Designation: D4209 - 07 (Reapproved 2013)

Standard Practice for Determining Volatile and Nonvolatile Content of Cellulosics, Emulsions, Resin Solutions, Shellac, and Varnishes¹

This standard is issued under the fixed designation D4209; 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 This practice is intended to serve as a guide to the selection of the proper ASTM test method for determining the volatile and nonvolatile content of cellulosics, emulsions, resin solutions, shellac, and varnishes.

Note 1—Standards for determining the composition of the volatile fraction are not covered by this practice.

1.2 The standards referenced in the practice are as follows:

Classification	Section	ASTM Standard
Cellulosics	5.1	D871
		D914
		D1347
Emulsions	5.2	D2369
Resin Solutions	5.3	D1259
		D1490
Shellac	5.4	D29
		D1650
Varnishes	5.5	D115
		D1644

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

2. Referenced Documents

- 2.1 ASTM Standards:²
- D16 Terminology for Paint, Related Coatings, Materials, and Applications
- D29 Test Methods for Sampling and Testing Lac Resins (Withdrawn 2005)³
- D115 Test Methods for Testing Solvent Containing Varnishes Used for Electrical Insulation
- D360 Specification for Shellac Varnishes
- D871 Test Methods of Testing Cellulose Acetate

D914 Test Methods for Ethylcellulose

D1259 Test Methods for Nonvolatile Content of Resin Solutions

D1347 Test Methods for Methylcellulose (Withdrawn 2003)³

D1490 Test Method for Nonvolatile Content of Urea-Formaldehyde Resin Solutions

D1644 Test Methods for Nonvolatile Content of Varnishes

D1650 Test Methods for Sampling and Testing Shellac Varnish (Withdrawn 1997)³

D2369 Test Method for Volatile Content of Coatings

D4758 Test Method for Nonvolatile Content of Latexes (Withdrawn 2007)³

3. Terminology

- 3.1 Definitions:
- 3.1.1 The definitions contained in Terminology D16 are applicable to this practice.
- 3.1.2 *cellulose ester*, *n*—derivatives of cellulose in which one or more of the hydroxyl hydrogens have been replaced by acyl groups.
- 3.1.3 *cellulose ether*, *n*—derivatives of cellulose in which one or more of the hydroxyl hydrogens have been replaced by alkyl groups.
- 3.1.4 *cellulose nitrate (nitrocellulose)*, *n*—derivatives of cellulose in which one or more of the hydroxyl hydrogens have been replaced by nitrate groups.
- 3.1.5 *emulsion vehicle*, *n*—an emulsion of binder in water. The binder may be oil, oleoresinous varnish, resin, or other emulsifiable liquid.
- 3.1.6 *latex*, *n*—a stable aqueous dispersion of synthetic resin, produced by emulsion polymerization, as the principal constituent of the binder.
- 3.1.7 *shellac varnish*, *n*—a solution or "cut" of a specified type and grade of dry lac resin in a suitable alcohol.
- 3.1.8 *varnish*, *n*—a liquid composition that is converted by oxidation or thermal cross-linking to a transparent or translucent solid film after application as a thin layer.

4. Significance and Use

4.1 The nonvolatile content of raw materials may be used to determine the total nonvolatile content (solids) of paint and

¹ This practice is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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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.

 $^{^{3}\,\}mbox{The last approved version of this historical standard is referenced on www.astm.org.$



related coatings. Such information may be useful to coatings producers and users for the determination of the total solids available for film formation and for the estimation of the volatile organic content.

5. Procedure

- 5.1 Cellulosics:
- 5.1.1 Test Methods D1644, Method A, should be used for determining the amount of alcohol present in nitrocellulose shipments. This method may also be used for other cellulose derivatives if mutually agreed upon by producer and user.
- 5.1.2 Moisture in three different cellulose derivatives may be determined using test methods given in the following standards:

Cellulose Derivative	ASTM Standard	Section
Cellulose acetate	D871	3–4
Ethylcellulose	D914	4–6
Methylcellulose	D1347	3–4

- 5.2 Emulsions and Latices:
- 5.2.1 Test Method D2369, Procedure B may be used to determine the nonvolatile and volatile content of most emulsions. Important considerations are the presence of volatile plasticizers and surfactants and the presence of oxidizable components in oleoresinous vehicles and drying oil modified alkyds.
- 5.2.2 Test Method D4758 should be used to determine the nonvolatile and volatile content of synthetic latex samples.
 - 5.3 Resin Solutions:
- 5.3.1 Test Methods D1259, Method A is recommended for solutions of non-heat reactive resins that remain stable and release solvents under the test conditions. Examples include alkyds and rosin esters.
- 5.3.2 Test Methods D1259, Method B is recommended for solutions of heat reactive resins that undergo condensation or other reactions or both under the influence of heat. Examples include the formaldehyde reaction products of urea, melamine, and phenols. Method B is also recommended for resin solutions that release solvent slowly, for example, epoxy resin and vinyl resin solutions when these contain high boiling solvents.
- 5.3.3 Method A and Method B of Test Methods D1259 differ primarily in the longer drying time specified for Method B (2 h versus 30 min). In both methods, a weighed specimen of resin solution is spread under pressure between two weighed sheets of aluminum foil, which are then separated and dried at

105°C. The method is unique in that it provides for drying of very thin films of resin, minimizing chances for volatiles to be trapped.

5.4 Shellac:

- 5.4.1 Sections 14 and 15 of Test Methods D29 describe two methods for the determination of volatile matter (moisture) in lac resins. Method A (Section 14) is recommended for orange shellac, button lac, garnet lac, and dry bleached lac. Method B (Section 15) is recommended for bleached lac in the form of hanks, bars, and crushed fresh ground (see Specification D360).
- 5.4.2 Sections 14 to 16 of Test Methods D1650 are recommended for the determination of the nonvolatile and volatile content of shellac varnishes.

5.5 Varnishes:

- 5.5.1 Test Methods D1644 describes two methods for the determination of the nonvolatile and volatile content of varnishes. Method A requires heating a 1.2 g specimen at 105°C for 3 h in an oven, while Method B requires heating a smaller specimen at 149°C for 10 min on a hotplate. Method A may give high results due either to incomplete elimination of volatile matter or to absorption of oxygen by oxidizing-type varnishes.
- 5.5.2 Sections 18 to 22 of Test Methods D115 is applicable to the following classifications of varnishes used for electrical insulation: alcohol-soluble varnishes, oxidizing air-drying varnishes, thermosetting varnishes, oxidizing baking varnishes, air-drying asphaltic varnishes, silicone varnishes, and thermosetting laminating varnishes. Determine nonvolatile matter in electrical insulating varnishes intended for electrical equipment operating at 180°C and above in accordance with Test Methods D115 except that the temperature used shall be 135 \pm 3°C (275 \pm 5.5°F) or at a temperature agreed upon between the producer and user.

6. Precision

6.1 Some of the referenced ASTM standards have precision limits. Reference to the individual standards for precision statements is recommended.

7. Keywords

7.1 nonvolatile content of cellulosics; emulsions; volatile content of cellulosics

SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue (D4209 - 82 (2002)) that may impact the use of this standard. (Approved July 1, 2007.)

(1) Updated the Terminology section.

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