



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

This standard is issued under the fixed designation D 3367; 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 Department of Defense.

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 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.* A specific safety precaution is given in 7.8.1.

1.4 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 and ISO 4608-1984 are identical in all technical details.

2. Referenced Documents

2.1 ASTM Standards:

D 883 Terminology Relating to Plastics²

D 1600 Terminology for Abbreviated Terms Relating to Plastics²

2.2 ISO Standards:

ISO 4608-1984 Homopolymers and Copolymers of Vinyl Chloride for General Use—Determination of Plasticizer Absorption at Room Temperature³

3. Terminology

3.1 Definitions—Definitions are in accordance with Terminology D 883 and Terminology D 1600, unless otherwise indicated.

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 (Section D20.15.08).

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² *Annual Book of ASTM Standards*, Vol 08.01.

³ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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 at the end of the centrifuging for 60 min.

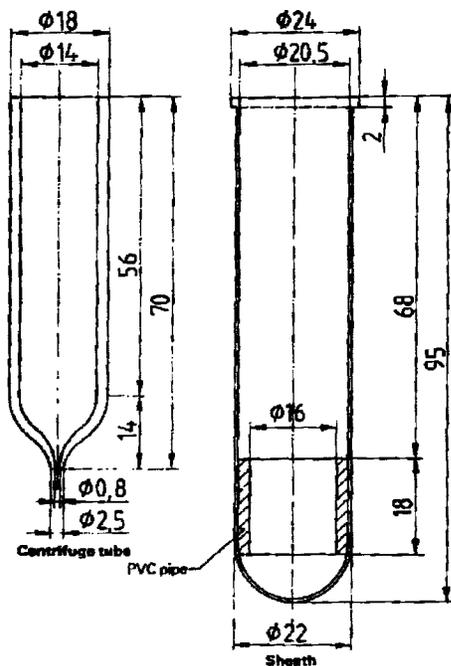
NOTE 2—It is permissible to use higher acceleration to reduce the centrifuging time, for example, 34 000 $\text{m} \cdot \text{s}^{-2}$ and 30 min, provided that it has been verified that the results obtained are equivalent.

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).

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 %.

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



NOTE—Dimensions in millimetres.

FIG. 1 Example of Centrifuge Tube and Sheath

NOTE 3—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)*.

7. Conditioning

7.1 The PVC and plasticizer shall be at $23 \pm 1^\circ\text{C}$ ($73.4 \pm 1.8^\circ\text{F}$) and $50 \pm 5\%$ relative humidity for at least 1 h before testing.

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

8. Procedure

8.1 Weigh a piece of cotton wool (100 ± 2 mg), 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 into the tube to the nearest 1 mg 2 g of the resin under test, 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 (p.h.r.) 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 sorption of the test sample, %.

11. Precision and Bias⁴

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; poly(vinyl chloride) resin

⁴ The round-robin precision data for this test method may be obtained from ASTM Headquarters. Request RR:D 20-1043.

SUMMARY OF CHANGES

Committee D-20 has identified the location of selected changes in this test method since the last issue that may impact the use of this test method.

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(1) Section 1.4 was added and clarified notes and footnotes.

(2) Section 6 was revised to describe apparatus and materials

rather than referencing sources.

(3) Section 8 was changed to ISO instruction detail.

(4) Section 9 was changed to ISO calculation detail.

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