NIST SPECIAL PUBLICATION 260-148

U. S. DEPARTMENT OF COMMERCE/Technology Administration
National Institute of Standards and Technology

Standard Reference Materials®

Certification of Standard Reference Material 1474a, A Polyethylene Resin

J. R. Maurey, K. M. Flynn, and C. M. Guttman

NIST
National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

NIST Special Publication 260-148

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Issued November 2003

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National Institute of Standards and Technology Special Publication 260-148
Natl. Inst. Stand. Technol. Spec. Publ. 260-148, 18 pages (November 2003)
CODEN: NSPUE2

U.S. GOVERNMENT PRINTING OFFICE
WASHINGTON: 2003

For sale by the Superintendent of Documents, U.S. Government Printing Office
Internet: bookstore.gpo.gov — Phone: (202) 512-1800 — Fax: (202) 512-2250
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Abstract

The melt flow rate of Standard Reference Material (SRM®) 1474a, a linear polyethylene (narrow MWD ethylene-hexene copolymer) resin, was determined to be 5.10 g/10 min at 190 ^{o}C under a load of 2.16 kg using the ASTM Method D 1238-00. The average results from 45 determinations on samples with a standard deviation of 0.056 g/10 min. The overall expanded uncertainty, including type A and type B uncertainties, is estimated as 0.42 g/10 min.

1.0 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 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).

This is a report on the melt flow rate certification of Standard Reference Material (SRM®) 1474a, a polyethylene resin. SRM 1474a is the designated successor to SRM 1474 the characterization of which was described in an earlier report (5). SRM 1474a is from a different material from a different supplier, but with a closely matched melt flow rate.

2.0 Experimental Procedure

2.1 Sampling and Randomization of Charge Sequence of SRM 1474a

Material for SRM 1474a came as pellets of polyethylene. The supplier of this resin identifies the resin as narrow molecular mass distribution linear ethylene-hexene copolymer with an estimated density of 0.948 g/cm^3. The NIST Standard Reference Materials Group (SRMG) blended the resin and divided the pellets into 404 units of 60 g each. Fifteen of these units were chosen by stratified random selection for homogeneity and certification studies. Three charges were taken for extrusion from each of the randomly chosen bottled samples of SRM 1474a during the course of the melt flow rate experiments. In preparation for the melt flow rate determinations, the charges to be extruded were identified by ordinal numbers. Three such charge numbers were assigned to each identified sample. The sequence of numbered charges taken from the bottled samples for extrusion was randomized according to a procedure described by Natrella (6).

2.2 Instrument Calibrations and Alignment

2.2.1 Temperature Indication

The temperature of the extrusion plastometer cylinder was measured by a mercury column thermometer of the form described in paragraph 5.7 of the ASTM method (7). Calibration of the thermometer is traceable to the Thermometry Group of the NIST Process Measurements Division. An iron-constantan thermocouple was calibrated by correlating its voltage with the scale readings of an ASTM 68 ^{o}C thermometer at 10 points from 185 ^{o}C to 194 ^{o}C, in a constant temperature oil bath. The ASTM 68 ^{o}C thermometer had been calibrated at the ice point and at 190 ^{O}C in the Thermometry Group of the NIST Process Measurements Division by means of a platinum resistance thermometer. The temperature indication by the scale of the cylinder thermometer in the extrusion plastometer was calibrated by correlating the thermometer scale readings with the temperature measured by the emf from the calibrated thermocouple with its hot junction stationed in a column of polyethylene melt in the bore, as described in paragraph 5.5.2 of the ASTM method. Thermal conductivity between cylinder and thermometer was enhanced by adding Wood's metal to the thermometer well in the cylinder. The standard uncertainty in the final temperature indication may be regarded as equal to the nominal limit of resolution on the scales of the ASTM 68 ^{o}C thermometer, and of the thermometer in the extrusion plastometer, 0.1 ^{o}C. The effect of a 0.1 ^{o}C standard uncertainty in temperature on the melt flow rate of SRM 1474a is described in the subsequent section on uncertainty analysis.

2.2.2 Metering of Plastometer Components

Many dies and piston feet were obtained from Tinius Olsen Testing Machine Co.[*] so that during any given day, a different die and piston foot was used for each melt flow determination made on that day. Methods of cleaning the dies and piston feet were designed to avoid dimensional changes (see section 3.0). The geometric dimensions of the plastometer cylinder, piston feet, and dies were all measured and found to comply with the specifications described in the ASTM method.

