

1 **New Approach to Calibrate the Mortar Flow Table**

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1 ABSTRACT

2

3 The flow table is a standardized test that is widely used to qualify a mortar for compressive

4 strength and air content. It is also the only standard test to quantify the workability of a mortar.

5 Therefore, the calibration of this device is paramount, and is done today by preparing a reference

6 material consisting of a mixture of silica powder and oil. The two materials are mixed and tests

7 are performed using the reference flow table located in the Cement and Concrete Reference

8 Laboratory (CCRL). This flow table is identical to commercial versions currently used, and the

9 values obtained with this flow table are considered the reference values for calibrating flow

10 tables in the United States. This is an empirical procedure, and relies heavily on one device that

11 could break or generate results that can drift over time. This study will review the manufacturing

12 process of the reference material, provide historical data, and propose a more scientifically-based

13 approach to developing an improved reference material.

14

1 INTRODUCTION

2 The flow table is a standardized test (AASHTO M 152 (1) and ASTM C 230 (2)) widely
3 used to qualify a mortar to be tested for compressive strength and air content. The flow table test
4 is the only standard test to quantify a mortar's workability. This test was revised in 2003 and
5 specifically states that, ". . . a reference material for calibration of the flow table is available from
6 the Cement and Concrete Reference Laboratory (CCRL) at NIST." The reference material
7 provided by CCRL is composed of an oil that is mixed with a finely ground silica powder.

8 The two materials are mixed and tests are performed. This flow table is identical to
9 commercial versions used in practice. The values obtained with this flow table are considered to
10 be reference values, and are used to calibrate all the flow tables in the U.S. Although the
11 reference flow table at CCRL is well maintained and has been employed successfully for
12 preparation of reference materials in the past, there are several issues that need to be considered:

- 13 1) The reference flow table is very old (over 30 years). If it breaks beyond repair, industry
14 will be unable to promulgate a flow table value
- 15 2) The production of the reference material is based on trial and error as the proportions of
16 oil and silica powder are adjusted to obtain a specified flow table value.
- 17 3) Due to normal wear-and-tear on the mechanical components, it is likely, but unknowable,
18 whether the flow table values for identical mixtures have remained constant over time;
- 19 4) The reference material is prepared in small batches since the properties of the silica are not
20 being controlled on a large scale. This is a very time-consuming process.

21 Considering these issues, it seems necessary to explore an alternative method of determining
22 the properties of the reference material using material science. Material properties can be
23 measured accurately and precisely, so future reference materials can be very reproducible,
24 avoiding the problem of drifting values over time.

25 The approach adopted in this study is to determine the properties required for the reference
26 material from historical data, and then determine the characteristics of each component as shown
27 today. The next step is to know how small variations of the properties of the material would
28 affect the results on the flow table. This knowledge will allow the development of a reference
29 material that does not rely solely on the results of one specific flow table.

30 HISTORICAL INFORMATION

31 The flow table reference material has been prepared by CCRL since 1965 using the same
32 method.

33 The original oil used was *Primol 355*¹. Its characteristics were measured at NIST
34 (Formerly NBS) to have a viscosity of 0.15 Pa·s (170 cSt) at 25 °C, and a specific gravity of
35 0.878 at 23 °C (74 °F). In 1970, a 55-gal drum was purchased, and was used. According to
36 laboratory notebooks, another mineral oil was used in 2007, called *Drakeol 35*. The viscosity
37 reported by the manufacturer is a value between 0.057 Pa·s (65.8 cSt) and 0.062 Pa·s (71 cSt) at
38 40 °C, as determined using ASTM D445 (3). The specific gravity is reported by the
39 manufacturer to be between 0.864 to 0.881 at 25 °C (77 °F), measured using ASTM D4052 (4).

