INTERNATIONAL STANDARD

ISO 6441-2

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Paints and varnishes — Determination of micro-indentation hardness —

Part 2: **Knoop hardness by measurement of indentation depth under load**

Peintures et vernis — Détermination de la dureté par micro-indentation —

Partie 2: Dureté Knoop par mesurage de la profondeur d'indentation sous charge

Reference number
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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 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.

Attention is drawn to the possibility that some of the elements of this part of ISO 6441 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 6441-2 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

Together with the other parts (see below), it cancels and replaces ISO 6441:1984, which has been technically revised.

ISO 6441 consists of the following parts, under the general title *Paints and varnishes — Determination of micro-indentation hardness*:

— *Part 1: Knoop hardness by measurement of indentation length*

— *Part 2: Knoop hardness by measurement of indentation depth under load*

— *Part 3: Creep and visco-elastic properties and elastic modulus using the Vickers indenter*

Annex A forms a normative part of this part of ISO 6441.

Introduction

This part of ISO 6441 is one of three dealing with the determination of the micro-indentation hardness of a dried film of paint, varnish or related product.

Part 1 specifies a method in which the hardness is calculated from the length, measured using a microscope, of a long diagonal of the indentation made by a Knoop indenter.

In this method, the indentation length is measured after the indenter has been removed.

Part 2 specifies a method in which the hardness is calculated from the depth, measured using an electronic technique, of the indentation made by a Knoop indenter.

In this method, the indentation depth is measured while the indenter is still under load.

Part 3 specifies a method in which the hardness, creep, visco-elasticity and modulus of elasticity are determined by measuring the depth of the indentation made by a Vickers indenter as a function of time.

Other methods have been standardized to measure the hardness of a paint film:

— ISO 1518, *Scratch test.*

— ISO 15184, *Determination of film hardness by pencil test.*

— ISO 2815, *Buchholz indentation test.*

— ISO 1522, *Pendulum damping test.*

The method chosen depends on the property which is to be measured. All these tests differ from each other technically, and in their accuracy. They also range from fairly simple to rather sophisticated, with the instruments prescribed in ISO 6441, part 1, part 2 and part 3, being of a sophisticated nature.

Paints and varnishes — Determination of micro-indentation hardness —

Part 2:
Knoop hardness by measurement of indentation depth under load

1 Scope

This part of ISO 6441 is one of a series of standards dealing with the sampling and testing of paints, varnishes and related products.

It specifies a method for determining the hardness of a dried film of a paint, varnish or related product by conducting an indentation test in which the indentation depth is measured with the indenter still under load, using electronic techniques.

It is applicable to single-coat and multicoat systems.

The result is expressed as a hardness value, in N/mm^2, calculated from the indentation produced by a pyramidal indenter of a specified size and shape applied to the coating under specified conditions.

This method give similar results to those of part 1 of this International Standard and Test Method A of ASTM D 1474-92 (see reference [1] in the Bibliography).

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 6441. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 6441 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 1513:1992, *Paints and varnishes — Examination and preparation of samples for testing.*

ISO 1514:1993, *Paints and varnishes — Standard panels for testing.*

ISO 2808:1997, *Paints and varnishes — Determination of film thickness.*

ISO 3270:1984, *Paints and varnishes and their raw materials — Temperatures and humidities for conditioning and testing.*

ISO 15528:—[1], *Paints, varnishes and raw materials for paints and varnishes — Sampling.*

1) To be published. (Revision of ISO 842:1984 and ISO 1512:1991)

3 Principle

The hardness of the surface of a dried paint or varnish film is measured, under specific conditions, with a pyramidal indenter (Knoop indenter) fitted to a micro-hardness tester.

The Knoop hardness is defined as the applied load divided by the projected area of the indentation in the plane of the surface of the paint film (see Figure 3).

During the test, the Knoop indenter is kept under a specified load for (18 ± 0,5) s.

At the end of the test, the depth of the indentation made by the indenter, still under load, is measured eletronically and, from the depth, the area of the indentation in the plane of the surface of the paint film is calculated, and hence the Knoop hardness.

This method is applicable provided that the indentation depth does not exceed approximately 25 % of the coating thickness.

Exceeding this limit will result in the substrate influencing the penetration depth.

Further, the hardness value of the coating can be influenced (lowered) by a substrate material of much lower hardness than the coating.

4 Required supplementary information

For any particular application, the test method specified in this part of ISO 6441 needs to be completed by supplementary information. The items of supplementary information are given in annex A.

