National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2497

Bingham Concrete Mixture for Rheological Measurements

This Standard Reference Material (SRM) is intended for use in calibrating rheometers for measuring the rheological properties of concrete. A unit of SRM 2497 consists of two 18.9 L (5-gallons) containers labeled P and A. Container P includes Limestone (12 kg) and 4 L of corn syrup. Container A includes one plastic bag each of 1 mm beads (6 kg) and 10 mm beads (20 kg).

Certified Values: A NIST certified value is a value for which NIST has the highest confidence in its accuracy and that all known or suspected sources of bias have been investigated or taken into account [1]. The certified Bingham yield stress and plastic viscosity for the paste values are given in Table 1 and are based on measurements from analyses made using a parallel serrated plate rheometer performed at NIST by a single operator and on a model developed at NIST. The fit curve for viscosity vs. shear rate is provided as a reference. The expanded uncertainties of the certified values are consistent with the NIST uncertainty policy described in the NIST Technical Note 1297 [2], and are computed using a random coefficient regression model evaluated by Monte Carlo methods.

Expiration of Certification: The certification of **SRM 2497** is valid, within the measurement uncertainty specified, until **31 December 2029**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage, Preparation, and Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur that affect the certification before the expiration of the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Preparation of the material and coordination of the technical measurements leading to certification were performed by C.F. Ferraris and M. Peltz of the NIST Materials and Structural Systems Division. The development of the computational model used to provide certified values was performed by N.S. Martys of the NIST Materials and Structural Systems Division and W.L. George of the NIST Applied and Computational Mathematics Division.

Statistical consultation on measurement design and analysis of the certification data was performed by B. Toman of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Certificate Issue Date: 11 July 2019

Steven J. Choquette., Director
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Certified Values: The certified values presented in this certificate were generated using a parallel plate rheometer for paste and a computational model [7]. To calibrate a rheometer [7] using the data provided in this certificate, two methods are suggested, Method A uses the approximation of the Bingham equation and Method B is based on the viscosity vs. shear rate curves. In Section "Instructions for Storage, Preparation, and Use", a methodology is described for both the methods. Method A is an easier method to use and would provide both the yield stress and the plastic viscosity as per the Bingham equation, however, it uses only the linear portion of the stress-rate curve and it is only recommended for characterizing concrete rheological behavior at high shear rates [7]. On the other hand, Method B utilizes the viscosity vs. shear rate curve and it is not model dependent.

Method A: Based on Bingham Parameters

The measurands are the parameters listed and the certified values are metrologically traceable to the indicated consistent SI units. Values of the Bingham parameters are provided in Table 1 by both a parallel plate rheometer and the computational model for the paste component used in SRM 2497. Bingham parameters are provided for the mortar and concrete using the computater model in Table 2 and Table 3, respectively.

Table l. Certified Values and Standard Uncertainty for Yield Stress and Plastic Viscosity for 0 % Concentration of Beads Based on Parallel Plate Measurement and the Computational Model

Yield Stress

	Certified Values (Pa)
Parallel Plate Measurement	12.5 ± 0.5
Computational Model	22 ± 4

Plastic Viscosity

	Certified Values (Pa·s)
Parallel Plate Measurement	7.0 ± 0.2
Computational Model	6.40 ± 0.06

Table 2. Certified Values and Standard Uncertainty for Yield Stress and Plastic Viscosity for 20 % Concentration of 1 mm Beads Based on the Computational Model [4]

Parameter	Certified Values
Yield Stress	35 ± 5 Pa
Plastic Viscosity	11.6 ± 0.1 Pa·s

Table 3. Certified Values and Standard Uncertainty for Yield Stress and Plastic Viscosity for the Concrete Mixture (mortar with 40% 10 mm beads) Based on the Computational Model [4]

Paramater	Certified Values
Yield Stress	129 ± 22 Pa
Plastic Viscosity	88.5 ± 0.4 Pa·s

Method B: Based on the Viscosity vs. Shear Rate Curve
The data are divided into two regions, above and below the shear rate equal to 1 s^{-1}. The data in each region were fit to functions f_1 and f_2 (see Equations 1 and 2 respectively). To represent the full dataset with a single function, Equation 3 was developed, which seamlessly combines the two functions without any significant deviations from f_1 and f_2 in their respective regions.

