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Strength Measurements of Archive K Basin Sludge Using a Soil Penetrometer

CH Delegard
AJ Schmidt
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December 2011

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Pacific Northwest National Laboratory
Richland, Washington 99352

Summary

The K East (KE) and the K West (KW) Basin fuel storage pools near the Columbia River at the U.S. Department of Energy (DOE) Hanford Site were used from the 1980s until 2004 for storage of a portion of the spent nuclear fuel from the Hanford N Reactor. Over this period, the spent fuel storage and packaging operations generated radioactive sludge in both basins. Transfer of sludge from the KE Basin to the KW Basin was completed in 2007. Sludge from both basins now resides in six large underwater engineered containers in the KW Basin.

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 the six engineered containers. The retrieved sludge will be hydraulically loaded into sludge transport and storage containers (STSCs) and transported to an interim storage facility in the Central Plateau. 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 (WIPP) in New Mexico.

During the period the STSCs are stored, the strength of K Basin sludge is expected to increase because of chemical reactions and intergrowth of sludge phase crystals whose rates increase with increasing temperature. Sludge strength also can increase by compaction and dewatering due to settling. Changes in sludge strength with time can impact the specialized equipment and the mechanical intensity of its operation when sludge is retrieved from STSCs for final sludge treatment and packaging.

Under current plans, water jets will be used to help mobilize K Basin sludge for retrieval from the STSCs after interim storage. It is important to determine whether water jets can mobilize and erode the stored K Basin sludge from the STSCs. Shear strength is known to be a key property to determine whether water jets can mobilize sludge from the STSCs.

Accordingly, the unconfined compressive strengths of archive K Basin sludge samples and sludge blends were measured using a pocket penetrometer modified for hot cell use. Based on known correlations, the unconfined compressive strength (UCS) values measured by the pocket penetrometer were converted to shear strengths. Using inventory logs, twenty-six sludge samples were identified and selected as potential candidates for sludge strength measurement. These samples had been stored in hot cells for varying numbers of years since last being disturbed. Valid UCS measurements could only be made for twelve samples with the remaining materials not being suitable for UCS measurements due to quantity, geometry, or texture limitations. Significantly, valid measurements were made for all seven of the key archive samples that have been maintained for future testing. The samples for which valid measurements were made were moist or water-immersed solids and at least ½-inch deep in their storage jars. Two of the samples were measured in quadruplicate, seven in triplicate, two in duplicate, and one had a single measurement. The UCS measurement reproducibility generally was 30%, relative, for these multiple determinations.

Ten of the twelve samples, including all of the key archive samples, were relatively weak, having UCS values between 0.26 and 0.49 kg_f/cm^2, with consistencies described as “soft”. The shear strengths, ranging from 11 kPa to 21 kPa, were determined based on prior published shear strength correlations to UCS for homogeneous materials. The consistencies according to the shear strength values were similarly described as “very soft” to “soft”.

Two of the twelve samples, KE Pit and KC-4 P250, were strong with UCS values of 2.2 and 2.6 kg_f/cm^2. These UCS values correspond to shear strengths of about 110 kPa and 140 kPa, respectively, based on an experimentally-determined correlation for heterogeneous materials (i.e., materials containing hard granules such as sand). The UCS values for both materials correspond to “very stiff” consistency

described as “readily indented by thumb nail”. The shear strength values for both materials correspond to “stiff” consistency described as “can be indented by thumb”. Both of these sludge samples are composites prepared from material collected from a number of locations on the KE Basin floor (KC-4 P250) and Weasel Pit (KE Pit). The KE Pit sample had been left undisturbed for 52 months before it was measured while the KC-4 P250 sample had been left undisturbed for 146 months before measurement. These are comparatively long times but are not markedly different than the 30 to 90 months that the ten weaker samples had been left undisturbed.

Although both KE Pit and, likely, KC-4 P250 have relatively high iron concentrations, attribution of their high strengths to this factor could not be made with confidence. This is because sludge samples KC-4 Whole, KC-4-2, and KE Floc Comp, all from the KE Basin floor and also having relatively high iron concentrations, were found to have low UCS and shear strengths. Therefore, a mechanistic reason could not be offered to explain the much greater strengths of the KE Pit and KC-4 P250 sludge materials compared with the other measured archive sludge materials. The observed UCS and shear strengths for these latter two sludges were greater than observed in any prior testing of K Basin sludge except for sludge that had been processed at 185°C under hydrothermal conditions. Note that the STP is no longer planning to use the 185°C hydrothermal process to treat the K Basin sludge but is evaluating warm water oxidation at~100°C and chemical treatment methods to react uranium metal present in K Basin sludge.

