



# Standard Test Method for Dilution Ratio of Active Solvents in Cellulose Nitrate Solutions<sup>1</sup>

This standard is issued under the fixed designation D 1720; 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.

*This standard has been approved for use by agencies of the Department of Defense.*

## 1. Scope

1.1 This test method covers the determination of the volume ratio of hydrocarbon diluent to active solvent required to cause persistent heterogeneity (precipitation) in a solution of cellulose nitrate.

1.2 *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 Section 6.

1.3 For hazard information and guidance, see the supplier's Material Safety Data Sheet.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 301 Test Methods for Soluble Cellulose Nitrate<sup>2</sup>

D 841 Specification for Nitration Grade Toluene<sup>3</sup>

D 4615 Specification for *n*-Butyl Acetate (All Grades)<sup>3</sup>

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *dilution ratio*—the maximum number of unit volumes of a diluent that can be added to a unit volume of solvent to cause the first persistent heterogeneity (precipitation) in the solution at a concentration of 8 g cellulose nitrate per 100 mL of combined solvent plus diluent and at a temperature of 25  $\pm$  3°C.

NOTE 1—The dilution ratio decreases as the cellulose nitrate concentration at the end point increases. It is, therefore, necessary to set an arbitrary concentration of cellulose nitrate as part of the dilution ratio term. For this purpose 8.0 g of cellulose nitrate per 100 mL of solvent plus diluent has been adopted.

## 4. Significance and Use

4.1 By use of standard or reference grade materials for any

<sup>1</sup> This test 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 D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

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

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 06.04.

two of the three components, namely, oxygenated solvent, diluent, or cellulose nitrate, the effect of different batches or different types of the third component can be determined.

4.2 This test method is applicable for the determination of the following:

4.2.1 The dilution ratio of toluene as the standard diluent to an oxygenated solvent under test, using as the solute standard cellulose nitrate as defined in 5.2.

4.2.2 The dilution ratio of a hydrocarbon diluent under test to *n*-butyl acetate as the standard solvent, using as a solute standard cellulose nitrate as defined in 5.2.

4.2.3 The dilution ratio of toluene, as the standard diluent, to *n*-butyl acetate as the standard solvent, using as the solute cellulose nitrate of varying solubility characteristics.

## 5. Materials

5.1 *n*-Butyl Acetate (90 to 92 %), conforming to Specification D 4615.

NOTE 2—This grade of *n*-butyl acetate contains 8 to 10 % *n*-butyl alcohol.

5.2 *Cellulose Nitrate*, conforming to the Sampling section (Appearance, Ash, and Stability requirements) of Methods D 301 and of such quality that, when used in determining the toluene dilution ratios of *n*-butyl acetate and methyl *n*-propyl ketone, it will give results between the following limits:

Toluene Dilution Ratio

<i>n</i> -butyl acetate	2.73 to 2.83
methyl <i>n</i> -propyl ketone	3.80 to 3.90

5.3 *Toluene (Toluol)*, conforming to Specification D 841.

## 6. Hazards

6.1 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. Do not store samples of dry cellulose nitrate at any time. Dry only that portion required for immediate test. Wear a face shield when the oven is opened after samples have been heated. Wet excess material and the samples left after testing with water and dispose of properly.

## 7. Drying Cellulose Nitrate

7.1 Dry not more than 20 g of cellulose nitrate at a time by spreading in a thin layer on a tray at room temperature for 12 to 16 h, or on top of a 100°C oven where the temperature is 35 to 40°C for about 8 h (**Warning**, see 6.1). Alternatively, use a steam or hot water-heated oven maintained at 45 to 50°C to dry specimens in about 8 h. For safety reasons, the oven should have the latch removed.

