Designation: D1474/D1474M – 13

Standard Test Methods for Indentation Hardness of Organic Coatings[1]

This standard is issued under the fixed designation D1474/D1474M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 These test methods cover the determination of the indentation hardness of organic materials such as dried paint, varnish, and lacquer coatings, when applied to an acceptable plane rigid surface, for example, metal or glass.

1.2 Two methods are covered as follows:

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Method A—Knoop Indentation Hardness	6 – 12
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1.3 Method A, which has the greater precision, provides hardness values in terms of Knoop Hardness Number (KHN). Method B provides hardness in terms of Pfund Hardness Number (PHN). Although the hardness value scales of these methods differ, the methods agree in the ranking of coating hardness.

1.4 Test Method A of these test methods is similar in content (but not technically equivalent) to ISO 6441-1and ISO 6441-2.

1.5 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.6 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D823 Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels

D1005 Test Method for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers

D7091 Practice for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to Ferrous Metals and Nonmagnetic, Nonconductive Coatings Applied to Non-Ferrous Metals

E384 Test Method for Knoop and Vickers Hardness of Materials

2.2 *Other Standards:*

ISO 6441-1 Paints and Varnishes—Determination of micro-indentation hardness—Part 1: Knoop hardness by measurement of the indentation length[3]

ISO 6441-2 Paints and Varnishes—Determination of micro-indentation hardness—Part 2: Knoop hardness by measurement of the indentation depth under load[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *indentation hardness, n*—the resistance to penetration by an indenter.

3.1.2 *Knoop indenter, n*—a pyramidal diamond of prescribed dimensions.

3.1.3 *Pfund indenter, n*—hemispherical quartz or sapphire indenter of prescribed dimensions.

3.1.4 *Knoop hardness number, KHN, n*—the indentation hardness determined with a Knoop indenter, and calculated as follows:

$$KHN = L/A_p = L/l^2C_p \quad (1)$$

where:

L = load applied to the indenter, kg,
l = measured length of long diagonal of the indentation, mm,
C_p = indenter constant relating l^2 to A_p, and
A_p = projected area of indentation, mm^2.

[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

***A Summary of Changes section appears at the end of this standard**

3.1.5 *Pfund hardness number, PHN, n*—the indentation hardness determined with a Pfund indenter, and calculated as follows:

$$\text{PHN} = L/A = 4L/\pi d^2 = 1.27\,(L/d^2) \quad (2)$$

where:

L = load/kg applied to the indenter, kg,
A = area of projected indentation, mm^2, and
d = diameter of projected indentation, mm.

4. Significance and Use

4.1 Indentation hardness measurements have proven to be useful in rating coatings on rigid substrates for their resistance to mechanical abuse, such as that produced by blows, gouging, and scratching. These measurements do not necessarily characterize the resistance to mechanical abuse of coatings that are required to remain intact when deformed.

5. Test Specimens

5.1 The substrate for the coating shall be an acceptable plane rigid surface such as glass or metal.

5.2 The coating thickness on any one panel shall be uniform within 3 µm [0.1 mil]. Coatings to be compared shall be of equal thickness within 5 µm [0.2 mil]. For maximum accuracy, the minimum permissible coating thickness shall be such that the depth of indentation does not exceed three fourths of the coating thickness, to minimize the effect of the substrate.

5.3 At least three replicate specimens shall be tested for each coating to be evaluated.

5.4 Coatings should be applied in accordance with Practices D823 and their dry film thickness should be measured in accordance with Test Methods D1005 or D7091.

5.5 From precoated sheets cut at least three specimens. Use only plane (flat) sheets and round the edges slightly. The coating shall be free of oil and other foreign matter. Measure the film thickness as in 5.4.

METHOD A—KNOOP INDENTATION HARDNESS

6. Summary of Method

6.1 This method consists of applying a load to the surface of a coating by means of a pyramidal shaped diamond having specified face angles, and converting the measurements of the resultant permanent impression to a hardness number.

7. Apparatus

7.1 *Hardness Tester*[4], consisting of a load applicator, a Knoop indenter, and a microscope fitted with a movable micrometer stage. The apparatus shall mechanically bring the indenter into contact with the test surface with negligible impact, apply the selected full load, maintain it for 18 ± 0.5 s, and withdraw the indenter.

7.2 *Knoop Indenter*—The Knoop indenter is a pyramidal diamond with included longitudinal angles of 172° 30' and included transverse angle of 130° 0'.

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

7.3 *Microscope*—The microscope shall have a filar micrometer eyepiece and sufficient objectives to permit the measurement of the length of impression to within ±1 %. The specimen shall be firmly supported on a movable micrometer stage attached to the microscope.

