



## Standard Test Methods for Soluble Cellulose Nitrate<sup>1</sup>

This standard is issued under the fixed designation D 301; 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 the material known as soluble cellulose nitrate (also known as soluble nitrocellulose), which is shipped wet in conformance with regulations of the Interstate Commerce Commission.

1.2 The test methods appear in the following sections:

	Sections
Ash	5-7
Drying Samples	4
Nitrogen	8-10
Stability	11-13
Toluene Dilution	22-24
Viscosity	14-18

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 specific hazard statements, see Notes 2-5.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 302 Specification for Ethyl Acetate (85 to 88 Percent Grade)<sup>2</sup>
- D 303 Specification for *n*-Butyl Acetate (90 to 92 % Grade)<sup>2</sup>
- D 362 Specification for Industrial Grade Toluene<sup>2</sup>
- D 1343 Test Method for Viscosity of Cellulose Derivatives by Ball-Drop Method<sup>3</sup>
- D 4795 Test Method for Nitrogen Content of Soluble Nitrocellulose—Alternative Method<sup>3</sup>
- E 1 Specification for ASTM Thermometers<sup>4</sup>

### 3. Sampling

3.1 Samples shall be taken from not less than 10 % (at least two barrels) of each lot or batch in the shipment. In sampling

<sup>1</sup> 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 D01.36 on Cellulose and Cellulose Derivatives.

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<sup>2</sup> Discontinued, see 1986 *Annual Book of ASTM Standards*, Vol 06.03.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 06.03.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.03.

the barrels, two samples of approximately 1 pt (0.5 dm<sup>3</sup>) each shall be taken from two well-separated points at least 1 ft (0.3 m) beneath the surface of the material in the barrel. These samples shall then be composited to represent each lot or batch in the shipment.

3.2 The samples shall meet the following requirements:

3.2.1 *Appearance*—The cellulose nitrate shall not be discolored and shall be free of lumps and foreign matter, such as charred particles.

3.2.2 *Ash*—Ash content shall not exceed 0.30 %, calculated on the basis of dry-weight soluble cellulose nitrate.

3.2.3 *Nitrogen*—The percent nitrogen, calculated on the basis of dry-weight soluble cellulose nitrate, shall be within the limits agreed upon by the purchaser and the manufacturer for the particular type of soluble cellulose nitrate.

3.2.4 *Stability*—The stability as determined by the 134.5 C test shall be not less than 25 min.

3.2.5 *Viscosity*—The viscosity shall be within the limits agreed upon by the purchaser and the manufacturer for the particular type of soluble cellulose nitrate.

3.2.6 *Solubility and Appearance of the Solution*—The solubility and appearance of the sample shall be equal to the reference standard for the particular type of soluble cellulose nitrate.

3.2.7 *Film Test*—The film test of the sample shall be equal to that of the reference standard for the particular type of soluble cellulose nitrate.

3.2.8 *Toluene Dilution Test*—The toluene dilution value of the sample shall be equivalent to that of the reference standard for the particular type of soluble cellulose nitrate.

### DRYING SAMPLES

### 4. Procedure

4.1 Soluble cellulose nitrate is a flammable material, the degree of flammability varying with the extent and nature of the wetting medium. Cellulose nitrate is always wet with water or alcohol in commercial handling, shipping, and storage, in which condition it presents no unusual hazard. Dry cellulose nitrate, if ignited by fire, spark, or static electricity, burns very rapidly. Samples of dry cellulose nitrate *must not* be stored at any time. Dry only that portion required for immediate test. Wet the excess material and the samples left after testing with water and dispose of by burning on a safe burning ground.

4.2 Dry small quantities required for ash and nitrogen tests by spreading in a thin layer on a tray at room temperature for 12 to 16 h, followed by oven-drying in crucibles or weighing bottles 1 h at 100 to 105°C. The oven used for drying cellulose nitrate should have the latch removed. Wear a face mask (see 12.2) when the oven is opened after samples have been heated.

4.3 Dry larger quantities of *water-wet* material required for viscosity and toluene dilution tests, or a small quantity for stability tests, by blowing warm compressed air (at a temperature of 60 to 65°C, and a pressure of 40 to 60 psi (275 to 415 kPa)) through the sample placed in a cylindrical holder with a screen over one end for ½ to 1 h. Provide the compressed air line with a safety plug (Note 1) of Wood's metal, which melts at 70 to 75°C, so the air will be diverted from the sample if a temperature of 70°C is exceeded.

NOTE 1—Information on the availability of a suitable fusible plug assembly may be obtained from ASTM Headquarters.

4.4 If the material is *alcohol-wet*, it is necessary to modify the drying procedure. After placing the required amount of cellulose nitrate in the cylindrical holder, pour in sufficient distilled or iron-free water to fill it. Allow the bulk of the liquid to drain off. Then dry by blowing warm air through the holder as described in 4.3.

