Designation: D1824 – 16

Standard Test Method for Apparent Viscosity of Plastisols and Organosols at Low Shear Rates[1]

This standard is issued under the fixed designation D1824; 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 U.S. Department of Defense.

1. Scope*

1.1 This test method covers the measurement of plastisol and organosol viscosity at low shear rates.

1.2 Apparent viscosity at high shear rates is covered in Test Method D1823.

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

1.4 *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.*

NOTE 1—This test method resembles ISO 3219-1977 in title only. The content is significantly different.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1755 Specification for Poly(Vinyl Chloride) Resins

D1823 Test Method for Apparent Viscosity of Plastisols and Organosols at High Shear Rates by Extrusion Viscometer

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 3219-1977: Polymers in the Liquid, Emulsified, or Dispersed State—Determination of Viscosity With a Rotational Viscometer Working at a Defined Shear Rate[3]

3. Summary of Test Method

3.1 The sample is conditioned to the proper temperature and its viscosity is determined.

4. Significance and Use

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

4.2 The viscosity defines the flow behavior of a plastisol or organosol under low shear. This viscosity relates to the conditions encountered in pouring, casting, molding, and dipping processes.

5. Apparatus

5.1 *Viscometer, Concentric Cylinder Rotational*—The essential instrumentation required providing the minimum rotational viscometer analytical capabilities include:

5.1.1 A *drive motor* to apply a unidirectional displacement to the specimen at a rate from 0.5 to 60 r/min constant to ±0.5 %.

5.1.2 A *force sensor* to measurement the torque developed by the specimen by the rotational element.

5.1.3 A *coupling shaft*, or other means, to transmit the rotational displacement from the motor to the spindle.

NOTE 2—It is helpful to have a mark on the shaft to indicate appropriate test fluid level.

5.1.4 A *rotational element, spindle or tool* of the right circular cylindrical shape as shown in Fig. 1, to fix the specimen between the drive shaft and a stationary position.

NOTE 3—The rotational element dimensions *L* and *D*, are selected so that the measured viscosity is between 10 and 90 % of the range of that element.

5.1.5 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for rotational viscometry are torque, rotational speed, temperature and time.

NOTE 4—Manual observation and recording of data are acceptable.

[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.

Current edition approved April 1, 2016. Published April 2016. Originally approved in 1961. Last previous edition approved in 2010 as D1824 - 95(2010). DOI:10.1520/D1824-16.

[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**

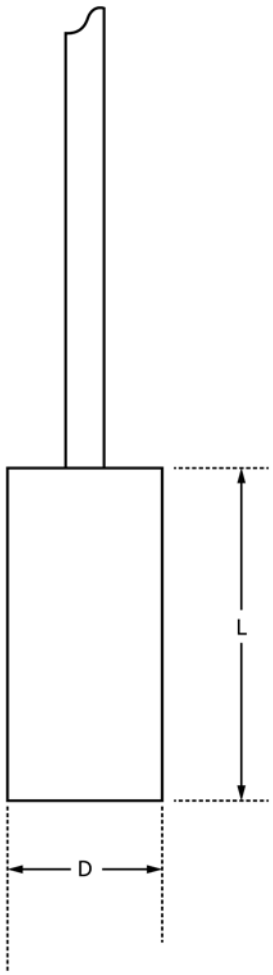

FIG. 1 Rotational Spindle Configuration

5.1.6 A *stand* to support, level, and adjust the height of the drive motor, shaft and rotational element.

5.1.7 Auxiliary instrumentation considered useful in conducting this method includes:

5.1.7.1 *Data analysis capability* to provide viscosity, stress or other useful parameters derived from the measured signals.

5.1.7.2 A *level* to indicate the vertical plumb of the drive motor, shaft and rotational element.

5.1.7.3 A *guard* or other method to protect the rotational element from mechanical damage due to contact between the rotational element and the container walls.

5.2 *Sample Containers, Tin Cans, or Glass Jars,* 500-mL (1-pint) capacity with minimum dimensions of 80 mm (3.15 in.) inside diameter by 80 mm (3.15 in.) depth.

5.3 *Temperature Measurement Device*—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.

