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Standard Guide for Testing Poly(Vinyl Chloride) Resins[1]

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ϵ^1 NOTE—Keywords were added editorially in October 1996.

1. Scope

1.1 This guide covers test methods for poly(vinyl chloride) resins and vinyl chloride copolymers for use in coatings applications. The test methods listed in Table 1 were tested by interlaboratory participation in accordance with ASTM guidelines. Also included are methods useful to test the suitability of resins for use in fluid nonaqueous vinyl dispersions.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1243 Test Method for Dilute Solution Viscosity of Vinyl Chloride Polymers[2]

D 1705 Test Method for Particle Size Analysis of Powdered Polymers and Copolymers of Vinyl Chloride[2]

D 1755 Specification for Poly(Vinyl Chloride) Resins[2]

D 1823 Test Method for Apparent Viscosity of Plastisols and Organosols at High Shear Rates by Extrusion Viscometer[2]

D 1824 Test Method for Apparent Viscosity of Plastisols and Organosols at Low Shear Rates by Brookfield Viscometer[2]

D 1895 Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Materials[2]

D 1921 Test Method for Particle Size (Sieve Analysis) of Plastic Materials[2]

D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer[3]

D 2222 Test Method for Methanol Extract of Vinyl Chloride Resins[2]

D 3030 Test Method for Volatile Matter (Including Water) of Vinyl Chloride Resins[4]

D 3205 Test Method for Viscosity of Asphalt with Cone and Plate Viscometer[5]

D 3680 Test Method for Residual Vinyl Chloride Monomer Content of Poly(Vinyl Chloride) Resins, Compounds, and Copolymers by Solution Injection Technique[6]

D 3749 Test Method for Residual Vinyl Chloride Monomer in Poly(Vinyl Chloride) Homopolymer Resins by Gas Chromatographic Head Space Technique[4]

D 4287 Test Method for High-Shear Viscosity Using the ICI Cone/Plate Viscometer[3]

E 442 Test Method for Chlorine, Bromine, or Iodine in Organic Compounds by Oxygen Flask Combustion[7]

TABLE 1 Test Methods for Vinyl Chloride Resins

Method	Section	ASTM Designation
Viscosity, dilute solution	5	D 1243
Particle size (sieve analysis)	11	D 1705
Poly(vinyl chloride) resins, specification	6	D 1755
Viscosity, vinyl dispersions, high shear		6D 1823
Viscosity vinyl dispersions, low shear	6	D 1824
Apparent density	10	D 1895
Particle size (sieve analysis)	11	D 1921
Methanol extract	7	D 2222
Volatile content	8	D 3030
Residual VC1 monomer-solution technique	9	D 3680
Residual VC1 monomer-head space technique	9	D 3749
Viscosity, high shear, cone and plate	6	D 4287
Total chlorine content	4	E 442

3. Significance and Use

3.1 The test methods listed and discussed in this guide are used to determine the purity and physical properties of poly-(vinyl chloride) and vinyl chloride copolymers. This information is useful to polymer producers and coatings manufacturers.

4. Total Chlorine Content

4.1 The determination of the total chlorine in vinyl chloride homopolymers and copolymers is useful for the compositional

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

[3] *Annual Book of ASTM Standards,* Vol 06.01.

[4] *Annual Book of ASTM Standards,* Vol 08.02.

[5] *Annual Book of ASTM Standards,* Vol 04.03.

[6] *Annual Book of ASTM Standards,* Vol 06.03.

[7] *Annual Book of ASTM Standards,* Vol 03.05.

analysis of the polymer and also to determine the amount of vinyl resin present in a coating binder, provided no other chlorine-containing ingredients are present.

4.2 Test Method E 442 involves the determination of chlorine in organic compounds by the oxygen flash technique (Schoeninger technique).

5. Dilute Solution Viscosity

5.1 The dilute solution viscosity provides a measure of the relative molecular weight of the vinyl resin. In Test Method D 1243, 0.2 g of the sample in 100 mL of cyclohexanone is employed. The flow time of the solution in a standard Ubbelohde viscometer at 30 ± 0.5°C is used to calculate the inherent viscosity (logarithm viscosity number).

5.2 The procedure is applicable to all vinyl chloride homopolymers and copolymers used in coatings.

6. Viscosity Measurement (of Vinyl Dispersions)

6.1 Viscosity behavior is important to the use of fine powder vinyl resins (dispersion resins) in fluid nonaqueous dispersions for coatings applications. These dispersions are called plastisols when only plasticizer(s) is(are) employed with the resin, and organosols when volatile diluents or solvents are also present.

6.2 The flow behavior of the dispersions at low shear rates, which determines the relative ease of mixing and pumping (transfer) and is also related to low shear coating applications for example and dip coating, is measured in Test Method D 1824 or Test Method D 2196 with a Brookfield viscometer. The flow at high shear rates, related to high shear application such as in coil coating, is characterized in Test Method D 1823 using an extrusion rheometer. High shear flow can also be determined using a cone-and-plate viscometer, following the general procedure of Test Method D 3205 or with the equivalent Test Method D 4287.

6.3 The preparation and testing of the resin in a standard vinyl plastisol, which contains 60 parts of di(2-ethylhexyl phthalate) and 100 parts of the test vinyl dispersion resin, are described in Specification D 1755.

7. Methanol Extract

7.1 The amount of nonvolatile, methanol-soluble material present in the vinyl resin, such as surface active agents, plasticizers, and other alcohol-soluble additives, is obtained in Test Method D 2222 by weighing the material soluble in refluxing methanol.

7.2 This procedure provides a relative measure of the polymeric purity of the resin.

7.3 With some low molecular weight vinyl copolymer resins with moderate comonomer content, the methanol extraction may also remove the low molecular fraction of the polymer.

8. Volatile Content

8.1 The volatile material present in the vinyl resin is determined from the loss in weight observed on heating a resin specimen to constant weight in an air-circulating oven at 110°C (see Test Method D 3030).

9. Residual Vinyl Chloride Monomer

9.1 Residual vinyl chloride monomer present in either vinyl chloride homopolymers or copolymers is measured in Test Method D 3680 using analysis of a solution of the test resin in tetrahydrofuran injected into a gas chromatograph.

9.2 Gas chromatography using a head space sampling technique is also applicable to poly(vinyl chloride) (Test Method D 3749).

10. Apparent Density

10.1 The apparent or bulk density of the solid resin is the weight of the powder per unit volume, that is, grams per cubic centimetre (or pounds per cubic foot). This parameter is a measure of the compactness of the powder. A cylindrical cup of 100 ± 0.5 mL is used in measuring this characteristic in Test Methods D 1895; a small quantity of powdered carbon black is used to dissipate the electrostatic charge present occasionally on vinyl resin powders or granules.

11. Particle Size

11.1 The median particle size and particle size distribution of the resin sample are determined using sieve analysis (Test Method D 1921). A small amount of carbon black is used to dissipate the static charge generated in the procedure. The lower size limit of the determination is about 38 μm; for smaller particle sized resins, sedimentation methods are recommended.

11.2 Test Method D 1705 describes a wet sieve method that can also be used to determine the particle size and distribution.

12. Keywords

12.1 poly(vinyl chloride); resins; vinyl chloride copolymers