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Steel wire and wire products - Non-ferrous metallic coatings on steel wire - Part 2: Zinc or zinc alloy coatings

Fils et produits tréfilés en acier - Revêtements métalliques non ferreux sur fils d'acier - Partie 2 : Revêtement de zinc ou d'alliage de zinc

Stahldraht und Drahterzeugnisse - Überzüge aus Nichteisenmetall auf Stahldraht - Teil 2: Überzüge aus Zink und Zinklegierungen

This European Standard was approved by CEN on 20 December 2008.

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Foreword

This document (EN 10244-2:2009) has been prepared by Technical Committee ECISS/TC 30 “Steel wires”, the secretariat of which is held by AFN0R.

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of an identical text or by endorsement, at the latest by October 2009, and conflicting national standards shall be withdrawn at the latest by October 2009.

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

This document supersedes EN 10244-2:2001.

Some significant technical changes from the previous edition of this standard have been made like the addition of new mass requirements for a coating of Zn95Al5 (in Table 2, see E) and the correction of the Formula in 5.2.3.6 "Expression of results".

This European Standard for non-ferrous metallic coating on steel wire is made up of the following parts:

— *Part 1: General principles*

— *Part 2: Zinc or zinc alloy coatings*

— *Part 3: Aluminium coatings*

— *Part 4: Tin coatings*

— *Part 5: Nickel coatings*

— *Part 6: Copper, bronze or brass coatings*

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1 Scope

This part of this European Standard specifies the requirement for coating mass, other properties and testing of zinc and zinc alloy coatings on steel wire and steel wire products of circular or other section.

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.

EN 1179, *Zinc and zinc alloys — Primary zinc*

EN 10218-1, *Steel wire and wire products — General – Part 1: Test methods*

EN 10244-1:2009, *Steel wire and wire products — Non-ferrous metallic coatings on steel wire — Part 1: General principles*

ISO 7802, *Metallic materials - Wire - Wrapping test*

ISO 7989-2:2007, *Steel wire and wire products — Non-ferrous metallic coatings on steel wire — Part 2: Zinc or zinc-alloy coating*

ASTM B 750, *Standard Specification for GALFAN (Zinc-5% Aluminium-Mischmetal) Alloy in Ingot Form for Hot-Dip Coatings.*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
wire with zinc or zinc alloy coating
wire to which zinc or zinc alloy coating has first been applied to protect it against corrosion.

NOTE The coating method may be hot dipping in a bath of molten zinc or by means of an aqueous solution of suitable electrolyte. In the hot dipping process wiping media may be used to modify the mass of coating

3.2
zinc or zinc alloy coating
zinc to which deliberately other elements are added in order to obtain particular characteristics.

NOTE In all cases the quantity of zinc in the alloy shall be minimum 50 %. The most common alloy elements are aluminium, tin, nickel but other elements may also be considered

3.3
mass of coating
mass of zinc per unit of surface area expressed in grams per square metre of surface of bare wire

Table 1 — Mass requirements for a coating of Zn

Diameter d mm	Classes [a]					
	A g/m²	AB g/m²	B g/m²	C g/m²	D g/m²	A x 3 [b] g/m²
$0,15 \leq d < 0,20$	—	—	15	—	10	—
$0,20 \leq d < 0,25$	30	20	20	20	15	—
$0,25 \leq d < 0,32$	45	30	30	25	15	—
$0,32 \leq d < 0,40$	60	30	30	25	15	—
$0,40 \leq d < 0,50$	85	55	40	30	15	—
$0,50 \leq d < 0,60$	100	70	50	35	20	—
$0,60 \leq d < 0,70$	115	80	60	40	20	—
$0,70 \leq d < 0,80$	130	90	60	45	20	—
$0,80 \leq d < 0,90$	145	100	70	50	20	—
$0,90 \leq d < 1,00$	155	110	70	55	25	—
$1,00 \leq d < 1,20$	165	115	80	60	25	—
$1,20 \leq d < 1,40$	180	125	90	65	25	540
$1,40 \leq d < 1,65$	195	135	100	70	30	585
$1,65 \leq d < 1,85$	205	145	100	75	30	615
$1,85 \leq d < 2,15$	215	155	115	80	40	645
$2,15 \leq d < 2,50$	230	170	125	85	45	690
$2,50 \leq d < 2,80$	245	185	125	95	45	735
$2,80 \leq d < 3,20$	255	195	135	100	50	765
$3,20 \leq d < 3,80$	265	210	135	105	60	795
$3,80 \leq d < 4,40$	275	220	135	110	60	825
$4,40 \leq d < 5,20$	280	220	150	110	70	840
$5,20 \leq d < 8,20$	290	—	—	110	80	870
$8,20 \leq d \leq 10,00$	300	—	—	110	80	900

[a] The coating class with a designation starting with A relates to thick coatings (generally final coating). Designations ending in B relate to classes usually but not always obtained by (zinc coating) and subsequent drawing. Classes C and D are standard classes for low mass coating which are usually produced but not exclusively, produced by hot zinc dipping and then wiping.

