

INTERNATIONAL STANDARD

ISO
8111-1

First edition
1988-11-01



INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

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

Part 1 : Sheets

Fer chromé électrolytique double réduction à froid —

Partie 1 : Feuilles

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Reference number
ISO 8111-1:1988 (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 8111-1 was prepared by Technical Committee ISO/TC 17, *Steel*.

ISO 8111 consists of the following parts, under the general title *Double cold-reduced electrolytic chromium/chromium oxide-coated steel*

Part 1 : Sheets

Part 2 : Coil for subsequent cutting into sheets

Annexes A and B form an integral part of this part of ISO 8111.

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

Part 1 : Sheets

1 Scope

This part of ISO 8111 specifies requirements for double 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 0,14 mm up to and including 0,29 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 8111.

It has not been possible, in preparing this first edition of ISO 8111-1, to specify mandatory requirements for mechanical properties. Test data are still limited and there are fundamental questions concerning what are the most relevant and reliable tests for evaluating the mechanical properties of double reduced tinplate. For guidance purposes, information is given in annex A on the levels of proof stress and hardness to be expected in practice; procedures for sampling and testing are recommended for the determination of these properties.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 8111. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 8111 are encouraged to investigate the possibility of applying the most recent editions of the standards listed 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)*.

ISO 6892 : 1984, *Steel — Tensile testing*.

ISO 8110-1 : —¹⁾, *Single cold-reduced electrolytic chromium/chromium oxide-coated steel — Part 1 : Sheets*.

3 Definitions

For the purposes of this part of ISO 8111, 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 bulk package; bulk : A multiple packaging unit comprising a stillage platform (see 3.8), the ECCS and packaging material.

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

3.4 finishes

3.4.1 stone finish : A finish on the steel base corresponding to that for single-reduced ECCS (see ISO 8110-1), 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 on single-reduced ECCS.

3.4.2 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.

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

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

1) To be published.

3.7 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.

3.8 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. double cold-reduced electrolytic chromium/chromium oxide-coated steel sheets;
- b) reference to this part of ISO 8111;
- 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 (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) 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 8111 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 the direction of rolling (see clause 13);
- b) the sheets shall be supplied with a stone surface finish (see 6.2);

- c) 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.

NOTE — Double cold-reduced ECCS is less ductile than single cold-reduced ECCS and has very distinct directional properties.

When ordering double cold-reduced ECCS, it is recommended that the purpose for which the material is intended should be stated. When double cold-reduced ECCS is used for built-up can bodies, it is essential that the rolling direction is around the circumference of the can so as to minimize the hazard of flange cracking. In such cases, it is imperative that the rolling direction be clearly stated in the contract.

5 Designations — mechanical property classification

For the purposes of this part of ISO 8111, the mechanical properties in which sheets complying with this part of ISO 8111 are supplied are designated in terms of a mechanical property classification based on the 0,2 % proof stress, as shown in table 1.

Table 1 — Mechanical property classification

Mechanical property classification	Nominal 0,2 % proof stress N/mm ²
DR 550	550
DR 620	620
DR 660	660

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 8111.

The purchaser shall be informed if any 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

Double cold-reduced ECCS sheets are usually provided with a stone surface finish characterized by a directional pattern, imparted to the strip by the use of ground work rolls in the final stages of the second reduction operation. Any other surface finish shall be agreed at the time of order. Sheets complying with this part of ISO 8111 are also supplied with a coating of oil on both sides.

6.3 Defects

Sheets shall not contain any defects as defined in 3.7.

7 General requirements

The sheets shall comply with the requirements of clauses 8 to 10.

When tests are carried out to verify compliance with the requirements of clauses 8 and 9, sample sheets shall be selected from consignments in accordance with clause 11.

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, test specimens shall be taken from the sample sheets and shall be tested in accordance with clause 12.