The diameter of the cylinder bore was determined by a Brown and Sharpe model 599-281 Intrimik inner diameter (ID) micrometer. The ID of the bore was measured from the top to the bottom at one centimeter intervals. The top diameter measurement was made at a depth of 2 cm into the bore; the bottom diameter measurement was made with the micrometer head resting on a die at the bottom of the bore. The resulting measurements varied from (0.9548 to 0.9554) cm, in compliance with the tolerance of this specification described in paragraph 5.2 of the ASTM method. The apparent mass of the nominal 2060 g load was determined in the Mass Group of the NIST Automated Production Technology Division, and found to be 2059.8 g. New detachable piston feet were used to conduct the extrusions; a different foot was used on the piston for each extrusion. The assembled piston varied in apparent mass from 100.027 g with the lightest foot attached, to 100.071 g with the heaviest foot attached. Thus the calculated combined apparent mass of piston and load varied from 2159.83 g with the lightest foot attached to the piston, to 2159.87 g with the heaviest foot attached to the piston, both limits well within the 0.5 % tolerance described in paragraph 5.4.3 of the ASTM method (7).

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

2.2.3 Alignment of Plastometer

The cylindrical axis of the bore was aligned with the gravity vector by the following plumb-line procedure. First, a die was selected as the "target" die and stationed on the structural base plate of the extrusion plastometer directly below the cylinder. Its position on the plane of the base plate was adjusted to have the axis of its bore coincide with the projection from the axis of the cylinder bore onto that plane. This was accomplished by viewing the target die through the bores of two "sighting" dies, one stationed at its operational position in the bottom end of the cylinder bore and the other stationed at the top end of the cylinder bore. The position of the target die on the plane of the base plate was adjusted until it appeared centered in the view from above the cylinder through the sighting dies in the cylinder bore.

Next a plumb-bob was suspended by a plumb-line from the axis of a die supported in the top end of the cylinder bore, with the pointer of the suspended bob extending down to a level where the pointer was almost touching the top surface of the target die. The leveling screws were adjusted until the pointer of the plumb-bob appeared to be centered over the bore of the target die.

The deviation of the plumb bob pointer was observed to be less than 1 mm from the point, which would indicate ideally vertical orientation of the cylinder bore, at the end of a pendulum length of 41 cm. Consequently, this procedure is considered to obtain alignment of the cylindrical axis of the cylinder bore with the gravity vector, with a calculated uncertainty of (1 mm/41 cm). Thus, the maximum uncertainty in terms of possible angle of displacement of the bore axis from the gravity vector is estimated as $\sin^{-1}$(1 mm/41 cm) = 0.14°. It is estimated that such a maximum displacement in angle from the gravity vector would diminish the force of the loaded piston to a level of its total mass multiplied by cos (0.14°), or (2.16 kg)(0.999997) in the present case. This factor would thus diminish the effective mass of the loaded piston by a decrement of 3×10^{-4} %, incomparably less than the (+/-)0.5 % tolerance in combined mass of piston and load described in paragraph 5.4.3 of the ASTM method. Standard uncertainties cited in this report are considered equivalent to those corresponding to a 95 % level of confidence, as explained in the subsequent section on uncertainty estimates in the melt flow rate data.

The initial bore alignment was conducted at ambient temperature in preparation for the characterization. The alignment was also occasionally tested, during the course of the characterization extrusions while the cylinder was hot, with a circular level which can be mounted atop the piston rod mounted in the bore, as described in the bore alignment section, Section 5.8 and Appendix X1 of ASTM method (7).

3.0 Melt Flow Rate of SRM 1474a

The melt flow rates of SRM 1474a samples were determined by procedure A described in Section 9 of ASTM Method D1238-00 (7). Standard test condition 190/2.16 was used. Thus the flow rate was determined at (190.0 ± 0.1) ºC using a load of 2.16 kg. The flow rate of the melt was measured by a manually operated extrusion plastometer manufactured by the Tinius-Olsen Testing Machine Co.