7.2 Another simple way to dry small quantities of cellulose nitrate is to use a drier assembled from common laboratory apparatus. The assembled drier is shown in Fig. 1. Hot air from a laboratory electric oven is drawn through wet cellulose nitrate contained in a brass tube hooked up through a thistle tube, or small funnel, and suction flask to a water aspirator or other vacuum source. The brass pipe should be about 40 mm in diameter and 200 mm long, these relative dimensions having been found to give efficient results. Such a tube will hold about 25 g, dry weight, of wet cellulose nitrate. The pipe is insulated to conserve heat. The suction flask end of the brass tube is fitted with a thistle tube, or a small funnel, over the mouth of which is tied a silk cloth screen. An indentation made in the funnel edge allows insertion of the thermometer. The funnel and thermometer are held in place by means of a rubber stopper. When using an oven temperature of 85°C and vacuum supplied by a water aspirator or other vacuum source, the alcohol-wet cellulose nitrate will be dried in about 4 h.

7.3 If larger quantities of cellulose nitrate are required, the drying equipment described in the Procedure section of Drying Samples of Methods D 301 may be used.

## 8. Preparation of Solution

8.1 When testing either a solvent or diluent, first estimate the probable dilution ratio for the unknown component in relation to the other to determine the amount of solvent required to dissolve the cellulose nitrate (Table 1). This volume of solvent should be such that there will be approximately 10 g of cellulose nitrate present per 100 mL of solvent plus diluent at the end point.

NOTE 3—Reference to published data on similar types of solvents or diluents will provide a good approximation of the amount of solvent required. If data are not available, several solutions with varying amounts of solvents may be required to arrive at a suitable volume to use.

8.2 On an analytical balance, weigh  $5 \pm 0.01$  g of the cellulose nitrate into a 125-mL cork-stoppered, preweighed Erlenmeyer flask, or other suitable container. From a buret add the volume of solvent indicated in Table 1. Swirl the flask until the cellulose nitrate is completely dissolved. When a high

TABLE 1 Volume of Solvent Required to Dissolve Cellulose Nitrate

Probable Dilution Ratio, volume of diluent:volume of solvent	mL of Solvent per 5 g of Cellulose Nitrate at 25°C
1	25.0
2	16.7
3	12.5
4	10.0
5	8.3

concentration of cellulose nitrate in solvent is required, dispersion may be more quickly accomplished by adding a measured portion of the diluent to the flask. This reduces the solids concentration and thus lowers the viscosity of the solution, making it easier to dissolve the cellulose nitrate.

## 9. Procedure

9.1 Add the diluent, maintained at  $25 \pm 3.0^\circ\text{C}$ , to the flask from a buret in small additions. Five-millilitre increments may be added at first, but these shall be decreased to about 0.5 mL as the end point is approached. After each addition, stopper the flask and swirl vigorously to disperse any gel or precipitate thrown down by local overconcentration of diluent (Note 4). When precipitation persists after at least 2 min of vigorous swirling, the initial end point has been reached, as indicated by the presence of gel particles in the solution or on the sides of the flask (Note 5). Determine the total volume of diluent added to the flask at this point.

NOTE 4—Take care to prevent loss of volatile components by evaporation. Avoid contact of the solution with the stopper.

NOTE 5—Presence of a uniform fine haze that is usually formed when aliphatic hydrocarbons are used as diluents must not be confused with the gel end point.

9.2 Determine a second end point using the same solution. This requires addition of solvent to redissolve the cellulose nitrate. The amount of solvent to add depends upon the amount of diluent used in the initial titration. The volume of solvent to be added is obtained directly from Fig. 2 and the volume of diluent used to reach the initial end point.

9.3 After addition of the required volume of solvent, swirl the flask to redisperse the cellulose nitrate. Then, continue the titration with diluent to the second end point, at which point there should be approximately 8 g of cellulose nitrate present per 100 mL of solvent plus diluent.

## 10. Calculation

10.1 Calculate the dilution ratio and cellulose nitrate concentration at both the initial and the second end points as follows:

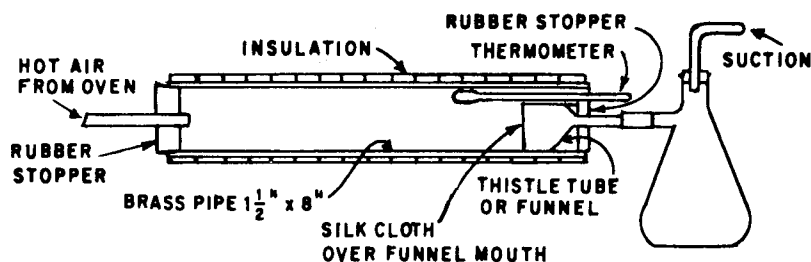
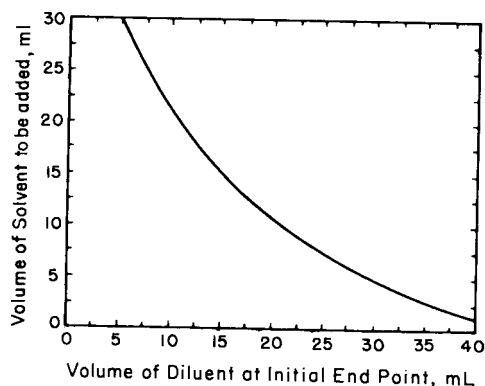


FIG. 1 Assembled Drier



**FIG. 2 Volume of Solvent to Be Added to Complete Titration versus Volume of Diluent Used to Reach Initial End Point**

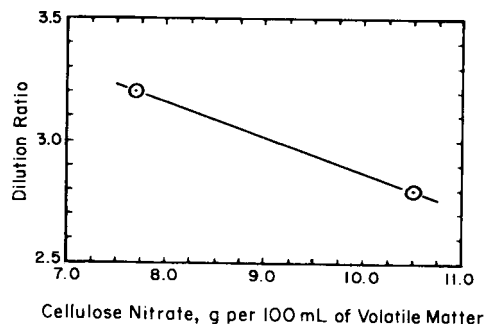
$$\text{Dilution ratio} = A/B \tag{1}$$

$$\text{Cellulose nitrate concentration per 100 mL of volatile matter} = C/(A + B)$$

where:

- A = diluent for the titration, mL,
- B = solvent used, mL, and
- C = cellulose nitrate used, g.

10.2 Construct a graph for dilution ratio versus cellulose nitrate concentration. Plot as two points on the graph (Fig. 3) the two sets of values calculated as described in 10.1. One of the points will be very close to 8 g/100 mL of volatile matter. The correct value at exactly 8 g/100 mL of volatile matter may be interpolated by drawing a straight line connecting the two experimentally determined points. While the curve showing



**FIG. 3 Grams of Cellulose Nitrate per 100 mL of Volatile Matter versus Dilution Ratio**

the relationship between the dilution ratio and cellulose nitrate concentration is not necessarily a straight line, the error made by interpolating or extrapolating from the straight line connecting the two points is negligible in the proximity of  $8 \pm 1$  g/100 mL of volatile matter.

NOTE 6—Example: Assuming a probable dilution ratio of approximately 3, the following results are obtained:

	Initial End Point	Solvent Added (from Fig. 2)	Second End Point
Cellulose nitrate, g	5.00	...	5.00
Solvent, mL	12.5	3	15.5
Diluent, mL	35.0	...	49.6
Solvent plus diluent, mL	47.5	...	65.1
Dilution ratio	$35/12.5 = 2.8$	...	$49.6/15.5 = 3.2$
Cellulose nitrate per 100 mL of volatile matter, g	10.5	...	7.7

From Fig. 3 the dilution ratio at 8.0 g of cellulose nitrate = 3.16

### 11. Report

11.1 When testing solvents, report the ratio of the volume of toluene to the volume of solvent at 8 g of cellulose nitrate per 100 mL of volatile matter.

11.2 When testing diluents, report the ratio of the volume of diluent to the volume of *n*-butyl acetate at 8 g of cellulose nitrate per 100 mL of volatile matter.

11.3 When testing cellulose nitrate, report the ratio of the volume of toluene to the volume of *n*-butyl acetate at 8 g of cellulose nitrate per 100 mL of volatile matter.

### 12. Precision and Bias

12.1 *Precision*—The following criteria should be used for judging the acceptability of results at the 95 % confidence level.

12.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 0.1.

12.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 0.2.

12.2 *Bias*—This test method has no bias because the value of the test result is defined only in terms of the test method.

### 13. Keywords

13.1 active solvents; cellulose nitrate solutions; dilution ratio; hydrocarbon diluent

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