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Paints and varnishes — Determination of resistance to liquids —

Part 4: Spotting methods

*Peintures et vernis — Détermination de la résistance aux liquides —
Partie 4: Méthodes à la tache*



Reference number
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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

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

ISO 2812-4 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

ISO 2812 consists of the following parts, under the general title *Paints and varnishes — Determination of resistance to liquids*:

- Part 1: *Immersion in liquids other than water*
- Part 2: *Water immersion method*
- Part 3: *Method using an absorbent medium*
- Part 4: *Spotting methods*
- Part 5: *Temperature-gradient oven method*

Paints and varnishes — Determination of resistance to liquids —

Part 4: Spotting methods

1 Scope

This part of ISO 2812 specifies spotting methods for determining the resistance of an individual layer or multi-layer system of coating materials to the effects of liquids or paste-like products.

These methods enable the testers to determine the effects of the test substance on the coating and, if necessary, to assess the damage to the substrate.

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 1513, *Paints and varnishes — Examination and preparation of samples for testing*

ISO 1514, *Paints and varnishes — Standard panels for testing*

ISO 2808, *Paints and varnishes — Determination of film thickness*

ISO 3270, *Paints and varnishes and their raw materials — Temperatures and humidities for conditioning and testing*

ISO 4628-1, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 1: General introduction and designation system*

ISO 4628-2, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 2: Assessment of degree of blistering*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Principle

A coated test panel is exposed to a test substance using the spotting method. The effects of the exposures are assessed in accordance with agreed criteria.

4 Apparatus

Ordinary laboratory equipment and the following apparatus.

- 4.1 **Pipette**, suitable for applying approximately 0,1 ml droplets of test substance.
- 4.2 **Burette**, 50 ml, for applying test substances.
- 4.3 **Petri dishes** with 60 mm diameter and 20 mm rim.

5 Test substances

One or more test substances, as agreed between interested parties, shall be used. Examples of test substances are given in Annex A.

6 Sampling

Take a representative sample of the coating material to be tested, in accordance with ISO 15528.

Pretest each sample in accordance with ISO 1513 and prepare it for further testing (see 7.2).

7 Test panels

7.1 Substrate

Unless otherwise agreed, use test panels conforming to the requirements of ISO 1514, with dimensions of approximately 150 mm × 100 mm and a thickness of 0,7 mm to 1,0 mm.

7.2 Preparation and coating

Prepare each test panel as described in ISO 1514 and then coat it by the specified application method with the product or system under test. Dry (or stove) and age (if applicable) each coated test panel for the specified time under specified conditions.

7.3 Coating thickness

Determine the dry film thickness of the coating, in micrometres, using one of the non-destructive methods specified in ISO 2808.

8 Procedure

8.1 Conditioning of the test panels

Immediately before testing, condition the test panels for at least 16 h under standard conditions as specified in ISO 3270, i.e. (23 ± 2) °C and (50 ± 5) % relative humidity.

8.2 Test conditions

Carry out the test at the standard temperature specified in ISO 3270, i.e. (23 ± 2) °C.

8.3 Determination

8.3.1 Method A — Horizontal test panel

Perform the test in duplicate.

Place the test panel horizontally. When using liquid test substances, apply them to the test panel with a pipette, making sure that the droplets do not touch one another and that the distance to the edges of the panel is at least 12 mm. Then immediately cover the test areas with the Petri dishes.

When using highly viscous or paste-like test substances, apply about 0,5 cm³ of each substance to the test panel and cover the areas with Petri dishes.

The test duration shall be agreed between the interested parties and should reflect the end use of the coating.

8.3.2 Method B — Inclined test panel

Perform the test in duplicate.

Place the test panel in a collecting vessel at an angle of 30° to the horizontal. With the burette, and at intervals of 1 s to 2 s, apply droplets of the liquid test substance to the upper section of the test panel, close to the centre of the panel, over a period of 10 min. The substance will flow down the test panel into the collecting vessel.

9 Evaluation

After the test period has expired, wipe the test panel with dry cotton wool. Clean off any dried residue of aqueous test substances under running water, and clean off the dried residue of any other test substances with a solvent that does not attack the coating.

Evaluate only the area which has been in direct contact with the test substance.

Immediately assess the test panel for blistering as specified in ISO 4628-2 and in comparison with the non-exposed areas of the panel. Rate any visible alterations as specified in ISO 4628-1.

Unless otherwise agreed, reassess the exposed areas after 24 h.

