



among polymers from different manufacturing processes. Hence, its correlation with other properties of polymers produced by different processes, by even one manufacturer, may be limited.

4.3 The viscosity of polymer solutions may be drastically affected by the presence of known or unknown additives in the sample. The use of solution viscosity may be of questionable value where ethylene polymers are known or suspected to contain colorants, carbon black, low molecular weight hydrocarbons, fillers, or other additives.

4.4 The measurement of dilute solution viscosity of ethylene polymers presents problems not ordinarily encountered in viscosimetry. Ethylene polymers are not soluble at room temperature in any known solvent. Some of the higher density materials are insoluble below 100°C. Extreme care must be exercised in transferring the solution to the viscometer for the test if the correct solution concentration is to be maintained. This test has no significance unless the sample is completely soluble.

4.5 The solution viscosity is a function of the root-mean-square size of the polymer molecules in solution. It is known that the solvent selected and the temperature of the determination have an effect on the root-mean-square size of the particles. Hence, where a viscometer, solvent, or temperature other than specified is used, data may not be comparable to that obtained by this procedure.

## 5. Apparatus

5.1 *Volumetric Flasks*, 100-mL, grade EXAX or better.<sup>4</sup>

5.2 *Transfer Pipets*, Grade EXAX or better.<sup>4</sup>

5.3 *Constant Temperature Bath*, capable of maintaining  $135 \pm 10.1^\circ\text{C}$ .

5.4 *Viscometer*, Ubbelohde No. 1, calibrated for kinetic energy correction constant.

NOTE 3—Other types of viscometers may be used provided they can be shown to agree with the type specified.

5.5 *Oven*, maintained at  $140 \pm 5^\circ\text{C}$ .

5.6 *Timer*, as specified in 4.5 of Test Method D445.

5.7 *Thermometer*—An ASTM High Softening Point Thermometer having a range from 30 to 200°C, and conforming to the requirements for Thermometer 16C in Specification E1. Thermometric devices such as resistance temperature detectors (RTDs), having accuracies equal to or better than Thermometer 16C in the specified temperature range are also appropriate for use.

## 6. Reagents and Materials

6.1 *Solvent*—Decahydronaphthalene, practical grade, purified and redistilled, as follows:

6.1.1 The solvent shall be purified by percolation through 100 to 200 mesh commercial grade silica gel. This treatment removes naphthalene, tetrahydronaphthalene, and oxy compounds, particularly peroxides.

6.1.2 The redistilled product shall conform to the following requirements when tested in accordance with Method D86:

Standard Distillation	ASTM Method D86
Initial boiling point	190°C min
10 mL	191°C min
20 mL	192°C min
80 mL	194°C max
90 mL	195°C max
Dry point	196°C max

NOTE 4—While use of other solvents, such as tetrahydronaphthalene or xylene, may sometimes be advantageous, they will generally yield different values for solution viscosities.

6.1.3 Immediately after redistillation of the decahydronaphthalene, add 0.2 % Tetrakis [methylene 3-(3',5'-di-tert-butyl-4'-hydroxyphenyl) propionate] methane to inhibit oxidation during the viscosity determination.

6.2 *Heat Transfer Medium*—Any liquid heat transfer medium that will not appreciably affect the accuracy of the test may be used. Care should be exercised in using fluids that discolor or smoke with prolonged heating.

NOTE 5—Silicone heat-transfer fluids are adequate for this use<sup>5</sup>.

## 7. Procedure

7.1 *Decahydronaphthalene Preparation*—Add 0.2 % weight/volume stabilizer (Irganox 1010 or equivalent), to the decahydronaphthalene, stir to dissolve, filter through fast filter paper, and store in a clean amber bottle.

7.2 *Cleaning the Viscometer*—Clean the viscometer thoroughly as follows: fill with cleaning solution, let stand overnight, empty and rinse several times with distilled water, then rinse with acetone and purge with dry nitrogen.

<sup>4</sup> Glassware used in this test method should be tested in accordance with the procedures described in the National Institute of Standards and Technology Circular No. C-434, "Testing of Glass Volumetric Apparatus," and should not exceed the limits of accuracy set forth in the circular.

<sup>5</sup> The silicone fluids available from the Dow Corning Corp., Midland, MI, or from the Union Carbide Corp., Linde Silicones Div., New York, NY, have been found satisfactory for this purpose.