(1) $$f_1(\dot{\gamma}) = \frac{A_1}{\dot{\gamma}^{B_1}} + C_1$$

(2) $$f_2(\dot{\gamma}) = \frac{A_2}{\dot{\gamma}^{B_2}} + C_2$$

(3) $$\mu = [(tanh(-a(\dot{\gamma} - 1)) + 1)f_1(\dot{\gamma}) + (tanh(a(\dot{\gamma} - 1)) + 1)f_2(\dot{\gamma})]/2$$

where

$\dot{\gamma}$ = Shear rate in units of s^{-1}
μ = Viscosity in units of Pa-s
A_i, B_i, *and* C_i *(i= 1,2)* = coefficients to fit the curve
a = parameter to smoothly combine curves f_1 and f_2.

Paste: The coefficients were estimated by a non-linear least square regression fit to the paste SRM 2497 data, obtained with the parallel plate geometry. The maximum difference was 3 % or less of the experimental values shown. The standard uncertainty of the estimates of the coefficients was calculated [9] and is given in parentheses below. The parameter, a, was set to four and was not estimated, therefore it has no uncertainty. The viscosity (μ) can be calculated at any given shear rate with Equation 3, and the parameters below:

A_1 = 8.62 (1.18)	A_2 = 8.89 (0.18)
B_1 = 0.94 (0.05)	B_2 = 0.53 (0.04
C_1 = 7.49 (0.35)	C_2 = 5.94 (0.24)
a = 4	

Mortar: To obtain the viscosity vs. shear rate with inclusions of 20 % by volume of 1 mm glass beads, the shear rate and the viscosity of Equation 3 need to be scaled by the factors shown in Table 4 by using a computer simulation [4]. These factors are used to multiply the paste shear rate and the viscosity, respectively, to obtain the values for mortar at 20 % of beads concentration.

Table 4. Unitless Scaling Parameters and Uncertainty to Obtain the SRM 2497 Curve of the Mortar with 20 % by volume of 1 mm Beads Concentration [7], Determined by Computer Simulation

μ_{sc} factor	$\dot{\gamma}_{sc}$ factor
1.8 ± 0.1	0.77 ± 0.01

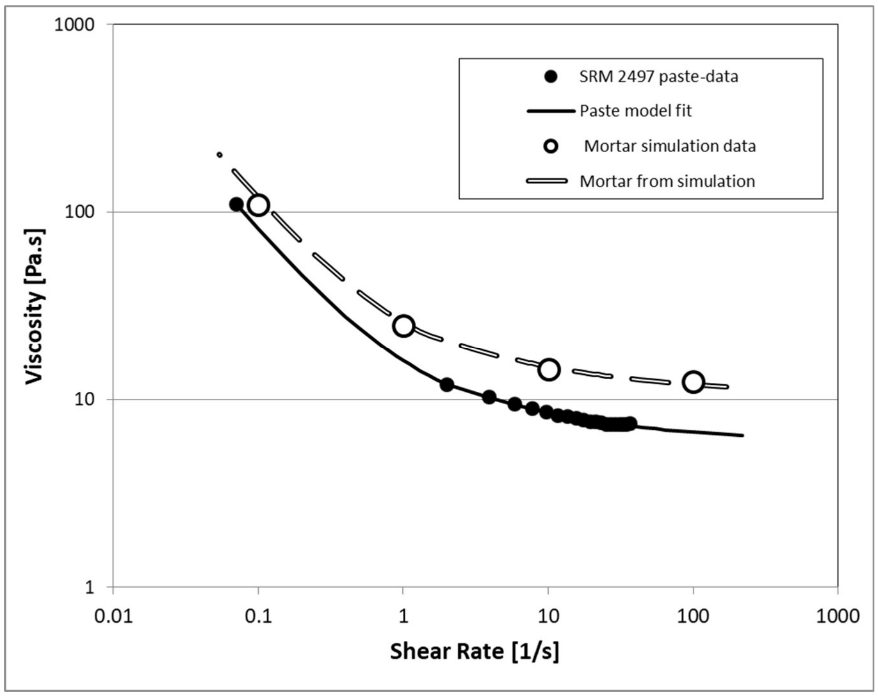

Figure 1. Simulation model data and predicted data from scaling parameters are compared to experimental data results. The relative uncertainty is about 5 % [7].

