

Designation: C1752 - 17 C1752 - 21

Standard Guide for Measuring Physical and Rheological Properties of Radioactive Solutions, Slurries, and Sludges¹

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. 1752-21
- 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.

¹ This test method guide 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.

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2. Referenced Documents

2.1 ASTM Standards:²

C859 Terminology Relating to Nuclear Materials

C1750 Guide for Development, Verification, Validation, and Documentation of Simulants for Hazardous Materials and Process Streams

C1751 Guide for Sampling Radioactive Tank Waste

D1193 Specification for Reagent Water

2.2 Other Standards: ISO Standard: 3

ISO13320;2009ISO 13320:2020 "Particle Size Analysis—Laser Diffraction Methods. Part 1: General Principles," International Organization for Standardization, Geneva, Switzerland (1999)Particle size analysis — Laser diffraction methods

3. Terminology

- 3.1 Definitions: Definitions—For definitions of terms used in this guide but not defined herein, refer to Terminology C859.
- 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.
- 3.1.3 Newtonian fluid, n—a fluid whose apparent viscosity is independent of shear rate.
- 3.1.4 non-Newtonian fluid, n—a fluid whose apparent viscosity varies with shear rate.
- 3.1.5 *rheogram*, *n*—plot of shear stress versus shear rate.
 - 3.1.5.1 Discussion—

A rheogram is also called a flow curve.

- 3.1.6 *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. log/standards/sist/e6c3d3cc-7daa-4b7b-9013-b56322988894/astm-c1752-21
- 3.1.7 shear strength, n—maximum shear stress measured during incipient motion.
- 3.1.8 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
- 3.1.9 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.

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 Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

- 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.
- 3.1.10 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.
- 3.1.11 weight percent (wt%) dried solids, n—percentage of the mass of the sample that remains after removing volatiles including free water by drying at $105 \pm \frac{5 \text{ °C}}{5} \text{ °C}$ to a stable mass.
 - 3.1.11.1 Discussion—
- Wt% wt% total dried solids and wt% centrifuged dried solids are the wt% dried solids in the bulk sample and centrifuged solids, respectively.
- 3.1.12 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.
- 3.1.13 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.
- 3.1.14 *yield stress*, *n*—minimum stress required to initiate fluid movement as determined by a flow curve using a rheological model.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *interstitial solution*, *n*—*in this guide*, interstitial solution is the solution contained between the suspended, settled, or centrifuged solid particles of a sludge sample.
- 3.2.2 sludge, n—in this guide, sludge is wet solids having little or no standing liquid (that is, mud-like).
- 3.2.3 slurry, n—in this guide, a slurry is a mixture of solids and solution.
- 3.2.4 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.
- 3.2.5 *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.
- 3.2.6 *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.
- 3.2.7 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.
 - 3.3 NomenclatureAbbreviations::
- 3.3.1 *b*—Hershel-Bulkley Herschel-Bulkley power law exponent (unitless):(unitless)

- 3.3.2 *B*—steady-state torque in a shear strength test in N·cm.N·cm
- 3.3.3 *D*—diameter of the shear vane in cm.cm
- 3.3.4 D_T —diameter of the shear strength sample cup in em.cm
- 3.3.5 *H*—height of the shear vane in cm.cm
- 3.3.6 k—Hershel-Bulkley Herschel-Bulkley consistency coefficient in Pa·s^b.
- 3.3.7 *m*—power law consistency coefficient in $Pa \cdot s^n$.
- 3.3.8 M_B —total mass of bulk slurry after centrifugation in g.g.
- 3.3.9 M_{CC} —mass of the centrifuge cone in g-g
- 3.3.10 M_{CR} —mass of the crucible in g.g.
- 3.3.11 M_{CS} —mass of the centrifuged solids and their interstitial liquid in g-g
- 3.3.12 M_{DCSDCL} —mass of the oven dried centrifuged solids supernatant liquid in g.g.
- 3.3.13 M_{DCLDCS} —mass of the oven dried centrifuged supernatant liquid solids in g-g
- 3.3.14 M_{FSC} —mass of the fired solids $\frac{(1000^{\circ}\text{C})}{(1000^{\circ}\text{C})}$ in the crucible in g.g.
- 3.3.15 M_{OSC} —mass of the oven dried solids (105°C) (105 °C) in the crucible in g.g.
- 3.3.16 M_s —mass of the decanted supernatant liquid after centrifugation in g-g
- https://standards.iteh.ai/catalog/standards/sist/e6c3d3cc-7daa-4b7b-9013-b56322988894/astm-c1752-2
- 3.3.17 M_{SL} —mass of the supernatant liquid after gravity settling in g.g.
- 3.3.18 M_{SS} —mass of the settled solids and interstitial liquid after gravity settling in g-g
- 3.3.19 M_{VL} —mass of a subsample of the decanted centrifuged supernatant liquid in g-g
- 3.3.20 M_{WCS} —mass of the wet sample in the crucible in $\frac{g}{g}$
- 3.3.21 *n*—power law exponent (unitless):(unitless)
- 3.3.22 *N*—rotational rate of the shear vane in revolutions per min.min
- 3.3.23 P_{MCS} —percent mass of centrifuged solids with the associated interstitial liquid in the slurry-slurry
- 3.3.24 P_{MDS} —percent mass of oven dried solids in the <u>crucible.crucible</u>
- 3.3.25 P_{MOX} —percent mass of oxides in the slurry.slurry
- 3.3.26 P_{MSS} —percent mass of settled solids with its associated interstitial liquid-liquid
- 3.3.27 P_{MTS} —percent mass of total solids.

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- 3.3.28 P_{MUS} —percent mass of undissolved solids in the slurry.slurry
- 3.3.29 P_{ODS} —percent mass of oven dried solids in the centrifuged solids including interstitial liquid liquid
- 3.3.30 P_{VCS} —percent volume of centrifuged solids with its associated interstitial liquid in the slurry-slurry
- 3.3.31 P_{VSS} —percent volume of settled solids with its associated interstitial liquid in the slurry.slurry
- 3.3.32 r^2 —correlation coefficient (unitless):(unitless)
- 3.3.33 R_i —radius of the inner cylinder of the viscometer concentric cylinder geometry in em.cm
- 3.3.34 R_2 —radius of the outer cylinder of the viscometer concentric cylinder geometry in em.cm
- 3.3.35 R_e—Reynolds number (unitless). (unitless)
- 3.3.36 R_t —maximum torque in a shear strength test in N·cm.N·cm
- 3.3.37 V_B —total volume of bulk sample after centrifugation in mL.mL
- 3.3.38 V_{CS} —volume centrifuged solids and the associated interstitial liquid in mL.mL
- 3.3.39 V_S —volume of decanted supernatant liquid after centrifugation in mL.mL
- 3.3.40 V_{SB} —total volume of bulk sample after gravity settling in mL.mL
- 3.3.41 V_{SL} —volume of supernatant liquid after gravity settling in mL.mL
- 3.3.42 V_{SS} —volume of settled solids with its associated interstitial liquid after gravity settling in mL:mL
- https://standards.iteh.ai/catalog/standards/sist/e6c3d3cc-7daa-4b7b-9013-b56322988894/astm-c1752-2
- 3.3.43 Z_I —height of the sample above the top of the immersed shear vane in em.cm
- 3.3.44 Z_2 —depth of the sample below the immersed shear vane in em.cm
- 3.3.45 ρ_B —bulk density of slurry in g/mL.g/mL
- 3.3.46 ρ_{CS} —density of centrifuged solids in g/mL.g/mL
- 3.3.47 ρ_S —density of supernatant liquid in g/mL.g/mL
- 3.3.48 ρ_{SS} —density of settled solids in g/mL.g/mL
- 3.3.49 τ —shear stress in Pa.Pa
- 3.3.50 τ_o —shear strength in Pa.Pa
- 3.3.51 τ_o^B —Bingham yield stress in Pa.Pa
- 3.3.52 τ_o^H —Herschel-Bulkley yield stress in Pa.Pa
- 3.3.53 η —Newtonian viscosity in Pa·s. Pa·s

- 3.3.54 η_p —plastic viscosity in Pa·s.Pa·s
- 3.3.55 γ —shear rate in s⁻¹.
- 3.3.56 μ—apparent viscosity in Pa·s. Pa·s
 - 3.4 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 before 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)(~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. Details on methods to develop representative simulants are provided in the Guide C1750. 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:below:
- 5.4.1 Bulk slurry density Settled solids density.
- 5.4.2 Settled solids densityBulk slurry density.
- 5.4.3 Centrifuged solids densitydensity.
- 5.4.4 Supernatant density density.
- 5.4.5 Settling raterate.

- 5.4.6 Volume percent centrifuged solidssolids.
- 5.4.7 Volume percent settled solids after settlingsettling.
- 5.4.8 Undissolved solids contentcontent.
- 5.4.9 Dissolved solids content content.
- 5.4.10 Weight percent centrifuged solids.
- 5.4.11 Weight percent total oxidesoxides.
- 5.4.12 Solids content of the centrifuged solids.
- **5**.4.13 Total solids contentcontent.
 - 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. 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(105) and 1000°C, 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).

⁴ 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.

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 $\frac{15 \text{ mL}}{15 \text{ mL}}$ 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-mL10 to the 4-mL4 mL graduation marks on the 15-mL15 mL centrifuge cone or the 30-mL30 to the 10-mL10 mL graduation marks on the 50-mL50 mL centrifuge cone). Volume-graduated centrifuge cones should be cylindrical between 10 and 4 mL and 30 and 10 mL for 15-mL15 and 50-mL50 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.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. 105 °C. Centrifuge cones used in this step should be rated for at least 105°C. 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. 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 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. 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 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 ~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. TM C1752-21
- 9.2.7 Return the samples to the drying oven controlled at 105°C 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. 1000 °C. Hold the temperature of the furnace between 1000°C 1000 and 1050°C for 30 min. 1050 °C for 30 min.
- 9.2.11 Allow the sample to cool to 150°C. 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 12, 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	 andards_	Location	
Institution		nple rds.ite	Preparation Da	te
Balance 1:	Calibration ID Calibration Expiration ASTM (1 Preview 11752-21 cc-7daa-4b7b-9013	Location:	astm-c1752-21
Balance 2:	Calibration ID Calibration Expiration		Location:	
Thermal couple:	Calibration ID Calibration Expiration		_Location:	
Digital Thermometer:	Calibration ID Calibration Expiration		_Location:	
Institution	Analyst	Sample	Preg	paration Date

Physical	Properties Data Collection Sheet	Sample ID:			
Line No.	Description		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	cm			
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 (MCS) (line 15 - line 1)	g			
17	Vial Tare weight	g			
18	Mass of Supernatant Liquid + Vial	g			
19	Mass of Supernatant Liquid Sample (MVL) (line 18 – line 17)	g			
20	Final Dry Weight of Solids in Centrifuge Cone	g			
21	Mass of Dried Centrifuged Solids (MDCS) (line 20 – line 1)	g			
22	Final Dry Weight of Supernatant Liquid Sample in Vial	g			
23	Mass of Dried Centrifuged Supernatant Liquide (MDCL) (line 22 – line 17)	g			

iTeh Standards (https://standards.iteh.ai)

TABLE 1 Continued

Settling Schedule		Volume (mL)						
		Sample V C 1/5/2		-2 Duplicate		Triplicate (if needed)		
Date	Time	Interval	Total	Solids	Total	Solids	Total	Solids
https://sta	ndards.hen.a	i/catalog/sta	idards/sist/e	ocsasce-/a	aa-40/0-90	13-0303229	00094/asum	·C1/32-2

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

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.