Designation: D 233 – 02

Standard Test Methods of Sampling and Testing Turpentine[1]

1. Scope

1.1 These test methods cover procedures for sampling and testing turpentine, as defined by the Code of Federal Regulations and Terminology D 804. These test methods are also used for the sampling and testing of pinenes, the major components of most turpentines.

1.2 These test methods primarily measure the physical rather than the chemical properties of turpentines and pinenes. As turpentines and pinenes are currently used chiefly as chemical raw materials for the production of resins and synthetic organic chemicals, chemical composition is also very important. Consequently, testing the chemical composition of turpentines and pinenes by gas chromatography has displaced these test methods to a large extent. (See for example Test Methods D 6387.)

1.3 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure [2]

D 270 Methods of Sampling Petroleum and Petroleum Products[3]

D 804 Terminology Relating to Naval Stores, Including Tall Oil and Related Products[4]

D 1193 Specification for Reagent Water[5]

D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)[6]

D 6166 Test Method for Color of Naval Stores and Related Products (Instrumental Determination of Gardner Color)[4]

D 6387 Test Methods for Composition of Turpentine and Related Terpene Products by Capillary Gas Chromatography[4]

E 1 Specification for ASTM Thermometers[7]

2.2 *Other Document:*

Code of Federal Regulations, Title 7, Part 160, “Regulations and Standards for Naval Stores,” 1999[8]

3. Significance and Use

3.1 The test procedures described in this standard were developed when the chief use for turpentine was as a solvent. Currently however, the chief use for turpentine (and pinenes) is as raw materials for the production of resins and synthetic organic chemicals. Thus the chemical composition of turpentines and pinenes is extremely important and tests, in addition to the ones described in these test methods, are required in order to fully characterize turpentines and pinenes. The most widely used technique for determining the chemical composition of turpentines (and pinenes) is gas chromatography (see Test Methods D 6387).

4. Purity of Reagents

4.1 Reagent grade chemicals shall be used in all tests. 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.[9] 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.

4.2 Unless otherwise indicated, references to water shall be

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[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 D01.34 on Naval Stores.

Current edition approved May 10, 2002. Published July 2002 Originally published as D 233 – 26. Last previous edition D 233 – 97.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] Discontinued; see *1983 Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 06.03.

[5] *Annual Book of ASTM Standards*, Vol 11.01.

[6] *Annual Book of ASTM Standards*, Vol 06.04.

[7] *Annual Book of ASTM Standards*, Vol 14.03.

[8] Available from U.S. Government Printing Office, Superintendent of Documents, 732 North Capitol St., NW, Mail Stop: SDE, Washington, DC 20401

[9] *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.

understood to mean reagent water conforming to Specification D 1193.

SAMPLING

5. Sampling

5.1 The method of sampling specified in 5.2 or 5.3 shall be used, according to the special conditions that apply.

5.2 *From Loaded Tank Car or Other Large Vessel*—The composite sample taken shall be not less than ½ gal (1.9 L) and should consist of small samples of not more than 1 qt (0.9 L) each, taken from near the top and bottom by means of a metal or glass container with removable stopper or top.[10] This device, attached to a suitable pole, shall be lowered to the desired depth, the stopper or top removed, and the container allowed to fill. If a sample from close to the bottom of a tank shows a decided difference in color or appearance from samples taken at other depths, an extra bottom sample shall be taken and examined separately from the composite sample. In such case, the composite sample shall not include any portion of such bottom sampling.

5.3 *From Barrels and Drums*—At least 5 % of the packages in any shipment shall be represented in the sample. The purchaser may increase the percentage of packages to be sampled at his direction, and it is recommended that every package be sampled in the case of expensive terpene hydrocarbons that are bought in small quantity. A portion shall be withdrawn from about the center of each package sampled by means of a "thief" or other sampling device. The composite sample thus obtained shall be not less than 1 qt (0.9 L) and shall consist of equal portions of not less than ½ pt (0.24 L) from each package sampled.

DETECTION AND REMOVAL OF SEPARATED WATER

6. Procedure

6.1 Draw a portion by means of a glass or metal container with a removable stopper or top,[10] or with a "thief," from the lowest part of the container, or by opening the bottom valve of the perfectly level tank car. If water is found in this sample, draw it all out, record the quantity, and deduct it from the total volume of liquid delivered.

APPEARANCE

7. Procedure

7.1 Examine a portion of the sample after agitation to determine whether its appearance conforms to specifications.

ODOR

8. Procedure

8.1 Compare the odor of the sample with an agreed-upon water-free reference sample kept in the dark in a completely filled, well-stoppered bottle. In the absence of such a sample, compare with samples of known purity similarly preserved.

SPECIFIC GRAVITY

9. Procedure

9.1 Determine the specific gravity at 15.6/15.6°C by any convenient method having a precision of 0.0005. Determinations made at any other temperature using apparatus standardized at 15.6°C shall be corrected by adding to or subtracting from the observed reading 0.00082 for each degree Celsius that the temperature of the liquid is above or below 15.6°C. If the determination is made with apparatus calibrated for a temperature other than 15.6°C, the observed reading shall first be calculated to density at the temperature of observation, then converted to density at 15.6°C by applying the above factor, and finally converted to specific gravity by dividing the calculated density by 0.999 (the density of water at 15.6°C).

REFRACTIVE INDEX

10. Procedure

10.1 Determine the refractive index with an accurate instrument, at 20°C if possible. If determined at any other temperature, correct the reading obtained to 20°C by adding or subtracting 0.00045 for each degree Celsius that the temperature at which the determination was made is, respectively, above or below 20°C.