Further tests on the exposed and non-exposed areas of the test panel may be performed (e.g., cross-cut test, hardness test, etc.) to determine the changes resulting from the effects of the test substances.

If the substrate of the test piece is to be examined for visible alterations, remove the coating using the specified procedure.

If the results of the evaluation of the duplicate determinations differ significantly, repeat the determination, again in duplicate.

Report the results of all determinations, including any repeat determinations.

10 Precision

No details are currently available for the repeatability limit (*r*) and reproducibility limit (*R*).

11 Test report

The test report shall contain at least the following information:

- a) all information necessary for identification of the coating tested, including the manufacturer, trade name, batch number, etc;
- b) a reference to this International Standard (ISO 2812-4:2006);
- c) details of the test panels, including:
 - 1) the material (including thickness) and surface pretreatment of the substrate;
 - 2) the application method for applying the sample coating to the substrate, including the drying time and drying conditions for all layers; where applicable, ageing conditions before the test;
 - 3) the dry film thickness of the coating, in micrometres, including the measuring method chosen in ISO 2808;
- d) the method used (A or B), including:
 - 1) the specification of the test substances;
 - 2) the duration of the test;
 - 3) the temperature;
- e) result(s) of the assessment, as indicated in Clause 9;
- f) the name of the person who conducted the test;
- g) any deviations from the procedure specified;
- h) any unusual features (anomalies) observed during the test;
- i) the date of the test.

Annex A (informative)

Examples of test substances

A.1 General

A range of fuels and chemicals that are typically used as test substances for automotive coatings is given in A.2, A.3 and A.4. Other test liquids may be used for testing both automotive and other coatings.

Use only analytical grade chemicals.

A.2 Fuels and operating fluids for the automotive industry

A.2.1 FAM test substance, conforming to the requirements of DIN 51604-1, DIN 51604-2 or DIN 51604-3.

A.2.2 Diesel fuel, conforming to the requirements of EN 590.

A.2.3 Premium gasoline, conforming to the requirements of EN 228.

A.2.4 Bio-diesel, conforming to the requirements of EN 14214.

A.2.5 Engine oil.

A.2.6 Hypoid gearbox oil.

A.2.7 Hydraulic oil.

A.2.8 Automatic transmission oil.

A.2.9 Brake fluid.

A.2.10 Radiator anti-freeze.

A.2.11 Body sealing compound.

A.2.12 Cavity sealing compound.

A.2.13 Windscreen-washer fluid.

A.2.14 Cold cleaner.

A.3 Laboratory chemicals

A.3.1 Ethanol.

A.3.2 Isopropanol.

A.3.3 Sodium hydroxide solution, with a mass fraction of 5 % sodium hydroxide.

A.3.4 Hydrochloric acid solution, with a mass fraction of 10 % hydrochloric acid.

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A.3.5 Sulfurous acid solution, with a mass fraction of 6 % sulfurous acid.

A.3.6 Sulfuric acid solution, with a mass fraction of 10 % sulfuric acid.

A.3.7 Sulfuric acid solution, with a mass fraction of 36 % sulfuric acid.

A.3.8 Water, conforming to the requirements of Grade 3 of ISO 3696.

A.4 Biological substances

A.4.1 Resin, consisting of:

- rosin 50 % (mass fraction)
- pine oil 50 % (mass fraction).

A.4.2 Fall-out test substance, consisting of:

- e.g. formic acid 47 % (mass fraction)
- tannic acid 24 % (mass fraction)
- albumin 5 % (mass fraction)
- honey 24 % (mass fraction).

A.4.3 Gum arabic, e.g. acacia gum.

A.4.4 Rosin.

A.4.5 Simulated bird droppings: pancreatin, 1:1 diluted with water of grade 3 in accordance with ISO 3696.

Pancreatin may be ground in a mortar, if agreed between the interested parties. If the pancreatin is ground, this shall be stated in the test report.

Bibliography

- [1] ISO 3696, *Water for analytical laboratory use — Specification and test methods*
- [2] EN 228, *Automotive fuels — Unleaded petrol — Requirements and test methods*
- [3] EN 590, *Automotive fuels — Diesel — Requirements and test methods*
- [4] EN 14214, *Automotive fuels — Fatty acid methyl esters (FAME) for diesel engines — Requirements and test methods*
- [5] DIN 51604-1, *FAM testing fluid for polymer materials; composition and requirements*
- [6] DIN 51604-2, *Methanolic FAM testing fluid for polymer materials; composition and requirements*
- [7] DIN 51604-3, *Methanolic lower layer FAM testing fluid for polymer materials; composition and requirements*

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