TECHNICAL REPORT

ISO/TR 15922

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Metallic and other inorganic coatings — Evaluation of properties of dark-stain phenomenon of chromated coiled or sheet products

Revêtements métalliques et autres revêtements inorganiques — Évaluation des propriétés du phénomène de tache foncée des produits chromés enroulés ou en tôle d'acier

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO should not be held responsible for identifying any or all such patent rights.

ISO/TR 15922 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 8, *Chemical conversion coatings*.

Introduction

The existence of the dark- or black- stain phenomenon related to chromated hot-dip metallic zinc and electroplated-zinc steel products has been known, but the cause for the occurrence of this phenomenon so far does not appear to have been rationalized. One possible reason for this incidence is due to the packed or stacked storage conditions of coils and sheets in high temperature, pressure and high relative humidity (RH) conditions.

The test method specified in this Technical Report employs exposure of unstacked specimens to accelerated environmental storage conditions. Specimens made from unstacked product representing the unstressed condition typify the outer surface of the coil-packed product whilst similar specimens, when subjected to applied stress, temperature and humidity, represent the inner-surface layers of the packed coil and stacked sheet products and signify the stressed condition of those layers.

The properties are determined qualitatively by test methods to confirm the presence of chromated layers and quantitatively by a colorimetric method. The colorimetric method measures the radiance of the surface and analyses the difference in luminosity or intensity before and after the tests are performed.

Metallic and other inorganic coatings — Evaluation of properties of dark-stain phenomenon of chromated coiled or sheet products

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1 Scope

This Technical Report describes the methods to evaluate the properties of dark-stain phenomenon observed in chromate conversion-coated coiled and sheet products.

Chromate conversion coatings, with trivalent or hexavalent chromium ions, are applied to hot-dip metallic zinc and electroplated-zinc steel products to enhance corrosion resistance of the components.

When zinc-coated packed coiled and sheet products with conversion coatings are unpacked after long-term storage at elevated temperature and high relative humidity (RH), the surface of the sheet products appears tarnished. This discolouration appears as dark or black stain and influences the appearance and corrosion resistance, when parts are produced using these affected sheets.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3613, *Metallic and other inorganic coatings — Chromate conversion coatings on zinc, cadmium, aluminium-zinc alloys and zinc-aluminium alloys — Test methods*

ISO 3892, *Conversion coatings on metallic materials — Determination of coating mass per unit area — Gravimetric methods*

ISO 4520, *Chromate conversion coatings on electroplated zinc and cadmium coatings*

ISO 6789, *Assembly tools for screws and nuts — Hand torque tools — Requirements and test methods for design conformance testing, quality conformance testing and recalibration procedure*

IEC 60068-2-30, *Environmental testing — Part 2-30: Tests — Test Db: Damp heat, cyclic (12 h + 12 h cycle)*

IEC 60068-3-5, *Environmental testing — Part 3-5: Supporting documentation and guidance — Confirmation of the performance of temperature chambers*

IEC 60068-3-6, *Environmental testing — Part 3-6: Supporting documentation and guidance — Confirmation of the performance of temperature/humidity chambers*

ASTM E1347, *Standard Test Method for Color and Color-Difference Measurement by Tristimulus Colorimetry*

ASTM D2244, *Standard Practice for Calculation of Color Tolerances and Color Differences from Instrumentally Measured Color Coordinates*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3613, ISO 3892, ISO 4520, ISO 6789, appropriate parts of ISO 60068 and the following apply.

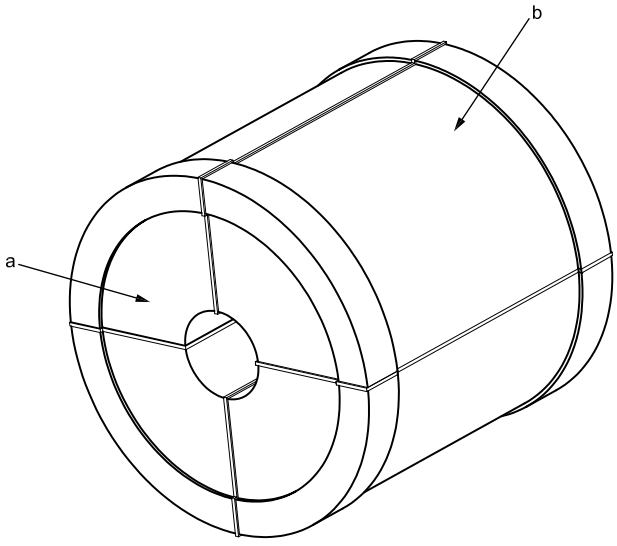
3.1
dark stain
phenomenon of discolouration of the surfaces of steel packed coil or stacked sheet products during storage at elevated temperature and high relative humidity

