

Rheological Characterization of Flow Table Reference Material

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Abstract

The flow table is a standardized test that is widely used to qualify a mortar to be tested for compressive strength and air content. It is also the only standard test to quantify a mortar workability property. Therefore, the calibration of this device is paramount and it is done today by preparing a reference material that consists of a mixture of silica powder and oil. Once the two materials are selected, they are mixed and tests are performed using the flow table at the Cement and Concrete Reference Laboratory (CCRL). This flow table is identical to commercial versions used in practice, and the values obtained with this flow table are considered the reference values that are used to calibrate all the flow tables in the United States (US). This procedure is an empirical procedure and relies heavily on one device that could break or, through mechanical wear, have a result that drifts over time. This study will review the production process of the reference material, provide historical data and then propose a more scientifically-based approach to developing an improved reference material. It was found that the flow table results are more sensitive to the particle size distribution of the powder than the viscosity of the oil. Fundamental rheological properties were determined using a rheometer and some correlations were drawn. A new method to produce the silica powder is proposed that would make the process more reliable, safe, and economically beneficial.

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CHAPTER 1. Introduction

The Flow Table is a standardized test (AASHTO M 152 (1), and ASTM C 230 (2)) that is widely used to qualify a mortar to be tested for compressive strength and air content. The flow table is also referenced in AASHTO T 71 (3), “Effect of Organic Impurities in Fine Aggregate on Strength of Mortar”; AASHTO T 137 (4) and ASTM C 185 (5), “Air content of Hydraulic Cement Mortar”; and AASHTO T 106 (6) and ASTM C 109 (7), “Compressive Strength of Hydraulic Cement Mortar Using 50-mm or 2-in cube Specimens”; ASTM C 311 (8), “Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete”. The Flow Table test is ubiquitous because it is the only standard test to quantify a mortar workability property.

This test was revised in 2003 as part of the ASTM review process and the following statement was added: “...a reference material for calibration of the flow table is available from the Cement and Concrete Reference Laboratory (CCRL) at NIST.” The reference material provided by CCRL is composed of oil that is mixed with a finely ground silica powder.

Once the two materials are selected by CCRL, they are mixed and tests are performed using the flow table at CCRL. This flow table is identical to commercial versions used in practice. The values obtained with this flow table are considered the reference values and are used to calibrate all the flow tables in the US. Although the reference flow table at CCRL is well maintained and has been used successfully for preparation of reference materials in the past, there are several issues with the method:

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

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

A step-by-step approach is adopted to address the above-mentioned issues: (1) determine the properties required for the reference material from historical data, (2) characterize each component used in the test, (3) investigate sensitivity of the flow table test results to the variations in the tested material properties, and (4) develop a new reference material that does not rely uniquely on the results of one specific flow table.

CHAPTER 2. Historical Information

The flow table reference material has been prepared by CCRL since 1965 using a method that has not changed much over the years. The components are oil and silica powder. The oil and powder are commercially available and the powder is refined further at CCRL. To obtain the historical information, laboratory notebooks were examined. The information below is a summary of the findings.

The oil used was initially *Primol 355*. Its characteristics were measured at NIST (Formerly NBS) as a viscosity of 0.15 Pa·s at 25 °C (170 cSt) and a specific gravity of 0.878 at 23 °C (74 °F). In 1970, a 55 gallon drum was purchased and was used for years. From these notebooks, there was no indication of when the oil source was switched other than the first time that the new oil *Drakeol 35* is mentioned for Lot H in 2007. There is no mention in the laboratory notebooks that the viscosity was measured at NIST but the manufacturer reported a value ranging between 0.057 Pa·s (65.8 cSt) and 0.062 Pa·s (71.0 cSt) at 40 °C as determined using ASTM D 445 (9). The specific gravity is reported by the manufacturer to be 0.864 to 0.881 at 25 °C (77 °F), measured using ASTM D 4052 (10).

The powder has been produced by grinding ASTM C778 (11) graded sand in a laboratory ball mill loaded with approximately 90 kg (200 lbs) of agate stones and 45 kg (100 lbs) of quartz sand. These materials, although mineralogically distinct, are chemically similar, thus avoiding any problems of contamination. Each resulting lot was labeled alphabetically starting with J in 1965, continuing through Z in 1993, continuing with AA (1996) to AF (May 2004), and returning to single letters ranging from G (July 2004) to J (2009).

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

The silica powder and the oil are mixed by hand in a 1 liter glass jar for 10 min using a spatula (Appendix A). Nominally, the proportions are 500 g of ground silica and 350 g of oil, but the proportions are adjusted to obtain a flow table value near (110 ± 5) %. Nevertheless, the value of 110 % is rarely assigned to the reference material as the average value obtained from the flow table was (104 ± 3) % based on the historical values recorded from 1965 to 2008 (Figure 1). The lot I was prepared for this project only and therefore, no flow table value was assigned to it. It seems that CCRL has determined that keeping the proportions of material constant is more important than having a set value of 110 %. The ASTM C 230 standard (***Error! Bookmark not defined.***) states that the acceptable standard deviation is 5 %.

These flow table values are plotted in Figure 2 as a function of the “Fineness Sieve” by the Fisher sizer (the data from PSD by laser diffraction are not shown as the two methods are not comparable). It seems that within the narrow range of particle size and flow table values reported, there is no apparent correlation between the two values. Later in this study, the influence of particle size distribution on the flow table value will be examined.

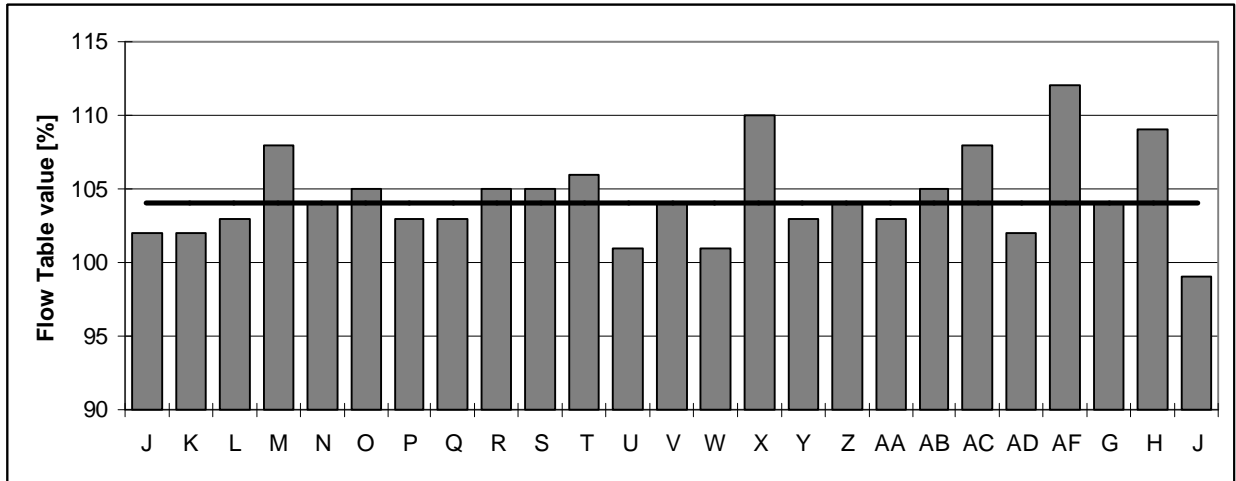


Figure 1: Historic flow table value assigned to reference material. The line is the median value of 104 %

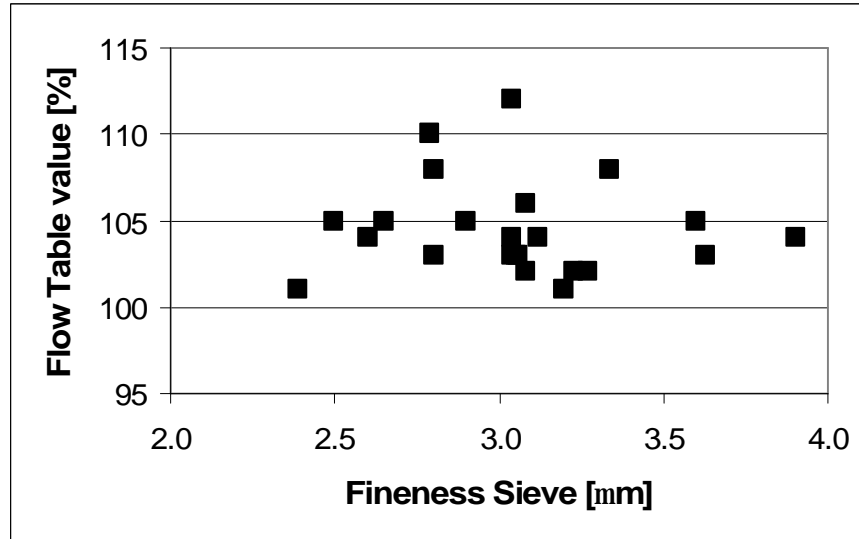


Figure 2: Relationship between fineness by Fisher size and flow table value from historic data. The data from laser diffraction PSD were omitted as non comparable with Fisher Size data.

