



Designation: D3367 – 21

Standard Test Method for Plasticizer Sorption of Poly(Vinyl Chloride) Resins Under Applied Centrifugal Force¹

This standard is issued under the fixed designation D3367; 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 determination of the plasticizer sorption of poly(vinyl chloride) (PVC) homopolymers using a controlled centrifugal force.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 This test method references notes and footnotes that provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of this test method.

NOTE 1—This standard is equivalent to ISO 4608.

1.4 *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.*

1.5 *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

D883 Terminology Relating to Plastics

2.2 *ISO Standards:*

ISO 4608 Homopolymers and Copolymers of Vinyl Chloride for General Use—Determination of Plasticizer Absorption at Room Temperature³

¹ This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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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 Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

3. Terminology

3.1 *Definitions*—For definitions that appear in this practice relating to plastics refer to Terminology **D883**.

4. Summary of Test Method

4.1 A sample of PVC is saturated with plasticizer in a screening tube with an amount of plasticizer equal to approximately twice the weight of the PVC. The PVC and plasticizer are subjected to centrifugation for a specified time. A small amount of cotton is weighed into the screening tube before weighing of the PVC to prevent PVC particles from escaping through the orifice of the tube during weighing and centrifugation. Plasticizer that is not absorbed by the PVC particles is removed by the centrifugation through the orifice of the screening tube.

5. Significance and Use

5.1 This test method provides a quantitative measure of the relative plasticizer absorption of general-purpose poly(vinyl chloride) resins under standard temperature conditions. Plasticizer sorption thus defined is one of the criteria useful for the description of the powder blend characteristics of poly(vinyl chloride) resins.

6. Apparatus and Materials

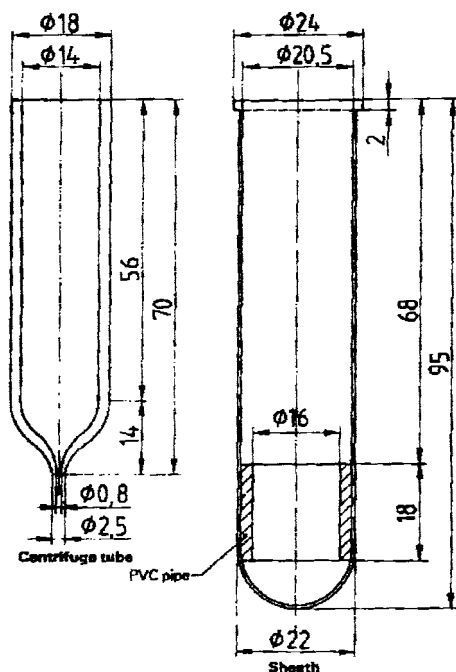
6.1 *Centrifuge*, whose rotor turns in a horizontal plane and has an acceleration under the test conditions of 24 500 to 29 500 $\text{m} \cdot \text{s}^{-2}$ measured at the level of the bottom of the tube, with, if necessary, a cooling system to prevent the temperature of the mixture from exceeding 30°C (86°F) at the end of the centrifuging for 60 min.

NOTE 2—It is permissible to use higher acceleration to reduce the centrifuging time, for example, 34 000 $\text{m} \cdot \text{s}^{-2}$ and 30 min, provided that it has been verified that the results obtained are equivalent.

6.2 *Centrifuge Tubes*, to fit the centrifuge used, of glass or plastic, with a conical bottom pierced by a hole of 0.8-mm diameter (see **Fig. 1**).

NOTE 3—It has been found using Fisherbrand Disposable Filter Column (Fisher HealthCare) #11-387-50, works well in this application. It is recommended removing the filter paper before placing the cotton in the column.

*A Summary of Changes section appears at the end of this standard



NOTE 1—Dimensions in millimetres.

FIG. 1 Example of Centrifuge Tube and Sheath

6.3 *Plastic Sheaths* (metal, polyamide, polyethylene, and so forth), with a piece of PVC pipe at the bottom to support the centrifuge tube (see Fig. 1).

6.4 *Cotton Wool*, pharmaceutical grade, having a DOP absorption measured under the test conditions (6.4.1) of approximately 10 %.

NOTE 4—Alternative materials to cotton wool may be used if it can be shown that they produce equivalent results; for example, glass wool, and PTFE-coated polyester felt.

6.4.1 *Measurement of DOP Absorbed by Cotton Wool*—In accordance with Section 8, carry out a test with a piece of cotton wool having a mass of 100 ± 2 mg, but without resin. Determine the mass of DOP absorbed by the cotton wool in grams (mass m_0).

6.5 *Di(2-ethyl hexyl)phthalate (DOP)* or alternative phthalate or non-phthalate plasticizers.

NOTE 5—Phthalate and non-phthalate plasticizers other than DOP can be used in this test. The plasticizer type will affect the plasticizer sorption % test value.

7. Conditioning

7.1 The PVC and plasticizer shall be at $23 \pm 1^\circ\text{C}$ ($73 \pm 2^\circ\text{F}$) and 50 ± 10 % relative humidity for at least 1 h before testing.

7.2 Perform the centrifugation with centrifuge and test samples at $23 \pm 1^\circ\text{C}$ ($73 \pm 2^\circ\text{F}$).

8. Procedure

8.1 Weigh 100 ± 2 mg of cotton wool and place it in the centrifuge tube, and pack it down slightly. Weigh the tube and

cotton wool to the nearest ± 0.1 mg, m_1 being the mass in grams of the prepared tube.

8.2 Weigh 100 ± 2 mg of the resin being tested and place it into the tube, m_2 being the mass in grams of the prepared tube plus resin, to the nearest ± 0.1 mg.

8.3 From a pipette, run into the tube 4 mL of DOP and allow it to stand for about 10 min.

8.4 Place the tube in its sheath and place the sheath into one of the compartments of the centrifuge rotor (the other compartments being occupied by tubes containing other resin samples, all the tubes being balanced).

8.5 Set the centrifuge to give an acceleration of 24 500 to 29 500 $\text{m} \cdot \text{s}^{-2}$ at the level of the bottom of the tube for 60 min. If necessary, the cooling device shall be switched on during centrifuging. Check that the temperature does not exceed 30°C .

8.6 Remove the tube from its sheath, carefully wipe it to remove any DOP on the outside, and weigh it to the nearest 0.1 mg, m_3 being the mass in grams of the tube containing the resin and the absorbed DOP.

9. Calculation

9.1 The room temperature plasticizer absorption, reported as parts of DOP absorbed per 100 parts resin (phr) is given by the following formula:

$$\frac{(m_3 - m_0) - m_2}{m_2 - m_1} \times 100$$

where:

m_0 = mass, of DOP absorbed by cotton wool, g,

m_1 = mass, of centrifuge tube with cotton wool, g,

m_2 = mass, of centrifuge tube with resin, g, and

m_3 = mass of centrifuge tube with resin and absorbed DOP, g.

10. Report

10.1 Report the plasticizer type and plasticizer sorption of the test sample, %.

11. Precision and Bias

11.1 The precision of the test method calculated by analysis of round-robin data from six laboratories is as follows:

11.1.1 *Repeatability*—Coefficient of variation (average of replicates) within a laboratory of 3.6 %.

11.1.2 *Reproducibility*—Coefficient of variation (average of replicates) between laboratories of 8.0 %.

11.2 *Bias*—No justifiable statement of bias can be made for this test method since the true value of the property cannot be established by an accepted referee method.

12. Keywords

12.1 DOP; plasticizer sorption; plasticizer type; poly(vinyl chloride) resin