8. Calibration

8.1 Adjust the illumination in the microscope to give maximum contrast when viewing an indentation.

8.2 By means of a calibrated scale, determine the factor for each microscope objective that converts the filar scale units of the eyepiece to millimetres.

8.3 With a 25-g load on the indenter, determine the KHN of a calibrated standard (Note 2) with an assigned value not greater than 50 KHN. If the obtained value is within ±5 % of the assigned value, the instrument is considered to be in calibration.

NOTE 2—A suitable source of standard reference materials in this hardness range is available from the U.S. National Institute for Standards and Technology.[5] By agreement of the parties concerned, a stable specimen (such as an aged coating or a baked enamel applied to a flat substrate) could be used to calibrate the participating hardness testers.

9. Procedure

9.1 Unless otherwise specified, make the hardness determinations at 23 ± 2°C [73.5 ± 3.50°F] and 50 ± 5 % relative humidity after equilibrating the specimens under these conditions for at least 24 hours.

9.2 Rigidly attach the specimen to the movable stage so that the surface to be measured is normal to the direction of indentation. Mount the panel so that is cannot move with respect to the stage in any direction during the test.

9.3 Use the microscope 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 3—If good impressions cannot be obtained because of the roughness of the surface of the specimen, gently polish the surface with No. 400 carborundum and finish off with jewelers rouge before making the impression.

9.4 Present the apparatus to apply a load that permits the length of identification to be read accurately but does not cause the depth of indentation to exceed three-quarters of the coating film thickness. Start the test cycle so that the indenter is mechanically brought into contact with the mounted specimen under a load of 25 g and full load is applied, maintained for 18 ± 0.5 seconds, and removed. For maximum accuracy, ensure that the indenter has not penetrated the coating to a depth

[4] A hardness tester meeting the apparatus requirements for this method is the Tukon Microhardness Tester, available from the Wilson Instruments, Inc., Division of Instron, 100 Royall St., Canton, MA 02021.

[5] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

beyond three-fourths of the coating thickness. This is necessary to eliminate any major substrate effect on the hardness values.

NOTE 4—For maximum accuracy, care must be taken that the indenter does not penetrate the coating to a depth beyond three fourths of the coating thickness. This is necessary to eliminate any major substrate effect on the hardness measurement.

9.5 Immediately after the completion of the cycle, adjust the movable stage so that the indentation is in the field of the microscope. Focus the microscope on the indentation so that both extremities of the long diagonal (that is, where the upper edges of the indentation just converge) are as sharp as possible. Measure the length of the long diagonal of the impression with the filar micrometer eyepiece.

NOTE 5—Select a microscope objective that will cause the length of impression to be between 200 and 800 filar units to assure maximum accuracy in measurement.

9.6 From the measurements obtained in 9.5, the information given in Note 1, and the measured film thickness at the place of indentation, calculate the depth of indenter penetration. If the depth of penetration exceeds three-fourths of the coating thickness, the results may be influenced by substrate proximity. Consequently, repeat the test with specimens having a greater film thickness or with a lighter load on the indenter.

9.7 Since the applied load is not always the same and, practically never 25 g, calculate the Knoop Hardness Number using the equation provided by the manufacturer:

$$KHN = L/l^2C_p \quad (3)$$

where:

L = load applied, kg, to the indenter,
l = length of long diagonal of indentation, mm, and
C_p = indenter constant = 7.028×10^{-2}.

9.8 The Knoop Hardness Numbers for a test load of one (1) gf and length of long diagonal of indentation from 1.0 to 200.9 µm are given in Table 1 of Test Method E384.

10. Calculation

10.1 Calculate the mean indentation length in filar units.

10.2 Convert this mean indentation length to KHN by means of the appropriate tables supplied with the instrument.

NOTE 6—If a conversion table is not available, the KHN may be calculated as follows:

$$KHN = 0.025/l^2C_p \quad (4)$$

where:

0.025 = load applied, kg, to the indenter,
l = length of long diagonal of indentation, mm, and
C_p = indenter constant = 7.028×10^{-2}.

11. Report

11.1 Report the following information:

11.1.1 Mean and range of KHN values obtained for each specimen, stating the number of indentations made and the indenter load used,

11.1.2 Mean film thickness of each specimen, based on the measurements made at the points of indentation,

11.1.3 Specimen preparation and conditioning techniques used,

11.1.4 Mean and range of KHN values of the replicate panels, and

11.1.5 Substrate material of panel.

12. Precision and Bias

12.1 *Precision*—On the basis of an interlaboratory test of this method in which operators in six laboratories tested seven coated panels having a broad range of hardness, the within-laboratory coefficient of variation was found to be 3 % with 21 degrees of freedom and the between-laboratories coefficient of variation 8 % with 30 degrees of freedom. Based upon these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

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

12.1.2 *Reproducibility*—Two results, each the mean of three determinations on a specimen, obtained by operators in different laboratories should be considered suspect if they differ by more than 24 % of their mean value.