3.2
unstacked plate specimen
specimen to represent the outer-surface layer of coiled or sheet product that is not stacked

3.3
stacked plate specimen
specimen to represent the inner-surface layer of coiled or packed sheet product that is stacked

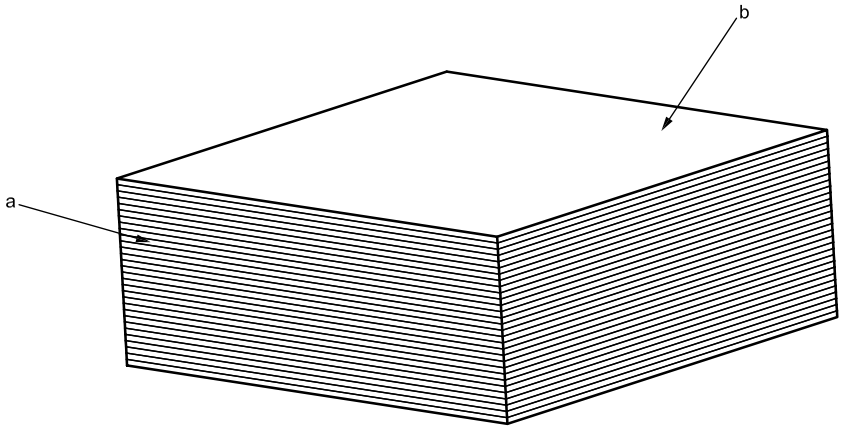
NOTE The inner surface is under stress due to stacking.

Figures 1 and 2 exhibit inner and outer layers of packed coil and sheet products in stacked and unstacked conditions.



a Represents inner-surface layer of stacked coil.
b Represents outer-surface layer of stacked coil.

Figure 1 — Illustration of packed coil product



a Represents inner-surface layer of packed sheet product.

b Represents outer-surface layer of packed sheet product.

Figure 2 — Illustration of packed sheet product

4 Information to be supplied by the producer to the user or purchaser

4.1 Essential information

When ordering the product in accordance with this Technical Report, the purchaser or user should be provided by the producer with the following information in writing, in, for example, the contract or purchase order, or on engineering drawings:

a) number and distribution of defects, and time elapsing before the appearance of the first sign of defect;

b) temperature, relative humidity, applied pressure in N/m^2 and test duration;

c) the AQL level and sampling plan used;

NOTE Widely used sampling plans are provided in Technical Reports (see Bibliography).

4.2 Additional information

The following additional information should also be provided by the purchaser, when appropriate:

a) any deviation from the agreed test procedures;

b) whether test samples are packed with vinyl before placing them to apparatus used for stacking pressures.

5 Apparatus

Normal laboratory apparatus and the following:

5.1 Environmental test cabinet, capable of environmental testing in accordance with IEC 60068-2-30, Test Db: Damp heat, cyclic (12 h + 12 h cycles).

Ensure that the conditions inside the working area of the test chamber are identical at all points, within the specified tolerances, including the immediate vicinity of measurement sensing devices. The chamber should conform to the requirements of IEC 60068-3-6.

Ensure that no condensed water is allowed to drop on the specimens.

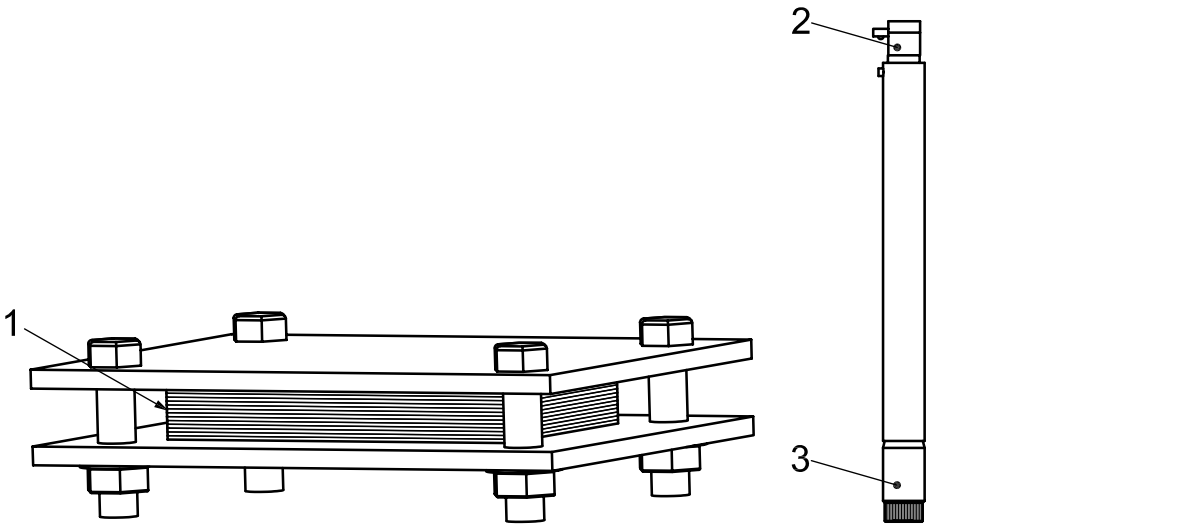
As specified in IEC 60068-2-30, the properties and dimensions of the test specimens should not influence the conditions within the chamber.