The KE Basin sludge samples that were measured represent composites of distinct sludge types collected prior to consolidation of the KE Basin sludge into underwater engineered containers (SCS-CON-240, -250, -260, and -230) in the KW Basin. Consolidation of these KE Basin sludge types into engineered containers has resulted in the mixing of KE Basin floor, pit, and some canister sludge. Thus, it is noted that this consolidation and mixing could affect the magnitude of strength changes with time.

Terms and Acronyms

CHPRC	CH2M Hill Plateau Remediation Company
DOE	U.S. Department of Energy
KE Basin	K-East Basin
kPa	kilopascal
KW Basin	K-West Basin
OIER	organic ion exchange resin
Pa	pascal
PNNL	Pacific Northwest National Laboratory
RPL	Radiochemical Processing Laboratory
STP	Sludge Treatment Project
STSC	sludge transport and storage container
TI	test instruction
UCS	unconfined compressive strength
WIPP	Waste Isolation Pilot Plant
XRD	X-ray diffraction or diffractometry

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1.0 Introduction

The K East (KE) and the K West (KW) Basin fuel storage pools near the Columbia River at the U.S. Department of Energy (DOE) Hanford Site were used from the 1980s until 2004 for storage of a portion of the spent nuclear fuel from the Hanford N Reactor. Over this period, the spent fuel storage and packaging operations generated radioactive sludge in both basins. Transfer of sludge from the KE Basin to the KW Basin was completed in 2007. Sludge from both basins now resides in six large underwater engineered containers in the KW Basin.

Under the Sludge Treatment Project (STP), K Basin sludge disposition will be managed in two phases (Honeyman and Rourk 2009). The first phase is to retrieve the sludge that currently resides in engineered containers in the KW Basin pool. The retrieved sludge will be hydraulically loaded into sludge transport and storage containers (STSCs) and transported to an interim storage facility in the Central Plateau where it will be stored under a water cover. 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 (WIPP) in New Mexico.

During the period the STSCs are stored, the strength of K Basin sludge is expected to increase because of chemical reactions and intergrowth of sludge phase crystals whose rates increase with increasing temperature. Sludge also can alter by compaction and dewatering due to settling. 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.

Under current plans, water jets will be used to help mobilize K Basin sludge for retrieval from the STSCs after interim storage. It is important to determine whether water jets can mobilize and erode the stored K Basin sludge from the STSCs.

1.1 Prior Sludge Strength Observations

Shear strength is one of the key properties to determine the energy of water jets needed to mobilize sludge from the STSCs (Onishi et al. 2010). Among other factors, shear strength is affected by its time at rest. Most prior sludge strength measurements were obtained for sludge that had been quiescent for several days to several weeks (Poloski et al. 2002). Shear strengths ranging from 1 to 8200 Pascals (Pa) have been reported for K Basin sludge samples predominantly collected from the KE floor, KE pits, and KE canisters from 1995 to 2002 (Poloski et al. 2002; Schmidt and Sexton 2009; Schmidt 2010). These measurements were mostly conducted for samples that had settled for up to 20 to 30 days.

To investigate the influence of settling time and long-term storage, a 28-month study was conducted from May 2002 to September 2004 with six KE Basin sludge samples (Delegard et al. 2005). These samples were kept under ~32 to 38°C hot cell conditions, immersed in liquid water and in a ~5 Rad/hour radiation field. Five of the six KE Basin sludge samples were readily remobilized at the end of the 28-month settling test. However, the sixth sample, 96-13, collected from a fuel storage canister in the KE Basin, was found to be self-cemented and showed considerable strength such that the glass graduated cylinder in which the settling test took place had to be broken to remove the 96-13 monolith. The 96-13 sample contained 82 wt% uranium (dry basis) and had previously dried-out during storage but, approximately 6 months before initiation of the long-term storage tests, was reconstituted by being rewetted and blended. The self-cementing behavior was tentatively linked to its high total uranium concentration (Hill 2010). Review of video records of handling the 96-13 sample after the settling test was used to estimate the material's strength. The heterogeneous cohesive sediment was judged to be comprised of a “paste” material with estimated shear strength of 3 to 5 kPa joining “chunks” with estimated shear strength ranging from 380 to 770 kilopascal (kPa). The combined bulk “paste” and “chunk” material shear strength was estimated to be 15 to 65 kPa based on assessments of written and video records (Wells et al. 2009).

