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.

Designation: C1752 – 17

Standard Guide for
Measuring Physical and Rheological Properties of Radioactive Solutions, Slurries, and Sludges[1]

This standard is issued under the fixed designation C1752; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 *Intent:*

1.1.1 The intent of this guide is to provide guidance for the measurement and calculation of physical and rheological properties of radioactive solutions, slurries, and sludges as well as simulants designed to model the properties of these radioactive materials.

1.2 *Applicability:*

1.2.1 This guide is intended for measurement of mass and volume of the solution, slurries, and sludges as well as dissolved solids content in the liquid fraction and solids content associated with the slurries and sludges. Particle size distribution is also measured.

1.2.2 This guide identifies the data required and the equations recommended for calculation of density (bulk, settled solids, supernatant, and centrifuged solids), settling rate, volume and weight percent of the centrifuged solids and settled solids, and the weight percent undissolved solids, dissolved solids, and total oxides.

1.2.3 This guide is intended for measurement of shear strength and shear stress as a function of shear rate.

1.2.4 Rheological property measurement guidelines in this guide are limited to rotational rheometers.

1.2.5 This guide is limited to measurements of viscous and incipient flow and does not include oscillatory rheometry.

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

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C859 Terminology Relating to Nuclear Materials
C1751 Guide for Sampling Radioactive Tank Waste
D1193 Specification for Reagent Water

2.2 *Other Standards:*[3]

ISO13320;2009 "Particle Size Analysis—Laser Diffraction Methods. Part 1: General Principles," International Organization for Standardization, Geneva, Switzerland (1999)

3. Terminology

3.1 *Definitions:*

3.1.1 *apparent viscosity, n*—measured shear stress divided by the measured shear rate.

3.1.2 *density, n*—in the United States, mass per unit volume.

3.1.3 *interstitial solution, n*—in this guide, interstitial solution is the solution contained between the suspended, settled, or centrifuged solid particles of a sludge sample.

3.1.4 *Newtonian fluid, n*—a fluid whose apparent viscosity is independent of shear rate.

3.1.5 *non-Newtonian fluid, n*—a fluid whose apparent viscosity varies with shear rate.

3.1.6 *rheogram, n*—plot of shear stress versus shear rate.

3.1.6.1 *Discussion*—A rheogram is also called a flow curve.

3.1.7 *shear rate, n*—in laminar flow, the velocity gradient perpendicular to the direction of shear flow in parallel adjacent layers of a fluid body under shear force.

---

[1] This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.13 on Spent Fuel and High Level Waste.

Current edition approved July 1, 2017. Published August 2017. Originally approved in 2011. Last previous edition approved in 2011 as C1752 – 11. DOI: 10.1520/C1752-17.

[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] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, D. C. For suggestions on the testing of reagents not listed by the American Chemical Society see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U. K., and the United States Pharmacopeia and National Formulary, U. S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. United States

3.1.8 *shear strength, n*—maximum shear stress measured during incipient motion.

3.1.9 *shear stress, n*—shear force per unit area.

3.1.10 *sludge, n*—in this guide, sludge is wet solids having little or no standing liquid (that is, mud-like).

3.1.11 *slurry, n*—in this guide, a slurry is a mixture of solids and solution

3.1.12 *supernatant liquid, n*—a liquid phase overlying material deposited by settling, precipitation, or centrifugation.

3.1.13 *solids settling rate, n*—in this guide, the rate at which solids in a homogenized sample settle as defined by the change in the settled solids height as a function of time.

3.1.14 *volume percent (vol%) settled solids, n*—in this guide, the percentage of the volume of the slurry sample that the settled solids with its interstitial liquid occupy after settling for a specified time under one gravity.

3.1.15 *volume percent (vol%) centrifuged solids, n*—in this guide, the volume of the solids layer with its interstitial liquid that separates from the bulk slurry after centrifugation at a specified time and centrifugal force divided by the total sample volume on a percentage basis.

3.1.16 *weight percent (wt%) total oxides, n*—percentage of the mass of the bulk sample that remains after converting all non-volatile elements to oxides at 1000°C.

3.1.17 *weight percent (wt%) centrifuged solids, n*—in this guide, the mass of the solids layer with its interstitial liquid that separates from the bulk slurry after centrifugation at a specified time and centrifugal force divided by the total bulk slurry sample mass on a percentage basis.

3.1.18 *weight percent (wt%) dissolved solids, n*—mass of dissolved species in the supernatant liquid divided by the total mass of the supernatant liquid on a percentage basis.

3.1.19 *weight percent (wt%) dried solids, n*—percentage of the mass of the sample that remains after removing volatiles including free water by drying at 105 ± 5°C to a stable mass.

3.1.19.1 *Discussion*—Wt% total dried solids and wt% centrifuged dried solids are the wt% dried solids in the bulk sample and centrifuged solids, respectively.

3.1.20 *weight percent (wt%) undissolved solids, n*—calculated value reflecting the percent mass of solids remaining if all the supernatant liquid and interstitial solution were removed from the bulk slurry.

3.1.21 *yield stress, n*—minimum stress required to initiate fluid movement as determined by a flow curve using a rheological model.

3.2 *Nomenclature*:

3.2.1 b—Hershel-Bulkley power law exponent (unitless).

3.2.2 B—steady-state torque in a shear strength test in N·cm.

3.2.3 D—diameter of the shear vane in cm.

3.2.4 D_T—diameter of the shear strength sample cup in cm.

3.2.5 H—height of the shear vane in cm.

3.2.6 k—Hershel-Bulkley consistency coefficient in $\text{Pa}\cdot\text{s}^b$.

3.2.7 m—power law consistency coefficient in $\text{Pa}\cdot\text{s}^n$.

3.2.8 M_B—total mass of bulk slurry after centrifugation in g.

3.2.9 M_{CC}—mass of the centrifuge cone in g.

3.2.10 M_{CR}—mass of the crucible in g.

3.2.11 M_{CS}—mass of the centrifuged solids and their interstitial liquid in g.

3.2.12 M_{DCS}—mass of the oven dried centrifuged solids in g.

3.2.13 M_{DCL}—mass of the oven dried centrifuged supernatant liquid in g.

3.2.14 M_{FSC}—mass of the fired solids (1000°C) in the crucible in g.

3.2.15 M_{OSC}—mass of the oven dried solids (105°C) in the crucible in g.

3.2.16 M_S—mass of the decanted supernatant liquid after centrifugation in g.

3.2.17 M_{SL}—mass of the supernatant liquid after gravity settling in g.

3.2.18 M_{SS}—mass of the settled solids and interstitial liquid after gravity settling in g.

3.2.19 M_{VL}—mass of a subsample of the decanted centrifuged supernatant liquid in g.

3.2.20 M_{WCS}—mass of the wet sample in the crucible in g.

3.2.21 n—power law exponent (unitless).

3.2.22 N—rotational rate of the shear vane in revolutions per min.

3.2.23 P_{MCS}—percent mass of centrifuged solids with the associated interstitial liquid in the slurry.

3.2.24 P_{MDS}—percent mass of oven dried solids in the crucible.

3.2.25 P_{MOX}—percent mass of oxides in the slurry.

3.2.26 P_{MSS}—percent mass of settled solids with its associated interstitial liquid.

3.2.27 P_{MTS}—percent mass of total solids.

3.2.28 P_{MUS}—percent mass of undissolved solids in the slurry.

3.2.29 P_{ODS}—percent mass of oven dried solids in the centrifuged solids including interstitial liquid.

3.2.30 P_{VCS}—percent volume of centrifuged solids with its associated interstitial liquid in the slurry.

3.2.31 P_{VSS}—percent volume of settled solids with its associated interstitial liquid in the slurry.

3.2.32 r^2—correlation coefficient (unitless).