## ASH

### 5. Significance and Use

5.1 Ash accounts for the nonsoluble, nonfilm forming portion of the polymer. It may affect solution clarity and film properties.

### 6. Apparatus

- 6.1 *Porcelain Crucibles*, Coors No. 3 or equivalent.
- 6.2 *Muffle Furnace*, maintained at  $550 \pm 25^\circ\text{C}$ .

### 7. Reagents

- 7.1 *Ethyl Alcohol*.
- 7.2 *Acetone*.
- 7.3 *Castor Oil*.

### 8. Procedure

8.1 Dry the cellulose nitrate as described in 4.2 and place a specimen of approximately 4.0 g in a tared and ignited crucible. Moisten the sample in the crucible with ethyl alcohol, then gelatinize by adding a sufficient amount of 5 % solution of castor oil into the acetone. Place the crucible in a draft-free hood and ignite the contents with a Bunsen flame. Allow the material to burn without further addition of heat until a charred residue remains. Place the crucible in a muffle furnace at  $550 \pm 25^\circ\text{C}$  for 90 min. Remove carefully the crucible from the muffle furnace to avoid loss of ash, cool in a desiccator, and weigh accurately.

### 9. Calculation

9.1 Calculate the percent ash as follows:

$$\text{Ash, \%} = (\text{wt of ash/wt of dry sample}) \times 100$$

### 10. Precision and Bias

10.1 *Precision*—Statistical analysis of intralaboratory (repeatability) test results on a sample containing approximately 0.015 % ash indicates a precision of  $\pm 0.015$  % absolute at the 95 % confidence level.

10.2 *Bias*—No statement of bias can be made as no suitable reference material is available as a standard.

## NITROGEN

### 11. Significance and Use

11.1 The nature and strength of solvent systems required for cellulose nitrate are dependent upon the nitrogen content. Mismatches of solvent with nitrogen level can result in poor solution quality and colloid and gel formation.

11.2 An alternative preferred method can be found in Test Method D 4795.

### 12. Apparatus

12.1 *Nitrometer*—Use the duPont Nitrometer, which is illustrated in Figs. 1-4.

12.2 *Face Mask*—A face mask, so constructed that a heavy piece of cellulose acetate sheeting protects the face.

NOTE 2—**Precaution:** The cellulose acetate mask must be worn during the generation and measurement of the gas as a precaution in case of an explosion.

### 13. Procedure

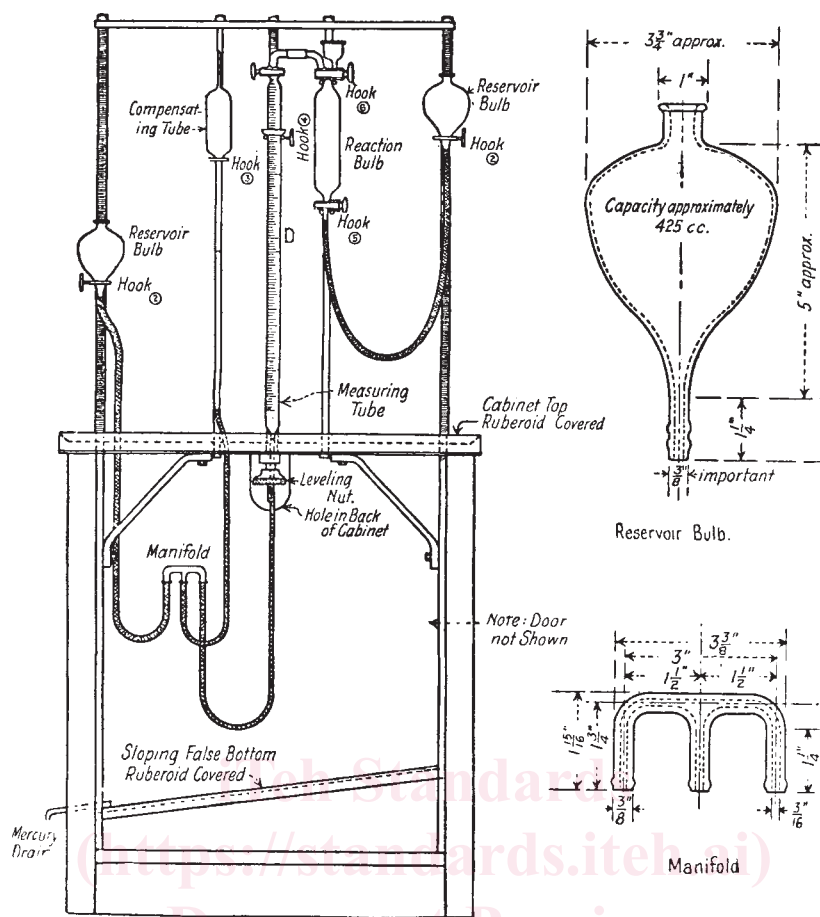
13.1 Calibrate the measuring tube accurately in the usual manner, using mercury as the calibrating liquid.