7.3 *Solution Preparation*—Weigh 20 to 30 mg of the UHMWPE (see **Note 6**) into the Erlenmeyer flask. Add the decahydronaphthalene at room temperature, using a burette to measure in milliliters a volume equal to 4.5 times the UHMWPE weight in milligrams (for example, 26 mg of UHMWPE and 117 ml of decahydronaphthalene). Heat the flask, with stirring, to 150°C ± 2°C. Continue stirring at 150°C ± 2°C for 1 h, with the flask lightly stoppered. Examine the flask to see if undissolved UHMWPE remains. If so, heat an additional 15 min and reexamine. Heat for an additional 15 min, if necessary, but do not heat longer than 2 h total.

#### 7.4 *Viscosity Measurement:*

7.4.1 Place the clean viscometer into the constant-temperature bath. Fill the viscometer with decahydronaphthalene, and allow it and the solvent to equilibrate at 135 ± 0.1°C. Measure the flow time of the solvent,  $t_o$ , three times, and average the results. The viscometer may have to be cleaned if replicate measurements of flow time differ by more than 0.3 s (see 7.4.3). Remove the decahydronaphthalene from the viscometer with vacuum and thoroughly rinse the viscometer with xylene at 110 to 120°C or decahydronaphthalene at 135°C. Remove the xylene or decahydronaphthalene with vacuum and aspirate dry air or nitrogen to dry the viscometer (2 or 3 min). It is essential that the viscometer be completely dry.

7.4.2 Transfer the hot (150°C) polymer solution to the viscometer with sufficient quantity to fill to the mark (see **Note 7**), allow to equilibrate at 135°C ± 0.1°C, and measure the flow time of the solution,  $t_s$ , in triplicate, and average the results.

7.4.3 Between uses, clean the viscometer by rinsing with hot xylene or decahydronaphthalene, drying with air or nitrogen, and storing filled with hot decahydronaphthalene. If solvent flow time changes by more than 0.3 s, or if difficulty is encountered in obtaining reproducible flow times with any solution or solvent, clean the viscometer with cleaning solution as described in 7.2.

**NOTE 6**—If the UHMWPE is suspected to contain moisture or other volatiles, it shall first be dried in a vacuum oven at 60°C for 2 h.

**NOTE 7**—Filling of the viscometer is made easier by the use of a glass funnel warmed with a heating mantle. This helps to prevent the UHMWPE solution from precipitating.

## 8. Calculation

8.1 Calculate the relative solution viscosity as follows:

$$\eta_r = (t_s - k/t_s)/(t_o - k/t_o) \quad (1)$$

where:

$k$  = kinetic energy correction constant for the particular viscometer used,

$t_s$  = flow time of solution at 135°C (average of three measurements), and

$t_o$  = flow time of pure solvent at 135°C (average of three measurements).

## 9. Report

9.1 Report the following information:

9.1.1 Complete identification of the material tested including type, source, manufacturer's code numbers, and trade name.

9.1.2 Conditioning procedure used, if any.

9.1.3 The relative viscosity (viscosity ratio) of one or more concentrations, depending on whether it is desired to obtain relative, inherent, or intrinsic viscosity (viscosity ratio, logarithmic viscosity number, or limiting viscosity number) (see **Appendix X4**).

9.1.4 The intrinsic viscosity (limiting viscosity number), when desired, to three significant figures (see **Appendix X4**).

## 10. Precision and Bias

10.1 **Table 1** is based on a round robin conducted in 1997 in accordance with Practice E691, involving two materials tested by six laboratories. One laboratory distributed the two commercial UHMWPE samples chosen for the evaluation. Each laboratory obtained six test results for each material.

**TABLE 1 Relative Solution Viscosity**

Material	Average	$S_r^A$	$S_R^B$	$r^C$	$R^D$
UHMWPE No. 1	1.454	0.015	0.049	0.042	0.136
UHMWPE No. 2	1.653	0.017	0.048	0.047	0.136

<sup>A</sup> $S_r$  = within-laboratory standard deviation for the indicated material. It is obtained by first pooling the within-laboratory standard deviations of the test results from all of the participating laboratories.

$$S_r = [ \{ (s_1)^2 + (s_2)^2 \dots + (s_n)^2 \} / n ]^{1/2}$$

<sup>B</sup> $S_R$  = between-laboratory reproducibility, expressed as standard deviation:

$$S_R = [ S_r^2 + S_L^2 ]^{1/2}$$

where  $S_L$  = standard deviation of laboratory means.

