The Characterization of Linear Polyethylene SRM 1475. IV. Melt Flow Rate.

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The melt flow rate of SRM 1475 was determined to be 2.07 g/10 min at 190 °C under a load of 325 g by a method similar to procedure A of ASTM method D 1238–65T. This value is the average of determinations on 42 samples with a standard deviation of a single measurement of 0.040 g/10 min, and a range of 1.991 g/10 min to 2.132 g/10 min.

Key words: Extrusion plastometer; load; melt flow rate; orifice; orifice die; preliminary extrudate; test extrudate.

1. Introduction

Melt flow rate is widely used in polymer technology as a product specification since this value, which includes a statement of the load and temperature under which it is obtained, gives an indication of the processing properties of the polymer. The value of melt flow rate is expressed as the mass of polymer melt pushed from the heated cylinder of the extrusion plastometer through its precision bore orifice by its piston in a period of time, the standard units of the value being grams per ten minutes (g/10 min).

In this paper, we report the determination of melt flow rate for linear polyethylene SRM 1475. Use of the same measurements to investigate the uniformity of the material is described elsewhere [1].[1]

2. Experimental Procedure

2.1. Instrument and Method

The melt flow rate determinations were made with a commercial extrusion plastometer,[2,3] by a method similar to Procedure A described in "Tentative Method of Measuring Flow Rates of Thermoplastics by Extrusion Plastometer," ASTM Designation: D 1238–65T.[4]

The dimensions of the plastometer cylinder, piston assembly and orifice, and the combined masses of the piston and passenger weight were found to comply with the instrument specifications described by the ASTM method.

In compliance with the ASTM method, the temperature indicator of the extrusion plastometer was calibrated with respect to a standardized iron-constantan thermocouple mounted in an undisturbed column of polymer melt in the plastometer cylinder. The thermocouple junction was fixed at the axis of the cylinder, 12.7 mm above the top surface of the orifice die in the bottom of the cylinder. An equilibrium temperature reading of 189.9 °C was observed on the mercury column thermometer in the cylinder while the thermocouple indicated the equilibrium temperature of the polyethylene melt to be 190.0 °C at the prescribed calibration point. Thus, prior to each series of flow rate determinations, the cylinder of the extrusion plastometer was brought to thermal equilibrium with a constant reading of 189.9 °C on the mercury column thermometer.

Further study of the temperature at other distances up to 48 mm above the orifice, under conditions required by the calibration procedure, indicated that the temperature was uniform above the 12.7 mm height in the undisturbed melt column. However, the temperature of the melt 1 mm above the orifice was 0.7 deg lower than the temperature of the melt 12.7 mm above the orifice. This thermal gradient at the bottom of the undisturbed melt column is probably at least partially erased in an actual flow rate determination by the downward flow of the piston-driven melt through the orifice.

A separate study of the effect of temperature indicated that the flow rate of SRM 1475 was not significantly altered by a temperature change of 1 deg from the specified test temperature, 190 °C.

The orifice die was found to have a bore diameter of 2.096 mm, well within the ASTM specification tolerance limits. Bore diameter tolerance gauges were used to test the bore of the orifice die frequently between series of flow rate determinations. According to this test, no detectable change occurred in

[1] Figures in brackets indicate the literature references at the end of this paper.

[2] Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the material or equipment identified is necessarily the best available for the purpose.

[3] Model 3, Tinius Olsen Testing Machine Company, Willow Grove, Pennsylvania 19090.

[4] Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

the bore diameter during the entire course of the flow rate study.

2.2. Procedure

Because of the relatively high fluidity of the melt of the linear polyethylene, the light load of conditions D of the ASTM method was chosen for the flow rate determination. Conditions D of the ASTM method specify the temperature of the melt to be 190 °C, and the load (combined apparent masses of piston assembly and passenger weight) to be 325 grams.

In accordance with the ASTM procedure, each sample was first preheated in the plastometer for 6 minutes, and the preliminary extrudate cut from the bottom of the orifice. Test segments were then cut from the extrudate at 1 minute intervals for 11 minutes, and were examined for bubbles as they emerged from the plastometer. The ASTM procedure calls for the use of bubble-free samples obtained during the first 3 minutes. We found, however, that it was virtually impossible to obtain bubble-free samples of SRM 1475 within 3 minutes. Attempts were therefore made, as suggested in the ASTM procedure, to eliminate the bubbles by manually forcing out some of the melt during the preheat period. These attempts were unsuccessful and this modification was therefore not employed. Among the first five segments collected, however, at least two were completely free of bubbles, and generally one or two additional segments contained only bubbles so few and small that their individual flow rate values were not distinguishable from the values for the completely bubble-free segments. The extrudate from the fourth through the eleventh 1 min intervals contained no bubbles. In order to obtain measurements on bubble-free materials while staying as close as possible to the conditions specified by the ASTM procedure, we therefore determined flow rate for the certificate from the bubble-free segments obtained during the first 5 min. In order to investigate the effect of exceeding the prescribed time of 3 min, we also determined the flow rate from all the bubble-free segments obtained during the entire 11 min interval. The results obtained are described in the following section.

It was found unnecessary to apply the severe orifice-cleaning techniques, described in the ASTM method, between flow rate determinations of this particular polyethylene. It was found satisfactory to ram out the residual polyethylene with a soft copper wire of diameter nearly as large as that of the orifice bore, while the die was still hot. No effect on the flow rate values could by detected when this technique was applied between determinations.

3. Results and Discussion

Flow-rate measurements were obtained on a total of 42 samples, taken from thirteen different regions of the total supply of SRM 1475. As described elsewhere [1], no variation could be found from region to region. The results for the 42 samples were therefore pooled. The mean flow rate, based on segments obtained during the first 5 min as described in the preceding section, was found to be 2.07 g/10 min, with a sample standard deviation of 0.040 g/10 min, a range of 1.991 to 2.132 g/10 min, and a sample standard deviation of the mean of 0.0062 g/10 min. The mean value and the sample standard deviation of the mean are the values reported on the certificate for SRM 1475.

Measurements on segments collected during the entire 11 min period, well beyond the 3 min limit specified in the ASTM procedure, yielded a mean flow rate of 2.06 g/10 min, with a sample standard deviation of 0.038 g/10 min and a range of 1.987 to 2.142 g/10 min. The close agreement between the two calculations in mean value, standard deviation, and range suggests that either a longer preheat period or a longer measurement period could be used for this material without affecting the observed flow rate.

4. References

[1] Hoeve, C. A. J., Wagner, H. L., and Verdier, P. H., J. Res. Nat. Bur. Stand. (U.S.), NBS **76A**, (Phys. and Chem.), No. 2, 137–140 (Mar.–Apr. 1972). Paper I of this series.

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