Concrete: To obtain the viscosity vs. shear rate with inclusions of 40 % by volume of 10 mm glass beads, the shear rate and the viscosity of Equation 3 need to be scaled by the factors shown in Table 5. These factors are used to multiply the paste or mortar shear rate and the viscosity respectively to obtain the values at the concrete composition.

Table 5. Unitless Scaling Parameters and Uncertainty to Obtain the SRM 2497 Curve of Concrete with 40 % Concentration of 10 mm Beads [7]

Material	$\mu_{\text{sc-m}}$ factor	$\dot{\gamma}_{\text{sc-m}}$ factor
Paste	14.3 ± 1.1	0.25 ± 0.03
Mortar	7.9 ± 0.7	0.33 ± 0.06

Alternatively, the parameters A_1, A_2, B_1, etc., in Equation 3 can be replaced by:

$A_1 = 35$ (6.6)	$A_2 = 62.3$ (8.1)
$B_1 = 0.94$	$B_2 = 0.53$
$C_1 = 107.1$ (9.7)	$C_2 = 84.9$ (7.4)
$a = 4$	

to give the full viscosity vs. shear rate curve of the concrete SRM (uncertainty in parenthesis).

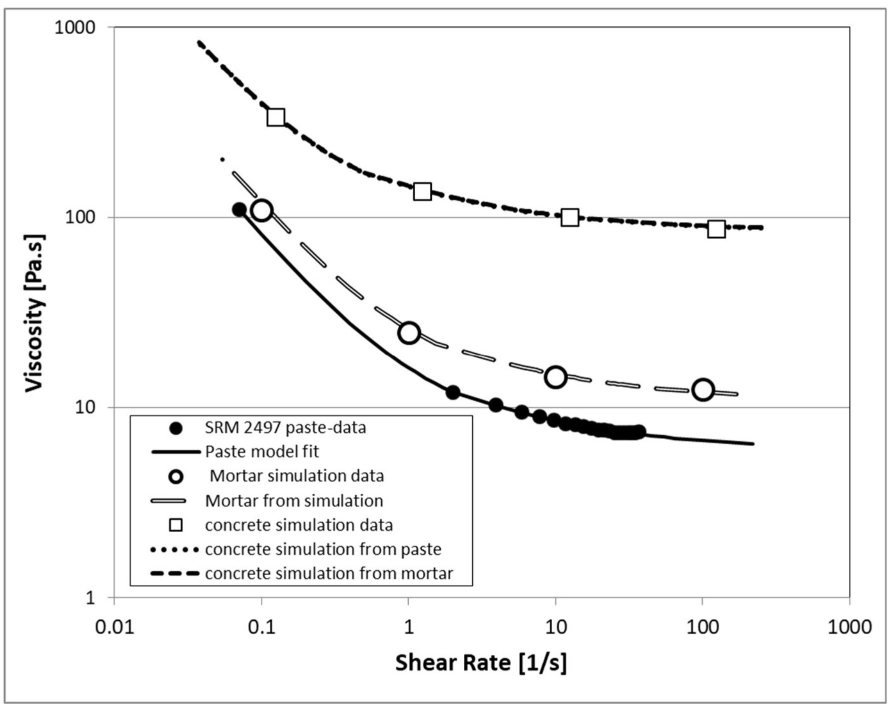

Figure 2. Simulation model data and predicted data from scaling parameters are compared to experimental data results. The concrete model curve is representing two overlapping curves, generated either from the paste or the mortar model. The relative uncertainty is about 10 % throughout the shear rates shown [7].

INSTRUCTIONS FOR STORAGE, PREPARATION, AND USE

Storage: Store the bulk, unmixed material at room temperature. The SRM batch needs to be prepared by the operator before it can be used. Once the components are mixed as described below, the SRM should be stored at 23 °C ± 2 °C in a sealed plastic jar and used within 7 days [5].