A 6.2 g charge of pellets was used for each extrusion. The end of the 7.0 min preheat period was marked as the beginning of timed test extrusion by making the initial extrudate cut at 7.0 min and discarding the preheat segment. (The reader should note that in previous certifications of melt flow rate a 6.0 min preheat was used. The 6.0 min preheat was allowed in ASTM D-1238 methods with a date before 1990. In the current methods a 7.0 min preheat is prescribed with a range of (+/-) 0.5 min allowed.) It was also observed that the 4 mm start section of the piston had always entered the top of the guide collar part way at the moment of the initial cut to begin collecting timed test extrudate. Three timed test extrudate segments were cut at 1.0 min intervals thereafter. After the third timed test extrudate segment had been cut, the remaining melt in the cylinder was purged and discarded. Following ASTM D1238-00 only the first timed extrudate was used in the final data analysis for certification of SRM 1474a. The piston and bore were cleaned free of the polymer at the end of each extrusion. Many dies and piston feet were obtained from Tinius-Olsen Testing Machine Co. During a given day a different die and piston foot was used for each separate charge. The dies and piston feet were then cleaned in preparation for the next day's run. Tools of brass and copper, considerably less hard than steel, were applied in the cleaning process. The use of steel tools was avoided in order to prevent changing dimensions of instrument components due to cleaning wear. Cotton gun cleaning patches and 38 caliber gun bore brushes with bronze bristles were used to clean the cylinder bore. The dies and piston feet were exposed to a vapor of boiling xylene for the final cleaning.

Two operators were used in the operation of the equipment. Each operator made measurements on six extrusions (charges) on randomly chosen days.

4.0 Data Analysis on SRM 1474a

Data from 45 charges were analyzed for the 2.16 kg load following the ASTM Method D1238-00. The average melt flow rate of SRM 1474a was found to be 5.10 g/10 min with a standard deviation of 0.056 g/10 min with 44 degrees of freedom. This standard deviation includes bottle- to-bottle, charge-to-charge, operator-to-operator and day-to-day variability. The standard deviation of the mean was calculated to be 0.0083 g/10 min. Our estimates of systematic uncertainties in the measurement will be discussed in the following sections.

5.0 Uncertainty Estimates of Melt Flow Rate Data

The measurement uncertainties encountered in the process of determining the melt flow rate were determined or estimated in compliance with the NIST policy governing the reporting of uncertainties in measurement (8). The uncertainty due to instrument variability among the results from a large population of laboratories was derived from expected precision limits tabulated in the ASTM method description (7).

5.1 Repeatability and Sampling Uncertainties

In this section, the uncertainty will be discussed in terms of variations of the measured melt flow rate arising from sampling and bottling differences.

For SRM 1474a, the standard deviation was 0.056 g/10 min for 45 melt flow rate determinations in a range from 4.95 g/10 min to 5.21 g/10 min. The standard uncertainty arising from the overall experimental extrusion repeatability is taken as the standard deviation of the mean of all the melt flow rate determinations, $u = 0.0083$ g/10 min.

5.2 Charge-to-Charge Variability Within a Bottle

As described in Section 2.1,15 bottles were selected for measurement from the original bottling. Three charges from each bottle were measured so a study of charge-to-charge variation within an individual bottle was easily performed. The mean charge-to-charge standard deviation of the melt flow rate value from charges removed from an individual bottle was 0.055 g/10 min, with a range from (0.0091 to 0.11) g/10 min. These data are not reported separately in Table 1, since this uncertainty is included in the relative standard uncertainty reported in line 1 of Table 1.

5.3 Bottle-to-Bottle Variability

The bottle-to-bottle variability was estimated from the population of all charges taken from all 15 bottles. The mean value of the melt flow rate for any bottle was found to lie within two standard deviations of the mean melt flow rate for all the charges. The mean of any bottle was not found to be significantly different at the 95 % confidence level from any other mean in the group. The bottle-to-bottle variability is not reported separately in Table 1 since this uncertainty is included in the overall standard uncertainty reported in line 1 of Table 1.

5.4 Day-to-Day Variability

Six melt flow rate experiments could be conducted in a single day. The day-to-day variability was small compared to the charge-to-charge variability. The day-to-day variability is not reported separately in Table 1, since this uncertainty is included in the overall standard uncertainty reported in line 1 of Table 1.

5.5 Operator-to-Operator Variability

Six melt flow rate determinations could be conducted in a single day. On any one day a single operator used the instrument. The operator for any given day was chosen at random. A two-way ANOVA run comparing the two operators concluded the two operators were not different. This uncertainty is not reported separately in Table 1 since it is included in the overall standard uncertainty reported in line 1 of Table 1.