3.2.33 R_1—radius of the inner cylinder of the viscometer concentric cylinder geometry in cm.

3.2.34 R_2—radius of the outer cylinder of the viscometer concentric cylinder geometry in cm.

3.2.35 R_e—Reynolds number (unitless).

3.2.36 R_t—maximum torque in a shear strength test in N·cm.

3.2.37 V_B—total volume of bulk sample after centrifugation in mL.

3.2.38 V_{CS}—volume centrifuged solids and the associated interstitial liquid in mL.

3.2.39 V_S—volume of decanted supernatant liquid after centrifugation in mL.

3.2.40 V_{SB}—total volume of bulk sample after gravity settling in mL.

3.2.41 V_{SL}—volume of supernatant liquid after gravity settling in mL.

3.2.42 V_{SS}—volume of settled solids with its associated interstitial liquid after gravity settling in mL.

3.2.43 Z_1—height of the sample above the top of the immersed shear vane in cm.

3.2.44 Z_2—depth of the sample below the immersed shear vane in cm.

3.2.45 ρ_B—bulk density of slurry in g/mL.

3.2.46 ρ_{CS}—density of centrifuged solids in g/mL.

3.2.47 ρ_S—density of supernatant liquid in g/mL.

3.2.48 ρ_{SS}—density of settled solids in g/mL.

3.2.49 τ—shear stress in Pa.

3.2.50 τ_o—shear strength in Pa.

3.2.51 τ_o^B—Bingham yield stress in Pa.

3.2.52 τ_o^H—Herschel-Bulkley yield stress in Pa.

3.2.53 η—Newtonian viscosity in Pa·s.

3.2.54 η_p—plastic viscosity in Pa·s.

3.2.55 $\dot{\gamma}$—shear rate in s^{-1}.

3.2.56 μ—apparent viscosity in Pa·s.

3.3 Other unique terms used throughout the nuclear industry are defined in the terminology standard for the ASTM committee on the nuclear fuel cycle (Terminology C859).

4. Summary of Guide

4.1 Guidance for the measurement and calculation of physical and rheological properties of radioactive solutions, slurries, and sludges is provided. Methods are applicable to remote handling and measurement of samples with significant radiation doses.

4.2 Physical properties including bulk density, settled solids density, centrifuged solids density, supernatant density, settling rate, and volume and weight percent centrifuged and settled solids are determined by measuring the solids with their interstitial liquid and supernatant liquid masses and volumes as a function of time during settling and centrifugation of slurry and sludge samples.

4.3 Dissolved and undissolved solids content of solutions, slurries, and sludges as well as the solids separated by settling or centrifugation, or both, are calculated by measuring the mass of the sample prior to and after drying the sample. Oxide content is determined by measuring the mass of the sample before and after heating the sample in air at high enough temperatures (~1000°C) to oxidize the solids in the sample.

4.3.1 Automated moisture analyzers may be used to measure solids content after the automated method has been verified to provide comparable results for similar samples to the oven drying method described in this standard.

4.4 The flow behavior of solutions, slurries, and sludges is characterized by the shear strength, apparent viscosity, and yield stress of the material by measuring the shear stress of a sample as the sample shear rate is systematically varied.

5. Significance and Use

5.1 Measurements performed in this guide are limited to radioactive solutions, slurries, and sludges as well as simulants designed to model the properties of these radioactive materials.

5.2 Data obtained from the measurement and calculation of physical and rheological properties of radioactive solutions, slurries, and sludges are essential in developing appropriate simulants for design and testing of retrieval, transport, mixing, and storage systems for treatment of radioactive materials. These data also provide input parameters for modeling the flow behavior, processing, transport, safety, and storage of these radioactive materials.

5.3 Consistency in the handling of samples, measurement methods, and calculations is essential in obtaining reproducible results of rheological and physical property measurements.

5.4 This guide will be used to measure or calculate the physical properties listed below.

5.4.1 Bulk slurry density
5.4.2 Settled solids density
5.4.3 Centrifuged solids density
5.4.4 Supernatant density
5.4.5 Settling rate
5.4.6 Volume percent centrifuged solids
5.4.7 Volume percent settled solids after settling
5.4.8 Undissolved solids content
5.4.9 Dissolved solids content
5.4.10 Weight percent centrifuged solids
5.4.11 Weight percent total oxides
5.4.12 Solids content of the centrifuged solids
5.4.13 Total solids content

5.5 This guide describes the process of performing measurement of the rheological properties. The rheological measurements and calculations described in this guide are limited to shear strength, shear stress versus shear rate, apparent viscosity, consistency, and yield stress.

5.6 Due to the nature of some solutions, slurries, and sludges, not all of the measurements described in this standard may be applicable to all samples. For example, some sludges do not settle; therefore, settling rate measurements are not applicable for these samples.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all measurements. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water as defined by Type II of Specification D1193 or purer.

6.3 Viscosity standards certified to a national standards body should be used to calibrate the rheometer.

7. Hazards

7.1 Radiological hazards including external dose, internal dose, and contamination are present when handling radioactive solutions, slurries, and sludges.

7.2 Drying and muffle furnaces are maintained at high temperatures (105°C and 1000°C, respectively) during the measurement of solid and oxide content.

8. Sampling, Test Specimens, and Test Units

8.1 At least duplicate samples should be analyzed for physical and rheological property determinations.

8.2 Care should be taken to obtain representative samples during all sampling activities. Details on methods to obtain representative samples from slurries and sludges are provided in the Guide for Sampling Radioactive Tank Waste (Guide C1751).

9. Procedure

9.1 *Physical Properties Determination:*

9.1.1 Mix the sample to combine any separated liquid and solids phases.

9.1.2 Weigh and record the masses of the clean, volume-graduated containers for both the sample and replicate.

9.1.2.1 Volume-graduated labware, appropriate to accommodate 10 to 50 mL of sample, should be used when determining physical properties requiring volume measurements. Generally, this labware is either a graduated cylinder or a volume graduated centrifuge cone with a cap. For solutions and slurries with low solids content, such that the sample can be easily poured without smearing on the labware, a volumetric flask may be used for density measurements.

(1) Some samples may stick to the side of the volume-graduated labware. For these types of samples, it is preferred to perform the work in volume-graduated centrifuge cones. Volumes will normally be measured to ±0.2 mL when using a 15 mL centrifuge cone, and ±1 mL when using the 50 mL centrifuge cones. The recommended sample volumes are ≥ 10 mL and ≥ 30 mL for the 15 mL and 50 mL centrifuge cones, respectively.

9.1.2.2 If centrifuge cones are used as the volume-graduated container, measure the distance between the graduation marks on the cylindrical portion of each centrifuge cone (at least from the 10-mL to the 4-mL graduation marks on the 15-mL centrifuge cone or the 30-mL to the 10-mL graduation marks on the 50-mL centrifuge cone). Volume-graduated centrifuge cones should be cylindrical between 10 and 4 mL and 30 and 10 mL for 15-mL and 50-mL centrifuge cones, respectively. Record these data as well as the volume mark just above where the centrifuge cone departs from a cylindrical shape.