NOTE — No sampling or testing requirements are specified in this part of ISO 8111 for verifying that any oil coating applied to the sheets is suitable for food packaging in accordance with clause 10.

The method of packaging the sheets shall be in accordance with clause 13.

8 Chromium/chromium oxide coating mass

The average values of the coating mass of the sample selected in accordance with clause 11 shall not be less than the following (see note 1) :

- chromium as metal : 30 mg/m² on each surface;
- chromium in the oxide : 5 mg/m² on each surface.

Metallic chromium is expressed as milligrams of chromium metal per square metre (mg/m²) on each surface of the sheet.

Chromium oxides are expressed as the amount of chromium in the oxide, in milligrams per square metre (mg/m²), 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 be 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 :

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

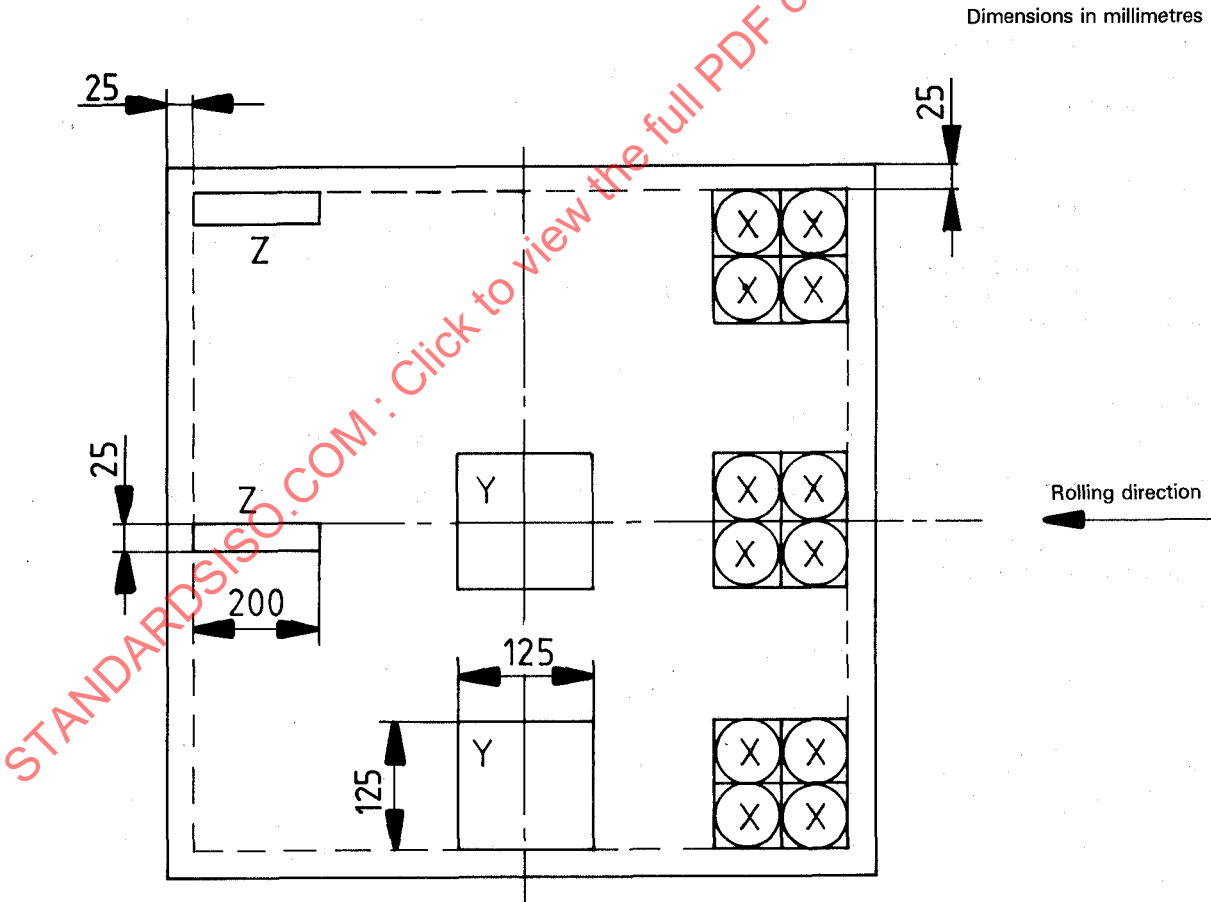
9.2.1.2.2 To determine the average thickness for a consignment, calculate the arithmetic mean of the calculated thicknesses of all the sample sheets representing the consignment.

