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Development of K-Basin High-Strength Homogeneous Sludge Simulants and Correlations Between Unconfined Compressive Strength and Shear Strength

Y Onishi EBK Baer J Chun ST Yokuda AJ Schmidt SA Sande WC Buchmiller

February 2011



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# Development of K-Basin High-Strength Homogeneous Sludge Simulants and Correlations Between Unconfined Compressive Strength and Shear Strength

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Pacific Northwest National Laboratory Richland, Washington 99352

## **Executive Summary**

K-Basin sludge will be stored in the Sludge Transport and Storage Containers (STSCs) at an interim storage location on Central Plateau before being treated and packaged for disposal. During the storage period, sludge in the STSCs may consolidate/agglomerate, potentially resulting in high-shear-strength material. The Sludge Treatment Project (STP) plans to use water jets to retrieve K-Basin sludge after the interim storage.

STP has identified shear strength to be a key parameter that should be bounded to verify the operability and performance of sludge retrieval systems. Determining the range of sludge shear strength is important to gain high confidence that a water-jet retrieval system can mobilize stored K-Basin sludge from the STSCs. The shear strength measurements will provide a basis for bounding sludge properties for mobilization and erosion. Thus, it is also important to develop potential simulants to investigate these phenomena.

Long-term sludge storage tests conducted by Pacific Northwest National Laboratory (PNNL) show that high-uranium-content K-Basin sludge can self-cement and form a strong sludge with a bulk shear strength of up to 65 kPa. Some of this sludge has "paste" and "chunks" with shear strengths of approximately 3~5 kPa and 380~770 kPa, respectively. High-uranium-content sludge samples subjected to hydrothermal testing (e.g., 185°C, 10 hours) have been observed to form agglomerates with a shear strength up to 170 kPa. These high values were estimated by measured unconfined compressive strength (UCS) obtained with a pocket penetrometer. Due to its ease of use, it is anticipated that a pocket penetrometer will be used to acquire additional shear strength data from archived K-Basin sludge samples stored at the PNNL Radiochemical Processing Laboratory (RPL) hot cells.

It is uncertain whether the pocket penetrometer provides accurate shear strength measurements of the material. To assess the bounding material strength and potential for erosion, it is important to compare the measured shear strength to penetrometer measurements and to develop a correlation (or correlations) between UCS measured by a pocket penetrometer and direct shear strength measurements for various homogeneous and heterogeneous simulants.

This study developed 11 homogeneous simulants, whose shear strengths vary from 4 to 170 kPa. With these simulants, we developed correlations between UCS measured by a Geotest E-280 pocket penetrometer and shear strength values measured by a Geonor H-60 hand-held vane tester and a more sophisticated bench-top unit, the Haake M5 rheometer. This was achieved with side-by-side measurements of the shear strength and UCS of the homogeneous simulants.

The homogeneous simulants developed under this study consist of kaolin clay, plaster of Paris, and amorphous alumina CP-5 with water. The simulants also include modeling clay. The shear strength of most of these simulants is sensitive to various factors, including the simulant size, the intensity of mixing, and the curing time, even with given concentrations of simulant components. Table S.1 summarizes these 11 simulants and their shear strengths.

	Simulant Initial Composition, wt%					Averaged	
_	CP-5				Shear		
Simulant			Plaster of	Amorphous	Modeling	Strength,	
Designation	Water	Kaolin	Paris	Alumina	Clay	kPa	
А	35	50	15			3.7	
В	34	66				7.7	
С	43	28	29			15	
D	41	30	29			17	
Е					100	44	
F	39	24.5	36.5			72	
G	64.5			35.5		74	
Н	63			37		95	
Ι	37.5	24.5	38			130	
J	37.5	23.5	39			140	
K	56		44			170	

Table S.1. Eleven Homogeneous Simulants and Measured Averaged Shear Strengths

The correlation of unconfined compressive strength measured by the Geotest E-280 pocket penetrometer and shear strength measured by the Haake M5 rheometer and the Geonor H-60 hand-held vane tester is shown in Figure S.1 and Equation S.1.



#### Correlation between UCS and Shear Strength

Figure S.1. Correlation Between Unconfined Compressive Strength and Shear Strength

$$\tau_s = 42.8(UCS)$$
 (S.1)  
with  $R^2 = 0.957$ 

where the unconfined compressive strength is in kg(f)/cm<sup>2</sup>, and  $\tau_{s}$  is the shear strength in kPa.

This correlation applies to shear strength values ranging from 4 kPa to 170 kPa. The 11 simulants have four different solid materials, i.e., kaolin clay, plaster of Paris, amorphous alumina, and modeling clay, as stated above. However, Figure S.1 does not show any different trends among the simulants. Thus, this correlation, as indicated in Equation S.1, applies to various solid materials, including at least these four solid materials or similar materials. Additional test data implies that the error of conversion of the pocket penetrometer UCS reading to shear strength with Equation S.1 may be up to 27%.

Applying Equation S.1 to the bounding UCS measurement  $[3.54 \text{ kg}(f)/\text{cm}^2]$  made on hydrothermally treated sludge (185°C) with a pocket penetrometer gives a shear strength prediction of 152 kPa vs. the previously reported estimate of 174 kPa (as reported as 170 kPa). The hydrothermal treated sludge was a heterogeneous material, and Equation S.1 was developed from the testing of homogeneous simulants. However, the work in this study confirms that the sludge strength being used by STP as the anticipated bounding range (i.e., 150 to 200 kPa) is reasonable for sludge during retrieval from STSCs.

After performing the testing to develop the strength correlation, follow-on investigations were conducted to evaluate the effect of simulant batch size, cure time, and the simulant component mixing approach. To examine the effects of simulant scale-up, we successfully prepared sixteen ~22-L homogeneous simulant (Simulants I and II) batches with shear strengths of approximately 60 kPa and 190 kPa. Additionally, a number of 0.2- and 0.5-L homogeneous simulants batches, of shear strength varying from 40 to 220 kPa, were also prepared.

This additional limited testing reveals that simulant sizes and container materials have only minor effects on the unconfined compressive strength and shear strength values. These test data and their analysis indicate that they themselves are not a main cause of the shear strength variation, but the manner and degree of simulant mixing and some non-uniformity of the simulant within a mixing bucket may be more important factors to determine the shear strength of a simulant for the given simulant compositions and their concentrations. Thus, it is important to uniformly mix simulants in a very consistent way to obtain a specific shear strength.

The simulants gained most of the final unconfined compressive strength and shear strength values after 1 day of curing and generally reached their final values in the first 3 days. However, a simulant containing plaster of Paris would start to solidify 15~20 minutes after plaster of Paris started to be mixed with other simulant components. Thus, it is important to pour the necessary amount of slurry of this simulant into a test container within 15 to 20 minutes after the start of plaster mixing.

Simulant II, consisting of 44 wt% of plaster of Paris and 56 wt% of water, has quite uniform distributions of the unconfined compressive strength and shear strength in both lateral and vertical directions. Simulant I, consisting of 27 wt% kaolin clay, 33 wt% of plaster of Paris, and 40 wt% of water, also has reasonably uniform distributions of the unconfined compressive strength and shear strength, but its vertical distribution is less uniform than for Simulant II.

It may be expected that with the minimum mixing of simulant in a 6-gallon container, the maximum unconfined compressive strength and shear strength variation may be expected to be approximately  $\pm 12\%$  or 27%. The mixing degree alone may produce up to a  $\pm 30\%$  or 85% variation of the shear strength. This variation is true for both 40 ~ 80 kPa Simulant I and 150~ over 260 kPa Simulant II.

Because these tests indicate that the degree and manner of mixing during the simulant preparation can significantly affect the simulant strength, it is important to uniformly mix the simulants in a very consistent manner to obtain the specific shear strength.

The simulants gained most of the final UCS and shear strength values after 1 day of curing and generally reached their final values in the first 3 days. The simulants tested here also have relatively steady values of unconfined compressive strength and shear strength over 7 and 10 days; thus, they have fairly long shelf-lives of 7 to 10 days to keep the strengths measured after 1 day of curing.

Under this study, we successfully made up to 22-L homogeneous simulants with shear strengths of approximately 60 kPa and 190 kPa. It is possible to obtain the vertical distributions of UCS and the shear strength of a simulant by cutting the simulant vertically and measuring its UCS and shear strength along the vertical axis with the use of a Geotest E-280 pocket penetrometer, a Haake M5 rheometer, and/or a Geonor H-60 hand-held vane tester because we have obtained the vertical distributions of UCS of the homogeneous simulants. For producing larger simulant volumes, the simulant make-up may be achieved by using multiple mixing setups to obtain the necessary amount of a simulant, all within 15~20 minutes.

The evaluations listed below should be considered as potential follow-up activities to this homogeneous simulant experimental study:

- Continue to develop and evaluate heterogeneous simulants whose shear strengths range from around 4 to 170 kPa.
- Evaluate performing side by side water-jet erosion testing with high-strength homogeneous and heterogeneous simulants to confirm that homogeneous simulants have more resistance to being eroded by a high-speed water jet than heterogeneous simulants with an equal shear strength. The stored K-Basin sludge would be heterogeneous sludge. Mining and construction industries use the "hydrodemolition" technique to remove some rocks and damaged concrete by eroding weaker parts of these heterogeneous materials. However, this concept is not yet tested for Hanford waste conditions. Thus, it is important to test this concept by conducting side-by-side testing with high-strength homogeneous and heterogeneous simulants with equal shear strength. To facilitate this side-by-side water jet testing, it will be necessary to develop high-strength heterogeneous simulants, the first item listed for consideration above.
- Evaluate the use of the high-strength homogeneous simulants developed under this study and possibly heterogeneous simulants being developed at PNNL to conduct small- and full-scale water jet testing to support the development of a suitable water jet retrieval system for stored K-Basin sludge in STSCs:
  - Small-scale water jet testing with 2-, 3-, and 4-mm nozzles to meet stored sludge erosion requirements for erosion distance (effective cleaning radius) and erosion rate.

- Full-scale water jet testing to repeat selected sets of the small-scale testing conditions with 3and/or 4-mm nozzles with an expected erosion distance of 10 inches or less. Under these test conditions, the required pressure would be expected to be less than 5,000 psi.

# Acronyms

APEL	Applied Process Engineering Laboratory
BASF	BASF Catalysts LLC is a chemical manufacturer; the producer of CP-5 amorphous alumina powder used in the testing
CHPRC	CH2M HILL Plateau Remediation Company
CP-5	product designation of the amorphous alumina powder used in the testing and is produced by BASF
DAP®	DAP Incorporated is a construction products manufacturer; the producer of the plaster of Paris used in testing
DOE	U. S. Department of Energy
ЕРК	product designation of the kaolin clay used in the testing and is produced by Edgar Minerals, Inc.
KE	K-East Basin
KW	K-West Basin
LRB	Laboratory Record Book
PNNL	Pacific Northwest National Laboratory
RPL	Radiochemical Processing Laboratory
STP	Sludge Treatment Project
STSC	sludge transport and storage container
UCS	unconfined compressive strength in $kg(f)/cm^2$

# List of Symbols

$AI_{SHAFT}$	A term of the denominator of Equation [B.2] (the surface area information, in cubic
	meters, of the vane shaft whose length is between the simulant surface and the top surface of vane rotation) in Appendix B
$AI_{VANE}$	A term of the denominator of Equation [B.2] (the surface area information of vane, in
	cubic meters) in Appendix B
D	vane diameter in meters
D <sub>S</sub>	shaft diameter in meters
Н	vane height in meters
h	vane immersion depth (the distance between the simulant surface and the top surface of the vane rotation) in meters $T_{R-PEAK}$ peak torque in N-m
T <sub>R-PEAK</sub>	peak torque in N-m
$\tau_{\rm S}$	shear strength in pascal, or kilopascal
$ au_{S-HG}$	strength measured by the Humboldt hand-held Geovane soil shear strength tester
$ au_{S-HH}$	shear strength measured by the Geonor H-60 Hand-Held Vane Tester
$ au_{\mathit{S-SHAFT}}$	Shear strength, in pascal, to which the vane shaft contributes, defined by Equation [B.7] in Appendix B.
$ au_{\mathit{S-VANE}}$	Shear strength, in pascal, to which the vane rotation area contributes, defined by Equation [B.8] in Appendix B.

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## 1.0 Introduction

Spent nuclear fuel from the N-Reactor along the Columbia River in Eastern Washington State was stored in the K-East (KE) and the K-West (KW) Basin fuel storage pools at the Hanford Site of the U.S. Department of Energy (DOE). The spent fuel storage and packaging operations resulted in the generation of radioactive sludge in these two basins. The fuel has been removed from the K-Basins, and the sludge currently resides in the KW Basin in large underwater engineered containers.

Under the Sludge Treatment Project (STP), K-Basin sludge disposition will be managed in two phases. The first phase is to retrieve the sludge that currently resides in engineered containers in the KW Basin pool at ~10 to 18°C. The retrieved sludge will be hydraulically loaded into sludge transport and storage containers (STSCs) and transported to an interim storage in Central Plateau before being treated and packaged for disposal (Honeyman and Rourk 2009). In the second phase of the STP, sludge will be retrieved from interim storage and treated and packaged in preparation for eventual shipment to the Waste Isolation Pilot Plant in New Mexico.

Structural details and dimensions of the STSCs, which include a removable voided inner annulus, are shown in Figure 1.1 (Johnson 2010). The purpose of the annulus is to enhance heat dissipation. The STSC design capacities with and without the annulus are 2.89 m<sup>3</sup> and 3.5 m<sup>3</sup>, respectively. Under current STP plans, STSCs fitted with the annulus will be used for the more radioactive containerized sludge originating from the settler tanks and is currently present in engineered container SCS-CON-230. The improved heat dissipation afforded by the annulus is unnecessary for the lower activity KE and KW sludges from the canisters, floors, and pits currently present in the engineered containers SCS-CON-210, 220, 240, 250, and 260. Each STSC will contain 0.5 to 2.1 m<sup>3</sup> of settled sludge with the specific loading dependent upon sludge type.



Figure 1.1. STSC Shown with an Inner Annulus

The inner annulus will be used with settler sludge but will be removed for other sludge materials (schematic given in Figure 9 of Johnson [2010]; diagram above reproduced from CHPRC ENGINEERING SKETCH SK-4K-M-002-1R0).

While the STSCs are stored, chemical and physical properties of K-Basin sludge are expected to change with time because of chemical reactions (whose rates increase with increasing temperature) and from compaction due to settling. For example, the sludge strength can increase by the intergrowth of sludge phase crystals. Changes in solids strength with time can impact the intensity and specialized equipment needed when sludge is retrieved from STSCs for final sludge treatment and packaging.

It is currently planned to use water jets to retrieve K-Basin sludge after interim storage. It is important to determine whether water jets can mobilize and erode the stored K-Basin sludge from the STSCs, especially the high-uranium-content KW settler sludge. The shear strength of the sludge is one of the key sludge properties to determine whether water jets would mobilize the stored sludge in the STSCs (Onishi et al. 2010). The shear strength is affected by sample history among other factors, and most of the measurements given in Poloski et al. (2002) were obtained from sludge samples that had been settled/gelled for several days to several weeks. These include the shear strength range of 1 to 8200 Pa reported for K-Basin sludge samples (predominantly collected from the KE floor, KE pits, and KE canisters) from 1995–2002 (Poloski et al. 2002, Plys and Schmidt 2009, Schmidt 2010). These measurements were mostly conducted for samples that had settled/gelled for 20 to 30 days. During the multi-year storage period, some sludge in the STSCs may consolidate/agglomerate, thus, potentially resulting in high-shear-strength material.

A 28-month study was conducted with six KE Basin sludge samples from May 2002 to September 2004 to characterize the behavior of sludge samples during long-term storage (Delegard et al. 2005). These samples were stored under hot cell storage conditions (~32 to 38°C, ~30 to 41% relative humidity, slightly below atmospheric pressure, and ~5 Rad/hour radiation field). One of the KE sludge samples, 96-13 (82 wt% uranium) used in that study had previously dried-out during storage, but was reconstituted (i.e., it was rewetted/mixed and prepared as a settled slurry, approximately 6 months before initiation of the long-term storage tests). The 96-13 sample at the end of the 28 months was found as agglomerate and was self-cemented, due possibly to its higher total uranium concentration. The 96-13 sample was described as heterogeneous cohesive sediment with "paste" material with estimated shear strength of 3 to 5 kPa joining "chunks" with an estimated shear strength ranging from 380 to 770 kPa. The bulk material shear strength was estimated at 15 to 65 kPa based on an assessment of written and video records (Wells et al. 2009).

Under a separate study, various sludge samples were subjected to hydrothermal conditions (e.g., 185°C, 10 to 72 hours), and the sludge agglomerated to form relatively high-strength material. Shear strengths were estimated to range from 9 kPa to 170 kPa. To date, this is the highest strength measured/estimated for any K-Basin sludge sample.

The shear strength of K-Basin sludge following hydrothermal treatment of up to 170 kPa was estimated by the unconfined compressive strength (UCS) measured by a pocket penetrometer in a PNNL hot cell. It is uncertain that penetrometer measurements from the hydrothermal testing provided the actual shear strength of the material. There is no universally accepted correlation to convert UCS to shear strength, and the relationship is likely material-dependent (Wells et al. 2009).

To assess the bounding material strength and potential for erosion by water jets, it is important to compare the measured shear strength to penetrometer measurements and to develop a correlation (or correlations) between UCS measured by a pocket penetrometer and direct shear strength measurements for various homogeneous and heterogeneous simulants (Wells et al. 2009).

The objective of the current evaluation was to correlate UCS measured by a Geotest E-280 pocket penetrometer to shear strength for homogeneous cohesive materials whose shear strength covers an anticipated range of several thousand pascal to 170 kPa. Homogeneity in this study is considered as spatially uniform materials. An accompanying separate study conducts similar work for heterogeneous simulants.

As part of the activity to develop the shear strength measurement comparison/correlations, a number of high-strength simulants were developed and tested under this study. The high-strength simulants developed here may also be used in developing and testing the water jet retrieval system as well as testing large-scale retrieval equipment, which will be conducted by STP.

This report describes the development of 11 high-strength homogeneous simulants whose shear strength ranges from 4 kPa to 170 kPa to cover the potential range of the stored K-Basin sludge. The simulants consist of kaolin clay, plaster of Paris, and amorphous alumina CP-5 with water. These simulants also include modeling clay purchased at a local craft store. The report also presents derived correlations between UCS measured by a Geotest E-280 pocket penetrometer and measured shear strength obtained by a bench-top Haake M5 rheometer and a Geonor H-60 hand-held vane tester (which is functionally equivalent to a Durham Geo S-162 hand-held field vane tester). Measurements made with the hand-held vane tester were also compared to shear strength values measured with the more sophisticated Haake M5 rheometer. The possible effects of simulant container sizes and simulant curing time on UCS and shear strength were also evaluated by conducting additional testing with high-strength simulants.

The composition and preparation of the simulants are described in Section 2. Section 3 presents the experimental setup, including measuring devices, simulant containers, and test procedures. The homogeneous simulant experiments, data analysis, and results are provided in Section 4. The conclusions and recommendations are presented in Section 5, while cited references are listed in Section 6. Appendix A presents operating steps and example data sheets. Appendix B discusses the vane shaft evaluation, and Appendix C provides plots of Haake M5 rheometer measurements. Appendix D presents results of re-evaluation of unconfined compressive strength measurements previously reported in PNNL-16496 (Delegard et al. 2007). Appendix E presents additional test date and their analysis results to elucidate the simulant container materials and sizes, degree of simulant mixing, and curing time on UCS and shear strength.

## 2.0 Simulants

### 2.1 Simulant Composition and Component Descriptions

Materials and compositions used for homogeneous high-shear strength simulants are shown in Table 2.1. Mixtures of water and one or two components at varying solids concentrations were prepared to achieve a range of shear strength values. (Only Simulant E, the modeling clay, contained no water.) Simulant designations are listed in increasing order of shear strength values achieved in testing; see Section 4.2 for reported shear strength values.

It should be noted that the compositions shown are as-prepared weight percentages; simulants containing water experienced small amounts of water loss to evaporation during the curing time. Also, all water used in simulant preparation was City of Richland utility water. Tests of Simulants C and H were each repeated for confirmation (only the Simulant C repeat testing values are reported here, as discussed in Section 4).

_	Simulant Composition, wt% (as prepared)				
				CP-5	
Simulant			Plaster of	Amorphous	Modeling
Designation	Water	Kaolin	Paris	Alumina	Clay
А	35	50	15		
В	34	66			
С	43	28	29		
D	41	30	29		
Е					100
F	64.5			35.5	
G	39	24.5	36.5		
Н	63			37	
Ι	37.5	24.5	38		
J	37.5	23.5	39		
K	56		44		

Table 2.1. Homogeneous Simulant Compositions

#### 2.1.1 EPK Kaolin Clay

EPK Kaolin clay, by Edgar Minerals Inc. (<u>http://edgarminerals.com/EPK-Clay.html</u>) was chosen as a low-strength simulant component that has been well characterized in previous Pacific Northwest National Laboratory (PNNL) work (Burns et al. 2010). Because of its low strength, kaolin was mixed with higher strength plaster of Paris and water to achieve varying shear strengths in several simulant formulations (Figure 2.1). Only Simulant B, 66 wt% kaolin with 34 wt% water, used kaolin and water alone.



**Figure 2.1.** Photograph of a Preliminary Kaolin-Plaster-Water Simulant Sample (note penetrometer impression visible on left-hand side)

### 2.1.2 DAP® Plaster of Paris

Commercially available plaster of Paris dry mix, by DAP Inc. (DAP 2005), was used as our highest strength simulant component. Simulant K, 44 wt% plaster of Paris and 56 wt% water, produced shear strengths in the upper range of the Geonor H-60 hand-held shear vane's capacity (it was also near the capacity of the test personnel to turn the handle of the hand-held vane). Figure 2.2 shows a preliminary test sample. As mentioned above, plaster of Paris was mixed with lower strength kaolin clay and water to achieve varying shear strengths in several simulant formulations. It should be noted that plaster of Paris mixtures will generate heat as the calcium sulfate hemi-hydrate rehydrates to form gypsum after combining with water.

#### 2.1.3 EZ Shape® Modeling Clay

EZ Shape modeling clay (<u>http://www.sculpey.com/products/clays/ez-shape</u>) is a colored, non-drying, wax-based modeling clay. It was used in testing as a mid-range strength simulant; it is the same product tested by Burns et al. (2009). For test purposes, it was formed into two cylinders each roughly 12 cm in diameter and 9 cm high. The test samples with labels indicating measurement locations are shown in Figure 2.3.



Figure 2.2. Photograph of a Preliminary Plaster-Water Simulant Sample



Figure 2.3. Photograph of Modeling Clay Samples Used in Testing

## 2.1.4 BASF CP-5 Amorphous Alumina Powder

BASF Catalysts LLC (BASF) CP-5 Amorphous Alumina powder was used as an alternative high-strength simulant component based on researcher's observations during simulant development work for a Waste Treatment Plant project.<sup>(a)</sup> Various CP-5 concentrations were tested for scoping analysis,

<sup>(</sup>a) Personal communication from RL Russell at PNNL to Y Onishi at PNNL.

ranging from 35 to 48 wt%. For the actual analysis, the CP-5 alumina powder was tested at two concentrations, 35.5 wt% and 37 wt%, in water. Figure 2.4 shows a preliminary test sample. When used in testing, these simulants had a propensity to form cracks during the curing process; see Section 2.3 for further discussion. The manufacturer notes that CP-5 alumina powder will readily adsorb water and should be protected from humidity during storage and handling. During simulant preparation, it was observed that like the plaster of Paris, CP-5 alumina will generate heat after being mixed with water.



Figure 2.4. Photograph of a Preliminary CP-5 Alumina-Water Simulant Sample

### 2.2 Simulant Preparation

Powdered simulant materials, such as kaolin and plaster of Paris, were prepared by mixing pre-weighed aliquots of each component, including the water. Dry powders were added to the water during mixing. It is not recommended to add water to plaster of Paris because this produces plaster clams and will require more intensive simulant mixing to make the simulant uniform. Kaolin and CP-5 alumina were added in small quantities and mixed in to avoid the formation of clumps. Based on the manufacturer's instructions, plaster of Paris mix was added one-half portion at a time and quickly mixed into the water or water-kaolin mixture. Both plaster of Paris and to a lesser extent the CP-5 alumina begin to set up rapidly, and the mixing time must be monitored during preparation (for plaster of Paris, a 15 to 20 minute wet working time is recommended). Mixing was accomplished both by hand and by using mechanical mixing devices (a variable speed drill with dry-wall mixing tool and several standard kitchen stand mixers).

After confirming that the mixing appeared to be uniform, the simulant was loaded into the simulant containers to the desired depth. The surface was smoothed to level, and/or surface bubbles were removed as needed based on the fresh simulant consistency. For higher-strength simulants, the hand-held shear vanes and dummy vane shafts (or shafts alone without vanes) were placed in the fresh simulant. The prepared simulant was then allowed approximately 24 hours of curing time for the material strength to develop before taking test measurements. The manufacture of the plaster of Paris, DAP, Inc., indicates that 3 days is the final curing time for this plaster of Paris product; thus, we conducted our initial 3-day

scoping test and selected the 24-hour cutting time for the actual testing to derive correlations between the unconfined compressive strength (UCS) and shear strength. Appendix E presents a series of additional tests to examine UCS and shear strength variations with a simulant curing time over up to 10 days. As indicated in Appendix E, two simulants consisting of 1) kaolin, plaster of Paris, and water and 2) plaster of Paris and water gained most of the final UCS and shear strength values after 1 day of curing and generally reached their final values in the next few days.

## 2.3 Observations on Simulant Preparation

Much of the practical knowledge needed for simulant preparation was discovered during the scoping test work. However, observations during actual testing reported in Chapter 4 and additional testing reported in Appendix E also proved important. While understanding the mechanisms behind simulant behavior was beyond the scope of our tests, several observations are important to consider in future testing:

- Sample drying at ambient laboratory temperatures during the curing period appeared to be minimal with evaporative losses of <1.5 wt% for kaolin-plaster-water mixtures and ~2.2 to 2.4 wt% for CP-5 alumina in water; thus, samples were generally left uncovered during curing. However, in preparing Simulant B (66 wt% kaolin in water) for testing, it became apparent that weaker simulants could form a hard crust at the surface; therefore, Simulants A and B were covered during curing, and testing on Simulant C was repeated to confirm that differences in drying between covered and uncovered samples did not affect test measurements.
- 2. Initial scoping test samples were prepared in 250-mL plastic beakers (example shown cut in half in Figure 2.1); however, when preparing plaster-water simulants of the same composition in the larger 4-cm square with 10-cm-deep steel test containers, larger-than-expected differences were often observed in strength measurements compared to the samples in plastic beakers. While the difference in container geometry may not be the sole or even primary factor in the strength differences seen, we concluded during the scoping that for a given test, it was best to make sure that the sample volume and geometry were the same for each container. Geometry and container composition (plastic vs. steel) would affect the rate at which samples cooled during curing; thus, it may be possible that these container properties could impact the sample strength.
- 3. Simulant container sizes and materials have only minor effects on the UCS and shear strength values of simulants of kaolin-plaster of Paris-water and plaster of Paris-water mixtures, based on additional limited tests (see Appendix E). They themselves are not a main cause of the shear strength variation, but the manner and degree of simulant mixing and some non-uniformity of the simulant within a mixing bucket may be more important factors to determine the shear strength of a simulant for the given simulant compositions and their concentrations, as reported in Appendix E in more detail.
- 4. The simulants gained most of the final UCS and shear strength values after 1 day of curing and generally reached their final values in the first 3 days (see Appendix E).
- 5. Scoping samples of CP-5 alumina in water prepared in small beakers had no cracks form in the cured simulant, while test samples of CP-5 amorphous alumina in water suffered various degrees of cracking. Cracks were mostly apparent on the simulant surface, but some of the cracks appeared to extend 1 cm or more in depth when the simulant was removed from the containers. For the CP-5 alumina, geometry was not the only concern. Testing was performed using product that was fresh from the manufacturer, while the scoping samples were prepared from older material that had been

stored for over a year. The intensity of simulant mixing (i.e., using mechanical mixing rather than stirring by hand) also appeared to increase the degree of cracking. Determining the cause of the cracking was beyond the scope of our tests. Photographs of test samples (from the same test) of 37-wt% CP-5 alumina in water are shown in Figure 2.5 and Figure 2.6. Note that for samples without vanes inserted, some samples cracked during curing while others did not; yet all samples with vanes or dummy shafts inserted formed cracks. The potential effect of cracks on shear strength is discussed in Section 4.

6. A paradox was noted in mixing these simulants: weaker materials, such as kaolin and bentonite clay (used in scoping work), achieve most of their strength as soon as they are combined with water, while stronger materials build their strength more gradually, hence requiring a curing time to achieve the desired strength for testing. This results in weaker simulants, in particular Simulants A and B, being more difficult to mix and load into simulant containers than the higher strength simulants that are more fluid immediately after mixing.



Figure 2.5. Examples of Cracked and Uncracked Samples of 37 wt% CP-5 Amorphous Alumina



Figure 2.6. Cracking in 37 wt% CP-5 Amorphous Alumina with a Shear Vane

The observations listed above describe several challenges encountered with simulant preparation during bench-scale tests. It is anticipated that these or similar issues would present challenges when preparing simulants at larger scales. For example, the challenge of uniform mixing is likely to scale with simulant volume as the high-strength simulants will begin to harden in the same period regardless of volume. Because the manner and degree of simulant mixing and some non-uniformity of the simulant within the mixing bucket appear to be very important factors to determine the shear strength of a simulant for the given simulant compositions and their concentrations, it is important to uniformly mix simulants in a very consistent way to obtain specific shear strength. The simulant container sizes and materials, curing time, and degree of simulant mixing on UCS and shear strength are evaluated and presented in Appendix E.

# 3.0 Experimental Setup

This section describes three measuring devices and a test procedure for homogeneous simulant experiments. Detailed operating steps and data sheets are provided in Appendix A.

## 3.1 Measuring Devices

Measuring devices used in this study were

- Haake M5 Rheometer
- Geonor H-60 Hand-Held Vane Tester
- Geotest E-280 Pocket Penetrometer.

The first two devices were used to measure shear strengths of the 11 homogeneous simulants, and the third device was used to measure their UCSs.

#### 3.1.1 Haake M5 Rheometer

The Haake M5 viscometer, referred to here as the Haake M5 rheometer, used in this study is a bench-top system and is connected to a computer that controls its operation, as shown in Figure 3.1 (Burns et al. 2009). This M5 system is located in the PNNL Applied Process Engineering Laboratory (APEL) Building. It was used to measure shear strengths or yield stresses in the shear of the simulants. The M5 rheometer determines shear strength by rotating a shear vane of known geometry at a constant rate to measure the resulting torque with time. The rotation speed was set to 0.3 rpm in this study.





The peak torque and the diameter and height of a shear vane were used to calculate shear strength without accounting for a vane shaft effect on the torque. Section 4.1 and Appendix B discuss this

calculation in detail. After the vane geometry is entered into the operational control computer, the computer displays the shear stress change over time and the shear strength and stores the measurements; examples are shown in Appendix C. This study used the following three shear vane sizes:

- 6 mm diameter and 6 mm height, which can measure shear strength up to about 108 kPa
- 8 mm diameter and 17 mm height, which can measure shear strength up to about 25 kPa
- 16 mm diameter and 8 mm height, which can measure shear strength up to about 9 kPa.

Rheometer performance was verified with viscosity performance checks encompassing the testing time period. In accordance with RPL-COLLOID-02 (Daniel 2007) the performance checks were done approximately every 30 days starting before testing and concluding after the last test date. A 101-centipoise Brookfield viscosity standard (lot # 122109, expiration date 3/15/2011) was used for the performance checks. An example data sheet used for the M5 performance check is included in Appendix A.

As discussed by Daniel (2007) and Burns et al. (2009), the performance checks of the Haake M5 using standard concentric-cylinder geometry confirm that the torque measurement system is operating properly, but does not verify shear strength measurement (there is no reference standard for shear strength).

#### 3.1.2 Geonor H-60 Hand-Held Vane Tester

The maximum shear strength that can be measured using the Haake M5 rheometer with the  $6 \text{ mm} \times 6 \text{ mm}$  vane is approximately 108 kPa, while the stored K-Basin sludge may have a shear strength of up to approximately 170 kPa (Delegard et al. 2007). Thus, a Geonor H-60 hand-held vane tester, which is also sold as a Durham Geo S-162 hand-held vane tester, was selected as the second device to measure shear strength. It is capable of measuring shear strength of up to 260 kPa (Geonor). As shown in Figure 3.2, it has a T-handle to push a vane to the desired test depth and to apply the shearing torque to have good control of shear-strength measurements.



Figure 3.2. Geonor H-60/Durham Geo S-162 Hand-Held Vane Shear Tester

The tester comes with the following three vanes:

- 16 mm diameter and 32 mm height, which can measure shear strength up to about 260 kPa
- 20 mm diameter and 40 mm height, which can measure shear strength up to about 130 kPa
- 25 mm diameter and 51 mm height, which can measure shear strength up to about 65 kPa.

Its accuracy is reported by its manufacturer as  $\pm 10\%$  of the measuring range.

The Geonor H-60 hand-held vane tester also comes with a dummy (shaft alone) probe (Figure 3.2), which replaces the vane to measure the shaft skin friction to provide a shaft correction factor for the shear strength estimate. Thus, it can provide the shear strength measured by a shear vane alone.

#### 3.1.3 Geotest E-280 Pocket Penetrometer

K-Basin sludge following hydrothermal treatment shows shear strength of up to 170 kPa in a PNNL hot cell. The shear strength was estimated by UCS measured by a Geotest E-280 pocket penetrometer in a PNNL hot cell. The purpose of the current study was to correlate UCS measured by a Geotest E-280 pocket penetrometer to the shear strength for homogeneous cohesive materials. This device, shown in Figure 3.3, was used in this study. It measures UCS of up to  $4.5 \text{ kg}(f)/\text{cm}^2$ . Its accuracy is reported by its manufacturer to be approximately  $0.25 \text{ kg}(f)/\text{cm}^2$ . The user calibration of pocket penetrometer is presented in Figure 3.4. This pocket penetrometer #1 with this calibration was used for all actual measurements of UCS to derive correlations of UCS and shear strength, as reported in Chapter 4 and Appendix E. In this study, values converted from pocket penetrometer readings with the calibration shown in Figure 3.4 are regarded as UCS values.



Figure 3.3. Geotest E-280 Pocket Penetrometer



Figure 3.4. Pocket Penetrometer User Calibration

The benchmark testing was achieved by head-to-head comparison of pocket penetrometer values with those of the Haake M5 rheometer and the Geonor H-60 hand-held vane tester.

#### 3.2 Simulant Containers

Based on the experience of previous PNNL researchers (Powell et al. 1997), it was recommended that for very high shear strength simulants, like plaster of Paris, the shear vanes need to be inserted into the simulant containers at the appropriate depths before simulants are cured to avoid breaking and/or cracking of hardened simulants by the insertion of the shear vanes. Simulant containers with holders for securing the shear vanes were built for these tests. The containers were constructed from stainless steel, 0.1 inch thick, and formed dimensions approximately 7 cm long, 7 cm wide, and 12.5 cm deep. Removable vane holders allowed the simulant to cure for 24 hours with the vane or a shaft secured at the appropriate depth. A photograph of one the containers used in testing is shown in Figure 3.5 with a typical vane mounted in the vane holder.



Figure 3.5. Simulant Container Holding a Geonor H-60 Hand-Held Shear Vane

The use of the vane holders for the M5 vanes was found to provide inadequate stability for curing the vanes in the simulant (the vanes were not perfectly aligned with the M5 shaft rotor and thus could not be connected to the Haake M5 rheometer). Thus, curing "in-place" for the M5 vanes was abandoned for these tests. However, no visible cracking or breaking of simulant was observed as the M5 vanes were inserted during testing because the M5 vanes used in this study are small.

During testing, it was found that these containers were not suitable for all of the softer simulants used. However, because these were weaker materials, breaking the simulant by vane insertion was not observed and not considered to be an issue. This is because the standard practice for shear vane measurement is to insert the vane into the sample just before the measurement is performed. Thus, for Simulants A and E, alternate containers were used and provided for the geometry requirements for each of the measurement devices to be met for at least 12 measurements. Each of these samples was 9 to 10 cm deep. For shear vanes, the axial geometry requirement is to have at least half a vane diameter between the vane and the container wall (Daniel 2007). For pocket penetrometers, it is recommended to have at least the diameter of the penetration foot between measurements (Durham Geo Slope Indicator). Thus, providing a distance between measurements equal to or greater than the largest device diameter for two adjacent measurements should minimize interference between them. For subsequent additional testing, 250-mL, 500-mL and 5-gallon plastic containers and 500-mL and 6-gallon carbon steel containers were used, as reported in Appendix E.

## 3.3 Test Procedure

The following steps were taken to conduct shear strength and UCS measurements with the three devices described in Section 3.1:

- Step 1: Prepare simulant (see Section 2).
- Step 2: Pour the freshly made simulant into stainless steel simulant containers to a depth of 9 to 10 cm.
- Step 3: Obtain initial simulant weight.
  - For high-strength simulants, shear vanes or shafts for the Geonor H-60 hand-held vane tester are inserted in the simulant at an appropriate depth.
- Step 4: Cure the simulant for 24 hours.
- Step 5: Obtain the simulant weight after 24-hour curing.
- Step 6: Measure shear strength and UCS by the three devices.

Twelve simulant samples were typically made for each simulant and triple measurements were usually obtained by the shear vane devices and the pocket penetrometer:

- Shear strength measured by the Haake M5 rheometer
- Shear strength measured by the Geonor H-60 Hand-Held Vane Shear Tester with an appropriate shear vane
- Shear strength measured by the Geonor H-60 Hand-Held Vane Shear Tester with a shaft alone without a shear vane
- UCS measured by the Geotest E-280 Pocket Penetrometer.

Generally, only one measurement was performed for each simulant container, usually at the center of the container cross-section (with exceptions noted at the end of Section 3.2). Thus, there should be no interference from the other measurements. When the expected shear strength was over 100 kPa, Haake M5 measurements were not performed. In this case, only nine simulant samples were made.

Appendix A presents detailed step-by step test procedures for these three devices and associated example data sheets. The Haake M5 rheometer was performance checked every month, while the pocket penetrometer was user-calibrated at the beginning of this study; their example datasheets are also shown in Appendix A.

# 4.0 Experimental Results and Analysis

### 4.1 Experimental Results

The UCS and shear strength were measured for the 11 homogeneous simulants, Simulants A through K, discussed in Section 2. These measurements were performed to obtain a correlation between the UCS and shear strength. The stress maximum occurs at the transition between the visco-elastic and fully viscous flow, and the shear strength is shear stress at this transition.

The shear strength of the K-Basin sludge is affected by sample history among other factors. Most measurements given in Poloski et al. (2002) were obtained from sludge samples that been settled/gelled for several days to several weeks. For some K-Basin sludge samples that had settled/gelled for 20 to 30 days, the shear strength ranges from 1 to 8200 Pa (Poloski et al. 2002, Plys and Schmidt 2009, Schmidt 2010). The shear strength of the 96-13 sample of K East Basin sludge was estimated to be 15 to 65 kPa after 28 months stored under hot cell storage conditions. The highest shear strengths measured for K-Basin sludge samples were between 120 and 170 kPa obtained after hydrothermal treatment (7 to 10 hours at 185°C) of high-uranium-content KE canister sludge (Delegard et al. 2007). These values were inferred from UCSs of samples measured by a pocket penetrometer. Thus, shear strengths of the 11 simulants tested in the current experiments were varied from approximately 4 kPa to 170 kPa to cover the expected shear strength values of the stored K-Basin sludge.

For each simulant, the following four sets of measurements were conducted:

- Shear strength measured by the Haake M5 rheometer
- Shear strength measured by the Geonor H-60 Hand-Held Vane Shear Tester with an appropriate shear vane
- Shear strength measured by the Geonor H-60 Hand-Held Vane Shear Tester with a shaft alone without a shear vane
- UCS measured by the Geotest E-280 Pocket Penetrometer.

The shear strength measurements with a hand-held vane tester were conducted with and without a shear vane to identify a possible effect of the shaft of the shear vane on the shear strength measurement. Shear vanes were immersed in the simulants at an appropriate immersion depth of the shear vane, based on established standards (Burns et al. 2010). For each of these four sets of measurements, three separate simulant samples were typically made to examine the repeatability of these measurements. Thus, 12 measurements with 12 samples were usually made for each simulant to avoid any interference from the other 11 measurements.

For the higher strength simulants (> 100 kPa), only three sets of measurements were typically made for each simulant. This was because the maximum shear strength that the Haake M5 rheometer could measure was approximately 108 kPa with the smallest vane (a 6 mm x 6 mm vane). Thus, the Haake M5 rheometer was not used for any simulants whose shear strengths were greater than 108 kPa. Simulant C experiments were made twice at two different occasions to examine the repeatability of the simulant making. All these measured values are reported in this section.

There was some uncertainty regarding the effect of a shear vane's shaft on a shear strength measurement. This possible effect was examined with data obtained by the Geonor H-60 hand-held vane tester with and without a shear vane.

The Haake M5 unit calculates the shear strength directly from the indicated torque by the following equation (Burns et al. 2009):

$$\tau_{s} = \frac{T_{R-PEAK}}{\frac{\pi D^{3}}{2} \left(\frac{H}{D} + \frac{1}{3}\right)}$$
(4.1)

where D = vane diameter in meters H = vane height in meters  $T_{R-PEAK}$  = peak torque in N-m  $\tau_S$  = shear strength in Pascal.

To approximate the effect of simulant stress acting on the vane and its shaft, the following equation may be used (Burns et al. 2009):

$$\tau_{s} = \frac{T_{R\_PEAK}}{\frac{\pi D_{s}^{3}}{2} \left(\frac{h}{D_{s}} - \frac{1}{6}\right) + \frac{\pi D^{3}}{2} \left(\frac{H}{D} + \frac{1}{3}\right)}$$
(4.2)

where  $D_s$  is the shaft diameter in meters, and h is the vane immersion depth (the distance between the simulant surface and the top surface of the vane rotation) in meters.

Equation 4.2 is valid, when 1) sludge failure around the shaft due to stress is equivalent to sludge shear strength, 2) the diameter of the cylindrical slip plane is equal to the vane shaft diameter, 3) the vane diameter is similar to the shaft diameter, and/or 4) the vane immersion depth is not similar to the vane height (Burns et al. 2009).

Because the Geonor H-60 hand-held vane tester comes with a shaft probe without a vane (to measure the shaft resistance alone, without a shear vane), measured data using a shaft with and without a vane were used to determine the adequacy of Equation 4.2 to estimate the shear strength values with the Haake M5. As discussed in Appendix B, Equation 4.2 does not accurately reflect the current test condition to accurately estimate the effect of shaft resistance. For example, 1) the failure of the current simulants around the shaft may be to some extent due to adhesion or friction, 2) the vane diameter is greater than the shaft diameter, and 3) the vane immersion depth is relatively similar to the vane height.

Moreover, without correcting for the shaft effect (Equation 4.1), a shear strength value would be slightly conservative (a greater shear strength value). Thus, Equation 4.1 was used to obtain the shear strength value with the Haake M5 rheometer values without compensating for the shaft effect on the shear strength.

Table 4.1 through Table 4.12 show the measured UCS and shear strength for Simulants A through K, respectively. Most of them have triple measurements. As noted above, Simulant C measurements were
repeated twice, as listed in Table 4.3 and Table 4.4. Appendix C presents plots of shear strength measurements with the Haake M5 rheometer. As discussed in Section 2.3, strengths of the simulant vary with several factors, e.g., sample size and curing time. Thus, all these simulants have a 7-cm by 7-cm cross-section with 9- to 10-cm depth, except Simulants A and E. All these simulants were cured for 24 hours before their measurements. Simulants A and E each had two large samples used to obtain all 12 measurements because of the nature of these simulants mixing and/or loading simulant containers would have been impractical in the time required. However, the sample size and geometry were chosen to meet the geometry requirements for each measurement type to prevent interference, and each of these samples was 9 to 10 cm deep.

These 12 tables show that most of the measurements, either of unconfined compressive shear strength or shear strength, have reasonably close values among these three values for each simulant. Simulant C has two sets of simulant data, and these values also reveal reasonable repeatability of making this simulant. Simulant H also has two sets. However, the first set of the Simulant H set had significantly more surface cracks than the second set of Simulant H. Thus, the first set of simulant data was not used for analysis. Even for fixed concentrations of a given CP-5 alumina simulant, surface cracks may or may not appear during simulant curing, affected by various factors, e.g., method and degree of vigorous mixing. Cracks were not observed in the other simulants tested. Any data that appeared to be directly affected by surface cracking were not considered in the current analysis.

	Simu	ılant A	
	Shear Strength, kPa		
Geonor	Geonor H-60		
Hand-Held V	Hand-Held Vane Tester		Geotest E-280
Vane and Shaft	Shaft Alone	M5 Rheometer	Pocket Penetrometer
4	0	3.517	0.044
4	0	3.434	0.041
3.5	0	3.499	0.041

Table 4.1. Shear Strength and UCS Measurements for Simulant A

**Table 4.2.** Shear Strength and UCS Measurements for Simulant B

	Simu	ılant B	
	Shear Strength, kPa		
Geonor	Geonor H-60		
Hand-Held V	Hand-Held Vane Tester		Geotest E-280
Vane and Shaft	Shaft Alone	M5 Rheometer	Pocket Penetrometer
7	0	7.87	0.10
7	0	8.76	0.10
7	0	8.53	0.10

	Simu	ılant C	
	Shear Strength, kPa		
Geonor	Geonor H-60		
Hand-Held V	Hand-Held Vane Tester		Geotest E-280
Vane and Shaft	Shaft Alone	M5 Rheometer	Pocket Penetrometer
11.5	0	16.24	0.24
11.5	0	17.58	0.23
10	0	17.02	

### Table 4.3. Shear Strength and UCS Measurements for Simulant C

Table 4.4. Shear Strength and UCS Measurements for Simulant C (repeated)

	Simulant	t C (repeat)		
	Shear Strength, kPa			
Geonor	Geonor H-60			
Hand-Held V	Hand-Held Vane Tester Haake			
Vane and Shaft	Shaft Alone	M5 Rheometer	Pocket Penetrometer	
17	0	18.71	0.22	
16		15.59	0.23	
16		16.16		
11				
10				

### Table 4.5. Shear Strength and UCS Measurements for Simulant D

	Simu	ılant D		
	Shear Strength, kPa			
Geonor	Geonor H-60			
Hand-Held V	Hand-Held Vane Tester		Geotest E-280	
Vane and Shaft	Shaft Alone	M5 Rheometer	Pocket Penetrometer	
16	0	21.01	0.23	
13	0	18.21		
14	0	18.61		

### Table 4.6. Shear Strength and UCS Measurements for Simulant E

	Simu	ılant E	
	Shear Strength, kPa		
Geonor	· H-60		
Hand-Held V	Hand-Held Vane Tester		Geotest E-280
Vane and Shaft	Shaft Alone	M5 Rheometer	Pocket Penetrometer
50	2	45.45	0.66
42	2	41.54	0.55
45	2	41.87	0.55

	Sim	ılant F	
Shear Strength, kPa			UCS, $kg(f)/cm^2$
Geonor H-60			
Hand-Held Vane Tester		Haake	Geotest E-280
Vane and Shaft	Shaft Alone	M5 Rheometer	Pocket Penetrometer
76	0	62.54	1.47
78		71.18	1.78
78		68.19	1.93

### Table 4.7. Shear Strength and UCS Measurements for Simulant F

**Table 4.8.** Shear Strength and UCS Measurements for Simulant G

	Simu	ılant G			
	Shear Strength, kPa				
Geonor	Geonor H-60				
Hand-Held V	Hand-Held Vane Tester Haake				
Vane and Shaft	Shaft Alone	M5 Rheometer	Pocket Penetrometer		
55 <sup>(a)</sup>	0 <sup>(a)</sup>	68.66	1.47		
50 <sup>(a)</sup>		77.19			
45 <sup>(a)</sup>		75.38			
(a) Surface cracks were se	en around the vane shaft.				

### Table 4.9. Shear Strength and UCS Measurements for Simulant H

Shear Strength, kPa			UCS, kg(f)/cm <sup>2</sup>
Geonor	Н-60		
Hand-Held Vane Tester Haake		Geotest E-280	
Vane and Shaft	Shaft Alone	M5 Rheometer	Pocket Penetrometer
93 <sup>(a)</sup>	0 <sup>(a)</sup>	88.54	1.78
98 <sup>(a)</sup>		94.10	1.93
		101.40	1.68

	Sim	ulant I	
	Shear Strength, kPa		
Geonor H-60			
Hand-Held Vane Tester		Haake	Geotest E-280
Vane and Shaft	Shaft Alone	M5 Rheometer	Pocket Penetrometer
141	6		2.85
131	8		3.00
129	7		2.70

#### Table 4.10. Shear Strength and UCS Measurements for Simulant I

Table 4.11. Shear Strength and UCS Measurements for Simulant J

	Sim	ulant J	
	Shear Strength, kPa		
Geonor	H-60		
Hand-Held Vane Tester		Haake	Geotest E-280
Vane and Shaft	Shaft Alone	M5 Rheometer	Pocket Penetrometer
149	6		3.71
143	2		3.51
155	8		3.46

Table 4.12. Shear Strength and UCS Measurements for Simulant K

	Simu	ılant K	
	Shear Strength, kPa		
Geonor	Geonor H-60		
Hand-Held V	Hand-Held Vane Tester		Geotest E-280
Vane and Shaft	Shaft Alone	M5 Rheometer	Pocket Penetrometer
198	0		3.00
174	0		3.31
144	0		2.80

### 4.2 Experimental Data Analysis

The measurements for each UCS measured by the pocket penetrometer and the shear strength measured by the Haake M5 and the hand-held vane tester were averaged to obtain the values shown in Table 4.13 and Table 4.14. Most cases have three values, but some have more than or less than three values, as shown in Table 4.1 through Table 4.12.

	UCS,				
	kg(f)/cm <sup>2</sup>		Shear Streng	th, kPa	
	Geotest E-280		Geonor H-	60 Hand-Held V	ane Tester
Simulant	Pocket	Haake M5	Vane and		Vane
Designation	Penetrometer	Rheometer	Shaft	Shaft Alone	Alone <sup>(b)</sup>
А	0.042	3.5	3.8	0	3.8
В	0.1.0	8.4	7.0	0	7.0
С	0.24	16.9	11.0	0	11.0
$C^{(a)}$	0.22	16.8	14.0	0	14.0
D	0.23	19.3	14.3	0	14.3
Е	0.59	43.0	45.7	2.0	43.7
F	1.73	67.3	77.3	0	77.3
G	1.74	73.7	$50^{\circ}$	$0^{\mathbb{C}}$	50
Н	1.80	94.7	95.5 <sup>(c)</sup>	0 <sup>(c)</sup>	95.5
Ι	2.85		134	7.0	127
J	3.56		149	5.3	144
Κ	3.04		172	0	172

Table 4.13. Average Values of UCS and Shear Strength for Each of the 11 Simulants

(a) Repeat.

(b) Calculated as the difference between shear strength values of a vane with a shaft and a shaft alone.

(c) Surface cracks were seen around the vane shaft.

	Simulant Initial Composition, wt%							
-	CP-5							
Simulant			Plaster of	Amorphous	Modeling	Strength <sup>(a)</sup>		
Designation	Water	Kaolin	Paris	Alumina	Clay	kPa		
А	35	50	15			3.7		
В	34	66				7.7		
С	43	28	29			15		
D	41	30	29			17		
Е					100	44		
F	39	24.5	36.5			72		
G	64.5			35.5		74		
Н	63			37		95		
Ι	37.5	24.5	38			130		
J	37.5	23.5	39			140		
Κ	56		44			170		

Table 4.14. Eleven Homogeneous Simulants and Measured Averaged Shear Strengths

The correlation between the UCS measured by the Geotest E-280 Pocket Penetrometer and the shear strength measured by the Haake M5 is shown in Figure 4.1.



Figure 4.1. Correlation Between UCS Measured by the Geotest E-280 Pocket Penetrometer and Shear Strength Measured by the Haake M5 Rheometer

As shown in Figure 4.1, the correlation between the UCS measured by the Geotest E-280 Pocket Penetrometer and the shear strength measured by the Haake M5 rheometer is

$$\tau_s = 41.9(UCS)$$
 (4.3)  
with  $R^2 = 0.937$ 

where the unit of UCS is  $kg(f)/cm^2$ , and the shear strength unit is kPa.

The corresponding correlation between the UCS measured by the Geotest E-280 Pocket Penetrometer and the shear strength measured by the Geonor H-60 Hand-Held Vane Tester is shown in Figure 4.2.



Correlation between UCS and Shear Strength of Hand Vane

**Figure 4.2.** The Correlation Between UCS Measured by the Geotest E-280 Pocket Penetrometer and the Shear Strength Measured by the Geonor H-60 Hand-Held Vane Tester

The correlation between the UCS measured by the Geotest E-280 Pocket Penetrometer and the shear strength measured by the Geonor H-60 Hand-Held Vane Tester is

$$\tau_s = 43.1(UCS)$$
 (4.4)  
with  $R^2 = 0.959$ 

Figure 4.3 shows Figure 4.1 and Figure 4.2 in a single plot for the comparison between these two correlations (Equations 4.3 and 4.4).

Figure 4.3 shows that these two correlations fit measured data reasonably well and are similar to each other, although the fit between the pocket penetrometer and the hand-held vane tester values is slightly better than that between the pocket penetrometer and the Haake M5 rheometer data.

The correlation of shear strength measured by the Haake M5 rheometer and the Geonor H-60 Hand-Held Vane Tester is shown in Figure 4.4.



**Figure 4.3.** Correlations Between UCS Measured by the Geotest E-280 Pocket Penetrometer and the Shear Strength Measured by the Haake M5 Rheometer and the Geonor H-60 Hand-Held Vane Tester



**Figure 4.4.** The Shear Strength Correlation Between that Measured by the Haake M5 Rheometer and the Geonor H-60 Hand-Held Vane Tester

As shown in Figure 4.4, the correlation of shear strength between the Haake M5 rheometer and the Geonor H-60 Hand-Held Vane Tester is

$$\tau_{S-HH} = 1.06 \tau_{S-M5}$$
 (4.5)  
with  $R^2 = 0.968$ 

where  $\tau_{S-M5}$  is the shear strength measured by the Hake M5 rheometer, and  $\tau_{S-HH}$  is the shear strength measured by the Geonor H-60 Hand-Held Vane Tester. Equation 4.5 fits the measured data well.

Simulants G and H consist of CP-5 amorphous alumina and may have a potential effect of cracks on the hand-held vane tester data. However, these figures do not show the effect, even though some cracks existed around a vane shaft. If the shear strength of Simulants G and H measured by the Geonor H-60 Hand-Held Vane Tester is also included in Figure 4.4, then it becomes Figure 4.5.



# **Figure 4.5.** The Shear Strength Correlation Between Measured by the Haake M5 Rheometer and the Geonor H-60 Hand-Held Vane Tester Including Simulants G and H's Hand-Held Vane Tester Data

As shown in Figure 4.5, the correlation of shear strength between the Haake M5 rheometer and the Geonor H-60 Hand-Held Vane Tester is

$$\tau_{S-HH} = 0.944 \tau_{S-M5}$$
 (4.6)  
with  $R^2 = 0.926$ 

The correlation (Equation 4.5) shown in Figure 4.4 has a better fit than Correlation (Equation 4.6) shown in Figure 4.5.

Burns et al. (2009) provide some insight to the shaft correction on the M5 measurements. Burns et al. (2009) previously developed a correlation between the Haake M5 rheometer and a Humboldt hand-held H-4221 Geovane soil shear strength tester in a shear strength range up to approximately 30 kPa. Although the Humboldt hand-held device is different from the Geonor H-60 Hand-Held Vane Tester used in the current study, their correlation between the two devices is presented in Figure 4.6, as information.



**Figure 4.6.** Comparison of Shear Strength Between the Haake M5 and the Humboldt Hand-Held H-4221 Geovane Soil Shear Strength Tester (Burns et al. 2009)

Burns' shear strength correlation between the Haake M5 rheometer and the Humboldt hand-held H-4221 Geovane soil shear strength tester is

$$\tau_{S-M5} = 0.96\tau_{S-HG} \tag{4.7}$$

where  $\tau_{S-HG}$  is the shear strength measured by the Humboldt hand-held Geovane soil shear strength tester, and  $\tau_{S-M5}$  is the shear strength measured by the Haake M5 rheometer. The correlation shown in Figure 4.6 and Equation 4.5 can be arranged to be

$$\tau_{S-M5} = 0.941 \tau_{S-HH} \tag{4.8}$$

Burns et al. (2009) reported that there was a significant difference of shear strength measured by the Haake M5 rheometer and the Humboldt hand-held Geovane tester. They "speculated that the difference results from (1) overestimation of shaft contribution for the M5 results, and (2) possible disruption and weakening of material by insertion and removal of the Geovane" (Burns et al. 2009). Note that the current study 1) did not use the shaft correction for the Haake M5 shear strength reading (see Appendix B), 2) a vane and a shaft alone without a vane of the Geonor H-60 Hand-Held Vane Tester were inserted to the simulant before curing the simulant for harder simulants to avoid potential disruption and weakening of the hardened simulants, and 3) a single measurement was conducted for each separate simulant container to eliminate any interference from any other measurements.

Table 4.13 and Figure 4.1 through Figure 4.6 have not clearly identified 1) whether the Haake M5 rheometer is more accurate than the Geonor H-60 Hand-Held Vane Tester for the shear strength measurements, and 2) the shaft adjustment for the M5 values. Haake M5 data without shaft adjustment would provide a greater (more conservative) shear strength value for a given UCS value. Thus, all UCS data measured by the Geotest E-280 pocket penetrometer and shear strengths measured by the Haake M5 rheometer without shaft adjustment and the Geonor H-60 hand-held vane tester with shaft adjustment were used to develop the overall correlation between the UCS and the shear strength. The result is shown in Figure 4.7. As stated above, this correlation does not include the shear strength values of Simulants G and H measured by the Geonor H-60 hand-held vane tester.



#### Correlation between UCS and Shear Strength

Figure 4.7. Correlation Between UCS and Shear Strength

The overall correlation between UCS and shear strength is shown in Equation 4.9, together with Figure 4.7.

$$\tau_s = 42.8(UCS)$$
 (4.9)  
with  $R^2 = 0.957$ 

This correlation applies to shear strengths ranging from 4 kPa to 170 kPa. The 11 simulants have four different solid materials: kaolin clay, plaster of Paris, amorphous alumina, and modeling clay. However, Figure 4.7 does not show any different trends among the simulants. Thus, considering this correlation, Equation 4.9 applies to various solid materials, at least these four solid materials or similar materials.

### 5.0 Conclusions and Recommendations

### 5.1 Conclusions

K-Basin sludge will be stored in STSCs at an interim storage location on Central Plateau before being treated and packaged for disposal. During the storage period, sludge in the STSCs may consolidate/agglomerate, potentially resulting in high-shear-strength material. Water jets will be used to retrieve K-Basin sludge after the interim storage. The Sludge Treatment Project has identified shear strength to be a key parameter that should be bounded to verify the operability and performance of retrieval systems. Determining the range of sludge shear strength is important to gain high confidence that a water-jet retrieval system can mobilize stored K-Basin sludge from the STSCs. The shear strength measurements will provide a basis for bounding sludge properties for mobilization and erosion and thus also potential simulants to investigate these phenomena.

The shear strength of K-Basin sludge varied greatly up to possibly 170 kPa. This value was estimated by measured UCS obtained by a pocket penetrometer for K-Basin sludge that underwent hydrothermal treatment (e.g., 185°C, 10 to 72 hours) in a PNNL hot cell. To date, this is the highest strength measured/estimated for any K-Basin sludge sample.

This study developed 11 homogeneous simulants, whose shear strength varies from 4 to 170 kPa. With these simulants, we developed correlations between UCS measured by a Geotest E-280 pocket penetrometer and shear strength values measured by a Geonor H-60/Durham Geo S-162 hand-held vane tester as well as a more sophisticated bench-top unit, the Haake M5 rheometer. This was achieved with side-by-side measurements of shear strengths and UCSs of homogenous samples.

The homogeneous simulants consist of kaolin clay, plaster of Paris, amorphous alumina CP-5 with water, and modeling clay. The shear strengths of most of these simulants are sensitive to many factors, including the simulant sample size and the curing time, even with given concentrations of simulant components. Table 5.1 summarizes these 11 simulants and their average measured shear strength.

The correlation of UCS measured by the Geotest E-280 pocket penetrometer and the shear strength measured by the Haake M5 rheometer and the Geonor H-60 hand-held vane tester is shown in Figure 5.1 and Equation 5.1.

		Simulan	t Initial Compos	sition, wt%		Average
- Simulant Designation	Water	Kaolin	Plaster of Paris	CP-5 Amorphous Alumina	Modeling Clay	Shear Strength, kPa
А	35	50	15			3.7
В	34	66				7.7
С	43	28	29			15
D	41	30	29			17
Е					100	44
F	39	24.5	36.5			72
G	64.5			35.5		74
Н	63			37		95
Ι	37.5	24.5	38			130
J	37.5	23.5	39			140
Κ	56		44			170

 Table 5.1.
 Eleven Homogeneous Simulants and Measured Averaged Shear Strengths

Correlation between UCS and Shear Strength



Figure 5.1. Correlation Between UCS and Shear Strength

$$\tau_s = 42.8(UCS)$$
 (5.1)  
with  $R^2 = 0.957$ 

This correlation applies to shear strengths ranging from 4 kPa to 170 kPa. The 11 simulants contain four different solid materials: kaolin clay, plaster of Paris, amorphous alumina, and modeling clay. However, Figure 5.1 does not show any different trends among the simulants. Thus, considering this correlation, Equation 5.1 applies to various solid materials, at least these four solid materials or similar materials. Additional test data implies that the error of conversion of the pocket penetrometer UCS to shear strength with Equation 5.1 may be up to 27%.

Applying Equation 5.1 to the bounding UCS measurement  $[3.54 \text{ kg}(f)/\text{cm}^2]$  made on hydrothermally treated sludge (185°C) with a pocket penetrometer gives a shear strength prediction of 152 kPa vs. the previously reported estimate of 174 kPa (reported as 170 kPa). The hydrothermally treated sludge was a heterogeneous material, and Equation 5.1 was developed from the testing of homogeneous simulants. However, the work in this study confirms that the sludge strength being used by STP as the anticipated bounding range (i.e., 150 to 200 kPa) is reasonable for sludge during retrieval from STSCs.

Additional limited testing reveals that simulant sizes and container materials have only minor effects on the unconfined compressive strength and shear strength values. These test data and their analyses indicate that simulant sizes and container materials are not a main cause of the shear strength variation, but the manner and degree of simulant mixing and some non-uniformity of the simulant within a mixing bucket may be more important factors to determine the shear strength of a simulant for the given simulant compositions and their constituent concentrations. Thus, it is important to uniformly mix simulants in a very consistent way to obtain the specific shear strength.

Under this study, we successfully made 16-  $\sim$  22-L homogeneous simulants with shear strengths of approximately 60 kPa and 190 kPa, besides the 0.2- and 0.5-L homogeneous simulants of shear strengths varying from 40 to 220 kPa. It is possible to obtain the vertical distributions of UCS and shear strength by cutting the simulant vertically and to measure the UCS and shear strength with the use of a Geotest E-280 pocket penetrometer, a Haake M5 rheometer, and/or a Geonor H-60 hand-held vane tester, as we have obtained the vertical distributions of UCS and estimated shear strength of the homogeneous simulants.

The simulants gained most of the final unconfined compressive strength and shear strength values after 1 day of curing and generally reached their final values in the first 3 days. However, a simulant containing plaster of Paris would start to solidify 15~20 minutes after the plaster of Paris begins to be mixed with the other simulant components. Thus, it is important to pour the necessary amount of slurry of this simulant into a test container within 15 to 20 minutes after the plaster of Paris begins to be mixed. For a large-volume simulant, the simulant making may be achieved with multiple mixing setups to obtain the necessary amount of a simulant, all within 15~20 minutes.

Simulant II, consisting of 44 wt% of plaster of Paris and 56 wt% of water, has quite uniform distributions of the unconfined compressive strength and shear strength in both lateral and vertical directions. Simulant I, consisting of 27 wt% of kaolin clay, 33 wt% of plaster of Paris, and 40 wt% of water, has also reasonably uniform distributions of the unconfined compressive strength and shear strength, but the vertical distribution of these strengths is less uniform.

It may be expected that with the minimum mixing of simulant in a 6-gallon container, the maximum unconfined compressive strength and shear strength variations will be approximately  $\pm 12\%$  or 27%. The

mixing degree alone may produce up to a  $\pm 30\%$  or 85% variation of the shear strength. This variation is true for both 40~ 80 kPa Simulant I and 150 ~ above 260 kPa Simulant II.

Because these tests indicate that the degree and manner of mixing during the simulant preparation can significantly affect the simulant strength, it is important to uniformly mix the simulants in a very consistent manner to obtain the specific shear strength.

The simulants gained most of the final UCS and shear strength values after 1 day of curing and generally reached their final values in the first 3 days. The simulants tested here also have relatively steady values of unconfined compressive strength and shear strength over 7 and 10 days; thus, they have fairly long shelf-lives of 7 to 10 days to maintain the strengths measured after the first day of curing.

### 5.2 Recommendations

The evaluations listed below should be considered as potential follow-up activities to this homogeneous simulant experimental study:

- Continue to develop and evaluate heterogeneous simulants whose shear strength ranges from around 4 to 170 kPa.
- Evaluate performing side by side water-jet erosion testing with high-strength homogeneous and heterogeneous simulants to confirm that homogeneous simulants have more resistance to being eroded by a high-speed water jet than heterogeneous simulants with an equal shear strength. The stored K-Basin sludge would be heterogeneous sludge. Mining and construction industries use the "hydrodemolition" technique to remove some rocks and damaged concrete by eroding weaker parts of these heterogeneous materials. However, this concept is not yet tested for Hanford waste conditions. Thus, it is important to test this concept by conducting side-by-side testing with high-strength homogeneous and heterogeneous simulants with equal shear strength. To facilitate this side-by-side water jet testing, it will be necessary to develop high-strength heterogeneous simulants, the first item listed for consideration above.
- Evaluate the use of the high-strength homogeneous simulants developed under this study and heterogeneous simulants being developed at PNNL to conduct small- and full-scale water-jet testing to support the development of a suitable water jet retrieval system for stored K-Basin sludge in STSCs.
  - Small-scale water-jet testing with 2-, 3-, and 4-mm nozzles to meet stored sludge erosion requirements for erosion distance (effective cleaning radius) and erosion rate.
  - Full-scale, water-jet testing to repeat selected sets of the small-scale testing conditions with 3and/or 4-mm nozzles with an expected erosion distance of 10 inches or less. Under these test conditions, the required pressure would be expected to be less than 5,000 psi to meet the required erosion distance and the required erosion rate.

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## Appendix A

Operating Steps and Data Sheets for Unconfined Compressive Strength and Shear Strength Measurements

### Appendix A: Operating Steps and Data Sheets for Unconfined Compressive Strength and Shear Strength Measurements

### A.1 Haake M5 Rheology Measurements

This section describes operating steps and presents an example data sheet for Haake M5 Rheometer measurements. It also provides an example data sheet for a Haake M5 rheology performance check.

### A.1.1 Operating Steps

The following operating steps refer to Data Sheet A.1 for recording measurements. These steps assume that the pre-test performance check of the Haake M5 rheometer has been completed per RPL-COLLOID-02 (Daniel 2007).

- 1. Enter a description of the specific testing to be conducted in the project-specified laboratory record book (LRB) (e.g., date, time, simulant, simulant container/batch designation/description).
- 2. Select the appropriate shear vane to be used with the simulant being tested based on recommendations generated from scoping tests.
- 3. If not already provided, enter the shear vane designation/name and accompanying description in the LRB. For the description, measure and record in the LRB the height and diameter of the shear vane and the number of individual vanes. The height and diameter of the shear vane should be obtained with calibrated calipers to a tolerance of  $\pm 0.01$  inch ( $\pm 0.2$  mm).
- 4. Mark the target depth of insertion on the selected shear vane. Provide some scale markings above and below the target mark in case the target depth is not achieved.
- 5. Complete the pre-test information on Data Sheet A.1. This includes the measurement designation of the device used and the sequence of the measurements taken with the Haake M5. Example: M5-3 is the third measurement taken with the Haake M5.
- 6. With minimal disturbance to the simulant, mark the surface of a cohesive simulant as to where a shear vane measurement is to be made. The shear vane is expected to be inserted at the center of the simulant surface.

For high-shear-strength testing, continue on to Step 7. For low-shear-strength testing, skip to Step 16.

- 7. On a simulant curing table, insert the shear vane into the simulant in a slow continuous process closely maintaining a vertical orientation near the center of the container to the designated depth, based on the marking on the simulant and the vane shaft. Record the shear vane depth on Data Sheet A.1.
- 8. Confirm that the shear vane shaft is secured to make certain that the shear vane will not move during the simulant curing.

- 9. Allow the simulant to cure in the sample container for the desired length of time (24 hours is anticipated).
- 10. Using a spare shear vane identical to the one placed in the simulant, install the shear vane in the instrument per the manufacturer's operating instructions. Make sure that the vane is securely installed with no vertical or rotational slip.
- 11. Verify the instrument settings on the control unit: Rate controller knobs set to 100 and 10, Maximum torque dial set to 100, Filter dial set to 0.
- 12. Use the zero adjust dial to achieve an indicated torque of 0% on the digital display.
- 13. Remove the spare shear vane from the M5 device.
- 14. After the simulant is adequately cured, install the shear vane (in the simulant container) into the M5 device per the manufacturer's operating instructions. Make sure that the vane is securely installed with no vertical or rotational slip while being careful not to disturb it in the simulant. Likewise, carefully raise the laboratory jack so that it just supports the simulant container.
- 15. Skip to Step 22.

For low-shear-strength testing cases, continue at Step 16.

- 16. Install the shear vane per the manufacturer's operating instructions. Make sure that the vane is securely installed with no vertical or rotational slip.
- 17. Verify the instrument settings on the control unit: Rate controller knobs set to 100 and 10, Maximum torque dial set to 100, Filter dial set to 0.
- 18. Use the zero adjust dial to achieve an indicated torque of 0% on the digital display.
- 19. Position the simulant container on the laboratory jack and raise the container until the simulant surface is just below (several mm) the bottom of the vane. Adjust the position of the container so the shear vane is directly above an indicated measurement location.
- 20. Raise the simulant container slowly with the laboratory jack. Note: After initiating the insertion of the shear vane into the simulant, no lateral adjustment is to be made to the position of the shear vane. The laboratory jack should only travel upward during the insertion.
- 21. Insert the shear vane to the target depth by positioning the target measurement depth mark on the shear vane shaft even with the simulant surface. Record the measurement depth on Data Sheet A.1.
- 22. Make sure that device settings and the name of the output file to be generated are indicated on Data Sheet A.1.
- 23. Record the measurement designation on Data Sheet A.1 and in the plan view of the simulant container.
- 24. Obtain the rheogram by measuring the torque as a function of time with a vane rotational rate of approximately 0.3 rpm. Refer to the manufacturer's operating instructions for the operation of the rheometer. If the material possesses shear strength, the rheogram will show a peak torque at the beginning, then level-off with time, and finally drop-off to a lower value. Record the time the test was performed and the shear strength measured on Data Sheet A.1. Verify that the units of the measurement are labeled correctly at the head of each column.

- 25. Lower the laboratory jack to remove the simulant container and extract the shear vane.
- 26. Take a simulant temperature reading. The temperature device should be inserted into the location where the shear vane measurement was taken. Record the temperature on Data Sheet A.1.
- 27. Clean the shear vane in preparation for the next measurement.
- 28. Repeat Steps 6 through 27 as needed.

## A.1.2 Example Data Sheet A.1: Haake M5 Shear Strength Measurements (M5 Unit)

	]	Inserted in LRB	No.:	on page:	
Date:		Related LRB	entries on page(s):		
Test Personn	el:				
Device:		LRB entry for	Shear Vane design	ation/ID/diameter/height:	
Simulant:					
Location of S	Simulant Description	n:			
	Measurement	Measured	Instrument	D	Temperature <sup>(d)</sup>
Time <sup>(a)</sup>	Designation <sup>(b)</sup>	Depth <sup>(c)</sup>	Calculated T <sub>ss</sub>	Electronic File Name for	of Simulant
(hh:mm)	(device-sequence)	(mm)	(Pa)	Stress-vsTime Data	(°C)
			$\langle V \rangle$		
		7	7/		
			<		
			/		
(a) Recorde	d in 24-hour clock for	mat.			
	s the designation for the asurement acquired w			ment was taken. Example: "M5-3"	designates the

- (c) Depth from simulant surface to top of shear vane.
- (d) Take the temperature after the shear vane measurement.

Completed by:

Printed Name and Signature

Date

Technical Reviewer:

Printed Name and Signature

Date

### A.1.3 Example Data Sheet of a Haake M5 Rheometer Performance Check

### 1. Opening and Closing Performance Checks

Verify that a rheometer performance check has been conducted within the past 30 days (as per RPL-COLLOID-02) and was acceptable. If a performance check has been run within the past 30 days, enter the relevant performance check information in the "Opening" section below. If not, then complete a performance check and fill in the table below. The acceptable range is defined as follows:

- For fluids with  $\eta_{list} > 10$  cP, the acceptable range of measured viscosity ranges from  $0.90 \times \eta_{list}$  to  $1.10 \times \eta_{list}$ .
- For fluids with  $\eta_{list} \leq 10$  cP, the acceptable range of measured viscosity ranges from  $0.85 \times \eta_{list}$  to  $1.15 \times \eta_{list}$ .

#### Performance Check of RV20-M5 and/or the RS600 Using a Brookfield Viscosity Standard

Standard lot #	Expiration date:
List Viscosity:	Acceptable range:
Thermocouple Calib. ID:	Expiration Date:
Measuring Geometry M5/RS600: MV1/Z41	Other:

					-	
Performance		Temperature		ity (cP)		
Check	Instrument	(°C)	List <sup>(b)</sup>	Measured	Acceptable <sup>(a)</sup>	File Name
Opening Date:	RS600	25				
	M5	Ambient T	N/A			
	RS600	Ambient T	N/A			
Closing Date:	R\$600	25				
	M5	Ambient T	N/A			
	RS600	Ambient T	N/A			

(a) As per RPL-COLLOID-02, Rev. 1, the acceptable range for Brookfield Fluid 50 (calculated as ±10% of the list viscosity of 48.0 cP) is 43.2 to 52.8 cP at 25°C.

(b) Viscosities at temperatures other than 25°C are not provided by the manufacturer. Viscosity measurements at ambient cell temperatures are conducted on two measurement systems (RV20-M5 and RS600); results are to agree within 10% to show acceptable performance.

Completed by:

Printed Name and Signature

#### 2. Comments

Enter any comments/observations on rheometer performance check in the table below:

Date	Comments

### 3. Records

The following records are to be attached to the LRB with completed performance check sheet:

- 1. Shear strength plots from rheometer software (Note: Multiple runs can be included on a single plot).
- 2. Certification for viscosity standard.

### 4. Technical Review (check boxes and sign)

- Software data files have been reviewed and I concur with the interpretation.
- Certification of viscosity standard is attached.
- The two pages of this "Data Collection Sheet for Haake M5 Performance Checks" have been reviewed.

Completed by:

Printed Name and Signature

Date

Technical Reviewer:

Printed Name and Signature

Date

### A.2 Geonor H-60 Hand-Held Vane Tester Measurements

This section describes operating steps and presents an example data sheet for Geonor H-60 Hand-Held Vane Tester measurements.

### A.2.1 Operating Steps

The following operating steps refer to Figure A.1 and Data Sheet A.2 for recording measurements.

- 1. Enter a description of the specific testing to be conducted in the project-specified LRB (e.g. date, time, simulant, and simulant container/batch designation/description).
- 2. Select the appropriate shear vane or the shaft probe to be used with the simulant being tested based on anticipated shear strengths obtained from scoping tests with the M5 unit or shear strengths greater than an M5 unit operating range.
- 3. If not already done, provide a shear vane designation/name and accompanying description in the LRB. The description should include the height and diameter of the shear vane and the number of individual vanes. The height and diameter of the shear vane should be obtained with calibrated calibrate of ±0.01 inch (±0.2 mm).
- 4. Mark the target depth of insertion on the selected shear vane or the shaft probe. Provide some scale markings above and below the target mark in case the target depth is not achieved.
- 5. Complete the pre-test information on Data Sheet A.2. This includes the measurement designation of the device used and the sequence of the measurements taken with the hand-held vane tester. Example: HH-3 is the third measurement taken with the hand-held vane tester.
- 6. With minimal disturbance to the simulant, mark the surface of a cohesive simulant as to where the shear vane measurements are to be made. The shear vane is expected to be inserted at around the center of the simulant surface. For very high shear strength cases, a shear vane will be inserted to the simulant before curing the simulant.

For high-shear-strength testing cases, follow Steps 7 through 11.

- 7. Insert the shear vane or the shaft probe into the simulant in a slow continuous process closely maintaining a vertical orientation near the center of the container to the designated depth, based on the markings on the simulant surface and the vane shaft. Record the measurement depth on Data Sheet A.2.
- 8. Confirm that the shear vane shaft or the shaft probe is secured to make certain that the shear vane or shaft probe will not move during the simulant curing.
- 9. After the simulant is adequately cured, install the shear vane or the shaft probe (Item 11 shown in Figure A.1) already inserted in the simulant container into the handheld vane tester per the manufacturer's operating instructions. Make sure that the vane or the shaft probe is securely installed with no vertical or rotational slip while being careful not to disturb it in the simulant.
- 10. Record the measurement designation on Data Sheet A.2. Provide the measurement designation of the device used and the sequence the measurement was taken with the Geonor H-60 Hand-Held Vane

Tester. Example: HH-3 is the third measurement taken with the hand-held vane tester with the shear vane, and HS-2 is the second measurement taken with the hand-held vane tester with the shaft probe.

11. Skip Steps 12 and 13 and then follow Steps 14 through 22.

For low-shear-strength testing cases, follow Steps 12 through 22.

- 12. Install the shear vane or the shaft probe (Item 11 shown in Figure A.1) into the hand-held device per the manufacturer's operating instructions. Make sure that the vane or the shaft probe is securely installed with no vertical or rotational slip.
- 13. Insert the shear vane or the shaft probe to the target depth by positioning the target depth mark on the shear vane shaft or the shaft probe even with the simulant surface. Insert the vane or the shaft probe into the simulant in a slow continuous process maintaining a vertical orientation. Do not twist or "wiggle" the vane or the shaft probe during the loading process. Record the measurement depth on Data Sheet A.2.
- 14. Make sure that the graduated scale (Item 5 shown in Figure A.1) is set to the "0" position.
- 15. Turn the handle clockwise as slowly as possible with constant speed. (Note: Shakedown testing will identify an appropriate rotation speed.) The lower part (Item 8 shown in Figure A.1) of the Geonor H-60 device initially follows the upper part (Item 4 shown in Figure A.1) around. When the lower part falls behind the upper part, failure has occurred, and the maximum shear strength has been obtained at the vane.
- 16. Holding the handle firmly, allow it to return to the "0" position. Do not allow the handle to spring back uncontrolled. Read the graduated scale.
- 17. Record the shear strength reading in kPa together with position of the hole and the depth on Data Sheet A.2.
- 18. Turn the graduated scale anti-clockwise back to the "0" position.
- 19. Extract the shear vane or the shaft probe from the simulant with minimal disturbance.
- 20. Take a simulant temperature reading. The temperature device should be inserted into the location where a shear vane measurement was taken. Record the temperature on Data Sheet A.2.
- 21. Turn the vane or the shaft probe quickly at least 25 revolutions. Zero the scale.
- 22. Remove the vane or the shaft probe from the hand-held vane tester body and clean the shear vane or the shaft probe in preparation for the next measurement.
- 23. Repeat Steps 6 through 21 as needed.



Figure A.1. Geonor H-60 Hand-Held Vane Tester

## A.2.2 Example Data Sheet A.2 for Geonor H-60 Shear Strength Measurements (Hand-Held Unit)

		Inserted in LRB	No.:		on page	
Date:		Related LRB	entries on page	e(s):		
Test Personnel	l:				<u>&gt;</u>	
Device and she	ear vane/shaft proł	be designation/de	escription:		-//	<u> </u>
LRB entry for	shear vane/shaft p	robe designation	n/description: _			
Simulant:				/	$\searrow$	
Location of Si	mulant Description	n:			$\sim$	
Time <sup>(a)</sup> (hh:mm)	Measurement Designation <sup>(b)</sup> (device-sequence)	Measured Depth <sup>(c)</sup> (mm)	Device Reading	Multiplier (0,5, 1, 2)	Shear Strength Reading, τ <sub>ss</sub> (kPa)	Temperature <sup>(d)</sup> of Simulant (°C)
(a) Recorded	d in 24-hour clock fo	yrmát.				

(b) Provides the designation for the device and the sequence the measurement was taken. Example: "HH-3" designates the third measurement acquired with the hand-held vane shear tester.

(c) Depth from simulant surface to top of shear vane.

(d) Take the temperature after the shear vane measurement.

Completed by:

Printed Name and Signature

Date

Technical Reviewer:

Printed Name and Signature

Date

### A.3 Geotest E-280 Pocket Penetrometer Measurements

This section describes the operating steps and presents an example data sheet for Geotest E-280 Pocket Penetrometer measurements. It also provides an example data sheet for Geotest E-280 pocket penetrometer user calibration.

### A.3.1 Operating Steps

The following operating steps refer to Figure A.2 and Data Sheet A.3 for recording measurements.

- 1. Verify that that a user-calibration check has been performed on the pocket penetrometer (or that the unit has been calibrated by an approved calibrations laboratory).
- 2. Choose a test location with care to avoid disturbed areas based on geometry requirements for measurements or non-homogeneous areas. (See Sample Geometry Guidelines below.)
- 3. Return indicator ring (shown in Figure A.2) to back position against the penetrometer body.
- 4. Complete the pre-test information on Data Sheet A.3. This includes the measurement designation of the device used and the sequence of the measurements taken with the pocket penetrometer. Example: PP-3 is the third measurement taken with the pocket penetrometer.
- 5. Grip the handle firmly and slowly insert the shaft with a smooth constant force into the simulant to a depth of <sup>1</sup>/<sub>4</sub> inch (shown in Figure A.3).
- 6. Take the unconfined compressive strength (UCS) reading in units of tons/ft<sup>2</sup> or kg/cm<sup>2</sup> from the top of the indicator ring.
- 7. Record the UCS reading together with the position of test location. If the 1-inch foot adapter is used, divide the reading by 16 to obtain UCS in  $tons/ft^2$  or  $kg/cm^2$ , as discussed below.
- 8. Extract the probe from the simulant with minimal disturbance.
- 9. Take a simulant temperature reading. The temperature device should be inserted into the location where a shear vane measurement was taken. Record the temperature on Data Sheet A.3.
- 10. Clean the probe in preparation for the next measurement.



Figure A.2. Geotest E-280 Pocket Penetrometer

The use of a penetrometer adapter foot is recommended when testing very low-strength cohesive simulants. The adapter foot has a diameter of 1 inch (25.4 mm) compared to the  $\frac{1}{4}$ -inch (6.35-mm) penetration piston. The effective area of the piston will be increased 16 times when the adapter foot is attached to the penetrometer piston; therefore, the reading must be divided by 16 to obtain the correct UCS (in tons/ft<sup>2</sup> or kg/cm<sup>2</sup>) when the adapter foot is used.

## Sample Geometry Guidelines for Unconfined Compressive Strength Measurement with the Pocket Penetrometer:

- Penetrometer foot diameter  $D_f$  (typically  $\frac{1}{4}$  inch).
- Measurements should be made  $\geq D_f$  from the sample edge/container wall.
- For multiple measurements on one sample (if they are taken in that way), subsequent measurements should be beyond the area that is visibly disturbed by previous measurements (i.e., separation distance may be  $\geq D_f$ ).
- The sample depth should be at least 2 times the foot insertion depth (i.e., <sup>1</sup>/<sub>2</sub> inch).

### A.3.2 Example Data Sheet A.3 for Geotest E-280 Pocket Penetrometer Measurements (Penetrometer Unit)

		Inserted in LRI	3 No.:		on page:	
Date:		Related LRE	B entries on p	page(s):		
Test Personn	el:					
Device and s	hear vane/shaft pro	obe designation/	description:_		$\square$	
LRB entry fo	or shear vane/shaft	probe designation	on/description	n:		2
Simulant:						
	Simulant Descripti					
Time <sup>(a)</sup> (hh:mm)	Measurement Designation <sup>(b)</sup> (device-sequence)	Penetration Foot Size (inch) ¼ or 1 inch	Inserted Depth <sup>(c)</sup> (inch) <sup>1</sup> / <sub>4</sub> inch		JCS or kg/cm <sup>2</sup> ) True <sup>(d)</sup>	Temperature <sup>(e)</sup> of Simulant (°C)
		$\land$		$\sim$		
(a) Recorde	ed in 24-hour clock f	ormat.				

(b) Provides the designation for the device and the sequence the measurement was taken. Example: "PP-3" designates the third measurement acquired with the pocket penetrometer.

(c) Depth of the inserted penetrometer head from the simulant surface. It should be <sup>1</sup>/<sub>4</sub> inch.

(d) Based on foot size (divide by 16 for a 1-inch foot) and instrument calibration.

(e) Take the temperature after the penetrometer measurement.

Completed by:

Printed Name and Signature

Date

Technical Reviewer:

Printed Name and Signature

Date

## A.3.3 Example Data Sheet for the Geotest E-280 Pocket Penetrometer User-Calibration

Calibration Steps:

- 1. Press pocket penetrometer onto a calibrated analytical balance to target balance load.
- 2. Record penetrometer measurement (top of indicator ring).
- 3. Repeat sequence three times.
- 4. Plot True vs. As-Read values and obtain linear correlation with its slope of m (forced through zero).

Device nat	me:		Date:		Foot Diam.:	1"
Balance II	O (Make, model, loc	eation):				
Balance C	alibration ID:		C	al. Exp. Date:		
			Mass measured:		g	
Check wei	ght mass 2:		Mass measured:	$\langle Q \rangle$	g	
Check wei	ght mass 3:		Mass measured:	$\searrow$	g	
	Balance Load	Un	confined Compressive	Strength, kg(f)	$/cm^2$	]
	kg(f)	$True^{(a)} (\pm 5\%)$		As-Read		
	0.00	0.00		/		
	0.50	0.29				
	1.00	0.58				
	2.00	1.17				
	3.00	1.75				
	4.00	2.33				
	5.00	2.92				
	6.00	3.50				
	7.00	4.08				
Completes	S-170/170B Du	urham Geo Slope Indi	e taken to ¼-inch penetrati cator, Pocket Penetrometer	on is 0.58 × Lo Instruction Ma	ad, kg(f) (Reference: nual, PN S-212M).	-
Completed	l by:		ame and Signature		- Date	

Technical Reviewer:

Printed Name and Signature

Date

Appendix B

Shear Vane Shaft Correction Evaluation
# **Appendix B: Shear Vane Shaft Correction Evaluation**

#### **B.1** Introduction

In the work of this report, instruments used to measure the shear strength of 11 simulants included the Haake M5 rheometer and the Geonor H-60 hand-held vane shear tester. Both of these instruments use four-bladed vanes that are attached to a vane shaft for a shear strength measurement. In this appendix, the unit consisting of four-bladed vanes and a vane shaft is referred to as a "Shear Vane Unit."

For the H-60 hand-held tester, an angular displacement of the upper part of the instrument is produced by hand rotation, and the shear strength is obtained by direct reading of the angular displacement on the graduated scale of the instrument. The scale factor is determined from the vane size information used for the measurement, and the shear strength obtained by the angular displacement reading is multiplied by this factor to evaluate the correct shear strength of the simulant.

In addition to the Shear Vane Units, the H-60 hand-held tester is provided with vane shafts without vane blades. The vane shaft without vane blades is referred to as "Shaft Alone" herein. The Shaft Alone is used for the vane shaft correction of the shear strength measured with the H-60 hand-held tester.

The M5 rheometer evaluates the shear strength of the simulant from the measured peak torque on the simulant with the vane size information of the used Shear Vane Unit which is provided with the computer software. Because, as a standard method, the vane shaft information of the M5 Shear Vane Unit is not included in the vane size information, the significance of the vane shaft effect of the Shear Vane Unit on the M5 shear strength evaluation was a concern.

In this appendix, the vane shaft effect on the shear strength evaluation with the M5 rheometer is studied. From the study conducted in the following sections, it is concluded that the vane shaft correction on the M5 shear strength evaluation, by including the vane shaft information of the Shear Vane Unit into the vane size information, is unnecessary.

#### **B.2 Study Procedure and Results**

As a standard method, the M5 rheometer evaluates the shear strength from measured peak torque via Equation B.1 of

$$\tau_{s} = \frac{T_{R-PEAK}}{\frac{\pi D^{3}}{2} \left(\frac{H}{D} + \frac{1}{3}\right)}$$
(B.1)

where  $\tau_s$  is the evaluated shear strength in pascal, D is the shear vane diameter in meters, H is the shear vane height in meters, and  $T_{R-PEAK}$  is the measured peak torque in Newton-meters.

Because Equation B.1 does not take the vane shaft effect, caused by the shaft area of the Shear Vane Unit, into account, Burns et al. (2009) modified Equation B.1 by including the vane shaft area information in the shear strength evaluation. Their modified equation is

$$\tau_{s} = \frac{T_{R-PEAK}}{\frac{\pi D_{s}^{3}}{2} \left(\frac{h}{D_{s}} - \frac{1}{6}\right) + \frac{\pi D^{3}}{2} \left(\frac{H}{D} + \frac{1}{3}\right)}$$
(B.2)

where  $D_s$  is the vane shaft diameter in meters, and h is the vane immersion depth (distance between the simulant surface and the top surface of vane rotation) in meters. The first term of the denominator in the right hand side of Equation B.2 is the surface area information, in cubic meters, of the shaft whose length is between the simulant surface and the top surface of vane rotation. The second term of the denominator in the right hand side of Equation B.2 is the rotation surface area information of vane, in cubic meters.

In order to evaluate the vane shaft effect quantitatively, Equation B.2 is subtracted from Equation B.1 as

$$\tau_{s-SHAFT} = \frac{T_{R-PEAK}}{\pi D^3} \left(\frac{H}{D} + \frac{1}{3}\right)^{-1} \frac{T_{R-PEAK}}{\pi D_s^3} \left(\frac{h}{D_s} - \frac{1}{6}\right) + \frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3}\right)$$

$$= \frac{\left(\frac{\pi D_s^3}{2} \left(\frac{h}{D_s} - \frac{1}{6}\right) + \frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3}\right) - \frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3}\right)\right) T_{R-PEAK}}{\frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3}\right) \left(\frac{\pi D_s^3}{2} \left(\frac{h}{D_s} - \frac{1}{6}\right) + \frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3}\right)\right)}$$

$$= \frac{\frac{\pi D_s^3}{2} \left(\frac{H}{D} + \frac{1}{3}\right) \left(\frac{\pi D_s^3}{2} \left(\frac{h}{D_s} - \frac{1}{6}\right) + \frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3}\right)\right)}{\frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3}\right) \left(\frac{\pi D_s^3}{2} \left(\frac{h}{D_s} - \frac{1}{6}\right) + \frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3}\right)\right)}$$

$$= \left(\frac{\frac{\pi D_s^3}{2} \left(\frac{h}{D_s} - \frac{1}{6}\right)}{\frac{\pi D_s^3}{2} \left(\frac{h}{D_s} - \frac{1}{6}\right)} + \frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3}\right)} \right) \tau_s$$
(B.3)

where  $\tau_s = \frac{T_{R-PEAK}}{\frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3}\right)}$ , Equation (B.1), was used.

 $\tau_{S-SHAFT}$ , Equation B.3, is a quantitative expression of the vane shaft effect and is the shear strength to which the vane shaft area of the Shear Vane Unit contributes.  $\tau_{S-SHAFT}$  is referred to as "shaft shear strength" in this appendix.

Equation B.3 is divided by the shear strength to obtain an alternative form of

$$\frac{\tau_{S-SHAFT}}{\tau_{S}} = \left(\frac{\frac{\pi D_{S}^{3}}{2}\left(\frac{h}{D_{S}} - \frac{1}{6}\right)}{\frac{\pi D_{S}^{3}}{2}\left(\frac{h}{D_{S}} - \frac{1}{6}\right) + \frac{\pi D^{3}}{2}\left(\frac{H}{D} + \frac{1}{3}\right)}\right)$$
(B.4)

The H-60 hand-held tester provides the Shear Vane Units with three sizes of four-bladed vanes to measure the shear strength in the range of 0 to 200 kPa. In addition, vane shafts without vane blades, Shaft Alones, are provided by the H-60 hand-held tester. The Shaft Alone is used to measure the shear strength produced by the friction between the vane shaft and the simulant to correct the shear strength measured with the H-60 hand-held tester by subtracting the shear strength measured by Shaft Alone from the shear strength measured by Shear Vane Unit. If Equation B.2 correctly takes the effect of the vane shaft area of the Shear Vane Unit into account, the shear strength measured by Shaft Alone of the H-60 hand-held tester is equivalent to the shaft shear strength,  $\tau_{S-SHAFT}$ , given by Equation B.3.

In this appendix, the shaft shear strength,  $\tau_{S-SHAFT}$ , and the ratio of the shaft shear strength to the

shear strength,  $\frac{\tau_{S-SHAFT}}{\tau_s}$ , are used to study the vane shaft correction on the shear strengths measured with

the M5 rheometer.

In the work of this report, shear strengths of eight simulants were measured with both the M5 rheometer and the H-60 hand-held tester. The measured shear strength,  $\tau_s$ , Shear Vane Unit information, and measurement conditions used for the measurements were used to evaluate the shaft shear strengths,  $\tau_{S-SHAFT}$ , and the ratio of the shaft shear strength to the shear strength,  $\frac{\tau_{S-SHAFT}}{\tau_s}$ , by applying Equation B.3 and Equation B.4.

Four Shear Vane Units for each size of four-bladed vanes for the M5 rheometer and H-60 hand-held tester and four Shaft Alones with one shaft size for the H-60 hand-held tester were provided to measure the shear strengths of simulants. For each simulant, multiple measurements were taken; therefore, measured shear strengths, evaluated ratios of the shaft shear strengths to the shear strengths, and evaluated shaft shear strengths were averaged over a number of the measurements taken for each simulant and presented in Table B.1. Table B.1 also includes the shear strength measured by Shaft Alone, which was also averaged over a number of the measurements taken.

	Data f	from M5 Rheometer M	easurements	Data	Data from H-60 Hand-held Tester Measurements				
Simulant	Measured Shear Strength <sup>(a)</sup> [kPa]	$\frac{\tau_{S-SHAFT}}{\tau_{S}}$ (a)	τ <sub>S-SHAFT</sub> <sup>(a)</sup> [kPa]	Measured shear strength <sup>(a)</sup> [kPa]	$rac{{{ au }_{S-SHAFT}}}{{{ au }_{S}}}$ (a)	τ <sub>S-SHAFT</sub> <sup>(a)</sup> [kPa]	$ au_{S}^{(a)}$ Measured by Shaft Alone [kPa]		
А	3.5	10	0.3	3.8	2	0.1	0.0		
В	8.4	5	0.4	7.0	2	0.2	0.0		
С	16.9	5	0.9	11.0	2	0.2	0.0		
C (repeated)	16.8	5	0.9	14.0	2	0.3	0.0 <sup>(b)</sup>		
D	19.8 (17.1)	11 (5)	2.2 (0.9)	14.3	4	0.5	0.0		
E	43.0	11	4.7	45.7	3	1.6	2.0		
F	67.3	11	7.4	77.3	4	2.8	0.0 <sup>(b)</sup>		
G	73.7	11	8.1						
Н	94.7	11	10.4						

**Table B.1.** Measured Shear Strengths, Evaluated Ratios of Shaft Shear Strengths to Shear Strengths, Evaluated Shaft Shear Strengths, and ShaftShear Strengths Measured by Shaft Alone

(a) Averaged value over measurements.

(b) The measurement was taken only once.

() Different shear vane size dimension was used.

#### **B.3 Discussion and Conclusion**

To take the vane shaft effect into account, for the shear strengths evaluated from the M5 rheometer measurements, Burns et al. (2009) developed Equation B.2. According to the theory described in the previous section of B.2, if Equation B.2 correctly takes the vane shaft effect into account, the shear strength measured by Shaft Alone of the H-60 hand-held tester is equivalent to the shaft shear strength,  $\tau_{S-SHAFT}$ , given by Equation B.3.

A number of observations are made from Table B.1.

- 1. For all simulants, finite values of shaft shear strengths,  $\tau_{S-SHAFT}$ , were evaluated from Equation B.3 whereas all of the shear strength readings by Shaft Alone are zero except for simulant E.
- 2. The ratios of the shaft shear strengths to the shear strengths,  $\frac{\tau_{S-SHAFT}}{\tau_s}$ , obtained from the M5

rheometer measurements are higher than those obtained from the H-60 hand-held tester measurements.

- 3. For simulant E, the shaft shear strength obtained from the H-60 hand-held tester measurement via Equation B.3 is comparable to that measured with Shaft Alone.
- 4. For simulant E, the shaft shear strength obtained from the M5 rheometer measurement via Equation B.3 is more than twice as large as that measured with Shaft Alone.

According to Equation B.3, except for the condition of  $\frac{h}{D_s} = \frac{1}{6}$  or  $D_s = 0$ , the finite values of the

shaft shear strength,  $\tau_{S-SHAFT}$ , are obtained for the finite values of shear strength measurements. The zero readings of the shear strength by Shaft Alone, especially for the cases of high-shear-strength simulants, are considered to be caused by the slip condition between the vane shaft and the simulant, and Equation B.3 does not satisfy this presumed slip condition.

The shear strength is measured by four-bladed vanes and a solid rod shaft for the M5 rheometer and H-60 hand-held tester. The simulant is contained between the vanes, and the trapped simulant moves along with the four-bladed vanes to form vane rotation surfaces. Therefore, the adhesion and/or friction between the simulant and the solid rod shaft are expected to be different from the adhesion and/or friction between the simulant and the vane rotation surfaces. It is pointed out especially that the simulant around the solid rod shaft is fractured once by the shaft when the Shear Vane Unit is inserted into the simulant whereas the simulant between the inside and outside of the vane rotation surfaces remains undisturbed until the vanes rotate. However, Equation B.2 does not take this difference into account.

From the discussion conducted above, it is considered that there is uncertainty in Equation B.2 and the uncertainly is considered to become more significant for a higher ratio of the shaft shear strength to the shear strength,  $\frac{\tau_{S-SHAFT}}{\tau_S}$ , as seen from Equation B.3 and Equation B.4. Table B.1 shows that the ratios of the shaft shear strengths to the shear strengths of the M5 rheometer are higher than those of the

H-60 hand-held tester (Note that the ratio of the shaft shear strength to the shear strength is determined from the information of the shaft surface and the vane rotation surfaces). Therefore, the M5 rheometer measurements are considered to have higher uncertainty in the shear strength evaluation by using Equation B.2. Because of this high uncertainty, the vane shaft correction on the shear strength evaluation by using Equation B.2 for the M5 rheometer measurements is not recommended. Generally, it is recommended to perform the vane shaft correction when measurements are taken for large vane immersion depths. The vane immersion depths for the measurements in the work of this report are not considered to be large as they are approximately the sizes of the vane rotation diameters.

The objective of the work of this report is to develop a correlation between UCSs measured with the Geotest E-280 pocket penetrometer and shear strengths measured with the M5 rheometer and H-60 hand-held tester to convert UCS to shear strength. To make the conversion conservative, the shear strengths obtained with the M5 rheometer are not recommend to be corrected for the vane shaft effect by Equation B.2 because, without the vane shaft correction, the higher shear strengths are given for the conversion of the Geotest E-280 pocket penetrometer readings.

As the conclusion of the study conducted in this appendix, the vane shaft correction on the M5 shear strength evaluation by using Equation B.2 is unnecessary.

### **Acronyms and Symbols:**

 $\tau_{S-SHAFT}$  Shaft shear strength, in pascal, defined by Equation B.5 in Appendix B.

Appendix C

Haake M5 Rheometer Measurements

# **Appendix C: Haake M5 Rheometer Measurements**



ThermoHaake RheoWin 11/18/2010 / 3:53 PM

Company / Operator: PNNL / Ellen Baer Date / Time / Version: 14.07.2010 / 13:20:56 PM / RheoWin Pro 296 Substance / Sample no: Ka50 -PI15 -Wa35 -3 / 07142010 Ka50 -PI15 -Wa35 -3 2: C:\Rheology Results\K -Basin homogeneous\07142010\07142010\_Ka50 -PI15 -Wa35 -1.rwd Company / Operator: PNNL / Ellen Baer

Date / Time / Version: 14.07.2010 / 13:04:11 PM / RheoWin Pro 296 Substance / Sample no: Ka50 -PI15 -Wa35 -1 / 07142010 Ka50 -PI15 -Wa35 -1

3: C:\Rheology Results\K-Basin homogeneous\07142010\07142010\_Ka50-PI15-Wa35-2.rwd

Company / Operator: PNNL / Ellen Baer

Date / Time / Version: 14.07.2010 / 13:12:36 PM / RheoWin Pro 296 Substance / Sample no: Ka50 -PI15 -Wa35 -2 / 07142010\_Ka50 -PI15 -Wa35 -2

Figure C.1. Shear Strength Measurement of Simulant A (water of 35 wt%, kaolin of 50 wt%, and plaster of Paris of 15 wt%) by a Haake M5 Rheometer

ThermoHaake RheoWin 11/18/2010 / 2:27 PM



1: C:\Rheology Results\K -Basin homogeneous\07072010\07072010\_Ka66 -Wa34 -4.rwd Company / Operator: PNNL / Ellen Baer Date / Time / Version: 07.07.2010 / 18:32:40 PM / RheoWin Pro 296 Substance / Sample no: Ka66 -Wa34 -4 / 07072010 Ka66 -Wa34 -4 2: C:\Rheology Results\K -Basin homogeneous\07072010\07072010\_Ka66 -Wa34 -2.rwd Company / Operator: PNNL / Ellen Baer Date / Time / Version: 07.07.2010 / 18:17:13 PM / RheoWin Pro 296 Substance / Sample no: Ka66 -Wa34 -2 / 07072010 Ka66 -Wa34 -2 3: C:\Rheology Results\K -Basin homogeneous\07072010\07072010\_Ka66 -Wa34 -3 3: C:\Rheology Results\K -Basin homogeneous\07072010\07072010\_Ka66 -Wa34 -3.rwd Company / Operator: PNNL / Ellen Baer Date / Time / Version: 07.07.2010 / 18:25:30 PM / RheoWin Pro 296 Substance / Sample no: Ka66 -Wa34 -3 / 07072010\_Ka66 -Wa34 -3

**Figure C.2.** Shear Strength Measurement of Simulant B (water of 34 wt% and kaolin of 66 wt%) by a Haake M5 Rheometer

ThermoHaake RheoWin 11/18/2010 / 3:51 PM



1: C:\Rheology Results\K -Basin homogeneous\07092010\07092010\_Ka28 -PI29 -Wa43 -3.rwd Company / Operator: PNNL / Ellen Baer Date / Time / Version: 09.07.2010 / 17:32:58 PM / RheoWin Pro 296 Substance / Sample no: Ka28 -PI29 -Wa43 -3 / 07092010 Ka28 -PI29 -Wa43 -3 2: C:\Rheology Results\K -Basin homogeneous\07092010\07092010\_Ka28 -PI29 -Wa43 -1.rwd 2. C. (Rheology Results & Basil Hollingeneous 07092010(07092010\_Ka28-Pl2 Company / Operator: PNNL / Ellen Baer Date / Time / Version: 09.07.2010 / 17:16:28 PM / RheoWin Pro 296 Substance / Sample no: Ka28 -Pl29 -Wa43 -1 / 07092010 Ka28 -Pl29 -Wa43 -1 3: C:\Rheology Results\K-Basin homogeneous\07092010\07092010\_Ka28-Pl29-Wa43-2.rwd Company / Operator: PNNL / Ellen Baer Date / Time / Version: 09.07.2010 / 17:26:15 PM / RheoWin Pro 296 Substance / Sample no: Ka28 -Pl29 -Wa43 -2 / 07092010\_Ka28 -Pl29 -Wa43 -2

Figure C.3. Shear Strength Measurement of Simulant C (water of 43 wt%, kaolin of 28 wt%, and plaster of Paris of 29 wt%) by a Haake M5 Rheometer

ThermoHaake RheoWin 11/18/2010 / 3:58 PM



1: C:\Rheology Results\K -Basin homogeneous\07222010\07222010\_Ka28 -PI29 -Wa43 -5.rwd Company / Operator: PNNL / Yasuo Onishi Company / Operator. FINE / rasuo Onisin Date / Time / Version: 22.07.2010 / 17:09:29 PM / RheoWin Pro 296 Substance / Sample no: Ka28 -PI29 -Wa43 -5 / 07222010 Ka28 -PI29 -Wa43 -5 2: C:\Rheology Results\K -Basin homogeneous\07222010\07222010\_Ka28 -PI29 -Wa43 -2.rwd 2. C. (Rheology Results & Basil Homogeneous 07222010/07222010\_Ka28-Pi2 Company / Operator: PNNL / Yasuo Onishi Date / Time / Version: 22.07.2010 / 16:45:37 PM / RheoWin Pro 296 Substance / Sample no: Ka28-Pi29-Wa43-2 / 07222010\_Ka28-Pi29-Wa43-2 3: C:\Rheology Results\K-Basin homogeneous\07222010\07222010\_Ka28-PI29-Wa43-3.rwd Company / Operator: PNNL / Yasuo Onishi Date / Time / Version: 22.07.2010 / 16:54:00 PM / RheoWin Pro 296 Substance / Sample no: Ka28-Pl29-Wa43-3 / 07222010 Ka28-Pl29-Wa43-3 4: C:\Rheology Results\K-Basin homogeneous\07222010\07222010 Ka28-PI29-Wa43-4.rwd Company / Operator: PNNL / Yasuo Onishi Date / Time / Version: 22.07.2010 / 17:01:20 PM / RheoWin Pro 296 Substance / Sample no: Ka28 -PI29 -Wa43 -4 / 07222010\_Ka28 -PI29 -Wa43 -4 Figure C.4. Repeated Shear Strength Measurement of Simulant C (water of 43 wt%, kaolin of 28 wt%,

and plaster of Paris of 29 wt%) by a Haake M5 Rheometer

ThermoHaake RheoWin 11/18/2010 / 4:06 PM



5: C:\Rheology Results\K-Basin homogeneous\08052010\08052010 Ka30-Pl29-Wa41-4.rwd

Company / Operator: PNNL / Yasuo Onishi Date / Time / Version: 05.08.2010 / 16:26:48 PM / RheoWin Pro 296

Substance / Sample no: Ka30 -PI29 -Wa41 -4 / 08052010 Ka30 -PI29 -Wa41 -4

Figure C.5. Shear Strength Measurement of Simulant D (water of 41 wt%, kaolin of 30 wt%, and plaster of Paris of 29 wt%) by a Haake M5 Rheometer

ThermoHaake RheoWin 11/18/2010 / 4:00 PM



1: C:\Rheology Results\K-Basin homogeneous\07302010\07302010\_ModelClay -3.rwd Company / Operator: PNNL / Yasuo Onishi Date / Time / Version: 30.07.2010 / 11:39:12 AM / RheoWin Pro 296 Substance / Sample no: ModelClay -3 / 07302010 ModelClay -3 2: C:\Rheology Results\K-Basin homogeneous\07302010\07302010\_ModelClay -1.rwd Company / Operator: PNNL / Yasuo Onishi Date / Time / Version: 30.07.2010 / 11:25:21 AM / RheoWin Pro 296 Substance / Sample no: ModelClay -1 / 07302010 ModelClay -1 3: C:\Rheology Results\K-Basin homogeneous\07302010\_ModelClay -1 3: C:\Rheology Results\K-Basin homogeneous\07302010\_ModelClay -1 3: C:\Rheology Results\K-Basin homogeneous\07302010\_ModelClay -2 Company / Operator: PNNL / Yasuo Onishi Date / Time / Version: 30.07.2010 / 11:32:13 AM / RheoWin Pro 296 Substance / Sample no: ModelClay -2 / 07302010\_ModelClay -2

**Figure C.6.** Shear Strength Measurement of Simulant E (modeling clay of 100 wt%) by a Haake M5 Rheometer

ThermoHaake RheoWin 11/19/2010 / 2:49 PM



1: C:\Rheology Results\K -Basin homogeneous\09102010\09102010\_Ka24.5Pl36.5Wa39 -3.rwd Company / Operator: PNNL / Yasuo Onishi Date / Time / Version: 10.09.2010 / 14:49:27 PM / RheoWin Pro 296 Substance / Sample no: Ka24.5Pl36.5Wa39 -3 / 09102010 Ka24.5Pl36.5Wa39 -3 2: C:\Rheology Results\K -Basin homogeneous\09102010\09102010\_Ka24.5Pl36.5Wa39 -3 2: C:\Rheology Results\K -Basin homogeneous\09102010\09102010\_Ka24.5Pl36.5Wa39 -1.rwd Company / Operator: PNNL / Yasuo Onishi Date / Time / Version: 10.09.2010 / 14:37:03 PM / RheoWin Pro 296 Substance / Sample no: Ka24.5Pl36.5Wa39 -1 / 09102010 Ka24.5Pl36.5Wa39 -1 3: C:\Rheology Results\K -Basin homogeneous\09102010\09102010\_Ka24.5Pl36.5Wa39 -2.rwd Company / Operator: PNNL / Yasuo Onishi Date / Time / Version: 10.09.2010 / 14:44:27 PM / RheoWin Pro 296 Substance / Sample no: Ka24.5Pl36.5Wa39 -2 / 09102010\_Ka24.5Pl36.5Wa39 -2

**Figure C.7.** Shear Strength Measurement of Simulant F (water of 39 wt%, kaolin of 24.5 wt%, and plaster of Paris of 36.5 wt%) by a Haake M5 Rheometer

#### ThermoHaake RheoWin 11/18/2010 / 4:10 PM



1: C:\Rheology Results\K -Basin homogeneous\08272010\08272010\_CP35.5Wa64.5 -5.rwd Company / Operator: PNNL / Jaehun Chun Date / Time / Version: 27.08.2010 / 14:45:21 PM / RheoWin Pro 296 Substance / Sample no: CP35.5Wa64.5 -5 / 08272010 CP35.5Wa64.5 -5 2: C:\Rheology Results\K -Basin homogeneous\08272010\08272010\_CP35.5Wa64.5 -1.rwd Company / Operator: PNNL / Jaehun Chun Date / Time / Version: 27.08.2010 / 14:21:58 PM / RheoWin Pro 296 Substance / Sample no: CP35.5Wa64.5 -1 / 08272010 CP35.5Wa64.5 -1 3: C:\Rheology Results\K -Basin homogeneous\08272010\_CP35.5Wa64.5 -1 3: C:\Rheology Results\K -Basin homogeneous\08272010\_CP35.5Wa64.5 -3.rwd Company / Operator: PNNL / Jaehun Chun Date / Time / Version: 27.08.2010 / 14:33:18 PM / RheoWin Pro 296 Substance / Sample no: CP35.5Wa64.5 -3 / 08272010\_CP35.5Wa64.5 -3

**Figure C.8.** Strength Measurement of Simulant G (water of 64.5 wt% and CP-5 amorphous alumina of 35.5 wt%) by a Haake M5 Rheometer

ThermoHaake RheoWin 11/29/2010 / 6:26 PM



1: C:\Rheology Results\K -Basin homogeneous\09082010\09082010\_CP37Wa63\_r2-6.rwd Company / Operator: PNNL / Ellen Baer Date / Time / Version: 08.09.2010 / 15:56:52 PM / RheoWin Pro 296 Substance / Sample no: CP37Wa63 r2-6 / 09082010 CP37Wa63 r2-6 2: C:\Rheology Results\K -Basin homogeneous\09082010\09082010\_CP37Wa63\_r2-2.rwd Company / Operator: PNNL / Ellen Baer Date / Time / Version: 08.09.2010 / 15:29:05 PM / RheoWin Pro 296 Substance / Sample no: CP37Wa63 r2-2 / 09082010 CP37Wa63 r2-2 3: C:\Rheology Results\K -Basin homogeneous\09082010\09082010\_CP37Wa63\_r2-3.rwd Company / Operator: PNNL / Ellen Baer Date / Time / Version: 08.09.2010 / 15:34:25 PM / RheoWin Pro 296 Substance / Sample no: CP37Wa63 r2-3 / 09082010 CP37Wa63 r2-3 4: C:\Rheology Results\K -Basin homogeneous\09082010\_CP37Wa63 r2-3 4: C:\Rheology Results\K -Basin homogeneous\09082010\_CP37Wa63\_r2-4.rwd Company / Operator: PNNL / Ellen Baer Date / Time / Version: 08.09.2010 / 15:34:25 PM / RheoWin Pro 296 Substance / Sample no: CP37Wa63 r2-3 / 09082010\_CP37Wa63\_r2-4.rwd Company / Operator: PNNL / Ellen Baer Date / Time / Version: 08.09.2010 / 15:42:01 PM / RheoWin Pro 296 Substance / Sample no: CP37Wa63\_r2-4 / 09082010\_CP37Wa63\_r2-4. Figure C.9. Shear Strength Measurement of Simulant H (water of 63 wt% and CP-5 amorphous alumina

of 37 wt%) by a Haake M5 Rheometer

Appendix D

Re-Evaluation of UCS Measurement Previously Reported in PNNL-16496

# Appendix D: Re-Evaluation of UCS Measurement Previously Reported in PNNL-16496

The shear strength of K-Basin sludge following hydrothermal treatment of up to 170 kPa was estimated by unconfined compressive strength (UCS) measured by a pocket penetrometer in a PNNL hot cell (Delegard et al. 2007). Although there is a published correlation from the unconfined compressive strength to shear strength,<sup>(a)</sup> this correlation is not universally accepted. The short-duration hydrothermal tests were conducted at temperatures much greater than the projected maximum temperature of the T Plant canyon cells (i.e., 33°C); however, the strength results provide an initial bounding target for sludge stored for many years as well as an upper range for simulants. To date, this is the highest strength measured/estimated for any K-Basin sludge sample.

As noted, there is no universally accepted correlation to convert UCS to shear strength, and the relationship is likely material-dependent. In this Appendix D, the UCS measurements made by Delegard et al. (2007) have been re-evaluated using the correlation for the UCS developed in this current work.

The information and data from Table 3.5 of Delegard et al.<sup>(a)</sup> is reproduced below in Table D.1. The UCS of the sludge products from the five hydrothermal treatment tests was determined using a soil penetrometer. The original estimates of shear strengths<sup>(a)</sup> of the product sludges were derived from the UCS values using a conversion provided by Holtz and Kovacs (1981). Table D.1 also includes the shear strength estimates based on the correlation of UCS measured by the Geotest E-280 pocket penetrometer and shear strength measured by the Haake M5 rheometer and the Geonor H-60 hand-held vane tester with homogeneous simulants (see Equation 5.1).

The two approaches for shear strength estimates from the UCS measurements provided similar results. The relative percent difference between the two techniques is  $13 \sim 22\%$  for all five hydrothermal tests.

Therefore, the work in this study confirms the sludge strength being used by STP as the anticipated bounding range (i.e., 150 to 200 kPa) for sludge during retrieval from STSCs.

#### Reference

Holtz RD and WD Kovacs. 1981. An Introduction to Geotechnical Engineering, Table 11-6, pp. 572-573, Prentice-Hall, Inc., Englewood Cliffs, NJ. See also pages 9-3 and 9-9 of P Blum. 1997. *Physical Properties Handbook: A Guide to the Shipboard Measurement of Physical Properties of Deep-Sea Cores*. Technical Note 26, Ocean Drilling Program, Texas A&M University, College Station, Texas. Available at: http://www-odp.tamu.edu/publications/tnotes/tn26/CHAP9.PDF.

Shear strength, kPa = 
$$\frac{\text{UCS, kg(f)/cm}^2}{2} \times \frac{9.81 \text{ m}}{\text{sec}^2} \times \frac{10^4 \text{ cm}^2}{\text{m}^2} \times \frac{\text{kPa}}{(10^3 \text{ kg/m} \cdot \text{sec}^2)}$$

<sup>(</sup>a) Delegard et al. 2007 estimated shear strength from UCS according to Holtz and Kovacs (1981):

	Unconfined Compressive Strength, kg(f)/cm <sup>2</sup>					<b>Original Shear</b>	New Shear	Consistency		
Test	As-Read			True <sup>(a)</sup>		Strength	Strength Estimate	Consistency		
1051	Rep. 1 Re	Rep. 2 Rela Diff.	n. 1 Ren. 2	Relative	Rep. 1	Rep. 2	Estimate, <sup>(b)</sup> kPa	Based on New	Corp of	British
			Кер. 1		Kepi 2		nopri	nep: 2	(PNNL-16496)	<b>Correlation</b> <sup>(c)</sup>
1	3.15	3.35	6	3.33	3.54	170	<mark>150</mark>	Very stiff	Very stiff	
2	2.50	2.00	22	2.64	2.12	120	<mark>100</mark>	Very stiff	Stiff	
3	0.35	0.25	33	0.37	0.26	16	<mark>13</mark>	Soft	Very soft	
4	0.15	0.20	29	0.16	0.21	9	8	Very soft	Very soft	
5	0.25	0.25	0	0.26	0.26	13	<mark>11</mark>	Soft	Very soft	

Table D.1. Strengths of Processed Sludges

(a) True UCS =  $1.0577 \times \text{As-Read UCS}$ . See Section 2.4.

(b) Shear strength estimated from UCS according to Holtz and Kovacs (1981):

Shear strength, kPa = 
$$\frac{\text{UCS, kg(f)/cm}^2}{2} \times \frac{9.81 \text{ m}}{\text{sec}^2} \times \frac{10^4 \text{ cm}^2}{\text{m}^2} \times \frac{\text{kPa}}{(10^3 \text{ kg/m} \cdot \text{sec}^2)}$$

(c)  $\tau_s = 42.8(UCS)$ 

(d) Consistency descriptions obtained from Corps of Engineers (1994) and "Consistency/strength of clay mixtures" (Solum 2005) are based on UCS. These descriptions are used in the present document.

- Fluid mud (UCS  $< 0.02 \text{ kg/cm}^2$ ).
- Very Soft (UCS 0.02-0.25 kg/cm<sup>2</sup>) Easily penetrated several inches by thumb. Exudes between fingers and thumb when squeezed.
- Soft (UCS 0.25-0.5 kg/cm<sup>2</sup>) Easily penetrated one inch by thumb. Molded by light finger pressure.
- Medium (UCS 0.5-1.0 kg/cm<sup>2</sup>) Can be penetrated <sup>1</sup>/<sub>4</sub>" by thumb with moderate effort. Molded by strong finger pressure.
- Stiff (UCS 1.0-2.0 kg/cm<sup>2</sup>) Indented about  $\frac{1}{4}$ " by thumb but penetrated only with great effort.
- Very stiff (UCS 2.0-4.0 kg/cm<sup>2</sup>) Readily indented by thumb nail.
- Hard (UCS > 4.0 kg/cm<sup>2</sup>) Difficult to indent by thumb nail.
- (e) Consistency descriptions by Clayton et al. (1995) and British Standard (1999; *in italics*) are based on shear strengths and are similar to those given for UCS but with ~50% higher strength thresholds. These descriptions are used for comparison and completeness but are not otherwise used in the present document.
  - Very soft (shear strength <20 kPa) Exudes between fingers when squeezed in hand. Finger easily pushed in up to 25 mm.
  - Soft (shear strength 20-40 kPa) Molded by light finger pressure. Finger pushed in up to 10 mm.
  - Firm (shear strength 40-75 kPa) Can be molded by strong finger pressure. *Thumb makes impression easily*.
  - Stiff (shear strength 75-150 kPa) Cannot be molded by fingers. Can be indented by thumb. Can be indented slightly by thumb.
  - Very stiff (shear strength 150-300 kPa) Can be indented by thumb nail. Can be indented by thumb nail.
  - Hard (shear strength >300 kPa) Cannot be indented by thumb nail. Can be scratched by thumb nail.

D.2

Appendix E

Preliminary Evaluation of Possible Effects of Simulant Size, Simulant Container Materials, Degree of Mixing, and Simulant Curing Time on Shear Strength

# Appendix E: Preliminary Evaluation of Possible Effects of Simulant Size, Simulant Container Materials, Degree of Mixing, and Simulant Curing Time on Shear Strength

## **E.1** Introduction

After performing the testing to develop the strength correlation, follow-on investigations were conducted to evaluate effects of simulant batch size, simulant curing time, and the degree of simulant component mixing. Before collecting the final measurements used to develop the correlations between the unconfined compressive strength (UCS) and shear strength of simulants, a series of scoping tests were conducted. These scoping tests were used to identify and develop simulants that are homogeneous and have a shear strength of approximately 4 kPa to 170 kPa and to select appropriate simulant curing times. Most of these scoping tests used 250-mL plastic simulant containers and 500-mL stainless steel simulant containers, as shown in Figure 2.1 and Figure 3.5. The scoping tests revealed that, with identical simulant components and concentrations, the UCS and shear strength of simulants measured in 250-mL plastic containers were often greater than those measured in 500-mL stainless steel containers.

The manufacturer indicates that 3 days is the final curing time for the plaster of Paris product; hence, the initial test in this study was a 3-day test. Figure E.1 shows a scoping test that evaluated the curing time of a simulant consisting of 45 wt% plaster of Paris and 55 wt% water over 3 days. The UCS was measured with the Geotest E-280 pocket penetrometer. The UCSs of the simulants appear to change only moderately from 1 to 3 days of curing.



Unconfined Compressive Strength, kg/cm2

**Figure E.1.** Measured Unconfined Compressive Strength of the Simulant Consisting of 45 wt% of Plaster of Paris and 55 wt% of Water in a 250-mL Plastic Container for 3 Days

The shear strength may be estimated from the measured UCS with Equation 4.9 (same as Equations S.1 and 5.1) and shown below as Equation E.1:

$$\tau_s = 42.8(UCS) \tag{E.1}$$

Replotting Figure E.1, the estimated shear strength of this simulant over 3 days is shown in Figure E.2. These two figures indicate that the UCS and possibly the shear strength were already near their final/maximum value after 1 day, and further changes were gradual over 0.5 to 3 days.



**Figure E.2.** Estimated Shear Strength of the Simulant Consisting of 45 wt% of Plaster of Paris and 55 wt% of Water in a 250-mL Plastic Container for 3 Days

To avoid the potential effects of simulant size, materials, and curing duration on shear strength, all actual tests to derive correlations between the UCS and shear strength were conducted with 500-mL stainless steel containers, and the UCS and shear strength were measured at approximately 24 hours after the simulant was made, as previously discussed.

Upon a request from the project client, CHPRC, additional limited tests, beyond those reported in the Client Review Draft,<sup>(a)</sup> were then performed to evaluate the possible effects of simulant sizes, simulant materials, and curing durations. These test results, including the potential mixing degree effect on shear strength, are presented here.

<sup>(</sup>a) 53451-RPT14, Rev A, "Client Review Draft: Development of K Basin High-Strength Homogeneous Sludge Simulants and Correlation Between Unconfined Compressive Strength and Shear Strength," issued on December 10, 2010 (via transmittal Letter 53451-L34).

## E.2 Test Data

#### E.2.1 Test Materials

Most of 11 simulants developed under this study consist of kaolin clay and plaster of Paris with water, as shown in Table 4.14. Thus, simulants similar to or the same as Simulants F and K were selected for these evaluations.

- Simulant I consists of 27 wt% kaolin clay, 33 wt% plaster of Paris, and 40 wt% water.
- Simulant II consists of 44 wt% plaster of Paris and 56 wt% water.

Particle-size distributions of kaolin clay and plaster of Paris used in this study are shown in Figures E.3 and E.4.



Figure E.3. Particle-Size Distribution of Kaolin Clay



Figure E.4. Particle-Size Distribution of Plaster of Paris

The following five simulant containers were used for these additional tests:

- 250-mL plastic container (see Figure 2.1)
- 500-mL stainless steel container (see Figure 3.5)
- 500-mL plastic container
- 5-gallon plastic container with the top diameter of 28.4 cm and height of 37 cm
- 6-gallon carbon steel container with the top diameter of 34.5 cm and height of 20.5 cm.

Simulants I and II were made and poured in the simulant containers within 15 to 20 minutes after plaster of Paris was mixed with other simulant components (i.e., kaolin and water).

The following two sets of Simulant I (27 wt% kaolin clay, 33 wt% plaster of Paris, and 40 wt% water) were made:

- Simulant I-a was mixed vigorously with electric drill/mixing devices (a variable-speed electric drill with mixing vanes) and was poured in 250-mL, 500-mL and 5-gallon plastic containers and 500-mL stainless steel and 6-gallon carbon steel containers.
- Simulant I-b was mixed gently, mainly by hand, using a spatula and whisk (an electric mixer was used only for combining kaolin and water) and was poured in 500-mL and 500-mL stainless steel containers.

In both simulant preparations, the mixing process was sufficient to achieve uniformity.

The following four sets of Simulant II (44 wt% plaster of Paris and 56 wt% water) were also prepared:

- Simulant II-a was mixed both by hand and with an electric drill/mixer to evaluate a possible container size effect of 250-mL, 500-mL, and 5-gallon plastic containers and the 500-mL stainless steel container on shear strength.
- Simulant II-b was mixed gently by hand and slightly with an electric drill/mixer to evaluate a possible container size effect of 500-mL stainless steel and 6-gallon carbon steel containers on shear strength.
- Simulant II-c was also mixed gently by hand (no electric drill/mixer was needed) to examine possible effects of container material of 500-mL stainless steel and plastic containers on shear strength.
- Simulant II-d was mixed vigorously with electric drill/mixing devices to check the possible effects of the degree of mixing with 250-mL, 500-mL, and 5-gallon plastic containers, as well as 500-mL stainless steel containers.

The 5-gallon plastic container has a diameter of 28 cm at the top, and a depth of 37 cm. The 6-gallon carbon steel container has a 35.5-cm diameter at the top and a 23-cm depth. Figures E.5 and E.6 show photographs of Simulant I-a and II-a, respectively, both made in the 5-gallon plastic container.



Figure E.5. Simulant I-a Consisting of 27 wt% of Kaolin Clay, 33 wt% of Plaster of Paris, and 40 wt% of Water Made in the 5-gallon Plastic Container. The Top and Bottom Diameters of the Simulant Are 27.5 and 25.5 cm, Respectively, and Its Height Is 26.5 cm.



**Figure E.6.** Simulant II-a Consisting of 44 wt% of Plaster of Paris and 56 wt% of Water Made in the 5-gallon Plastic Container. The Top and Bottom Diameters of the Simulant Are 28.0 and 25.5 cm, Respectively, and Its Height Is 32.0 cm.

The UCS was measured by pushing the E-280 pocket penetrometer to the depth of about 6 mm from the simulant surface. The M5 rheometer had a vane with 6-mm diameter and 6-mm height, and the vane was inserted into the simulants to a depth of about 6 mm from the simulant surface. Thus, it measured the shear strength in the depth from the 6-mm to 12-mm portion of the simulants. The H-60 hand-held vane tester has a 16-mm diameter and a 32-mm height, and the vane was inserted into the simulant to a depth of about 16 mm from the simulant surface. Thus, it measured the shear strength in the depth from the simulant surface. Thus, it measured the shear strength in the depth from the simulant surface. Thus, it measured the shear strength in the depth from the 16-mm to 48-mm portion of the simulant.

#### E.2.2 Test Data

Test results that Simulants I and II made in different simulant containers are summarized in Table E.1. The UCS and shear strength data shown in this table were measured after 1 day of simulant curing.

	Simu	lant	Simulant Con	tainer	Unconfined	
Simulant Composition			Size	Compressive Strength kg/cm <sup>2</sup>	Shear Strength kPa	
p		192	Plastic	250 mL	1.80	79
	т	464	Stainless Steel	500 mL	1.70	71
27 wt% Kaolin	I-a	472	Plastic	500 mL	1.90	79
33 wt% Plaster		15,750	Plastic	5 gallons	1.80	63
40 wt% Water		482	Plastic	500 mL	1.34	43
	I-b	494	Stainless Steel, #A	500 mL	1.09	41
		499	Stainless Steel, #B	500 mL	1.24	
		207	Plastic	250 mL	3.93	226
	II-a	445	Stainless Steel	500 mL	3.73	148
	11 <b>-</b> a	477	Plastic	500 mL	3.63	190
		18,620	Plastic	5 gallons	4.49	192
	II-b	454	Stainless Steel, #B	500 mL	3.83	196
44 wt% Plaster		460	Stainless Steel, #A	500 mL	3.33	160
56 wt% Water		21,920	Carbon Steel	6 gallons	3.53	188
	II-c	445	Stainless Steel	500 mL	2.73	160
	11-0	477	Plastic	500 mL	2.93	162
			Plastic	250 mL	> 4.5	> 260
	II-d		Stainless Steel	500 mL	> 4.5	> 260
	11-0		Plastic	500 mL	> 4.5	> 260
			Plastic	5 gallons	> 4.5	> 260

 Table E.1. Test Results for Five Simulant Containers (One-Day Cure Time)

Test Results for UCS and shear strength measured over 7 days for 15,750-mL Simulant I-a in the 5-gallon plastic container are summarized in Table E.2. As indicated above, Simulant I-a consists of 27-wt% of kaolin clay, 33 wt% of plaster of Paris, and 40 wt% of water.

**Table E.2.** Unconfined Compressive Strength and Shear Strength, Measured Over 7 Days, of SimulantI-a Consisting of 27 wt% of Kaolin Clay, 33 wt% of Plaster of Paris, and 40 wt% of Water,Made in the 5-gallon Plastic Simulant Container

	Simulant	Unconfined Compressive Strength kg/cm <sup>2</sup>	Shear Strength kPa		
Simulant	Curing Days	5-gallon Plastic Simulant Container			
	1	1.80	64		
	2	1.72	73		
	3	1.74	70		
I-a	4	1.70	_		
	5	1.64	_		
	6	1.64	67		
	7	1.59	69		

Tables E.3 and E.4 summarize the UCS and shear strength of 18,620-mL Simulants II-a and 21,920-mL II-b measured over 10 and 7 days, respectively. These simulants consist of 44 wt% of plaster of Paris and 56 wt% of water. Table E.1 provides additional simulant information.

		Unconfined Compressive Strength	Shear Strength		
	Simulant	kg/cm <sup>2</sup>	kPa		
Simulant	Curing Days	5-gallon Plastic Simulant Cor	ntainer		
	1	4.49	192		
	2	3.95	—		
	3	3.64	—		
	4	3.83	—		
II-a	5	3.74	—		
11 <b>-</b> a	6	3.46	—		
	7	3.69	—		
	8	3.59	—		
	9	3.59			
	10	3.56			

**Table E.3.** Unconfined Compressive Strength and Shear Strength, Measured Over 10 Days, ofSimulant II-a, Consisting of 44 wt% of Plaster of Paris and 56 wt% of Water, Made in the5-gallon Plastic Simulant Container

**Table E.4.** Unconfined Compressive Strength and Shear Strength, Measured Over 7 Days, ofSimulant II-b, Consisting of 44 wt% of Plaster of Paris and 56 wt% of Water

		Unconfir	ned Compressive kg/cm <sup>2</sup>	e Strength	Shear Strength kPa			
	Cinculant	6-gallon	500-mL	500-mL	6 gallon	500-mL	500-mL	
Simulant	Simulant Curing Days	Carbon Steel Container	Container #A	Stainless Steel Container #B	Carbon Steel Container	Stainless Steel Container #A	Stainless Steel Container #B	
Sinuant	0.5	4.03	3.38	4.27	192			
	1	3.53	3.33	3.83	192	160	106	
	1					100	196	
	2	3.41	3.18	3.63	182			
II-b	3	3.36	3.03	3.68	158	—		
11-0	4	3.33	3.13	3.63				
	5	3.36	2.93	3.78	160	—		
	6	3.28	3.03	3.48				
	7	3.29	2.83	3.43	168	—	—	

## E.3 Data Analysis

# E.3.1 Effects of Simulant Sizes, Container Materials, and Degree of Mixing on Shear Strength

#### E.3.1.1 Overall Plots

To examine the possible effects of simulant container sizes on UCS and shear strength, test data shown in Table E.1 were plotted against the simulant container sizes. Figures E.7 and E.8 show the measured UCS versus simulant container sizes for Simulants I-a, I-b, II-a, II-b, and II-c.



#### Unconfined Compressive Strength, kg/cm2

**Figure E.7.** Unconfined Compressive Strength, Measured at a 24-hour Curing Time, of Simulants I–a and I-b, Consisting of 27 wt% Kaolin Clay, 33 wt% Plaster of Paris, and 40 wt% Water Versus Volumes of Simulants Made in 250-mL, 500-mL, and 5-Gallon Plastic Containers, and a 500-mL Stainless Steel Container



Figure E.8. Unconfined Compressive Strength, Measured at a 24-hour Curing Time, of Simulants II-a, II-b, and II-c, Consisting of 44 wt% Plaster of Paris and 56 wt% Water Versus Volumes of Simulants Made in 250-mL, 500-mL, and 5-Gallon Plastic Containers, and 500-mL Stainless Steel and 6-gallon Carbon Steel Containers

These two figures do not show any discernable trend of UCS as a function of simulant container sizes.

Measured and estimated shear strengths of Simulants I-a, I-b, II-a, II-b, and II-c are shown as a function of simulant container sizes in Figures E.9 and E.10. The estimated shear strength was obtained using the measured UCS and the conversion Equation E.1.



Figure E.9. Measured and Estimated Shear Strength of Simulants I-a and I-b Consisting of 27 wt% Kaolin Clay, 33 wt% Plaster of Paris, and 40 wt% Water Versus Volumes of Simulants Made in 250-mL, 500-mL and 5-Gallon Plastic Containers and a 500-mL Stainless Steel Container, Measured at a 24-hour Curing Time



Figure E.10. Measured and Estimated Shear Strength of Simulants II-a, II-b, and II-c Consisting of 44 wt% Plaster of Paris and 56 wt% Water Versus Volumes of Simulants Made in 250-mL, 500-mL, and 5-Gallon Plastic Containers, and 500-mL Stainless Steel and 6-Gallon Carbon Steel Containers, Measured at a 24-hour Curing Time

Figures E.9 and E.10 do not show any identifiable relationships between the shear strength and simulant container sizes. We may expect to get the target UCS and shear strength with  $\pm 30\%$  or 85% variations. Comparing Simulant I and II variations on shear strength also indicates that these simulants have about the same maximum variations because of the difference in the mixing degree, about  $\pm 30\%$  or 85% variations.

Figures E.7 through E.10 indicate that the UCS and shear strength are not a direct function of the simulant container sizes. However, during the scoping tests before conducting the actual simulant testing to derive the correlations between UCS and shear strength, it was observed that the UCS and shear strength of simulants in 250-mL plastic containers were often greater that those in 500-mL stainless steel containers with the identical simulant components and concentrations. Thus, possible factors affecting the UCS and shear strength were examined further, such that some of the 11 simulants developed under this study might be used for the K-Basin Project in containers larger than the 500-mL stainless steel container used in this study.

#### E.3.1.2 Simulant I

Figure E.11 shows a comparison of the UCS of Simulant I-a made in the 250-mL plastic, 500-mL plastic, 500-mL stainless steel, and 5-gallon plastic containers measured at a 24-hour curing time, depicting the simulant size vs. UCS measured by the Geotest E-280 pocket penetrometer.



#### Measured Unconfined Comressive Strength, kg/cm2

Figure E.11. Unconfined Compressive Strength, Measured at a 24-hour Curing Time, of Simulant I-a Consisting of 27 wt% Kaolin Clay, 33 wt% Plaster of Paris, and 40 wt% Water Versus Volumes of Simulants Made in 250-mL, 500-mL, and 5-Gallon Plastic Containers, and a 500-mL Stainless Steel Container

Figure E.11 shows that the measured UCS is the smallest for the 0.5-L simulant made in the 500-mL stainless steel container, and the greatest value was that of the 0.5-L simulant made in the 500-mL plastic
container. The smallest (0.2-L) and largest (16-L) simulants made in 250-mL and 5-gallon plastic containers have UCS values between these two maximum and minimum UCS values.

Figure E.12 presents the shear strength of the same Simulant I-a measured with the Haake M5 rheometer. This figure also includes estimated shear strength values. The estimated shear strength was obtained from the measured UCS values and Equation E.1.

The Haake M5 rheometer had a vane with 6-mm diameter and 6-mm height, and the vane was inserted into the simulants to about 6 mm from the simulant surface. Thus, it measured the shear strength in the depth of the simulants from 6-mm to 12-mm. The UCS was measured by pushing the pocket penetrometer into the simulants to a depth of about 6 mm from the simulant surface.



Shear Strength, kPa

Figure E.12. Measured and Estimated Shear Strength of Simulant I-a Consisting of 27 wt% Kaolin Clay, 33 wt% Plaster of Paris, and 40 wt% of Water Versus Volumes of Simulants Made in 250-mL, 500-mL, and 5-gallon Plastic Containers, and a 500-mL Stainless Steel Container, Measured at a 24-Hour Curing Time

Figure E.12 indicates that the measured shear strength was the smallest for the 16-L simulant made in the 5-gallon plastic container, and the simulants made in the smaller plastic containers are very similar to each other and had the greatest shear-strength values. The shear strength of the 0.5-L simulant made in the 500-mL stainless steel container is greater than that of the 16-L simulant, but is below those of the 0.2- and 0.5-L simulants made in the 250-mL and 500-mL plastic containers.

Figure E.12 also provides some insights as to the accuracy of the conversion equation, Equation E.1. The large discrepancy between measured and estimated shear strength of the 16-L simulant may be due to possible non-uniformity of the simulant strength, especially near the simulant surface. This figure implies that the error of conversion of the pocket penetrometer UCS measurement to shear strength using Equation E.1 may be up to 22%.

Corresponding figures to Figures E.11 and E.12 are shown in Figures E.13 and E.14 for Simulant I-b. Simulant I-b had the minimum amount of mixing, but still allowed enough mixing to have simulant uniformity in the 500-mL plastic and stainless steel containers. Simulant I-b was primarily hand-mixed, a much gentler mixing process than used for Simulant I-a.



#### Measured Unconfined Compressive Strength, kg/cm2

Figure E.13. Measured Unconfined Compressive Strength, Measured at a 24-hour Curing Time, of Simulant I-b, Consisting of 27 wt% Kaolin Clay, 33 wt% Plaster of Paris, and 40 wt% Water Versus Volume of Simulants Made in 500-mL Plastic and 500-mL Stainless Steel Containers



Figure E.14. Measured and Estimated Shear Strength of Simulant I-b Consisting of 27 wt% Kaolin Clay, 33 wt% Plaster of Paris, and 40 wt% of Water Versus Volumes of Simulants Made in 500-mL Plastic and 500-mL Stainless Steel Containers, Measured at a 24-Hour Curing Time

Figures E.13 and E.14 show that UCS and shear strength of simulants in the plastic and stainless steel containers of about the same sizes are very similar, although the values for the simulants made in the stainless steel container are slightly smaller than those for the plastic containers. This suggests that the container materials may not be an important factor affecting the shear strength of the simulant.

Comparing the strength between Simulants I-a and I-b (see Figures E.11 through E.14) shows that Simulant I-a made with vigorous mixing is more than 50% stronger than Simulant I-b made with minimum mixing, revealing the effect of the degree of simulant mixing on the simulant's UCS and shear strength. Thus, the mixing degree alone may produce up to a  $\pm 30\%$  or 85% variation in both UCS and shear strength.

#### E.3.1.3 Simulant II

The measured UCS of Simulant II-a (44 wt% plaster of Paris and 56 wt% water) made in the 250-mL, 500-mL, and 5-gallon plastic and 500-mL stainless steel containers at 1 day of simulant curing time are shown in Figure E.15.



Measured Unconfined Compressive Strength, kg/cm2

Figure E.15. Comparison of Measured Unconfined Compressive Strength, Measured at a 24-hour Curing Time, of Simulant II-a, Consisting of 44 wt% Plaster of Paris and 56 wt% Water Made in 250-mL, 500-mL, and 5-Gallon Plastic and 500-mL Stainless Steel Containers

Figure E.15 indicates that the UCS of the 19-L simulant made in the 5-gallon plastic container was the highest, while the 0.5-L simulant in the 500-mL plastic container has the smallest UCS value. The 0.5-L simulant in the 500-mL stainless steel container has a UCS value between those of the 0.2-L and 0.5-L simulants made in the 250-mL and 500-mL plastic containers.

Measured and estimated shear strengths for these four containers of Simulant II-a are presented in Figure E.16. As noted previously, the estimated shear strength was obtained with the measured UCS and the use of Equation E.1.



#### Figure E.16. Measured and Estimated Shear Strength of Simulant II-a, Consisting of 44 wt% Plaster of Paris and 56 wt% of Water Versus Volumes of Simulants Made in 250-mL, 500-mL, and 5-gallon Plastic Containers and a 500-mL Stainless Steel Container, Measured at a 24-Hour Curing Time

The Geonor hand-held vane tester used for these simulants has a vane of 16-mm diameter and a height of 32-mm. It has an accuracy of approximately  $\pm 10\%$ , according to its manufacturer. It was inserted into the simulant, before simulant curing, to a depth of approximately 20 mm for Simulant II-a and 16 mm for Simulant II- b and II-c, measured from the simulant surface. The vane tester measured the shear strength of the simulant in the depth from 16~20-mm to 48~52-mm after 1 day of curing time. The UCS was measured by pushing the pocket penetrometer into the simulant to depth of about 6-mm from the simulant surface.

Figure E.16 indicates that the 0.2-L simulant made in the 250-mL plastic container has the largest shear strength, while the 0.5-L simulant made in the 500-mL stainless steel container has the smallest shear strength. The 0.5-L and 19-L simulants made in the 500-mL and 5-gallon plastic containers have basically the same shear strength. The measured and estimated shear strength values matched well for the simulants in the 500-mL stainless steel and 5-gallon plastic containers, but for the other two simulants, the estimated shear strength values are less than the measured shear strength values.

The measured and estimated shear strength variations shown in Figures E.12 and E.16 imply that the simulant container sizes themselves may not be a major factor consistently causing different shear

strengths for the same simulants of different sizes. This is consistent with Figure E.10 discussed previously.

Another test was conducted to further evaluate a possible effect of simulant size on shear strength, as well as how well the simulant was mixed as a large batch. This test with Simulant II-b used 500-mL stainless steel and 6-gallon carbon steel containers. After the simulant was mixed gently, mostly by hand in a large mixing bucket, the top layer of the mixed simulant was first poured in the 500-mL stainless steel Container #A. Then, the bulk of the simulant in the mixing bucket was poured into a 6-gallon carbon steel container, and the simulant remaining near the bottom of the mixing bucket was poured into the 500-mL stainless steel Container #B.

Figure E.17 shows measured UCS values of Simulant II-b in these three steel containers. Figure E.18 shows the measured shear strength by the Geonor H-60 hand-held vane tester and the estimated shear strength converted from UCS with Equation E.1.



Figure E.17. Comparison of Measured Unconfined Compressive Strength, Measured in 500-mL Stainless Steel and 6-Gallon Carbon Steel Containers at a 24-hour Curing Time, of Simulant II-b, Consisting of 44 wt% Plaster of Paris and 56 wt% Water Versus Volumes of Simulants



**Figure E.18.** Measured and Estimated Shear Strength of Simulant II-b Consisting of 44 wt% Plaster of Paris and 56 wt% of Water Versus Volumes of Simulants, Measured in 500-mL Stainless Steel and 6-Gallon Carbon Steel Containers at a 24-Hour Curing Time

Figures E.17 and E.18 indicate that the UCS and shear strength of 20-L Simulant II-b in the 6-gallon carbon steel container are between the values of 0.5-L Simulant II-a in the 500-mL Stainless Steel Containers A and B. This implies that potential non-uniformity of the simulant within the mixing bucket may cause a larger variation of shear strength than the possible effect of simulant size on shear strength.

Comparing Figures E.15 and E.17 with Figures E.16 and E.18 indicates that Simulant II-a was slightly stronger than Simulant II-b, thus revealing the possible effect of the degree of simulant mixing on UCS and shear strength.

Figures E.17 and E.18 show that the UCS variation of Simulant II-b among the three simulant containers is  $\pm$  8% from the average value for these three simulants. The shear strength variation among these three simulants is  $\pm$  12% from the average value. Thus, it may be expected that with the minimum mixing of simulant in a 6-gallon container, the maximum simulant strength variation may be expected to be approximately 27%.

Simulant II-c was used to examine the possible effect of simulant container materials by using the 500-mL plastic and 500-mL stainless steel containers. Figures E.19 and E-20 show the measured UCS and measured/estimated shear strength of this simulant in these two containers.



Figure E.19. Comparison of Measured Unconfined Compressive Strength, Measured at a 24-hour Curing Time, of Simulant II-c, Consisting of 44 wt% Plaster of Paris and 56 wt% Water Versus Volumes of Simulants Made in 500-mL Plastic and 500-mL Stainless Steel Containers



**Figure E.20.** Measured and Estimated Shear Strength, Measured at a 24-Hour Curing Time, of Simulant II-c, Consisting of 44 wt% Plaster of Paris and 56 wt% of Water Versus Volumes of Simulants Made in 500-mL Plastic and 500-mL Stainless Steel Containers

Figures E.19 and E.20 reveal that the UCS and shear strength in the plastic and stainless steel containers are quite similar to each other, although the values in the stainless steel container are slightly smaller than those in the plastic container. Simulant II-c in these two containers was prepared with the minimum agitation/mixing. As compared to Simulants II-a and II-b, Simulant II-c has the smallest shear strength, again reflecting the difference in degree of simulant mixing.

Because these two 500-mL containers are small, the simulants in there were expected to be similar to other and quite uniform. Thus, Figure E.20 indicates that an error of the measured UCS to predict the shear strength with Equation E.1 is  $23 \sim 27\%$ .

Simulant II-d was made with vigorous mixing with the electric mixer in a large mixing bucket. This simulant in all 250-mL plastic, 500-mL plastic, 500-mL stainless steel, and 5-gallon plastic containers exceeded the measurable upper limits of UCS as measured with the pocket penetrometer, and shear strength measured with the Geonor H-60 hand-held vane tester. The upper limit of the penetrometer is approximately 190 kPa, while the upper limit of the vane tester is 260 kPa. Thus, all the Simulant II-d made in these different containers exceeded 260 kPa.

Note that 260 kPa of shear strength is 75% greater than 148 kPa of shear strength of Simulant II-a in the 500-mL stainless steel container, and 62% greater than 160 kPa of Simulant II-b in the 500-mL stainless steel container. This indicates that the variability introduced by simulant mixing can exceed  $60\sim70\%$ .

Figures E.11 through E.20 indicate that although the UCSs and shear strengths of these homogeneous simulants in metal containers were generally slightly less than those in plastic containers, it appears that effects of simulant size and container materials on shear strength were not significant. The variation of these values among different simulant sizes and materials is probably due more to the different degree of mixing to make the simulants and possibly some non-uniformity of simulants within the mixing buckets.

Because these tests indicate that the degree and manner of mixing during the simulant preparation can significantly affect the simulant strength, it is important to uniformly mix the simulants in a very consistent manner to obtain the specific shear strength.

#### E.3.2 Effect of Simulant Curing Time on Shear Strength

#### E.3.2.1 Simulant I

Simulant I-a (27 wt% kaolin clay, 33 wt% plaster of Paris, and 40 wt% water), which was made with vigorous mixing by the electric drill/mixer, as stated above, was used for these tests. The UCS and shear strength were measured with the Geotest E-280 pocket penetrometer and the Haake M5 rheometer over 7 days to evaluate the effect of the simulant curing time on the shear strength of the simulant. Figure E.21 shows the measured UCS for Simulant I-a in 250-mL, 500-mL, and 5-gallon plastic containers, and 500-mL stainless steel containers. Note that data at the 24-hour curing time are the same as those shown in Figure E.11. To measure UCS and shear strength of the simulant in the 5-gallon container over 7 days, measuring points on the simulant surface were fairly evenly spaced, so measurements were not concentrated in any specific segment of the simulant surface area. Thus, the UCS and shear strength

values over 7 days not only show the variations over 7 days, but also represent spatial distributions of shear strength.



Figure E.21. Measured Unconfined Compressive Strength, Over a 7-Day Curing Time, of Simulant I-a, Consisting of 27 wt% Kaolin Clay, 33 wt% Plaster of Paris, and 40 wt% Water Made in 250-mL, 500-mL, and 5-Gallon Plastic Containers, as well as a 500-mL Stainless Steel Container

The measured UCS of 16-L simulant made in the 5-gallon plastic container gradually reduced from 1.80 to 1.59 kg/cm<sup>2</sup> (12% reduction) over a 7-day period.

The shear strength measured by the Haake M5 for the same simulant over the same 7-day period is shown in Figure E.18.



Figure E.22. Measured Unconfined Compressive Strength, Over a 7-Day Curing Time, of Simulant I-a, Consisting of 27 wt% Kaolin Clay, 33 wt% Plaster of Paris, and 40 wt% Water Made in 250-mL, 500-mL, and 5-Gallon Plastic Containers, as well as a 500-mL Stainless Steel Container

Figure E.22 indicates that the measured shear strength of the 16-L simulant made in the 5-gallon plastic container varied from 63 kPa to 73 kPa over 7 days. Its value was the greatest at the second day after the simulant was made, but over 7 days, it changed little (13% variation). The measured shear strength and estimated shear strength (converted from the measured UCS with Equation E.1) over the 7-day curing period are shown in Figure E.23 for the 16-L simulant.



**Figure E.23.** Measured and Estimated Shear Strength, Over a 7-Day Curing Time, of Simulant I-a, Consisting of 27 wt% Kaolin Clay, 33 wt% Plaster of Paris, and 40 wt% Water Made in the 5-Gallon Plastic Container

Figure E.23 reveals that the simulant of the kaolin-plaster-water mixture attained most of its final shear strength in the first 1 to 2 days. Figures E.22 and E.23 also indicate that the lateral distributions of UCS and shear strength are reasonably uniform because these values also reflect their distributions in the lateral direction (near the simulant surface).

On the 8<sup>th</sup> day, the 16-L simulant (Simulant I-a) in the 5-gallon plastic container was vertically cut in half (because a regular saw was used to cut the simulant, it could not be cut very cleanly). At various points along the vertical axis, as well as on the top and bottom surfaces, UCS measurements were made with the pocket penetrometer. The penetrometer marks are visible in Figure E.24.



**Figure E.24.** Vertically-Cut Half of Simulant I-a, Consisting of 27 wt% Kaolin Clay, 33 wt% Plaster of Paris, and 40 wt% Water Made in the 5-Gallon Plastic Container, Showing Marks of UCS Measurements with the Pocket Penetrometer (Note: The top of the figure is the top surface of the simulant.) The Top and Bottom Diameters of the Simulant Are 27.5 and 25.5 cm, Respectively, and Its Height Is 26.5 cm.

The measured vertical distributions of the measured UCS and shear strength estimated with Equation E.1 are shown in Figure E.25 and E.26, respectively. Note that UCS values of the top and bottom measuring points are on the top and bottom surfaces of Simulant I-a.



Figure E.25. Vertical Distribution of Unconfined Compressive Strength of Simulant I-a, Consisting of 27 wt% Kaolin Clay, 33 wt% Plaster of Paris, and 40 wt% Water Made in the 5-Gallon Plastic Container Measured on 8<sup>th</sup> Day. (UCS values of the top and bottom measuring points are on the top and bottom surfaces of Simulant I-a.)

#### Estimated Shear Strength, kPa



Figure E.26. Vertical Distribution of Estimated Shear Strength of Simulant I-a, Consisting of 27 wt% Kaolin Clay, 33 wt% Plaster of Paris, and 40 wt% Water Made in the 5-Gallon Plastic Container Measured on 8<sup>th</sup> Day. (Shear Strength values of the top and bottom measuring points are on the top and bottom surfaces of Simulant I-a.)

The UCS and shear strength vertical distributions have some non-uniformity, with the largest values at the top and bottom surfaces of the simulant. As shown in Figure E.26, the estimated shear strength varies from 43 to 71 kPa, with the average value of 56 kPa and a variation of  $\pm 25\%$ . If only the simulant interior is considered, the variations of UCS and estimated shear strength are less than  $\pm 20\%$ .

#### E.3.2.2 Simulant II

The maximum shear strength measurable using the Haake M5 rheometer with a 6-mm diameter and 6-mm-high vane (the smallest vane available) is 108 kPa. Because the shear strength of Simulant II exceeds that upper limit, the Geonor H-60 hand-held vane tester was used to measure the shear strength of Simulant II over time. For these measurements, 16-mm diameter, 32-mm-high shear vanes were inserted into the simulants before the simulants began to solidify, as shown in Figure E.27. Figure E.27 shows seven vanes embedded in Simulant II in the 5-gallon plastic container to enable the measurement of shear strength at seven different times. The UCS was measured by the Geotest E-280 pocket penetrometer in the vicinity of the shear strength measurement point, but not close enough to interfere with the shear strength measurement. Figure E.27 also shows that these measurement locations are spatially spread to represent lateral distributions of the UCS and shear strength values, as discussed in E.3.2.1.



Figure E.27. Seven Shear Vanes Embedded in Simulant II in the 5-Gallon Plastic Container

Simulant II-b was used to examine the shear strength change over 7 days. It consisted of 44 wt% plaster of Paris and 56 wt% water and was made with minimal use of the electric drill/mixer for simulant mixing, as described in Section E.3.1.3. The simulant was used in 500-mL stainless steel containers #A and #B and a 6-gallon carbon steel container. As stated in Section E.3.1.3, the top layer of the mixed simulant in the large mixing bucket was first poured into 500-mL stainless steel Container #A. Then, the bulk of the simulant in the mixing bucket was poured into the 6-gallon carbon steel container, and the simulant remaining near the bottom of the mixing bucket was poured into 500-mL stainless steel Container, and the Container #B.

The measured UCS over the 7-day curing period is shown in Figure E.28. Note that the UCS values shown in this figure at a 1-day curing time are the same as those shown in Figure E.17. Figure E.28 indicates that the plaster-water mixture reached its final UCS value after 1 day of curing for the simulants in all three containers. Figure E.29 shows the measured shear strength for the same three containers, together with the estimated shear strength converted from the measured UCS and the use of Equation E.1. The shear strength values shown in Figure E.29 at the first-day curing time are the same as those shown in Figure E.18.

#### Unconfined Compressive Strength, kg/cm2



**Figure E.28.** Measured Unconfined Compressive Strength, Over a 7-Day Curing Time, of Simulant II-b, Consisting of 44 wt% Plaster of Paris and 56 wt% Water Made in the 500-mL Stainless Steel Containers #A and #B, and the 6-Gallon Carbon Steel Container



**Figure E.29.** Measured Shear Strength, Over a 7-Day Curing Time, of Simulant II-b, Consisting of 44 wt% Plaster of Paris and 56 wt% Water Made in the 500-mL Stainless Steel Containers #A and #B, and the 6-Gallon Carbon Steel Container

The shear strength reduced from 192 kPa at 0.5 day to 158 kPa on the third day and then progressed back up to 168 kPa on the seventh, having an average value of 170 kPa over 7 days for the 22-L simulant in the 6-gallon carbon steel container.

As discussed in Section E.3.1.3, the shear strength of the 22-L simulant was between those of the simulants in 500-mL stainless steel Containers #A and #B, but is very close to shear strength of the simulant in stainless steel Container #B. The estimated shear strength of the 22-L simulant is very close to the measured shear strength of the simulant in the stainless steel Container #A.

These tests again indicate that the simulant container size itself may not be a major factor in the sheer strength of the simulant. However, the degree of mixing affects the shear strength of the simulant. Thus, it appears that as the simulant size changes, there is a potential that the mixing agitation will change, affecting the shear strength.

To further examine the simulant container materials on shear strength over time, Simulant II-a in the 5-gallon plastic container was used to measure the UCS for 10 days. Note that UCS and shear strength after 1 day are also shown in Figure E.15 and E.16. As shown in Figure E.30, the measured UCS decreased for the first 3 days and became steady for the next 7 days. The estimated shear strength with the measured UCS and Equation E.1 are shown in Figure E.31, which also includes the measured shear strength on the first day. This figure also includes the measured shear strength of Simulant II-b in the 6-gallon plastic container (see Figure E.29) for comparison.



#### Unconfined Compressive Strength, kg/cm2

**Figure E.30.** Measured Unconfined Compressive Strength, Over a 10-Day Curing Time, of Simulant II-a, Consisting of 44 wt% Plaster of Paris and 56 wt% Water Made in the 5-gallon Plastic Container



**Figure E.31.** Shear Strength of Simulants II-a and II-b, Consisting of 44 wt% Plaster of Paris and 56 wt% Water Made in 5-gallon Plastic and 6-gallon Carbon Steel Containers, up to a 10-Day Curing Time

Figure E.31 indicates that the shear strengths of simulants are similar between the simulants made in the 6-gallon carbon steel container and the 5-gallon plastic container. Thus, the container materials do not appear to be a major factor affecting shear strength.

Although there are some variations, the simulants reached the bulk of the steady-state values of UCS and shear strength on the first day and reached their steady-state values during the first 3 days. This figure also indicates that the lateral distribution of the shear strength is quite uniform because each measurement at a different time also was measured at a different lateral location (see Figure E.27).

The 19-L simulant (Simulant II-a) in the 5-gallon plastic container was vertically cut in half by a regular saw after 10-day measurements of the UCS, as shown in Figure E.32. UCS was measured with the pocket penetrometer on the 11<sup>th</sup> day; penetrometer marks are visible in the figure. The UCS values on the top and bottom surfaces of Simulant II-a were also measured at that time.



**Figure E.32.** Vertically-Cut Half of Simulant II-a, Consisting of 44 wt% Plaster of Paris and 56 wt% Water Made in the 5-Gallon Plastic Container, Showing Marks of UCS Measurements with the Pocket Penetrometer (Note: The top of the figure is the top surface of the simulant.) The Top and Bottom Diameters of the Simulant Are 28.0 and 25.5 cm, Respectively, and Its Height Is 32.0 cm.

The vertical distributions of the measured UCS and estimated shear strength using Equation E.1 are shown in Figure E.33 and E.34, respectively.



Figure E.33. Vertical Distribution of Unconfined Compressive Strength of Simulant II-a, Consisting of 44 wt% Plaster of Paris and 56 wt% Water Made in the 5-Gallon Plastic Container Measured on 11<sup>th</sup> Day. (UCS values of the top and bottom measuring points are on the top and bottom surfaces of Simulant II-a.)



Figure E.34. Vertical Distribution of Estimated Shear Strength of Simulant II-a, Consisting of 44 wt% Plaster of Paris and 56 wt% Water Made in the 5-Gallon Plastic Container Measured on 11<sup>th</sup> Day. (Shear strength values of the top and bottom measuring points are on the top and bottom surfaces of Simulant II-a.)

The UCS and estimated shear strength vertical distributions are fairly uniform, with the largest values at the top and bottom surfaces of the simulant. As shown in Figure E.34, the estimated shear strength varies from 128 to 154 kPa, with an average value of 139 kPa and a variation of  $\pm$  10%. Excluding the shear strength values of the top and bottom surfaces of the simulant, the vertical variation of the estimated shear strength is within  $\pm$  6%.

Figures E.31 and E.34 indicate that the distributions of shear strengths in both lateral and vertical directions are quite uniform for Simulant II.

#### E.4 Summary

These preliminary tests presented in Appendix E indicate that the simulant sizes and container materials have only minor effects on the unconfined compressive strength and shear strength values. These test data and their analyses indicate that simulant sizes and container materials are not a main cause of the shear strength variation, but the manner and degree of simulant mixing and some non-uniformity of the simulant within the mixing bucket may be more important factors in determining the shear strength of a simulant for the given simulant compositions and constituent concentrations. Thus, it is important to uniformly mix simulants in a very consistent way to obtain a specific shear strength.

Simulant II, consisting of 44 wt% of plaster of Paris and 56 wt% of water, has quite uniform distributions of the unconfined compressive strength and shear strength in both lateral and vertical directions. Simulant I, consisting of 27 wt% kaolin clay, 33 wt% of plaster of Paris, and 40 wt% of water, has also uniform distributions of the unconfined compressive strength and shear strength, but the vertical distribution was less uniform than it was for Simulant II.

It may be expected that with the minimum mixing of simulant in a 6-gallon container, the maximum UCS and shear strength variations may be expected to be approximately  $\pm 12\%$  or 27%. The mixing degree alone may produce up to  $\pm 30\%$  or 85% variations of the UCS and shear strength.

The simulants gained most of the final UCS and shear strength values after 1 day of curing and generally reached their final values in the first 3 days. The simulants tested here also have relatively steady values of unconfined compressive strength and shear strength over 7 and 10 days; thus, they have fairly long shelf-lives of 7 to 10 days to maintain the strengths measured after 1 day of curing.

The test data also imply that the error of conversion UCS measured with the pocket penetrometer to shear strength using Equation E.1 may be up to 27%.

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