Designation: D3591 – 22

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, health, and environmental 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.

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]

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
D883 Terminology Relating to Plastics
D1243 Test Method for Dilute Solution Viscosity of Vinyl Chloride Polymers
D1600 Terminology for Abbreviated Terms Relating to Plastics
E456 Terminology Relating to Quality and Statistics
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids
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*—For definitions of terms pertaining to plastics used in this test method, refer to Terminology D883. For definitions of abbreviations used in this test method, refer to Terminology D1600, unless otherwise indicated. For definitions of terms that appear in this test method relating to quality and statistics, refer to Terminology E456.

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.

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[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.

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

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.

6.4 *Volumetric Flasks,* 100-mL.

6.5 *Viscometers.*[4]

6.6 *Infrared Spectrophotometer.*

6.7 *Filter Funnel,* funnel, Hirsch-Type; borosilicate glass; with coarse fritted disc, pore size: 40–60 µm.

6.8 *Thermometer,* standard, in accordance with Test Method E2251.

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:

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

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.

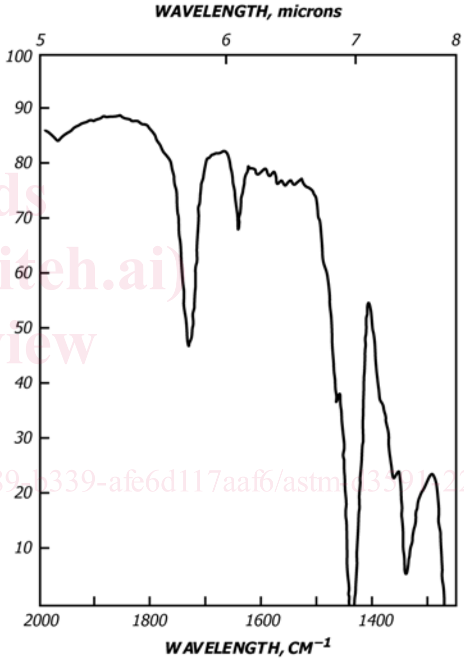

FIG. 1 Calibration Curve, 1 % Plasticizer in PVC Resin at 5.8 µm

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

$$F = \frac{100 - P}{R} \times 0.2 \tag{2}$$

where:

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

8.10 Weigh a sample of extracted film as determined in 8.9 and transfer to a 100-mL glass-stopped volumetric flask. Take care to transfer all of the weighed sample.

8.11 Add 50 to 70 mL of cyclohexanone to the flask. Make sure that all the sample is in the solvent and not attached to the neck of the flask.

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

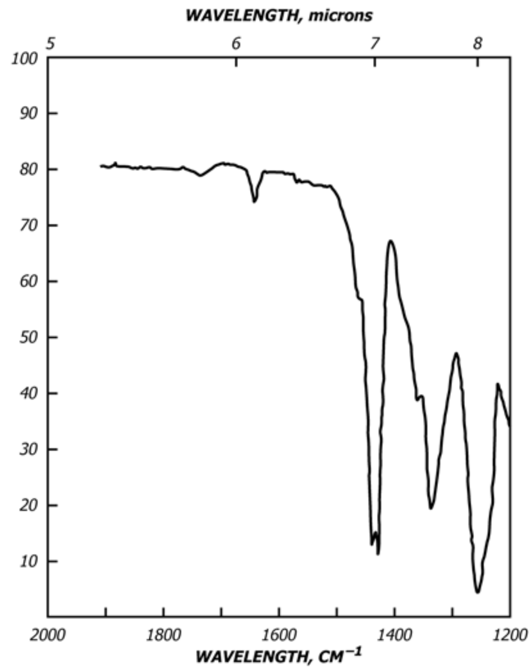

FIG. 2 Acceptable Residual Plasticizer, Less than 0.05 % at 5.8 µm

8.11.1 Use freshly distilled cyclohexanone. Serious errors of greater than 10 % can occur by not observing this factor. Details of the distillation are discussed in Annex A1.

8.12 Heat the flask to 85 ± 10°C until the resin is dissolved. Occasional shaking will reduce the time required for solution. Heating should not exceed 12 h and should preferably be less to minimize degradation.

8.13 Cool the solution and adjust to a solution volume of 100 mL.

8.14 Centrifuge for 30 min at about 2500 rpm until the solution is reasonably clear. A slight haze has been found to make no significant difference in the measurements. (As a standard for this haze level, disperse 3 mg of Dythal powder in 100 g of cyclohexanone; this 30-ppm suspension is known to give no effect on the measurements.)

8.15 Decant the supernatant liquid through a fritted-glass 40–60 µm filter directly into the viscometer.

8.16 Place the viscometer in a water bath at a temperature of 30 ± 0.5°C controlled to within ±0.01°C. Allow at least 10 min for the viscometer to come to equilibrium.

8.17 Measure the efflux time of the solution and the pure solvent (aged at 85 ± 10°C) in the viscometer. The efflux time of the solution or the solvent should be within 0.1 % for repeated runs on the same filling.

8.18 Duplicate determination should be run.

9. Calculation

9.1 Calculate the logarithmic viscosity number as follows:

$$\text{Logarithmic viscosity number} = \ln(t/t_o)/C \qquad (3)$$

where:

ln = natural logarithm,
t = efflux time of solution, s,
t_o = efflux time of solvent, s, and
C = concentration, weight of PVC sample used per 100 mL of solution, g/100 mL.

9.2 The units of logarithmic viscosity number are millilitres per gram.

10. Report

10.1 Report the following information:

10.1.1 Complete identification of the sample tested,
10.1.2 Date,
10.1.3 Efflux time of solution,
10.1.4 Efflux time of solvent, and
10.1.5 Logarithmic viscosity number.

TABLE 1 Logarithmic Viscosity Number (Inherent Viscosity) of Formulated PVC Compounds at 30°C

Material	Mean	S_r	S_R
PVC–1	1.0006	0.00953	0.0430
PVC–4	1.2425	0.01138	0.0430
PVC–5	1.2322	0.00584	0.0609
PVC–6	1.2619	0.00971	0.0411
PVC–7	0.9605	0.01062	0.0695
Overall average		0.0097	0.053

Based on five laboratories, two determinations. PVC–1 used four laboratories.

11. Precision and Bias

11.1 Table 1 is based on a round robin[5] conducted in 1976 in accordance with Practice E691, involving five materials tested by five laboratories. For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories which tested them. Each test result was the average of two individual determinations. Each laboratory obtained two test results for each material.

NOTE 2—The explanations of *r* and *R* (11.2 – 11.2.2) only are intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 should not be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots formulations, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E691 to generate data specific to their materials and laboratory (or between specific laboratories).

11.2 *Concept of r and R in Table 1*—If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing two specimens for each test result, then:

11.2.1 *Repeatability* precision under repeatability conditions in accordance with Terminology E456.

11.2.2 *Reproducibility* precision under reproducibility conditions in accordance with Terminology E456.

NOTE 3—Only five laboratories participated in this round robin, making this value somewhat questionable.

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1057.