Designation: D 5225 – 98

Standard Test Method for Measuring Solution Viscosity of Polymers with a Differential Viscometer[1]

1. Scope *

1.1 This test method covers the determination of the solution viscosity of polymers using a differential or the modified differential viscometer. It is applicable to all polymers that dissolve completely without chemical reaction or degradation to form solutions that are stable with time and temperature. Results of the test are usually expressed as specific viscosity, intrinsic viscosity (limiting viscosity number), inherent viscosity (logarithmic viscosity number), or relative viscosity (viscosity ratio).

1.2 Since there is more than one type of viscometer available to measure a differential pressure, follow the manufacturer's directions applicable to the equipment being used.

1.3 The solution viscosity values are comparable with those obtained using a glass capillary of Test Method D 2857. This test method differs from the glass capillary in that the solvent and the solution are compared at the same time that a test is run. With a glass capillary, each solution must be referenced back to the solvent run in the same capillary at the same temperature.

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.* For specific hazard statements, see Section 8.

NOTE 1—There is no ISO equivalent method.

2. Referenced Documents

2.1 *ASTM Standards:*

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

D 2857 Test Method for Dilute Solution Viscosity of Polymers[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *inherent viscosity*—the ratio of the natural logarithm of the relative viscosity to the concentration. The IUPAC term for inherent viscosity is *logarithmic viscosity number.*

3.1.2 *intrinsic viscosity*—limit of the reduced and inherent viscosities as the concentration of the polymeric solute approaches zero and represents the capacity of the polymer to increase viscosity. The IUPAC term for intrinsic viscosity is *limiting viscosity number.*

3.1.3 *reduced viscosity*—the ratio of the specific viscosity to the concentration. Reduced viscosity is a measure of the specific capacity of the polymer to increase the relative viscosity. The IUPAC term for reduced viscosity is *viscosity number.*

3.1.4 *relative viscosity*—the ratio of the polymer solution pressure to the pressure of the solvent.

3.1.5 *specific viscosity*—the relative viscosity minus one.

3.1.6 *viscosity constant, K*—baseline reading when solvent is present in both capillaries.

4. Summary of Test Method

4.1 *Differential Viscometer* (Fig. 1):

4.1.1 The viscosity measurement with the differential viscometer is based on a fluid analog of the Wheatstone Bridge. Pure solvent at constant inlet pressure P_i enters a balanced capillary network and flows through, producing a zero or baseline pressure on the differential pressure transducer. Solution is loaded into the sample reservoir A and then injected onstream by means of the switching valve S_A. The differential pressure begins to rise until it reaches a steady state value of ΔP proportional to the specific viscosity of the solution. The differential pressure is monitored continuously on a strip chart recorder or computer, providing a baseline where ΔP is measured. The equation relating ΔP to specific viscosity is:

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[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

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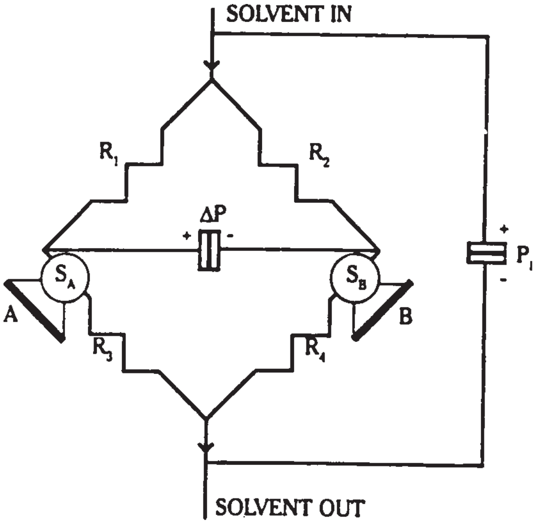
[2] *Annual Book of ASTM Standards*, Vol 08.01.

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

[4] *Annual Book of ASTM Standards*, Vol 14.02.