9.1.3 Transfer sub-samples into each of the containers and weigh the filled container. Record the total mass of each filled container.

9.1.4 Mobilize the settled solids in the container to obtain a homogenous sample. This may be done by shaking the container, using a vortex mixer, or a comparable method. Homogenization of thick sludges such as pastes may be difficult and require longer mixing times with more rigorous mixing systems.

9.1.5 Record the volume of the total sample and the volume of the settled solids as a function of settling time. If multiple solid layers are visible, record the volume at the interface of each solids layer as a function of time. Some samples may not settle; therefore, settling measurements may not be applicable for all samples.

9.1.5.1 Measurements should be recorded at appropriate time intervals based on the settling rate of the sample. The final measurement provides the data for the volume percent settled solids.

9.1.5.2 Volume data could be biased due to entrained gas as well as the inability to clearly measure the total sample volume due to material smeared on the sides of the centrifuge cone.

9.1.6 Centrifuge the cones containing the sample at a specific centrifugal force and time. Record the centrifugal force, time, volume of the total sample, and the volume of the centrifuged solids.

9.1.7 Decant the centrifuged supernatant liquid to a pre-weighed graduated cylinder and record the mass and volume of the supernatant liquid.

9.1.8 Weigh and record the mass of the centrifuge cone with the remaining solids (centrifuged solids) after decanting the supernatant liquid.

9.1.9 Transfer the decanted supernatant liquid to a pre-weighed vial with a lid. The vial shall be rated to at least 105°C. Weigh the vial with the transferred liquid. Record the masses of the vial and the vial plus the transferred liquid.

9.1.10 Air dry the solids and liquids overnight to minimize possible splattering during the oven drying steps.

9.1.11 Transfer the air dried samples (without lids) to a drying oven controlled at 105°C. Centrifuge cones used in this step should be rated for at least 105°C. Allow the samples to dry for at least 24 h.

9.1.12 Remove the dried solid and liquid samples from the oven and cap the vials and centrifuge cones. Allow the samples to cool for at least 10 min, or to ambient temperature, in a desiccate environment. Weigh the samples and record the masses.

9.1.13 Remove the lids and return the solid and liquid samples to the drying oven controlled at 105°C. Allow the samples to dry for at least another 24 h.

9.1.14 Repeat the drying and weighing steps (9.1.11 and 9.1.12) until a stable mass is achieved. A stable mass is defined as a change in mass between the last two measurements of less than 0.1 %. Record this mass which is the mass of the oven dried centrifuged solids.

9.1.14.1 Calculation of the total solids content, including both the dissolved and undissolved solids, is based upon the assumption that all of the volatile components lost during the drying process at 105°C are water molecules and that all of the water is lost during this drying process. Waters of hydration may not be lost at this temperature; therefore, they will bias the calculation of the total solids content high since the mass of the dried centrifuged solids will be high. Volatile organics may bias the total solids content low, because the mass of the dried centrifuge solids may be biased low.

9.1.15 An example of a data sheet for recording the physical property determination data is provided in Table 1.

9.2 *Total Oxides Content:*

9.2.1 Mix the bulk slurry sample to combine any separated liquid and solids phases.

9.2.2 Weigh (±0.001 g) pre-fired crucibles rated for at least 1200°C. Record this tare weight.

9.2.3 Transfer sub-samples of approximately three or more grams to each crucible. Weigh (±0.001 g) the samples in the crucibles and record the mass.

9.2.4 Air dry the material in the crucibles overnight to minimize splattering during the next drying step.

9.2.5 Transfer the filled crucibles to a drying oven controlled at 105°C and allow the samples to dry for 24 h. Record the time and date the samples were placed in the drying oven.

9.2.6 Remove the samples from the drying oven and transfer the sample to a dessicator. Allow the samples to cool for ~1 h or until at ambient temperature. Weigh the samples in the crucibles and record the date and time the samples were removed from the drying oven and the time the samples were weighed.

9.2.7 Return the samples to the drying oven controlled at 105°C and allow the samples to dry for an additional 24 h.

9.2.8 Remove the samples from the drying oven and transfer them to a dessicator. Allow the samples to cool for ~1 h or until at ambient temperature. Weigh the samples in the crucibles and record the date and time the samples were removed from the drying oven and the time the samples were weighed.

9.2.9 Repeat these drying steps until the sample reaches a stable mass. A stable mass is defined as a change in mass between the last two measurements of less than 0.1 %.

9.2.10 Once a stable mass is achieved, place the crucibles in the furnace and heat the furnace to 1000°C. Hold the temperature of the furnace between 1000°C and 1050°C for 30 min.

9.2.11 Allow the sample to cool to 150°C. If the sample is cooled too fast, the crucible could shatter; therefore, appropriate precautions should be taken to mitigate the potential for cracking the crucible and losing the sample.

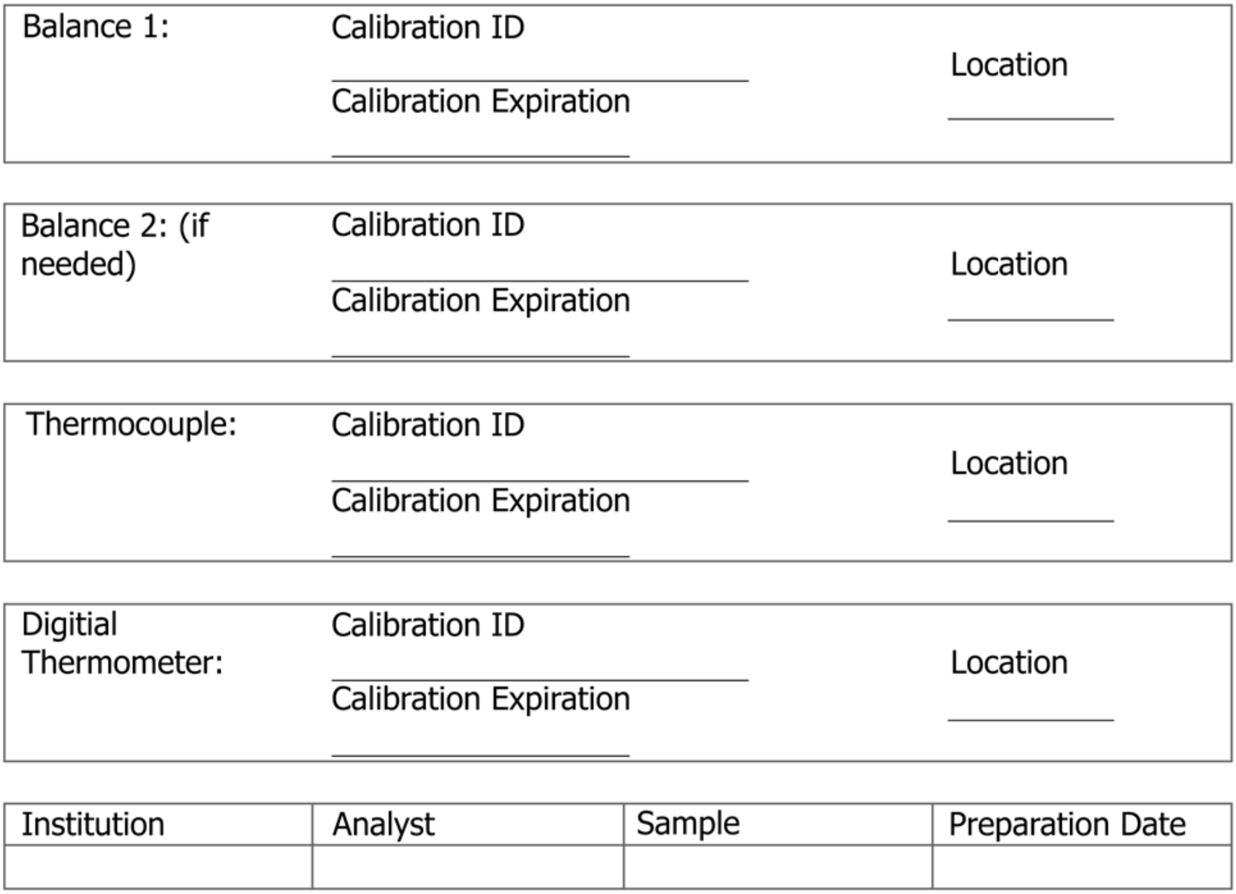
9.2.12 Transfer the sample to a desiccator, and cool the sample to ambient temperature (~1 h).