In a separate study, various sludge samples were subjected to hydrothermal processing at 185°C for 10 to 72 hours (Delegard et al. 2007). The sludge from these tests agglomerated to attain shear strengths estimated to range from 9 kPa to 170 kPa (rounded from 174 kPa) based on unconfined compressive strengths (UCS) measured by a pocket penetrometer. A later refinement of the correlation between UCS values measured by pocket penetrometer and shear strength has re-estimated the highest strength to be 152 kPa (Onishi et al. 2011). To date, the 170 kPa value is the highest strength measured or estimated for any bulk K Basin sludge sample although this was for hydrothermally-treated material. Although the STP is no longer planning to use the 185°C hydrothermal process to treat the K Basin sludge, warm water oxidation (~100°C) and chemical treatment methods to react uranium metal present in K Basin sludge are being evaluated (Honeyman et al. 2011).

Shear strength measurements also have been performed on samples collected from sludge consolidated in the six engineered containers in the KW Basin, SCS-CON-210, -220, -230, -240, -250, and -260.[1] Most of the strengths, which were measured after several days to several weeks of settling, ranged from 80 to 800 Pa. The highest strength, 2300 Pa, was obtained from a settler sludge sample (SCS-CON-230) that had settled for 3 weeks.

During the anticipated multi-year storage period in STSCs, sludge may consolidate and agglomerate to produce material with higher strength that is more difficult-to-retrieve. Therefore, separate longer-term settling tests are currently underway to examine the strengths for consolidated KE sludge samples from all six engineered containers SCS-CON-210, -220, -230, -240, -250, and -260.

1.2 Sludge Strength Measurements for the Present Testing

Measurements of the UCS of 26 archive K Basin sludge samples and sludge blends were attempted using a pocket penetrometer modified for hot cell use. Based on known correlations, the UCS values measured by the pocket penetrometer were converted to shear strengths. The testing was performed by the Pacific Northwest National Laboratory (PNNL) for the CH2M Hill Plateau Remediation Company

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(1) Fiskum SK, JM Billing, SJ Bos, CA Burns, CD Carlson, DS Coffey, JV Crum, RC Daniel, CH Delegard, MK Edwards, OT Farmer, LR Greenwood, SA Jones, D Neiner, BM Oliver, KN Pool, AJ Schmidt, RW Shimskey, SI Sinkov, CZ Soderquist, CJ Thompson, ML Thomas, T Trang-Le, and MW Urie. 2011. *Characterization of K-Basin Containerized Sludge Samples Collected from Engineered Containers SCS-CON-240, 250, 260, and 220*. PNNL-19035 Rev. 1, Pacific Northwest National Laboratory, Richland, WA.

Fountain MS, SK Fiskum, DL Baldwin, SJ Bos, CA Burns, DS CD Carlson, DS Coffey, RC Daniel, CH Delegard, MK Edwards, LR Greenwood, D Neiner, BM Oliver, KN Pool, AJ Schmidt, RW Shimskey, SI Sinkov, LA Snow, CZ Soderquist, CJ Thompson, T Trang-Le, and MW Urie. 2011. *Characterization Data Package for Containerized Sludge Samples Collected from Engineered Container SCS-CON-210*. PNNL-20650, Rev. 1, Pacific Northwest National Laboratory, Richland, WA.

Shimskey RW, JM Billing, SJ Bos, CA Burns, CD Carlson, DS Coffey, RC Daniel, CH Delegard, MK Edwards, SK Fiskum, LR Greenwood, SA Jones, M Luna, D Neiner, BM Oliver, KN Pool, AJ Schmidt, SI Sinkov, LA Snow, CZ Soderquist, ML Thomas, CJ Thompson, T Trang-Le, and MW Urie. 2011. *Characterization Data Package for Containerized Sludge Samples Collected from Engineered Container SCS-CON-230*. PNNL-20470, Pacific Northwest National Laboratory, Richland, WA.