[b] A x 3 relates to very high mass requirement three times higher than class A. Other multiples of Class A are possible and these classes will be identified in the same way, e.g. A x 4.

4 Coating requirements

4.1 Requirements for the coatings material

The zinc or zinc alloy used for the coating shall comply with the standard EN 1179. For zinc-alloy grades not mentioned in the EN the alloy shall be specified at the enquiry and order. For Zn95Al5 reference is made to ASTM B 750 with or without mischmetal (MM).[1]

1) Mischmetal is a mixture of rare earth metals (La and others) which is a by product from zinc production.

The ingot of the material used for the zinc coating shall be of minimum 99,95 % purity (according to Z3 of EN 1179) unless otherwise stated in the relevant product standard or other specification in the order. Coatings applied by electrolysis shall contain a minimum of 99 % zinc.

4.2 Requirements relating to coating on the wire

4.2.1 Coating mass

The minimum mass of zinc per unit of surface area of the wire shall comply with the requirements of Table 1.

If no class of zinc coating or no coating mass is specified the coating shall be called "regular coating". Such a coating shall be not less than 1 g zinc mass per Kg of zinc coated wire (equivalent to the coating mass in g/m^2 not less than two times the wire diameter expressed in mm).

The requirements for zinc-aluminium coatings of type Zn95Al 5 are given in Table 2.

For other zinc alloy coatings, the manufacturer and supplier shall agree on the required coating mass.

4.2.2 Appearance of coating

The coating applied to the wire shall be reasonably smooth and as evenly distributed as industrial technology allows and not show discontinuities such as bare patches, dross contamination, etc.

NOTE The Zn95Al5 zinc-aluminium alloy might show difference in colour and become darker with time. This does not affect the corrosion protection performance.

4.2.3 Dipping test

When specified the dipping (immersion) test shall be carried out according to the procedure detailed in 5.3. However, it should be pointed out that there is no correlation between the number of dips and the mass of the coating and that the result is influenced as much by the conditions of manufacture of the coating as by the uniformity of the coating.

Table 3 gives the minimum number of immersions for coatings of classes A and AB.

The dipping test does not apply to class B, C and D.

Table 2 — Mass requirements for a coating of Zn95Al5

Diameter mm	Mass of coating [a] g/m²			
	A	AB	B	E [b]
$0,20 \le d < 0,25$	—	20	20	40
$0,25 \le d < 0,40$	—	30	30	
$0,40 \le d < 0,50$	85	55	40	
$0,50 \le d < 0,60$	100	70	50	
$0,60 \le d < 0,70$	115	80	60	
$0,70 \le d < 0,80$	130	90	60	
$0,80 \le d < 0,90$	145	100	70	
$0,90 \le d < 1,00$	155	110	70	
$1,00 \le d < 1,20$	165	115	80	
$1,20 \le d < 1,40$	180	125	90	60
$1,40 \le d < 1,65$	195	135	100	
$1,65 \le d < 1,85$	205	145	100	
$1,85 \le d < 2,15$	215	155	115	
$2,15 \le d < 2,50$	230	170	125	
$2,50 \le d < 2,80$	245	185	125	
$2,80 \le d < 3,20$	255	195	135	
$3,20 \le d < 3,80$	265	210	135	
$3,80 \le d < 4,40$	275	220	135	
$4,40 \le d < 5,20$	280	220	150	
$5,20 \le d < 8,20$	290	—	—	
$8,20 \le d < 10,00$	300	—	—	

[a] The coating class with a designation starting with A relates to thick coatings (generally final coating). Designations ending in B relate to classes usually but not always obtained by (zinc coating) and subsequent drawing.

