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Abstract

The melt flow rate of Standard Reference Material (SRM) 1474c, a linear polyethylene with narrow molar mass distribution (MMD) ethylene-hexene copolymer resin, was determined to be 5.04 g/10 min at 190 °C under a load of 2.16 kg using the ASTM Method D1238-23a, *Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer*. The average is a result from 30 determinations on samples with a standard deviation of 0.016 g/10 min. The overall expanded uncertainty, including type A and type B uncertainties, is estimated as 0.20 g/10 min. This document describes details of the measurement and analysis.

Keywords

extrusion plastometer; melt flow index; melt flow rate; pellets; polyethylene; resin; thermoplastic

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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 [1-4]. The value of melt flow rate is expressed as the mass of polymer melt extruded from a heated cylinder of an 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). Measurements are carried out in accordance with a protocol that is described in ASTM D1238-23a, *Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer* [5].

Here the melt flow rate certification of Standard Reference Material (SRM) 1474c, a polyethylene resin, is described. SRM 1474c is the designated successor to SRM 1474b, which is the successor to SRM 1474a [6]. SRM 1474c is from the same source material as SRM 1474b.

The flow rate obtained with the extrusion plastometer is not a fundamental polymer property. It is an empirically defined parameter critically influenced by the physical properties and molecular structure of the polymer and the conditions of measurement. In order that measurements are meaningful and comparable across laboratories, measurements must be made in accord with a standard procedure. For SRM 1474c, measurements are carried out following ASTM D1238-23a. Within this standard, Procedure A at a temperature of 190 °C and a load of 2.16 kg was utilized.

2. Material and Bottling

The base material, as reported by the supplier, is a linear polyethylene with narrow molar mass distribution (MMD) ethylene-hexene copolymer resin. The density reported by the supplier is 0.948 (g/cm^3). The material was procured in two 25 kg bags in April 2002. The material was received in pellet form. The bag from which SRM 1474c was produced was kept sealed at The National Institute of Standards and Technology (until 2025). While in storage, the material was maintained at room temperature. The pellets were dry mixed, bottled and numbered 1 through 414. Fifteen of these units were chosen by stratified random selection for homogeneity and certification studies, as described below.

A unit of SRM 1474c consists of approximately 60 g of uncolored polyethylene pellets in an amber glass bottle.

3. Instrumentation

Melt flow measurements were performed with an extrusion plastometer. A common alternative name for an extrusion plastometer is a melt flow indexer, or melt indexer. Critical geometrical features, dimensions and tolerences are described in ASTM D1238-23a.

4. Calibrations, Tolerances and Cleaning

Temperature, mass and geometrical tolerances are prescribed by ASTM D1238-23a and were followed herein. The temperature calibration of the extrusion plastometer cylinder was conducted by the instrument vendor, utilizing the method that is prescribed by ASTM D1238-23a, using a NIST traceable resistance temperature detector (RTD). Mass measurements of the piston, foot and load were performed via balances that were calibrated via NIST traceable procedures. The combined mass was within the ASTM D1238-23a prescribed tolerance of 0.5 % of total mass, or 0.01 kg. The diameter of the cylinder bore was determined by the instrument vendor - the resulting measurements were in compliance with the tolerance of this specification described in the aforementioned ASTM method. Dimensional measurements of all dies and feet were performed utilizing a micrometer calibrated by NIST traceable procedures. All the above measurements and calibrations were carried out within one year of the melt flow measurements. Prior to each measurement, the dimension of the inner bore of the die was checked with a Go/No Go gauge, per ASTM D1238-23a specifications with a micrometer traceable to NIST.

Rigorous cleaning of the bore was conducted after each measurement using cotton gun cloth. Cleaning of the foot, die, piston was conducted via brass wire brush. Satisfactory cleaning of the bore and of the outside surfaces of the die were confirmed by dropping the die into the bore and checking that it falls freely to the base of the bore. Cleanliness of the foot was ascertained visually and by confirmation of unhindered motion of the piston-foot assembly through the bore upon full manual insertion and retraction.