Preparation: The composition of SRM 2497 is a paste where 1 mm beads (20 % by volume) and 10 mm beads (40 % by volume) are added. As the total volume using most of the materials provided is to produce 20 L of the SRM 2497 to be used in the calibration of concrete rheometers. To achieve that goal, the constituents need to be added in stages during mixing:

- Corn syrup and water
- Addition of limestone
- Addition of 1 mm beads
- Addition of 10 mm beads

The 5-gallon containers used for shipment of the materials can be used as the mixing vessel. The mixer that was used is a handheld grout two-speed mixer (slow and high) with a single helical blade.

The steps to prepare the mixture and the amounts with some tips are provided here.

1. Store and use the corn syrup, limestone, beads and the water needed at 23 °C ± 2 °C at least a day prior of the mixing. The corn syrup container provided should be placed upside down to facilitate emptying it.

2. Weigh the bucket to be used for mixing, M_b [g]

3. Weight the paste ingredients:
    a. Weigh the corn Syrup directly in the mixing bucket: 5257.8 g
    b. Weigh the distilled water (set aside) 1660.4 g
    c. Weigh the limestone (set aside): 12043 g

4. Mix procedure for the corn syrup solution
    a. Add water to corn syrup
    b. Mix 2 min at slow speed
    c. Mix 2 min a high speed
    d. Scrape side for 30 s
    e. Mix at high speed for 4 min (scrape again after 2 min)

5. Mix procedure for the paste
    a. Add about 200 g to 300 g of limestone into the bucket containing the corn syrup solution
    b. While mixing at slow speed for 2 min, add slowly the rest of the limestone
    c. Mix for 1 min at medium speed
    d. Scrape the sides
    e. Mix for 4 min a high speed

6. Calculation of the amount of 1 mm beads needed for 20 % by volume
    a. Weigh the bucket with the paste, M_{bp} [g]
    b. Densities:
        i. paste, ρ_p, is: 1930 kg/m^3 ± 30 kg/m^3 (or measure it)
        ii. beads: 2465 kg/m^3
    c. Volume of paste: $V_p\ [m^3] = (M_{bp}-M_b)/\ (\rho_p/1000)$
    d. Mass of beads [g] = V_p*0.25*2.465.

7. Mix procedure of the mortar (20 % beads by volume)
    a. Add the beads into the paste while mixing for 2 min on medium
    b. Mix for 2 min on high

8. Calculation of the amount of 10 mm beads needed for 40 % by volume:
    a. Weigh the bucket, M_{bm} [g]
    b. Densities:
        i. mortar, ρ_m is 2020 kg/m^3 ± 5 kg/m^3 (or measure it)
        ii. Beads: 2465 kg/m^3
    c. Volume of mortar, $V_m\ [m^3] = (M_{bm}-M_b)/\ (\rho_m/1000)$
    d. Mass of beads [g] = V_m*0.67*2.465

9. Mix procedure of the concrete (40 % beads by volume)
    a. Add the beads to mortar while mixing for 2 min on medium
    b. Mix for 2 min on high (Two persons are needed, one to hold the bucket or it will rotate with the mixer)

The SRM can be stored in the bucket with the provided lid at 23 °C. The shelf life of the concrete is 7 day (limited by the shelf life of the paste [5]). Discussion on the type of design of the rheometer can be found in Reference 3.

NOTICE TO USERS
If some material was removed for intermediate measurements (paste or mortar), it is advisable that the density be measured, in case some water or beads were not removed uniformly.

Use: The goal is to calibrate any rotational rheometer with a geometry selected by the user. The following steps need to be followed:

1) Prepare a paste or a mortar or concrete SRM, following the instructions above
2) Place the SRM mixture in the rheometer to be calibrated and conduct a sweep rotational speed controlled test. Increase the speed from zero to any desired value (span at least three decades of rotational speed, for instance from 0.0104 rad/s to 10.4 rad/s (0.1 rpm to 100 rpm)) and then decrease it while recording the torque (newton meter). Repeat at least three tests with a geometry of choice on a sample of SRM 2497 and find the average values from the produced torque and respective rotational speed.
3) Since the raw data measured do not directly correspond to the SRM 2497 certified values, the torque and rotational speed need to be scaled to match the known certified shear stress and shear rate. Comparison of the raw data measured, either the paste, the mortar or the concrete, to the model data from this certificate, should be performed following either method A or B. A module is available on the NIST website to perform these calculation.