[b] the corrosion resistance of this class E must be at least equivalent of these from a zinc coating according Table 1 class B

Table 3 — Minimum number of dips

Nominal diameter mm	Class A of coating		Class AB of coating	
	Number of dips		Number of dips	
	of 1 min	of 1/2 min	of 1min	of 1/2 min
$0,40 \le d < 0,60$	—	1	—	—
$0,60 \le d < 0,90$	1	—	—	1
$0,90 \le d < 1,00$	1	1	—	1
$1,00 \le d < 1,40$	1	1	1	—
$1,40 \le d < 1,65$	2	—	1	—
$1,65 \le d < 1,85$	2	—	1	—
$1,85 \le d < 2,15$	2	—	1	1
$2,15 \le d < 2,80$	2	1	1	1
$2,80 \le d < 4,40$	3	—	2	—
$4,40 \le d < 5,20$	3	1	2	—
$5,20 \le d < 8,20$	3	1	—	—
$8,20 \le d < 10,00$	4	—	—	—

4.2.4 Special finishes

If drawing after galvanising is required it shall be stipulated at the time of enquiry and order or in the appropriate product standard. The same also applies for other special finishes such as wax coating, a polished surface or an exceptionally smooth surface.

4.2.5 Adherence of coating

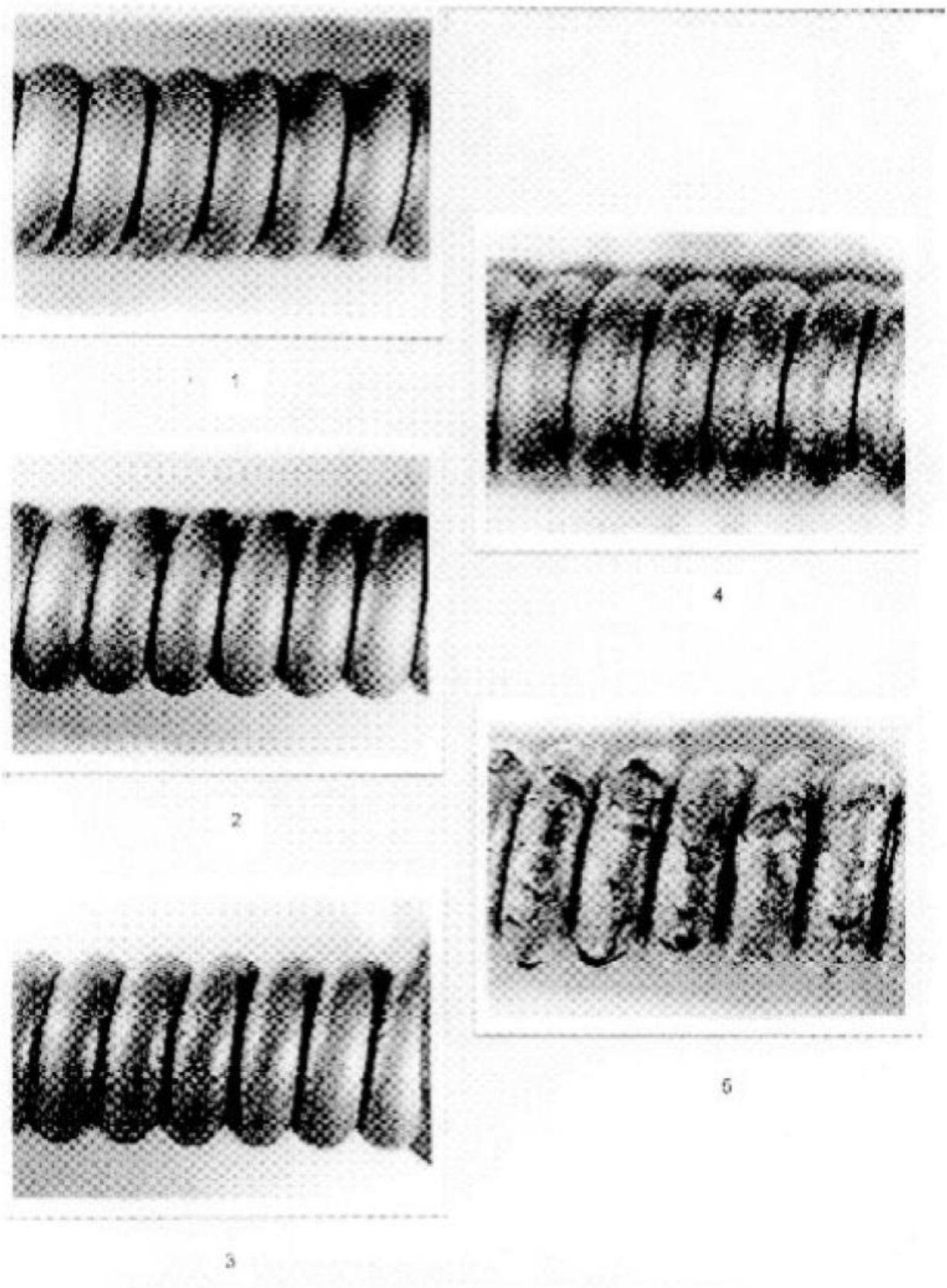
4.2.5.1 During the test carried out in accordance with EN 10218-1, the coating shall adhere to the steel when subjected to the conditions of wrapping test for adherence. It shall not crack or split to such an extent that slivers of coating can be removed by simply rubbing with the bare fingers. The loosening or detachment during testing of small particles of zinc resulting from mechanical polishing of the surface of the zinc or zinc alloy coating shall not be considered to be a cause for rejection.