Proper vertical extrusion bore alignment was confirmed on a daily basis, before any melt flow measurements were conducted, via a circular level that was mounted on the inserted piston rod, per ASTM D1238-23a specifications.

5. Measurement Procedure

For each of the fifteen selected bottles, two measurements were carried out for a total of 30 measurements. Six measurements were made each day. For any given bottle, both measurements were made on the same day. The sequence of numbered charges taken from the bottled samples for extrusion was randomized according to a procedure described by Natrella [7].

This nested measurement approach (bottle nested within day) allows for the separate estimation of a day effect and bottle effect as described in the nested random effects model. Since the particular days and bottles sampled are not of direct interest, but rather represent a larger population for which we are interested, a random effects model (vs. fixed effects) is appropriate.

Measurements were conducted between May 20 to 30, 2025.

The melt flow rate of SRM 1474c samples was determined by ASTM D1238-23a, Procedure A, a manual procedure described in section 10. The melt flow rate was determined at (190.0) °C using a load of (2.16) kg , a standard condition for polyethylene (This test condition is sometimes abbreviated as 190/2.16). Prior to daily measurements, the cylinder was allowed sufficient time to heat up and for the temperature to stabilize to (190.0 ± 0.1) °C.

Before each measurement and prior to the pellet loading, the die and the piston-foot assembly were inserted into the heated bore for a period of at least 15 min. A (5.80 $\pm$ 0.01) g charge of pellets was then loaded into the bore, taking care to pack the pellets to minimize air bubbles. The load was applied - as described by ASTM D1238-23a. Following load placement, a pre-heat period of duration (7.0 $\pm$ 0.5) min occured during which molten plastic extrudes from the orifice. This extrudate from the pre-heat period was discarded.

The mass of the charge, (5.80 $\pm$ 0.01) g, was used because it was found through trial and error to allow the piston land to reliably translate to a point 46 $\pm$2 mm from the top of the die at the end of the pre-heat period, as ascertained by scribe marks on the piston. Provided that the piston translated this amount during the pre-heat period, three timed extrudate test segments were then cut at 1.0 min intervals. After the third timed test extrudate segment was cut, the remaining melt in the cylinder was purged and discarded. Following ASTM D1238-23a only the first timed extrudate was used in the final data analysis. Following each measurement, the piston, bore, foot, and die were cleaned as described previously. Tools of brass and cloth, considerably less hard than steel, were applied in the cleaning process.

In practice, obtaining a clean extrudate cut can be problematic as the extruded polymer may make contact with a hot surface of the underside of the extrusion plastometer, causing unwanted adherence and preventing an accurate extrudate mass measurement. It was found that using a simple hair tweezer to cut and pull off the extrudate allowed for acceptable cuts nearly every time.

6. Data Analysis

The data consists of measurements made from the 15 randomly selected bottles (of 414 available bottles) of SRM 1474c. Figure 1 plots the data in run order. This plot does not indicate any drift in the data by day.

Figure 2 plots the data by bottle. This plot indicates there is some between bottle variability.

Figure 3 generates a block plot with replication as the primary factor. This plot indicates that replication is not a significant factor (six bottles have higher values for the first replication, seven bottles have higher values for the second replication, and two bottles are essentially equal for the replications).

6.1. Consensus Means Analysis

Based on the above plots, we can generate a consensus means analysis with bottle as the laboratory factor. Figure 4 shows the consensus means plot.

This plot shows that the DerSimonian-Laird (DSL) and Vangel-Rukhin (VR) methods give similar results. The bootstrap uncertainty is slightly more conservative than the Horn-Horn-Duncan uncertainty, so we will use that result. The DSL consensus value is $\hat{\mu}_{DSL} = 5.0394$ (g/10 min), and the associated standard uncertainty is $u_{\text{meas}} = 0.0158893$ (g/10 min) This will be used for the within-bottle and between-bottle uncertainty components.

