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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Single cold-reduced electrolytic chromium/chromium oxide-coated steel —

Part 1 :
Sheets

Fer chromé électrolytique simple réduction à froid —

Partie 1 : Feuilles

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Reference number
ISO 8110-1 : 1988 (E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8110-1 was prepared by Technical Committee ISO/TC 17, *Steel*.

ISO 8110 consists of the following parts, under the general title *Single cold-reduced electrolytic chromium/chromium oxide-coated steel*:

Part 1 : Sheets

Part 2 : Coil for subsequent cutting into sheets

Annex A forms an integral part of this part of ISO 8110.

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Single cold-reduced electrolytic chromium/chromium oxide-coated steel —

Part 1 : Sheets

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1 Scope

This part of ISO 8110 specifies requirements for single cold-reduced electrolytic chromium and chromium oxide-coated low carbon steel sheet. It applies to sheets in nominal thicknesses that are multiples of 0,01 mm from 0,15 mm up to and including 0,49 mm.

The term "electrolytic chromium/chromium oxide-coated steel" is usually abbreviated, for convenience, to ECCS and this abbreviation is used throughout this part of ISO 8110.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 8110. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 8110 are encouraged to investigate the possibility of applying the most recent edition of the standard given below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1024 : —[1], *Metallic materials — Hardness test — Rockwell superficial test (scales 15N, 30N, 45N, 15T, 30T and 45T).*

3 Definitions

For the purposes of this part of ISO 8110, the following definitions apply.

3.1 anvil effect : The effect which a hard anvil can produce on the numerical hardness value obtained when a hardness test is performed on very thin sheet supported on such an anvil.

3.2 batch (box) annealed : Annealed by the process in which the cold-reduced strip is annealed in tight coil form, within a protective atmosphere, for a predetermined time-temperature cycle.

3.3 bulk package; bulk : A multiple packaging unit comprising a stillage platform (see 3.10), the ECCS and packaging material.

3.4 consignment : A quantity of single cold-reduced ECCS sheets of the same specification made available for despatch at the same time.

3.5 continuously annealed : Annealed by the process in which cold-reduced coils are unwound and annealed in strand form within a protective atmosphere. The resulting product is harder and exhibits a finer grain size than the corresponding batch-annealed product.

3.6 finishes

3.6.1 *shot blast finish* : *A finish on the steel base resulting* from the use of temper mill work rolls that have been shot blasted.

3.6.2 smooth finish : A finish on the steel base resulting from the use of temper mill work rolls that have been ground to a high degree of polish.

3.6.3 stone finish : A finish on the steel base, characterized by a directional pattern, resulting from the use of final mill work rolls that have been ground to a lower degree of polish than those used for the smooth finish.

3.6.4 surface finish : The appearance of the surface of ECCS sheets, governed by the surface characteristics of the steel base which result from controlled preparation of the work rolls during the final stages of rolling.

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[1] To be published.

3.7 line inspection : The final inspection of the finished product performed by instruments and/or by visual examination at normal line speeds.

3.8 rolling width : The width of the sheet perpendicular to the direction of rolling.

3.9 standard grade ECCS : Material in sheet form which does not contain any of the following :

a) pinholes, i.e. any perforation through the whole thickness of the plate;

b) areas of thickness outside the tolerance range specified in 9.2;

c) significant surface defects which render the material unsuitable for the intended use;

d) significant damage or shape related defects which render the material unsuitable for the intended use.

3.10 stillage platform : Base platform on which ECCS sheets are stacked to facilitate packing and transportation.

4 Information to be supplied by the purchaser

4.1 General

The following information shall be given on the enquiry and order to assist the manufacturer in supplying the correct material :

a) a description of the material required, i.e. single cold-reduced electrolytic chromium/chromium oxide-coated steel sheets;

b) reference to this part of ISO 8110;

c) the quantity, expressed on an area basis, and the dimensions of the sheets required;

d) the designation for the mechanical property classification of the sheets required and whether batch (BA) or continuous (CA) annealing is required (see clause 5);

NOTE — Certain classifications are suitable for shaping operations such as stamping, drawing, folding, beading and bending, and assembly work such as joint forming and welding although soft soldering is impracticable and welding is only recommended if, prior to welding, removal of the coating layer is efficiently undertaken at surfaces comprising the weld area. The end use should be borne in mind when the classification is selected.

e) the finish required (see 6.2);

f) any further special requirements.

4.2 Options

In the event that the purchaser does not indicate his wish to implement any of the options included in this part of ISO 8110 and does not specify his requirements at the time of the enquiry and order, the product shall be supplied on the following basis :

a) the direction of the runners of the stillage platform shall be at the discretion of the manufacturer (see clause 14);

b) the larger of the two dimensions of the sheet shall be the rolling width (provided this is within the capability of the mill).

4.3 Additional information

In addition to the information in 4.1 and 4.2, the purchaser may wish to provide further information to the supplier to ensure that the order requirements are consistent with the end use of the product.

The purchaser shall inform the supplier of any modification to the fabrication methods that will significantly affect the way in which the purchased product is used.

When ordering single cold-reduced ECCS, it is recommended that the purpose for which the material is intended should be stated.

5 Designations

For the purposes of this part of ISO 8110, the tempers in which sheets complying with this part of ISO 8110 are supplied are designated in terms of a temper classification based on the Rockwell 30T hardness test as given in table 2.

For batch-annealed and continuously annealed materials, the designations BA and CA respectively are added after the temper classification (for example, T57 BA).

6 Manufacture, finish and defects

6.1 Manufacture

The methods of manufacture of the sheets are left to the discretion of the producer and are not specified in this part of ISO 8110.

The purchaser shall be informed if an alteration is made to the method of manufacture that will affect the properties of the purchased ECCS.

NOTE — It is recommended that the manufacturer supplies to the purchaser such details of the steelmaking process as may assist the purchaser in his efficient use of the material.