The wrapping test is defined in ISO 7802 and specific requirements for wires are defined in EN 10244-1:2009, 5.3.

4.2.5.2 Assessment of adherence

To assess the adherence of the coating as may be specified in the relevant product standard or for evaluating different conditions of manufacturing the following procedure may be applied.

Coil the wire around its own diameter compare to the reference chart (see Figure 1). Allocate a value of 1 to 5 to the quality of adherence of the coating in accordance with the reference chart in Figure 1.



Key

Descending scale based on quality of adherence from 1 (highest quality) to 5 (lowest quality) (see 4.2.5.2)

Figure 1 — Assessment of the adherence of the coating

5 Test conditions

5.1 Selection of samples

5.1.1 The number and the basis for the selection of test pieces shall be defined in the product standard. In the absence of such a standard, the manufacturer and purchaser shall agree between them on the degree of sampling.

5.1.2 When taking the samples, care shall be taken to avoid damaging the surface. Parts of the wire which are visibly damaged shall not be used.

5.1.3 A test piece of wire, which is of a suitable length for the specified tests shall be taken from one or both ends of each reel selected for sampling.

In the case of coated wire products, the product standard shall specify the length of the samples to be taken.

5.2 Determination of mass of coating

5.2.1 General

In the case of wire with a zinc or zinc alloy coating, the mass of the coating shall be determined either by the gravimetric method or the volumetric method. The second offers an acceptable degree of accuracy and has the advantage of being quick. This is why it is the preferred method for routine testing of wire of all dimensions which can easily be fitted into the measuring equipment. In the event of any dispute, the gravimetric method shall be used as the reference test method.

NOTE the test methods in this standard do not necessarily include in detail all the precautions necessary to satisfy hygiene and safety requirements in the workplace, etc. Care should be taken to ensure that all necessary precautions are taken and procedures are implemented only by people who have received suitable training.

5.2.2 Gravimetric method

The method shall be applied in accordance with EN 10244-1 with the following additional requirements:

5.2.2.1 Reagents

The stripping solution shall be hydrochloric acid with a concentration of 1,13 g/ml to 1,19 g/ml by density to which an appropriate inhibitor is added. Dissolve 3,2 g of antimony chloride ($SbCl_3$) or 2 g antimony oxide (III) (Sb_2O_3) in 500 ml concentrated hydrochloric acid (ρ = 1,190 g/ml). Dilute this solution with distilled water to 1 l. Hexamethylenetetramine ($C_6H_{12}N_4$), formaldehyde (HCHO) or any other suitable inhibitor may be used.

NOTE It is recommended that preference be given to inhibitors which do not contain antimony.

5.2.2.2 Calculation of mass of coating

The coating mass of zinc or zinc alloy shall be calculated according the following formulae:

$$m_A = \frac{\Delta m}{A} \times 10^6 \qquad (1)$$

where

A is the coated surface of the sample in mm^2 (surface of the stripped wire);

Δm is mass loss of the sample by chemical stripping in grams;

m_A is coating mass in g/m^2.

or

$$m_A = 1\,962 \times d \times \frac{\Delta m}{m_2} \quad (2)$$

where

m_2 is the mass of the sample after chemical stripping expressed in grams;

d is the diameter of (round) wire in mm;

Δm is mass loss of the sample by chemical stripping in grams;

m_A is the coating mass in g/m^2.

