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# International Standard



# 6441

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

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## Paints and varnishes — Indentation test (spherical or pyramidal)

*Peintures et vernis — Essai d'indentation (sphérique ou pyramidal)*

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Descriptors : paints, varnishes, tests, indentation hardness tests.

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

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.

International Standard ISO 6441 was developed by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the member bodies in January 1983.

It has been approved by the member bodies of the following countries :

Australia	Hungary	South Africa, Rep. of
Belgium	India	Sri Lanka
Canada	Iran	Sweden
China	Israel	Switzerland
Egypt, Arab Rep. of	Italy	United Kingdom
France	Netherlands	USSR
Germany, F.R.	Poland	

No member body expressed disapproval of the document.

# Paints and varnishes — Indentation test (spherical or pyramidal)

## 0 Introduction

This International Standard is one of a series of standards dealing with the sampling and testing of paints, varnishes and related products.

The measurement of the resistance to indentation of any particular coating applied to a substrate which does not deform under the conditions of test is dependent, to a greater or lesser extent, on the shape and size of the indenter, the load applied, the time under load, the temperature and the coating thickness. The effect of such variables on the resistance to indentation of the coating may be used to characterize mechanical properties in the form of relationships between load and indentation depth or between indentation depth and temperature.

Although this International Standard defines a procedure for measuring the resistance to indentation of a coating under defined conditions with a greater measure of reproducibility than that obtainable using the Buchholz method described in ISO 2815, it is considered that the present state of knowledge concerning the experimental factors that can affect indentation measurement does not permit a full definition of a method of test. Nevertheless the procedure described below is recommended for the conduct of such a test, to be reviewed in the light of further knowledge and experience.

For any particular application, the test method described in this International Standard needs to be completed by the following supplementary information. This information should be derived, in part or totally, from an (inter)national standard or other document related to the product under test or, if appropriate, should be agreed between the interested parties.

- a) Material and surface preparation of the substrate.
- b) Method of application of the test coating to the substrate.
- c) Duration and conditions of drying the coat (or conditions of stoving and ageing, if applicable) before testing.
- d) Thickness, in micrometres, of the dry coating and method of measurement in accordance with ISO 2808, and whether it is a single coating or a multi-coat system (see the note to 6.2).
- e) Temperature and humidity for the test, if different from those specified in 6.3 (see ISO 3270).
- f) The type of indenter.

g) The mass, in grams, and the application time, in seconds, of the contact load (see also the note to 7.2.2).

h) The mass, in grams, and the application time, in seconds, of the test load.

## 1 Scope and field of application

This International Standard describes a test procedure for the conduct of an indentation test on a single or multi-coat system of paint, varnish or related product. The result is expressed as the depth of indentation produced when an indenter of specified size and shape is applied to the coating under defined conditions.

The method is applicable if the indentation does not exceed approximately 10 % of the total coating thickness.

## 2 References

- ISO 1512, *Paints and varnishes — Sampling.*
- 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 2815, *Paints and varnishes — Buchholz indentation test.*
- ISO 3270, *Paints and varnishes and their raw materials — Temperatures and humidities for conditioning and testing.*
- ISO 6507/1, *Metallic materials — Hardness test — Vickers test — Part 1 : HV 5 to HV 100.*

## 3 Definitions

**3.1 contact load** : The load that is first applied to the indenter in order to obtain reproducible contact between the indenter and the surface of the test specimen without causing undue deformation of the surface.

**3.2 test load** : The load that is applied to the indenter in order to deform the specimen to a measurable extent.

## 4 Apparatus

### 4.1 Indentation apparatus

The indentation apparatus shall be constructed so that two loads, the contact load and the test load, can successively be applied to an indenter which is free to press into the test specimen. The depth of indentation of the specimen by the indenter shall be capable of measurement whilst under load to  $0,2 \mu\text{m}$ . The apparatus shall secure the test specimen in such a way that it does not move under the applied load.

NOTE — Reproducibility and repeatability of results can be markedly affected by the temperature control of the stage to which the specimen is attached. Without careful temperature control of the specimen the precision of the measurement as indicated in 9.1 and 9.2 will not be attained, especially in those cases where the glass transition temperature  $T_g$  of the coating is in the region of the test temperature.

### 4.2 Indentor

The tip of the indentor shall be either spherical or pyramidal in form.

#### 4.2.1 Spherical indentor

The spherical indentor shall have a non-deformable hemispherical tip.

#### NOTES

1 If an indentor with a diameter of 1 mm is to be used, it may conveniently be made by soldering a 1 mm diameter steel ball of the type used in ball bearings (1 % C, 1 % Cr type steel) to a steel shank with the end recessed to facilitate soldering.

