



Designation: D509 – 20

# Standard Test Methods of Sampling and Grading Rosin<sup>1</sup>

This standard is issued under the fixed designation D509; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 These test methods cover procedures for sampling and determining the grade of rosin delivered in commercial bags, barrels, drums or in molten form. Although developed for rosin, these methods can also be used for the grading of modified rosins and rosin based resins.

NOTE 1—All rosin sold in interstate commerce must be described by reference to the U. S. Standards for rosin, and is therefore subject to grading prior to such sale. The grading procedure described in these test methods is used for checking grades or regrading after the rosin has moved from the primary markets to distributing or consuming points.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

**D465** Test Methods for Acid Number of Pine Chemical Products Including Tall Oil and Other Related Products

**D1544** Test Method for Color of Transparent Liquids (Gardner Color Scale)

**D5974** Test Methods for Fatty and Rosin Acids in Tall Oil

<sup>1</sup> 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 Pine Chemicals and Hydrocarbon Resins.

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<sup>2</sup> 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.

**D6090** Test Method for Softening Point Resins (Mettler Cup and Ball Method)

**D6166** Test Method for Color of Pine Chemicals and Related Products (Instrumental Determination of Gardner Color)

**E28** Test Methods for Softening Point of Resins Derived from Pine Chemicals and Hydrocarbons, by Ring-and-Ball Apparatus

## 3. Significance and Use

3.1 Rosin is an important product of the centuries old Naval Stores industry, now known as the Pine Chemical industry, and is produced and consumed in many countries throughout the world. Consequently, reliable methods of sampling and grading rosin are necessary. The test methods based on the use of the USDA Official Standards described herein, were developed many years ago for the sampling and grading of rosin and are similar to those included in the Naval Stores Act.<sup>3</sup>

3.2 Although these test methods based on the use of the USDA Official Standards are still applicable, many additional test methods are now used to grade rosin. In particular, the color of rosin is now more commonly reported using the Gardner color scale rather than the USDA Official Standards. In addition, Test Methods **E28**, **D465**, and **D5974** are widely used to assess the quality of rosin in addition to the color grading methods described in this standard. The Naval Stores Act of 1923 established the methods for grading rosin and authorized the "Official Naval Stores Standard of the United States". Based upon a request from the chairman of the ASTM D01.34 Pine Chemicals and Hydrocarbon Resins subcommittee requesting that the Gardner color scale be used for grading the color of rosin, a letter from the Food and Drug Administration was received and is archived in Pine Chemicals Association<sup>4</sup> files. It documents a no-objection response to the request to use the Gardner color scale as a standard for grading rosin and rosin derivatives. The no-objection response was conditioned on having a reliable correlation between the USRG Scale (the "Official Naval Stores Standard of the United

<sup>3</sup> Naval Stores Act of 1923 (42 Stat 1435.7 USC-91-99) as amended in 1951 and regulations promulgated thereunder by the United States Department of Agriculture.

<sup>4</sup> Pine Chemicals Association International, P.O. Box 17136 Fernandina Beach, FL 32035; +1-404-994-6267

States”) and the Gardner Color Scale. A correlation study of color measurements on the Gardner Color Scale and the USRG Scale was carried out by scientists at Tintometer, Inc, Amesbury, England. The results of the correlation were submitted to the Food and Drug Administration as supporting evidence for the use of the Gardner Color Scale in measuring or grading the color of rosin and rosin derivatives.

#### 4. Sampling

4.1 *Number of Packages to Be Sampled*—A preliminary sampling of 20 % of the entire lot or shipment shall be made. If the grade of 85 % or more of the number of such preliminary samples agrees with the grade indicated on the package or invoice, with none of the samples disagreeing by more than one grade, the original grading shall be considered confirmed and the shipment accepted as a good delivery. If the grades of more than 15 % of the preliminary samples are in disagreement, or if any appreciable number are under grade by more than one grade, additional packages in the lot to make up a total equal to not less than 50 % of the entire shipment (preferably the entire shipment if accessible and not too great) shall be sampled. The findings on such larger quantity shall be accepted as the basis for settlement between the purchaser and the seller.

4.2 *Method of Sampling*—Take samples that are approximately cubical in shape and exactly 22.225 mm ( $\frac{7}{8}$  in.) thick in the direction in which they are to be viewed, by any of the following methods:

(a) by cutting or cleaving the same from a lump of rosin removed from the solid mass in the barrel or drum, the top side of which lump shall come from not less than 101.6 mm (4 in.) below the surface of the rosin.

(b) by means of a tin mold of suitable design which has been placed inside the barrel or drum through an opening in the side, the top of which opening is not less than 203.2 mm (8 in.) from the top of the container, in order to provide a sample that shall have come from a position not less than 4 in. below the surface of the rosin. The mold thus placed must be entirely within the barrel or drum and completely encased in the rosin.

