Designation: D2697 − 03 (Reapproved 2021)

# Standard Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings[1]

This standard is issued under the fixed designation D2697; 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.

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

## 1. Scope

1.1 This test method is believed to be applicable to the determination of the volume of nonvolatile matter of a variety of coatings. An interlaboratory study to establish the precision of this test method included a water-reducible exterior latex paint and three automotive coatings that included a solvent-reducible primer surfacer, water reducible primer surfacer, water reducible enamel topcoat, and acrylic dispersion lacquer topcoat. Earlier collaborative studies included a gloss enamel, a flat wall paint, a gloss house enamel, an industrial baking enamel, an interior latex paint, and an exterior latex paint.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1475 Test Method for Density of Liquid Coatings, Inks, and Related Products

D2369 Test Method for Volatile Content of Coatings

D3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings

D3980 Practice for Interlaboratory Testing of Paint and Related Materials (Withdrawn 1998)[3]

## 3. Summary of Test Method

3.1 The weight and volume of a stainless steel disk is to be determined; after the disk is coated with the material being tested. The weight and volume of the disk plus dried coating is determined by weighing in air and then by weighing in a liquid of known density. The volume being equal to the quotient of the weight loss of the coated disk (due to the Archimedes buoyancy effect) divided by the density of the liquid displaced. The liquid may be water, organic liquid such as low-solvency mineral spirits or kerosine, or with special modifications not covered specifically in this method, mercury. The choice of liquid depends upon the nature of the coating tested.

NOTE 1—Distilled water is suitable for most paints. Exceptions are coatings that contain ingredients that are readily leached out of the dry film by the water and low-gloss coatings, the surface of which is poorly wet by water even with surfactant added. (Note 2) Low-solvency hydrocarbon solvent (KB below 36) is also practical for most paints and is preferred by some workers.[4] It is considered to be particularly good for paint films not readily wet by water. Analogously, organic solvents must not be used if the coating to be tested contains ingredients that will be dissolved readily by the solvent. Lacquers containing monomeric plasticizers would be examples where hydrocarbon solvents should definitely not be used. Coatings formulated much above the CPVC present a special problem, where mercury might be the desired "suspending" liquid (Note 3), and for solvent-reducible paints hydrocarbon solvent might be considered the poorest (unless it is the objective to obtain values closer to "theoretical" spaces between pigment particles not filled with binder, becoming partially filled with solvent during the test).

NOTE 2—Concentration of surfactant must be kept very low or literature values for the density of the water cannot be used.

NOTE 3—Details of the mercury displacement techniques can be found in the literature.[5]

3.2 From the measured weights and volumes of the disk before and after coating, the weight and volume of the dried coating film are calculated. Based on the density of the liquid

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

Current edition approved Feb. 1, 2021. Published February 2021. Originally approved in 1968. Last previous edition approved in 2014 as D2697 – 03 (2014). DOI: 10.1520/D2697-03R21.

[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] The last approved version of this historical standard is referenced on www.astm.org.

[4] Bissey, J. E., *Official Digest*, Federation of Paint and Varnish Production Clubs, Vol 35, 1963, p. 1072, and Ashton, H. E., *Materials Research and Standards*, Vol 1, 1961, p. 549.

[5] Cole, R. J., *Journal*, Oil Colour Chemists' Assn., Vol. 45, 1962, p. 776.

coating and the weight percent nonvolatile matter, the volume of the liquid coating deposited on the coated disk is calculated. The volume of the dried coating divided by the volume of liquid coating, multiplied by 100, provides the volume percent nonvolatile matter in the total liquid coating.

## 4. Significance and Use

4.1 This test method is intended to provide a measure of the volume of dry coating obtainable from a given volume of liquid coating. This value is useful for comparing the coverage (square feet of surface covered at a specified dry film thickness per unit volume) obtainable with different coating products.

4.2 For various reasons the value obtained may not be equal to that predicted from simple additivity of the weights and volumes of the raw materials in a formulation. One reason is that the volume occupied by a solution of resin in solvent may be the same, greater, or less than the total volume of the separate ingredients: such contraction or expansion in resin solutions is governed by a number of factors, one of which is the extent and direction of spread between solubility parameters of the resin and solvent.

4.3 The spatial configuration of the pigment particles and the degree to which the spaces between the pigment particles are filled with the binder also affect the volume of a dry coating formulation. Above the critical pigment volume concentration, the apparent volume of the dry film is significantly greater than theoretical due to the increase in unfilled voids between pigment particles. The use of volume nonvolatile matter values in such instances should be carefully considered as the increased volume is largely due to air trapped in these voids.

## 5. Apparatus

5.1 *Analytical Balance.*

5.2 *Steel Disk,* preferably stainless steel, 60 mm (2⅜ in.) in diameter and 22 gage (0.65 mm) in thickness with a small hole near the circumference. A fine wire, such as Chromel A, 28 gage (0.32 mm), is attached through the hole and made the appropriate length for subsequent suspension of the disk in a liquid. The wire should have a small loop on the upper end so the disk and wire can be hung by this loop on the balance.

NOTE 4—Instead of steel disks, some analysts use aluminum tubes. In the round-robin results, essentially no difference was found in the precision obtained by both methods. Source and dimensions of these tubes are described in the annex.

5.3 *Counterweight,* to be placed on the balance stirrup after hanger bow and pan are removed.

5.4 *Beaker*, 1-L—For easier manipulation during the weighing of disk in liquid it is advisable to cut the beaker to a height of 115 mm (4½ in.).

5.5 Support for holding the beaker under the balance stirrup without jamming the pan damper in the floor of the balance. A cork or neoprene ring is suitable when a single-pan balance is used.