5.6 Systematic Uncertainties

Obtaining a systematic uncertainty analysis of the melt flow rate is a difficult matter since the melt flow rate is not a fundamental property of the material and there is no simple relationship describing its estimation. Nonetheless an effort shall be made in this section to estimate the uncertainties from their possible contributing sources, and their contribution to the combined uncertainty to be computed.

5.6.1 Instrument Variability

As noted before, the estimates of the measurements repeatability are in line 1 of Table 1. These data reflect the repeatability of the experiments and do not reflect any instrument-to-instrument variation since only one instrument was used.

However, the results in Table 4 in ASTM D1238-00 (7) provide a means of estimating the uncertainty among the results from a large population of instruments and operators applying procedure A. For the purposes of this estimation for SRM 1474a with a melt flow rate of 5.10 g/10 min, the uncertainty data for the polyethylene using the same melt flow conditions with a melt flow rate of 2.04 g/10 min was used. The table of precision limits lists the within laboratory standard deviation of the average, S_r, and the between laboratory standard deviation of the average, S_R, in addition to other related statistical parameters, all defined in ASTM Standard E 691-99, "Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method" (9). The standard deviation of the mean of an array of average melt flow rate results from a large population of laboratories can be derived from the S_R given in the precision tables of ASTM D 1238-00 by applying their relationship with the more fundamental standard deviation of the mean described in ASTM E 691-99. Thus a standard deviation of the mean of 0.079 g/10 min was obtained for the average melt flow rate 2.04 g/10 min from its corresponding S_R listed in Table 4 of ASTM D 1238-00. Assuming the measurement has the same percent standard deviation of the mean arising from this instrument variability, the uncertainty due to instrument variability in the results from a large population of laboratories is estimated as 0.20 g/10 min. This standard uncertainty due to instrument variability is also listed in Table 1 of this report. Predicated on statistical analysis of measurements from nine laboratories in a study coordinated by ASTM Committee D-20, this is a Type A uncertainty in the nomenclature described in NIST Technical Note 1297(8).

5.6.2 Measurement Uncertainties

An effort was made to estimate the intrinsic uncertainty in the measurement. This was done by considering the uncertainties in the measured quantities (mass and time) as well as the uncertainties in the controlled quantities (temperature). The melt flow rate, F, is given by

$$F = \text{Mass}/\text{Time}$$

Thus the relative standard uncertainty in the melt flow rate, $\delta F/F$, is then obtained as the root-sum-of-squares of the uncertainties in the systematic physical factors upon which the melt flow rate depends (8).

$$(\delta F/F)^2 = (\delta m/m)^2 + (\delta t/t)^2 + (cT\ \delta T/F)^2.$$

where the relative standard uncertainty in mass of extrudate is $\delta m/m$, $\delta t/t$ is the relative standard uncertainty in timing of the extrudate cut and $\delta T/T$ term arises from the uncertainty in the temperature control and in the calibration of temperature indication. The factor cT is the sensitivity coefficient, cT=(dF/dT). The causes of these uncertainties are discussed in the next few paragraphs. These uncertainties together with the other uncertainties are given in Table 1. An estimate of the combined standard uncertainty resulting from all sources is also given in Table 1.

5.6.2.1 Weighing Uncertainty

The extrudate segments were weighed on a balance with 0.1 mg resolution and an estimated uncertainty of 0.1 mg. Replicate weighings of the segments always agreed to within 0.1 mg. Paragraph 9.12 of ASTM Method D 1238-00 instructs the experimenter to "weigh the extrudate to the nearest 1 mg when cool."

The extrudate segments were routinely weighed within one hour after having been cut, in compliance with the instruction in paragraph 9.12 of the ASTM method. Considering the hydrophobic character of polyethylene it would not be anticipated that the extrudate would accumulate moisture beyond the initial cooling stage prior to being weighed. On a few occasions during the characterization of another polyolefin, extrudate segments, which had been weighed at the end of a day, were weighed again on the following day without detecting any statistically valid change of mass within the groups. All individual changes, either positive or negative, did not exceed 0.1 mg.

The extrudate mass was about 500 mg. With an estimated overall weighing uncertainty of 0.1 mg from the above sources, the relative standard uncertainty

$$\delta m/m = 0.02\ \%.$$

Since this weighing uncertainty appears negligible in comparison with larger uncertainties from other sources, the weighing uncertainty was taken to be zero.