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



NOTE—
R_1, R_2, R_3, R_4= Matched SS Capillary Tubing
A, B = Solution Holdup Reservoirs
S_A, S_B= Switching Valve
P_i= Solvent Inlet Pressure Transducer
ΔP = Differential Pressure Transducer

FIG. 1 Differential Viscometer

$$\eta_{sp} = \frac{4\Delta P}{P_i - 2\Delta P} \quad (1)$$

4.1.2 Derivation of the equation is in Annex A1.

4.2 *Modified Differential Viscometer* (Fig. 2):

4.2.1 The modified differential viscometer has two stainless steel capillaries connected in series with a sample loading/injection valve before the second capillary. Two differential pressure transducers, P_1 and P_2, are connected in parallel across the capillaries. A pump continuously supplies solvent flow. The ratio of the pressures P_2 and P_1 is proportional to the ratio of the viscosities of the fluid in capillary 2 to that in capillary 1.

$$\frac{P_2}{P_1} = K\frac{\eta_2}{\eta_1} = K \cdot \text{Relative Viscosity} \quad (2)$$

4.2.1.1 *K,* the viscosity constant, is obtained from the baseline reading when solvent is present in both capillaries, so η_2/η_1 is unity.

4.2.1.2 With the valve in LOAD position, the sample is flushed through the sample loop by the syringe pump. A baseline reading is established and recorded by the computer data acquisition system. When the valve is switched to the INJECT position, solvent flowing from capillary 1 pushes the sample into capillary 2. The differential pressure P_2 will increase due to the higher viscosity of the sample solution. The steady state value of P_2/P_1 then yields the value of relative viscosity of the sample.

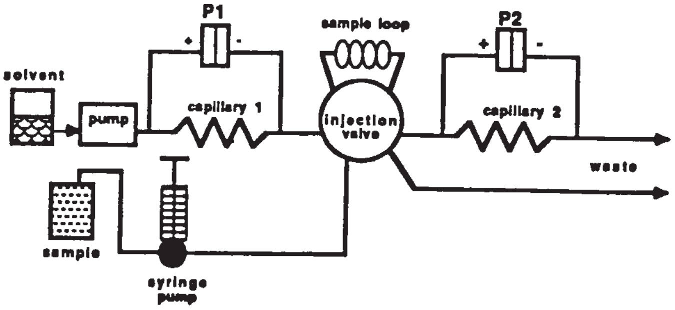


FIG. 2 Schematic of Relative Viscometer

$$\text{Relative Viscosity} = \frac{P_2}{KP_1} \quad (3)$$

4.2.1.3 Absolute viscosity of the sample may be calculated from relative viscosity, *RV,* assuming the viscosity of the solvent is known.

$$\eta(\text{sample}) = RV \cdot \eta(\text{solvent}) \quad (4)$$

5. Significance and Use

5.1 Solution viscosity values for polymers are related to the average molecular size of that portion of the polymer which dissolves in the solvent.

6. Apparatus

6.1 *Differential Viscometer*, Viscotek Model 100-01 viscometer,[5] or equivalent; or *Relative Viscometer*, Viscotek Model Y-500,[5] or equivalent,

6.2 *Kit for conversion to GPC Detector,*

6.3 *Computer*, IBM PC or equivalent, with 640K RAM, coprocessor, and graphics adapter,

6.4 *PC Based Software*, for data acquisition and processing.

6.5 *PC interface with Data Translation Board and external signal box and cable,*

6.6 *Printer,*

6.7 *Autosampler*, Viscotek #100-06, or equivalent,

6.8 *Glass Vial*, 25 mL,

6.9 *Screw Type Cap*, open top, size 24-400,

6.10 *Septa*, aluminum foil (household grade) or acceptable substitute,

6.11 *Balance*, accurate to 0.0001 grams,

6.12 *Filter Funnel*, and

6.13 *Filter medium*, coarse filter paper or metal screen of 140 mesh or finer.

