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Standard Guide for Measurement of the Rheological Properties of Hydraulic Cementious Paste Using a Rotational Rheometer¹

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1. Scope*

1.1 This guide covers description of several methods to measure the rheological properties of fresh hydraulic cement paste. All methods are designed to determine the yield stress and plastic viscosity of the material using commercially available instruments and the Bingham model. Knowledge of these properties gives useful information on performance of cement pastes in concrete.

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 guide offers an organized collection of information or a series of options and does not recommend a specific course of action. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this guide may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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.*

¹ This guide 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. Referenced Documents

2.1 ASTM Standards:²

C305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency

C511 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes

C1738 Practice for High-Shear Mixing of Hydraulic Cement Pastes

E2975 Test Method for Calibration or Calibration Verification of Concentric Cylinder Rotational Viscometers

2.2 Other Standards:

API Recommended Practice 10B Testing Well Cements, American Petroleum Institute, Washington, DC (1997)

ISO 10426-2 (2003) Petroleum and Natural Gas Industries—Cements and Materials for Well Cementing—Part 2: Testing of Well Cements—Section 5.2

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology C125 and C219.

3.2 Definitions of Terms Specific to This Standard:^{3,4}

3.2.1 *apparent viscosity, n* —the shear stress divided by rate of shear, in units of Pa.s.

3.2.2 *plastic viscosity, n* —in the plastic (Bingham) model, the slope of the shear stress – shear rate curve, in units of Pa.s.

3.2.3 *thixotropy, n* —a decrease of the apparent viscosity under constant shear stress or shear rate followed by a gradual recovery when the stress or shear rate is removed.

3.2.4 *yield stress, n* —the stress corresponding to the transition from elastic to plastic deformation, in units of Pa; it is also referred to as the stress needed to initiate flow. It would be calculated using the Bingham model in this guide.

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 H.A. Barnes, J.F. Hutton and K. Walters, *An Introduction to Rheology*, Elsevier (1989).

⁴ Hackley V.A., Ferraris C.F., "The Use of Nomenclature in Dispersion Science and Technology" NIST Recommended Practice Guide, SP 960-3, 2001.

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

3.2.5 *Bingham model, n*—a rheological model for materials with non-zero yield stress and a linear relationship between shear rate and shear stress, following the equation: $\tau = \tau_B + \dot{\gamma}\eta_{pl}$; where τ_B Yield stress in Pa, $\dot{\gamma}$ Shear rate in 1/s, τ Shear stress in Pa, and η_{pl} Plastic viscosity in Pa.s.

4. Significance and Use

4.1 Rheological properties determined using this guide include plastic viscosity and yield stress as defined by the Bingham model and apparent viscosity.

4.2 Rheological properties provide information about the workability of hydraulic cementitious paste. As an example, the yield stress and plastic viscosity indicate the behavior of a specific cement paste composition. As another example, the apparent viscosity indicates what energy is required to move the suspension at a given strain rate. This test may be used to measure flowability of a cement paste or the influence of a specific material or combination of materials on flowability.

4.3 Rheological properties may be sensitive to the procedure being used. This guide describes procedures that are expected to provide reproducible results.

5. Summary of Guide

5.1 This guide provides procedures for the determination of rheological properties of fresh cement paste using a rotational rheometer with geometries, such as parallel plate, narrow-gap and wide gap concentric cylinders.

6. Interferences

6.1 Rheological properties may be sensitive to the procedure, so a comparison of properties obtained using different procedures is not recommended, unless relative viscosity (ratio between the plastic viscosity of a materials and the plastic viscosity of a reference material, both measured using the same rheometer) is considered.

6.2 Rheological properties may be sensitive to the shear history of the sample, so comparison of properties using different mixing procedures is not recommended.

6.3 Paste mixtures (water and cement particles) that are very fluid may yield erroneous data using this procedure due to settling of particles. Such settling is especially likely in shear thinning and thixotropic mixtures.

6.4 Larger cement particles or aggregations of cement particles may block flow in a narrow-gap rheometer and thereby increase the shear stress. The gap between the shearing surfaces needs to be selected with consideration of the particle size of the material to be tested. Depending on the gap size, it may be necessary to remove larger particles by sieving or otherwise prevent segregation.

6.5 Incorporation of air in the paste 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 General Description:

7.1.1 The apparatus shall be a rotational rheometer in which the sample is confined between two surfaces (called the shearing surfaces), one of which is rotating at a constant rotational speed, Ω and the other being stationary. The apparatus shall measure both the rotational speed and the torque required to maintain that speed.

7.1.2 The rheometer geometry shall provide a simple shearing flow (laminar, without turbulence). Allowable geometries and their equations for computing stress and strain rate from the measured values of rotational speed and torque are described in 7.4.

7.2 The rotational rheometer shall be capable of measuring shear stress at strain rates in the range from 0.1 s^{-1} to 600 s^{-1} . The range of shear rates will be selected by the operator depending on the geometry used. At least five measurements need to be recorded.

NOTE 1—Most experiments found in the literature do not use the full range of shear rates prescribed here. For example, most parallel plate measurements are done between 0.1 s^{-1} to 50 s^{-1} . The selection of the shear rate range might take into account the exact geometry of the rheometer.

7.3 Regularly check the calibration and zeroing of the apparatus, as discussed in 7.9.

7.4 Rheometer Geometry:

7.4.1 The rheometer geometries described in this section provide simple shearing flow, essential for reliable computation of stress and strain rates. The equation for computation of stress and strain rates is given for each geometry.

NOTE 2—The following assumptions were made to develop the equations that appear in this section: (1) the fluid is homogeneous, (2) slip at the wall is negligible, and (3) the flow regime is laminar.

7.4.2 Selection of the geometry of the rheometer. Three geometries are described here: narrow-gap concentric cylinders, wide-gap concentric cylinders, and parallel plates. The selection of the geometry should be based on the type of rotational rheometer available. One criterion to select between the narrow-gap and the wide-gap should be based on the maximum size of the particles in the cement tested.

7.4.2.1 *Narrow-Gap Concentric Cylinder*—With this type of rheometer, the sample is confined between two concentric cylinders of radii R_1 and R_2 ($R_2 > R_1$), one of which, the rotor, is rotating at a constant rotational speed Ω and the other is stationary. The rotation of the rotor in the presence of the sample produces a torque that is measured at the wall of the inner cylinder. The cylinder radii should be selected such that the shear stress is uniform across the gap. This condition is assumed to be satisfied if:

$$\left(\frac{R_1}{R_2}\right) > 0.92 \quad (1)$$

where R_1 is the radius of the inner rotating cylinder (m) and R_2 is the radius of the outer stationary cylinder (m).⁵

To prevent slip (development of a liquid layer at the wall of the rotating cylinder that produces an anomalously low stress), the surface of cylinders may be serrated or at least rendered

⁵ DIN 53019-1:2008, *Viscometry—Measurement of viscosities and flow curves by means of rotational viscometers—Part 1: Principles and measuring geometry.*