9.2.13 Weigh the crucibles with the fired samples and record the mass of the fired sample plus crucible.

9.2.14 An example of a data sheet for recording the drying data is provided in Table 1, Wt% Oxides Data Collection Sheet and Wt% Oxides Drying Data Collection Sheet.

9.3 *Rheological Properties Measurements:*

TABLE 1 Physical Properties Data Sheet Format



Balance 1:
Calibration ID ____________________
Calibration Expiration ______________
Location __________

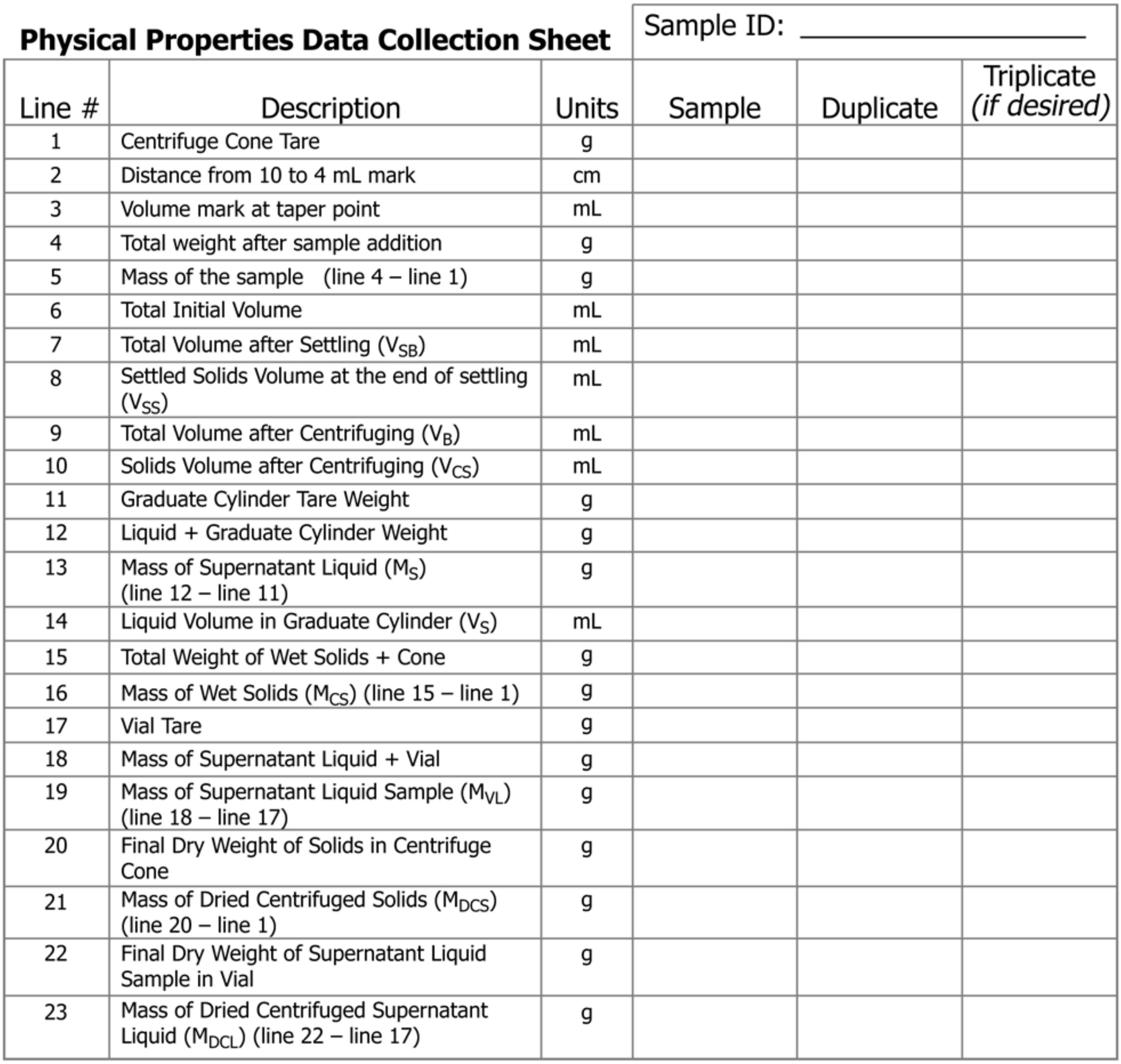
Balance 2: (if needed)
Calibration ID ____________________
Calibration Expiration ______________
Location __________

Thermocouple:
Calibration ID ____________________
Calibration Expiration ______________
Location __________

Digitial Thermometer:
Calibration ID ____________________
Calibration Expiration ______________
Location __________

Institution	Analyst	Sample	Preparation Date

TABLE 1 *(continued)*



Physical Properties Data Collection Sheet

Sample ID: ______________________

Line #	Description	Units	Sample	Duplicate	Triplicate *(if desired)*
1	Centrifuge Cone Tare	g			
2	Distance from 10 to 4 mL mark	cm			
3	Volume mark at taper point	mL			
4	Total weight after sample addition	g			
5	Mass of the sample (line 4 – line 1)	g			
6	Total Initial Volume	mL			
7	Total Volume after Settling (V_{SB})	mL			
8	Settled Solids Volume at the end of settling (V_{SS})	mL			
9	Total Volume after Centrifuging (V_B)	mL			
10	Solids Volume after Centrifuging (V_{CS})	mL			
11	Graduate Cylinder Tare Weight	g			
12	Liquid + Graduate Cylinder Weight	g			
13	Mass of Supernatant Liquid (M_S) (line 12 – line 11)	g			
14	Liquid Volume in Graduate Cylinder (V_S)	mL			
15	Total Weight of Wet Solids + Cone	g			
16	Mass of Wet Solids (M_{CS}) (line 15 – line 1)	g			
17	Vial Tare	g			
18	Mass of Supernatant Liquid + Vial	g			
19	Mass of Supernatant Liquid Sample (M_{VL}) (line 18 – line 17)	g			
20	Final Dry Weight of Solids in Centrifuge Cone	g			
21	Mass of Dried Centrifuged Solids (M_{DCS}) (line 20 – line 1)	g			
22	Final Dry Weight of Supernatant Liquid Sample in Vial	g			
23	Mass of Dried Centrifuged Supernatant Liquid (M_{DCL}) (line 22 – line 17)	g			

9.3.1 Rheological properties are measured in either a controlled-rate or a controlled-stress rheometer. In a controlled-rate rheometer, the resisting torque generated by rotating a solid shape of known geometry is measured over a range of fixed rotational rates. In a controlled-stress rheometer, the rotational rate of the solid shape of known geometry is measured over a range of fixed torques.