6.2 Finish

Single cold-reduced ECCS can be supplied with either a smooth, stone or shot blast finish, and the finish required shall be specified at the time of order [see 4.1 e)].

6.3 Defects

Sheets shall not contain any defects as defined in 3.9.

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7 General requirements

The sheets shall comply with the requirements of clauses 8 to 11.

When tests are carried out to verify compliance with the requirements of clauses 8, 9 and 10, sample sheets shall be selected from consignments in accordance with clause 12.

For the determinations of dimensions and shape, the sample sheets shall be tested in accordance with the methods described in clause 9.

For the determination of coating mass and hardness, test specimens shall be taken from the sample sheets and shall be tested in accordance with clause 13.

NOTE — *No sampling or testing requirements are specified in this part* of ISO 8110 for verifying that any oil coating applied to the sheets is suitable for food packaging in accordance with clause 11.

The method of packaging the sheets shall be in accordance with clause 14.

8 Chromium/chromium oxide coating mass

The average values of the coating mass of the sample selected in accordance with clause 12 shall not be less than the following (see note 1) :

- chromium as metal : 30 mg/m^2 on each surface;
- chromium in the oxide : 5 mg/m^2 on each surface.

Metallic chromium is expressed as milligrams of chromium metal per square metre (mg/m^2) on each surface of the sheet.

Chromium oxides are expressed as the amount of chromium in the oxide, in milligrams per square metre (mg/m^2), on each surface of the sheet.

NOTES

1 The total chromium is made up of chromium metal and chromium oxides. The amounts of each are determined separately.

2 It is not possible to specify maximum limits for the average values for chromium metal and chromium in the oxides as the requirements for these values are closely related both to the producer's process and the user's end use.

3 The values specified, in principle, permit the material to be classified as ECCS. However, conformance with the specified coating mass minima does not guarantee acceptable performance under all conditions of use. It is therefore essential for the supplier and the user to agree upon the suitability of the particular supplier's product for the intended end use.

9 Tolerances on dimensions and shape

9.1 Introduction

Tolerances on dimensions (i.e. thickness and linear dimensions) and shape (i.e. out-of-squareness and edge camber) are specified in 9.2 to 9.5, together with appropriate methods of measurement.

Other geometrical features may be present in cold-reduced chromium/chromium oxide-coated steel sheet, such as :

burr : metal displaced beyond the plane of the surface of the sheet by shearing action;

edge wave : an intermittent vertical displacement occurring at the sheet edge when the sheet is laid on a flat surface;

centre buckle (full centre) : an intermittent vertical displacement or wave in the sheet occurring other than at the edges;

longitudinal bow (line bow) : residual curvature in the sheet along the direction of rolling;

transverse bow (cross bow) : curvature in the sheet such that the distance between its edges parallel to the rolling direction is less than the sheet width.

Although it is not possible at present to specify methods of measuring or to specify limits for these geometrical features, certain of which are subject to the equipment employed by the purchaser, the producer should endeavour to keep the occurrence and magnitude of burr, edge wave, centre buckle, longitudinal bow and transverse bow to a minimum.

9.2 Thickness

9.2.1 Thickness measurements

9.2.1.1 General

Thickness shall be measured either by the weighing method described in 9.2.1.2, or by direct measurement using the micrometer method described in 9.2.1.3.

In cases of dispute and for all retests, the weighing method shall be the referee method.

9.2.1.2 Weighing method

9.2.1.2.1 Determine the thickness of each sample sheet as follows :

a) Weigh the sheet to obtain the mass to the nearest 2 g.

b) Measure the length and width of the sheet to the nearest 0,5 mm and calculate the area.

c) Calculate the thickness of the sheet, to the nearest 0,001 mm, using the following formula :

$$\text{thickness (mm)} = \frac{\text{mass (g)}}{\text{area (mm}^2) \times 0{,}007\,85\ (\text{g/mm}^3)}$$

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Table 1 — Ordered thickness and thickness tolerances

Ordered thickness mm	Tolerance ± mm
0,15	0,015
0,16	0,015
0,17	0,015
0,18	0,020
0,19	0,020
0,20	0,020
0,21	0,020
0,22	0,020
0,23	0,025
0,24	0,025
0,25	0,025
0,26	0,025
0,27	0,025
0,28	0,030
0,29	0,030
0,30	0,030
0,31	0,030
0,32	0,030
0,33	0,035
0,34	0,035
0,35	0,035
0,36	0,035
0,37	0,035
0,38	0,040
0,39	0,040
0,40	0,040
0,41	0,040
0,42	0,040
0,43	0,045
0,44	0,045
0,45	0,045
0,46	0,045
0,47	0,045
0,48	0,050
0,49	0,050

9.2.1.2.2 To determine the average thickness for a consignment, calculate the arithmetic mean of the calculated thicknesses of all the sample sheets representing the consignment.

9.2.1.2.3 To determine the variation of thickness within each sample sheet, take two specimens Y (see figure 1) from the sheet. Weigh each specimen to the nearest 0,01 g, measure the length and width of each specimen to the nearest 0,1 mm, and calculate the thickness of each specimen to the nearest 0,001 mm using the formula given in 9.2.1.2.1 c).

9.2.1.3 Micrometer method

9.2.1.3.1 Determine the thickness of each sample sheet by direct measurement using a hand-operated, spring-loaded micrometer which permits readings to 0,001 mm. Measure the thickness to an accuracy of 0,001 mm, at least 10 mm from the trimmed edge of the sheet.

NOTE — It is recommended that the micrometer should have a ball-ended shank anvil of approximately 3 mm diameter, a curved surface base anvil of approximately 25 mm radius and a face diameter of approximately 13 mm.

9.2.1.3.2 To determine the average thickness for a consignment, calculate the arithmetic mean of the measured thicknesses of all the sheets representing the consignment.