5.6.2.2 Timing Uncertainty

The 1 min interval ($t = 60$ s) between extrudate cuts for SRM 1474a was measured with a battery powered stopwatch having a 0.01 s resolution in time indication and an uncertainty of less than 0.05 s. Thus the extrudate cut was assumed to be timed to better than 0.1 s. Consequently, 0.1 s is taken as a practical estimate of the timing uncertainty. Hence, the relative standard uncertainty in time interval may be expressed

$$\delta t/t = 0.1\ \text{s}/60\ \text{s} = 0.17\ \%$$

The timing uncertainty is taken as zero, since it appears negligible in comparison with larger uncertainties from other sources.

5.6.2.3 Temperature Uncertainty

As described in Section 2.2.1, the extrusion cylinder temperature was measured by a mercury column thermometer of the form described in paragraph 5.7 of the ASTM method D1238-00. The uncertainty of the thermometer is certified to be within the tolerances of the ASTM method, by comparison to standards traceable to NIST. The standard uncertainty in the temperature indication calibration is less than 0.1 ºC.

Paragraph 5.7 in the ASTM method acknowledges that the temperature in the thermometer well may not necessarily be the temperature of the polymer melt at the calibration point in the bore. This is due to the steady state heat transfer gradients in the plastometer cylinder. Thus, the thermal profile of an undisturbed column of polyethylene melt was scanned along the cylindrical axis of the cylinder bore while the temperature was maintained at 190.0 ºC at the calibration point in the melt column. This experiment was conducted in another extrusion plastometer during an earlier determination of the melt flow rate of SRM 1475. The column of melt was held stationary by plugging the flow. The temperature in the stationary melt column was measured with a thermocouple hot junction stationed at different heights above the top surface of the die, along the cylindrical axis of the bore. Throughout the experiment the reading of the mercury column thermometer remained at (190.0 ± 0.1) ºC. The results are listed in Table 2.

Inspection of the tabulated results indicates that the departure of melt temperature from the measured cylinder temperature is within 0.1 ºC at any location in the melt column from 12 mm above the die upward. There is a 0.7 ºC drop in temperature between the 12 mm and 1 mm levels above the die. This temperature drop is probably at least partially erased by the downward flow of melt during an extrusion. Thus only part of that total drop is taken as the uncertainty in the temperature. The 0.4 ºC temperature drop determined at the bottom end of the melt column adjacent to the die, observed in Table 2, was taken as an estimate of the temperature uncertainty due to possible thermal gradients in the melt column. This uncertainty is combined with the nominal standard uncertainty in temperature indication, 0.1 ºC, by root-sum-of-squares, and the result rounded up to 0.5 ºC, as an estimate of maximum expected standard uncertainty in temperature, δT, due to temperature indication and to possible gradients in the thermal profile of the melt column.

The effect of small variations in temperature on the apparent melt flow rate was determined during the characterization of SRM 1473a reported earlier (10). In that study, the effect of temperature variation on melt flow rate was determined by conducting a set of five extrusions at 188.4 ºC, and another set of five extrusions at 191.5 ºC. The two sets of extrusions at the different temperatures were conducted with charges of polyethylene resin all taken from the same bottle of SRM 1473a.

From the resulting average melt flow rates from the first timed extrudate segments of those extrusions, and from that at 190 ºC it was estimated for SRM 1473a

$$(dF/dT) = 0.040 \text{ g/10 min / ºC}$$

For the purpose of estimating uncertainty, this value of (dF/dT) was taken as the quantitative expression for the temperature dependence and the sensitivity coefficient, cT, of the melt flow rate of SRM 1474a at temperatures near 190 ^{o}C.

Considering the standard uncertainty in temperature, δT = 0.5 ^{o}C, this result yields an estimate of 0.020 g/10 min for the uncertainty in melt flow rate of SRM 1474a due to uncertainty in temperature.

5.7 Combined Standard Uncertainty

The combined standard uncertainty (10) for the melt flow rate, u_c, is obtained as the root-sum-of-squares of component uncertainties from all sources. The combined standard uncertainty computed thus from the sources discussed in the preceding paragraphs is u_c = 0.21 g/10 min.

5.8 Expanded Uncertainty

With the object of presenting an uncertainty consistent with the reproducibility's tabulated in the ASTM method, the combined standard uncertainty from Table 1 of this report is expanded to the form of a 95 % confidence interval estimate. This is accomplished by applying a coverage factor of 2 to the combined standard uncertainty to obtain an expanded uncertainty. The resulting expanded uncertainty is 0.42 g/min for the melt flow rate of SRM 1474a.

