

Standard Test Methods of Sampling and Grading Rosin¹

This standard is issued under the fixed designation D 509; 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.

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.

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 inch-pound 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 465 Test Methods for Acid Number of Naval Stores Products Including Tall Oil and Other Related Products²

D 5974 Test Method for Fatty and Rosin Acids in Tall Oil Fractionation Products by Capillary Gas Chromatography²

D 6090 Standard Test Method for Softening Point of Resins (Mettler Cup and Ball Method)²

E 28 Test Method for Softening Point by Ring-and-Ball Apparatus²

3. Significance and Use

3.1 Rosin is an important product of the centuries old Naval Stores 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 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.³

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.34 on Naval Stores.

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² *Annual Book of ASTM Standards*, Vol 06.03.

³ 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.

3.2 Although these test methods are still applicable, many modifications of these methods and many additional test methods are now used to grade rosin. For example, Test Methods E 28, D 465, and D 5974 are widely used to assess the quality of rosin in addition to the color grading described in those methods. Further, as rosin is often available in molten form, it is common practice to pour liquid rosin into molds in order to prepare specimens for color grading. The need for a test method describing such a technique is under discussion by Subcommittee D01.34. Thus, even though some parts of these test methods are somewhat outdated, these test methods are still important from both a practical and historical viewpoint.

4. Apparatus

4.1 *Official Rosin Standards*—The official standards for use in grading rosin (*G*, Fig. 1, 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.⁴ 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.⁵ 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.

4.2 Secondary standards or “type samples” are sometimes used for approximate grading, in the absence of permanent official standards of glass.⁵ Such samples may consist of cubes

⁴ 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.

⁵ The Standards XA, XB, and XC and secondary standards may be purchased directly from Tintometer, Ltd., Salisbury, England, or from their United States representative HF Scientific Inc. 3170 Metro Parkway Fort Myers, FL 33916.



A—Rosin spike.
B—Comparison box.
C—Sampling hatchet.
D—Sampling adz.
E—Smoothing device.

F—Rosin type samples.
G—Official rosin standards.
H—Spiked lump sample.
I—Sample for grading.

FIG. 1 Apparatus for Sampling and Grading Rosin

TABLE 1 Colorimetric Specifications for U.S. Rosin Standards (Master Set No. 200)^A

Grade	x	y	T	λ	p
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	0.1114	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

^A x and y are CIE trilinear coordinates; T is the luminous transmission factor; λ 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)^A

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

^Ax and y are CIE trilinear coordinates; T is the luminous transmission factor.

of specially selected rosin or other colored transparent medium; solutions are also sometimes used (Note 2). Secondary standards or “types” are usually not permanent in color and must be protected from excessive exposure to sunlight or heat. Wrapping and storage in a cool dark place when not in use is recommended. To ensure correctness of grading therewith,

they should be tested periodically by comparison with permanent glass standards.

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 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.

4.3 *Rosin Color Molds*, 7/8 in. (22.225 mm), standard size or *Rosin Sample Trays*, 7/8 by 7/8 by 1 in. (22.225 by 22.225 by 22.225 mm), white cover without labels.

5. Sampling

5.1 *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.

5.2 *Method of Sampling*—Take samples that are approximately cubical in shape and exactly 7/8-in. (22.225-mm) 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 4 in. (101.6 mm) 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 8 in. (203.2 mm) 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 $\frac{7}{8}$ in² (22.225 mm²) (inside) and 1.5 in. (38.1 mm) 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 150 lb (67.5 kg) 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.

6. Grading

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. Softening Point

8.1 Determine the softening point in accordance with Test Method E 28.

8.2 Test Method D 6090 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 Method E 28.

9. Acid Number

9.1 Determine the acid number in accordance with Test Method D 465.

10. Gas Chromatographic Analysis

10.1 Determine the amounts of the major rosin acid isomers present in the rosin in accordance with Test Method D 5974.

11. Precision and Bias

11.1 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.

12. Keywords

12.1 colorimetric standards; rosin; USRG color

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