7. Reagents and Materials

7.1 *Polymer Sample.*

7.2 *Solvent.*

7.3 *Compressed Gas Cylinder* of nitrogen or helium.

8. Hazards

8.1 Flammable solvents are to be used in a hood or a well ventilated area.

8.2 Solvents are to be dated and to be used on a first-in first-out basis.

8.3 Stored solvents prone to develop peroxides should be tested on a regular schedule for peroxide development.

9. Procedure

9.1 Make solvent and gas cylinder connections as required for the equipment.

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[5] Available from Viscotek Corporation, 13600 West Hardy Road, Houston, TX 77060.

9.2 Connect computer cables to autosampler, viscometer, PC interface board and external signal box, monitor, and printer.

9.3 Set viscometer oven temperature to maintain solution of the test polymer.

9.4 Adjust inlet solvent pressure to obtain the pressure differential and test time desired.

9.5 Prepare polymer solution. Weigh the polymer accurately to the nearest 0.0001 g and record weight.

9.6 Filter polymer solution into a 25-mL vial. Cover vial with an aluminum septa or equivalent and cap.

9.7 Position vial into autosampler.

9.8 Activate the computer and printer.

9.9 Program the instrument and processing parameters for the desired viscosity calculations.

9.10 Enter the data for the sample solutions positioned in the autosampler into the computer acquisition program queue.

9.11 Initiate viscosity acquisition.

9.12 After the last sample has been run, flush the sample lines and sample holder with fresh solvent.

9.13 Turn off viscometer, leaving the sample lines filled with solvent.

10. Report

10.1 Report viscosity data for each sample in the run queue.

11. Precision and Bias

11.1 Table 1 is the precision data for eight polymers which were measured using this test method. Each result is an average of three or more independent tests made by a single laboratory.

NOTE 2—**Caution:** The following explanations of I_r and I_R(11.2 thru 11.2.3) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 should not be rigorously applied to acceptance or rejection of material, as those data are specific to the one laboratory and may not be representative of other lots, conditions, materials, or laboratories.

NOTE 3—Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 11.2.1 through 11.2.3 would then be valid for such data.

11.2 *Concept of I_r and I_R*—If S_r and S_R have been calculated from a large enough body of data, then the following applies:

11.2.1 *Repeatability*—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 I_r for that material.

$$I_r = 2.83 \times S_r \quad (5)$$

11.2.2 *Reproducibility*—comparing two test results for the same material, obtained by different operators using different equipment on different days, the two test results should be judged not equivalent if they differ by more than the I_R for that material.

$$I_R = 2.83 \times S_R \quad (6)$$

11.2.3 Any judgment per 11.2.1 and 11.2.2 would have an approximate 95 % (0.95) probability of being correct.

11.3 Bias is systematic error which contributes to the difference between a test result and a true (or reference) value. There are no recognized standards on which to base an estimate of bias for this test method.

12. Keywords

12.1 differential solution viscosity; differential viscometer; polymer solution viscosity

ANNEX

(Mandatory Information)

A1. CALCULATIONS FOR DIFFERENTIAL VISCOMETER

A1.1 Assume R_3 is filled with solution and R_1, R_2, R_4 are filled with solvent. The measured quantities are the inlet pressure P_i and the differential pressure ΔP.

