Designation: D1823 – 16

Standard Test Method for
Apparent Viscosity of Plastisols and Organosols at High Shear Rates by Extrusion Viscometer[1]

This standard is issued under the fixed designation D1823; 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 measurement of plastisol and organosol viscosity at high shear rates by means of an extrusion viscometer.

1.2 Apparent viscosity at low shear rates is covered in Test Method D1824.

1.3 The values stated in SI units are to be regarded as standard. The values in parentheses are given for information only.

1.4 *This standard does not purport to address the safety concerns 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.*

NOTE 1—This standard and ISO 4575-2007 address the same subject matter, but differ in technical content.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1475 Test Method For Density of Liquid Coatings, Inks, and Related Products

D1755 Specification for Poly(Vinyl Chloride) Resins

D1824 Test Method for Apparent Viscosity of Plastisols and Organosols at Low Shear Rates

E1 Specification for ASTM Liquid-in-Glass Thermometers

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

2.2 *ISO Standard:*

ISO 4575-2007 Poly Vinyl Chloride Pastes—Determination of Apparent Viscosity Using the Severs Rheometer[3]

3. Summary of Test Method

3.1 The sample is conditioned to the proper temperature, placed into an extrusion chamber, and extruded under standard conditions. The viscosity is calculated from the extrusion pressure and the rate of flow through the orifice.

4. Significance and Use

4.1 The suitability of a dispersion resin for any given application is dependent upon its viscosity characteristics.

4.2 The extrusion viscosity defines the flow behavior of a plastisol or organosol under high shear. This viscosity relates to the conditions encountered in mixing, pumping, knife coating, roller coating, and spraying processes.

5. Apparatus

5.1 *Extrusion Rheometer.*[4]

5.2 *Orifice,* 3.17 ± 0.13 mm (0.125 ± 0.005 in.) inside diameter and 50 ± 1.0 mm (1.97 ± 0.04 in.) long.

5.3 *Sample Containers, Tin Cans, or Glass Jars,* 1-pt (500-mL) capacity.

5.4 *Paper Cups,* 8-oz (250-mL) capacity.

5.5 *Nitrogen Cylinder,* equipped with pressure regulator and gage.

5.6 *Thermometer*—ASTM Solvents Distillation Thermometer having a range from − 2 to +52°C (28 to 126°F) and conforming to the requirements for Thermometer 37C as prescribed in Specification E1. Use of temperature measuring devices such as liquid-in-glass thermometers, thermocouples, or platinum resistance thermometers having equivalent or better accuracy and precision, while covering the temperature range of Thermometer 37C shall be permitted. (**Warning—**Thermometers referenced in Specification E1 contain mercury, mercury thallium eutectic alloy, or toluene or other suitable liquid colored with a permanent red dye. Mercury has been

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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 D20.15 on Thermoplastic Materials (Section D20.15.08).

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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.

[4] The sole source of supply of the Burrell Severs, Model A-120 known to the committee at this time is Burrell Corp., 2223 Fifth Ave., Pittsburgh, PA 15219. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

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

designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Safety Data Sheet (SDS) for details and EPA's website- http://www.epa.gov/mercury/faq.htm - for additional information. Users should be aware that selling mercury and/or mercury containing products into your state may be prohibited by state law.)

5.7 *Timer.*

6. Conditioning

6.1 Maintain the plastisol or organosol samples at 23± 1°C (73 ± 2 °F) and 50 ± 10 % relative humidity at all times after mixing and throughout the period of viscosity determinations.

7. Procedure

7.1 *Set Up Rheometer*—Attach the pressure regulator to the nitrogen tank. Connect the nitrogen supply to the rheometer by means of the copper tubing. Do not use oxygen or liquid pressure sources (Note 2). Set the three-way quick-acting valve to the IN position. Regulate the tank pressure to give 1.04 MPa (150 psi) pressure to the instrument. Do not use input or line pressure over 1.38 MPa (200 psi). Insert the medium-size orifice (approximately 3.2 mm (⅛ in.) inside diameter) in the orifice retaining cap, with the orifice and the barrel, then screw the cap solidly in place. Mount the barrel in the instrument.

NOTE 2—Air may be used instead of nitrogen.

7.2 Weigh four empty paper cups for each sample to be tested. Record tare weight of each cup to the nearest 0.1 g. Fill the barrel with the sample to be tested to within 13 mm (½ in.) from the top of the barrel. Measure the sample temperature.

