Designation: D 3133 – 96

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

Standard Test Method for Quantitative Determination of Cellulose Nitrate in Alkyd Modified Lacquers by Infrared Spectrophotometry[1]

This standard is issued under the fixed designation D 3133; 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 test method covers the quantitative determination of the content of cellulose nitrate (also known as nitrocellulose) in lacquers containing alkyd resins.

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.* For a specific hazard statement, see Note 1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1644 Test Methods for Nonvolatile Content of Varnishes[2]

D 2372 Practice for Separation of Vehicle from Solvent-Reducible Paints[2]

E 168 Practices for General Techniques of Infrared Quantitative Analysis[3]

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers[3]

3. Summary of Test Method

3.1 The method of standard additions is employed. Increments of cellulose nitrate, in solution, are added to aliquots of the sample. Absorbance measurements are made of the band at 848 cm^{-1} (11.8 µm) for each addition. The original content is then calculated from absorbance versus concentration.

4. Significance and Use

4.1 Coating compositions based on a mixture of synthetic resins and cellulose nitrate dissolved in organic solvents are quantitatively analyzed for the cellulosic derivative without isolating it. The test method is applicable to lacquers for which the grade of nitrocellulose is known and available. Other cellulosics, alkyd resins, many vinyl resins, and solvents do not interfere. Components, such as acrylic resins and some vinyl polymers, that absorb infrared near 848 cm^{-1}(11.8 µm) interfere with the determination. High boiling ester solvents, in particular methyl cellosolve acetate, may also interfere with the determination if not removed in the evaporation procedure (see 8.3).

5. Apparatus

5.1 *Infrared Spectrophotometer*, automatic recording, double-beam. Most infrared spectrophotometers operate from 4000 to 650 cm^{-1} (2.5 to 15 µm), but in this test method only the range between 1000 and 750 cm^{-1} (10 to 14 µm) is used. See Practices E 168.

5.2 *Absorption Cells*, sealed, with sodium chloride (NaCl) windows, 0.1-mm path length, one pair approximately matched.

5.3 *Film Vacuum Evaporator*, rotary thin or equivalent apparatus, to obtain redissolvable lacquer solids without decomposition of the cellulose nitrate.

5.4 *Oven*, vacuum drying, thermostatically controlled to operate at 65 ± 2 C.

6. Reagents

6.1 *Purity of Reagents*— 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.[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 *Cellulose Nitrate*, of the same grade as in the sample.

[1] This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved Nov. 10, 1996. Published January 1997. Originally published as D 3133 – 72. Last previous edition D 3133 – 72 (1989)$^{\epsilon1}$.

[2] *Annual Book of ASTM Standards*, Vol 06.01.

[3] *Annual Book of ASTM Standards*, Vol 03.06.

[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.