9.3.2 *Calibration and Standardization:*

9.3.2.1 Performance checks should be performed on rheometers at regular intervals during the generation of data. These performance checks verify the performance of the rheometer and limit the range of potentially impacted data should the rheometer, or its supporting systems, malfunction.

9.3.2.2 The viscosity standard and rheological program used to check the calibration of the viscometer should be appropriate for the torque and rotation rate used during analysis.

9.3.2.3 An appropriate deviation from the reported calibration standard should be determined by a statistical evaluation of the rheometer performance, requirements of the data, or requirements in an appropriate quality control or quality assurance program. Documentation of performance checks should include the identification of the viscosity standard used and the date of the performance check.

9.3.3 *Shear Strength:*

9.3.3.1 Shear strength is measured using the vane method developed by Dzuy and Boger (**1**).[4] Heath (**2**) and Barnes and Nguyen (**3**) provide additional references on this method.

9.3.3.2 Since shear strength is dependent upon the shear history of the sample, shear strength samples should remain

---

[4] The boldface numbers in parentheses refer to a list of references at the end of this standard.

TABLE 1 *(continued)*

Wt% Oxides Data Collection Sheet			Sample ID ____________		
Line #	Description	Units	Sample	Duplicate	Triplicate *(if needed)*
1	Pre-fired crucible tare	g			
2	Crucible + wet sample mass	g			
3	Wet sample mass (M_{WCS}) (line 2 – line 1)	g			
4	Stable dry mass @ 105°C (crucible + dired sample)	g			
5	Dried sample mass @ 105°C (M_{OSC}) (line 4 – line 1)	g			
6	Crucible + fired sample mass (1000°C)	g			
7	Fired sample mass @ 1000°C (M_{FSC}) (line 6 – line 1)	g			

TABLE 1 *(continued)*

Wt% Oxides Drying Data Collection Sheet Sample ID - ____________

Time	Date	Sample + Container Mass (g)		
		Sample	Duplicate	Triplicate *(if needed)*
Stable Mass at 105°C				
Furnace at 1000 - 1050°C for 30 minutes				

undisturbed with minimal shear forces placed upon the sample for a specified period of time prior to testing. The specific time should allow the sample to return to near its original rheological properties or specified properties. This time period may be based on the process that is being tested or monitored or upon experimental data.

(1) Experimental data or process knowledge should be evaluated when determining the time required for the sample to return near to its original shear strength or other specified rheological properties. Shear strength measurements at multiple storage times may be required to determine the appropriate storage time required to achieve a stable (reproducible) shear strength.

(2) While the sample is being stored, undisturbed, a lid should be placed on the sample to minimize evaporative losses.

(3) During the specified storage time prior to measurement, the sample should be held at the pre-determined analysis temperature.

9.3.3.3 For this standard, controlled-rate rheometry is used to measure shear strength. A vane of known geometry with the geometric constraints given in Fig. 1 is rotated through the sample medium to obtain shear strength.

9.3.3.4 Install the shear vane on the viscometer. The dimensions of the shear vane must meet the requirements provided in Fig. 1.

9.3.3.5 Perform all rheometer specific pre-measurement steps (for example, zeroing the torque offset, determining the geometry vertical gap, etc.).

9.3.3.6 Carefully raise the sample until the shear vane is immersed to a depth such that the height of the sample above the top of the immersed shear vane (Z_1 in Fig. 1) is equivalent to the height of the shear vane (H in Fig. 1). It is recommended that the shaft of the shear vane be marked to provide an indication of the depth of the shear vane in the sample since it is often difficult to judge the depth of the shear vane in the sample. **Warning—**When raising the sample, avoid sideways movement of the sample which may deform the sample resulting in lower shear strength.

9.3.3.7 It is normal for the rheometer to read a nonzero torque after inserting the shear vane. Do not re-zero the

TABLE 1 *(continued)*

Physical Properties Settling Rate Data Collection Sheet

Settling Schedule			Volume (mL)					
			Sample		Duplicate		Triplicate (if needed)	
Date	Time	Interval	Total	Solids	Total	Solids	Total	Solids

TABLE 1 *(continued)*

Physical Properties Drying Data Collection Sheet

Time	Date	Sample + Container Mass (g)					
		Sample		Duplicate		Triplicate *(if needed)*	
		Centrifuged Solids	Supernatant Liquid	Centrifuged Solids	Supernatant Liquid	Centrifuged Solids	Supernatant Liquid
Container (Tare)							
Final Dried Sample							

viscometer at this point in the procedure. Some rheometers automatically correct for torque offset. In such systems, this feature needs to be disabled.

9.3.3.8 Rotate the shear vane in the sample at a fixed rotational rate (N) for a period of at least 2 min. The preferred rotational rate is 0.3 revolutions per minute (RPM), but a rotational rate of up to 0.6 RPM is also acceptable. Additional information about the rheological properties of the sample can be determined by making this measurement at multiple shear rates.

9.3.3.9 Record the dimensions of the shear vane (height and diameter), the diameter of the sample container (D_T in Fig. 1), the depth of sample above and below the shear vane (Z_1 and Z_2 in Fig. 1), and the rotational rate (N).

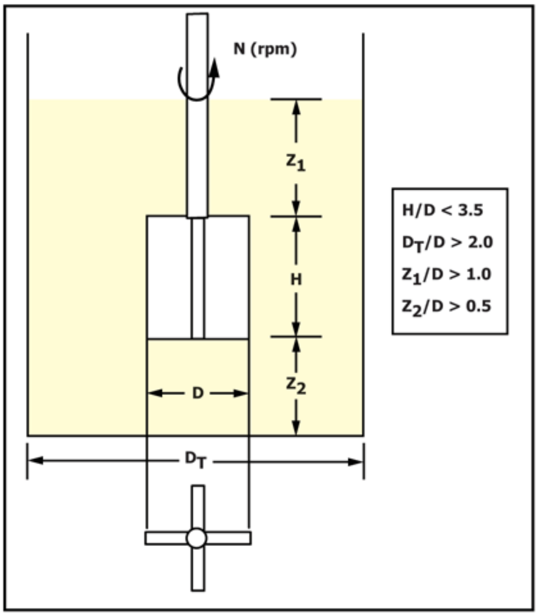


FIG. 1 Vane Geometry Dimensions (1)

9.3.3.10 During the measurement the torque should quickly increase at a near linear rate until it reaches a maximum torque (R_t) and then decline until a steady state torque (B) is reached (Fig. 2). Record both torque values, R_t and B, if observed. Some materials do not have a distinct maximum torque or do not reach steady state torque in the time frame of the measurement; these materials should be noted.

9.3.3.11 Calculate the shear strength (τ_o) according to Eq 19 provided in 10.2.1.

9.3.4 *Shear Stress vs. Shear Rate:*

9.3.4.1 Confirm an acceptable performance check has been performed as defined in 9.3.2 (*Calibration and Standardization*).

9.3.4.2 Select and install an appropriate sensor in the rheometer. Record the sensor system used along with the parameters for that sensor. A double gap sensor is preferred for solutions with little or no solids. A single gap sensor is preferred for samples with high solids content. Cone and plate or plate and plate sensors may be required for materials that cannot be poured. Vane geometries may be necessary for thick pastes, which tend to slip on the cone and plate or plate and plate systems.