CHAPTER 3. Materials Used

3.1 Oils

The oil that is used today for the flow table reference material is *Drakeol 35*. Two other mineral oils were selected from the same manufacturer: *Drakeol 600* and *Drakeol 21*. These oils were selected because their viscosity was at least 40% higher or lower than the standard oil used, as shown on Table 1. The measurements were made using a coaxial rotational rheometer at NIST.

Table 1: Oil characteristics

Oil name	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

Notes: * Uncertainty in the density measurement is less than 1 %

[#] *Drakeol 35* viscosity at 23 °C was measured by several methods (see section *Oil Viscosity*)

⁺ The viscosity shown here is the average of the range.

3.2 Silica Powders

The ground silica was produced by CCRL grinding of ASTM C778 standard sand (see section *Grinding Process*). This material is referred to as ground silica in this report. Three other silica powders were purchased from the same manufacturer, U.S. Silica, *Min-u-sil 10*, *Sil-co-sil 90*, and *Sil-co-sil 52*. These powders are sold with a specific PSD. Their PSDs were measured at NIST using a laser diffraction technique (LD) and are shown in Figure 3. *Min-u-sil 10* is a powder with a median (d_{50}) size of 3.45 μm, a little coarser than the first peak in the ground silica distribution (2.5 μm). The two other sands are closer to the second peak of the ground silica distribution (19 μm), namely d_{50} for *Sil-co-sil 52* of 15 μm and for *Sil-co-sil 90* of 21 μm. The scope of using these commercially available powders is to replace the ground silica with a dry material that is easier to obtain and possibly less expensive to produce.

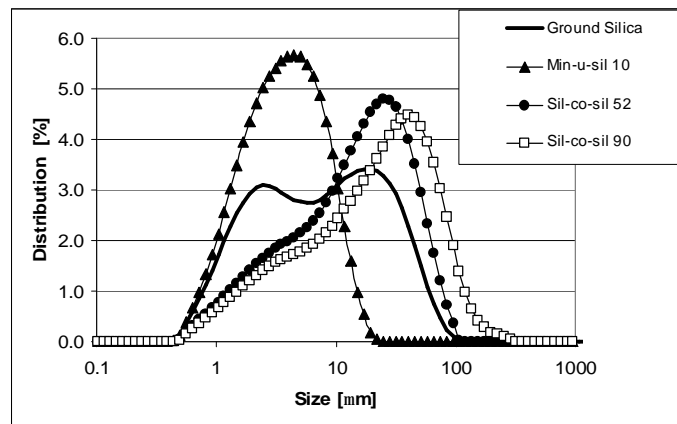


Figure 3: Particle size distribution of the silica powders selected. The uncertainty on the measurement is estimated to be 1 %.

CHAPTER 4. Methods Used

4.1 Flow Table and Mixture Preparation

The flow table measurements were done following the description in ASTM C230. The flow table used is not the CCRL reference flow table, but is identical as it follows the ASTM specifications. The values reported are as defined in the standard test method as the sum of four diameters of the spread of the material created after 25 drops of the table.

The procedure to prepare the mixture followed the recommended practice by CCRL as described in detail in Appendix A. The silica powder and the oil are mixed by hand in a 1 liter glass jar for 10 min using a spatula (Appendix A). Nominally, the proportions are 500 g of ground silica and 350 g of oil unless otherwise stated in this report.

4.2 Particle Size Distribution

The most widely used technique to measure the particle size distribution (PSD) of a powder is based on laser diffraction (LD). Most cement manufacturers that measure PSD use this technique. Therefore, it is the one used in this report.

In the LD technique (13), the angular distribution of light scattered from dilute particle dispersion is measured. To be precise, light can be *scattered*, *diffracted* or *absorbed* by the dispersed particles (14). Scattered light consists of reflected and refracted waves, and depends on the form, size, and composition of the particles. Diffracted light arises from edge phenomena, and is dependent only on the geometric shadow created by each particle: diffraction is independent of the composition of the particles. Absorption occurs when light is converted to heat or electrical energy by interaction with the particles, and is influenced by both size and composition. The so-called laser diffraction (LD) technique incorporates all three of these effects, but is generally limited to the more forward scattering angles. The key material parameter for LD is the complex refractive index, $m = n - ik$, where n is the real component and k is the imaginary (absorptive) component. Scattering arises due to differences in the refractive index of the particle and the surrounding medium (and internal variations in the case of heterogeneous particles). Values of n have been published for many bulk materials (15). Absorption becomes important primarily in the fine fraction, especially below 1 μm . The n value used for silica is 1.544 and the k value is 0.1.

There are two principal methods of data analysis for LD: Mie and Fraunhofer. Mie theory describes scattering by homogeneous spheres of arbitrary size, and is the most rigorous optical scattering model available. For non-spherical particles, Mie provides a volume-weighted equivalent spherical diameter. Further discussion on the benefit of each data analysis method is beyond the scope of this project and can be found in (16). The LD method requires that the particles be dispersed in liquid. In this study, isopropyl alcohol (IPA) was used with a refractive index of 1.39.

4.3 Rheological Parameters

Rheology is the study of the flow of matter. Therefore, it was essential that a flow table reference material and the oil be characterized using rheological tools.

Rheological measurements usually produce a shear stress-shear rate plot [17]. The apparent viscosity [18] is defined as the ratio of the shear stress over the shear rate at a given shear rate. For a Newtonian fluid, it is also equal to the slope of the fitted line of the shear stress-shear rate plot, going through zero, as the relationship is linear. But most granular materials are non-Newtonian. Their main characteristic is that they exhibit a yield stress, which is regarded as the stress needed to initiate deformation or flow of the material. There are several methods to measure the yield stress. The two most common methods are the stress growth method and the extrapolation from the Bingham test method.

In the case of the stress growth method, a small shear rate is applied and the induced shear stress is monitored. This stress increases linearly until the sample yields and starts to flow. Figure 4 shows the various stages of this test.

However, most researchers use the method based on the Bingham equation (Eq. (1)) to determine the plastic viscosity, η_{pl} , and the yield stress, σ_B . This procedure assumes that the plastic viscosity is defined as the slope of the shear stress – shear rate curve and the yield stress is the intercept of the curve at zero shear rate. This point is generally not measured so this constitutes an extrapolation (Figure 5). The Bingham rheological parameters, yield stress and plastic viscosity, will characterize the flow curve within a range of shear rates if it is not linear in a wide range of shear rates, as shown in Figure 5.

$$s = s_B + h_{pl} \dot{\gamma} \quad [1]$$

where s = shear stress, s_B = yield stress, h_{pl} = plastic viscosity, and $\dot{\gamma}$ = shear rate.