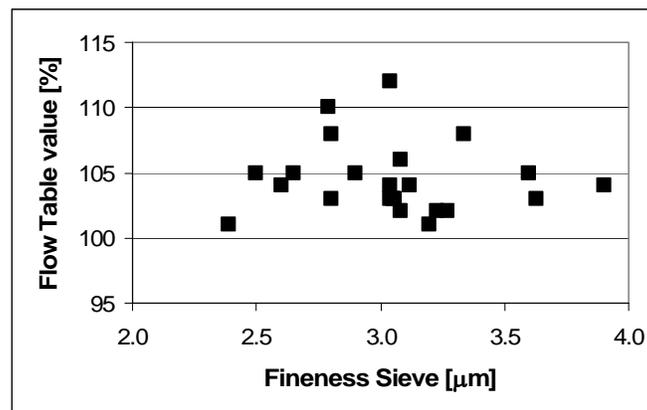
40 The powder has been produced by grinding ASTM C778 (5) graded sand in a laboratory
41 ball mill loaded with approximately 90 kg (200 lbs) of agate stones and 45 kg (100 lbs) of quartz
42

¹ Certain commercial products are identified in this paper to specify the materials used and procedures employed. In no case does such identification imply endorsement or recommendation by the National Institute of Standards and Technology, nor does it indicate that the products are necessarily the best available for the purpose.

1 sand. These materials, although mineralogically distinct, are chemically similar, and thus avoid
 2 any problems of contamination. Each resulting lot was labeled alphabetically starting with J in
 3 1965, through Z in 1993, continuing with AA in 1996 to AF in May 2004, and returning to single
 4 letters ranging from G in July 2004, to J in 2009.

5 The powder fineness was measured using a “Fisher sub-sieve sizer,” as described in
 6 ASTM B330 (6). This instrument measures the permeability of a bed of powder and, through
 7 calibration using a reference powder, calculates the particle size distribution of the test sample.
 8 The calculation and calibration assumes that all particles are spherical and identical in size. The
 9 value targeted by the grinding was 3 μm . Using data recorded in the laboratory notebooks, the
 10 average calculated particle size was $(3.0 \pm 0.4) \mu\text{m}$, thus achieving the targeted particle size.
 11 Since 2004, the Particle Size Distribution (PSD) was measured using a laser diffraction device
 12 (7). The median particle size (d_{50}) for the four (4) lots produced since 2004 averaged $6.5 \mu\text{m} \pm$
 13 $0.6 \mu\text{m}$. The difference in the calculated median particle size obtained using these two techniques
 14 is not surprising as they are based on completely different physical measurements and underlying
 15 assumptions.

16 The silica powder and the oil are mixed by hand in a 1 L glass jar for 10 min using a
 17 spatula. Nominally, the proportions are 500 g of ground silica and 350 g of oil, but the
 18 proportions are adjusted until a flow table value near $110 \% \pm 5 \%$ is obtained. The average
 19 value acquired from the flow table was $104 \% \pm 3 \%$, based on the historical values recorded
 20 from 1965 to 2008. These flow table values are plotted in Figure 1 as a function of the “Fineness
 21 Sieve” by the Fisher sizer. (The data from PSD by laser diffraction are not shown, as the two
 22 methods are not comparable.) It seems that within the narrow range of particle size and flow
 23 table values, there is no apparent correlation between the two values. This would need to be
 24 explored to determine whether the particle median size or particle size distribution is a critical
 25 parameter.



27 Figure 1: Relationship between fineness and flow value from historic data by Fisher size.

29 METHOD AND MATERIAL USED

30 In this study, several methods and materials are employed to explore the best procedure
 31 to characterize the powder and the oil used.

33 Material used

34 The materials used were three oils, ground silica, and a commercially available silica
 35 powder. The oils used were purchased from *Drakeol*¹, and they were selected to have nominally
 36

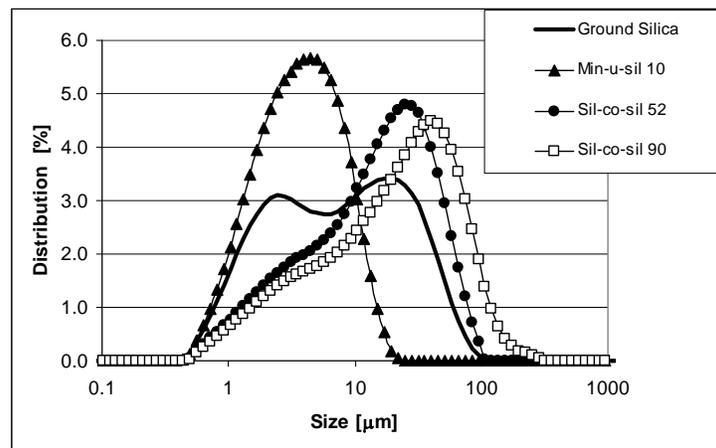
1 different viscosities, as shown on Table 1. The measurements were made using a coaxial
 2 rotational rheometer.

