Designation: ~~C1874 – 19~~ C1874 – 20

Standard Test Method for Measuring ~~the~~ Rheological Properties of Cementitious Materials Using ~~a~~ Coaxial Rotational Rheometer[1]

This standard is issued under the fixed designation C1874; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. ~~Scope~~ Scope*

1.1 This test method is used to measure the rheological properties such as plastic viscosity and yield stress of cementitious materials with particles up to 1 mm in diameter.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C125 Terminology Relating to Concrete and Concrete Aggregates
C219 Terminology Relating to Hydraulic and Other Inorganic Cements
C305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency
C1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the Physical Testing of Hydraulic Cements
C1602/C1602M Specification for Mixing Water Used in the Production of Hydraulic Cement Concrete
C1738/C1738M Practice for High-Shear Mixing of Hydraulic Cement Pastes
C1749 Guide for Measurement of the Rheological Properties of Hydraulic Cementious Paste Using a Rotational Rheometer

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology C125 and Terminology C219.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *shear rate, n*—the rate of change of the shear strain with time.

3.2.1.1 *Discussion*—

The shear strain **(1)**[3] is the relative in-plane displacement of two parallel layers in a material body divided by the separation distance, in units of s^{-1}.

3.2.2 *shear stress, n*—~~The~~the component of stress that causes successive parallel layers of a material body to move, in their own planes (that is, plane of shear), relative to each other, in units of Pa.

[1] This test method is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.22 on Workability.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] ~~Alex Olivas, Michelle A. Helsel, Nicos Martys, Chiara F. Ferraris, Raissa Ferron, "Rheological Measurement of Suspensions Without Slippage: Experimental and Model" NIST-TN 1946, Dec. 2016 - dx.doi.org/10.6028/NIST.TN.1946.~~The boldface numbers in parentheses refer to the list of references at the end of this standard.

***A Summary of Changes section appears at the end of this standard**

3.2.3 *relative viscosity, n*—the ratio between the viscosity of a material and the viscosity of a reference material as a function of shear rate, both viscosities measured using the same rheometer and with the same procedure.

4. Summary of Test Method

4.1 This method allows the measurement of the shear stress of cementitious paste or mortar, using a rotational rheometer with various spindle geometries. The plastic viscosity and yield stress are determined per the Bingham model.

4.2 As cementitious slurries are thixotropic, the shear history has an impact on the rheological measurements; mixing the sample, for instance 200 s^{-1} for 60 s, is needed to ensure correct preparation of the sample, prior to the measurements.

4.3 This mixing procedure is followed by the measurements of the shear stress evolution as a function of the shear rate in the range of ~~0.1 s~~0.1 to^{-1} ~~to~~ 50 s^{-1}. Before the standardization of the rheometer, the shear rate needs to be measured in terms of rotational speed in units of rad/s.

5. Significance and Use

5.1 Rheological properties provide information about the workability of cementitious paste or mortar. This test method may be used to measure flowability of a cement paste or the influence of a specific material or combination of materials on flowability.

5.2 As rheological properties are sensitive to sample heterogeneities, special care is needed using this procedure so that sedimentation, particle migration away from the spindle or slippage at the surface of the spindle and container, is avoided.

6. Interferences

6.1 Rheological properties may be sensitive to sample preparation (mixing method) and testing procedure (shear history, for example), so a comparison of properties obtained using different procedures is not recommended, unless relative viscosity is considered.

6.2 Paste or mortar mixtures that are very fluid are prone to sedimentation and may produce erroneous data using this procedure. Such sedimentation is especially likely in shear thinning and thixotropic mixtures. Paste or mortar mixtures that are too viscous may result in a torque that exceeds the capability of the instrument.

6.3 Paste or mortar mixtures may have a shear stress versus shear rate that does not follow the Bingham model, such as in a shear thinning or thickening behavior. In this case, this test method shall not be used.

6.4 Larger cementitious materials particles or sand particles may lead to jamming if the gap between the shearing surfaces is too small and thereby increase the shear stress. This gap needs to be selected considering the particle size of the material to be tested.

6.5 Incorporation of air in the pastes and mortars during mixing reduces viscosity and increases flow.

6.6 The time of testing after initial contact of cement with water influences the results.

7. Apparatus

7.1 *Rotational Rheometer*—conforming to Guide C1749. The instrument imparts a controlled rotation speed to the spindle, Ω in rad/s, and measures the resulting torque, Γ in Nm.

7.2 *Spindle*—The rotational tool used for testing **(2)** (see Note 1). The diameter of the spindle shall be such that the gap between the spindle and the container follow Guide C1749 recommendation (see Notes 1 and 2).

