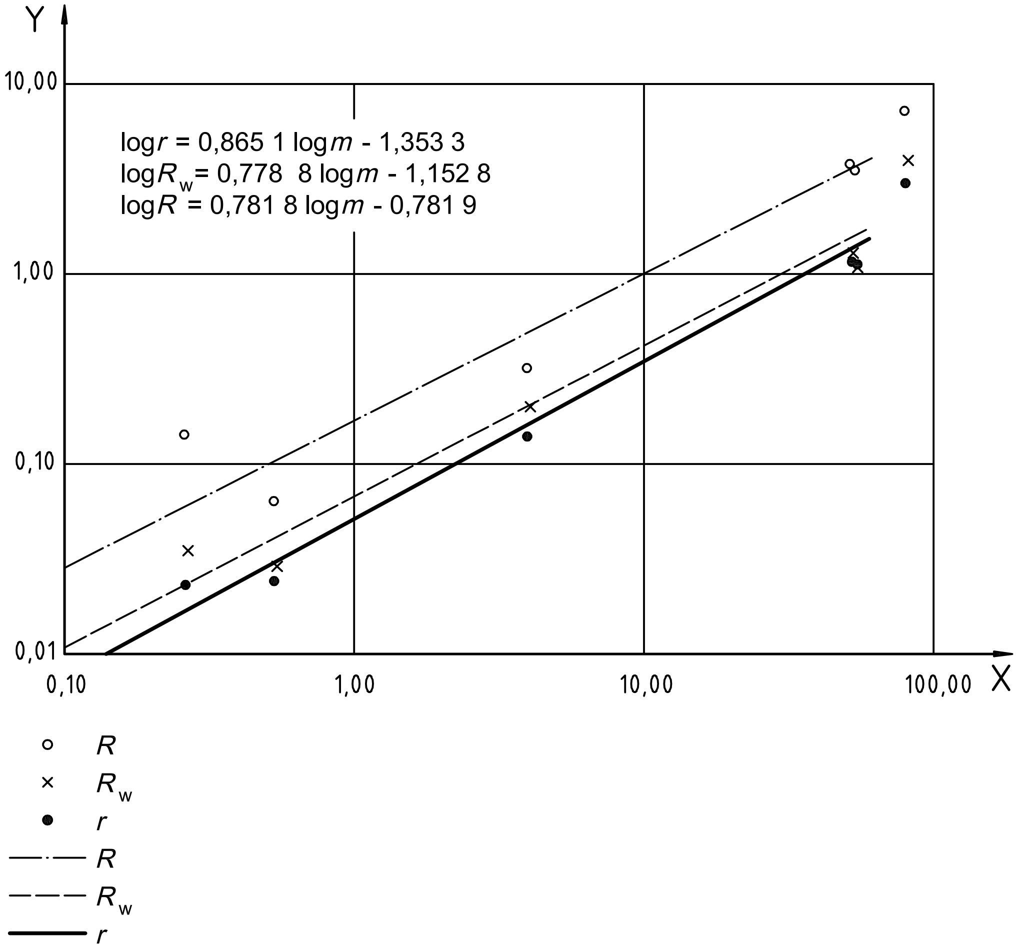


Key

X zinc content, % (mass fraction)

Y precision, % (mass fraction)

