Designation: D 3133 – 01

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

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 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 and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see 7.1.1.

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 11.8 µm (848 cm^{-1}) 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 2.5 to 15 µm (4000 to 650 cm^{-1}) , but in this test method only the range between 10 to 14 µm (1000 and 750 cm^{-1}) 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 D01 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 May 10, 2001. Published July 2001. Originally published as D 3133 – 72. Last previous edition D 3133 – 96.

[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.

6.3 *N, N-Dimethylacetamide* (*DMA*), spectrograde quality.

6.4 *Nitrogen Gas*, dry.

7. Standard Solution

7.1 Thoroughly dry a quantity of cellulose nitrate at 65°C, in the vacuum oven (7.1.1). Transfer 4000 g, weighed to 0.1 mg, to a 200-mL volumetric flask. Add DMA, shake to dissolve the cellulose nitrate, and dilute to volume with DMA. One millilitre of solution is equivalent to 20 mg of cellulose nitrate.

7.1.1 **Warning;** Every precaution must be exercised in the handling and drying of cellulose nitrate because of its explosive nature. No more than 15 g should be dried at any one time.

8. Procedure

8.1 If the material is pigmented, remove the pigment quantitatively in accordance with Practice D 2372, and proceed with the determination on the vehicle.

8.2 Determine the nonvolatile content of the clear lacquer, on the vehicle, in accordance with Test Methods D 1644.

8.3 Transfer, by any convenient means, 10.000 g of unpigmented material to a 50-mL volumetric flask. Attach the flask to the rotary evaporator and evaporate off the bulk of the solvents. Detach the flask, dissolve the lacquer solids in DMA, and dilute to volume with DMA. If the presence of methyl cellosolve acetate is suspected in the solvents, care must be taken to evaporate off the solvents as completely as possible.

8.4 Pipet 10.0 mL aliquots of this solution into each of three 25-mL volumetric flasks. To each, in turn, add 0, 5.0, and 10.0 mL of the standard solution. Dilute to volume with DMA.

8.5 *Infrared Analysis*:

8.5.1 Schedule the operating parameters of the infrared spectrometer to attain highly accurate absorbance values (refer to the directions of the manufacturer of the instrument). To accomplish this, set for lengthier scanning time and high signal-to-noise ratio, in accordance with Practice E 275.

8.5.2 Place one cell, containing DMA, in the reference beam. Place the other cell, filled in turn, with each of the solutions (Note 1), in the sample beam. Record the spectrum between 10 and 14 µm (1000 and 750 cm^{-1}) three times for each solution. Rinse the cell with DMA, and purge with dry nitrogen gas after each solution has been examined.

NOTE 1—To facilitate the transfer of the solutions to the cell, a small glass syringe fitted with a 75-mm (3-in.) hypodermic needle may be used.

8.5.3 Measure the difference in absorbance, ΔA, between the maximum absorbance at 11.8 µm (848 cm^{-1}) and the minimum at 12.3 µm (813 cm^{-1}) . Determine the mean ΔA for the three runs on each solution.

9. Calculation

9.1 Plot the absorbance difference, ΔA, versus the cellulose nitrate additions and extrapolate the curve to zero absorbance. The intersection of the curve with the abscissa (concentration axis) gives the original cellulose nitrate content in the diluted sample. See Fig. 1.

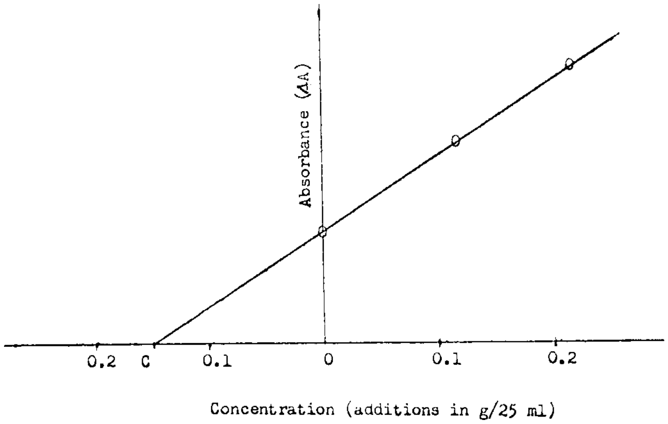

FIG. 1 Plot of ΔA verses Cellulose Nitrate Additions

9.2 *Alternative Method*—Solve for C in the three simultaneous equations derived from the expression for the Beer-Bourguer law, $A = abc$

$$\Delta A_0 / (C + 0.0) = \Delta A_{0.1} / (C + 0.1) = \Delta A_{0.2} / (C + 0.2) \qquad (1)$$

where:

C = original concentration of cellulose nitrate, in grams per 25 mL and the subscripts for ΔA correspond to the amounts of cellulose nitrate added in grams per 25 mL. Calculate the mean of C.

9.3 Calculate the weight percent of cellulose nitrate E in the original lacquer as follows:

$$E = (C \times D \times 100) / S \qquad (2)$$

where:

D = dilution factor,
S = weight of specimen, and
C = grams of cellulose nitrate in 25 mL.

9.4 Calculate weight percent cellulose nitrate E in nonvolatile portion of original lacquer as follows:

$$E = (C \times 5 \times 100 \times 100) / (S \times N) \qquad (3)$$

where:

N = nonvolatile content (see 8.2).

10. Precision and Bias

10.1 *Precision*—On the basis of a study in which four laboratories analyzed two different samples for percent of cellulose nitrate relative to the total samples, T, and relative to the nonvolatile matter, NVM, the within and between laboratory standard deviations were found to be:

Percent Relative to	Standard Deviations	
	Within	Between
T	0.14	0.12
NVM	0.39	0.64

Based on these standard deviations the following criteria should be used in judging the acceptability of results at the 95 % confidence level.

10.1.1 *Repeatability*—Two results obtained by one operator on one instrument should be considered suspect if they differ by more than the following:

Relative to	Percent
T	0.48
NVM	1.34

10.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than the following:

Relative to	Percent
T	0.71
NVM	2.51

10.2 *Bias*—No information can be presented on bias of the procedure of this test method for measuring weight percent cellulose nitrate since no material having an acceptable reference value is available.

11. Keywords

11.1 alkyl modified lacquers; cellulose nitrate; infrared spectrophotometry; quantitative determination

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).