<sup>C</sup> $r$  = within-laboratory critical interval between two test results = 2.8 ×  $S_r$ .

<sup>D</sup> $R$  = between laboratories critical interval between two test results = 2.8 ×  $S_R$ .

NOTE 8—**Caution:** The explanations of  $r$  and  $R$  (10.1.1-10.1.4) 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 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 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 laboratories). The principles of 10.1.1-10.1.4 would then be valid for such data.

10.1.1 *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 from testing one sample for each test result, then the following applies:

10.1.2 *Repeatability*— $r$  is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory. Two test results shall be judged not equivalent if they differ by more than the  $r$  value for the material.

10.1.3 *Reproducibility*— $R$  is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories, not necessarily on the same day. Two test results shall be judged not equivalent if they differ by more than the  $R$  value for that material.

10.1.4 Any judgement in accordance with 10.1.2 and 10.1.3 would have an approximate 95 % (0.95) probability of being correct.

10.2 *Bias*—There are no recognized standards by which to estimate bias of this method.

## 11. Keywords

11.1 dilute solution viscosity; ethylene polymers; inherent viscosity (logarithmic viscosity number); intrinsic viscosity (limiting viscosity number); reduced viscosity (viscosity number); relative viscosity; specific viscosity; viscosity ratio

## APPENDIXES

(Nonmandatory Information)

### X1. PROCEDURE FROM PREVIOUS VERSION (D1601 – 86 (1991)<sup>(\*)</sup>) DATING BACK TO 1957

X1.1 If it is desired to determine only the relative viscosity (viscosity ratio) or the inherent viscosity (logarithmic viscosity number), weigh one specimen of 0.18 to 0.22 ± 0.0002 g and transfer it quantitatively to a 100-mL volumetric flask which has been purged with nitrogen.

NOTE X1.1—Usually the determination of relative or inherent viscosity (viscosity ratio or logarithmic viscosity number) may suffice.

<https://standards.iteh.ai/catalog/standards/sist/c06931ba-f38-4d5b-a519-27d6311f52fd/astm-d1601-12>

X1.2 To determine the intrinsic viscosity (limiting viscosity number), weigh four specimens of the following approximate weights to ± 0.0002 g:

Specimen 1	0.09 to 0.11 g
Specimen 2	0.18 to 0.22 g
Specimen 3	0.28 to 0.32 g
Specimen 4	0.38 to 0.42 g

NOTE X1.2—The intrinsic viscosity (limiting viscosity number) may also be measured using successive dilutions of only one, or duplicate, solutions. This has the advantage of requiring that only one polymer specimen be weighed. Dilutions to the solution of this specimen in the viscometer are a time-saving device. The technique is as follows: Weigh one, or a duplicate, specimen of 0.38 to 0.42 g to an accuracy of ±0.0002 g and transfer it quantitatively to a 100-mL volumetric flask, which has been purged with nitrogen, using a funnel and washing the watch glass and funnel down with solvent. Place the specimen into solution and measure the relative viscosity (viscosity ratio).

Separate dilutions of the original solution to desired concentrations can be made by adding solvent at 135°C in precisely measured quantities to the original solution in the viscometer and the relative viscosity (viscosity ratio) at each dilution tested before the next dilution is made. For example, the following technique might be used.

*First Dilution*—Add exactly 5 mL of solvent at 135°C to the filter stick of the viscometer and calculate the solution concentration from the known weight of solute and total volume of solvent in the viscometer. (This concentration should be approximately 0.3 g/100 mL.) Mix the solution in the viscometer thoroughly by pulling it into the viscometer bulb and back again three times. Determine the relative viscosity (viscosity ratio) at this concentration.

*Second Dilution*—Add exactly 10 mL of solvent at 135°C and calculate the concentration, as above. (This concentration should be approximately 0.2 g/100 mL.) Determine the relative viscosity (viscosity ratio) at this concentration.

Volumetric limitations of the Ubbelohde viscometer require that approximately 15 mL of solution be added to the instrument for the first determination of relative viscosity (viscosity ratio). Also, the viscometer will not hold and properly mix liquid volumes very much in excess of 30 mL. Hence, use of the above procedure will provide only three values of relative viscosity (viscosity ratio) from which intrinsic viscosity (limiting viscosity number) can be determined. Other dilution techniques may be used to obtain four or more values of relative viscosity (viscosity ratio), but the range of solute