NOTE 1—Acceptable shapes for the spindles are cylinders, vanes with four or more blades, and double helical. Unless standardization is properly done (see Section 13), the raw data from the various spindles cannot be directly compared.[4]

NOTE 2—The gap between the spindle and the container is calculated as the distance between the highest points on any surface protrusion.

7.3 *Container*—The rheometer container shall be made of stainless steel or a material that does not react with the cement. The wall of the container shall have surface protrusions to prevent slippage~~.~~ **(2).** The height and the diameter of the container shall be adapted to the selected spindle, namely suitable diameter larger than the spindle to accommodate the paste or the mortar (see 7.2), and the height shall be at least ~~20 mm~~ 20 mm longer than the spindle.

NOTE 3—The additional 20 mm is to provide at least 10 mm space at the bottom and 10 mm cover at the top.

7.4 *Cover at ~~the~~ Top of ~~the~~ Container*—The container shall be covered with a vapor barrier or a water saturated material to prevent evaporation of water from the paste or mortar. Care shall be taken to ensure that the water saturated material does not drip water onto the sample.

7.5 *Temperature Control System*—the container temperature shall be controlled to the nearest ±2 °C. The temperature may be selected to reflect the conditions at which the material will be used (cold weather or hot weather concreting, for example).

[4] This SRMs can be obtained from the National Institute of Standards and Technology ~~(www.nist.gov).~~ (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

8. Reagents and Materials

8.1 *Standard Material*—A paste or fluid that has a linear correlation between the shear stress and the shear rate for a significant range of shear rates (at least one order of magnitude, that is ~~1 s~~1 to^{-1} ~~to~~ 10 s^{-1}) (see Note 4). The standard materials shall behave like a Bingham material and shall have certified rheological properties obtained from fundamental procedures.

NOTE 4—SRM 2492,[4]~~,~~ Bingham Paste Mixture for Rheological Measurements, and SRM 2493,[4]~~,~~ Bingham Mortar Mixture for Rheological Measurements, may be used for this purpose. SRM 2493 is a combination of SRM 2492 and 1 mm beads.

8.2 *Water*—Unless specified, use potable water as specified in Specification C1602/C1602M. When specified, use job-specific water when performing a specific test series that is related to field application. Follow Specification C1602/C1602M in reporting the type of water used.

9. Hazards

9.1 **Warning—**Fresh cementitious mixtures are caustic and may cause burns to skin and tissue upon prolonged exposure. The use of gloves, protective clothing, and eye protection is recommended. Wash contact area with copious amounts of water after contact. Wash eyes for a minimum of 15 min. Avoid exposure of the body to clothing saturated with the liquid phase of the unhardened material. Remove contaminated clothing immediately after exposure.

10. Test Specimens

10.1 The matrix of materials and mix proportions depend on the purpose of the testing program. To evaluate field mixtures, use materials and proportions based on the concrete mixture design without the coarse aggregate. The fine aggregates used in this test method shall not contain particles larger than 1 mm.

10.2 Mix the paste according to Practice C1738/C1738M or the mortar according to Practice C305.

11. Preparation of Apparatus

11.1 Place the container in the rheometer ~~as per~~ in accordance with the manufacturer instructions.

11.2 Attach the spindle ~~as per~~ in accordance with the manufacturer instructions.

12. Conditioning

12.1 The rheometer temperature controller shall be turned on and set to the desired temperature at least 30 min before any testing or standardization, to ensure that the temperature of the container has stabilized.

13. Standardization

13.1 *Background*—The standardization procedure essentially aims to establish the two constants $\boldsymbol{K}_{\dot{\gamma}}$ and K_τ defined respectively as the ratio of the shear rate $\dot{\boldsymbol{\gamma}}$ over the rotational speed Ω and as the shear stress τ over the torque Γ, using a standard material (see 8.1).

13.2 *Procedure to Determine ~~the~~ Two Constants K_τ and $\boldsymbol{K}_{\dot{\gamma}}$:*

13.2.1 Measure the torque, Γ, versus rotational speed of the standard material (see 8.1) using the following procedure:

13.2.1.1 Introduce the standard fluid material in the measuring container. Insert the spindle into the container, providing sufficient clearance between the spindle and the bottom of the container at the bottom to avoid interference between the spindle and the container (see Note 5).

NOTE 5—For aggregates up to 1 mm in diameter, it was found that a clearance of 15 mm between the spindle and the bottom of the container is sufficient.