(1) The gap must be of sufficient width to accommodate the solids. The gap should be at least three times larger than the diameter of the largest particles. A gap of at least ten times larger than the diameter of the largest particles is desired.

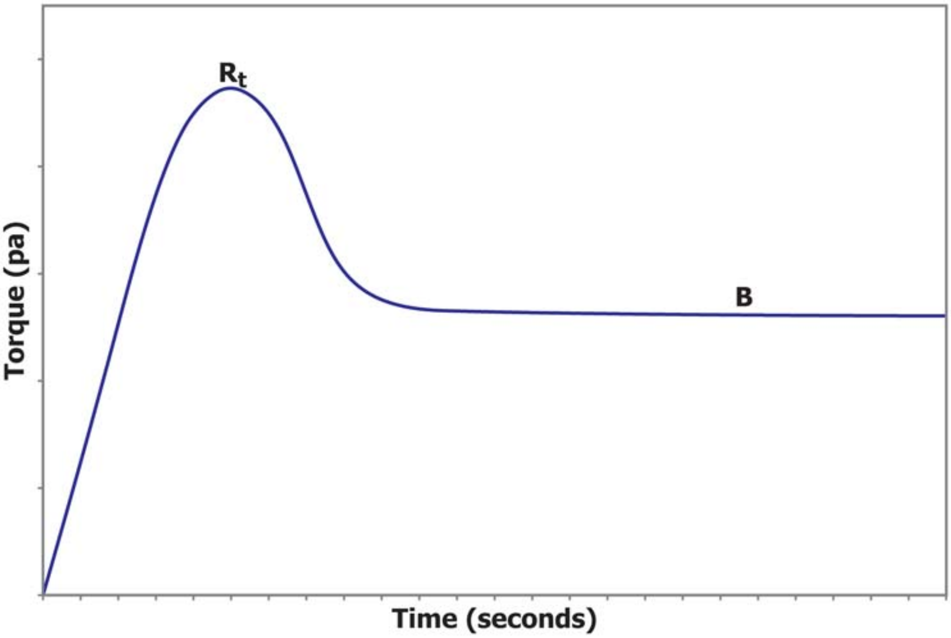


FIG. 2 Shear Strength Measurement

9.3.4.3 Set the rheometer temperature to the test temperature and allow sufficient time for the system to thermally equilibrate. Analyses temperatures should represent the temperatures at which the material will be processed or handled, because the viscosity and yield stress can be sensitive to temperature.

9.3.4.4 Mix the sample to obtain a homogeneous sample.

9.3.4.5 Transfer the sample to the rheometer and initiate the analysis as soon as possible, ensuring that the sample has reached the test temperature.

9.3.4.6 Increase the shear rate from 0 to 1000 s^{-1}, or the maximum shear rate of the selected sensor, at a rate selected for the sample and the desired data analysis. Hold the shear rate at the maximum value for one minute, and then decrease the shear rate to 0 at the same rate as the increase. The ramp rates should be selected according to the specific shear rate range of interest. Equilibrium flow curves can be obtained from controlled-stress experiments.

9.3.4.7 Repeat the analysis after mixing the sample. If the results from this second analysis are different from the first run, perform a third analysis. These replicates will be used to assess the samples sensitivity to shear history.

9.3.4.8 Examine the rheogram for Taylor vortices. Taylor vortices are the result of a secondary flow that occurs as the inner cylinder of the concentric cylinder viscometer rotates. Taylor vortices result from performing rheological measurements at too high of a shear rate for the rheological properties of the sample. Taylor vortices are anticipated when the following two equations are satisfied (**4**):

$$R_e > 41.3\left(\frac{R_2}{(R_2 - R_1)}\right)^{\frac{1}{2}} \quad (1)$$

$$R_e = \dot{\gamma}\frac{(R_2 - R_1)^2 \rho}{\mu} \quad (2)$$

9.3.4.9 Report any Taylor vortices observed and discard this part of the data set prior to modeling the data.

9.3.4.10 Model the increasing shear rate curves (excluding portions effected by Taylor vortices) using the following models in Section 10.2:

(1) Newtonian Fluid (Eq 20)

(2) Ostwald or Power Law (Eq 21)

(3) Bingham Plastic (Eq 22)

(4) Herschel-Bulkley (Eq 23)

9.4 *Particle Size Distribution:*

9.4.1 Multiple techniques can be used to measure the particle size distribution of sludges and slurries. These methods include microscopy, wet and dry sieving, sedimentation, electrical pulse counting, hydrodynamic, electroacoustics, and light scattering.

9.4.2 Different sizing methods can be expected to give different results because each method is measuring a different aspect of the particle size distribution. Since each of these methods interacts with the particle in different ways, comparison of the results obtained from each of these methods provides insight into the deviation of the particles in the sample from monodisperse spherical particles.

9.4.3 One of the primary variables used in selecting the appropriate particle size distribution method is the range of sizes to be measured by the method. Fig. 3 provides estimated size ranges for different particle size distribution measurement methods.

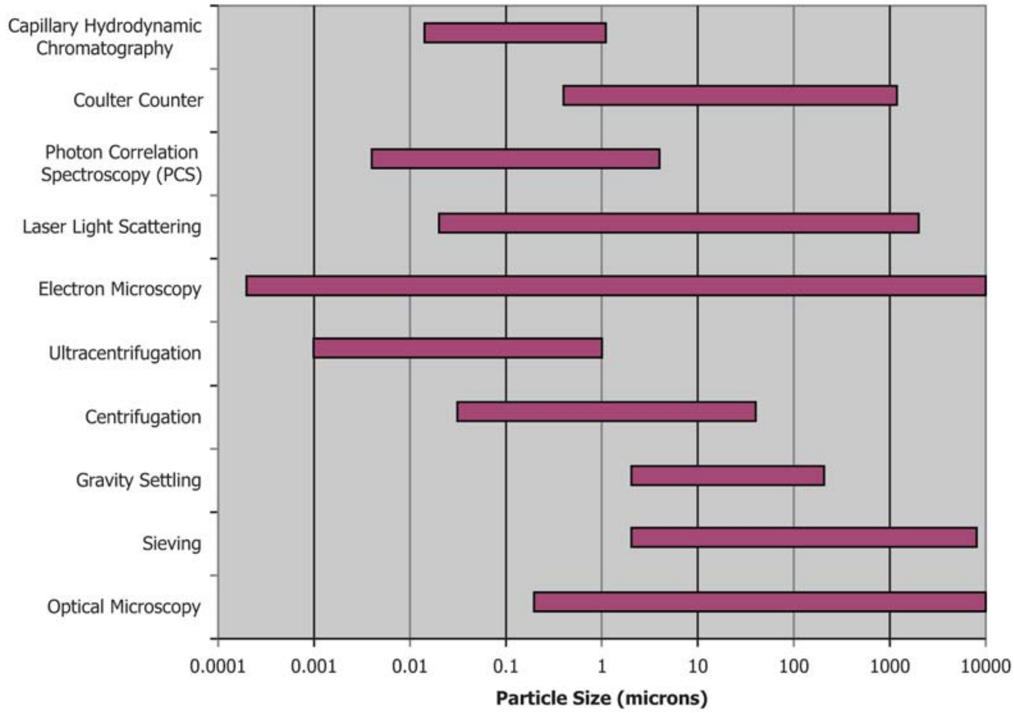


FIG. 3 Approximate Measuring Range of Particle Size Distribution Methods

9.4.4 Measurements should be made according to the instrument instructions and the guidelines provided in ISO13320-1 and the NIST practice for particle size distribution (**5**).