Method A (Based on Bingham Parameters): It is assumed that the shear stress (τ), shear rate ($\dot{\gamma}$), and viscosity (μ) are fundamentally proportional to the torque (Γ), rotational speed (N), and angular momentum (Γ/N), respectively. Using reference 7, the *(Γ vs. N)* results from the new geometry are scaled to match the *(τ vs. $\dot{\gamma}$)* certified SRM 2497 values by calculating scaling factors K_τ and K_μ. The scaling factors convert the three raw variables into fundamental rheology units by using the proportionality relationships. Also, if preferable, the two scaling factors listed are able to produce a direct shear rate scaling factor, K_γ, by means of Equation 4 which is derived from the known relationship for Bingham materials shown in Equation 5.

$$\mu = \tau / \gamma \quad [\text{Pa·s}] \tag{4}$$

Thus, the shear rate scaling factor would be:

$$K_\gamma = K_\tau / K_\mu \tag{5}$$

Figure 3 provides a schematic representation of the process.

Obtain the average of the raw data at day x.
(Torque, *Γ* [μNm] and rotational speed, *N* [rpm]) for at least three runs.
Convert N units to [rad/s] and Γ to [Nm].

Use the average data to find the slope (*S*) and y-intercept (*I*) of the torque vs. rotational speed relationship.
Note: use only the linear part of the curve, with at least 10 data points.

Obtain known SRM 2497 certified values from certificate. *For ex.:*
(Plastic viscosity) Slope= $\mu_{SRM2497}$
(*yield Stress*) Y-int = $\tau_{0,SRM2497}$

Find calibration factors using these equations:

$$K_\tau = \frac{\tau_{0,SRM2497}}{I} \quad [\mathrm{Pa/N{\cdot}m}] \qquad K_\mu = \frac{\mu_{SRM2497}}{S} \quad [\mathrm{Pa{\cdot}s/N{\cdot}m{\cdot}s}]$$

Calibrate raw data into shear stress (τ), shear rate ($\dot{\gamma}$) and apparent viscosity (μ) using calibration factors:

$$\mu = K_\mu \frac{\Gamma}{N} \quad [\mathrm{Pa{\cdot}s}]$$

$$\dot{\gamma} = K_{\dot{\gamma}}\, N = \frac{K_\tau}{K_\mu}\, N \quad [\mathrm{s^{-1}}]$$

$$\tau = K_\tau\, \Gamma \quad [\mathrm{Pa}]$$

Figure 3. Process Diagram for the Calibration of Rheometer Data from Raw Torque and Rotational speed to fundamental rheological variables, viscosity and shear ratebased on the Bingham parameters [7]
Note: This process is applied for calibration of any geometry used in a coaxial rheometer. The conversion from rpm to rad/s requires multiplication by 2π/60

Method B (Based on the Viscosity vs. Shear Rate Curve): The viscosity vs. shear rate curve is extracted from the SRM 2497 certificate and compared with the experimental data measured using the SRM 2497. Then, a least square fit of the experimental data is calculated to determine the scaling factors, L_γ, L_τ, *and* L_μ (See Figure 4). This method is recommended for the concrete.