9.2.1.3.3 To determine the variation of thickness within each sample sheet, use the micrometer to measure the thicknesses at two locations on each of the specimens Y (see figure 1). State the average thickness for each specimen to the nearest 0,001 mm.

9.2.1.3.4 To determine the transverse thickness profile, use either the micrometer or some other suitably precise means to measure the thickness at a location 6 mm in from the mill-trimmed edges of the sheet at right angles to the rolling direction.

NOTE — If means other than the micrometer are used to measure the transverse thickness profile, such means should be agreeable to both the producer and the purchaser.

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9.2.2 Thickness tolerances

9.2.2.1 Individual sheets

The thickness of each of the individual sample sheets selected from a consignment in accordance with clause 12, determined in accordance with 9.2.1.2 or 9.2.1.3, shall not deviate from the ordered nominal thickness by more than

a) ± 8,5 % if the weighing method is employed, or

b) the tolerance given in table 1 if the micrometer method is employed.

9.2.2.2 Average thickness for a consignment

The average thickness for a consignment, determined in accordance with 9.2.1.2 on the sample sheets selected in accordance with clause 12, shall not deviate from the ordered nominal thickness by more than

a) ± 2,5 % for a consignment of more than 20 000 sheets, or

b) ± 4 % for a consignment of 20 000 sheets or less.

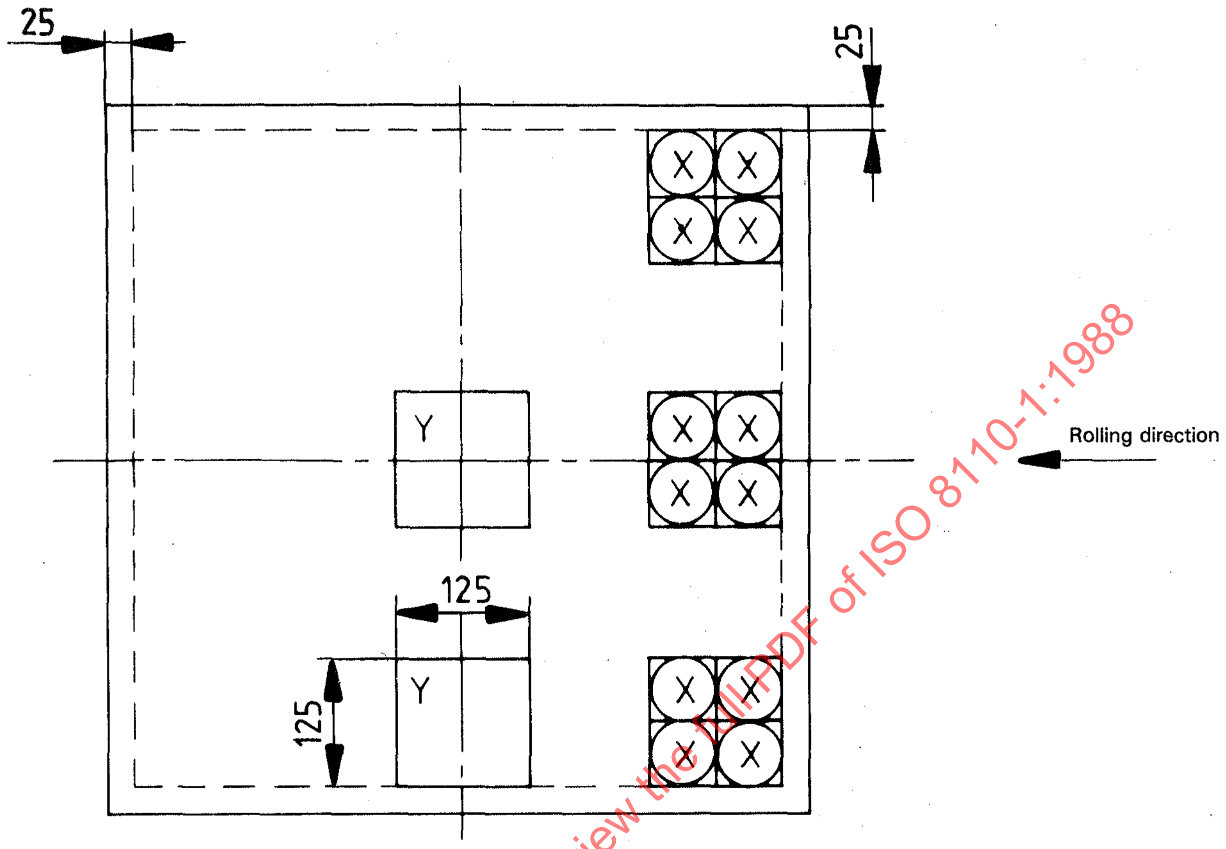
9.2.2.3 Permissible local thickness variations within a sheet

The thickness of either of the two individual specimens determined in accordance with 9.2.1.2.3 or 9.2.1.3.3 shall not deviate by more than 4 % from the average thickness of the whole sheet.

9.2.2.4 Transverse thickness profile (feather edge)

Transverse thickness profile is the reduction in sheet thickness at right angles to the rolling direction, close to the edge. The minimum thickness, when measured at 6 mm from the mill-trimmed edge in accordance with 9.2.1.3.4, shall not be more than 16 % below the ordered nominal thickness and/or shall not vary more than 10 % when compared to the centre thickness of the sheet being measured.

Dimensions in millimetres



X : specimens for coating mass tests

Y : specimens for determination of local thickness variation within a sheet

Figure 1 — Locations of test specimens

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9.3 Linear dimensions of sheets

9.3.1 Size of sheet

Each sample sheet shall be such that a rectangle of the ordered dimensions is available in it.

9.3.2 Tolerances on linear dimensions

In addition to being not less than the ordered dimensions, each sheet shall be trimmed on both edges and the trimmed (coil width) dimension shall not exceed the ordered dimension by more than 3 mm. Normally the cut in the rolling direction will not exceed the ordered dimension by more than 3 mm, but in no case shall it exceed the ordered dimension by more than 5 mm.

