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**Vitreous and porcelain enamels for  
aluminium — Determination of the adhesion  
of enamels on aluminium under the action  
of electrolytic solution (spall test)**

*Émaux vitrifiés déposés sur l'aluminium — Détermination de l'adhérence  
à l'aide d'un électrolyte (essai d'écaillage)*

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## Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

International Standard ISO 13805 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

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## Introduction

The spalling of porcelain enamelled aluminium, a defect characterized by separation of the porcelain enamel from the aluminium base material, is caused by poor adhesion. The spall test is therefore capable of controlling the quality of the enamelling process including the enamel formulation, the preparation of the base metal prior to enamelling, the application and firing procedures, and the aluminium alloy selected for enamelling.

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# Vitreous and porcelain enamels for aluminium — Determination of the adhesion of enamels on aluminium under the action of electrolytic solution (spall test)

## 1 Scope

This International Standard specifies a test method for the accelerated determination of the resistance of porcelain enamel coatings on aluminium and aluminium alloys to spalling as a result of exposure to moisture or weathering. Because spalling is caused by the lack of adhesion between the coating and the base metal, the spall test is a test of adhesion. The greater the extent of spalling in this test, the greater the likelihood that the article will spall in service.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative document indicated below. For undated references, the latest edition of normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3819, *Laboratory glassware — Beakers*.

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*.

ISO 10141, *Vitreous and porcelain enamels — Vocabulary*.

ISO 13805, *Vitreous and porcelain enamels on aluminium — Production of specimens for testing*.

## 3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 10141 apply.

## 4 Principle

A scored enamelled aluminium specimen is exposed to an aqueous test solution consisting of antimony trichloride,  $\text{SbCl}_3$ , having a concentration of 10 g/l, at an ambient temperature of 18 °C to 28 °C for 20 h.

## 5 Designation

The test method for the determination of the adhesion of enamel coatings on aluminium and aluminium alloys by means of the spall test described in this International Standard shall be designated as follows:

**Test ISO 13805**

## 6 Apparatus

### 6.1 Balance.

6.2 **Graduate measuring cylinder**, with a capacity of 1 000 ml in accordance with ISO 4788.

6.3 **Beaker**, in accordance with ISO 3819 or **plastic vessel** of suitable size.

6.4 **Plastic or glass hook**, or **stand**, for holding the specimen in the test solution.

6.5 **Viscose sponge**.

6.6 **File**.

6.7 **Steel needle**.

## 7 Reagents

### 7.1 Test solution

The test solution shall be an aqueous solution of technical grade antimony trichloride,  $\text{SbCl}_3$ , with a concentration of 10 g/l.

Prepare the test solution by dissolving 10 g of antimony trichloride,  $\text{SbCl}_3$ , in 1 000 ml of distilled or demineralized water and stir continuously.

NOTE Hydrochloric acid and a white precipitate consisting of a mixture of antimonious oxychloride,  $\text{SbOCl}$ , and antimony trioxide,  $\text{Sb}_2\text{O}_3$ , will be formed. The precipitate is an integral part of the test solution.

Antimony trichloride is hygroscopic. Therefore keep it in sealed containers. Do not use moist or wet antimony trichloride.

Prepare a fresh solution for each test on the same day as it is to be used.

### 7.2 Cleaning solution

The cleaning solution shall be a sodium *n*-alkyl ( $\text{C}_{10}$ - $\text{C}_{13}$ ) benzene sulphonate with a concentration of active substance of 0,1 % dissolved in distilled or demineralized water.

## 8 Specimen

### 8.1 Selection

The specimen may be a commercially available article or a part of it. Alternatively, it may be a specially prepared specimen in accordance with ISO 13804.

### 8.2 Preparation

Prepare at least one cut edge of the specimen immediately prior to the test. The angle between the cut edge and the surface of the specimen shall be approximately  $90^\circ$ . If required, refinish the cut edge with a file (6.6) where the filing direction shall be from enamel to metal. Wet the cut edge completely with the test solution (7.1) along its whole length.

Score a cross along the whole specimen surface using the steel needle (6.7) such that it extends to the base metal.

### 8.3 Cleaning

The specimen shall be clean and free of grease or similar impurities which may affect the test results. Therefore immerse the specimen in the cleaning solution (7.2) at a temperature of about 40 °C for approximately 3 min. Rinse it in tap water and then in distilled or demineralized water.

## 9 Procedure

The quantity of test solution depends on the size of the specimen and shall be at least 3 ml for each cm<sup>2</sup> of surface area.

Immerse the specimen completely in the test solution (7.1) in a beaker or plastic vessel of suitable size (6.3) for 20 h, suspending the specimen by a plastic or glass hook or by means of a stand (6.4). During this time the ambient temperature shall be between 18 °C and 28 °C.

The test solution (7.1) shall not come into contact with any metal other than the specimen.

After the 20 h test period, take the specimen out of the test solution (7.1), rinse it in water and clean it with the viscose sponge (6.5) to remove loose fragments of enamel. Then dry the specimen in air.

## 10 Expression of results

Examine each specimen visually from a distance of 250 mm. Describe the following types of damage in the test report (see clause 11):

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- any spall area where the basis metal is visible, that is located more than 3 mm from the edge or score mark, and that is longer than 25 mm;
  - any spall area on the specimen surface not touching an edge or a score mark, that is greater than 10 mm<sup>2</sup>;
  - more than one spall area (even if individual areas are less than 10 mm<sup>2</sup>) per dm<sup>2</sup> of the surface area, disregarding pinholes.

## 11 Test report

The test report shall include the following information:

- the type of specimen;
- reference to this International Standard, i.e., determined according to Test ISO 13805;
- the type of damage that occurred, according to the criteria specified in clause 10.

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