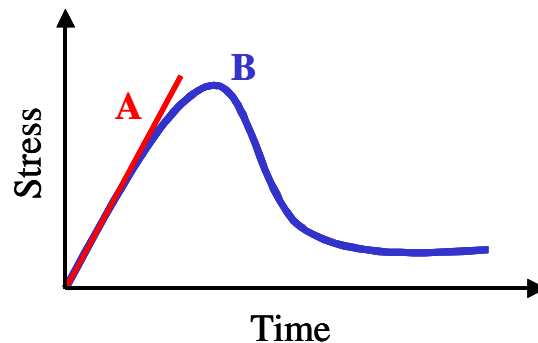


Figure 4: Stress growth schematic. Point A is the end of the linear portion, i.e., elastic limit, and it is considered to be the static yield stress point. Point B is the peak stress associated with the dynamic yield stress and it is taken as an approximation of the true yield stress as it is easier to determine than point A. [19, 20]

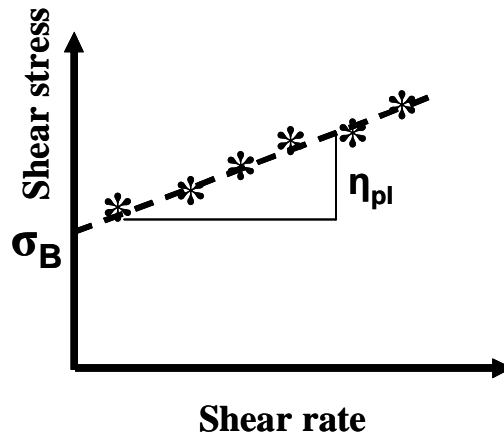


Figure 5: Bingham model and calculation of the plastic viscosity and yield stress.

The configurations of the rotational rheometer (21) used were:

- Coaxial: gap of 2.5 mm, a cup diameter of 43 mm and a bob diameter of 38 mm. The length of the bob is 55 mm
- Parallel plate: diameter 35 mm, serrated surface and with gap varying from 0.4 mm to 1 mm as indicated in the test. The gap was optimized to be 0.8 mm.

The shear rate range was 0 s^{-1} to 50 s^{-1} , as determined by analytical calculation from the rotational speed (22). It was established that the best results, e.g. the most linear curve of shear stress-shear rate, are obtained with the maximum shear rate at 20 s^{-1} . The induced shear stresses were measured, corresponding to 10 shear rates when increasing the rotational velocity, and 10 levels when decreasing the rotational velocity. Each measured point was recorded after the shear stress reached equilibrium or after 20 s, whichever occurred first. The descending data were regressed to a line using ordinary least squares (Figure 5), and the slope and intercept were calculated. The plastic viscosity and yield stress were calculated using the Bingham equation (Eq. (1)).

To measure the oil viscosity at various temperatures a vibrational viscometer was used. This type of viscometer can only be used with a Newtonian fluid whose viscosity does not change with shear rate. It consists of a rod that is immersed in the fluid to be measured. The rod vibrates at a high frequency, and it measures the damping due to the fluid. The amplitude is small and the power consumed is converted to viscosity (23). The temperature can also be measured at the same time. The viscosity measured is the kinematic viscosity or the viscosity/density of the fluid.

4.4 Grinding Process

As stated in the section *Historical Information*, the silica powder for the reference material has been produced by grinding ASTM C778 graded sand in a laboratory ball mill loaded with approximately 90 kg (200 lbs) of agate stones, replacing the steel balls usually used in a ball mill, and 45 kg (100 lbs) of ASTM C778 graded sand. A large quantity of stones was acquired over 30 years ago and is still used today.

After 20 h of grinding, the material from the ball mill is scooped by hand without removing the stones and placed in a 5-gal bucket. Then most of the stones are removed and the rest of the material still in the ball mill is placed in another 5-gal bucket. For Lot J, the two buckets were labeled *Can A* and *Can B*. Figure 6 shows that the particle size distribution from *Can A* and *Can B* are nominally identical (13). It should be noted that in the procedure used by CCRL, the two cans obtained from the grinding process are not blended before preparing bags for shipment. These bags are usually of 500 g each. The small difference measured in the PSD of the two can justify the procedure of not blending the two cans.

After the grinding process, the stones are washed, dried, and examined visually. The stones found to be damaged are discarded and replaced by new stones from a reserve. The damage types are cracked, broken, or spalled stones. The granulometry of the stones is unknown and the damaged stones are replaced by similar sized stones.

In this study, material from lot H (2007) was available and lots I (2008) and J (2009) were produced. Data from Lot G (2004) were also available.

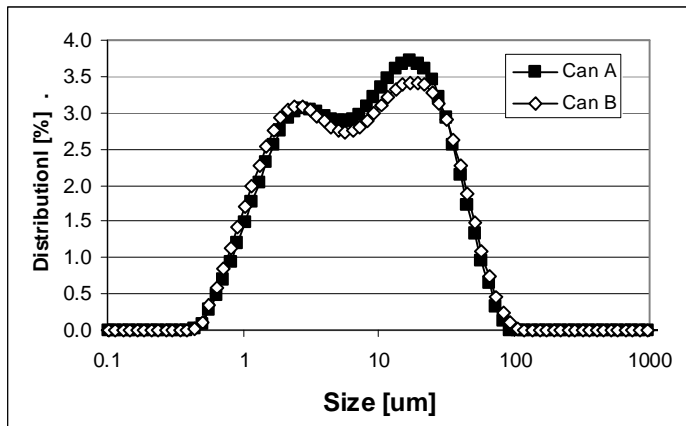


Figure 6: Particle size distribution of Lot J, *Can A* and *Can B*.

CHAPTER 5. Characteristics of Reference Material

5.1 Oil Viscosity

The viscosity of the oil currently used, *Drakeol 35*, as determined by a calibrated coaxial rheometer, was $0.1590 \text{ Pa}\cdot\text{s} \pm 0.0001 \text{ Pa}\cdot\text{s}$ at $23 \text{ }^\circ\text{C}$ (Table 1). The oil viscosity was also measured using a vibrational viscometer and a parallel plate rotational rheometer at various temperatures to compare with the manufacturer values. Figure 7 shows the results obtained. The values obtained with the coaxial rheometer are, within the error bars, identical to samples measured using the vibrational viscometer. Also, the values given by the manufacturer were 65.8 cSt ($0.057 \text{ Pa}\cdot\text{s}$) and 71.0 cSt ($0.062 \text{ Pa}\cdot\text{s}$) at $40 \text{ }^\circ\text{C}$, corresponding to an average viscosity of $0.059 \text{ Pa}\cdot\text{s}$ (marked by μ in Figure 7) which was also within the measurement uncertainty. This implies an average change in viscosity of $0.0076 \text{ Pa}\cdot\text{s}/^\circ\text{C}$ between $15 \text{ }^\circ\text{C}$ and $40 \text{ }^\circ\text{C}$. The viscosity will change by $0.038 \text{ Pa}\cdot\text{s}$ between $20 \text{ }^\circ\text{C}$ and $25 \text{ }^\circ\text{C}$ which is the specified range of testing. It should be determined if such a difference in viscosity will affect the flow table values.

It should be noted that the viscosity of this oil is approximately the same as the *Primol 355* oil used initially (1965), namely a viscosity of $0.15 \text{ Pa}\cdot\text{s}$ at $25 \text{ }^\circ\text{C}$ (see section *Historical Information*).

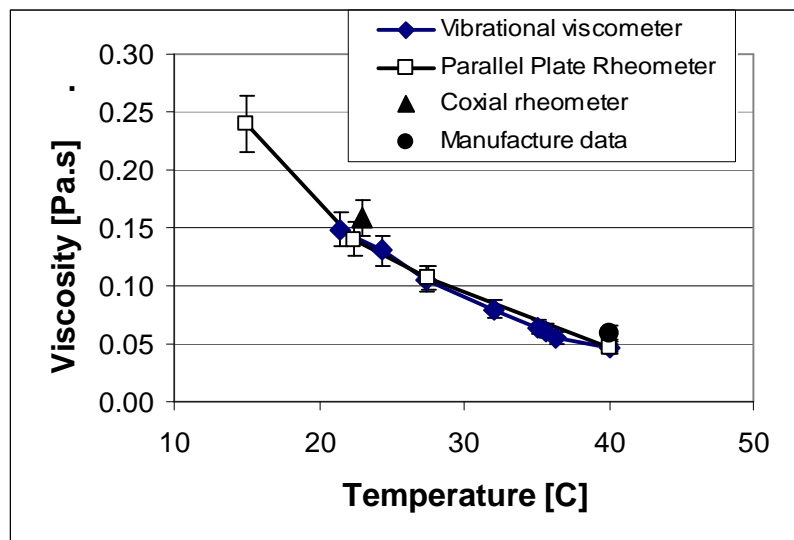


Figure 7: Viscosity of the *Drakeol 35* oil versus temperature using the various rheometers. The uncertainty is estimated at 10 % and it is indicated with error bars on the graph.