(c) by suspending in the barrel or drum of molten rosin a clean tin plate mold 22.225 mm<sup>2</sup> ( $\frac{7}{8}$  in.<sup>2</sup>) (inside) and 38.1 mm (1.5 in.) or more in length, in such a manner that it will be in a horizontal position at least 4 in. below the surface of the rosin after it has thoroughly cooled. Such samples shall not be spiked from the barrel until it is completely cooled.

(d) by withdrawing from a full package of 67.5 kg (150 lb) or less, a quantity of the molten rosin, and allowing the same to cool and solidify in any suitable mold. Samples representing a single charge (or intermittent distillation) of oleoresin shall be taken from not less than 2 packages, one of which shall be selected after  $\frac{1}{4}$  of the rosin has been placed in the containers, and the other shall be the last package filled.

(e) by withdrawing a quantity of molten rosin from a full drum, filled after a preliminary cooling period, and taken 1 h after the drum was filled, and pouring some withdrawn portion into a suitable mold.

(f) by pouring a portion of molten rosin sampled during production into a color mold.

#### 5. Apparatus

5.1 *Official Rosin Standards*—The official standards for use in grading rosin, plus three standards lighter than “X” consists of assemblies of colored glass plates, cemented together, as issued on loan by the U.S. Department of Agriculture.<sup>5</sup> The grades and standards are designated as follows: XC, XB, XA, X, WW, WG, N, M, K, I, H, G, F, E, and D. A special grade, FF, is used for dark wood rosins. Grades XA, XB, and XC are not available from the U.S. Department of Agriculture but may be purchased commercially.<sup>6</sup> The standards issued by the Department of Agriculture (except FF), consist of combinations of plates cut, ground, and polished to specified thicknesses from selected melts of Corning and Jena colored glass. The colorimetric specifications of the standards, based on the 1931 CIE Coordinate System, for a standard observer using standard Illuminant C, are given in **Table 1**. The colorimetric specifications for the U.S. Rosin Standards, Master Cubes XA, XB, and XC, are given in **Table 2**.

5.2 Secondary standards or “type samples” are sometimes used for approximate grading, in the absence of permanent official standards of glass.<sup>6</sup> Such samples may consist of cubes of specially selected rosin or other colored transparent medium; solutions are also sometimes used (**Note 2**).

5.3 *Rosin Color Molds, 22.225 mm ( $\frac{7}{8}$  in.), standard size or Rosin Sample Trays, 22.225 by 22.225 by 22.225 mm ( $\frac{7}{8}$  by  $\frac{7}{8}$  by 1 in.), white cover without labels.*

**NOTE 2**—It is not possible to dissolve a specified quantity of rosin in a solvent and use such solution as a standard for grading the rosin because there is no constant relationship between the color of the original solid

<sup>5</sup> The U.S. Rosin Standards are issued only by the Agricultural Marketing Service, Tobacco Division, U.S. Department of Agriculture, P.O. Box 96456, Washington, DC 20090-6456. To obtain a set of standards, a Form N.S.A. 2 “Request for Loan of Official United States Rosin Standards” and a security deposit of \$100.00 should be sent to the above address.

If an applicant is unable to borrow a set of the U. S. Rosin Standards, because of the limited number of sets in existence, secondary sets are available. These standards are excellent duplicates of the U. S. Rosin Standards but could not be classified as identical and do not have official recognition of the U.S. Department of Agriculture. (Note: Recent attempts to obtain either the official sets or the secondary sets at the USDA address listed have elicited no response and it is possible that these standards are no longer available.)

<sup>6</sup> Glass discs of the secondary standards and the XA, XB, and XC standards may be purchased directly from Tintometer, Ltd., Salisbury, England.

**TABLE 1 Colorimetric Specifications for U.S. Rosin Standards (Master Set No. 200)<sup>A</sup>**

Grade	<i>x</i>	<i>y</i>	<i>T</i>	$\lambda$	<i>p</i>
X	0.4339	0.4663	0.609	575.0	0.755
WW	0.4579	0.4732	0.531	576.8	0.851
WG	0.4785	0.4741	0.466	578.5	0.905
N	0.5001	0.4704	0.396	580.5	0.944
M	0.5212	0.4619	0.322	582.8	0.969
K	0.5430	0.4483	0.245	585.5	0.985
I	0.5649	0.4310	0.178	588.7	0.993
H	0.5879	0.4102	0.114	592.4	0.997
G	0.6116	0.3874	0.0723	596.8	0.999
F	0.6364	0.3632	0.0398	602.1	1.000
E	0.6640	0.3358	0.0131	609.4	1.000
D	0.6943	0.3057	0.0021	621.4	1.000

<sup>A</sup> *x* and *y* are CIE trilinear coordinates; *T* is the luminous transmission factor;  $\lambda$  is the dominant wavelength in nanometers; *p* is the colorimetric purity.