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.



X : specimens for coating mass tests

Y : specimens for determination of local thickness variation within a sheet (see also A.2.2.3.1 in annex A regarding the recommended specimens for determination of hardness)

Z : specimens for tensile or springback tests if mechanical properties are to be determined in accordance with annex A.

Figure 1 — Locations of test specimens

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.

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 11, 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) $\pm 8,5$ % if the weighing method is employed, or
- b) the tolerance given in table 2 if the micrometer method is employed.

Table 2 — Ordered thickness and thickness tolerances

Ordered thickness mm	Tolerance $\pm$ mm
0,14	0,015
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

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 11, shall not deviate from the ordered nominal thickness by more than

ance with clause 11, shall not deviate from the ordered nominal thickness by more than

- a) $\pm 2,5$ % 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 15 % below the ordered nominal thickness and/or shall not vary more than 9 % when compared to the centre thickness of the sheet being measured.

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 11, 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 :

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

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 %.

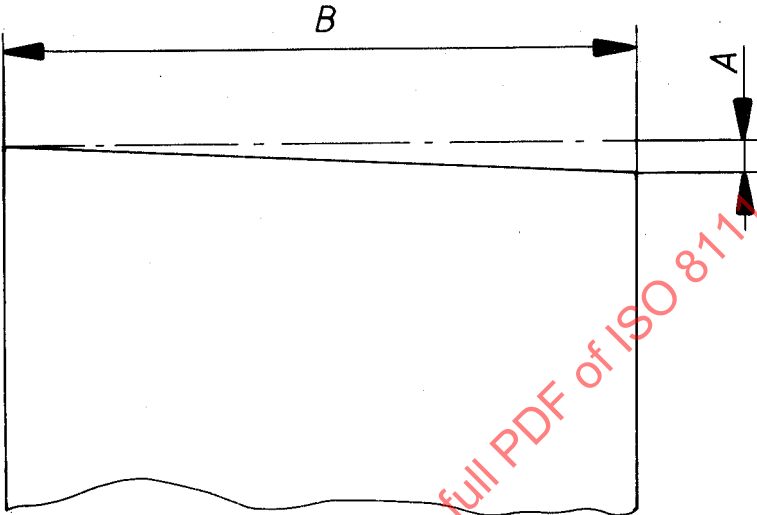
9.5 Edge camber

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).

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

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

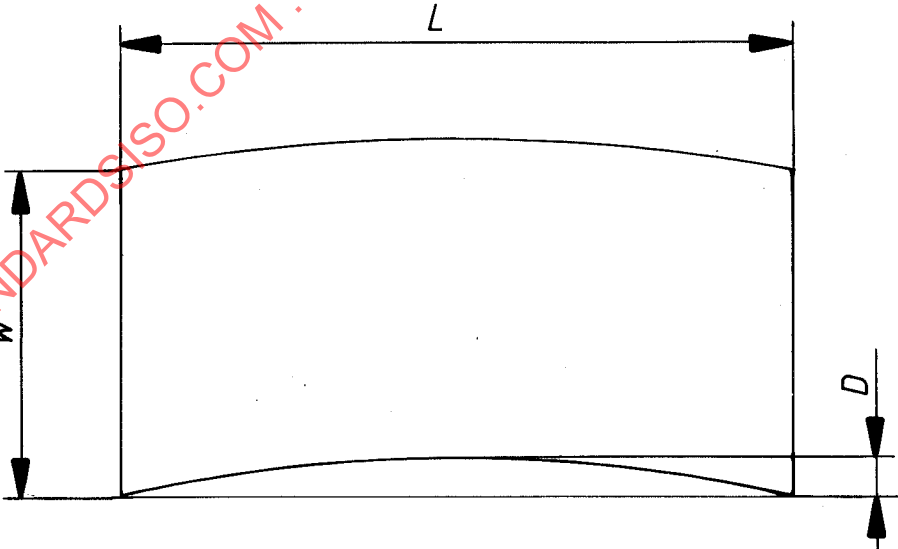
For each sample sheet, the camber shall not exceed 0,15 %.