5 Apparatus

5.1 Hardness tester, including a Knoop indenter (5.1.1), a microscope (5.1.2) and electronic equipment to measure the identation depth. This apparatus shall be capable of:

— Bringing the indenter into contact with the test surface at a specified speed, in accordance with the manufacturer's instructions, with negligible impact under a nearly zero load. Any impact by the indenter on the test surface will result in errors in readings and the calculated hardness will be too low.

— Progressively applying a selected load to the indenter and maintaining the load for (18 ± 0,5) s.

— At the end of the 18 s indentation period, measuring the depth of the indentation before unloading the indentor.

NOTE This part of ISO 6441 is based on a new type of instrument, connected to a computer operated using suitable software. The main characteristics of the instrument are:

— the load on the indenter is applied under electronic control and can be increased in small steps;

— during indentation, the indentation depth is continuously measured electronically;

— the apparatus adjusts its zero point, the point from which the actual measurement starts, automatically.

5.1.1 Knoop indenter: a pyramidal diamond with a longitudinal included angle at the vertex of 172° 30' and a transverse included angle of 130° (see Figure 1).

The four faces shall be equally inclined to the axis of the indenter (within ± 0,2°) and shall meet at the vertex, any offset between opposite faces not exceeding 1 µm (the usual shape of the point as it would appear under high magnification is shown in Figure 2).

NOTE 1 The ratio of the long diagonal to the short diagonal of the indentation is approximately 7:1; the ratio of the long diagonal to the depth of penetration is approximately 30:1.

NOTE 2 The manufacturer normally provides with each indenter a certificate stating the dimensions of the indenter. The certificate also gives the indenter constant, calculated from the dimensions.

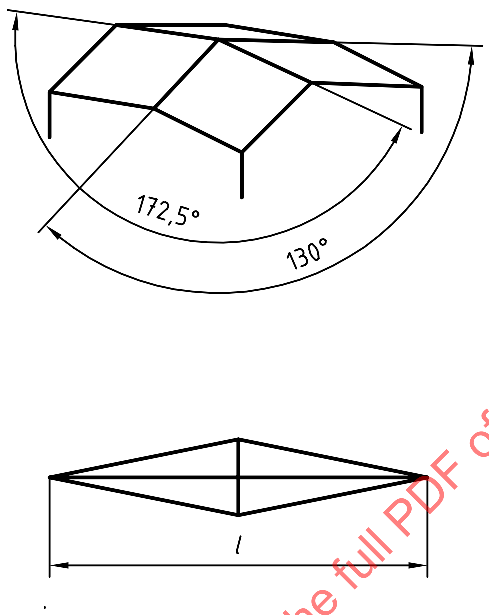
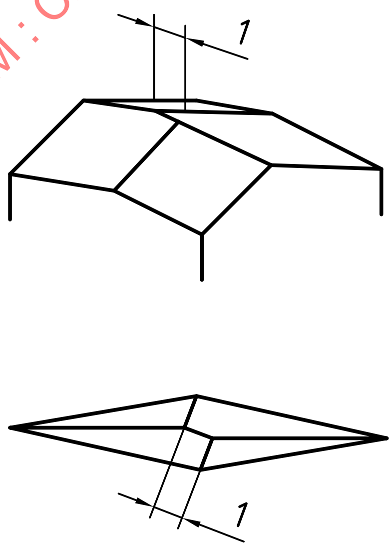


Figure 1 — Knoop indenter



Key

1 Offset (1 µm max.)

Figure 2 — Indenter offset

5.1.2 Microscope, with a movable stage, to find clean, dust-free areas on the surface of the test specimen.

5.2 Standard test blocks, for verifying the accuracy of the measurements.

It is recommended that the measurements be compared with those obtained using standard test blocks chosen so that their hardnesses cover the whole hardness range of interest.

Each test block shall be of compact-grained metal and shall have a known uniform hardness measured at the particular test force specified by the calibration authorities or the test instrument manufacturer. The test force shall be the same as that used in the actual tests.

NOTE A suitable source of standard reference materials in this hardness range is available from NIST[2]. By agreement between the interested parties, a stable specimen (such as an aged coating or a baked enamel with a Knoop hardness H_K < 200 N/mm^2, applied to a flat substrate) could be used to calibrate the hardness tester.

5.3 Means of fixing the test specimen rigidly to the moving table of the hardness tester.

NOTE Vibration represents a serious source of error, irrespective of the force applied, but the effects are far more evident with small forces. In general, lower hardness values are obtained if vibrations are present. This source of error can be detected by comparative measurements on a specimen of known hardness nearly equal to the hardness of the test surface. The effects of vibration can be reduced by mounting the test specimen on a rigid support.