5.6 *Weight per Gallon Cup,* acrometer, or other suitable means for determining the density of the coating material and the suspending liquids if not known.

## 6. Volume Determination of Uncoated Disks

6.1 Dry the disk in an oven at 110 ± 5°C for 10 min. Cool and weigh the disk in air.

6.2 Weigh the disk in the liquid to be used for suspension of the coated disk. If water is used as the suspending liquid, a few drops of wetting agent (Note 2) added to the liquid will help to ensure rapid and thorough wetting of the disk. Be careful that no air bubbles form on the disk or wire. Mark the level of liquid in the 1-L beaker necessary for complete immersion of the disk which should be at least 20 mm (¾ in.) above the disk. Maintain this level in subsequent weighings when the disk is coated.

6.3 Record the temperature of the liquid. Obtain the density of the liquid at the temperature used, from a table, such as is found for pure water in *Handbook of Chemistry and Physics*,[6] or determine it to 0.001 g/mL.

6.4 Calculate the volume of the disk, $G$, in millilitres as follows:

$$G = (w_1 - w_2)/D \tag{1}$$

where:

$w_1$ = weight of disk in air, g
$w_2$ = weight of disk in liquid, g, and
$D$ = density of liquid at temperature of test, g/mL.

## 7. Procedure

7.1 Take a representative sample of the liquid coating in accordance with Practice D3925. Mix thoroughly before taking specimens for the individual tests.

7.2 Determine the weight nonvolatile of the liquid coating by drying 1 h at 110° ± 5°C in accordance with Test Method D2369.

NOTE 5—If this method does not apply, then the method used should be agreed upon between producer and user.

7.3 Determine to 0.001 g/mL the density of the liquid coating in accordance with Test Method D1475.

7.4 Dip the disk in the liquid coating and allow the liquid to come up on the wire a distance from 5 to 15 mm (¼ to ½ in.). Allow about 10 min for draining, and blot the coating material off the bottom edge of the disk so that beads or drops do not dry on the bottom edge of the disk.

NOTE 6—In some cases the paint or varnish may be of such consistency that the amount of solid matter remaining on the disk after drying is too small for an accurate volume determination. The use of a flat pan with a sidewall about 10 mm in height in place of the disk enables the operator to obtain a more desirable volume of solid matter. However, extra care must be observed to prevent trapping of air at the point where the sidewall meets the bottom of the pan. In no case should bubbles be allowed to be present in cast films. This procedure has not been evaluated and no precision statement is available.

7.5 When beads or drips stop forming, hang the disk in the oven for 1 h at 110°C (Note 5). Remove and cool. Weigh the coated disk in air.

[6] CRC Press, Inc., West Palm Beach, FL, 1986.

7.6 Weigh the coated disk in the chosen medium in the same manner as for the uncoated disk, recording the temperature of the liquid at the time of the test.

## 8. Calculations

8.1 Calculate the volume of the coated disk, $H$, in millilitres, as follows:

$$H = (w_3 - w_4)/D \tag{2}$$

where:

$w_3$ = weight of coated disk in air, g,
$w_4$ = weight of coated disk in liquid, g, and
$D$ = density of liquid at temperature of test.

8.2 Calculate the volume of the dried coating, $F$, in millilitres, as follows:

$$F = H - G \tag{3}$$

8.3 Calculate the volume of wet coating, $V$, in millilitres, from which the dried coating was obtained, as follows:

$$V = (w_3 - w_1)/(w \times \rho) \tag{4}$$

where:

$w$ = nonvolatile matter in 1 g of wet coating, g, and
$\rho$ = density of liquid coating material.

8.4 Calculate the percent volume nonvolatile content in a liquid coating as follows:

$$(F/V) \times 100 \tag{5}$$

NOTE 7—The displacement liquid used should be reported with volume percent nonvolatile results. The method of drying the films should also be stated if different from that specified.

## 9. Precision

9.1 *Precision (In accordance with Practice D3980)*—In an interlaboratory study of this test method in which one operator in each of five laboratories analyzed in duplicate on two days four coatings (two solvent-reducible and two water-reducible) with nonvolatile contents ranging from 24 to 35 volume %, the pooled within-laboratory standard deviation was found to be 0.444 % with 17 degrees of freedom (DF) and the pooled between-laboratories standard deviation 1.195 % with 16 DF, after discarding one day's results from two laboratories on one sample, one day's results from one laboratory on another sample, and one duplicate result from one laboratory on a third sample. Based on these standard deviations the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—Two results, each the mean of duplicates, obtained by the same operator on different days should be considered suspect if they differ by more than 1.32 % absolute at volume nonvolatile contents of 24 to 35 %.

9.1.2 *Reproducibility*—Two results, each the mean of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 3.59 % absolute at the same levels.

9.2 *Bias*—Bias cannot be determined because there are no accepted standards for volume nonvolatile matter of clear or pigmented coatings.

## 10. Keywords

10.1 volume nonvolatile content



## ANNEX

**(Mandatory Information)**

### A1. ALUMINUM TUBES

A1.1 Aluminum tubes,[7] uncoated, plain with no cap or liner, #16 neck and orifice, 32 mm by 160 mm (1¼ by 6¼ in.).

A1.2 Cut two 75 mm (3-in.) lengths of tube from the aluminum tubing. Make a 20 mm (¾-in.) cut on the flattened end of the tube about 6 mm (¼ in.) from the end. Slip the tube over a short length of 25 mm (1 in.) inside diameter electrical conduit and return the tube to a round condition. Remove the tube from the pipe and press 1 in. wide strip at an end of the tube toward the center to serve as a hangar attachment.

[7] The sole source of supply of the tubes manufactured by Teledyne known to the committee at this time is Teledyne, 2290 W. Townsend St., Chester, PA 19016. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.