5.2 Powder Characteristics

Four lots were available for this study. Figure 8 shows the PSD distributions as measured by laser diffraction (13) for lots G (2004), H (2007), I (2008) and J (2009). Lots H, I and J were measured for this study while G data were provided by CCRL. These distributions are remarkably similar, despite the fact that the grinding stones size distribution in the ball mill is not controlled. On the other hand, it is clear that one value, whether the fineness by the sub-sieve

Fisher sizer or d_{50} from the laser diffraction, could not adequately describe this distribution as it has two peaks. The only significant difference is that there are some variations in the finer particles.

After each hour of grinding of Lot I, up to 12 h, a small sample, about 2 g to 5 g, was removed from the ball mill and placed in a labeled vial. After each of the following grinding durations, 12 h, 14 h, 16 h, 18 h, and 20 h, a sample of about 600 g was extracted.

During the grinding of lot J about 1 kg of material was taken from the ball mill at specific times (8 h, 12 h, 16 h, 17 h and 20 h). Then, the material from the ball mill was scooped by hand without removing the stones and placed in a 5-gal bucket labeled *Can A*. Then, most of the stones were removed and the rest of the material was placed in *Can B*. As there is no difference in the PSD of *Can A* and *B* (Figure 6), in the rest of this project, either can will be used for testing.

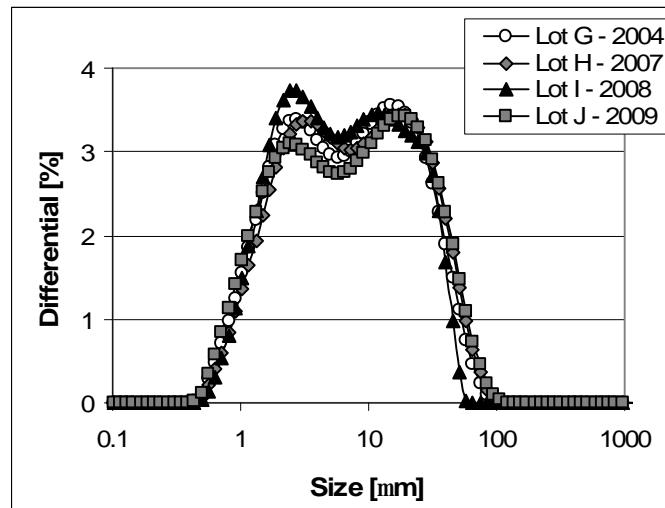


Figure 8: Particle size distribution for the four different lots (G to J).

The particle size distributions of all samples collected during grinding (lot I and J) were measured by laser diffraction and are shown in Figure 9 and Figure 10, respectively. As the ball mill was open and a small vial of material was extracted without any attempt to homogenize the whole batch, there is some doubt whether the samples measured represent the whole batch at the time of extraction. Nevertheless, in both cases (lot I and J), 16 h to 17 h of grinding yielded the same distribution as the final product. Therefore, the first simplification in the process to produce the powder would be to reduce the grinding time to 17 h.

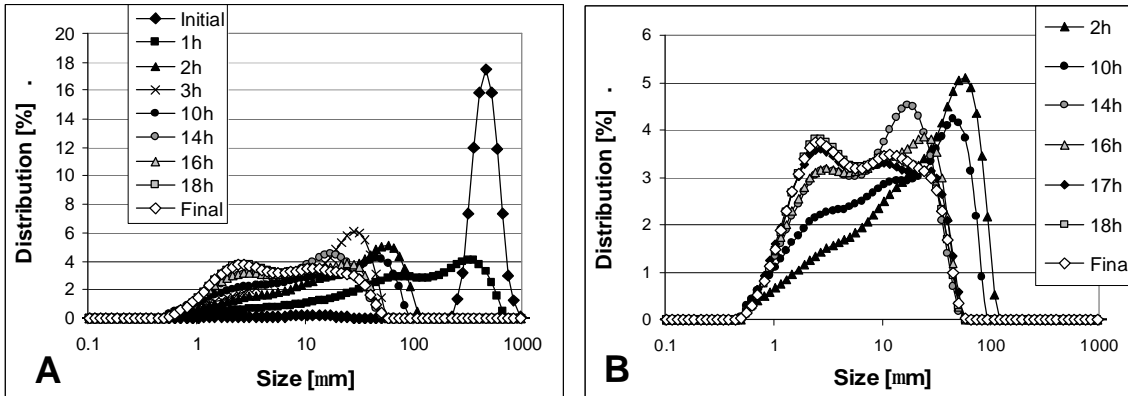


Figure 9: PSD for lot I after different durations of grinding. A) all data; B) selected data to enhance readability of data after 14h.

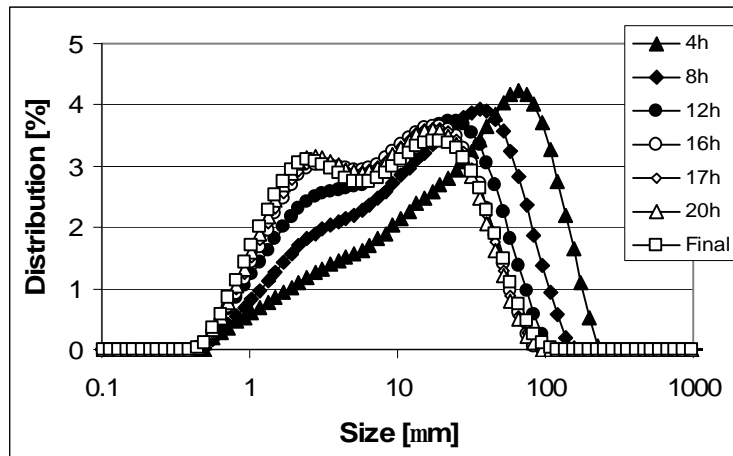


Figure 10: PSD for Lot J after different durations of grinding.

5.3 Flow Properties of the Reference Material

5.3.1 Influence of Grinding Duration

For lot J, 1 kg of material was collected at various grinding stages, namely after 8 h, 12 h, 16 h, 17 h and 20 h of grinding and final product, *Can B*. Therefore, enough material was available to conduct flow table tests and rheological tests for each grinding duration and for *Can B*. Figure 11 shows the results obtained as a function of grinding time.

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

The PSD (Figure 10) and the median, d_{50} , do not change significantly after 17 h as well. The rheological measurements were done using a parallel plate rotational rheometer with a gap of 0.4 mm. The results are shown in Figure 11. The yield stress values have not stabilized at 17 h while the plastic viscosity seems to have reached the value of *Can B* after 16 h within the error bars. The difference in yield stress is in contrast with the data obtained with the flow table, implying that some rheological measurements are more sensitive to small changes in the powder particle size distribution than the flow table.

In the section *Proposed New Procedure*, a better and more reproducible rheological procedure is described. Therefore, these rheological data should be considered as indicative of a trend only.