13.2.1.2 Maintain the system (container, with material and spindle) at the specified temperature by the standard material certificate. Record the temperature of the material in the rheometer before taking the first reading.

13.2.1.3 Select the rotational speed range to be used (see Note 6). Start rotation at the lowest rotational speed of the pre-selected rotational speed range. Maintain continuous rotation at this rotational speed for 30 s and then record the torque.

NOTE 6—Typical rotational speeds are 0.01 ~~rad/s~~ to 10 rad/s.

13.2.1.4 Increase the rotational speed following the pre-selected range, in at least ten steps. Shift to the next speed immediately after torque is stable for at least 5 s (see Note 7). Record the ascending rotational speed, Ω, and the respective measured torque, Γ.

NOTE 7—A torque is considered stable if there is less than 5 % variation in the torque during 5 s.

13.2.1.5 Decrease the rotational speed in the same pre-selected range and steps used for the ascending rotational speed. Shift to the next speed immediately after torque is stable for at least 5 s (see Note 7). Record the descending rotational speed, Ω, and the respective measured torque, Γ. The calculations are done using the descending portion of the curve.

13.2.2 *Calculation of ~~the constants~~ Constants K_τ and $\boldsymbol{K}_{\dot{\gamma}}$:*

•*(1)* Calculate the slope, *S*, and intercept, *I*, of the descending angular speed [rad/s] versus torque [N·m], using only the linear part of the curve (see Note 8).

~~NOTE 8—The linear part of the curve is determined by fitting a line using at least five points. A regression of a Bingham material yields R^2 higher than 0.90 and the intercept should be a positive value (>0).~~

•*(2)* Read the yield ~~stress,~~stress ~~τ~~to$_0$ ~~[Pa],~~the nearest 0.01 Pa, and plastic ~~viscosity,~~viscosity ~~μ [Pa.s],~~to the nearest 0.01 Pa.s, in the certificate of the standard material ~~selected~~selected.

•*(3)* Calculate the standardization ~~constants:~~constants to the nearest one-hundredth:

$$K_\tau = \tau_0 / I [\text{Pa/N.m}] \quad (1)$$

$$K_\mu = \mu / S [\text{Pa.s/N.m.s}] \quad (2)$$

~~$$K_\gamma = K_\tau / K_\mu [\text{unitless}] \quad (3)$$~~

$$K_{\dot{\gamma}} = K_\tau / K_\mu [\text{dimensionless}] \quad (3)$$

NOTE 8—The linear part of the curve is determined by fitting a line using at least five points. A regression of a Bingham material yields R^2 higher than 0.90 and the intercept should be a positive value (>0).

14. Testing Procedure for Cementitious Pastes or Mortars

14.1 Select the rotational speed range to be used (see Notes 9 and 10).

NOTE 9—A typical range of shear rate is ~~0.1 s~~0.1 to^{-1} ~~to~~ 50 s^{-1}.

NOTE 10—Ten to twenty steps of rotational speeds are recommended.

14.2 Prepare the materials (see Section 10).

14.3 Maintain the measuring tool at the testing temperature within ±2 °C for the duration of he test.

14.4 Record the elapsed time from initial contact of the water with the cementitious material and the start of the rheological testing.

14.5 Introduce the paste or mortar mixture in the measuring container. Insert the spindle into he container, providing sufficient clearance between the spindle and the bottom of the container to avoid interference between the spindle and the container (see Note 5).

14.6 Start rotation at the lowest rotational speed of the pre-selected rotational speed range. Maintain continuous rotation at this rotational speed for 20 s and then record the torque.

14.7 Increase the rotational speed following the pre-selected range. Rotate continuously for 20 s at each shear rate before taking a reading or when the shear stress is stable for at least 5 s (see Note 7). Shift to the next speed immediately after each reading. Record the ascending rotational speed, Ω, and the respective measured torque, Γ.

14.8 Decrease the rotational speed following the pre-selected range. Rotate continuously for 20 s at each shear rate before taking a reading or when the shear stress is stable for at least 5 s (see Note 7). Shift to the next speed immediately after each reading. Record the descending rotational speed, Ω, and the respective measured torque, Γ.

14.9 Calculate shear rate, shear stress, plastic viscosity and yield stress for the descending curve (see Section 15).

14.10 Plot the rheological measurements as torque as a function of the speed or shear stress as a function of shear rate.

15. Calculation of Rheological Properties

15.1 Convert the raw data (rotational speeds Ω and torque Γ readings) to shear rate and shear stress using the constants calculated under Section 13 and equations Eq 4-7.

NOTE 11—Use the descending portion of the curve.