5.2 Photoelectric absorptiometer (colorimeter), with a filter having a mean transmission of 520 nm. The absorptiometer should be used with an absorption cell of optical path length 10 mm.

5.3 Apparatus for application of constant pressure.

It is used to simulate stacking conditions. Parts of the apparatus are made from high corrosion resistance material, such as plastic.

The apparatus to simulate stacking conditions (Figure 3) should be placed in the appropriate working area, in accordance with IEC 60068-3-5.



a) Cross section view **b) Setting torque tool**

Key

1 specimens
2 socket wrench
3 torque adjustment

Figure 3 — Apparatus to simulate stacking conditions

6 Test methods

6.1 General

The test surface, prior to the following tests, should be free of all contaminants, finger prints and other extraneous stains. If the surface is coated with thin oil, it should be removed prior to the tests by degreasing using a suitable solvent at room temperature (not exceeding 35 °C). For test purposes, the samples should not be subjected to be forced drying at temperatures in excess of 35 °C. Treatment in alkaline solutions should not be performed as chromate conversion coatings are broken down by alkalis.

Test materials are selected from chromate conversion-coated (ISO 4520) hot-dip zinc and zinc electroplated steel products.

The test methods for the qualitative test for the determination of dark- or black- stain properties is given in 6.3 and quantitative method by colorimetric method is given in 6.4.

6.2 Procedures

Sectioning test specimens of 120 mm × 80 mm dimensions from the outer and inner surfaces of unstored or unstacked and unpacked coiled or sheet products. The test specimens are to be selected from unstacked sheets to satisfy the AQL level of the sampling plan [see 4.1 c)] and assemble the test specimens in accordance with ISO 6789.

Assess the presence of trivalent or hexavalent chromium content of each chromated test sample in accordance with ISO 3613. Calculate mass per unit area of the coating in accordance with ISO 3892.

Place unstacked specimens in an environmental test cabinet for the test duration. The temperature, humidity, applied pressure to reproduce stacked condition, and duration period of test should be as designated by the specification covering the material or product being tested [see 4.1 b)]. Unless specified otherwise, conditions are as follows.

Temperature	(60 ± 1) °C
Humidity	(95 % ± 1 %) RH
Applied Pressure	(147 ± 0,98) N/m^2
Period	24 h, 48 h, 72 h, 96 h, 120 h, 240 h, 500 h, 1 000 h

6.3 Qualitative test method for determination of the property of dark stain

6.3.1 Test method

The specimens from outer and inner surfaces of unstored, unstacked products are exposed to the agreed temperature, pressure and humidity [see 4.1 b)] or, alternatively, to the condition specified in 6.2, for an agreed duration, to replicate respective unstacked and stacked products in stored conditions.

For comparison purposes, also expose specimens without chromate conversion coating similarly.

Criteria for evaluation to this test are:

a) appearance after test;

b) distribution of defects per sq cm, (black or stain, white rust, etc.);

c) time elapsing before the appearance of the first sign of defect;

d) mass per unit area of the coating.

6.3.2 Test report

The test report should contain the following information:

a) a reference to this Technical Report, ISO/TR 15922;

b) detailed information of the test methods, including temperature, relative humidity, applied pressure in N/m^2 and test duration;

c) number of samples tested;

d) mass per unit area of conversion coatings;

e) appearance after test;

f) distribution of defects per sq cm (black stain , white rust, etc.);

g) time elapsing before the appearance of the first sign of defect;

h) dates of the tests and date of the report.

6.4 Quantitative test method for determination of property of dark stain

6.4.1 Test method

The specimens from outer and inner surfaces of unstored, unstacked products are exposed to the agreed temperature, pressure and humidity [see 4.1 b)] or, alternatively, to the condition specified in 6.2, for an agreed duration, to replicate respective unstacked and stacked products in stored conditions.