9.3.3 Measurement of linear dimensions

Lay each sample sheet, selected in accordance with clause 12, on a flat surface and measure the length and width to the nearest 0,5 mm across the centre of the sheet.

9.4 Out-of-squareness

Out-of-squareness is the deviation of an edge from a straight line drawn at a right angle to the adjacent edge of the sheet, from a corner to the opposite edge (see figure 2).

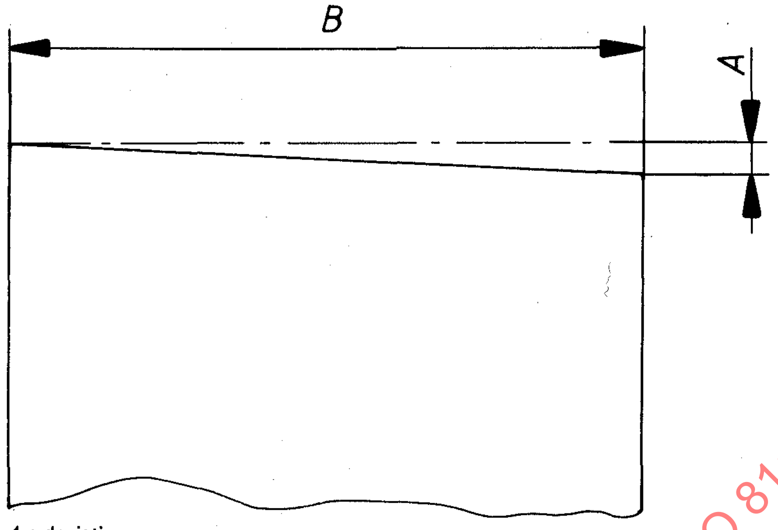
The out-of-squareness expressed as a percentage is calculated as follows :

$$\text{out-of-squareness (\%)} = \frac{\text{deviation } (A)}{\text{sheet dimension } (B)} \times 100$$

For each sheet in the sample, the out-of-squareness will not normally exceed 0,15 %, but in no circumstances shall it exceed 0,25 %.

9.5 Edge camber

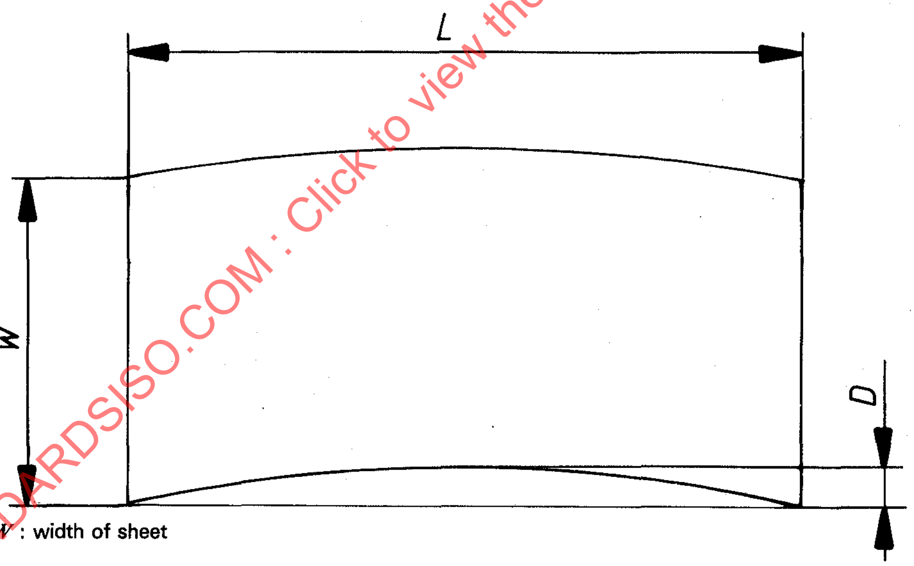
Edge camber is the maximum deviation (in the plane of the sheet) of an edge from a straight line forming a chord to it (see figure 3).



A : deviation

B : length or width of the sheet measured at right angles to an edge

Figure 2 — Measurement of out-of-squareness



W : width of sheet

L : length of sheet

D : deviation

Figure 3 — Edge camber of sheet

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The edge camber expressed as a percentage of the chord length is calculated as follows :

$$\text{edge camber (\%)} = \frac{\text{deviation } (D)}{\text{length of chord } (L)} \times 100$$

For each sample sheet, the camber shall not exceed 0,15 %.

10 Hardness

The term "temper" when applied to single cold-reduced ECCS sheets summarizes a combination of inter-related mechanical properties. The primary consideration for ECCS sheets is that they perform satisfactorily in subsequent processing and their intended end uses, but no single mechanical property test can measure all the various factors that contribute to the fabrication characteristics of the material. However, the Rockwell 30T hardness test (HR 30T) has come into use as a quick test and forms the basis for temper designation.

When the material is tested as described in 13.3, the hardness values shall be as given in table 2.

NOTES

1 For a given temper classification, and therefore hardness, other mechanical properties will vary with steel type.

2 For a given temper classification, and therefore hardness, other mechanical properties will also be different for batch-annealed (BA) material as compared with continuously annealed (CA) material.

3 On relatively thin material, the HR 15T test may be employed and the values obtained converted to the equivalent HR 30T values (see table 3).

11 Oiling

ECCS sheets are supplied with an oil coating. The oil shall be one that is recognized (i.e. by the relevant national or international authority) as being suitable for food packaging.

12 Sampling

12.1 General

If tests are carried out to ascertain whether the sheets in a consignment comply with the requirements for coating mass (see clause 8) and the requirements for the tolerances on dimensions and shape (see clause 9), sample sheets shall be selected in accordance with 12.2.

12.2 Selection of sample sheets

12.2.1 Number of bulk packages

Sample bulk packages shall be selected at random from the total number of bulk packages at the rate of 20 % rounded to the nearest greater whole number of bulk packages and subject to a minimum of four bulk packages. For consignments comprising less than four bulk packages, each bulk package shall be taken as a sample.

