BS EN 4195:2011

BSI Standards Publication

# Aerospace series — Paints and varnishes — Test method for determination of chromate leaching

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## National foreword

This British Standard is the UK implementation of EN 4195:2011.

The UK participation in its preparation was entrusted to Technical Committee ACE/65/-/3, Paints, Surface Finish and Protective Treatments for Aerospace Purposes for Aerospace Purposes.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

# Aerospace series - Paints and varnishes - Test method for determination of chromate leaching

Série aérospatiale - Peintures et vernis - Test de lixiviation de chromate

Luft- und Raumfahrt - Beschichtungsstoffe - Chromatauslaugbarkeitstest

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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## Foreword

This document (EN 4195:2011) has been prepared by the Aerospace and Defence Industries Association of Europe - Standardization (ASD-STAN).

After enquiries and votes carried out in accordance with the rules of this Association, this Standard has received the approval of the National Associations and the Official Services of the member countries of ASD, prior to its presentation to CEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2012, and conflicting national standards shall be withdrawn at the latest by June 2012.

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# 1 Scope

This European Standard defines a test procedure for the determination of the leaching rate of hexavalent chromium from the dry paint film of a chromate containing primer for aerospace use.

The rate can be related to requirements either to prescribe the type of primer for an intended use or for the purpose of batch quality consistency.

The procedure is applicable to products intended for use in aerospace applications.

# 2 Normative references

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

EN 2090, *Aerospace series - Aluminium alloy AL-P2024-T3 - Clad sheet and strip 0,3 mm <a <6 mm*

EN 2334, *Aerospace series — Chromic-sulphuric acid pickle of aluminium and aluminium alloys*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

# 3 Principle

The test involves placing a dry film of chromate primer in distilled water in accordance with EN ISO 3696 for a period of time at a controlled temperature to allow soluble chromate to leach from the film into solution in the water. The water is collected for the measurement of dissolved hexavalent chromate by a suitable means.

Because the rate of leaching can vary considerably over a period of time the collection time is divided into a number of periods of different duration appropriate to the rate of leaching over the collection period.

Some of the water collected in the collection periods is not used for measurement because normally sufficient data is gained from other collections.

Values of chromate leaching can be numerically expressed or for convenience plotted on a graph.

# 4 Specimens and test apparatus

The apparatus shall consist of a stoppered test tube to hold the water and the painted test specimens. The test tube shall be maintained at a constant temperature, as specified, in a suitably controlled chamber.

Test specimens shall be of aluminium alloy EN 2090.

Test specimens shall be prepared by chrome sulphuric acid pickling in accordance with EN 2334 or other non chromate leaching pretreatment as may be specified in the relevant material specification. They shall then be sprayed with primer within four hours to give the paint thickness prescribed in the relevant material specification.

When dried and preconditioned as prescribed in the relevant material specification an area of 40 $cm^2$ shall be cut from the panel for leaching test immersion. All edges shall be uncoated. Using a test tube for the immersion, it is convenient to cut two panels of dimensions $(10 \times 2)$ cm. This assumes that the panels are coated on one side only and are then placed back to back in the test tube.

# 5 Test procedure

A test specimen, prepared as above, shall be totally immersed in a stoppered tube containing 50 ml of distilled water maintained at a temperature of $(40 \pm 2)$ °C.

The distilled water shall be renewed after 1, 2, 3, 4, 7, 10, 14, 18, 23, 29 and 36 days. The samples collected at the end of 2, 10 and 36 days only shall be analysed for chromate content as normally this is sufficient to determine the leach rate characteristics of the paint sample under test. However samples from other collection periods can also be measured if additional data is required.

At the end of the appropriate immersion time, the water in which the painted specimens have been immersed shall be poured into a 100 ml volumetric flask.

The specimen and the test container shall be rinsed with a volume of distilled water not exceeding 25 ml and this shall be added to the volumetric flask containing the first solution. Another 50 ml of distilled water shall be added to the original container to proceed with the test for the next immersion period.

A suitable method of analysis for chromate determination has been outlined in Annex A. Whether this or another method is employed for chromate concentration determination, the final volume of chromate containing solution, tube washings and reagents shall be 100 ml.

The concentration of hexavalent chromium in the solution shall be measured and calculated as milligrams of chromate leached per square decimetre of paint film.

## 6 Analysis of hexavalent chromium

A sample shall be taken from the 100 ml container and analysed for hexavalent chromium content.

The result shall be determined as mg Cr per l and expressed as mg $CrO_4^{2-}$ per $dm^2$ of paint film tested.

NOTE For a 40 $cm^2$ exposed primer area this involves multiplying the quantity of hexavalent chromium in mg/l by a factor of 0,557 7. Details of the calculation of the factor for this case are given in Annex B.