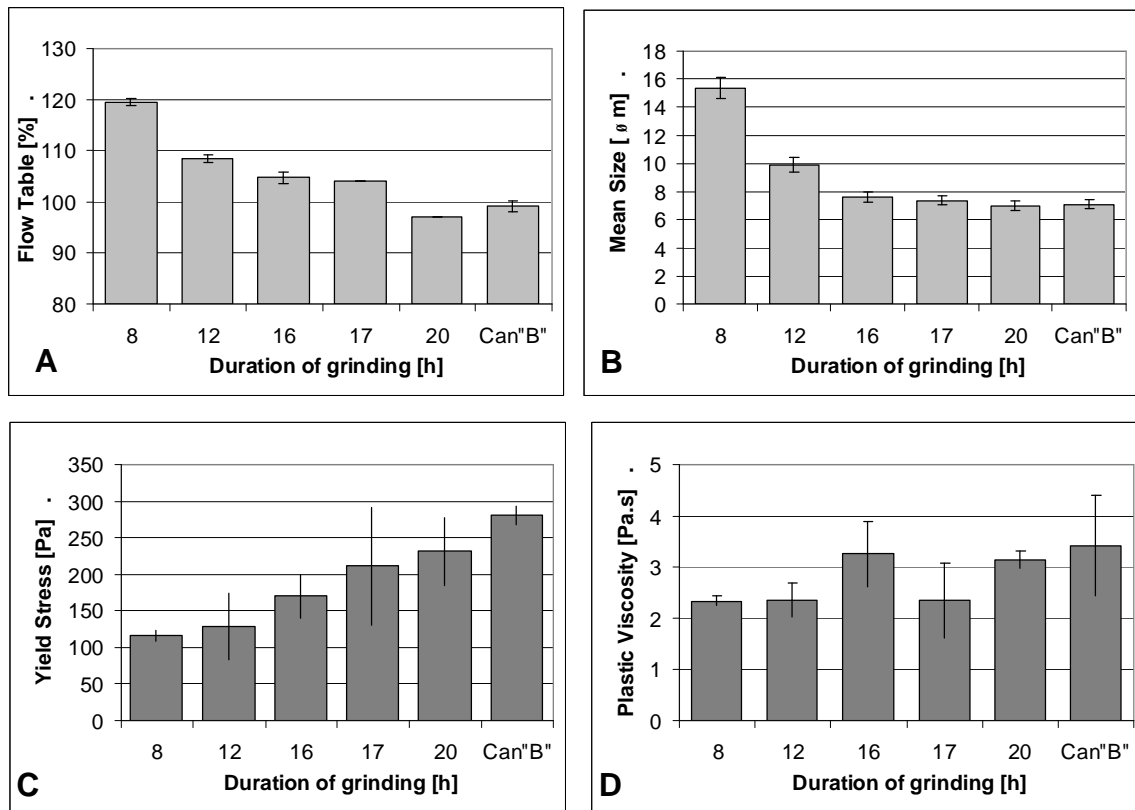


Figure 11: Influence of grinding duration on flow table results (A), particle size (B), yield stress (C), and plastic viscosity (D). Lot J was used here. The error bars represent one standard deviation of the mean. Influence of Test Temperature

A mixture with the composition of 500 g of ground silica (Lot H) and 350 g of *Drakeol 35* was prepared. The rheological properties of the mixture were examined at various temperatures (15 °C to 40 °C). Figure 12 shows the results obtained. The flow table values were also determined and the results were:

- At 21 °C as measured on the flow table: 63 % (only 10 drops)
- At 30 °C as measured on the flow table: 74 % (only 10 drops)

This would imply an increase of 18 % in flow value, while the rheological parameters increased by 79% for the viscosity and 24% for the yield stress as measured by the rheometer with a temperature controlled at 21 °C and at 30 °C. It seems in these preliminary data that the flow table is less sensitive to temperature changes than the rheological properties. But it should be noted that the flow table data were only conducted with 10 drops instead of 25. The difficulty in these tests is to maintain the temperature of the material during the flow table test. The table itself needs to be at the same temperature; however, since it is made of metal and as soon as it is retrieved from the chamber at the desired temperature it starts to cool down from 40 °C or to heat up from 15 °C to the laboratory temperature of 23 °C. The selection of only 10 drops was made to reduce the changes in temperature during the measurements of the flow table. On the other hand, the rheometer has a build in temperature controller leading to a stable temperature setting during testing. Therefore, these observations on the flow table should be considered only as an indication of trends.

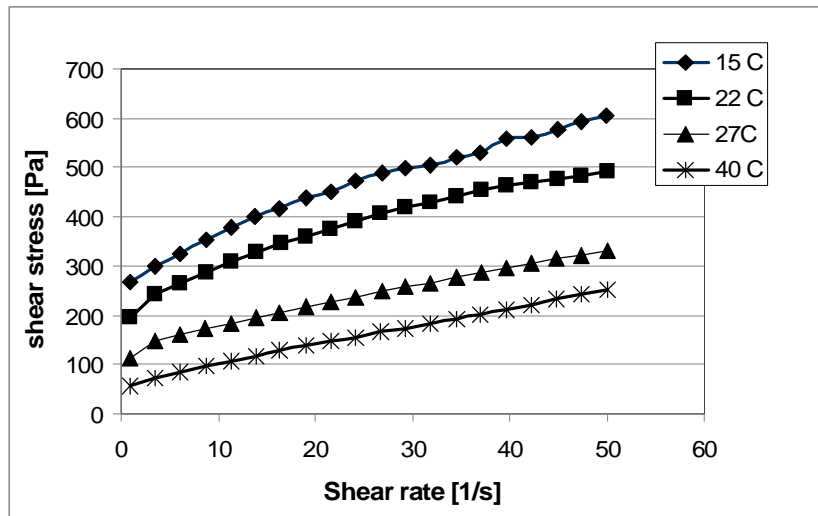


Figure 12: Shear stress vs. shear rate for Lot H + Drakeol 35 mixture at various temperatures. These data were not generated using the procedure proposed in the section Proposed New Procedure. (These tests were performed only once and are given here are indication only so not uncertainty is determined)

5.3.2 Influence of Oil Viscosity

The same mixture was prepared with each of the three oils (*Drakeol 35, 600, 21*). The proportions were 500 g of ground silica (lot J) and 350 g of oil. For each mixture, the following measurements were conducted: flow table, stress growth to determine dynamic yield stress and the Bingham parameters of yield stress and plastic viscosity.

Figure 13 shows the relationship between the flow table and the various rheological parameters for the three mixtures. As expected, as the flow table value increases, all the parameters decrease. It could be noticed that the range of oil viscosity was large, i.e., an increase of 177 % of the lower viscosity, or a range of nearly a factor of 2. On the other hand, the changes in flow table and in the rheological parameters are much smaller, as indicated by growth of dynamic yield stress by 6 %, Bingham yield stress by 16 %, Bingham viscosity by 27 %, and

flow table by 10 %. It could be stated that a small change in the oil viscosity will not affect the rheological properties or the flow table value. This is desirable to avoid fluctuation in the values with changes in oil viscosity due to temperature.

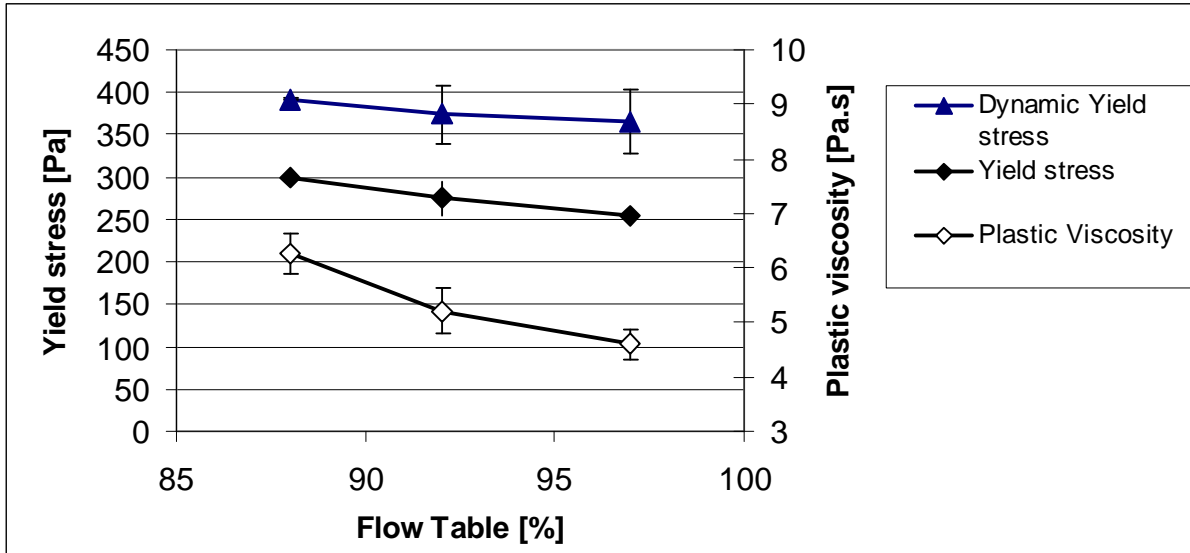


Figure 13: Relationship between the flow table and rheological parameters. The flow table values are in the inverse order than the oil viscosity, e.g. *Drakeol 21* has a flow of 88 %. These data were produced using the procedure described in section *Proposed New Procedure*. The error bars are the one standard deviation.