3 The ground silica was produced by CCRL grinding of ASTM C778 standard sand. Three
 4 silica powders were purchased from the same manufacturer as the standard sand, *Min-u-sil 10*,
 5 *Sil-co-sil 90*, and *Sil-co-sil 52*, but with different PSDs, as shown in Figure 2.

7 Table 1: Oil characteristics

Oil name ¹	Density* at 23 °C (measured at NIST) [g/cm ³]	Viscosity at 40 °C (from the manufacturer) [Pa·s]	Viscosity at 23 °C (measured at NIST) [Pa·s]
<i>Drakeol 21</i>	0.864	0.035	0.088 ± 0.005
<i>Drakeol 35</i>	0.852	0.059	0.159 ± 0.005 [#]
<i>Drakeol 600</i>	0.869	0.096	0.246 ± 0.001

8 Notes: * Uncertainty in the density measurement is less than 1%
 9 # *Drakeol 35* viscosity at 23 °C was measured by several methods (not described here)



11 Figure 2: Particle size distribution of the silica powders selected. The uncertainty on the
 12 measurement is estimated to be 5 %.

14 **Rheological measurements**

15 To put the results of the flow table on an absolute scale, the flow of a material as
 16 measured by the flow table needs to be linked to the rheological properties of the material.
 17 Rheological properties of the reference material (oil and ground silica) were measured using a
 18 rotational rheometer (8, 9). The configuration was parallel plates with serrated surfaces. The
 19 plates had a diameter of 35 mm and a variable gap between 0.4 mm and 1.0 mm. The shear rate
 20 range was 0 s⁻¹ to 20 s⁻¹, as determined by analytical calculation from the rotational speed (10).
 21 The induced shear stresses were measured, corresponding to 15 shear rates when increasing the
 22 rotational velocity, and 20 levels when decreasing the rotational velocity. Each measured point
 23 was recorded after the shear stress reached equilibrium or after 20 s, whichever occurred first.
 24 The descending data were expressed to a line using ordinary least squares, and the slope and
 25 intercept were calculated. The plastic viscosity and yield stress were calculated using the
 26 Bingham equation. This equation states that the slope of the shear stress vs. shear rate is the
 27 plastic viscosity, and the intercept at zero shear rate is the yield stress.
 28

1 The oil viscosity was measured using a parallel plate rotational rheometer (the same as
2 for the material) but also using a coaxial configuration of the same rheometer with a cup
3 diameter of 43 mm and a bob diameter of 38 mm. The length of the bob is 55 mm. Also, a
4 vibrational rheometer was used to characterize the oil.