(CHPRC) STP under a Test Instruction (TI) approved for use by PNNL authorities and the CHPRC Buyer's Technical Representative (BTR).[(1)]

The sludge samples had been retained since their collection in the period 1995-2003 in glass jars with plastic screw caps. The samples were held in archive in the Radiochemical Processing Laboratory (RPL) High Level Radiochemistry Facility (HLRF) and the Shielded Analytical Laboratory (SAL). These samples largely had remained undisturbed for long periods in the intervening time. However, sub-sampling and the creation of sample composites for test purposes or to consolidate like materials also had occurred (Delegard et al. 2011).

Aging processes occurred in the 31°C to 38°C (average ~34°C; Delegard et al. 2005) hot cell storage. Imperfect cap closure allowed some samples to dry out and all samples to undergo air exposure. As a result, chemically reduced uranium compounds, such as uraninite, UO_2, present in the sludge converted to hexavalent uranium compounds such as metaschoepite, $UO_3 \cdot 2H_2O$, as observed in prior characterization studies (Delegard et al. 2011). At the same time, uranium metal that might have been present in the initial sludge samples had opportunity to react with liquid water or water vapor during storage to form uraninite. Because the samples were not further characterized in the current testing, the extents of these oxidation reactions are unknown. However, characterization by X-ray diffractometry (XRD) of nine sludge samples in 2007 showed that the uranium was present only as U(VI) phases even though various uraninites (UO_2, U_4O_9, and U_3O_7) were found in earlier initial characterizations for three of these samples (Delegard et al. 2011). Based on these prior observations, it is expected that any uranium in the archived sludge samples measured in the present sludge strength testing now exists as U(VI) phases.

Twenty-six sludge samples were either suggested in the TI as potential candidates for sludge strength measurement or were examined in the course of accessing the samples identified in the TI. These samples were selected based on the expectation that they might have sufficient quantity (>½ inch depth) that valid measurements could be made using the soil penetrometer. However, of these 26, eight were either dry powders/granules/cakes or had sample depths too shallow to obtain a valid measurement. One of the 26 (the 96-13 sample from the 28-month settling test) was a solid cylinder held only loosely in the jar and could not be secured sufficiently steady to allow a penetrometer measurement to be made. Measurements of another two samples were judged to be invalid because the probe penetration depth could not be seen due to sample turbidity or jar wall smearing. In one case, with a dry sample, the measurement was invalid because the penetration depth was inadequate (<¼ inch) even when the penetrometer was compressed to near its maximum capacity.

The remaining twelve samples were present in their storage jars as moist or water-immersed solids of sufficient depth for valid measurements and thus represented sludge conditions anticipated for the STSCs. Two of the samples were measured in quadruplicate, seven in triplicate, two in duplicate, and one had a single valid measurement.

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(1) Delegard CH. 2011. "Soil Penetrometer Measurements of Strength of Archive K Basin Sludge." 53451-TI35, Pacific Northwest National Laboratory, Richland, WA.

2.0 Experimental Methods and Materials

Sludge strength measurements were made using soil penetrometers in prior strength testing of hydrothermally processed K Basin sludge (Delegard et al. 2007). Strength measurements using soil penetrometers were implemented owing to their simpler application under hot cell conditions compared with shear vane or other strength measurement devices and the penetrometer's ability to measure the smaller sludge quantities used in the hydrothermal testing. The correlation of the unconfined compressive strength (UCS) measured by the soil penetrometer with shear strength has been examined (Holtz and Kovacs 1981 and in other publications; see Delegard et al. 2007). Explicit correlations of UCS measured by soil penetrometer and shear strength have since been performed by Onishi and colleagues (2011) based on simulants meant to emulate K Basin sludge. This work has demonstrated the validity of using soil penetrometers to estimate sludge shear strength.

The experimental bases for penetrometer use and the penetrometer used in measuring UCS and, by extension, the sludge shear strength are examined in Section 2.1. The candidate sludge materials for UCS and shear strength measurement are described in Section 2.2.

2.1 Penetrometer

The operation of the penetrometer is described in Section 2.1.1, the correlation of penetrometer measurement of UCS with shear strength is described in Section 2.1.2, and the penetrometer calibration is described in Section 2.1.3.

2.1.1 Penetrometer Operation

Unconfined compressive strength measurements were performed for selected archive sludge samples using a Geotest Instrument Corporation model E-280 pocket penetrometer (Figure 2.1). The E-280 design is modeled on the Soiltest CL-700A pocket penetrometer whose operation is described in Appendix A (Soiltest 1984).