5.3.3 Influence of Particle Size Distribution

Researchers have found that the PSD influences rheological properties (24.) 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 3). Table 2 shows the PSD characteristics extracted from the data in Figure 3 and the flow table results obtained using the same mixture proportions. The mixture proportions used were 500 g of silica 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 by mass values, respectively. The span represents the width of the distribution. Figure 14 shows the same data as in Table 2 but in graphical mode.

Figure 15 shows flow table results as a function of median size. 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, these results are 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 92 %). This is seen by *Ground Silica*, *Mixture A*, and *Mixture B* in Table 2. *Mixture A* seems to have a higher flow than *Ground Silica* and *Mixture B* since its d_{90} is higher, implying fewer fine particles. From Table 2, it seems that the best powder combination to replace the ground silica would be *Mixture B*.

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 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.

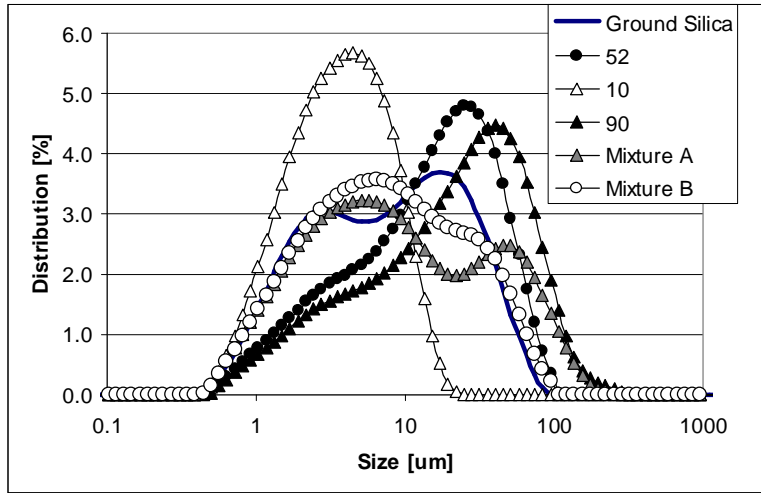


Figure 14: Particle size distribution of the powders investigated. See Table 2 for composition of Mixture A and B. The uncertainty on the measurement is estimated to be 5 %.

Table 2: Particle Size characteristics and flow table results. The uncertainty on the flow table measurement is estimated to be 5 % and for the PSD is 1 %.

Material	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	79
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	92
Mixture B	6.7	1.4	34.8	5.0	76

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

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

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

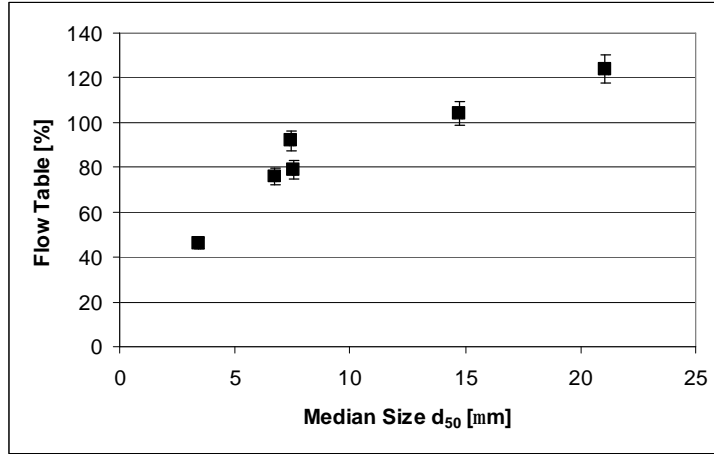


Figure 15: Median size particle and flow table results. The error bars are the one standard deviation.

CHAPTER 6. Relationship between Rheology and Flow Table

One of the goals of this study was to be able to characterize the flow table reference material by using rheological measurements. The advantage is that rheological measurements can be done using any calibrated rheometer. Rotational rheometer calibration is usually verified using standard oils. These oils are certified using various independent measurements. On the other hand, the flow table reference material value is assigned using only one specific flow table that is over 30 years old. The question to be answered is which rheological parameter will best correlate with the flow table values. Then a series of tests should be conducted using the standard flow table and the exact rheological value should be assigned. After that assignment, the flow table reference material can be certified using rheological measurements.

From all the tests performed in for this study using both ground silica mixtures and two-powder mixtures, and various types and amounts of oil, Figure 16 was generated. It can be seen that the yield stress, both dynamic and Bingham, are highly correlated with the flow table values, with R^2 factors for a linear relationship of 0.91 and 0.93, respectively. On the other hand the R^2 factor for the plastic viscosity is only 0.71.

Therefore, a set of tests was conducted using the standard flow table material and measuring the rheological properties to determine the target dynamic yield stress and Bingham yield stress. The results are shown in the section *Reproducibility and Stability*. The methodology developed during this study to obtain the rheological properties is described in the section *Proposed New Procedure*. These tests need to be repeated when the standard flow table is available to ensure that the mixture of oil and ground silica is optimized.

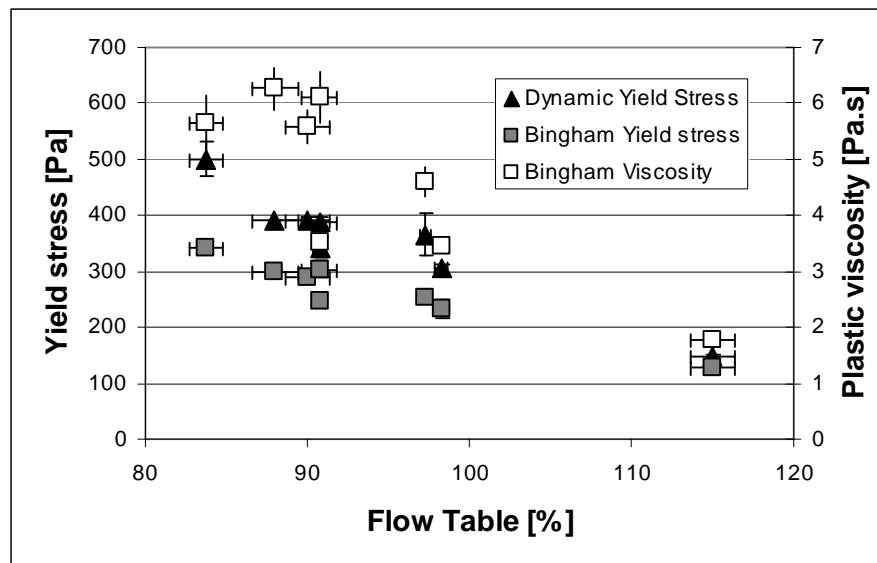


Figure 16: Relationship between rheological parameters and flow table values. The error bars are the one standard deviation.

CHAPTER 7. Reproducibility and Stability

During the duration of this study various batches of the reference material were prepared by mixing 350g of *Drakeol 35* and 500 g of the ground silica. At the end of the study, three batches were available that were 1.5 year, 0.5 year and just 1 day old. Each batch was stored undisturbed and no bleeding of the mixture was observed even after months of storage. All batches were tested during two consecutive days. The more recent batch was tested up to 29 days. The tests performed were flow table and rheological measurements that included stress growth (dynamic yield stress) and Bingham parameters. Table 3 shows the results obtained. The CV is the ratio of the standard deviation and the average reported in percent. The values presented are the average of several tests:

- Flow table was measured twice
- Rheological parameters were measured 5 times each. A new sample was placed on the rheometer each time.