6 **IMPACT OF OIL OR POWDER CHANGES**

8 **Influence of grinding time on flow properties**

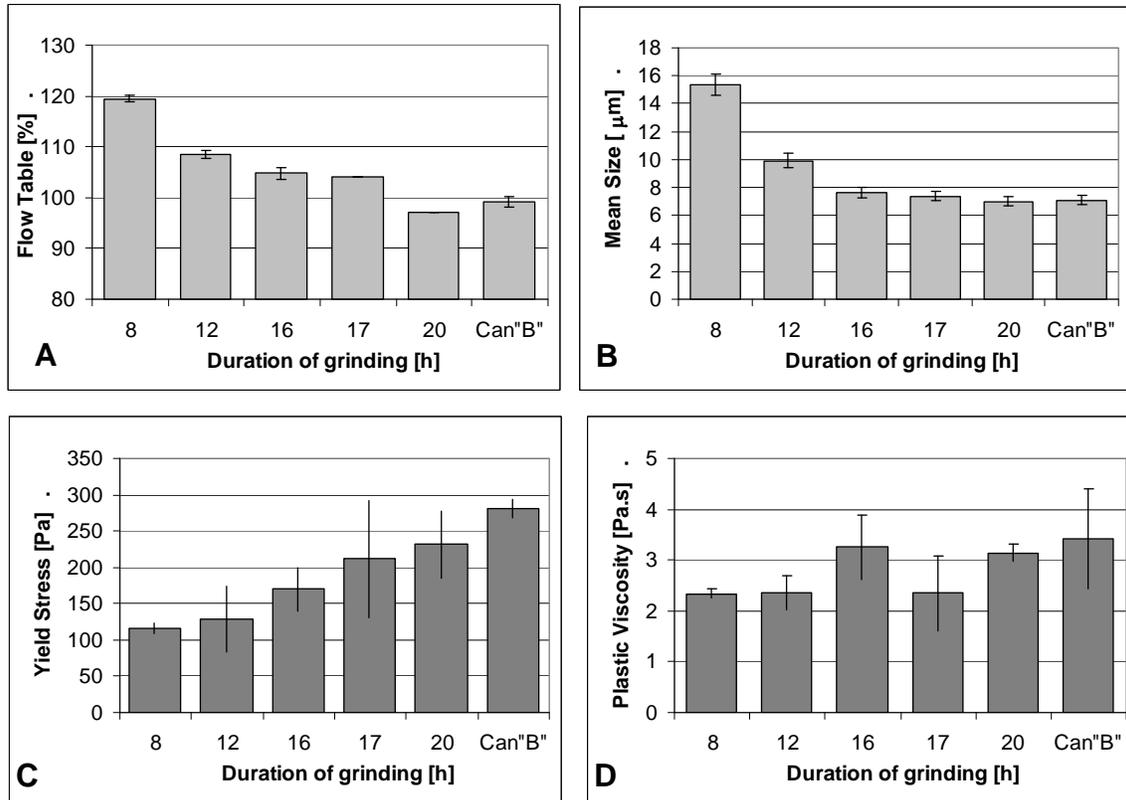
9 The grinding process implies that the material be continuously ground in a ball mill for
10 20 h. The question was how the grinding time would influence the results. Therefore, during the
11 grinding of lot J, about 1 kg of material was taken from the ball mill at specific times (8 h, 12 h,
12 16 h, 17 h, and 20 h). Subsequently, the ball mill was emptied by scooping the material by hand,
13 and was placed in a 5-gal bucket labeled *Can A*. Then, most of the balls were removed, and the
14 rest of the material was placed in *Can B*. This is the standard procedure developed by CCRL. In
15 this paper, the final product as collected after 20 h of grinding is labeled “ground silica”.

16 A mixture using 500 g of silica and 35 g of *Drakeol 35* was prepared. The silica powders
17 were the powder obtained at each grinding duration (8 h, 12 h, 16 h, 17 h, and 20 h) and with
18 *Can B*. Flow table test and rheological tests were performed for each grinding duration, and on
19 *Can B*. The PSD for each powder also was measured. Figure 3 shows the results obtained as a
20 function of grinding time.

21 The flow table value obtained for *Can B* (the end product) is 99 %. The uncertainty that is
22 assigned to the flow table by the ASTM standard is ± 5 %. The flow table values after 16 h and
23 17 h are 104 % and for 20 h, it is 97 %. Therefore, it could be inferred that the values obtained
24 after 16 h are within the error of the flow table for this mixture of 99 % (94 % - 104 %). This
25 could suggest that grinding could be stopped at 16 h to 17 h, instead of 20 h, saving time and
26 energy.

27 The median particle size does not change significantly after 16 h or 17 h, and the flow is
28 within the accepted uncertainty of 5%.

29 The rheological measurements were attained using a parallel plate rotational rheometer
30 with a gap of 0.4 mm. The results are shown in Figure 3. The yield stress values have not
31 stabilized at 17 h, while the plastic viscosity seems to have reached the value of *Can B* after 16 h
32 within the error bars. It should be noted here that the material is not perfectly Bingham, and
33 therefore the rheological values are an approximation at this stage. The difference in yield stress
34 is in contrast with the data obtained with the flow table, implying that some rheological
35 measurements are more sensitive to small changes in the powder particle size distribution than
36 the flow table. To assign the properties of the reference material by rheological measurement,
37 further measurements will be needed to determine the best target value.