12.2.2 Number of sheets

NOTE — As the number of sheets per bulk package may vary, for example between 1 000 and 2 000, the rate of sampling is specified on a percentage basis (except for verification of coating masses).

12.2.2.1 Verification of grade (see 3.9)

From each of the bulk packages selected in accordance with 12.2.1, sheets at the rate of 1 % per bulk package shall be taken at random and inspected. In case of dispute, further sheets at the rate of 5 % per bulk package shall be taken at random and inspected.

12.2.2.2 Hardness and verification of chromium/chromium oxide masses

From each of the sample bulk packages selected in accordance with 12.2.1, two sheets shall be taken at random.

12.2.2.3 Verification of dimensions

From each of the sample bulk packages selected in accordance with 12.2.1, sheets at the rate of 0,5 % per bulk package to the nearest greater whole number of sheets shall be taken at random.

12.3 Retests

12.3.1 Chromium/chromium oxide coating masses

In the event of the average chromium/chromium oxide masses failing to meet the specified requirements, two further sets of samples, from other bulk packages, shall be selected as specified in 12.2 and specimens taken as described in 13.2.1. If both retests are satisfactory, the consignment shall be deemed to comply with the requirements of this part of ISO 8110, but if either of the additional tests is a fail the consignment shall be deemed not to meet the requirements of this part of ISO 8110.

The retest determinations shall be made using the referee methods described in annex A.

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Table 2 — Hardness values

Temper	Thickness $\leq$ 0,21 mm		from 0,21 to 0,28 mm		> 0,28 mm	
	Nominal hardness value	Range	Nominal hardness value	Range	Nominal hardness value	Range
T50	53 max.		52 max.		51 max.	
T52	53	± 4	52	± 4	51	± 4
T55	56	± 4	55	± 4	54	± 4
T57	58	± 4	57	± 4	56	± 4
T61	62	± 4	61	± 4	60	± 4
T65	65	± 4	65	± 4	64	± 4

12.3.2 Dimensions

If any of the dimensions measured are unsatisfactory, further measurements shall be made on two further sets of samples selected, from other bulk packages, in accordance with 12.2. If both remeasurements are satisfactory, the consignment shall be deemed to comply with the requirements of this part of ISO 8110, but if either of the additional measurements fails to meet the relevant requirements, the consignment represented shall be deemed not to comply with this part of ISO 8110.

13 Test methods

13.1 Locations of test specimens

Test specimens for the determinations of coating mass, hardness and local thickness variation within a sheet shall be taken from each sample sheet selected in accordance with clause 12, at the locations shown in figure 1.

13.2 Determination of chromium/chromium oxide coating masses

13.2.1 Specimens

From each sheet selected in accordance with clause 12, four discs, each of area not less than 2 500 mm^2, shall be taken from each of the three sets of positions marked X in figure 1. The test area of each disc (see A.1.3.2) shall be accurately determined and not less than 2 000 mm^2. The specimens shall clear the edges and end of the sheet by 25 mm.

Two of the four discs from each position shall be used for the separate determinations of the mass of chromium in the metallic chromium layer and the mass of chromium in the chromium oxide layer on one surface of the sheet and the other two discs shall be used for the corresponding determinations on the other surface.

13.2.2 Method of determination

The masses of metallic chromium and chromium oxide shall be expressed in milligrams per square metre to the nearest 1 mg/m^2.

For routine test purposes, the coating masses may be determined by any of the recognized and acceptable analytical methods but, in cases of dispute and for all retests, the methods described in the annex shall be the referee methods.

Ay tests carried out using the methods in annex A shall be done on virgin ECCS, i.e. untreated material, in the as-produced state.

13.3 Determination of hardness

13.3.1 Specimens

From each of the sample sheets obtained in accordance with clause 12, take two specimens 125 mm × 125 mm from the positions marked Y in figure 1.

NOTE — The specimens (Y) taken in accordance with 9.2.1.2.3 and 9.2.1.3.3 for the determination of the thickness variations within the individual sample sheets taken in accordance with 12.2.2.3 may be used also for the hardness determinations where appropriate. For example, from a sample bulk package containing 200 sheets, two sheets would be required (12.2.2.2) for the coating mass determinations and one sheet (12.2.2.3) for verification of dimensions; clearly, the sheet taken in accordance with 12.2.2.3 (and therefore the specimens Y taken from it) could be one of the two sheets taken in accordance with 12.2.2.2.

Before carrying out the hardness tests in accordance with 13.3.2, artificially age the specimens at 200 °C for 20 min.

Polish shot blast finish plate with 600 grade emery paper.

13.3.2 Test method

Determine the Rockwell HR 30T indentation hardness either :

a) directly, in accordance with ISO 1024, or

b) indirectly, on relatively thin sheets (for example 0,22 mm and thinner), by determining the HR 15T hardness in accordance with ISO 1024 and then converting the HR 15T values to HR 30T values using table 3.

Make three hardness measurements on each of the specimens taken in accordance with 13.3.1.

Calculate the representative hardness for the consignment as the arithmetic mean of all the hardness results on all the sample sheets taken from the consignment.

Table 3 — Rockwell HR 15T values and their HR 30T equivalents

HR 15T value	Equivalent HR 30T value
90,0	76,0
89,5	75,5
89,0	74,5
88,5	74,0
88,0	73,0
87,5	72,0
87,0	71,0
86,5	70,0
86,0	69,0
85,5	68,0
85,0	67,0
84,5	66,0
84,0	65,0
83,5	63,5
83,0	62,5
82,5	61,5
82,0	60,5
81,5	59,5
81,0	58,5
80,5	57,0
80,0	56,0
79,5	55,0
79,0	54,0
78,5	53,0
78,0	51,5
77,5	51,0
77,0	49,5
76,5	49,0
76,0	47,5

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To measure the indentation hardness, use a Rockwell superficial hardness testing machine, employing the 30T or 15T scale (see ISO 1024), as appropriate. Provide the machine with an anvil having a diamond centre spot.