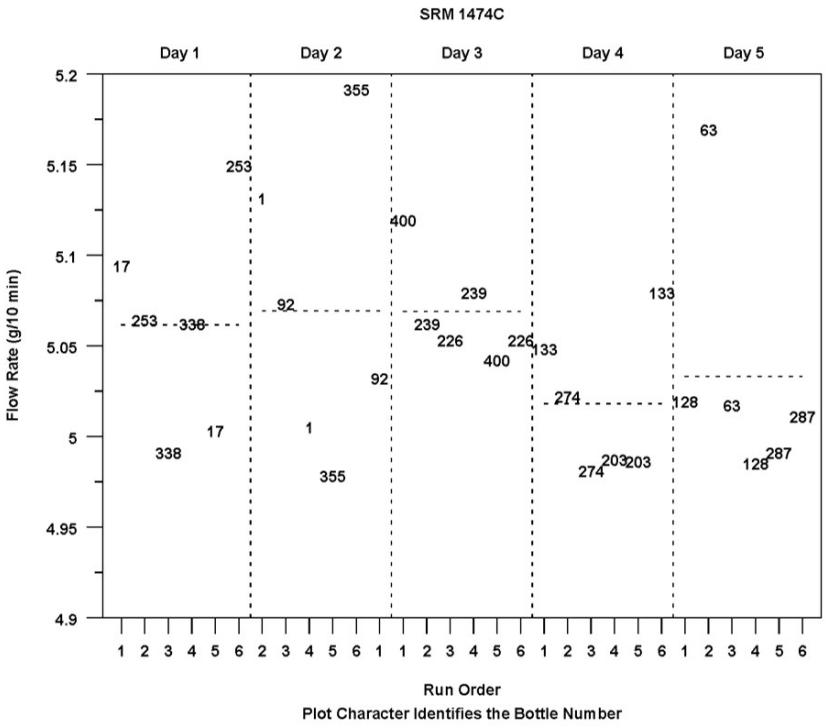

Fig. 1. Plot by Run Order. The horizontal dashed lines indicate the average flow rate for each corresponding day.

6.2. Additional Uncertainty Components

Several potential sources of systematic uncertainty have been identified.

In order to estimate the uncertainty in melt flow rate due to the allowable 0.2 °C uncertainty in the extrusion plastometer, melt flow rate measurements were conducted at temperatures ranging from (187 to 193) °C. The slope of melt flow rate versus temperature was determined by linear regression. It was ascertained that a temperature uncertainty of 0.2 °C leads to a melt flow rate uncertainty of $u_{\text{temp}} = 0.016$ (g/10 min) for this material under the current load and temperature conditions.

A robust discussion of measurement uncertainties for SRM 1474b is contained in a previous report [6]. Uncertainties due to the weighing of samples and timing of extrudate cuts were considered and found to be negligible compared to other sources of uncertainty.

The largest source of uncertainty stems from laboratory to laboratory variation based on round robin testing, as described in ASTM-D1238-23a [5]. Table 6 in this document provides an uncertainty of $u_{\text{ASTM}} = 0.103$ (g/10 min) for the case of a melt flow rate average of $\hat{\mu}_{ASTM} = 5.35$ (g/10 min). The relative standard deviation stemming from the interlaboratory study was computed by mul-