1 Figure 3: Influence of grinding duration on flow table (A) results and particle size (B), yield
 2 stress (C), and plastic viscosity (D). The error bars represent one standard deviation of the mean.
 3

4 **Influence of Oil Viscosity**

5 Three mixtures were prepared using the ground silica and three oils (Table 1) in the
 6 proportion of 500 g of silica and 350 g of oil. This composition is a typical proportion, even if
 7 the target value of 110 % is not reached. For each mixture, flow table and the Bingham
 8 parameters of yield stress and plastic viscosity were conducted.

9 Figure 4 shows the relationship between the flow table and the various rheological
 10 parameters. Each point in Figure 4 is the average of three measurements. As expected, as the
 11 flow table value increases, all the parameters decrease. It can be noted that the range of oil
 12 viscosity was large, i.e., an increase of 179 % of the lower viscosity. Conversely, the changes in
 13 flow table and in the rheological parameters are comparatively smaller: Bingham yield stress
 14 63 %, the Bingham viscosity by 13 % and flow table by 20 %. It could be stated that a small
 15 change in the oil viscosity will not affect the rheological properties or the flow table value. This
 16 is desirable, as it helps to minimize fluctuations in the rheological values with changes in oil
 17 viscosity due to small variations in temperature.

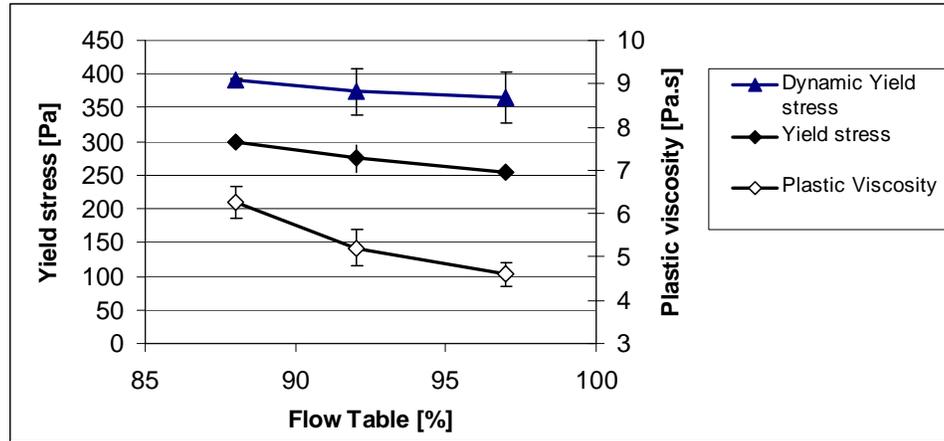


Figure 4: Relationship between the flow table and rheological parameters. The uncertainty bars represent one standard deviation of the mean.

Influence of particle size distribution

Researchers have found that the PSD influences rheological properties.⁽¹¹⁾ Therefore, it is necessary to determine an acceptable range of PSD. In this respect, the particle size distribution of the powder was varied by using commercially available silica powder (Figure 2). Table 2 shows the PSD characteristic extracted from the data in Figure 2 and the flow table results obtained using the same mixture proportions. The mixture proportions used were 500 g of powder and 350 g of *Drakeol 35*. The d_{50} represents the median of the PSD, while the d_{10} and d_{90} represent 10 % or 90 % PSD values, respectively. The span represents the width of the distribution.

Figure 5 shows the same data as in Table 2 but, in graphical mode. It is clear that the flow table value increases with the increase in d_{50} . As all the mixtures were prepared at constant oil content, this result is not too surprising. The smaller particles would increase the oil demand to produce the same flow. On the other hand, it seems that for the same d_{50} of $7.0 \mu\text{m} \pm 0.5 \mu\text{m}$, a wide range of flow table values can be obtained (76 % to 97 %). This is seen by *Ground Silica, Mixture A*, and *Mixture B* on Table 2. From Table 2 it is not clear if any of the other characteristics of the powder PSD could explain the wide scatter in flow table values. Therefore, the shape of the curve PSD is probably what influences the flow table results. In conclusion, the PSD shape and median size need to be monitored to ensure that flow table results are consistent.

