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Designation: D 1133 – 97 (Reapproved 2001)

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Designation: D 1133 – 02

Standard Test Method for Kauri-Butanol Value of Hydrocarbon Solvents¹

This standard is issued under the fixed designation D 1133; 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 Department of Defense.

¹ This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

Current edition approved-Nov: Jan. 10, 1997. 2002. Published August 1998. March 2002. Originally published as D 1133 – 50 T. Last previous edition D 1133 – 947(2001).

1. Scope^{*}

1.1 This test method covers the determination of the relative solvent power of hydrocarbon solvents used in paint and lacquer formulations. This test method is suitable for use with solvents having an initial boiling point over 40° C and a dry point under 300° C when determined in accordance with the procedures in Note 1.

NOTE 1—Test Method D 86 is used to determine the initial boiling point and dry point for mineral spirits and similar petroleum solvents. Test Method D 1078 is used for pure compounds and narrow boiling range cuts.

1.2 This standard does not purport to address all of 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.

1.3 For specific hazard information and guidance, consult the supplier's Material Safety Data Sheet.

2. Referenced Documents

2.1 ASTM Standards:

D 86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure²

D 304 Specification for *n*-Butyl Alcohol (Butanol)³

D 611 Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents²

D 841 Specification for Nitration Grade Toluene³

D 1078 Test Method for Distillation Range of Volatile Organic Liquids³

3. Terminology

3.1 Definitions:

3.1.1 *kauri-butanol value—of a solvent*, the volume in millilitres at 25°C of the solvent, corrected to a defined standard, required to produce a defined degree of turbidity when added to 20 g of a standard solution of kauri resin in normal butyl alcohol.

3.1.1.1 *Discussion*—The kauri resin solution is standardized against toluene, which has an assigned value of 105, and a mixture of 75 % *n*-heptane and 25 % toluene on a volume basis, which has an assigned value of 40.

4. Significance and Use

4.1 The kauri-butanol value is used as a measure of solvent power of hydrocarbon solvents. High kauri-butanol values indicate relatively strong solvency.

5. Apparatus

5.1 *Water Bath*, a clear-glass vessel, capable of being maintained at $25 \pm \pm 5^{\circ}$ C. Alternatively, a room maintained at $25 \pm 5^{\circ}$ C may be used.

5.2 Volumetric Flask, 200-mL capacity.

5.3 Erlenmeyer Flask, 250-mL capacity.

5.4 Buret, 50-mL capacity.

5.5 Print Specimen—A sheet of white paper having on it black 10 or 12 point print, No. 31 Bruce old style type.

² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 06.04.

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Note 2-Text in this published standard is satisfactory to use as print specimen.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests unless otherwise specified. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

 $6.2 \ Kauri-Butanol \ Solution^5$ —Place in a 3-L flask 400 g of clean, pale, bold kauri resin of Grade XXXX, XXX, or XX ground to pea-size or smaller. Add, while agitating vigorously, 2000 g of *n*-butyl alcohol, (conforming to Specification D 304). Shake on a mechanical shaker until the resin goes into solution, warming to about 55°C, if necessary to aid solution. If a mechanical shaker is not available, fit the flask with a reflux condenser and heat on a steam bath until all of the kauri resin is dissolved. Permit the solution to stand 48 h and then clarify by filtering through a Büchner funnel with suction, using double filter paper and changing as frequently as necessary.

6.3 Standard Toluene conforming to Specification D 841 for use as a high-solvency standard.

6.4 *Heptane-Toluene Blend* consisting of 25 ± 0.1 % toluene and 75 ± 0.1 % *n*-heptane on a volume basis, for use as a low-solvency standard. The heptane shall conform to the requirements for knock test grade *n*-heptane prescribed in Table 1 of Test Methods D 611.

NOTE 3—The blend of 25 ± 0.1 % toluene and 75 ± 0.1 % heptane can be prepared in any way that will give the desired accuracy. The following technique is adequate: Bring the toluene and heptane and a calibrated 200-mL volumetric flask to the same temperature, preferably in a constant-temperature room or thermostat. Run 50 mL of toluene into the 200-mL volumetric flask, using a buret or pipet calibrated to deliver 50 mL of toluene at the chosen temperature (preferably 25° C). Fill the volumetric flask to slightly below the calibration line with *n*-heptane, insert the ground-glass stopper of the volumetric flask, and mix carefully by repeatedly inverting the flask. Allow to stand for a few minutes; then bring to the 200-mL calibration mark with heptane and again carefully mix.

7. Standardization

7.1 Weigh out 20 ± 0.10 g of kauri-butanol solution in a 250-mL Erlenmeyer flask. Check that the temperature of the KB solution in the flask-and is $25 \pm 5^{\circ}$ C. If not, place in the Erlenmeyer flask and its content in a water bath maintained at $25 \pm 5^{\circ}$ C and allow to equilibrate for at least 30 min. T Remove the flask from the water bath and titrate with the standard toluene into the flask, with constant-swirling, while maintaining the mixture in the flask at $25 \pm 1^{\circ}$ C. swirling. Gradually reduce the successive amounts of toluene added as the end point is approached. The end point is reached when the sharp outlines of 10-point print placed directly beneath the water bath and observed through the liquid are obscured or blurred, but not to the point where the print becomes illegible. Check the temperature in the flask immediately after the end point has been reached, and if over -26° C $_{30^{\circ}}$ C or under 240° C, repeat the titration.