6.0 Conclusions of Melt Flow Rate Study

The melt flow rate of SRM 1474a was found to be 5.10 g/10 min, with a standard deviation of an average single measurement of 0.056 g/10 min, and a standard deviation of the mean of 0.0083 g/10 min. The combined standard uncertainty is 0.21 g/10 min, and the expanded uncertainty is 0.42 g/10 min.

Acknowledgment

The authors gratefully acknowledge Bruno Fanconi of the NIST Polymers Division for the technical counsel and direction and Stefan Leigh of NIST Statistical Engineering Division for guidance in the statistical analysis.

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Tables

Table 1 Estimates of Uncertainties in Melt Flow Rate of SRM 1474a Polyethylene Under Condition 190/2.16

Sources of standard uncertainty	u_i g/10 min	Type [c]
1. Due to experimental repeatability	0.056	A
2. Due to instrument variability as estimated from precision tables in ASTM D 1238-00	0.20	A
3. Due to weighing	<0.001	B
4. Due to timing [a]	<0.001	B
5. Due to temperature	0.020	B
Combined standard uncertainty[b], u_c	0.21	
Expanded uncertainty, $U = 2u_c$	0.42	

a. Sensitivity coefficient, cT = dF/dT, for variation of flow rate in response to small changes in melt temperature.

b. The combined standard uncertainty is computed by root-sum-of-squares of the component uncertainties (8).

c. Type of uncertainty(8)

Type A uncertainties are evaluated by statistical methods.
Type B uncertainties are evaluated by other means.

Table 2 Variation of Temperature with Height in Undisturbed Melt in Cylinder Bore

Height Above Die (mm)	Melt Temp. (ºC)
48	190.09
36	189.93
24	189.97
12	189.94
1	189.23

Certificate of Analysis

Standard Reference Material® 1474a

Polyethylene Resin

This Standard Reference Material (SRM) is intended for use in calibration and performance evaluation of instruments used in polymer technology and science for the determination of the Melt Flow Rate using ASTM Method D1238-00. A unit of SRM 1474a consists of approximately 60 g of white polyethylene pellets in an amber glass bottle.

Certified Values and Uncertainties: This material is certified for Melt Flow Rate using ASTM Method D1238-00, Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer [1] Standard Test Condition 190/2.16. The flow rate of the melt was determined at 190.0 ºC ± 0.1 ºC and a load of 2.16 kg by procedure A of the ASTM method. A manually operated extrusion plastometer was used. Under these conditions [2], the certified melt flow rate for this material is as follows:

Melt Flow Rate (FR) = 5.10 g/10 min ± 0.42 g/10 min

The uncertainty is an expanded uncertainty $U = 2ku_c$ with U determined from a combined standard uncertainty, u_c and coverage factor $k = 2$ with a level of confidence of approximately 95 %. Type A and Type B contributions to the expanded uncertainty include the standard deviation of the melt flow measurements, instrument-to-instrument variation as discussed in ASTM D 1238-00, operator dependence of the measurement, and temperature gradients in the apparatus [2]. The standard deviation for single measurement is 0.056 g/10 min, with 44 degrees of freedom [2].

Expiration of Certification: The certification of SRM 1474a is valid until **01 January 2008**, within the measurement uncertainties specified, provided that the SRM is handled in accordance with the storage instructions given in this certificate. This certification is nullified if the SRM is modified or contaminated.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Storage: The SRM should be stored in the original bottle with the lid tightly closed and under normal laboratory conditions.

Homogeneity: The homogeneity of SRM 1474a was tested by melt flow rate measurements using ASTM D1238-00. The characterization of this polymer is described in Reference 2.

The technical coordination leading to certification of this SRM was provided by B.M. Fanconi of the NIST Polymers Division.

The technical measurement and data interpretation were provided by K.M. Flynn, C.M. Guttman, J.R. Maurey, of the NIST Polymers Division.

Guidance in the statistical analysis was provided by S.D. Leigh of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.W.L. Thomas and B.S. MacDonald.

Eric J. Amis, Chief
Polymers Division

Gaithersburg, MD 20899
Certificate Issue Date: 18 March 2003

John Rumble, Jr. Chief
Measurement Services Division

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[3] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office; Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.