5.2.3 Gaseous volumetric method

5.2.3.1 Principle

The volumetric method for determining the mass of the coating depends on the property by which a metal dissolved in an acid releases a quantity of hydrogen proportional to the mass of metal dissolved, i.e. representing the chemical equivalent of the metal in question.

In the case of zinc, this relationship is very simple. For zinc alloys, each case shall be examined individually on the basis of the metals present in the zinc alloy. The method for zinc and the alloy zinc 95 % - Aluminium 5 % only is described in detail

By measuring the volume of hydrogen released by dissolving the coating, the quantity of coating can be determined. By relating this result to the surface area of the test piece, measured once the coating has dissolved, the mass of the coating per unit area is obtained.

5.2.3.2 Reagents

Hydrochloric acid: The acid used to dissolve the coating is hydrochloric acid with a density between 1,13 g/ml and 1,19 g/ml.

NOTE The concentration of the acid may affect the result of the volumetric method; a powerful acid will produce slightly lower results.

Inhibitor: An inhibitor shall be added to the stripping solution to protect the steel base against dissolving. The inhibitors may be hexamethylene-tetramine ($C_6H_{12}N_4$), formaldehyde (HCHO) or any other suitable inhibitor.

5.2.3.3 Apparatus (see 5.2.3.3 of ISO 7989-2:2007)

The apparatus used is made up of the following items (see Figure 2):

1) the tube shall be graduated in millimetres. The tube has a tap at each end;

2) flask with an opening near the bottom, connected by rubber tube to the opening near the bottom of the graduated tube;

3) container to hold the test piece once the zinc coating or zinc-alloy coating has been removed.

5.2.3.4 Test pieces

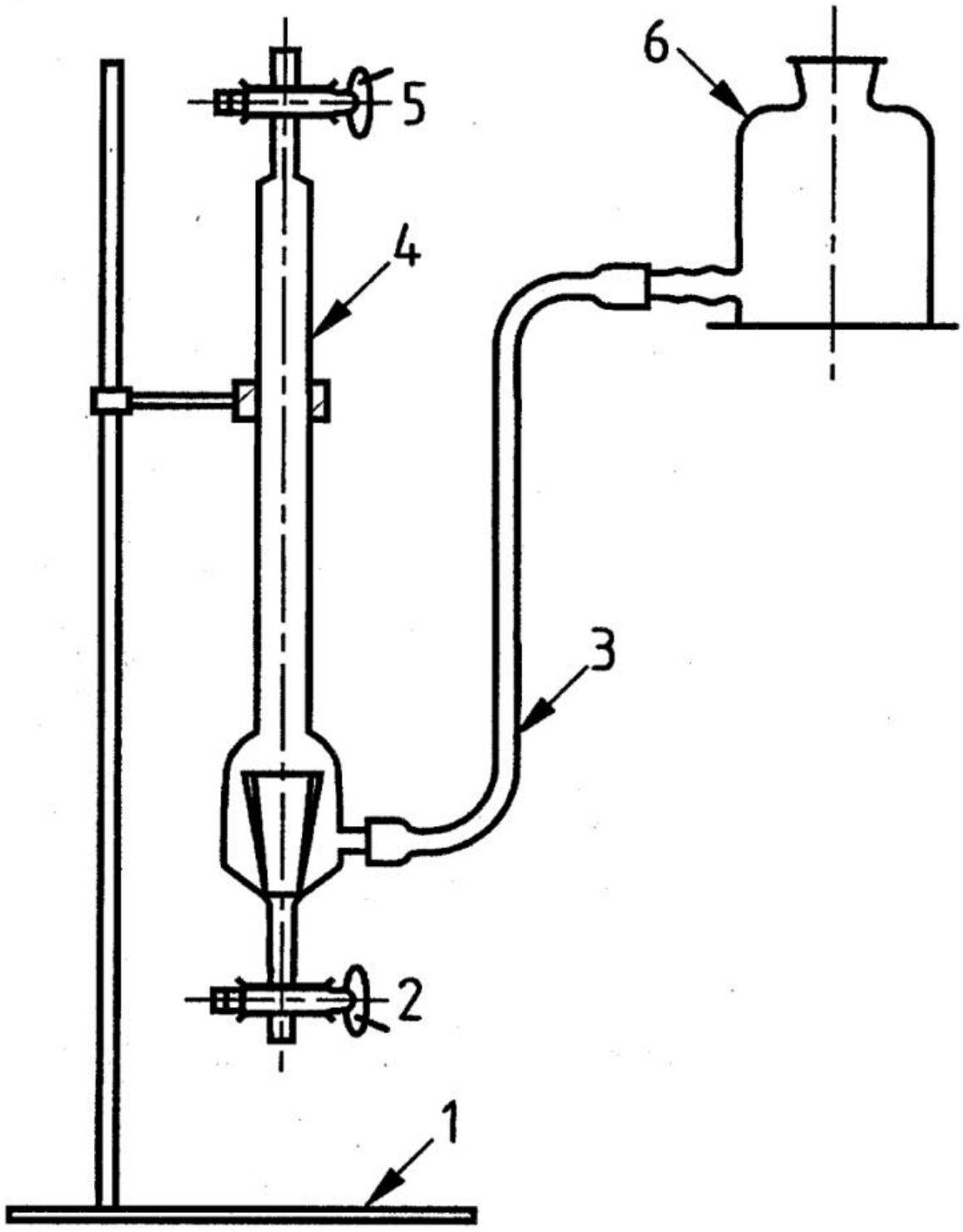
After carefully straightening out the wire samples, cut the test pieces accurately to a suitable length depending on the size of the tube, the diameter of the wire and the assumed mass of the coating. The lengths given in