10. Calculation or Interpretation of Results

10.1 *Physical Properties:*

10.1.1 Bulk Slurry Density

$$\rho_B = \frac{M_B}{V_B} \qquad (3)$$

10.1.2 Vol% Settled Solids

$$P_{VSS} = \frac{V_{SS}}{V_B} \times 100\,\% \qquad (4)$$

10.1.3 Centrifuged Solids Density

$$\rho_{CS} = \frac{M_{CS}}{V_{CS}} \qquad (5)$$

10.1.4 Vol% Centrifuged Solids

$$P_{VCS} = \frac{V_{CS}}{V_B} \times 100\,\% \qquad (6)$$

10.1.5 Wt% Centrifuged Solids

$$P_{MCS} = \frac{M_{CS}}{M_B} \times 100\,\% \qquad (7)$$

10.1.6 Supernatant Liquid Density

$$\rho_S = \frac{M_S}{V_S} \qquad (8)$$

10.1.7 Density of the settled solids is calculated in a three step process. The first calculation (Eq 9) is a determination of the mass of the supernatant liquid followed by the calculation of the mass of the settled solids (Eq 10). The density of the settled solids can then be calculated by dividing the mass of the settled solids by the measured volume of the settled solids (Eq 11). These equations are combined to obtain Eq 12.

10.1.7.1 Mass of the Supernatant Liquid

$$M_{SL} = \rho_S \times V_{SL} \qquad (9)$$

10.1.7.2 Mass of the Settled Solids

$$M_{SS} = M_B - M_{SL} \qquad (10)$$

10.1.7.3 Density of the Settled Solids

$$\rho_{SS} = \frac{M_{SS}}{V_{SS}} \qquad (11)$$

10.1.7.4

$$\rho_{SS} = \frac{(M_B - \rho_S \times V_{SL})}{V_{SS}} \qquad (12)$$

10.1.8 Wt% Settled Solids

$$P_{MSS} = \frac{M_{SS}}{M_B} \times 100\,\% \qquad (13)$$

10.1.9 Total solids content (wt%) is calculated from the physical properties samples and the weight percent oxide samples. The calculation from these two sets of data varies because the data collected is different. The calculation for the physical properties samples is provided in Eq 14 and the calculation for the weight percent oxide samples is given in Eq 15.

10.1.9.1 Percent Mass of Total Solids (wt%)

$$P_{MTS} = \left(\left(\frac{(M_{DCL} \times M_S)}{(M_{VL} \times M_B)}\right) + \left(\frac{M_{DCS}}{M_B}\right)\right) \times 100\,\% \qquad (14)$$

10.1.9.2 Percent Mass of Oven Dried Solids (wt%)

$$P_{MDS} = \frac{M_{OSC}}{M_{WCS}} \times 100\,\% \qquad (15)$$

10.1.10 Solids Content of the Centrifuged Solids (wt%)

$$P_{ODS} = \frac{M_{DCS}}{M_{CS}} \times 100\,\% \qquad (16)$$

10.1.11 Undissolved solids content in weight percent is calculated based on the assumption that the supernatant liquid and interstitial liquid have the same composition and that all mass loss during drying of the centrifuged solids is water loss from interstitial liquid. The equation for this calculation based on these assumptions is provided in Eq 17.

10.1.11.1 Percent Mass of Undissolved Solids (wt%)

$$P_{MUS} = \left(1 - \frac{1 - \dfrac{M_{DCS}}{M_{CS}}}{1 - \dfrac{M_{DCL}}{M_{VL}}}\right) \times \frac{M_{CS}}{M_B} \times 100\,\% \qquad (17)$$

10.1.12 Wt% Total Oxides

$$P_{MOX} = \frac{M_{FSC}}{M_{WCS}} \times 100\,\% \qquad (18)$$

10.1.13 An example of the physical properties calculation sheet is provided in Table 2.

10.2 *Rheological Properties:*

10.2.1 Shear Strength

$$\tau_o = \frac{R_t}{\dfrac{\pi \times D^3}{20\,000}\left(\dfrac{1}{3} + \dfrac{H}{D}\right)} \qquad (19)$$

10.2.2 Newtonian fluid

$$\tau = \eta\dot{\gamma} \qquad (20)$$

10.2.3 Ostwald model or Power Law

$$\tau = m\dot{\gamma}^n \qquad (21)$$

10.2.3.1 If n < 1, then the material is pseudoplastic (shear thinning). If n > 1 the material is dilatant (shear thickening). Since dilatant flow behavior is rare, a value of n > 1 is likely an indication of Taylor vortices or other measurement errors.

10.2.4 Bingham Plastic

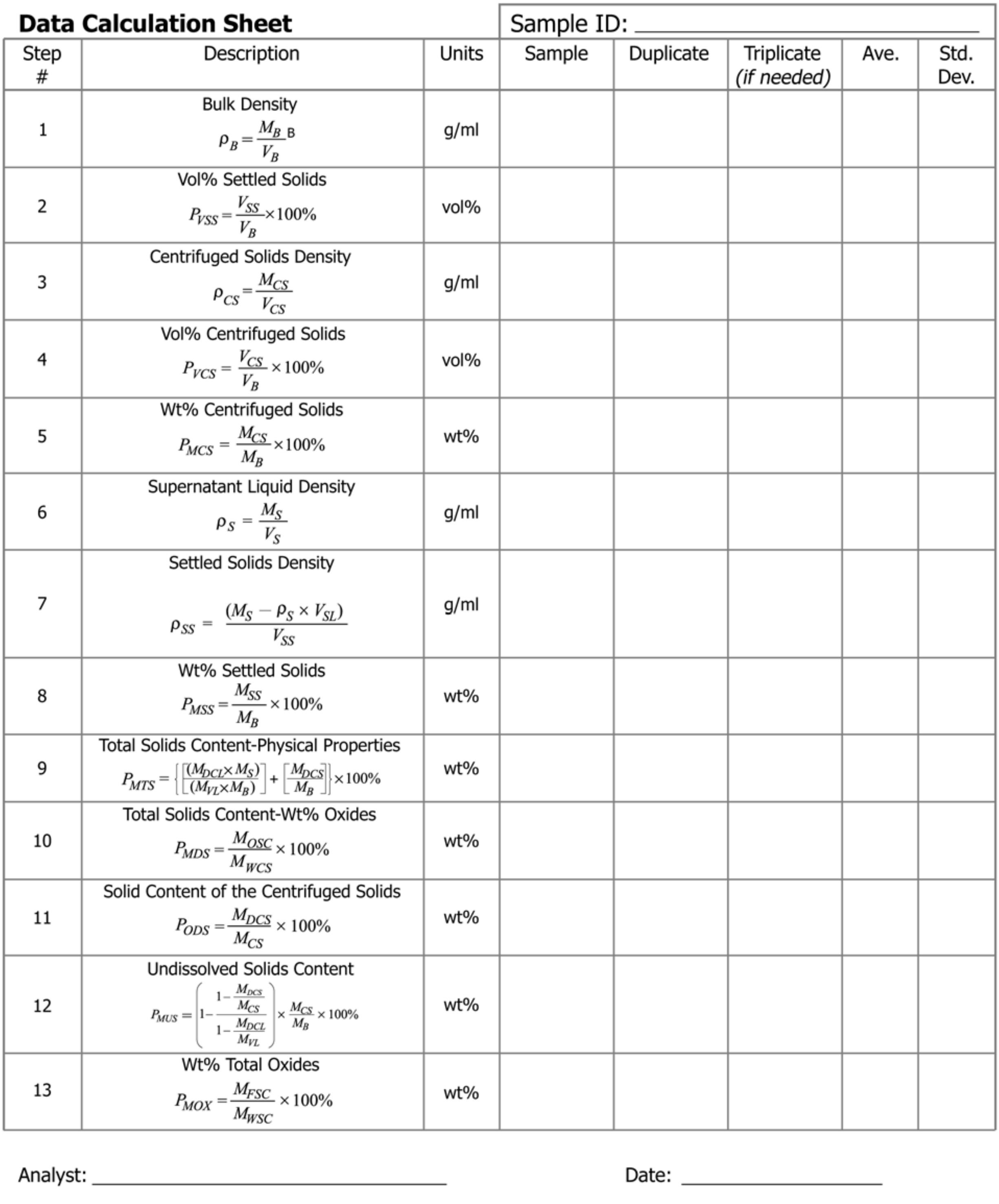
$$\tau = \tau_o^B + \eta_p\dot{\gamma} \qquad (22)$$