7.2 The volume of toluene used, in millilitres, represents the actual titer for the particular kauri-butanol solution at hand. This value should lie reasonably close to 105 mL, but not over 110 nor under 100 mL. If these limits are exceeded, adjust the concentration of the kauri-butanol solution to bring the total volume of toluene within them. Designate the final value using toluene as A.

7.3 Weigh out 20 ± 0.10 g of the kauri-butanol solution (adjusted as described in 7.2) in a 250-mL Erlenmeyer flask and place in the water bath. Titrate with the heptane-toluene blend in the same manner as described in 7.1. Designate the volume, in millilitres, of the blend used in this titration as *B*.

Note 4—If the composition of the blend is known to differ from 25 ± 1.0 % toluene, but is within the range from 22 to 28 % toluene, the constant in the blend factor equation will differ from 40.0 by 0.60 units for each 1 % toluene. For example, at 28 % toluene, the constant is 41.8 instead of 40.0.

Note 5—Freshly prepared kauri-butanol solution may change in standardization from day to day. It is, therefore, desirable to permit the solution to age before initial standardization and, in any case, the standardization should be rechecked on successive days until the toluene factor and blend factor remain constant.

8. Procedure

8.1 Weigh 20 \pm 0.10 g of the adjusted kauri-butanol solution into a 250-mL Erlenmeyer flask.-Place_Check that the flask temperature of the KB solution in the flask is 25 \pm 5°C. If not, place the Erlenmeyer flask and its content in a water bath maintained at 25 \pm -+ 5°C and allow to equilibrate for at least 30 min. Remove the flask from the water bath. Fill the 50-mL buret with the solvent being tested and titrate the solvent into the Erlenmeyer flask with constant-swirling while maintaining the mixture in the flask at 25°C. swirling. Gradually reduce the successive amounts of solvent added as the end point is approached. The end point is reached when the sharp outlines of 10-point print (see 5.5) placed directly beneath the water bath and observed through the liquid

⁴ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

⁵ The sole source of supply of prepared kauri-butanol solutions known to the committee at this time is the Chemical Service Laboratories, 5543 Dyer St., Dallas, TX 75206. If you are aware of alternative suppliers, please provide this information to ASTM <u>International</u> Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

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are obscured or blurred, but not to the point where the print becomes illegible. Check the temperature in the flask immediately after the end point has been reached and if over $26^{\circ}C$ 30°C or under $240^{\circ}C$, repeat the titration. Designate the volume of solvent, in millilitres, to produce turbidity as *C*.

9. Calculation

9.1 Calculate the kauri-butanol value, V, as follows:

$$V = [65(C - B)/(A - B)] + 40$$

where:

A = toluene required to titrate 20 g of kauri-butanol solution (7.2), mL,

B = heptane-toluene blend required to titrate 20 g of kauri-butanol solution (7.3), mL, and

C = solvent under test required to titrate 20 g of kauri-butanol solution (Section 8), mL.

9.2 If the buret is maintained at a temperature other than $25 \pm 1^{\circ}$ C, correct the volume of solvent used, S, in millilitres, to standard temperature as follows:

 $S = C(25 - T) \times 0.0009$

(2)

(1)

where:

C = solvent used in the titration, mL, and

T = temperature of the solvent in the buret, °C.

10. Report

10.1 Report the calculated kauri-butanol value to the nearest 0.5 KB unit.

11. Precision and Bias

101.1 The following criteria should be used for judging the acceptability of the results in the range from 30 to 90 at the 95 % confidence level.

101.1.1 Repeatability—Two results, each the mean of duplicates, obtained by the same operator on different days should be considered suspect if they differ by more than 0.01 K - 0.1, where K = mean kauri-butanol value.

101.1.2 Reproducibility—Two results, each the mean of duplicates, obtained by two laboratories should not be considered suspect unless they differ by more than 0.03 K + 1.0 where K = mean kauri-butanol value.

101.2 Bias—Test bias can result if the kauri-butanol solution is not carefully standardized and adjusted (see 7.2 and 7.3). The test method has no definitive bias statement because the value of the test result is defined only in terms of the test method.

112. Keywords

1+2.1 kauri-butanol value; hydrocarbon solvents; solvency power

SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue (D 1133 - 97(2001)) that may impact the use of this standard.

(1) All references to $25 \pm 1^{\circ}$ C have been changed to $25 \pm 5^{\circ}$ C.

(2) Section 9.2 and equation 2 have been deleted.

(3) Text of 7.1 and 8.1 were modified.

(4) A new Section 10 was added, and subsequent sections were renumbered.

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