Carry out the test on specimens from which all organic oxide coatings have been removed. Avoid testing near the edges of the specimen because of a possible cantilever effect.

Place the specimen on the anvil and bring it into contact with the ball indenter by turning the hand wheel until the indicator on the dial shows that the minor load is applied. Then turn the adjustable rim of the dial until the pointer reads zero and apply the major load by operating the handle. The rate of loading is controlled by a dash-pot incorporated in the machine. As soon as the loading is complete, remove the major load by pulling the handle forward and read the Rockwell hardness number directly from the appropriate scale.

14 Packaging

The sheets shall be supplied in bulk packages in which the numbers of sheets are multiples of 100.

NOTES

1 The sheets are customarily packed on a stillage platform forming a bulk package weighing between approximately 1 000 and 2 000 kg.

2 If the purchaser has any preference for the direction of the runners of the stillage platform, his requirements should be agreed with the producer and stated in the order [see 4.2 a)].

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Annex A
(normative)

Methods for the determination of metallic chromium and chromium in the oxide on the surface of electrolytic chromium/chromium oxide-coated steel

A.1 Determination of chromium in the oxide

A.1.1 Principle

The method involves the dissolution of the oxide in sodium hydroxide followed by oxidation with hydrogen peroxide. The absorbance of the coloured chromate ion is measured photometrically and the concentration of chromium is then obtained by reference to a calibration curve.

The effective range of the method is from 3 to 50 mg/m^2 and the reproducibility is better than ± 3 mg/m^2.

A.1.2 Reagents

During the analysis, use only reagents of recognized analytical grade, unless otherwise specified, and only deionized or distilled water.

Prepare freshly and, where necessary, filter all solutions.

A.1.2.1 Chromium, standard solution.

Dissolve 0,113 2 g of anhydrous potassium dichromate, primary standard grade, previously dried at 120 °C for 1 h, in approximately 200 ml of water in a 500 ml one-mark volumetric flask, dilute to the mark with water and mix. Pipette a 50 ml aliquot portion of this solution into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,004 mg of Cr.

A.1.2.2 Sodium hydroxide, 7,5 mol/l solution.

Dissolve 300 g of sodium hydroxide in approximately 700 ml of water, cool and dilute to 1 litre.

A.1.2.3 Hydrogen peroxide, approximately 60 g/l solution.

Use a solution supplied at this concentration or dilute a concentrated (for example 300 g/l) solution. (Ensure that the solution is at the recommended concentration. Hydrogen peroxide solutions may decompose if kept under non-ideal conditions.)

A.1.3 Apparatus

Ordinary laboratory apparatus and

A.1.3.1 Spectrophotometer, capable of measuring absorbance in the range 365 to 375 nm and of reading absorbance to ± 0,001.

A.1.3.2 Specimen holder, of the type shown in figure A.1, to permit stripping from an area of not less than 2 000 mm^2 from one surface of a specimen.

A.1.4 Preparation of the chromium calibration curve

Pipette 0 ml (blank), 10 ml, 20 ml, 30 ml, 40 ml and 50 ml aliquot portions of the dilute standard chromium solution (A.1.2.1) into a series of six 400 ml beakers, add 40 ml of the sodium hydroxide solution (A.1.2.2) and dilute to about 90 ml with water. Add 10 ml of the hydrogen peroxide solution (A.1.2.3), cover with a watch glass and boil until the excess peroxide is completely decomposed, replacing any loss of evaporated water by rinsing the wall of the beaker and the cover glass. Cool the solution, transfer to a 100 ml one-mark volumetric flask, dilute to the mark with water and mix well. Measure the absorbance using a cell of appropriate length and with water as a reference at the optimum wavelength for the specific instrument selected within the range 365 to 375 nm to obtain maximum sensitivity. Correct for the absorbance of the reagent blank and plot absorbance against milligrams of chromium per 100 ml.

A.1.5 Procedure

Handle sample material with care to prevent surface contamination. Do not subject specimens to any thermal stoving process before carrying out the following test. Take a disc of the material appropriate to the size of the specimen holder and fix it in position in the holder. Add 40 ml of hot sodium hydroxide solution (A.1.2.2) and place the cell on a hot-plate to maintain the temperature of the stripping solution at about 90 °C for 10 min. Transfer the contents of the cell quantitatively to a 250 ml beaker, add 10 ml of the hydrogen peroxide solution (A.1.2.3) and boil until the excess peroxide is decomposed. Cool, transfer to a 100 ml one-mark volumetric flask, dilute to the mark with water and mix well. Measure the absorbance at the selected wavelength (see A.1.4), using water as a reference. Correct for the absorbance of the reagent blank and obtain the concentration of chromium, in milligrams per 100 ml, from the calibration curve.

A.1.6 Calculation

The coating mass, expressed in milligrams of chromium per square metre, is given by the formula

$$\frac{m_1 \times 10^6}{S_1}$$

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where

m_1 is the mass of chromium, in milligrams, contained in 100 ml of test solution, obtained from the calibration curve;

S_1 is the area, in square millimetres, of specimen exposed to sodium hydroxide attack in the specimen holder.

A.2 Determination of metallic chromium — photometric method

A.2.1 Principle

The chromium oxide is first removed chemically. Metallic chromium is then stripped electrolytically in sodium carbonate solution, the completion of the reaction being indicated by a sharp rise in the cell voltage. The resultant solution is treated with hydrogen peroxide to ensure complete oxidation of the electrolytically stripped chromium to the hexavalent state. The absorbance of the coloured chromate ion is determined photometrically and the concentration of chromium is then obtained by reference to a calibration curve.

The effective range of the method is from 30 to 300 mg/m^2 and the reproducibility is better than ± 5 mg/m^2.