Table 4 are generally applicable for proper gas release. Different lengths may be necessary depending on the release of gas.

Table 4 —Recommended length of test pieces

Dimensions in millimetres

Diameter	Length of sample
$d < 1{,}00$	300
$1{,}00 \leq d < 1{,}50$	150
$1{,}50 \leq d < 3{,}00$	100
$3{,}00 \leq d \leq 5{,}00$	50



Key

1	Base	4	Graduated tube
2	Tap B	5	Tap A
3	Hose	6	Flask

Figure 2 — Apparatus for the determination of the mass of coating by the gaseous volumetric method

5.2.3.5 Procedure

With tap B closed, fill the graduated tube and part of the flask with as reagent an hydrochloric acid solution containing a suitable inhibitor.

Raise the level of liquid in the graduated tube to just below A by raising the acid reservoir C. The levels in the tube and flask shall be identical (see Figure 3a).

After placing the test piece in the graduated tube through tap A, close tap A and hydrogen released by the action of the acid on the coating will accumulate in the upper part of the graduated tube.

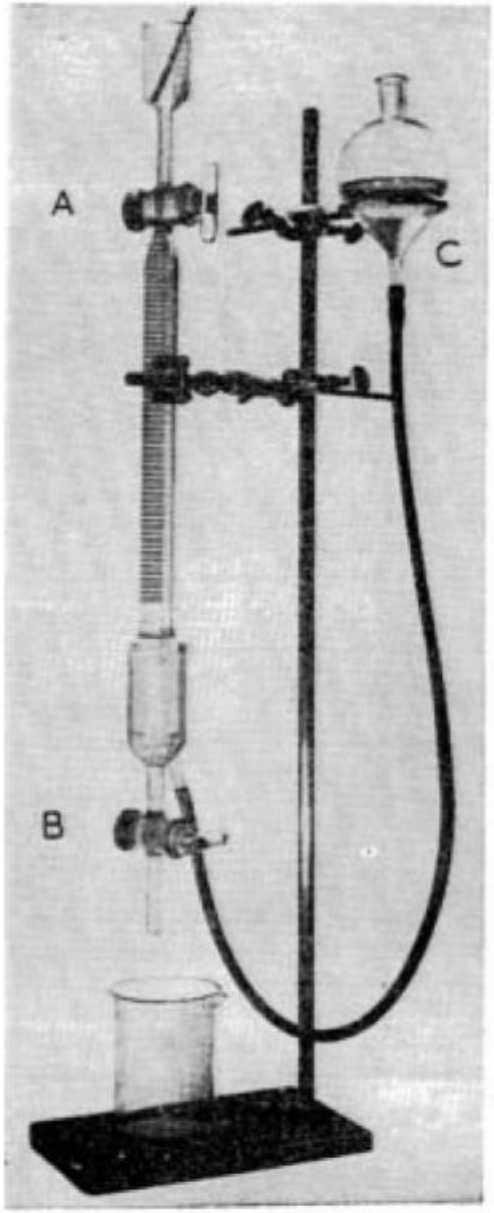


Figure 3a) — Arrangement of apparatus at start of test

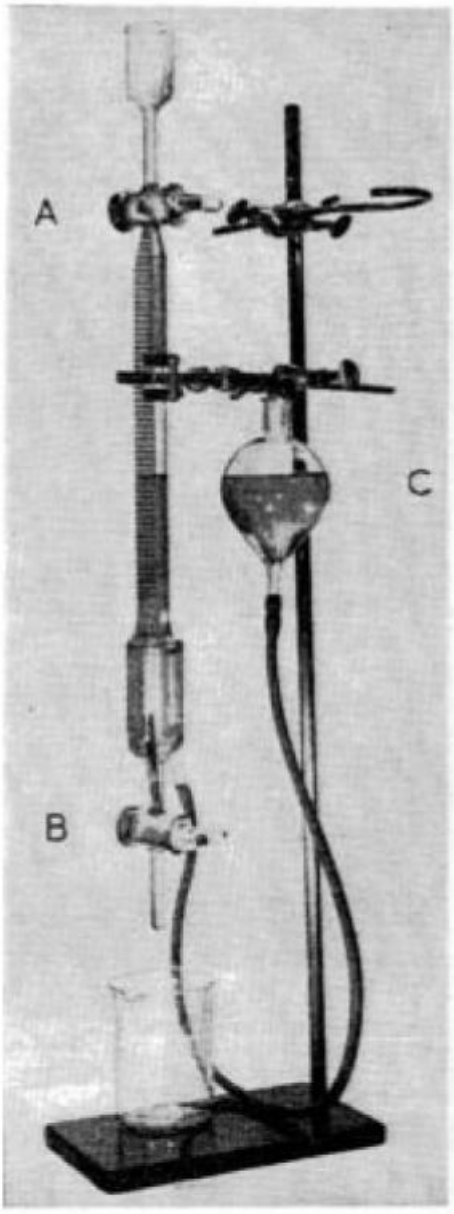


Figure 3b) — Arrangement of apparatus when measuring volume of hydrogen released

Key

A Tap A B Tap B C Acid Reservoir

Figure 3 — Apparatus to determine mass of coating by gaseous volumetric method

When the release of hydrogen is completed, apart from a few small bubbles, lower the flask (see Figure 3b) in relation to the graduated tube in order to bring the levels of solution in the tube and flask to the same height. The position of the surface of the liquid in the tube then shows the volume of hydrogen released. The volume shall be read from the lower level of the liquid surface to ensure minimum error.

When the test is completed, open tap A and remove all the acid reagent from the tube, placing the flask on a table.

Open tap B so that the test piece can be removed from the tube and placed in the container. The test piece is then washed and carefully wiped before its diameter is measured.