12.1.3 *Bias*—The bias for the procedure Test Method A for measuring indentation hardness using the Knoop indenter will be determined using data previously generated in round-robin testing.

METHOD B—PFUND INDENTATION HARDNESS

13. Summary of Method

13.1 This method consists of applying a load to the surface of a coating, by means of a transparent colorless quartz or synthetic sapphire hemisphere having a specified diameter, and converting the measurement of the resultant observed impression under load to a hardness number.

14. Apparatus

14.1 *Hardness Testers*—consisting of a load applicator, a Pfund quartz or sapphire indenter, and a microscope with a stage. The apparatus shall be constructed so as to permit the indenter to be brought manually into contact with the specimen surface with negligible impact.

14.2 *Pfund Indenter*[6]—The Pfund indenter (Fig. 1) is a transparent colorless quartz or synthetic sapphire hemisphere with a spherical radius of 3.18 mm [0.125 in.] and a maximum spherical eccentricity of 0.05 mm [0.002 in.].

14.3 *Microscope*—The microscope shall have a filar micrometer eyepiece and sufficient objectives to permit the measurement of the diameter of impression to within ±1 %. The specimen shall be rigidly supported on the microscope stage.

14.4 *Timer,* capable of measuring a time interval of 60 ± 0.5 seconds.

[6] A hardness tester meeting the apparatus requirements of this test method is the Pfund Indentation Hardness Tester. The instrument is no longer manufactured, but many are still in use.