A.2.2 Reagents

During the analysis, use only reagents of recognized analytical grade, unless otherwise specified, and only deionized or distilled water.

Prepare freshly and, where necessary, filter all solutions.

A.2.2.1 Chromium, standard solution.

Dissolve 1,132 g of anhydrous potassium dichromate, primary standard grade, previously dried at 120 °C for 1 h, in approximately 200 ml of water in a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix. Pipette a 50 ml aliquot portion of the solution into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 0,02 mg of Cr.

A.2.2.2 Sodium hydroxide, 7,5 mol/l solution.

Dissolve 300 g of sodium hydroxide in approximately 300 ml of water. Cool and dilute to 1 litre.

A.2.2.3 Sodium carbonate, 0,5 mol/l solution.

Dissolve 53 g of anhydrous sodium carbonate in water and dilute to 1 litre.

A.2.2.4 Hydrogen peroxide, approximately 60 g/l solution.

Use a solution supplied at this concentration or dilute a concentrated (for example 300 g/l) solution. (Ensure that this solution is at the recommended concentration. Hydrogen peroxide solutions may decompose if kept under non-ideal conditions.)

A.2.3 Apparatus

Ordinary laboratory apparatus and

A.2.3.1 Cell and electrodes, a cell for the electrolytic stripping of the metallic chromium, as shown in figure A.2, and consisting of a specimen holder, a platinum gauze cathode and a reference electrode (standard calomel).

It is essential that the cell/specimen holder exposes a minimum specimen area of 2 000 mm^2 from which the metallic chromium is electrolytically stripped.

A.2.3.2 Power supply, with a direct current stabilizer with a built-in milliammeter setting to 30 mA and an on/off switch.

A.2.3.3 Voltmeter, with a full-scale deflection of 2 V.

A.2.3.4 Spectrophotometer, capable of measuring absorbance in the range 365 to 375 nm and of reading absorbance to ± 0,001.

A.2.4 Preparation of the chromium calibration curve

Pipette 0 ml (blank), 5 ml, 10 ml, 15 ml, 20 ml, 30 ml, 40 ml and 50 ml of the dilute standard chromium solution (A.2.2.1) into a series of eight 250 ml beakers, add 120 ml of the sodium carbonate solution (A.2.2.3) and dilute to approximately 170 ml with water. Add 10 ml of the hydrogen peroxide solution (A.2.2.4), cover with a watch glass and boil until the excess peroxide is completely decomposed, replacing any loss of evaporated water by rinsing the wall of the beaker and the cover glass. Cool the solution, transfer to a 200 ml one-mark volumetric flask, dilute to the mark with water and mix well. Measure the absorbance using a cell of suitable length and with water as a reference at the optimum wavelength for the specific instrument selected within the range 365 to 375 nm to obtain maximum sensitivity. Correct for the absorbance of the reagent blank and plot absorbance against milligrams of chromium/200 ml for the different cell lengths (for example 2 cm, 4 cm, 5 cm) which have been used.

A.2.5 Procedure

A.2.5.1 General

Handle sample material with care to prevent surface contamination. Do not subject specimens to any thermal stoving process before carrying out the following test. Take a disc of the material appropriate to the size of the specimen holder.

Normally the determination of metallic chromium will follow the determination of chromium in the oxide and the same specimen discs may be used for both purposes. Where metallic chromium is to be determined without a previous determination of chromium in the oxide, remove the chromium oxide in accordance with A.2.5.2.

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A.2.5.2 Removal of chromium oxide layer

Remove the surface oxides from the specimen by treating with 40 ml of the sodium hydroxide solution (A.2.2.2) in a glass beaker at 90 °C for 10 min. Rinse the specimen with water and attach to the specimen holder (see figure A.1).

A.2.5.3 Removal and determination of metallic chromium

After the washed, oxide-free specimen has been attached to the specimen holder (see A.2.5.2), connect the leads as shown in figure A.3, add 120 ml of the sodium carbonate solution (A.2.2.3) and simultaneously switch on the power supply. Maintain the current density at a constant level within the range 0,5 to 1,5 mA/cm^2. The end-point of the reaction is indicated by a large potential jump. (The potential difference between the beginning and end of the dissolution is about 400 mV. This is noted by incorporating in the electrical circuit a voltmeter with the positive terminal connected to the d.c. stabilizer and the negative terminal to the reference electrode.)

Quantitatively transfer the contents of the specimen holder/cell to a glass beaker, add 10 ml of the hydrogen peroxide solution (A.2.2.4) and boil until the excess peroxide is decomposed. Cool the solution, transfer to a 200 ml one-mark volumetric flask, dilute to the mark with water and mix well. Using a cell of appropriate length, measure the absorbance at the selected wavelength (see A.2.4) using water as a reference.

A.2.6 Calculation

The coating mass, expressed in milligrams of chromium per square metre, is given by the formula

$$\frac{m_2 \times 10^6}{S_2}$$

where

m_2 is the mass of chromium, in milligrams, contained in 200 ml of test solution, obtained from the calibration curve;

S_2 is the area, in square millimetres, exposed to electrolytic attack in the specimen holder/cell.