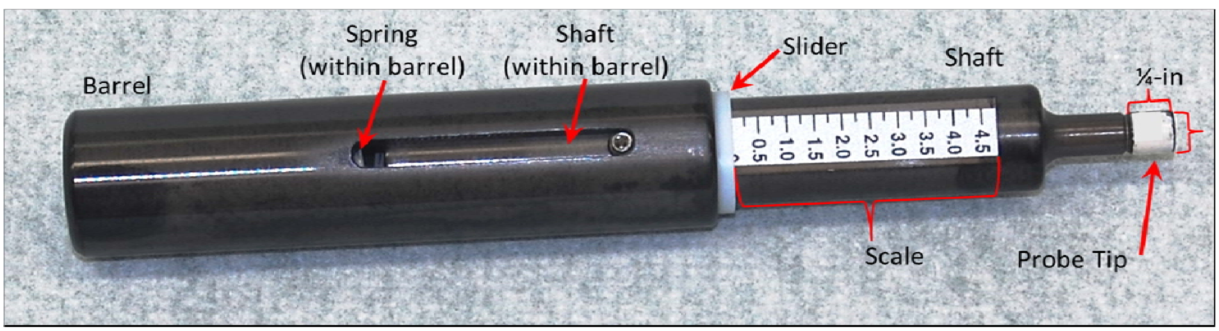


Figure 2.1. Geotest E-280 Pocket Penetrometer

The penetrometer is a hand-sized spring-loaded barrel ending with a ¼-inch (6.35 mm) diameter circular probe tip. To operate the penetrometer, the penetrometer probe, located on the right in Figure **2.1**, first is pressed perpendicularly into the surface to be tested until the ¼-inch diameter probe tip penetrates ¼-inch into the sludge or soil surface. The penetrometer then is removed from the sludge or soil. The maximum amount of resistance the sludge or soil offers to the downward pressure imposed by the compressed coil spring located in the penetrometer barrel shown in the left side of Figure 2.1 is registered by the white ring slider. The reading is taken from the edge closer to the probe tip (e.g., the slider position in Figure 2.1 indicates a reading of 0). The numerical gradations shown on the penetrometer scale are in units of kg_f/cm^2, where kg_f is kilograms of force. Units of kg_f/cm^2 are approximately equivalent to $tons/ft^2$.

However, the actual condition of any individual sludge sample may preclude the measurement or make it meaningless. For example, the sludge may be present as loose chunks within the container and thus not provide a stationary flat surface. The sludge also may be too thin to allow the probe to reach ¼-inch depth without interference with the container bottom. The necessary sample depth is about ½-inch (1.27 cm) based on UCS measurements of modeling clay. The sample diameter may permit multiple UCS measurements provided that the measurements are not too close to the vessel wall (less than two penetrometer diameters or ½ inch), in areas disturbed by prior measurements (e.g., at or near cracks), or, in any case, less than two penetrometer diameters (½ inch) from an earlier measurement.

Two modifications were made to the penetrometer for hot cell use. First, as seen in Figure 2.1, the penetrometer tip was painted white to the scored ¼-inch penetration depth. This was done to aid in viewing the entry of the tip into the sludge surface during operation in the limited visibility conditions afforded in the hot cell and with smeared sludge sample jars. A video camera within the hot cell and directed at the upper probed sludge surface was used to improve viewing during measurement. Second, the penetrometer barrel was lengthened with a grappling attachment to allow measurement of settled solids deep in jars and to facilitate handling the penetrometer in the hot cell by remote manipulators. The penetrometer modified for hot cell use is shown in Figure 2.2.

Figure 2.2. Geotest E-280 Pocket Penetrometer Modified for Hot Cell Use

The soil penetrometer measures the UCS of pliant solids such as soils. In most pocket penetrometer applications, UCS measurements are made to estimate the strengths of soils for civil engineering or construction activities with the goal to determine if the soil has sufficient strength to sustain vertical loads. The UCS measurements help indicate the stability of trenches to collapse (OSHA 1999). Penetrometer measurements also are made to determine soil compaction for agriculture. Vegetation has difficulty growing if soils are too highly compacted.