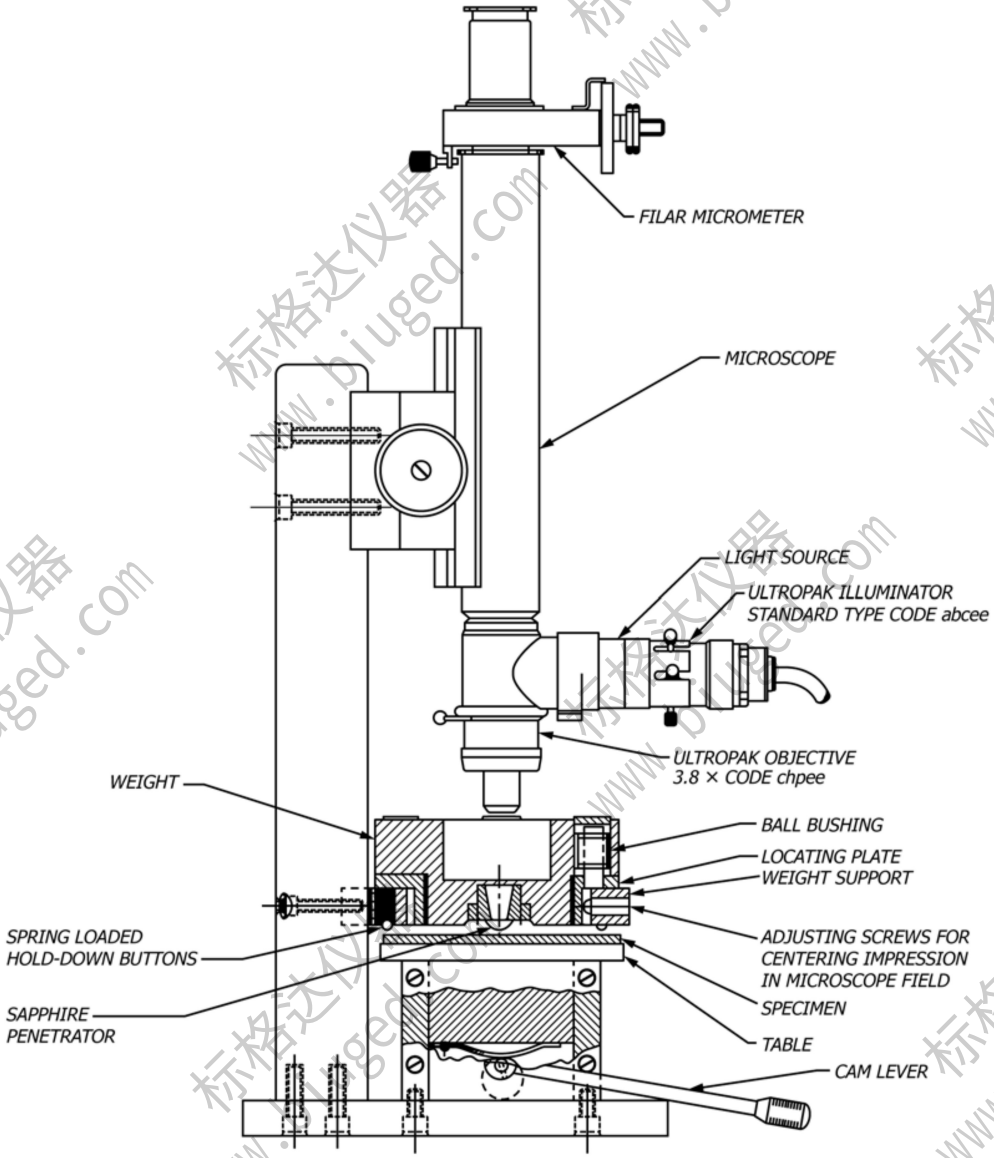

FIG. 1 A Pfund Hardness Tester (Shown Before Application of Weight)

15. Standardization

15.1 Adjust the illumination in the microscope to give maximum contrast when viewing an indentation.

15.2 By means of a calibrated scale, determine the factor for each microscope objective that converts the filar scale units of the eyepiece to millimetres.

15.3 With a 1.0-kg load on the indenter, determine the PHN of a calibrated standard (Note 2) with an assigned value not greater than 40 PHN. If the value obtained is within ±5 % of the assigned value, the instrument is considered to be in calibration.

16. Procedure

16.1 Unless otherwise specified, make the hardness determination at 23 ± 2°C [73.5 ± 3.5°F] and 50 ± 5 % relative humidity, after holding the specimens under these conditions for not less than 24 hours.

16.2 Rigidly attach the specimens to the instrument stage such that the surface to be measured is normal to the direction of indentation (Note 2).

16.3 With a 1.0-kg load on the indenter, carefully bring the indenter into contact with the specimen surface, apply the full load, and start the timer.

16.4 At the end of 60 seconds, while still under full load, rapidly measure the diameter of the circular impression with the filar eyepiece of the microscope (Note 5). It is very important that the diameter measurements be made rapidly so that the time the indenter is in contact with the specimen is closely controlled.

16.5 From the measurements obtained 16.4, the information given in 14.2, and the measured film thickness at the place of indentation, calculate the depth of indenter penetration. If the depth of penetration exceeds three fourths of the coating thickness, the results may be influenced by substrate proximity. Consequently, repeat the test with specimens having a greater film thickness or with a lighter load on the indenter.

16.6 Repeat the procedure in 16.3 and 16.4 until a total of at least five impressions have been made at widely spaced locations on the specimen.

17. Calculation

17.1 Calculate the mean indentation diameter in filar units.

17.2 Convert this mean indentation diameter to millimetres using the appropriate factor determined in 15.2.

17.3 Calculate the PHN as follows:

$$\text{PHN} = 1.27/d^2 \quad (5)$$

where:

d = mean indentation diameter, mm.

18. Report

18.1 Report the following information:

18.1.1 Mean and range of PHN values obtained for each specimen, stating the number of indentations made,

18.1.2 Mean film thickness of the specimen, based on measurements made near the points of indentation,

18.1.3 Specimen preparation and conditioning techniques used,

18.1.4 Mean and range of PHN values of the replicate panels, and

18.1.5 Substrate material.

19. Precision and Bias

19.1 *Precision*—On the basis of an interlaboratory test of this method in which operators in four laboratories tested seven coated panels having a broad range of hardness, the within-laboratory coefficient of variation was found to be 6 % with 28 df and the between-laboratories coefficient 12 % with 18 df. Based upon these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

19.1.1 *Repeatability*—Two results, each the mean of three determinations on a specimen, obtained by the same operator should be considered suspect if they differ by more than 18 % of their mean value.

19.1.2 *Reproducibility*—Two results, each the mean of three determinations on a specimen, obtained by operators in different laboratories should be considered suspect if they differ by more than 36 % of their mean value.

19.2 *Bias*—The bias for the procedure Test Method B for measuring indentation hardness using the Pfund indenter will be determined using data previously generated in round-robin testing.

20. Keywords

20.1 hardness (indentation); hardness (Knoop); hardness (Pfund); Knoop indenter; Pfund hardness tester; Pfund indenter

SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue (D1474-98(2008)) that may impact the use of this standard. (Approved November 1, 2013.)

(1) Various editorial changes, including consistency of SI units as primary throughout.

(2) Removed reference to Test Methods D1186 and D1400.

(3) Added reference to Practice D7091.