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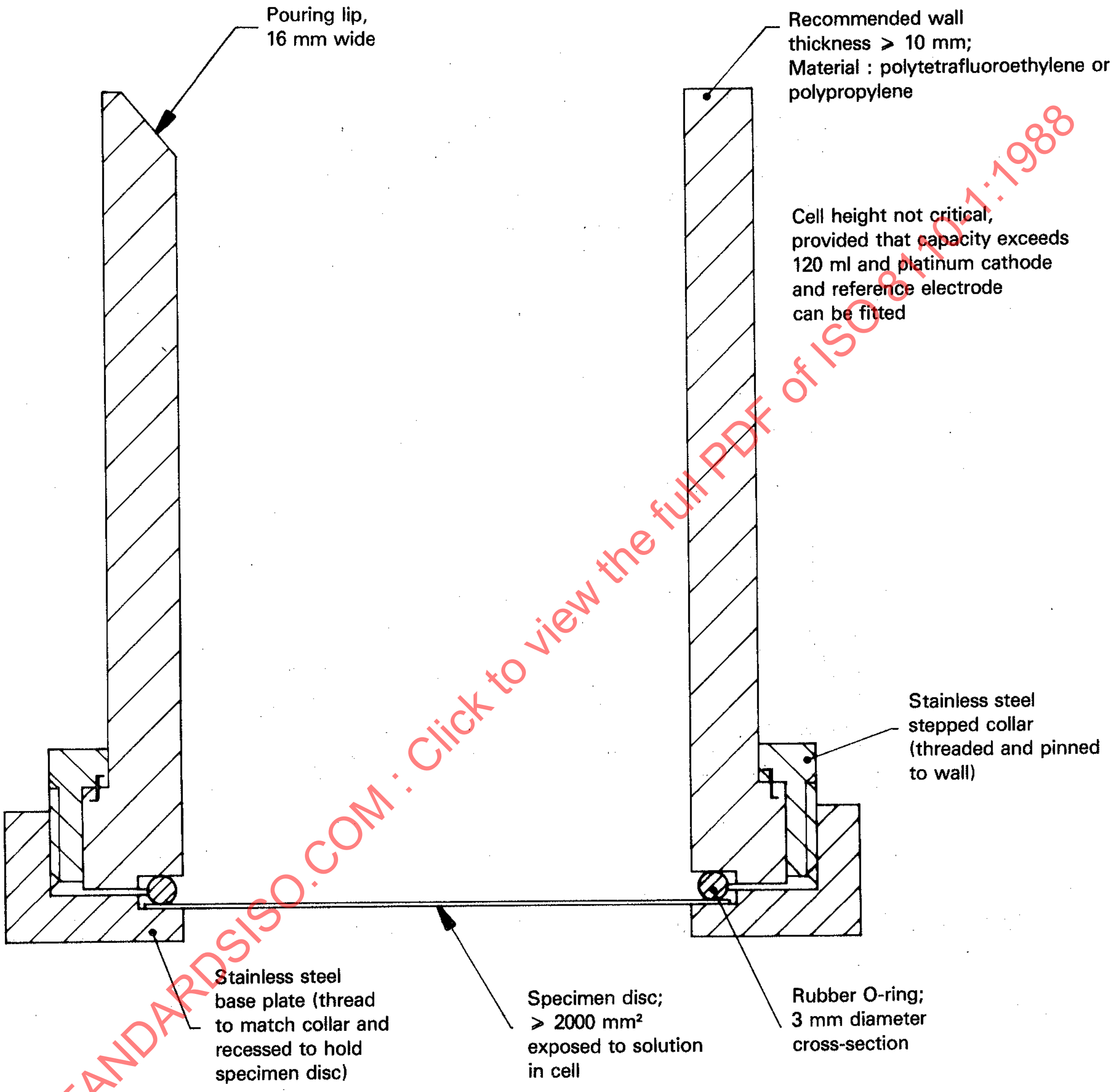

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Figure A.1 — Dual-purpose specimen holder showing details of construction

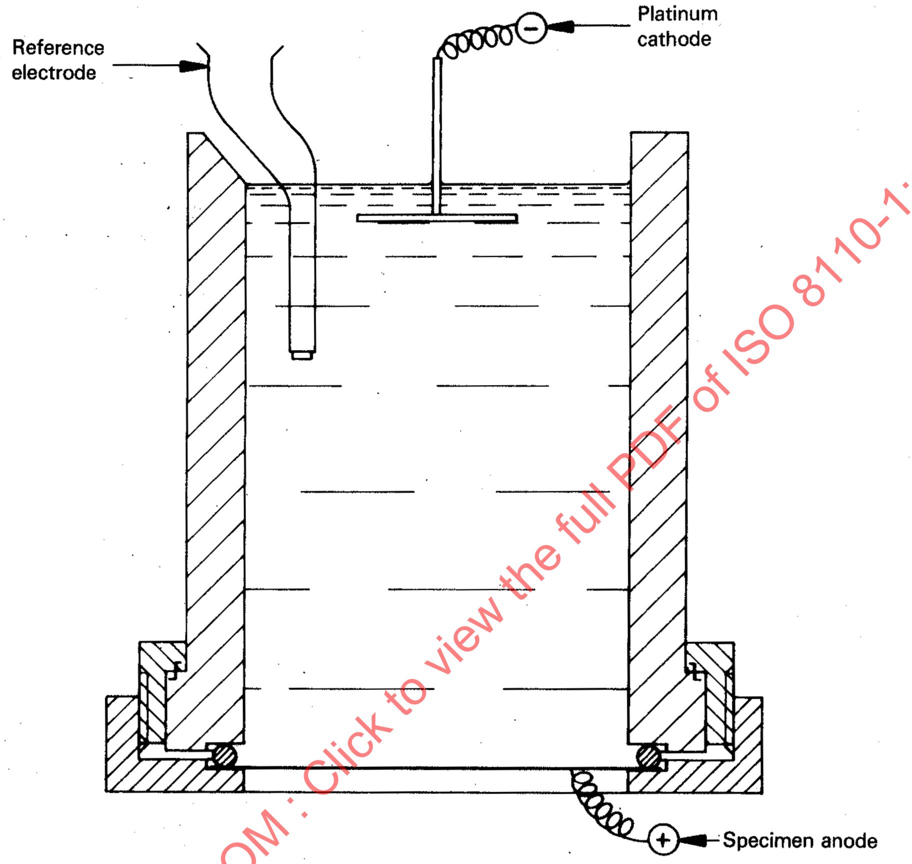

Figure A.2 — Electro-stripping of chromium metal using the dual-purpose specimen holder

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