
# Rheological Characterization of Flow Table Reference Material

## DETAILS

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# Research Results Digest 360

## RHEOLOGICAL CHARACTERIZATION OF FLOW TABLE REFERENCE MATERIAL

This digest summarizes key findings of NCHRP Project 20-07/Task 272, “Rheological Characterization of Flow Table Reference Material,” conducted by the AASHTO Materials Reference Laboratory under the direction of the principal investigator, Haleh Azari. The digest is based on the project final report authored by Haleh Azari and Chiara Ferraris of the National Institute of Standards and Technology. The full text of the project final report is available for download at http://apps.trb.org/cmsfeed/TRBNetProjectDisplay.asp?ProjectID=2600.

## INTRODUCTION

The flow table is described in AASHTO Standard Specification M 152, “Flow Table for Use in Tests of Hydraulic Cement,” as an apparatus “. . . used in making flow tests for consistency of mortars in tests of hydraulic cement.” In brief, a mortar’s flow value, which is an indication of its workability, is measured by unmolding a sample of the mortar on the flow table, dropping the table a specified number of times, and measuring the increase in diameter of the sample, expressed as a percent of its original diameter. Use of the flow table is referenced in AASHTO T 71, “Effect of Organic Impurities in Fine Aggregate on Strength of Mortar”; T 106, “Compressive Strength of Hydraulic Cement Mortar Using 50-mm or 2-in. Cube Specimens”; and T 137, “Air Content of Hydraulic Cement Mortar.”

Footnote 3 of the 2003 revision of AASHTO M 152 states that a reference material for calibration of a flow table is available from the Cement and Concrete Reference Laboratory (CCRL) at the National Institute of Standards and Technology, Washington, DC 20234. This reference material is compounded in batches by CCRL from a white mineral oil and a finely ground silica powder. The oil viscosity is not measured, but the value reported by its manufacturer is recorded. The powder fineness is obtained by a permeability method similar to the Blaine method (e.g., ASTM C204, “Fineness of Hydraulic Cement by Air-Permeability Apparatus”), although in recent years the particle size distribution of the powder has also been measured using a laser diffraction particle size analyzer. Each batch of silica powder is ground at CCRL from an Ottawa sand meeting the requirements of ASTM C778, “Standard Sand,” until its measured fineness matches that of the previous batch. The powder is then mixed with the oil and the flow value of the batch is determined using the flow table at CCRL. This flow table is identical to commercially available units. Thus, flow values obtained with the CCRL flow table are considered reference values for use in the calibration of all other flow tables in the United States.

Although the CCRL flow table is well maintained and has been successfully used for preparation of all flow table reference

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materials in the past, there are several concerns with the overall process of producing the CCRL flow table reference material:

1. If the CCRL flow table should break beyond repair, the single reference table is gone and new batches of reference material could not be produced.
2. The production of the reference material is based on trial and error, and the flow values provided by the CCRL flow table vary from one batch to another.
3. The CCRL flow table is itself calibrated using a previous batch of the reference material, leading to a potential risk of error propagation.
4. Due to normal wear-and-tear on the mechanical components of the CCRL flow table, it is likely, though undeterminable, that the flow values for "identical" batches of reference material are changing over time.
5. Since the oil and silica powder are obtained and processed in small-scale quantities, the reference material too must be prepared in small batches by a labor-intensive, time-consuming process.

These concerns highlight the need for development of alternative methods to both produce the reference material and accurately and precisely measure its flow value that are based on measurement of fundamental material properties of the reference material and its components and so are independent of the use of the CCRL flow table. With such alternative methods, future production of the reference material would be highly reproducible from batch to batch, avoiding the problem of an indeterminate drift in flow value over time.

## OBJECTIVES AND RESEARCH APPROACH

The objectives of NCHRP Project 20-07/Task 272 were to (1) fully characterize available batches of CCRL flow table reference material by measuring their fundamental material properties and those of their components and (2) propose a more efficient, stable, and reproducible method for future production of the reference material that is independent of the CCRL flow table.

To accomplish these objectives, the research team obtained samples of oils and silica powders used to produce several batches of reference material from 2004 to present. The viscosity of the oils was measured as were the fineness and particle size distribution of the silica powders. The rheological properties of the reference materials were measured with a parallel plate rheometer; measured properties included the dynamic yield stress, the Bingham viscosity, and the Bingham yield stress. The data were analyzed to establish correlations among the flow table values of the reference materials, their rheological properties, the viscosity of the oils, and the particle size distribution of the silica powders.

The results of this analysis established that the flow value of the reference materials was correlated to their rheological properties and, in turn, that these rheological properties were correlated with oil viscosity and particle size distribution of the silica powder. Experiments were also conducted to establish the sensitivity of the reference material rheology to variations in oil viscosity, powder particle size distribution, the proportion of oil and powder, and temperature.

Based on these results, a method was proposed to produce the flow table reference material with the required flow value without the use of the CCRL flow table. This method relies on the relationship established between the flow value of the reference material and its dynamic or Bingham yield stress as measured with a parallel plate rheometer.

## FINDINGS AND CONCLUSIONS

The project final report[1] presents a detailed discussion of the experiments conducted in the research and their results. These results promise a more reliable, less expensive, and more environmentally friendly process for producing the CCRL reference material.

The following are key findings of the research:

1. The time necessary to grind the ASTM C778 standard sand to the required particle size distribution could be reduced to 17 hours from present 20 hours.
2. Grinding the ASTM C778 standard sand could be replaced altogether by blending two commercially available silica powders, at a substantial reduction in labor hours and cost.
3. A methodology was developed to measure the rheological properties of the reference material

[1] Available for downloading at http://apps.trb.org/cmsfeed/TRBNetProjectDisplay.asp?ProjectID=2600.

based on strong correlation between flow table values and dynamic or Bingham yield stresses.

4. The reference material can be produced and its characteristics monitored with parallel plate rheometer measurements of dynamic or Bingham yield stress that demonstrate strong correlations with flow value. Use of the parallel plate rheometer makes the production of the reference material completely independent of the condition or, indeed, the existence of the CCRL flow table. The parallel plate rheometer is calibrated independently of the reference material with standard calibration fluids, ensuring that any change in the flow value of the reference material can be assigned to the condition or operation of the flow table and not to variability of the reference material.

The following procedure is proposed for producing the flow table reference material:

1. Buy the oil in bulk as usual and determine its viscosity using a vibrational viscometer (or other type of viscometer suitable for the oil's viscosity range).
2. Produce the silica powder by blending two commercially available silica powders in lieu of grinding ASTM C778 standard sand.
3. Blend the oil and powder to produce the reference material and determine its rheological properties using a calibrated parallel plate rheometer set up as follows:
   a. Serrated plates with a 0.8-mm gap.
   b. Stress growth sequence: a 30-s rest period followed by a shear rate of 0.2 $s^{-1}$ for 300 s. The dynamic yield stress is the peak of the shear stress v. time curve.
   c. Bingham test method: shear rate sweep increasing from 1 $s^{-1}$ to 20 $s^{-1}$ followed by a decrease from 20 $s^{-1}$ to 1 $s^{-1}$. The Bingham yield stress and plastic viscosity are calculated from the shear rate v. shear stress curve as the shear rate decreases through a linear regression calculation.

Initial target values for the rheological properties should be finalized using the CCRL flow table. Once this is done, future batches can be produced to the rheological target value with a rheometer that is calibrated independently of the flow table reference material, by checking the rheometer's torque and rotational speed with well-established procedures.

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