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 chord

D : deviation

Figure 3 — Edge camber of sheet

10 Oiling

The oil applied to the surfaces of ECCS sheets shall be one that is recognized (i.e. by the relevant national or international authority) as being suitable for food packaging.

11 Sampling

11.1 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 11.2.

11.2 Selection of sample sheets

11.2.1 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.

11.2.2 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).

11.2.2.1 Verification of grade (see 3.7)

From each of the bulk packages selected in accordance with 11.2.1, 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.

11.2.2.2 Verification of chromium/chromium oxide masses

From each of the sample bulk packages selected in accordance with 11.2.1, two sheets shall be taken at random.

11.2.2.3 Verification of dimensions

From each of the sample bulk packages selected in accordance with 11.2.1, sheets at the rate of 0,5 % per bulk package to the nearest greater whole number of sheets shall be taken at random.

11.3 Retests

11.3.1 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 11.2 and specimens taken as described in 12.2.1. If both retests are satisfactory, the consignment shall be deemed to comply with the requirements of this part of ISO 8111, 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 8111.

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

11.3.2 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 11.2. If both remeasurements are satisfactory, the consignment shall be deemed to comply with the requirements of this part of ISO 8111, 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 8111.

12 Test methods

12.1 Locations of test specimens

Test specimens for the determinations of coating mass and local thickness variation within a sheet shall be taken from each sample sheet selected in accordance with clause 11, at the locations shown in figure 1.

12.2 Determination of chromium/chromium oxide coating masses

12.2.1 Specimens

From each sheet selected in accordance with clause 11, four discs, each of area not less than 2 500 mm², shall be taken from each of the three sets of positions marked X in figure 1. The test area of each disc (see B.1.3.2) shall be accurately determined and not less than 2 000 mm². 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.

12.2.2 Method of determination

The masses of metallic chromium and chromium oxide shall be expressed in milligrams per square metre to the nearest 1 mg/m².

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 annex B shall be the referee methods.

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

13 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)

Mechanical properties

A.1 Nominal mechanical properties

No test or group of tests has been developed that adequately predicts all the factors affecting the fabricating performance of double cold-reduced ECCS sheets, the primary consideration for these products being that the sheets perform satisfactorily for the intended end use.

In the absence of such tests, it is customary to order the sheets to mechanical property classifications (see clauses 4 and 5), representing the 0,2 % proof stress (as determined by the tensile test or the springback test) and hardness (as determined by the Rockwell superficial hardness test). The proof stresses and hardnesses corresponding to the mechanical property classification levels are shown in table A.1. In practice, it is expected that the producer will aim to achieve the nominal values given in table A.1, within the ranges also given in the table.

The values included in table A.1 are based on average values determined in accordance with the sampling and test methods recommended in clause A.2; individual hardness values are not considered to have any representative significance in relation to a consignment of ECCS.

A.2 Recommended procedures for determining the nominal mechanical properties for consignments of double cold-reduced ECCS

A.2.1 Samples

For determination of the representative mechanical properties for a consignment of double cold-reduced ECCS sheets, use the two sheets taken in accordance with 11.2.2.2, for verification of the coating mass, from each of the bulk packages selected in accordance with 11.2.1. (However, see also the note to A.2.2.3.1.)