The quantity of $CrO_4^{2-}$ per $dm^2$ shall be converted into $CrO_4^{2-}$ per $dm^2$ per day leaching rate. This means that the value calculated for the test sample needs to be divided by the number of days over which the paint sample had been in contact with that sample of water since it had been previously renewed.

| | | |
|---|---|---|
| Sample at 2 days | duration 1 d to 2 d | divide by 1 |
| Sample at 10 days | duration 7 d to 10 d | divide by 3 |
| Sample at 36 days | duration 29 d to 36 d | divide by 7 |

The results can be numerically expressed or for convenience can be plotted on a semi-logarithmic graph.

## 7 Test report

The test report shall contain at least the following information:

a) Complete identification of the paint used including suppliers code numbers, date of manufacture, batch number, material standard, etc.;

b) Reference to this European Standard (EN 4195);

c) The number of specimens tested;

d) The paint thickness applied;

e) Dimensions of specimen tested;

f) The overall drying time between application and testing;

g) The method of analysis of chromate used for the determination;

h) Date of test and traceability to individuals performing the test work;

i) Any incident which may have affected the results and any deviation from the test method;

j) Reference to the material standard;

k) The test results.

## 8 Designation

EXAMPLE

| **Description block** | **Identity block** |
|---|---|
| **DETERMINATION OF CHROMATE LEACHING** | **EN4195** |

Number of this standard

# Annex A
(informative)

NOTE The following is one of many methods used for determining chromate concentration in a solution. It uses a colormetric determination of diphenylcarbazide complex. A set of standard solutions are prepared with increasing chromium concentrations and are used to construct a suitable calibration curve. The concentration of chromium in the "unknown" solution can then be read off the graph.

## A.1 Diphenylcarbazide solution

40 g of phthalic anhydride shall be dissolved in 1 l of hot industrial ethyl alcohol

The solution shall be cooled and 5 g of diphenylcarbazide added. This shall be stirred with a magnetic stirrer to dissolve.

## A.2 Phosphoric-sulphuric acid

375 ml of concentrated sulphuric acid (S.G. = 1,84 g/cm$^3$) and 375 ml of concentrated phosphoric acid (S.G. = 1,83 g/cm$^3$) shall be added to 1 l of distilled water.

## A.3 Preparation of standard chromium solutions

2,828 g of dried "Analar" potassium dichromate shall be accurately weighed, dissolved in about 100 ml of distilled water and diluted to 1 l in a volumetric flask. This solution shall now contain 1 g/l (or 1 000 µg/ml) of chromium.

In order to obtain a solution containing 5 µg/ml of chromium, 5 ml of the 1 g/l solution shall be pipetted into a 1 l volumetric flask and made up to the mark with distilled water.

In obtain a solution containing 100 µg/ml of chromium, 50 ml of the 1 g/l solution shall be pipetted into a 500 ml volumetric flask and made up to the mark with distilled water.

NOTE Standard solutions prepared in this way are stable when stored in closed glass containers for a maximum of 12 months.

## A.4 Calibration for diphenylcarbazide method

Using a burette, 2, 5, 10, 15 and 20 ml of 5 µg/ml standard solution shall be put into respective volumetric flasks.

NOTE 1 This is equivalent to 10, 25, 50, 75 and 100 µg of chromium for calibration.

50 ml of distilled water shall be added to another 100 ml volumetric flask. This shall be used as a blank.

The volume of solution in each flask shall be made up to approximately 50 ml with distilled water. 10 ml of the phosphoric/sulphuric acid solution and 10 ml of diphenylcarbazide shall be added.

The solutions shall be made up to the mark with distilled water, thoroughly mixed and allowed to stand for 10 minutes.

The standards shall be poured into 1 cm cells, the optical density values shall be measured using the blanks a reference on a suitable spectrophotometer set at a wavelength of 540 nm.

A graph of optical density versus chromium content of standards shall be plotted.

For test samples the steps outlined in clauses 3 to 5 of this section shall be followed and the chromium content equivalent to the measured optical density shall be read from the calibration graph.

NOTE 2 A fresh graph must be produced daily or with each batch of samples.

# Annex B
(informative)

# Calculation of factor

The result shall be determined as mg Cr per l and expressed as mg $CrO_4^{--}$ per $dm^2$ of paint film tested.

NOTE For a 40 $cm^2$ exposed primer area this involves multiplying the quantity of hexavalent chromium in mg/l by a factor of 0,557 7.

| | | |
|---|---|---|
| Mol wt of $CrO_4$ | a | 115,994 8 |
| Atomic wt of Cr | b | 51,996 |
| mg Cr per l | Y | – |
| mg Cr per 100 ml | Y/10 | – |
| mg $CrO_4$ per 100 ml | $Y \times a/b/10$ | 0,223 084 |
| For 40 $cm^2$ | $Y \times a/b/10$ | 0,223 084 |
| For 1 $cm^2$ | $Y \times a/b/10/40$ | 0,005 577 |
| For 100 $cm^2 = 1\ dm^2$ | $Y \times a/b/10/40 \times 100$ | 0,557 7 |

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