The test shall be carried out on one test piece at a time and the temperature in the tube shall be maintained at 20 °C ± 2 °C. The laboratory temperature shall be noted and when necessary a temperature correction shall be applied.

It is not recommended that the volumetric method be used for wire with a diameter greater than 5 mm.

5.2.3.6 Expression of results

The result is determined once the test piece has been tested. The mass of coating depends on the type of alloy. The volume of hydrogen released is directly related to the mass of zinc dissolved by the acid. For a non-alloy zinc coating, the mass of zinc coating per unit area, expressed in grams per square metre, is supplied by the equation:

$$m = \frac{2724\,V}{\pi \cdot d \cdot l} \tag{3}$$

NOTE 1 mol of hydrogen equals 24,0 l of hydrogen, corresponding to 65,38 grams of zinc. A volume V of hydrogen expressed in ml corresponds to a mass "m" of zinc expressed in grams.

$$m = \frac{65{,}38 \cdot V}{24{,}0 \cdot 10^3} = 2{,}724 \cdot V \cdot 10^{-3} \tag{4}$$

The mass per unit surface (m_A) in g/m^2 equals

$$m_A = \frac{2{,}724 \cdot V\,10^{-3}}{\pi \cdot d \cdot l \cdot 10^{-6}}\,\text{g/m}^2$$

$$= \frac{2724 \cdot V}{\pi \cdot d \cdot l} \tag{5}$$

Where:

d is the diameter of the wire without coating (in mm);

l is the length of the test piece (in mm);

V is the volume of hydrogen released during each of the tests (in ml).

In the case of shaped wire, then $\pi.d$ is replaced by the circumference of the shape; this generally obtained from a sketch of the section of the wire.

If the temperature (t °C) is very different from 20 °C, a temperature correction shall be applied for the volume by multiplying the right side of the equation above by the factor:

$$\frac{293}{t+273}$$

If the atmospheric pressure P is not between 987 mbar and 1 040 mbar, the same formula should be multiplied by:

$$\frac{P}{1{,}014}$$

In practice tables can be used to read the mass of zinc per square metre of surface of uncoated wire, as a function of the diameter of the wire, the length of the test piece and the volume of hydrogen released.