A.2.2 Determination of mechanical properties

A.2.2.1 General

It is unnecessary to remove the chromium/chromium oxide coating on the specimen, but remove all other coatings such as lacquers, varnishes and printing inks from the surface prior to testing.

NOTE — Attention is drawn to the effects of heat treatment during lacquering/printing which may influence the results of mechanical property tests.

Calculate the representative value of each property (proof stress and hardness) for the consignment as the arithmetic mean of all the values determined on all the sample sheets taken from the consignment.

A.2.2.2 0,2 % proof stress ($R_{p0,2}$)

A.2.2.2.1 Introduction

Two methods of determining the proof stress values of double cold-reduced ECCS are available. The first, the conventional tensile test, provides the more accurate measure, but is relatively slow and requires very careful, skilled preparation of the test specimens. The second, the springback test, was developed to give a reasonably accurate value and yet be fairly rapid. Therefore the tensile test is normally used for referee and calibration purposes while, for routine purposes, the springback test is considered generally to be adequate.

A.2.2.2.2 Tensile test

A.2.2.2.2.1 Specimens

For the determination of proof stress by the tensile test procedure, from each sheet selected in accordance with A.2.1 cut

Table A.1 — Mechanical properties

Mechanical property classification	Average proof stress (0,2 % non-proportional elongation) ¹⁾ , longitudinal		Average Rockwell hardness HR 30 T	
	Nominal N/mm ²	Range N/mm ²	Nominal	Range
DR 550 (DR 8)	550	480 to 620	73	70 to 76
DR 620 (DR 9)	620	550 to 690	76	73 to 79
DR 660 (DR 9 m)	660	590 to 730	77	74 to 80

1) The term tensile yield strength (0,2 % offset) is used in the USA and Canada.

two rectangular specimens approximately 200 mm long $\times$ 25 mm wide with the rolling direction parallel to the length of the specimen at the positions marked Z in figure 1. Ensure that the specimens clear the edges and end of the sheet by a minimum of 25 mm.

A.2.2.2.2 Test method

Make one test on each of the specimens selected in accordance with A.2.2.2.1 (i.e. two tests per sheet selected).

Determine the proof stress ($R_{p0,2}$) in accordance with ISO 6892 but take the following precautions if the thickness is less than 0,5 mm (ISO 6892 does not apply to thicknesses less than 0,5 mm, and thicknesses lower than this are commonly employed for double cold-reduced ECCS):

- Ensure that the edges of the specimen are entirely free of discernible burrs, if necessary by finishing the preparation of the edges with fine emery paper.
- Use specimens with a gauge length (effective length with parallel edges) of $50 \pm 0,5$ mm and a width of $12,5 \pm 1$ mm.
- Use a rate of straining of 1 mm/min.
- Employ grips that are capable of securing the sample such that no skew occurs during straining, to ensure that the applied stress is aligned centrally along the major axis of the test specimen.

A.2.2.2.3 Springback test

A.2.2.2.3.1 Specimens

For the determination of proof stress by the springback method, from each sheet selected in accordance with A.2.1 cut two rectangular specimens approximately 150 ± 1 mm long $\times$ 25 mm wide with the rolling direction parallel to the length of the specimen at the positions marked Z in figure 1.

A.2.2.2.3.2 Test method

Make one test on each of the specimens obtained in accordance with A.2.2.2.3.1 (i.e. two tests per sheet selected). Carry out the test using the springback Temper Tester model G.67.¹⁾

NOTE — Calibrate each new springback temper tester using the standard tensile test (see A.2.2.2.2) or another, "reference" springback Temper Tester. In addition, since malfunctions arising, for example, from excessive wear or inadvertent abuse of the test equipment may not be readily apparent, it is recommended that the springback temper test readings should be regularly compared with readings obtained from the standard tensile test or a "reference" springback Temper Tester. It is also recommended that such direct cross-checks should be supplemented by the frequent use of reference ECCS samples of known proof stress.