Figure C.2 — Precision data for zinc

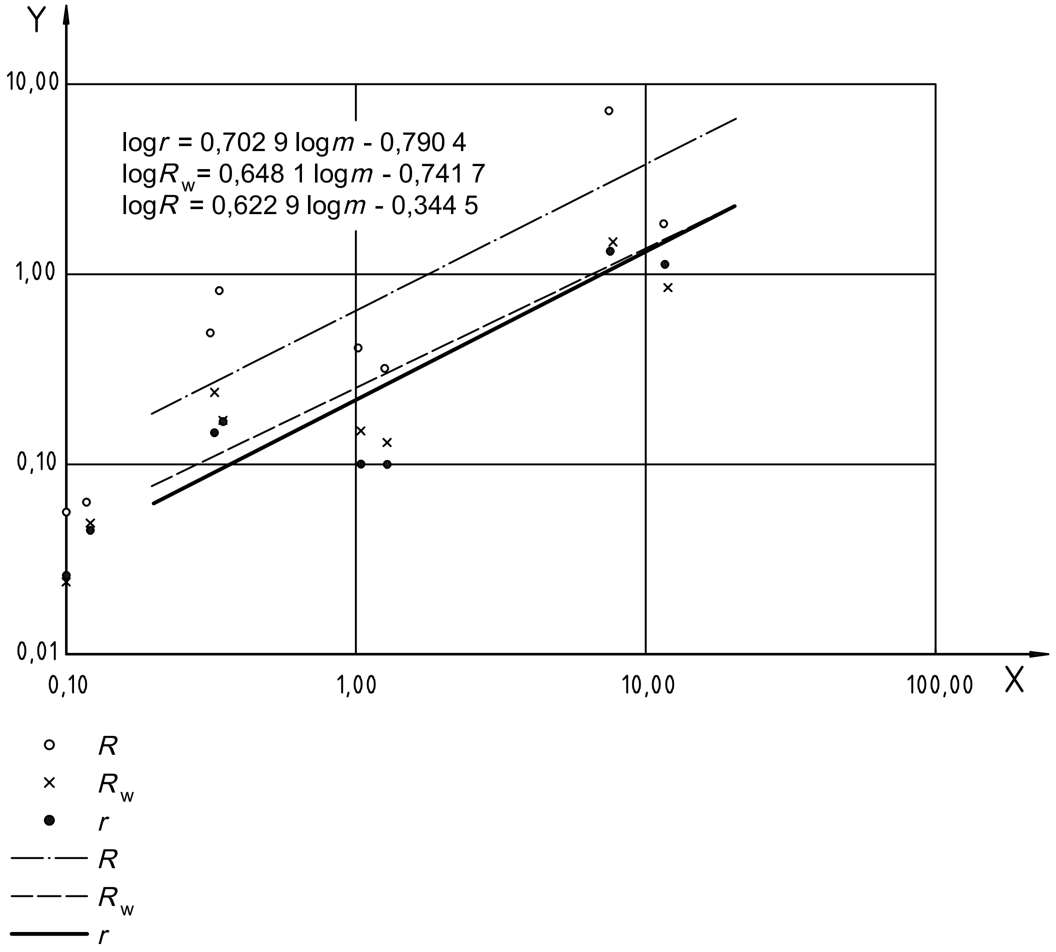


Key

X aluminium content, % (mass fraction)

Y precision, % (mass fraction)

Figure C.3 — Precision data for aluminium

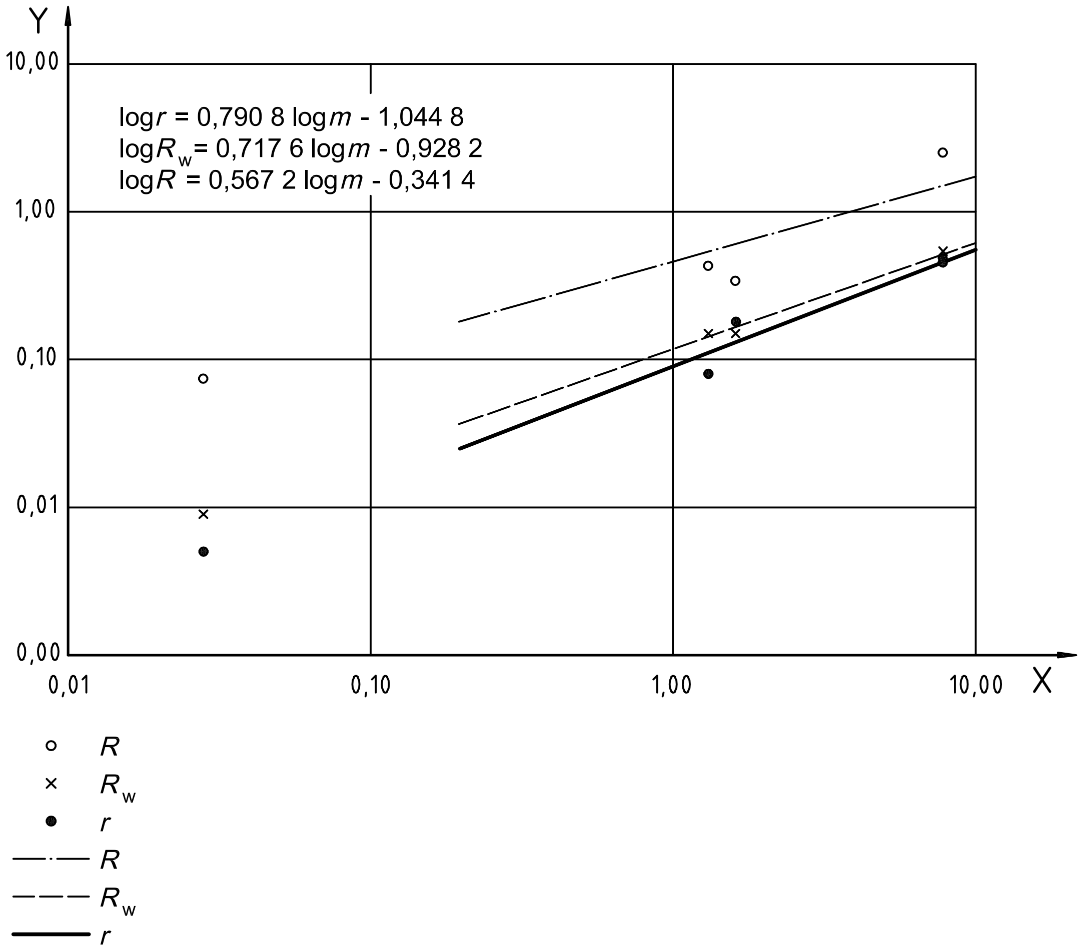


Key

X iron content, % (mass fraction)

Y precision, % (mass fraction)

Figure C.4 — Precision data for iron



Key

X silicon content, % (mass fraction)

Y precision, % (mass fraction)

Figure C.5 — Precision data for silicon

ICS 25.220.40; 77.080.20

Price based on 26 pages