NEW METHODOLOGY TO PRODUCE THE REFERENCE MATERIAL

From the above results on the influence of the PSD and oil viscosity on the flow table, it was determined that small variations in oil viscosity do not influence the results, but the PSD of the powder can change the flow table data. On the other hand, while the oil is purchased as-is, the silica powder is obtained through a lengthy (even if reduced to 17 h) grinding process. Also, as the stones used for grinding are unique, it might be hard or impossible to reproduce the same grinding effect in another laboratory or another ball mill. Therefore, it would be advantageous to be able to simply purchase a silica powder with a known PSD.

1 Table 2: Particle size characteristics (the uncertainty on the measurement is estimated to be 1 %
 2 (7)) and flow table results (the uncertainty on the measurement is estimated to be 5 %, as per the
 3 ASTM standard).

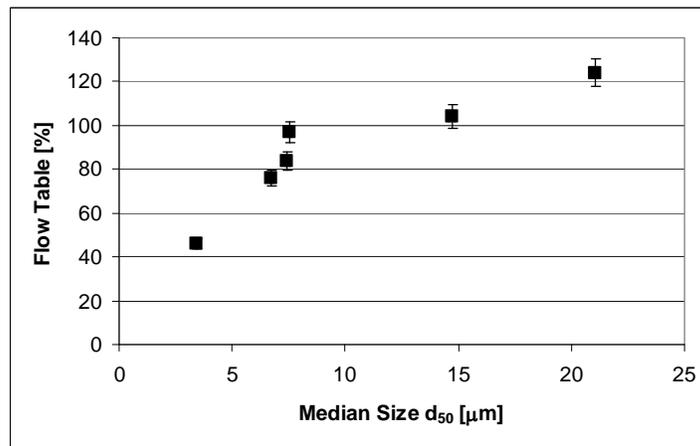
Material ¹	Particle size characteristics				Flow table [%]
	d ₅₀ [μm]	d ₁₀ [μm]	d ₉₀ [μm]	Span*	
Ground Silica	7.5	1.4	31.3	4.0	97
Min-u-sil 10	3.5	1.2	8.9	2.2	46
Sil-co-sil 52	14.7	2.1	44.3	2.9	104
Sil-co-sil 90	21.0	2.4	69.6	3.2	124
Mixture A	7.5	1.4	58.5	7.7	84
Mixture B	6.7	1.4	34.8	5.0	76

4 Note: * The span is calculated as $span = (d_{90} - d_{10}) / d_{50}$. It is a measure of the dispersion of the PSD.

5 Mixture A: 43 % Min-u-sil 10 and 57 % Sil-co-sil 90 by mass.

6 Mixture B: 44 % Min-u-sil 10 and 56 % Sil-co-sil 52 by mass

7



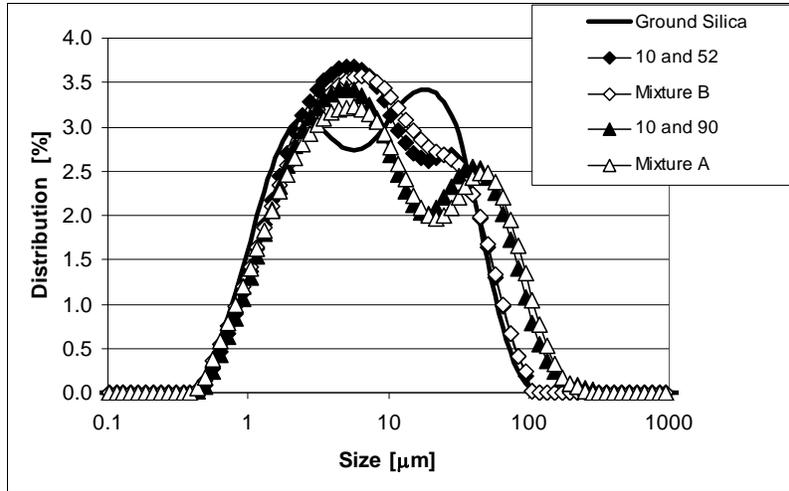
8 Figure 5: Median size particle (d₅₀) and flow table results. Particle size uncertainty on the
 9 measurement is estimated to be 1 % (7), and flow table uncertainty is estimated to be 5 %, as per
 10 the ASTM standard.