13.2 Standardize the apparatus as follows:

13.2.1 Fill the compensating, measuring, and reaction tubes and their rubber connections with mercury. Run 20 to 30 mL of  $\text{H}_2\text{SO}_4$  (ACS grade,  $94.5 \pm 0.5$  %) into the reaction bulb through the cup at the top and admit about 210 mL of air. Close the stopcocks, shake the bulb well, and allow to stand overnight. This desiccates the air which is then run into the compensating tube until the mercury is about on a level with the 12.50 % mark on the measuring tube, the two tubes being held at the same height. Then seal the compensating tube using a small blowpipe flame.

13.2.2 As a preferred alternative, nitrogen may be used in place of air.

13.2.3 Place in weighing bottles  $0.95 \pm 0.05$ -g portions of ACS grade  $\text{KNO}_3$  that has been recrystallized twice from distilled water and ground to pass a No. 100 (150- $\mu\text{m}$ ) sieve. Dry the specimens 2 to 3 h at 135 to 150°C. Stopper the bottles, cool in a desiccator, and weigh accurately. Transfer the  $\text{KNO}_3$  to the cup of the reaction bulb and weigh the weighing bottle to obtain the weight of sample used. Add 1.0 mL of water and stir the mixture in the cup with a small glass stirring rod to liberate the entrained bubbles of air; work the undissolved crystals into the lower part of the cup, keeping them below the surface of the solution. It is not necessary that the  $\text{KNO}_3$  dissolve before drawing it into the reaction bulb. *Make sure the lower stopcock is open*; then admit the mixture to the bulb by a series of quick openings of the upper stopcock, in the meantime keeping the crystals below the surface of the liquid.



NOTE 1—1 in. = 25.4 mm.  
**FIG. 1 General Assembly of Apparatus for Nitrogen Determination**

In this way, all but a small amount of the  $\text{KNO}_3$  may be run into the bulb. Rinse the cup with a second 1.0-mL portion of water; then repeat with a third 1.0-mL portion (3 mL in all). This should be sufficient to dissolve all remaining particles of  $\text{KNO}_3$  in the cup. Transfer 25 mL of the  $\text{H}_2\text{SO}_4$  ( $94.5 \pm 0.5\%$ ), divided in several portions, to the cup, and subsequently to the bulb by lowering the reservoir slightly and opening and closing the upper stopcock, care being taken that no air enters even the bore hole in the stopcock. There must always be a slight suction when introducing the specimen, the wash water, and the acid, but never enough to cause air to be sucked into the reaction bulb. The quantities of water and  $\text{H}_2\text{SO}_4$  used should be constant. Then with the bottom stopcock still open, lower the reservoir bulb to give reduced pressure in the reaction bulb and gently shake the reaction bulb to start the decomposition.

13.3 After the evolution of  $\text{NO}$  has become slow (**Precaution**, see Note 3), lower the reservoir bulb until all but 25 mL of the mercury in the reaction bulb is withdrawn, close the bottom stopcock, and shake the reaction bulb vigorously for 5 min.

NOTE 3—**Precaution:** It is extremely important that the bottom stopcock be left open until the major part of the decomposition has occurred; otherwise, sudden evolution of gas will burst the bulb, scattering acid and glass.

13.4 When the reaction is completed, allow the gas to cool for 20 min; then transfer the gas to a measuring tube. By means of the leveling device make careful adjustment of the mercury levels so that the mercury in the measuring tube is at the 13.85% mark (the theoretical percent nitrogen in  $\text{KNO}_3$ ) if an exactly 1.000-g specimen was used, or a proportional reading if less was used. Paste a strip of paper on the compensating tube at the level of the mercury, and the standardization is completed. It is advisable to make several check determinations, preferably on different days, to ensure accurate standardization. Determinations should check within  $\pm 0.01\%$ .

13.5 Dry the cellulose nitrate as described in 4.2 and place a specimen of 1.0 to 1.2 g in a weighing bottle. After drying at 100 to 105°C for 1 h, stopper, cool in a desiccator, and weigh accurately. Transfer the specimen to the cup of the decomposition bulb; then reweigh the empty bottle to get the weight of the specimen by difference. Add 5 to 10 mL of  $\text{H}_2\text{SO}_4$  ( $94.5 \pm 0.5\%$ ) to the cup and stir the mixture with a small stirring rod. Lower the mercury reservoir and then, with the lower stopcock open, draw the mixture in by opening the upper stopcock. Take care that no air is drawn in. Rinse the cup of the decomposing bulb several times with  $\text{H}_2\text{SO}_4$ , using a total of 25 mL for dissolving and rinsing. Complete the determination in accordance with the procedure described in 10.2 for standardization of the apparatus, and take a reading after adjusting the level of