In the case of the alloy Zn95Al 5, the mass (m_A) of the alloy is supplied by the equation:

$$m_A = \frac{2405 \cdot V}{\pi \cdot d \cdot l} \text{ g/m}^2 \quad (6)$$

This formula is only valid if the mean aluminium content is between 4,5 % and 5,5 %.

The equation above may be used for circular section wire. In the case of shaped wire, the factor $\pi \cdot d$ shall be replaced by the nominal circumference of the shape.

For other zinc alloys, the volumetric method may be applied in so far as a reliable relationship can be established between the gas released and the weight of the coating.

5.3 Dipping test

5.3.1 Principle

The test consists of immersing a wire sample for a given period once or several times consecutively in a saturated copper sulphate solution which gradually dissolves the zinc coating and reveals the defects in the continuity of this coating.

The dipping test is solely intended to reveal any significant eccentricity defect in the coating or any other significant uniformity defect which could exist even if the mass of zinc per unit of surface area complies with the mass requirements for the coating.

5.3.2 Reagents

The reagent used shall be a saturated copper sulphate solution prepared from copper sulphate crystals ($CuSO_4 . 5H_2O$) "in the pure for analysis state" in a proportion of 314 g of salts to 1 l of de-ionised water at a temperature of 20 °C ± 2 °C.

The solution shall be prepared completely from cold. The liquid shall under no circumstances be heated, even to complete the dissolving process. To prevent this operation taking too long, the procedure may be applied as follows: the salts to be dissolved are crushed then reduced bit by bit by successive amounts of the water used. Once the salts have dissolved completely, the various solutions which have been prepared separately are mixed together and stirred. A small quantity of salt shall remain undissolved at the bottom of the container as proof of the state of saturation.

The solution shall be neutralized using an excess of chemically pure copper oxide (1 g per litre of solution) and then the solution shall be left to rest for at least 24 h before decanting for use.

5.3.3 Preparation of test piece

The test piece shall consist of a piece of wire approximately 250 mm long, straightened by hand to a greater or lesser degree. This test piece shall be completely de-greased any suitable degreasing solvent. It is then rinsed in distilled water and wiped with a clean piece of cotton wool. After degreasing, the wire shall only be held at the end which is not to be dipped. In the case of a corroded wire or if other chemical substances remain present on the surface of the wire after degreasing (for example chromate or phosphates), the wire shall first be dipped in a 0,2 % sulphuric acid solution for 15 s and then rinsed.

5.3.4 Performing of the test

The test shall be performed in a glass container of least 8 cm internal diameter. It is filled with the reagent, prepared as in 5.3.2, to a depth of at least 10 cm. During the test the temperature shall be kept at 20 °C ± 2 °C. The real temperature shall be noted.

The test specimen shall be dipped in the reagent for half a minute if the required number of dips is only half a minute. In all other cases the test sample shall be dipped in the solution for a period of one minute.

During the period of immersion the test specimens shall be held vertically; the immersed part shall not touch the wall of the container and shall not be moved. After each dip any deposited but not adhering copper shall be removed by slightly rubbing with cotton wool under running water.

This procedure shall be repeated with additional dips until the sample shows adherent coating of metallic copper for the first time or until the number of dips in Table 3 is reached, whichever is the smallest. After the last dip the sample shall be rinsed again under running water and dried with cotton wool.

Where Table 3 prescribes a dip of half a minute the dip shall be done after execution of the prescribed number of one minute dips. The solution used shall be renewed whenever the series of tests carried out has resulted in dissolving a maximum of 5 g of zinc per litre of reagents. In order to save time several test samples with a maximum of six, may be dipped at the same time without touching each other.

5.3.5 Interpretation of results

The test sample shall satisfy the test if the number of dips without adherent copper is equal or larger than the prescribed number of dips in Table 3. Any deposit of metallic copper within 25 mm of the cut end shall be discarded.

In case of doubt about the adherence of the copper, the remaining zinc may be removed by dissolution in hydrochloric acid. If the copper is not adhering to the steel base but is sticking on top of the zinc surface it will be removed together with the zinc.

Bibliography

[1] ISO 7989-1, Steel wire and wire products — Non-ferrous metallic coatings on steel wire — Part 1: General principles