It should be noted that with the laboratory flow table none of the mixtures have the 104 % flow, but the flow is very consistent as it is within the 5% error required by ASTM test method. The following observations could be derived:

- For the mixture that is less than 8 days old at testing, the rheological values have a smaller CV of only 3 % or less. In general, the flow table error is not significantly affected by the age of the mixture.
- The flow table was not greased for a long period of time prior to these tests. This explains the low measured flow values of less than 90%. Once the table was greased and cleaned the flows increased as shown in Table 4. However, even after cleaning, the flow does not reach 104 %. It should be noted that this silica was never tested on the reference flow table and therefore the flow value on the reference table is not know. Maybe by adjusting the oil content (as done for lot H- Appendix A) would have yielded the 104 % target value; however, it was preferred to keep the oil content constant to be able to compare mixes with same solid concentration.
- Table 4 shows that there is a slight increase of the flow for the mixture prepared over 1 year ago, but the value is still within the error requested by ASTM.

Table 3 also provides the reproducibility of the rheological properties. As indicated from the table the rheological data are very consistent.

Table 3: Results of measurements on three different batches. All tests done in August 2010

Age at time of testing [d]	Rheological parameters							
	Flow Table [%]		Dynamic Yield Stress [Pa]		Bingham Viscosity [Pa.s]		Bingham Yield stress [Pa]	
	Value	ST. Dev	Value	ST. Dev	Value	ST. Dev	Value	ST. Dev
Batch prepared February 2009								
540	83.8	0.4	307	28	4.6	0.3	239	3
541	82.0	2.8	325	3	4.8	0.2	249	13
Batch prepared February 2010								
162	74.0	2.1	378	19	5.7	0.3	284	6
163	74.5	1.4	406	41	5.9	0.4	292	11
Batch prepared August 2010								
1	81.3	1.1	381	15	5.0	0.3	283	10
2	76.0	0.0	381	14	4.9	0.3	281	19
7	82.0	0.0	394	37	5.1	0.4	279	11
8	75.5	0.7	402	25	5.3	0.3	286	9
29	78.3	0.4	390	8	5.6	0.3	288	5
Overall - all data of three batches								
Average	78.6		372		5.2		274	
St. Dev.	4.2		36		0.4		19	
CV	5%		10%		8%		7%	
Only fresh batch prepared in August 2010								
Average	78.6		389		5.2		283	
St. Dev.	3.0		9		0.3		4	
CV	4%		2%		5%		1%	

Table 4: Flow measured after the table was cleaned and greased.

Age at time of testing [d]	Flow Table [%]	
	Value	ST. Dev
Batch prepared February 2009		
577	94.5	0.7
Batch prepared February 2010		
195	90.8	1.1
Batch prepared August 2010		
32	90.0	1.4
Overall - all data of three batches		
Average	91.8	
St. Dev.	2.4	
CV	3%	

To determine the validity of the results obtained, some limited tests were conducted using both the flow table at NIST (used for all the tests reported in this report) and the CCRL flow table traditionally used to develop the reference material. Flow measurements were conducted with a standard mixture (ground silica and Drakeol 35) using the following set-ups:

1. NIST flow table and NIST caliper and cone
2. NIST flow table and CCRL caliper and cone
3. CCRL flow table and NIST caliper and cone
4. CCRL flow table and CCRL caliper and cone

The materials used were ground silica used in Table 5 and a new batch with the same material (ground silica and Drakeol 35) with the same proportion (500 g of silica and 350 g of oil) were used. Table 5 shows the results obtained. The overall average for the new mixture is $96 \% \pm 3 \%$. Therefore, it is clear that all the data in this table are well within the error of the measurement (5 %). It can be stated that NIST table is properly maintained and the data obtained in this report are valid.

Table 5: Results comparing CCRL and NIST flow tables. See text for the test description

Test	Test 1	Test 2	Test 3	Test 4
New mixture [%]	95 ± 1	94 ± 2	99 ± 1	99 ± 1
Old mixture (same as prepared in February 2009 in Table 4)	95 ± 0	97 ± 0		

The low values obtained with the reference material in this report are due to the fact that the oil was kept constant, to be able to compare mixes with same solid concentration. Therefore, the conclusions reached regarding the use of a bought powder instead of grinding are valid. In theory any value obtained can be used as reference data. Two possible actions could be taken, either the oil or the flow are kept constant. Traditionally the oil was adjusted to obtain the same flow of 104 %, while in this report the oil was kept constant.
Proposed New 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 significantly (Table 2).

On the other hand, while the oil is purchased as-is with manufacturer quality control, 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.

The PSD of the powders selected are shown in Table 2 and Figure 3. *Min-u-sil 10* is a fine powder and the other two are coarser. Using the distributions of these powders, a spreadsheet was developed to calculate the combined distribution of two powders, one fine, and one coarse. The two mixtures are labeled “10 and 52” (44% *Min-u-sil 10* and 56% *Sil-co-sil 52* by mass) and “10 and 90” (43% *Min-u-sil 10* and 57% *Sil-co-sil 90* by mass) in Figure 17. These distributions were obtained by varying the proportion of each component to attempt to match the *ground silica* distribution. From Figure 17, it can be seen that either of the two simulations is able to match the ground silica, the span is perfectly matched but the peaks are slightly off.

The next step was to verify that the calculated distribution matches the measured PSD. The mixtures were prepared, blended using a Turbula¹, and the PSD was measured by laser diffraction. The composition of the blends of powders are:

- Mixture A is equal to “10 and 90” (43 % *Min-u-sil 10* and 57 % *Sil-co-sil 90* by mass)
- Mixture B is equal to “10 and 52” (44 % *Min-u-sil 10* and 56 % *Sil-co-sil 52* by mass)

From Figure 17, it can be seen that the measured values match very well the calculated distribution. Therefore, we could calculate other compositions such as Mixture C (33 % *Min-u-sil 10* and 67 % *Sil-co-sil 52* by mass) shown in Figure 18. These three mixtures were tested in attempt to obtain the same flow table value.

From Figure 18, it can be seen that *Mixture B* and *Mixture C* have the same particle size range (with an error of 3.5 μm) as the ground silica. The main difference between these two mixtures is the relative proportion of fine and coarse. *Mixture C* has less fine particles than *Mixture B*.

All the powder blends were prepared with oil *Drakeol 35* in the amounts shown in Table 5. The flow table and rheological tests were performed on all mixtures. Table 6 shows the results. A relative factor was calculated for each value. This relative factor is the ratio in percentage of value of the proposed mixtures over the ground silica mixture. The values for the ground silica were the average calculated from the reproducibility tests (Table 4). Therefore, a relative factor of (90 – 110) % for the rheological values and (95 – 105) % for the flow table should be desirable. The flow table should have a lower relative factor to meet the ASTM uncertainty requirement.

The following observations could be reached:

- The best mixture is “*Mixture B with 400g of oil*” as both the flow is within 5 % and the yield stresses are within 10 % of those of the ground silica. The plastic viscosity does not correlate as well with flow data (see section *Relationship Between Rheology and Flow Table*).
- The issue with *Mixture B* is that it uses more oil than the current reference material. To reduce the amount of oil if necessary, *Mixture A* might be an option as it has a higher flow table value and thus the oil can be reduced.
- The reduced flow table value of *Mixture C* could be explained by the significant increase in its yield stresses both dynamic and Bingham.

¹ A 3D mixer device that allows a material contained in jar to be tumbled and rolled at the same time. A Turbula was used for this mixing.

In summary, it seems that *Mixture B with 400 g of oil* is the optimized mixture with rheological properties within 10% and the flow table within 5 % of the ground silica based mixture.