6 Sampling

Take a representative sample of the product to be tested (or of each product in the case of a multicoat system), as described in ISO 15528.

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

7 Test panels

7.1 Substrate

Unless otherwise agreed, select the substrate from one of those described in ISO 1514, using, where possible, the same type of material as will be used in practice. The substrate panels shall be plane and free from distortion.

NOTE Surface curvature can introduce an error in the determination of the hardness, which increases as the radius diminishes. On convex surfaces, higher hardness readings are obtained than on plane surfaces, while on concave surfaces lower hardness readings are obtained.

7.2 Shape and dimensions

The shape and dimensions shall be such that the test panels can be fitted correctly in the apparatus.

NOTE Typical dimensions for a test panel are 100 mm × 100 mm. Depending on the type of test instrument, other dimensions are possible.

2) National Institute for Standards and Technology, Standard Reference Material Program, Building 202, Room 204, Gaithersburg, MD 20899, USA.

7.3 Preparation and coating

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

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 so that the expected depth of indentation does not exceed approximately 25 % of the coating thickness.

7.4 Drying and conditioning

Dry (or stove) and age, if applicable, each coated test panel for the specified time under the specified conditions. Before testing, condition the coated panels at a temperature of (23 ± 2) °C and a relative humidity of (50 ± 5) % in accordance with ISO 3270, unless otherwise agreed, for a minimum period of 16 h.

7.5 Thickness of coating

Determine the thickness, in micrometres, of the dried coating by one of the procedures specified in ISO 2808.

8 Calibration of the apparatus

Calibrate the apparatus in accordance with the manufacturer's instructions.

Determine the Knoop hardness on a standard test block (see 5.2). If the value obtained is within ± 5 % of the assigned value, the instrument is considered to be calibrated.

9 Procedure

9.1 Test conditions

Carry out the test at a temperature of (23 ± 2) °C and a relative humidity of (50 ± 5) %, unless otherwise agreed.

9.2 Preparation for the test

Attach the specimen securely to the moving table so that the test surface is normal to the direction of indentation and so that it cannot move in any direction with respect to the mount during the test.

Use the microscope at a magnification of for example × 10 to × 20 to select an area of the test specimen that is free of surface irregularities and imperfections.

Place this area under the indenter by means of the movable micrometer stage.

NOTE Rough surfaces can result in less accurate measurement of the indentation depth. In such cases, the surface may be gently polished, if agreed between the interested parties.

9.3 Determination

Adjust the test load to the value agreed by the interested parties.

Several paint systems of different hardnesses can be compared within one series by measuring all the systems with the same load despite the fact that not all the indentation depths are within 25 % of the coating thickness. If this is done, it shall be reported in the test report.

Perform the test in accordance with the manufacturer's instructions. After the instrument has adjusted itself to the zero point, the indenter will remain in contact with the specimen for (18 ± 0,5) s.

The indentation depth at the end of the 18 s period is measured automatically and recorded by the computer.

From the indentation depth, the Knoop hardness is normally calculated automatically by the computer.

NOTE In order for the computer to carry out this calculation, the indenter constant and the load will have had to have already been stored in the computer.

Repeat the determination at least two times at widely spaced locations on the specimen. This will result in three determinations.

10 Expression of results

10.1 The mean indendation depth and the mean Knoop hardness value will normally be calculated automatically by the computer. If not, calculate them as follows:

10.2 For each specimen, calculate the mean indentation depth for the three determinations and express this in millimetres.

10.3 Convert this mean indentation depth to the Knoop hardness value H_K by means of the equation

$$H_K = \frac{P}{A} = \frac{P}{d^2 \times C}$$

where

P is the load applied to the indenter, in newtons;

d is the mean indentation depth, in millimetres;

C is the indenter constant (= 65,438) (see note 2 to 5.1.1 and the note below);

A is the projected area of indentation, in square millimetres.

NOTE The indenter constant C is a factor for calculating the projected area, in mm^2, of the impression (see Figure 3). The value will vary slightly, depending on the actual dimensions of the indenter.

11 Precision

11.1 General

Precision data for this method are not available yet, but it is expected that the precision of this method is at least as good as the precision of the method described in ISO 6441-1. The precision data given below has been taken from ASTM D 1474-92 [1], simply to give an idea of the precision of the measurements.

11.2 Repeatability

Two results, each the mean of three determinations on a specimen, obtained by the same operator in one laboratory should be considered suspect if they differ by more than 9 % of their mean value.