**TABLE 2 Colorimetric Specifications for U. S. Rosin Standards (Master Cubes XA, XB, and XC)<sup>A</sup>**

Grade	x	y	T
XA	0.4048	0.4443	0.708
XB	0.3724	0.4117	0.788
XC	0.3406	0.3696	0.848

<sup>A</sup>x and y are CIE trilinear coordinates; T is the luminous transmission factor.

rosin and the color of the rosin after being put in solution. Consequently, the color of such solution is not a criterion of the color of the rosin itself.

## 6. Color Grading with the USDA Official Standards

6.1 The sample or “type” shall be compared with the appropriate standard, preferably against an open direct north sky light, or in a direction that excludes direct rays from the sun from passing through the rosin to the eye. The grade assigned to the rosin shall be considered that of the highest or lightest colored standard which the sample equals or excels (is lighter than) in color (Note 3). Should the color or appearance of the rosin be such that there is a doubt whether the rosin is as light or bright as the standard, the sample and the standard shall be viewed through a suitable colorimeter. Then in case of uncertainty the rosin shall be given the benefit of the doubt. For example, if a rosin sample is definitely darker than the WW standard but lighter than the WG standard, it is WG grade. If however, the grader is undecided whether the total color of the rosin is “as good as” or equals, or is darker than that of the WW standard, the grade is WW.

NOTE 3—The “color” of a sample of rosin is made up of three attributes: hue, saturation, and brightness. “Hue” alludes to the characteristic described by the words “yellow” or “red.” “Saturation” describes the purity or strength of the yellowness or redness of the color. “Brightness” depends on the relative amount of light transmitted by the rosin. The cleanliness of the rosin affects its transparency and therefore its brightness. The terms “lighter than” and its inverse, “darker than” are convenient to describe the difference between a sample and a standard. The rosin may be darker than the standard in one or more of the following ways: (1) a redder hue, (2) a more saturated hue, and (3) a lower brightness.

6.2 A sample showing two distinct colors, usually evidenced by darker streaks through the rosin, indicates a mixture of rosins in the package, in which case the darkest part of the sample or darkest rosin to be found in the package shall determine the grade.

6.3 A check sample taken from close to the bottom of a barrel or drum shall not be considered representative, because of a natural variation in color sometimes found between the top and bottom rosin as well as a reduced brightness of the bottom rosin due to a settling of finely divided suspended matter usually present in normal gum rosin. Should such bottom sample show not more than one grade lower than the top sample, the latter shall determine the grade of the rosin in the barrel. If, however, the bottom-head sample is more than one grade lower than the top sample, double filling or “mixed-packing” is indicated, and the bottom-head sample shall determine the grade of the rosin in the package.

6.4 Rosin that is only slightly cloudy or opaque (caused by occluded moisture or separation of crystals in the solid mass)

shall be graded in the usual way if the condition is not such as to prevent an accurate evaluation of the color in comparison with the standards. If, however, the opaqueness is of such a degree that the grade cannot be definitely determined in comparison with the standards, the rosin shall be designated “OPAQUE” and graded “OP,” in which case its acceptance and value shall be a matter for settlement between the purchaser and the seller without reference to grade. Such opaque rosin may be further identified or described as pale, medium, or dark opaque.

## 7. Tolerance

7.1 The tolerance or allowance of 15 % variation (Note 4) described in Section 5 is necessary because careful competent graders rarely get exact duplicate results on all individual samples when regrading a large number of rosin samples. Moreover, a regrade sample, coming from a different position in the package, and after storage and weathering for an indefinite period, may be slightly darker than the original sample on which the grade was based. An allowance for such slight variation must therefore be made.

NOTE 4—This tolerance is that recognized by the Federal Government in the administration of the U.S. Naval Stores Act.

## 8. Color Grading by Gardner Color Scale

8.1 Determine the color on the Gardner scale in accordance with Test Methods D1544 or D6166.

## 9. Softening Point

9.1 Determine the softening point in accordance with Test Methods E28.

9.2 Test Method D6090 may be used to determine the softening point provided the user determines the starting temperature and heating rate that give results equivalent to the ring and ball softening point in Test Methods E28.

## 10. Acid Number

10.1 Determine the acid number in accordance with Test Methods D465.

## 11. Gas Chromatographic Analysis

11.1 Determine the amounts of the major rosin acid isomers present in the rosin in accordance with Test Methods D5974.

## 12. Precision and Bias

12.1 Where the test method listed in this standard references another ASTM method, that method should be studied to determine the precision and bias. It is not practical to specify the precision and bias of the other procedures in this standard method for sampling and grading rosin, as the procedures are no longer widely used by industry and insufficient data could be developed to give meaningful statements.

## 13. Keywords

13.1 colorimetric standards; Gardner color; rosin; USRG color