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 Select a spindle that will read in the middle or upper portion of the viscometer indication at the highest rotational speed to be used.

7.2 Insert the spindle into the sample at an angle to wet it. Withdraw the spindle and attach it to the viscometer motor.

7.3 Taking care not to entrap an air bubble, lower the spindle into the specimen near the edge of the container. Then, adjust the spindle position so that it is centered in the container. Adjust its test specimen level to the level recommended by the instrument operations manual.

7.4 Initiate the rotation of the spindle at its lowest speed. Allow it to run 2 min.

7.5 Record the torque scale reading during the next rotation. Record the corresponding specimen temperature.

7.6 Without stopping the rotation of the spindle, increase the rotational speed by a factor of 2. Record the torque reading and temperature after 2 min.

7.7 Repeat 7.6 until the highest rotational speed available is reached.

8. Calculation

8.1 Calculate the viscosity using the procedure provided in the viscometer operations manual.

9. Report

9.1 Report the following information:

9.1.1 Complete sample identification.

9.1.2 Viscometer model and spindle used for test.

9.1.3 Viscosity and temperature at each spindle speed (shear rate) used.

9.1.4 Any sample conditioning time.

10. Precision and Bias[4]

10.1 Table 1 and Table 2 are based on a round robin conducted in 1983 involving six PVC dispersion resins tested by five laboratories at 2 r/min and 20 r/min. For each resin, all the samples were prepared at one source, but the individual plastisols were prepared according to Specification D1755 at the laboratories which tested them. Each test result consisted of one individual determination at the stated rotational speed. Each laboratory obtained five test results for each resin at 2 r/min and 20 r/min.

NOTE 5—**Caution:** 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 five laboratories, the between-laboratories results, in particular, should be viewed with extreme caution! The data in Table 1 through 4 should not be

[4] Supporting data are available from ASTM Headquarters. Request RR: D20-1137.

TABLE 2 Precision of Viscosity Data at (20 r/min)

NOTE 1—0.1 Pa·s = 1 Poise

	Average Viscosity	Values expressed as % of the Average			
Resin	(Poises)	v_r [A]	v_R [B]	r [C]	R [D]
V	24.72	8.5	12.1	23.8	34.0
IV	38.36	3.9	12.8	10.9	35.8
II	82.24	6.3	26.8	17.6	74.9
I	92.52	6.9	14.5	19.3	40.6
III	102.40	11.8	22.6	33.0	63.2
VI	226.05	11.6	27.1	32.5	75.9

[A] v_r is the within-laboratory coefficient of variation of the average.
[B] v_R is the between-laboratories coefficient of variation of the average.
[C] r is the within-laboratory Repeatability Limit (= 2.8 vr).
[D] R is the between-laboratories Reproducibility Limit (= 2.8 vR).

TABLE 1 Precision of Viscosity Data at (2 r/min)

NOTE 1—0.1 Pa·s = 1 Poise

	Average Viscosity	Values expressed as % of the Average			
Resin	(Poises)	v_r [A]	v_R [B]	r [C]	R [D]
I	33.20	8.1	12.0	22.7	33.7
IV	62.00	4.2	13.7	11.8	38.4
II	141.76	8.2	31.0	23.0	86.7
V	197.04	13.2	44.4	37.0	124.2
III	265.36	18.6	35.9	52.1	100.6
VI	607.68	23.6	45.1	66.1	126.3

[A] v_r is the within-laboratory coefficient of variation of the average.
[B] v_R is the between-laboratories coefficient of variation of the average.
[C] r is the within-laboratory Repeatability Limit (= 2.8 v_r).
[D] R is the between-laboratories Reproducibility Limit (= 2.8 v_R).

rigorously applied to acceptance or rejection of material, as those data are specific to the interlaboratory study 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; low shear rate viscometry; PVC organosol; PVC plastisol; rotational viscometer

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D1824 – 95(2010)) that may impact the use of this standard. (April 1, 2016)

(1) Expanded 6.1 to include a technical description of the rotational viscometer.

(2) Expanded 7.2 to 7.7 for readability.

(3) Added new Fig. 1.