10.2.5 Herschel=Bulkley

$$\tau = \tau_o^H + k\dot{\gamma}^b \qquad (23)$$

10.2.5.1 Constrain the Herschel-Bulkley yield stress to values greater than or equal to zero.

TABLE 2 Physical Properties Calculation Sheet Format



Data Calculation Sheet

Sample ID: ______________________________

Step #	Description	Units	Sample	Duplicate	Triplicate *(if needed)*	Ave.	Std. Dev.
1	Bulk Density $\rho_B = \frac{M_B}{V_B}$ B	g/ml					
2	Vol% Settled Solids $P_{VSS} = \frac{V_{SS}}{V_B} \times 100\%$	vol%					
3	Centrifuged Solids Density $\rho_{CS} = \frac{M_{CS}}{V_{CS}}$	g/ml					
4	Vol% Centrifuged Solids $P_{VCS} = \frac{V_{CS}}{V_B} \times 100\%$	vol%					
5	Wt% Centrifuged Solids $P_{MCS} = \frac{M_{CS}}{M_B} \times 100\%$	wt%					
6	Supernatant Liquid Density $\rho_S = \frac{M_S}{V_S}$	g/ml					
7	Settled Solids Density $\rho_{SS} = \frac{(M_S - \rho_S \times V_{SL})}{V_{SS}}$	g/ml					
8	Wt% Settled Solids $P_{MSS} = \frac{M_{SS}}{M_B} \times 100\%$	wt%					
9	Total Solids Content-Physical Properties $P_{MTS} = \left\{ \left[\frac{(M_{DCL} \times M_S)}{(M_{VL} \times M_B)} \right] + \left[\frac{M_{DCS}}{M_B} \right] \right\} \times 100\%$	wt%					
10	Total Solids Content-Wt% Oxides $P_{MDS} = \frac{M_{OSC}}{M_{WCS}} \times 100\%$	wt%					
11	Solid Content of the Centrifuged Solids $P_{ODS} = \frac{M_{DCS}}{M_{CS}} \times 100\%$	wt%					
12	Undissolved Solids Content $P_{MUS} = \left(1 - \frac{1 - \frac{M_{DCS}}{M_{CS}}}{1 - \frac{M_{DCL}}{M_{VL}}} \right) \times \frac{M_{CS}}{M_B} \times 100\%$	wt%					
13	Wt% Total Oxides $P_{MOX} = \frac{M_{FSC}}{M_{WSC}} \times 100\%$	wt%					

Analyst: ______________________________ Date: ________________

11. Report

11.1 Physical properties data should be reported as the average values of the sample and the replicates with a standard deviation.

11.2 Simulant formulation as well as the date the simulant was prepared should be included in the final report for all simulants tested.

11.3 A plot of torque versus time and a tabular report of the shear strength measurements should be included in the shear strength data report along with the geometry of the shear vane, the diameter of the sample container, the depth of sample above and below the shear vane, and the rotational rate. An example of a shear strength data sheet is provided in Table 3.

11.4 Flow curve parameters for rheological property determinations should be reported along with both a printout of the flow curve and a tabular report of the shear stress versus shear rate data. An example of a flow curve data sheet is provided in Table 4.

11.5 The results for applicable rheological models (Ostwald or Power Law, Bingham Plastic, and Herschel-Bulkley for non-Newtonian fluids and the Newtonian model for Newtonian fluids) should be reported along with the associated correlation coefficient (r^2). An example of the rheological properties summary report including these model parameters is provided in Table 5.

12. Keywords

12.1 physical properties; rheology; viscosity

TABLE 3 Shear Strength Data Sheet

Shear Strength Data Sheet	Sample ID: ____________________
Date	
File Name	
Operator	
Sensor	
Measurement System	
Temperature (°C)	
H (cm)	
D (cm)	
D_T (cm)	
Z_1 (cm)	
Z_2 (cm)	
N (RPM)	
Torque at Full Scale (N·cm)	
Time (seconds)	**Torque (N·cm)**

TABLE 4 Flow Curve Data Sheet

Flow Curve Data Sheet

Sample ID: ______________________________

Date			
File Name			
Operator			
Sensor			
Measurement System			
Temperature (°C)			
M Factor			
A Factor			
Flow Curve Data			
Shear Rate (1/s)	Shear Stress (Pa)	Viscosity (Pa•s)	Time (min)

TABLE 5 Rheological Property Data Sheet

Model/Model Parameter	**Value**
Shear Strength (by Vane Method):	
τ_o – Shear Strength (Pa)	
Ostwald or Power Law	
m – consistency coefficient (Pa•s^n)	
n – Power Law exponent	
r^2 – correlation coefficient	
Bingham Plastic	
τ_o^B – Bingham yield stress (Pa)	
η_p – plastic viscosity (Pa•s)	
r^2 – correlation coefficient	
Herschel-Bulkley	
τ_o^H – Herschel-Bulkley yield stress (Pa)	
k – Herschel-Bulkley consistency coefficient (Pa•s^b)	
b – Herschel-Bulkley power law exponent	
r^2 – correlation coefficient	

REFERENCES

(1) Dzuy, N. Q. and D. V. Boger, Direct Yield Stress Measurement with the Vane Method, *Journal of Rheology*, 29 (1985), p. 335-347.

(2) Heath, W. O., "Procedure for Measuring Sludge Shear Strength Using Shear Vanes," WHO-86-1, Pacific Northwest National Laboratory, Richland, Washington, May 1986.

(3) Barnes, H. A. and Q. D. Nguyen, Rotating Vane Rheometry—A Review, *Journal of Non-Newtonian Fluid Mechanics*, 98 (2001), p 1-14.

(4) Chang, C. Y., "Laboratory Procedure for the Rheological Characterization of Slurry Suspensions," PNNL-10985, Pacific Northwest National Laboratory, Richland, Washington, February 1996.

(5) Jillavenkatesa, A.; Dapkunas, S. J.; and Lum, Lin-Sien H., "Particle Size Characterization, NIST Recommended Practice Guide," Special Publication 960-1, National Institute of Standards and Technology, Washington, D. C. (2001)

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/