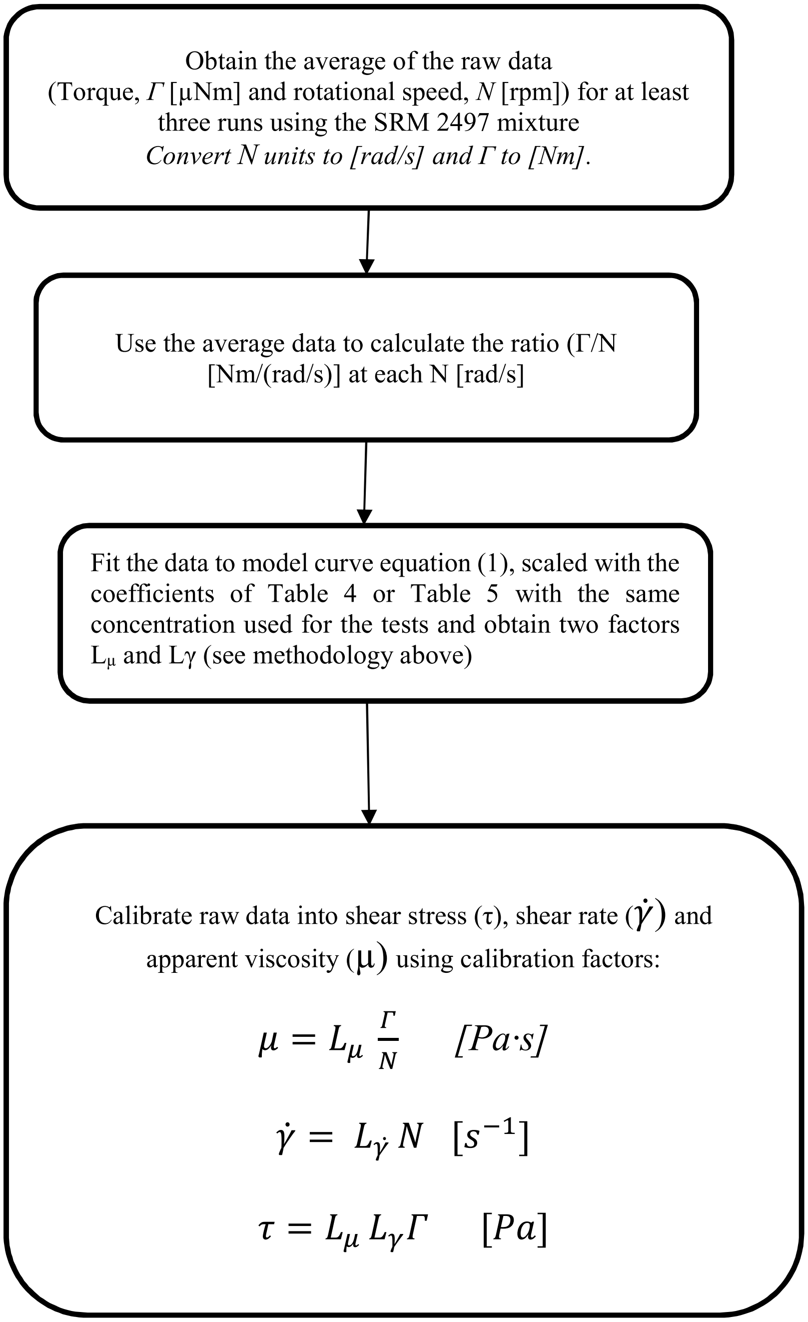

Figure 4. Process Diagram for the calibration of rheometer data from raw torque and rotational speed to fundamental rheological variables, viscosity and shear rate based on the viscosity vs. shear rate curves [7]
Note: This process is applied for calibration of any geometry used in a coaxial rheometer. The conversion from rpm to rad/s requires multiplication by 2π/60

Material Selection and Packaging: The limestone was purchased in bulk and the corn syrup was purchased in 1 gallon bottles. The beads, 1 mm and 10 mm, were purchased in 5 gallon buckets. The packaging contains enough material to prepare one batch of 20 L of material for testing.

Homogeneity Assessment and Certification Analyses: Certification analyses for Bingham parameters were performed at NIST on 2 randomly selected units of the SRM. The uncertainty reported for the certified values includes allowances for random measurement variability, day-to-day variability, and material heterogeneity between units.

Safety and Disposal: Refer to the SDS for further information. Dispose of contents and container according to local regulations.

REFERENCES

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[2] 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed July 2019).

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[7] Ferraris, C.F.; Martys, N.S.; Peltz, M.; George, W.L.; Garboczi, E.J.; Toman, B.; *Certification of SRM 2497: Standard Reference Concrete for Rheological Measurements*, NIST Special Publication SP-260-194; U.S. Government Printing Office: Washington, DC (2019); available at https://www.nist.gov/sites/default/files/documents/2019/04/24/sp260-194.pdf (accessed July 2019).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm.