Designation: D3591 – 97 (Reapproved 2011)

Standard Test Method for Determining Logarithmic Viscosity Number of Poly(Vinyl Chloride) (PVC) in Formulated Compounds[1]

This standard is issued under the fixed designation D3591; 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 logarithmic viscosity number of poly(vinyl chloride) (PVC) homopolymers after compounding or processing.

1.2 It is the basic assumption of this technique that the formulation of the compounded resin is known and that any additives present can be separated from the resin by extraction with diethyl ether. This is necessary to permit adjustment of the amount of sample used in the test to give a resin concentration in cyclohexanone of 0.2 ± 0.002 g/100 mL.

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.* Specific precautionary statements are given in 7.3 and 8.4.1.

NOTE 1—This test method and ISO 1628-2 are not equivalent.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers

D1243 Test Method for Dilute Solution Viscosity of Vinyl Chloride Polymers

D2124 Test Method for Analysis of Components in Poly(Vinyl Chloride) Compounds Using an Infrared Spectrophotometric Technique

E1 Specification for ASTM Liquid-in-Glass Thermometers

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

IEEE/ASTM SI-10 Standard for the Use of International System of Units (SI)

2.2 *ISO Standard:*

1628-2 Plastics—Determination of Viscosity Number and Limiting Viscosity Number—Part 2: Poly (Vinyl Chloride) Resins[3]

3. Terminology

3.1 Units and symbols used in this test method are those recommended in IEEE/ASTM SI-10.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 The term *logarithmic viscosity number* is defined by the equation is 9.1.

4. Summary of Test Method

4.1 The sample is pressed into a thin film and extracted to remove the plasticizer.

4.2 The plasticizer-free film is dissolved in cyclohexanone and centrifuged to remove insoluble matter.

4.3 The viscosity of the cyclohexanone solution is measured in accordance with Test Method D1243.

5. Significance and Use

5.1 The logarithmic viscosity number provides information on the effect of compounding or processing of PVC.

5.2 Exposure of PVC compositions to shear or to high temperatures can result in a change in the logarithmic viscosity number of the resin.

6. Apparatus

6.1 *Centrifuge,* capable of 2500 rpm with 100-mL sample container.

6.2 *Heated Hydraulic Press,* capable of 620-kN ram force and a temperature of 165°C.

6.3 *Soxhlet Extraction Apparatus* with a 150-mL flask and a 27 by 100-mm thimble.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

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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.

6.4 *Volumetric Flasks,* 100-mL.

6.5 *Viscometers.*[4]

6.6 *Infrared Spectrophotometer,* see 5.4 of Method D2124.

7. Reagents

7.1 *Cyclohexanone,* high-purity (see Annex A1).

7.2 *Diethyl Ether,* anhydrous, reagent grade.

7.3 (**Warning—**Safety precautions should be taken to avoid personal contact, to eliminate toxic vapors, and to guard against explosive hazards in accordance with the hazardous nature of the particular reagent being used.)

8. Procedure

8.1 Prepare the PVC sample for extraction by pressing a film. The film should be 0.02 to 0.5 mm (1 to 2 mil) thick. Prepare two films in order to make duplicate runs.

8.1.1 Heat the hydraulic press to 165°C (330°F).

8.1.2 Place 2 g of sample between two sheets of aluminum foil and insert into the press.

8.1.3 Allow the sample to come to temperature for 2.5 min. During the next 0.5 min, increase the force on the sample to 620 kN. Maintain the force for 3 min, then cool while maintaining the force.

8.2 Weigh, to ±0.2 mg, approximately 1 g of pressed film into a 27 by 100-mm extraction thimble.

8.3 Place the thimble in a Soxhlet extraction apparatus fitted with a tared 150-mL flask, and extract with 120 mL of diethyl ether for 20 h.

8.4 Remove the tared 150-mL flask containing the diethyl ether and extracted plasticizer from the extraction apparatus, and gently heat to boil off the ether.

8.4.1 (**Warning—**When evaporating a quantity of ether to near dryness, precautions should be taken to guard against an explosive hazard, due to peroxides which may be in the ether or which may have been formed during use.)

8.5 Place the flask in an evacuated desiccator for a minimum of 1 h to remove the last traces of ether.

8.6 Weigh, to ±0.2 mg, the flask containing the extracted plasticizer.

8.7 Calculate the percentage plasticizer as follows:

$$\text{Plasticizer, \%} = (A \times 100)/B \quad (1)$$

where:

A = weight of extracted plasticizer (7.6), and
B = sample weight (7.2).

8.8 Dry the film to remove all solvent.

8.8.1 The extracted film must be free of plasticizer. Errors in excess of 10 % will result from small residual amounts of plasticizer. Examine the extracted film by infrared spectroscopy to ascertain that the plasticizer level is less than 0.05 % in order to obtain satisfactory results. An example for a carbonyl containing plasticizer is shown in Fig. 1 and Fig. 2.

[4] Cannon Fenske No. 75 or Ubbelohde No. 1 have been found satisfactory for this purpose.

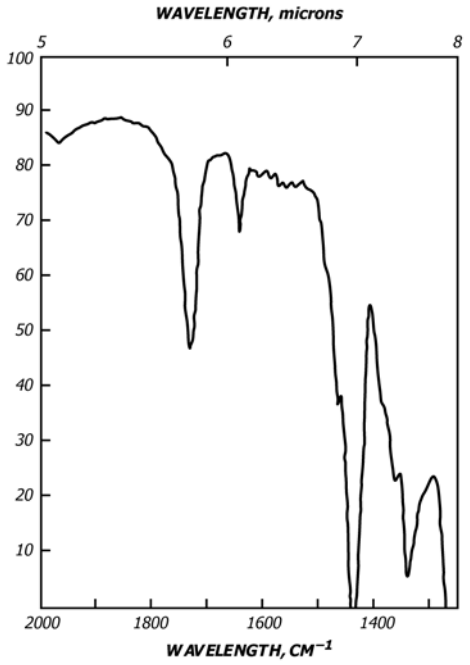

FIG. 1 Calibration Curve, 1 % Plasticizer in PVC Resin at 5.8 µm

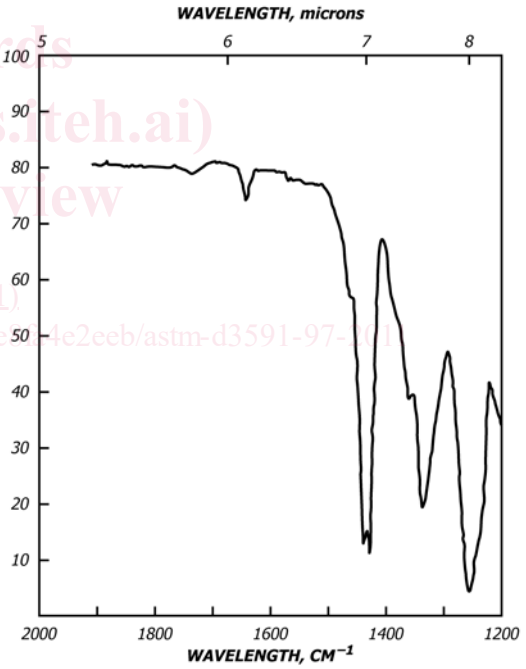

FIG. 2 Acceptable Residual Plasticizer, Less than 0.05 % at 5.8 µm

8.9 Determine the sample size of the extracted film that will yield 0.02 ± 0.002 g of PVC resin as follows:

$$F = \frac{100 - P}{R} \times 0.2 \quad (2)$$

where:

F = weight of extracted film, g,
P = plasticizer, %, and
R = PVC, %.