For comparison purposes, measure colour intensities, in accordance with ASTM E1347 and ASTM D2247, of a) untested specimens and b) tested specimens after these are cooled down to room temperature.

Assessment of colour intensities is given in Annex A.

6.4.2 Test report

The test report should contain the following information:

a) reference to this International Technical Report, ISO/TR 15922;

b) detailed information of the test methods, including temperature, relative humidity, applied pressure in N/m^2 and test duration;

c) number of samples tested;

d) mass per unit area of conversion coatings;

e) results of the tests, including the results of the individual determinations and their mean values;

f) any deviations from the procedures specified;

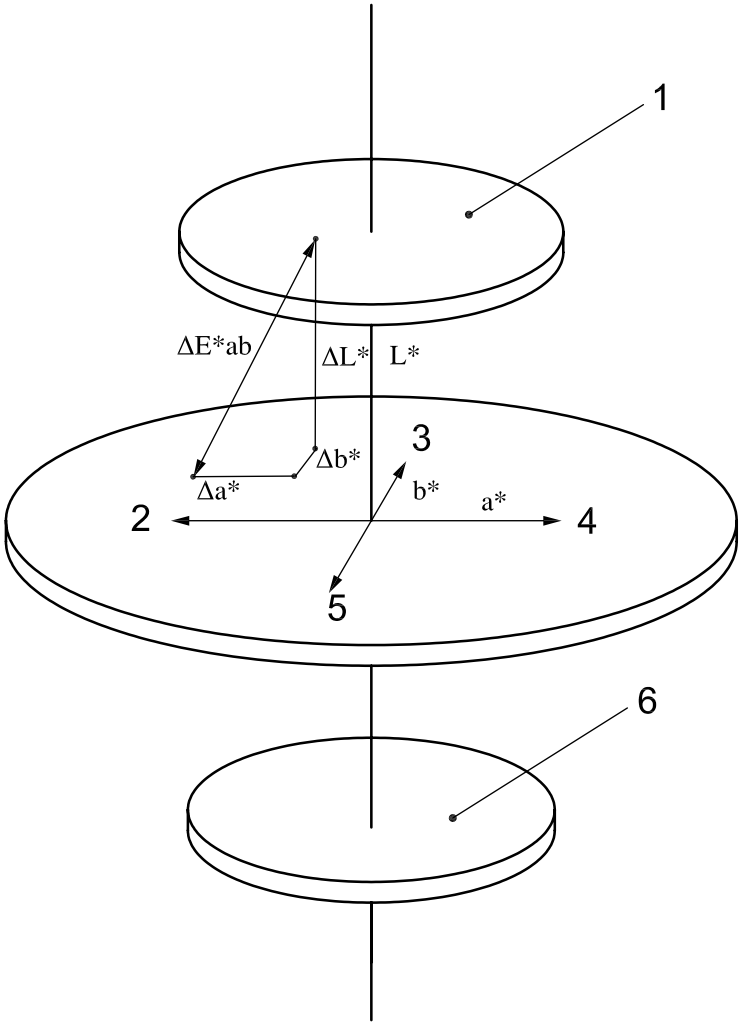
g) dates of the tests and date of the report.

Annex A
(normative)

Measurement of colour intensities

A.1 General

The colour intensity coordinates of the measurements by colorimetric methods are shown in Figure A.1.



Key

1 white
2 green
3 blue
4 red
5 yellow
6 black

Figure A.1 — Colour intensity coordinates

A.2 Expression of results

Difference in intensity of colour, prior to and after tests provide a measure of the characteristic of the product.

Δa^* = a^* (before test) – a^* (after test)

Δb^* = b^* (before test) – b^* (after test)

ΔL^* = L^* (before test) – L^* (after test)

Where

(+) Δa^* indicates colour intensity of red (less green);

(–) Δa^* indicates colour intensity of green (less red);

(+) Δb^* indicates colour intensity of yellow (less blue);

(–) Δb^* indicates colour intensity of blue (less yellow);

(+) ΔL^* indicates colour intensity of white (less black);

(–) ΔL^* indicates colour intensity of black (less white).

The mean of the difference of the total colour intensities, ΔEab^*, is calculated as:

$$\Delta Eab^* = [(\Delta a^*)^2 + (\Delta b^*)^2 + (\Delta L^*)^2]^{(0,5)}$$

Bibliography

[1] ISO 2859, *Sampling procedures for inspection by attributes*

[2] ISO 3497, *Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods*

[3] ISO 4519, *Electrodeposited metallic coatings and related finishes — Sampling procedures for inspection by attributes*

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