7.3 Insert the top air cap and gasket into the air cap ring, screw it in place on top of the barrel, and connect the air supply quick-connector.

7.4 Set the rheometer regulator gage (on the right side of the instrument) to 0.069 MPa (10 psi) pressure. Open the three-way quick-acting valve to the OUT position and allow the mix to extrude into an unweighed paper cup for 10 s. Adjust the gage pressure back to 0.069 MPa (10 psi).

7.5 Quickly place a preweighed and labeled paper cup under the nozzle and at the same time start the timer. Collect the extrudate until approximately 50 g of sample have entered the cup. Simultaneously remove the cup and stop the timer, again placing the unweighed cup under the nozzle. (Use a maximum flow time of 200 s for extremely viscous samples.)

7.6 Push the three-way quick-acting valve to the IN position to turn off the nitrogen supply to the chamber.

7.7 Record the extrudate weight to the nearest 0.1 g and efflux time to the nearest second.

7.8 Increase the gage pressure to 0.28 MPa (40 psi) and repeat 7.4 – 7.7. Increase to 0.48 MPa (70 psi) and repeat. Make a fourth determination at 0.69 MPa (100 psi). Report the exact efflux time for each determination. After all tests have been completed, turn off the nitrogen supply at the tank. Release the pressure in the instrument by pulling the three-way valve to the OUT position.

7.9 Clean the orifice between runs using pipe cleaners that have been wetted with mineral spirits. Take care that the inner surface of the orifice does not become scratched. After rinsing with mineral spirits, dry the orifice in air.

8. Calculation

8.1 Calculate the shear stress, shear rate, and viscosity as follows:

$$\text{Shear stress, MPa (or psi)} = PR/2L \tag{1}$$

where:

P = pressure in rheometer, MPa (or psi),
R = radius of orifice, cm (in.), and
L = length of orifice, cm (in.)

$$\text{Shear rate, s}^{-1} = 4W/3.1416R^3\,DT \tag{2}$$

where:

W = weight of material effluxed, g,
D = density of the sample, determined in accordance with Test Method D1475, except convert lb/gal to g/mL, and
T = efflux time, s.

NOTE 3—The preferred practice is to determine both the density and efflux time on deaerated material. If the efflux time of undeaerated material is specifically desired, the determination of density on an undeaerated sample may also be desirable.

$$\text{Viscosity, pascal seconds} = (\text{shear stress/shear rate}) \times 10^6 \tag{3}$$

if shear stress is in MPa.

$$\text{Viscosity, poises} = (\text{shear stress/shear rate}) \times 6.895 \times 10^4$$

if shear stress is in psi.

9. Report

9.1 The report shall include the following:

9.1.1 Complete sample identification,

9.1.2 Test temperature as measured in,

9.1.3 Conditioning time, and

9.1.4 Extrusion viscosity, in pascal seconds (or poises); shear rate, in reciprocal seconds; and shear stress, in pascals (or pounds-force per square inch), for each of the four pressures (0.069, 0.28, 0.48, and 0.69 MPa (or 10, 40, 70, and 100 psi)).

NOTE 4—If only one viscosity is to be reported, report the data obtained at 100 psi together with the shear rate and shear stress, for example: "Viscosity at a shear stress of Z psi and a shear rate of Y s^{-1} = X poises." The most information will be gained, however, by a plot of shear rate versus shear stress and would typify a true flow curve. In all cases where only one value is to be reported, the test must be run at each pressure in the order indicated in the procedure.

10. Precision and Bias[5]

10.1 Tables 1-4 are based on a round robin[5] conducted in 1983 involving six PVC dispersion resins tested by four laboratories at extrusion pressures of 10, 40, 70, and 100 psi.

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: RR:D20-1137.

TABLE 1 Precision of Viscosity Data at 10 psi Extrusion Pressure

Resin	Average Viscosity (Poises)	Values expressed as % of the Average			
		νr[A]	νR[B]	r[C]	R[D]
IV	79.95	10.5	18.3	29.4	51.1
V	81.05	7.0	25.8	19.6	72.2
I	87.95	7.1	11.9	19.9	33.4
III	99.90	6.1	8.6	17.1	24.1
II	121.55	12.0	13.7	33.6	38.5
VI	221.25	9.4	9.8	26.3	27.3

[A] νr is the within-laboratory coefficient of variation of the average.
[B] νR is the between-laboratories coefficient of variation of the average.
[C] r is the within-laboratory repeatability Limit (= 2.8 νr).
[D] R is the between-laboratories reproducibility Limit (= 2.8 νR).