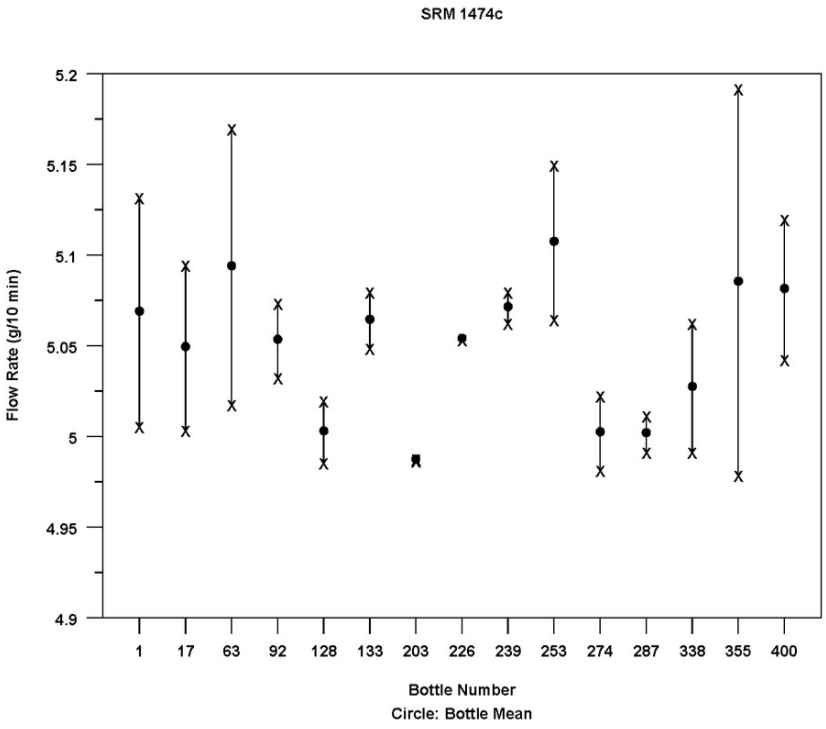

Fig. 2. Plot by Bottle. The round marker indicates the average for each bottle. The 'X' markers indicates the values of the two individual measurements made on each bottle.

tiplying the ASTM melt flow rate uncertainty by the ratio of melt flow values:

$$u_{\text{lab}} = u_{\text{ASTM}} * \frac{\hat{\mu}_{DSL}}{\hat{\mu}_{ASTM}} = 0.097\,(\text{g/ 10 min}) \tag{1}$$

6.3. Combined Uncertainty

The combined uncertainty was computed with two different methods. The first method uses

$$u_c = \sqrt{u_{\text{meas}}^2 + u_{\text{temp}}^2 + u_{\text{lab}}^2} \tag{2}$$

and substituting in the determined quantities, we have:

$$u_c = \sqrt{0.0158893^2 + 0.016^2 + 0.09702^2}\ (\text{g/10 min}). \tag{3}$$

The resulting expanded (k = 2) combined uncertainty is $U = ku_c = 0.19921$(g/10 min).

The second method uses a Monte Carlo simulation. The steps involved include:

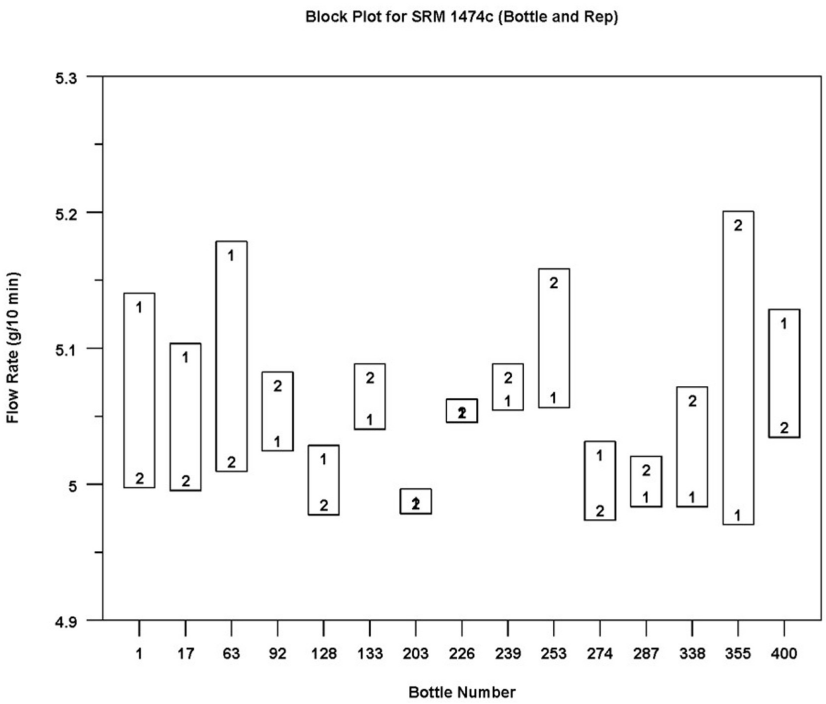

Fig. 3. Block Plot with Replication as Primary Factor

1. Generate one million sets of sample means over 15 bottles and the associated observed standard errors.
2. For each of the one million samples, calculate the DSL estimated consensus value.
3. Calculate a 95 % coverage interval based on the one million DSL values by ordering them and finding the 2.5 percentile and 97.5 percentile.

The completion of Step 1 is based on the following mixed effects model

$$y_{ij} = \mu + \beta_i + \varepsilon_{ij}, \quad \beta_i \sim N(0, \sigma_\beta^2), \quad \varepsilon_{ij} \sim N(0, \sigma_\varepsilon^2), \text{ for } \begin{cases} i = 1, \ldots, 15 \\ j = 1, 2 \end{cases} \tag{4}$$

where i denotes the bottle, j the replicated measurement, β_i is the random effect associated with a particular bottle, and ε_{ij} is the measurement error.

To generate the one million sample means we use the following equation:

$$\bar{x}_{ij} = \hat{\mu}_{DSL} + u.lab_i + u.temp_i + u.bot_i + u.meas_{ij}, \quad i = 1, \ldots, 1 \times 10^6; j = 1, \ldots, 15; \tag{5}$$

where $\hat{\mu}_{DSL} = 5.0394$ (g/10 min) is the consensus value of the observed data, $u.lab_i$ is the uncertainty component due to laboratory/instrument variability, $u.temp_i$ is the uncertainty component due to temperature, $u.bot_i$ is the between bottle uncertainty component, and $u.meas_{ij}$ is

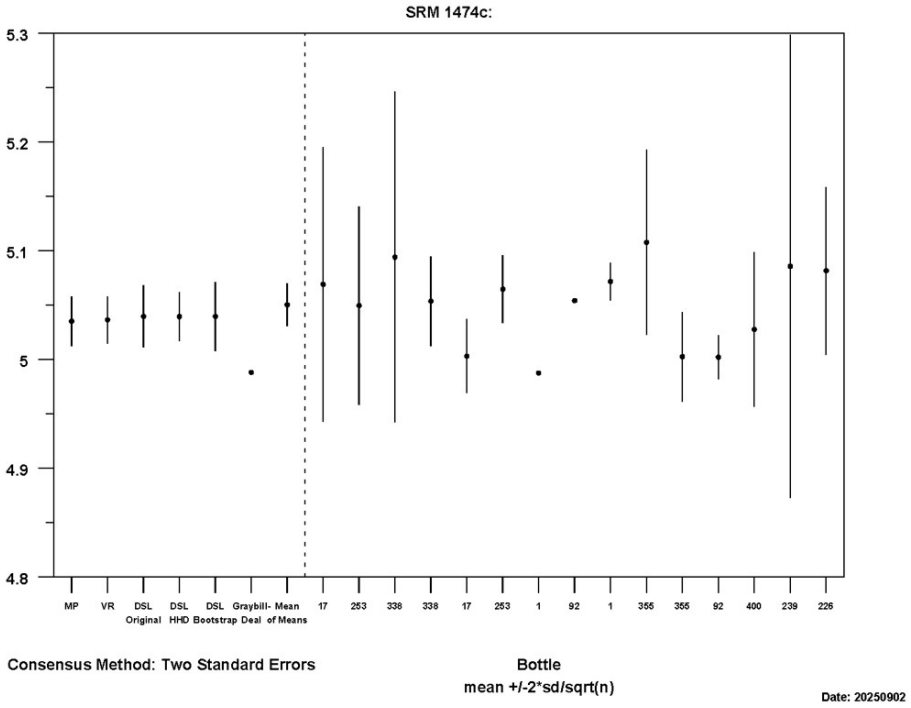

Fig. 4. Consensus Mean Plot

the measurement error associated with each individual bottle. The distributions for each of the uncertainty components are presented next:

$$u.lab_i \sim N(0, 0.0970^2); \quad u.temp_i \sim N(0, 0.016^2) \tag{6}$$
$$u.bot_i \sim N(0, \beta_i^2),$$

where $\beta_i^2 \sim A\left(\frac{m\hat{\tau}_{DL}^2+\hat{\sigma}^2}{m(n-1)}\right) - B\left(\frac{\hat{\sigma}^2}{mn(m-1)}\right)$ with $n = 15$ (the number of bottles), $m = 2$ (the number of measurements per bottle), $\hat{\tau}_{DL}^2 = 0.0003$ is the DSL estimated bottle-to-bottle variance, $\hat{\sigma}^2 = 0.0039$ is the within bottle pooled variance, $A \sim \chi_{n-1}^2$, and $B \sim \chi_{n(m-1)}^2$. Here N is shorthand for Gaussian distribution, so $N(\alpha, \beta^2)$ indicates a Gaussian distribution with a mean of α and standard deviation of β. This formulation for β_i^2 is based on the derivations from [8] on page 69:

$$u.meas_{ij} = \frac{(m-1)s_j^2}{C} \quad i = 1, \ldots, 1 \times 10^6; j = 1, \ldots, 15; \tag{7}$$

where $m = 2$ (the number of measurements per bottle), $\mathbf{s} = [0.063, 0.0455, 0.076, 0.0205, 0.017, 0.0155, 0.05, 0.0, 0.0085, 0.0425, 0.0205, 0.01, 0.0355, 0.1065, 0.0385]$ (the vector of the standard deviations of the observed bottle means), and $C \sim \chi_{(m-1)}^2$. Note that because the eighth sample

standard deviation is zero, we exclude this standard error and associated sample of means before proceeding to Step 2 above.

The 95 % coverage interval resulting from these simulations is $[4.838215, 5.242683]$ (g/10 min). To calculate the combined uncertainty from a coverage interval say, $[LB, UB]$, we find $\max(UB - \hat{\mu}_{DSL}, \hat{\mu}_{DSL} - LB)$. This simulation resulted in a combined uncertainty of 0.2032729 (g/10 min) which is in good agreement with the first method.

7. Summary - Certified Melt Flow Value

This material is certified for melt flow rate by methods described here using Procedure A of ASTM D1238-23a [5] at 190.0 $\pm$0.1 °C using a load of 2.16 kg. Under these conditions, the certified melt flow rate for this material is as follows:

Melt Flow Rate (FR) = 5.04 g/10 min $\pm$ 0.20 g/10 min

The measurand is the flow rate. Metrological traceability is to the SI units for mass and time (expressed as grams per ten minutes). Fifteen units of the SRM were measured in duplicate by NIST according to the procedures of ASTM D1238-23a [5]. The certified melt flow rate is the weighted mean of the NIST measurements estimated using a Gaussian random effects model [8–10] and the DSL procedure [11,12]. The associated measurement uncertainty was evaluated by an application of the parametric statistical bootstrap, consistent with the ISO/JCGM Guide and its Supplement 1 [13–16]. The expanded uncertainty, U, is calculated as U = ku_c , where u_c is intended to represent, at the level of one standard deviation, the combined effects of the within bottle and between bottle uncertainty 0.016 (g/10 min), uncertainty due to the temperature of the plastometer cylinder 0.016 (g/10 min), and the uncertainty due to laboratory-to-laboratory variability which is calculated based on Table 6 of ASTM D1238-23a, 0.097 (g/10 min). The expansion factor, k = 2, corresponds to an approximately 95 % confidence level.

This value falls within the uncertainty limits of SRM 1474b [17].

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