A rough cost analysis shows that this new reference material would be economically advantageous. From *US Silica* information, if the materials are bought in pallets, the costs of the sands are:

- Standard sand ASTM C778: \$3/kg (\$1.40/lb)
- *Min-u-sil 10* and *Sil-co-sil 52 or 90*: \$0.44/kg (\$0.20/lb)

It is a decrease of 85 % of the cost of raw material. The oil cost is about \$2.60/L (\$10/gal). Taking into account the density (Table 1), 350 g of oil will cost \$1.09 and 400 g will cost \$1.24. The increased cost of the oil (\$ 0.16) does not offset the saving in the materials. To these raw material prices, the cost of processing needs to be added. For the ASTM C778 standard sand, the cost of grinding needs to be considered and for the *Mixture B*, the material needs to be blended, although it could be sent as is and blended by the customer. These processing costs are not known but a guess would be that it is cheaper to blend than to grind. Considering the huge difference in cost of the raw materials, *Mixture B or C* should be significantly less expensive than the present ground silica produced.

Therefore, it was shown that a blend of two commercially available powders could be used to substitute the in house ground silica, possibly at a significant lower cost.

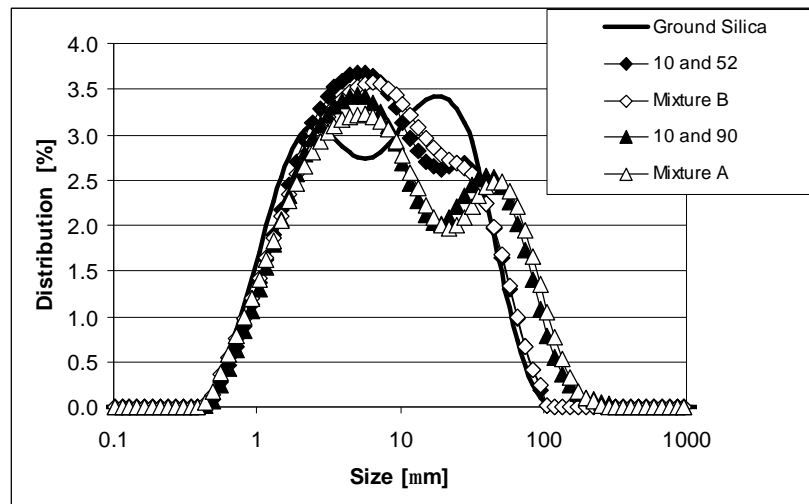


Figure 17: PSD of combination of powders. “10 and 52” and “10 and 90” were simulated distribution (see text) and *Mixture A* and *Mixture B* were measured distributions.

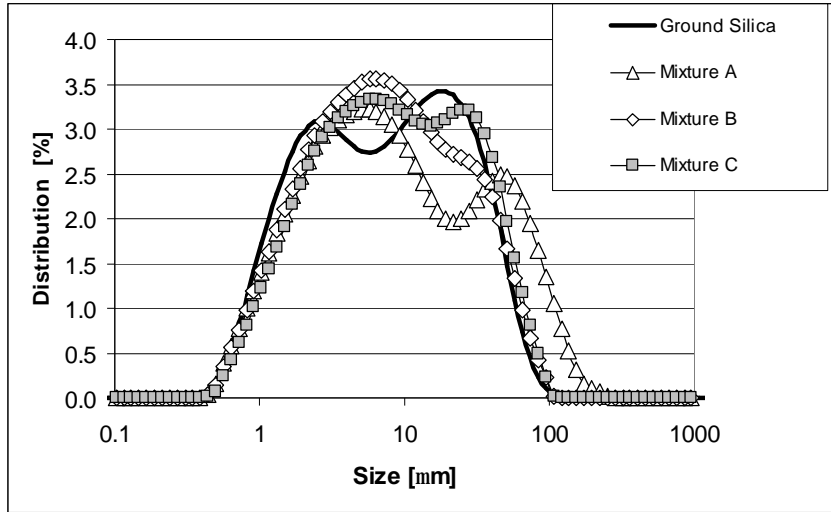


Figure 18: PSD of the combination powders *Mixture A* and *Mixture B* are measured PSD and *Mixture C* is only calculated.

Table 6: Rheological values for ground silica and mixtures of two-powders (PSD in Figure 18)

Material	Flow Table ² [%]		Dynamic Yield stress [Pa]			Bingham Yield stress [Pa]			Plastic viscosity [Pa.s]		
	Value	Relative	value	error	Relative to GS	value	error	Relative to GS	value	error	Relative to GS
Ground silica average	91.8		372	36		274	19		5.2	0.4	
Mixture A w/ 400 g of oil	98.3	107%	304	9	82%	233	16	85%	3.4	0.1	65%
Mixture B w/ 420 g of oil	115	125%	147	4	39%	127	7	46%	1.8	0.2	35%
Mixture B w/ 400g of oil	90.8	98%	343	18	92%	247	3	90%	3.5	0.2	67%
Mixture C w/ 350g of oil	83.8	90%	500	31	134%	343	11	125%	5.6	0.5	108%

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

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

Mixture C: 33% Min-u-sil 10 and 67% Sil-co-sil 52 by mass

² The flow table values reported here differ from the values in Table 2 because they were performed just after the flow table was greased

CHAPTER 8. Proposed New Procedure

The suggestion for quality control of the reference material could be determined by rheological data, especially if the dynamic yield stress and the Bingham yield stress are considered (see section *Relationship Between Rheology and Flow Table*). The following procedure to measure the properties of the material should be followed.

1. The oil can be bought in bulk as usual and its viscosity checked by vibration viscometer.
2. The powder can be purchased by blending two commercially available powders. CCRL needs to determine whether the amount of oil or the span of the distribution is more important.
3. The rheological properties of the material are determined using a calibrated parallel plate rheometer, using the following set-up:
 - Parallel plate rheometer with serrated plates
 - Gap between plates is 0.8 mm
 - Stress growth sequence: a 30 s rest period followed by a shear rate 0.2 s^{-1} for 300 s. The dynamic yield stress is the peak of the shear stress vs. time curve.
 - Bingham: 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 yield stress and plastic viscosity are calculated from the shear rate - shear stress curve with the decreasing shear rate. A linear regression allows the calculation of the two parameters.

The target values for the rheological properties need to be finalized using CCRL standard flow table. The advantage of using a rheological target value is that a rheometer is calibrated independent of reference flow material, by checking torque and rotational speed based on well established methods.

CHAPTER 9. Summary and Conclusions

The flow table is a very commonly used standard test that is easy to perform. According to the ASTM/AASHTO standard test method, a reference material, composed of silica powder and oil, is needed to calibrate the flow table. This reference material is tedious to produce, and needs to be characterized using fundamental measurements to ensure that there is no long-term bias from one batch to the next due to mechanical wear. The influence of oil viscosity, temperature, and grinding were examined and it was found that the major factor affecting the flow table value is the particle size distribution especially when it is widely changed. It was also determined that there is a strong correlation between the dynamic or Bingham yield stress and flow table values. This correlation will allow, in the future, the characterization of the reference material using rheological measurements. The procedure for such rheological measurements was described.

The main deliverables from this study are:

- Grinding of the ASTM C778 graded sand could be reduced to 17 h, a reduction of 3 h
- The in house ground silica could be replaced by a blend of two commercially available powders, reducing labor and cost. The final composition needs to be adjusted using CCRL reference flow table and decision needs to be made whether the amount of oil needs to be maintained at 350 g or not.
- A methodology was developed to measure the rheological properties of the reference material based on strong correlation between flow table values and dynamic or Bingham yield stresses.

In conclusion, this study yielded two very important findings that are part of the deliverables:

1. The powder used for the reference material can be purchased at a significant lower cost (over 85 % reduction of cost) and when the higher content of oil is factored in, the cost reduction is still 60 %. This reduction does not take into account the reduction in production as it completely eliminates the need for grinding the powder.
2. The reference material can be developed and its characteristics monitored using an independent measurements by using the rotational rheometer (see section “Proposed new procedure”). Presently, changes in the flow table values for a reference material could be linked either to the flow table device malfunctioning or to the reference material variation with time or composition. Instead, the rotational rheometer could be calibrated independently from the reference material to be characterized. Thus, ensuring that a change in the reference flow table value is due to the flow table device and not to the material.

These results will lead to a more reliable, less expensive, more environmental friendly production (no grinding) of the reference material.

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