DISTILLATION

11. Apparatus

11.1 Use the type of distillation apparatus described in Test Method D 86, with the following exceptions:

11.1.1 For testing turpentine or pinene use an ASTM Turpentine Distillation Thermometer, 3-in. (76-mm) partial immersion, having a range from 147 to 182°C, and conforming to the requirements for Thermometer 27C as prescribed in Specification E 1, or an ASTM Solvents Distillation Thermometer, 3.94-in. (100-mm) partial immersion, having a range from 95 to 255°C, and conforming to the requirements for Thermometer 42C as prescribed in Specification E 1.

11.1.2 Ice is not necessary in the condenser bath. It is permissible to use a glass Liebig condenser 22 in. (560 mm) in length with 16 in. (410 mm) in contact with the cooling water and filled with an adapter to extend 1 in. (51 mm) into the receiving graduate.

12. Procedure

12.1 Using the receiving graduate, transfer exactly 100 mL of the sample directly into the flask, allowing none to run into the side tube. If the sample contains dissolved or suspended water it is advisable to add a few small pieces of pumice or broken glass to promote smooth distillation. Insert the thermometer, so that the top of the mercury bulb (or the top of the contraction chamber if the Solvents Distillation Thermometer is used) is level with the bottom of the side tube. Connect the side tube to the condenser, with the bottom of the flask resting securely in the opening of the ceramic or other heat resistant board. Apply heat cautiously, and regulate it so that the first drop of condensate falls from the condenser in not less than 5 nor more than 10 min. Record as the *initial boiling point* the

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[10] Detailed description of equipment suitable for such sampling is given in Methods D 270.

thermometer reading when the first drop falls from the end of the condenser, correcting for barometric pressure as described in Section 13. When the distillation begins, regulate the heat so that the distillate is collected at a rate of not less than 4 nor more than 5 mL/min (approximately 2 drops/s). Discontinue the distillation when the temperature reaches that specified for the minimum percentage requirement (usually 170°C for turpentine), after correcting for barometric pressure. Allow the condenser to drain and read the percentage distilled.

12.2 The percentages distilled below successive selected temperatures and the temperature at which each successive 10 mL distills may also be determined, if desired, making the necessary correction of the temperature for barometric pressure.

13. Barometric Correction

13.1 The distilling temperature of turpentine (and pinene) is affected by 0.052°C for each millimetre (1.32°C for each inch) variation of the atmospheric pressure. Therefore, the distillation temperatures observed or specified shall be corrected to permit distillation to be conducted as though the barometer reading, corrected to 0°C, were exactly 760 mm (30 in.).

13.2 When about to begin the distillation, observe and record the barometric pressure and the temperature of the barometer. (No temperature correction is necessary for aneroid-type barometers.) From Table 1 determine the proper temperature correction corresponding to these atmospheric conditions, interpolating to the nearest 0.1°C. If the barometric pressure, corrected to 0°C, is below 760 mm, the temperature correction must be added to the initial boiling point and subtracted from the minimum percentage requirement temperature; if above 760 mm, the correction must be subtracted from the initial boiling point and added to the minimum percentage requirement temperature.

NOTE 1—*Example*—Suppose the observed barometric pressure is 748 mm at 32°C, and the initial boiling point is observed to be 155.2°C. From Table 1 the temperature correction is seen to be 0.8°C. Therefore, the corrected boiling point is 155.2 + 0.8 = 156°C. Furthermore, the temperature observation point at the minimum percentage requirement (170°C at 760 mm) must be altered to the same extent. Since the turpentine is distilling 0.8°C below what it would at normal pressure, distillation must be discontinued at 169.2°C to determine the percentage distilling below 170°C at 760 mm pressure.

TABLE 1 Temperature Corrections for Barometric Pressure[A]

Observed Barometric Pressure, mm	Aneroid Barometer	Mercurial Barometer Temperature of Barometer			
		20°C	25°C	30°C	35°C
780	− 1.04	− 0.91	− 0.88	− 0.84	− 0.81
770	− 0.52	− 0.39	− 0.36	− 0.32	− 0.29
760	0.00	+ 0.13	+ 0.16	+ 0.19	+ 0.22
750	+ 0.52	+ 0.65	+ 0.68	+ 0.71	+ 0.74
740	+ 1.04	+ 1.17	+ 1.20	+ 1.23	+ 1.26
730	+ 1.56	+ 1.68	+ 1.71	+ 1.75	+ 1.78
720	+ 2.08	+ 2.20	+ 2.23	+ 2.26	+ 2.29
710	+ 2.62	+ 2.72	+ 2.75	+ 2.78	+ 2.81
700	+ 3.16	+ 3.24	+ 3.27	+ 3.30	+ 3.33

[A] These corrections are calculated as follows: The observed barometric pressure is first corrected to what it would be at 0°C, by means of the table in Circular F, Instrument Division, U. S. Weather Bureau. The corrected barometric pressure is then subtracted from 760 mm (or *vice versa*) and the difference multiplied by 0.052°C to give the temperature correction shown in this table. The correction factor, C, of 0.052°C was calculated using the Sydney-Young equation:

$$C = 0.00012(760 - P)(273 + T)$$

where:

P = observed pressure, corrected to 0°C, in millimetres, and

T = median boiling temperature for turpentine, 160°C.

14. Color

14.1 Compare the color of the sample in any suitable or designated apparatus with the accepted or specific color standard. Accepted color standards are the Gardner color scale found in Test Method D 6166 and the platinum-cobalt scale found in Test Method D 1209.

15. Precision and Bias

15.1 The procedures described in these test methods were developed many years ago and were once widely used for the sampling and testing of turpentines and pinenes. Currently they are not widely used and it is not considered practical to redetermine the precision and bias of the individual methods at this time.

16. Keywords

16.1 pinene; sampling; turpentine