TABLE 1 Precision Data for the Solution Viscosities of Various Polymers

Polymer	Solvent	Concentration	Relative Viscosity			Inherent Viscosity			Intrinsic Viscosity		
			Mean	S_r	I_r	Mean	S_r	I_r	Mean	S_r	I_r
poly(1-butene)	decalin	0.1 g/100 mL	1.207	0.007	0.020	1.853	0.054	0.153	1.892	0.057	0.161
polycarbonate	methylene chloride	0.5 g/100 mL	. . .	. . .	. . .	0.460	0.003	0.0085	0.472	0.003	0.0085
poly(ethylene)	decalin	0.1 g/100 mL	1.124	0.005	0.014	1.166	0.044	0.125	1.181	0.045	0.127
poly(4-methyl-1-pentene)	decalin	0.1 g/100 mL	1.280	0.007	0.018	2.294	0.051	0.144	2.361	0.054	0.153
poly(propylene)	decalin	0.1 g/100 mL									
resin “A”			1.170	0.005	0.014	1.557	0.042	0.112	1.585	0.044	0.125
resin “B”			1.182	0.005	0.014	1.664	0.045	0.127	1.695	0.047	0.133
resin “C”			1.254	0.008	0.021	2.240	0.060	0.170	2.299	0.063	0.178
resin “D”			1.289	0.007	0.018	2.516	0.052	0.147	2.593	0.056	0.158
resin “E”			1.415	0.003	0.008	3.452	0.021	0.059	3.604	0.022	0.062
poly(phenylene oxide)	chloroform	0.1 g/100 mL	1.053	0.0005	0.001	0.500	0.003	0.0085	0.503	0.003	0.009
poly(styrene)	toluene	0.1 g/100 mL	1.095	0.0006	0.002	0.726	0.0015	0.004	0.737	0.0025	0.007
poly(vinyl chloride)	tetrahydrofuran	0.2 g/100 mL	1.225	0.0003	0.0009	1.015	0.001	0.003	1.039	0.001	0.003

$$\frac{\Delta P}{P_i} = \frac{P_1 - P_2}{P_i} \quad (A1.1)$$

P_1 = pressure drop across R_3 and
P_2 = pressure drop across R_4.

A1.2 R_2 and R_4 have equal flow resistance, so $P_i = 2P_2$, giving the following:

$$\frac{\Delta P}{P_i} = 1/2\left[\frac{P_1}{P_2} - 1\right] \quad (A1.2)$$

A1.3 Applying Poiseuelle's Law to capillaries R_3 and R_4:

$$\frac{P_1}{P_2} = \frac{\eta Q_1}{\eta_o Q_2} \quad (A1.3)$$

Q_1 = flow rate through R_1, R_3,
Q_2 = flow rate through R_2, R_4,
η = viscosity of solution, and
η_o = viscosity of solvent.

A1.4 The ratio of flow rates Q_1/Q_2 is equal to the inverse ratio of the total resistance in each side of the bridge.

$$\frac{Q_1}{Q_2} = \frac{\eta_o + \eta_o}{\eta_o + \eta} = \frac{2\eta_o}{\eta_o + \eta} \quad (A1.4)$$

A1.5 Combining Eqs (A1.2), (A1.3), and (A1.4), we find the following:

$$\frac{\Delta P}{P_i} = 1/2\left[\frac{\eta - \eta_o}{\eta + \eta_o}\right] \quad (A1.5)$$

A1.6 The definition of specific viscosity of a solution is as follows:

$$\eta_{sp} = \frac{\eta - \eta_o}{\eta_o} \quad (A1.6)$$

A1.7 Substituting into Eq (A1.5) yields the following:

$$\frac{\Delta P}{P_i} = \frac{\eta_{sp}}{2\eta_{sp} + 4} \quad (A1.7)$$

A1.7.1 Rearrange to give the following:

$$\eta_{sp} = \frac{4\Delta P}{P_i - 2\Delta P} \quad (A1.8)$$

A1.8 *Assumptions in Derivation:*

A1.8.1 Capillaries have equal flow resistance.

A1.8.2 Capillaries obey Poiseuelle's Law.

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D-20 has highlighted those changes that may impact the use of this test method. This section also may include descriptions of the changes or reasons for the changes, or both.

D 5225 – 98:

(*1*) An ISO equivalency statement (Note 1) was added and source reference (Footnote 5) was updated as part of the five-year review.