TABLE 2 Precision of Viscosity Data at 40 psi Extrusion Pressure

Resin	Average Viscosity (Poises)	Values expressed as % of the Average			
		νr[A]	νR[B]	r[C]	R[D]
V	86.30	7.3	10.2	20.4	28.6
IV	129.85	11.9	24.2	33.3	67.7
II	130.90	9.9	21.8	27.7	61.2
I	147.00	7.4	9.6	20.7	26.9
III	184.20	5.5	11.1	15.4	31.0
VI	211.80	5.9	8.7	16.5	24.3

[A] νr is the within-laboratory coefficient of variation of the average.
[B] νR is the between-laboratories coefficient of variation of the average.
[C] r is the within-laboratory repeatability Limit (= 2.8 νr).
[D] R is the between-laboratories reproducibility Limit (= 2.8 νR).

TABLE 3 Precision of Viscosity Data at 70 psi Extrusion Pressure

Resin	Average Viscosity (Poises)	Values expressed as % of the Average			
		νr[A]	νR[B]	r[C]	R[D]
V	79.10	7.6	11.1	21.3	31.2
II	119.50	4.9	6.4	13.7	18.0
IV	139.85	11.9	24.3	33.3	68.1
I	150.50	7.6	16.5	21.3	46.3
VI	182.40	5.9	10.3	16.5	28.7
III	233.95	6.6	13.8	18.5	38.5

[A] νr is the within-laboratory coefficient of variation of the average.
[B] νR is the between-laboratories coefficient of variation of the average.
[C] r is the within-laboratory repeatability Limit (= 2.8 νr).
[D] R is the between-laboratories reproducibility Limit (= 2.8 νR).

TABLE 4 Precision of Viscosity Data at 100 psi Extrusion Pressure

Resin	Average Viscosity (Poises)	Values expressed as % of the Average			
		νr[A]	νR[B]	r[C]	R[D]
V	70.20	6.6	10.8	18.5	30.3
II	107.50	5.3	7.3	14.8	20.6
IV	130.80	6.3	16.5	17.6	46.2
I	134.35	12.4	24.2	34.7	67.7
VI	153.05	5.0	10.1	14.0	28.4
III	249.05	7.4	14.3	20.7	39.9

[A] νr is the within-laboratory coefficient of variation of the average.
[B] νR is the between-laboratories coefficient of variation of the average.
[C] r is the within-laboratory repeatability Limit (= 2.8 νr).
[D] R is the between-laboratories reproducibility Limit (= 2.8 νR).

For each resin, all the samples were prepared at one source, but the individual plastisols were prepared according to Method D1755 at the laboratories which tested them. Each test result consisted of one individual determination at the stated extrusion pressure. Each laboratory obtained 5 test results for each resin at each extrusion pressure. (**Warning—**The following explanations of *r* and *R* (10.2 – 10.2.3) are only intended to present a meaningful way of considering the approximate precision of this test method. With data from only four laboratories, the between-laboratories results, in particular, should be viewed with extreme caution! The data in Tables 1-4 should not be rigorously applied to acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 10.2 – 10.2.3 would then be valid for such data.)

10.2 *Concept of r and R*—If S_r and S_R were calculated from a large enough body of data, and for test results consisting of one determination per test result.

10.2.1 *Repeatability Limit, r*—In comparing two test results for the same material, obtained by the same operator using the same equipment on the same day, the two test results should be judged not equivalent if they differ by more than the *r* value for that material.

10.2.2 *Reproducibility Limit, R*—In comparing two test results for the same material, obtained by different operators using different equipment in different laboratories on different days, the two test results should be judged not equivalent if they differ by more than the *R* value for that material.

10.2.3 Any judgment in accordance with 10.2.1 or 10.2.2 would have an approximate 95 % (0.95) probability of being correct.

10.3 There are no recognized standards by which to estimate bias of this test method.

11. Keywords

11.1 apparent viscosity; extrusion viscometer; high shear rate viscometry; PVC organosol; PVC plastisol

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D1823 - 95(2009)) that may impact the use of this standard. (May 1, 2016)

(1) Revised ISO statement in Note 1.
(2) Revised 5.6 on thermometers incorporating current wording from Specification E1.

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