In making the test, strictly observe the operational instructions provided with the springback Temper Tester. The principal steps in the test are :

- Measure the thickness of the ECCS specimen to the nearest 0,001 mm.
- Insert the specimen into the tester and fix it firmly in the testing position by gently tightening the clamping screw using light finger pressure.
- Bend the specimen through an angle of 180° against the mandrel by a gentle swing of the forming arm.
- Return the forming arm to its "start" position and read and record the springback angle by sighting directly over the specimen.
- Remove the specimen from the tester and, using the recorded thickness of the specimen and the springback angle, determine the appropriate springback index value from a suitable conversion formula (for example Bower) agreed between producer and purchaser.

A.2.2.3 Hardness

A.2.2.3.1 Specimens

For the determination of hardness, from each sheet selected in accordance with A.2.1 cut two specimens 125 mm $\times$ 125 mm at 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 11.2.2.3, may be used also for the hardness determinations where appropriate.

Polish each surface of the specimens with 600 grade emery paper.

A.2.2.3.2 Test method

Determine the Rockwell HR 30T indentation hardness either

- directly, in accordance with ISO 1024, or
- 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 A.2.

Make three hardness measurements on each of the specimens taken in accordance with A.2.2.3.1.

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. Avoid testing near the edges of the specimen because of a possible cantilever effect.

1) The Temper Tester model G.67 is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

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.

**Table A.2 — Rockwell HR 15T values
and their HT 30T equivalents**

HR 15T value	Equivalent HR 30T value
92,0	80,5
91,5	79,0
91,0	78,0
90,5	77,5
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

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Annex B (normative)

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

B.1 Determination of chromium in the oxide

B.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/m² and the reproducibility is better than ± 3 mg/m².

B.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.

B.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.

B.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.

B.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.)

B.1.3 Apparatus

Ordinary laboratory apparatus and

B.1.3.1 Spectrophotometer, capable of measuring absorbance in the range 365 to 375 nm and of reading absorbance to $\pm 0,001$.

B.1.3.2 Specimen holder, of the type shown in figure B.1, to permit stripping from an area of not less than 2 000 mm² from one surface of a specimen.

B.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 (B.1.2.1) into a series of six 400 ml beakers, add 40 ml of the sodium hydroxide solution (B.1.2.2) and dilute to about 90 ml with water. Add 10 ml of the hydrogen peroxide solution (B.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.

B.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 (B.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 (B.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 B.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.

B.1.6 Calculation

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

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

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.

B.2 Determination of metallic chromium — photometric method

B.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/m² and the reproducibility is better than ± 5 mg/m².

B.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.

B.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.

B.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.

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

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

B.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.)

B.2.3 Apparatus

Ordinary laboratory apparatus and

B.2.3.1 Cell and electrodes, a cell for the electrolytic stripping of the metallic chromium, as shown in figure B.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 mm² from which the metallic chromium is electrolytically stripped.

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

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

B.2.3.4 Spectrophotometer, capable of measuring absorbance in the range 365 to 375 nm and of reading absorbance to $\pm 0,001$.

B.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 (B.2.2.1) into a series of eight 250 ml beakers, add 120 ml of the sodium carbonate solution (B.2.2.3) and dilute to approximately 170 ml with water. Add 10 ml of the hydrogen peroxide solution (B.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.

B.2.5 Procedure

B.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 B.2.5.2.

B.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 (B.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 B.1).

B.2.5.3 Removal and determination of metallic chromium

After the washed, oxide-free specimen has been attached to the specimen holder (see B.2.5.2), connect the leads as shown in figure B.3, add 120 ml of the sodium carbonate solution (B.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/cm². 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

(B.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 B.2.4) using water as a reference.

B.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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