11

12 This idea was to use commercially available silica powder. Using the distributions of
 13 these powders, a spreadsheet was developed to calculate the combined distribution of two
 14 powders, one fine (*Min-u-sil 10*) and one coarse (either *Sil-co-sil 52* or *Sil-co-sil 90*). The two
 15 mixtures were labeled “10 and 52” and “10 and 90” on Figure 6. These distributions were
 16 obtained by varying the proportion of each component to attempt to match the ground silica
 17 distribution. From Figure 6, it can be seen that neither of the two simulations were able to match
 18 perfectly the ground silica. The next step was to verify that the calculated distribution matched
 19 the measured PSD. The mixtures were prepared, blended using a 3-D mixer², and the PSD was
 20 measured by laser diffraction. From Figure 6, it can be seen that the measured values match very
 21 well the calculated distribution.

22

² A 3D mixer is device that allows a material contained in jar to be tumbled and rolled at the same time. A Turbula was used for this mixing.



1
2 Figure 6: PSD of combination of powders. “10 and 52” and “10 and 90” were simulated
3 distribution (see text), and Mixture A and B were measured distributions.
4

5 Table 3: Rheological values for *ground silica* and *Mixture B*

Material	Flow Table [%]	Bingham Yield stress [Pa]			Bingham Plastic viscosity [Pa.s]		
		Value	Standard deviation	CV	Value	Standard deviation	CV
Ground silica (with 350 g of oil)	97	275	20	7 %	5.2	0.8	15 %
Mixture B (with 400 g of oil)	96	247	3	1 %	3.5	0.2	5 %
Relative difference	1%	10%			32%		

6 CV [%] = standard Deviation/value
7

8 In Table 2 and Figure 6, it can be seen that *Mixture B* has nearly the same particle size
9 range (with an error of 3.5 µm) as the ground silica. Therefore, it was selected as the best
10 candidate to replace the ground silica. However, there are more fines (peak at 3.5 %) than the
11 ground silica (peak at 2.5 %). We already know that the flow table value is lower (76 % instead
12 of 97 %). Therefore, to match the flow table value, more oil would be needed.

13 Tests were conducted to determine the amount of oil required to reach the same flow
14 value than obtained with the ground silica, namely 97 % ± 5 %, with *Mixture B*. The flow table
15 of *Mixture B* is 96 %, if 400 g of oil is used instead of the 350 g (as for the ground silica
16 mixture). Table 3 shows the results obtained. For the difference between the two mixtures
17 examined, the yield stress differences are within the error of the measurements, while the
18 viscosity difference is significantly larger. These preliminary data are encouraging, but need to
19 be repeated to obtain statistical verification. Nevertheless, a specific value of yield stress and
20 viscosity range could be established that would provide the same flow table results. Further tests
21 could allow the use of only 350g of oil if the mixture were adjusted, for instance, by reducing the
22 amount of fine particles.
23
24

1 CONCLUSION

2 The flow table is a very commonly used standard test that is easy to perform. According
3 to the ASTM/AASHTO standard test method, a reference material, composed of silica powder
4 and oil, is need to calibrate the flow table. This reference material is tedious to produce, and
5 needs to be characterized using fundamental measurements to ensure that there is long term bias
6 due to mechanical wear from one batch to the next. This study showed that there are two ways to
7 reduce the work needed to produce the powder: 1) grind only for 17 h (reduction of 3 h); 2) use
8 commercially available silica powders mixed in the correct proportion to obtain the same d_{50} and
9 span in the PSD as in the original ground silica powder. It was also shown that small variation in
10 oil viscosity does not impact the results of the flow table.

11 This study would be completed by performing more tests to ensure statistical
12 reproducibility and writing a procedure on how to ensure the required rheological characteristic.

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19 for providing the historic notebooks. Dr. Kenneth Snyder and Jeff Bullard from NIST and Dr.
20 Kejin Wang from Iowa State University should be thanked for their review, which improved this
21 paper.

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