2 It has been found that for a given depth of indentation, results obtained using the pyramidal indentor (4.2.2) are less dependent on variation in thickness of the coating under test than are those obtained with a 1 mm spherical indentor.

The results obtained with spherical indentors with diameters less than 1 mm are, however, less dependent on coating thickness.

#### 4.2.2 Pyramidal indentor

The pyramidal indentor shall have a diamond tip in the form of a right pyramid with a square base with dimensions that comply with the requirements of ISO 6507/1.

### 4.3 Timer

A stop-watch or other suitable timing device.

## 5 Sampling

Take a representative sample of the product to be tested (or of each product in the case of a multi-coat system) as described in ISO 1512.

Examine and prepare each sample for testing as described in ISO 1513.

## 6 Test panels

### 6.1 Materials and dimensions

The test panel shall be of steel, glass or hard aluminium complying with the requirements of ISO 1514. Each test panel shall be plane and free from distortion and the test surface shall be free from visible ridges or cracks.

Unless otherwise specified, the test panel shall be  $150 \text{ mm} \times 100 \text{ mm}$  and not less than 1 mm in thickness. The test panel may be cut to size after coating and drying, provided that no distortion occurs in the test coating.

### 6.2 Preparation and coating

Unless otherwise specified, prepare each test panel in accordance with ISO 1514 and then coat it by the specified method with the product or system under test to the thickness specified for each coat.

NOTE — In order to minimize the effect of coating thickness on the depth of indentation, it is recommended that the conditions of test and the thickness of the coating be specified where possible such that the expected depth of indentation does not exceed approximately 10 % of the coating thickness.

### 6.3 Drying and conditioning

Dry (or stove and age) the coated test panel for the specified time and under the specified conditions unless otherwise specified. Then condition the coated panels at  $23 \pm 2 \text{ }^\circ\text{C}$  and a relative humidity of  $50 \pm 5 \%$  for a minimum of 16 h. (See also ISO 3270.) Carry out the test procedure as soon as possible.

### 6.4 Thickness of coating

Determine the thickness, in micrometres, of the dry coating by one of the procedures described in ISO 2808.

## 7 Procedure

### 7.1 Test conditions

Carry out the test in duplicate at  $23 \pm 2 \text{ }^\circ\text{C}$  and a relative humidity of  $50 \pm 5 \%$ , unless otherwise agreed. (See also ISO 3270.)

### 7.2 Determination of indentation depth

**7.2.1** Fit the specified indentor (4.2) in the apparatus (4.1) and adjust the apparatus for sensitivity and/or calibration as required.

**7.2.2** Place the test panel in the apparatus and apply the specified contact load for the specified time, setting the apparatus to read zero indentation or otherwise establishing a vertical datum reading.

NOTE — As this contact forms the basis of the measurement of the penetration of the indentor under the test load, the contact load should

be specified to be of such magnitude as to make good contact between the indenter and the specimen surface without causing undue deformation of the specimen. The contact load should be the minimum required to ensure reproducible contact with the specimen and should not exceed 10 % of the test load.

**7.2.3** Measure, while under load, the indentation depth, in micrometres, after applying the specified test load to the contact load for the specified time. Record the result.

**7.2.4** Carry out six determinations on different parts of the same test panel.

## 8 Expression of results

Report the mean value of the six determinations, to the nearest 0,1  $\mu\text{m}$ , as the result.

## 9 Precision

### 9.1 Repeatability ( $r$ )

The value below which the difference between two test results, each the mean of six determinations, obtained by one operator in one laboratory, using the same equipment within a short interval of time and using the standardized test method on different panels prepared from the same sample, may be expected to lie with a 95 % probability, is 0,8  $\mu\text{m}$ .

### 9.2 Reproducibility ( $R$ )

The value below which the difference between two test results, each the mean of six determinations, obtained by operators in different laboratories, using the standardized test method on panels prepared from identical samples, may be expected to lie with a 95 % probability, is 1,5  $\mu\text{m}$ .

## 10 Test report

The test report shall contain at least the following information :

- a) the type and identification of the product tested;
- b) a reference to this International Standard (ISO 6441);
- c) the items of supplementary information referred to in the introduction to this International Standard;
- d) a reference to the (inter)national standard, product specification or other document supplying the information referred to in c) above;
- e) the results of the test, as indicated in clause 8;
- f) any deviation, by agreement or otherwise, from the test method specified;
- g) the date(s) of the test.

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