2.1.2 Measurement of Shear Strength Using the Pocket Penetrometer

The penetrometer measurement consists of pressing the ¼-inch diameter probe perpendicularly into the pliant soil or sludge material until it attains ¼-inch depth. The UCS is read directly from the penetrometer shaft by noting the displaced location of the sliding white ring. The UCS has been correlated with shear strength according to soil physics considerations (Holtz and Kovacs 1981) as shown in Equation 1:

$$\text{Shear strength, kPa} = \frac{\text{UCS, kg}_f\,/\,\text{cm}^2}{2} \times \frac{9.81\ \text{m}}{\text{sec}^2} \times \frac{10^4\,\text{cm}^2}{\text{m}^2} \times \frac{\text{kPa}}{(10^3\ \text{kg}/\text{m}\cdot\text{sec}^2)} \qquad \text{(Equation 1)}$$

$$\text{Shear strength, kPa} = 49.0 \times \text{UCS, kg}_f\,/\,\text{cm}^2$$

The correlation of UCS to shear strength also has been determined empirically based on comparative UCS and shear strength measurements made for homogeneous sludge strength simulants comprised of modeling clay and kaolin clay, plaster of Paris, kaolin with plaster of Paris, and amorphous alumina CP-5 mixed with water. This set is homogeneous because the constituents contain only fine particles and are free of coarse granules such as sand particles. The comparative measurements were made using a Geotest E-280 pocket penetrometer to determine UCS, a Geonor H-60 hand-held vane tester to measure shear strength manually, and the automated Haake M5 shear vane rheometer to determine shear strength (Onishi et al. 2011). Eleven different homogeneous mixtures, with shear strengths ranging from 3.5 to 95 kPa (and penetrometer UCS values ranging from about 0.04 to 1.8 kg_f/cm^2), were measured multiple times by the three instruments. However, shear strengths above about 127 kPa (to 172 kPa) exceeded the M5 range and could only be measured by the Geonor H-60 vane tester, comparable to UCS values ranging from 2.8 to 3.6 kg_f/cm^2.

The correspondence between the shear strength measurements made by the manual Geonor H-60 and automated Haake M5 devices was good. The manual Geonor H-60 readings were 1.06-times those found by the automated Haake M5 with a correlation coefficient, R^2, of 0.97. This excellent correspondence gives confidence in the validity of the Geonor readings in the >100 kPa shear strength range that was not attained by the Haake M5. The consonance of the Geonor H-60 and Haake M5 shear strength values for the range of rheology simulants tested was exploited to combine the shear strength data for the two instruments and arrive at Equation 2 for homogenous materials. This correspondence has a zero intercept, R^2 of 0.957, and is valid for UCS values from ~0.1 to 4.0 kg_f/cm^2, equivalent to 4 kPa to 170 kPa.

$$\text{Shear strength, kPa, homogeneous} = 42.8 \times \text{UCS, kg}_f / \text{cm}^2 \qquad \text{(Equation 2)}$$

The shear strength estimates based on penetrometer-measured UCS using Equations 1 and 2 thus differ by ~14%, relative. Given the ~4% to14% relative standard deviations observed by measuring the UCS by the Geotest E-280 pocket penetrometer in the various strength simulants (derived from Tables 4.6, 4.7, 4.9, 4.10. 4.11, and 4.12 of Onishi et al. 2011) and the even greater variabilities observed for multiple penetrometer measurements of genuine sludge in the present testing, both Equation 1 and Equation 2 are considered to be valid in converting penetrometer-measured UCS to shear strength for homogeneous (i.e., grit-free) sludge.

Correlations of UCS measured using a Geotest E-280 pocket penetrometer with shear strengths measured with a Geonor H-60 vane tester and a Haake M5 rheometer also have been performed for two sets of heterogeneous, sand-bearing, sludge simulants (Onishi et al. 2011). The weaker set of heterogeneous simulants, with shear strengths ranging from ~21 kPa to 46 kPa, contained kaolin clay, plaster of Paris, sand, and water. The stronger heterogeneous simulant set, with shear strengths ranging from ~160 kPa to 340 kPa, was made with plaster of Paris, sand, and water.

Again, the UCS values measured by the penetrometer were correlated with the shear strengths measured by the manual and automated shear vane devices (Onishi et al. 2011). The resulting heterogeneous simulant correlation shown in Equation 3 has an R^2 of 0.891 and a non-zero intercept. Because of the non-zero intercept, negative shear strengths are predicted at UCS values below ~0.7 kg_f/cm^2. This artifact and the fact that the UCS / shear strength correlation was developed with simulants having UCS values above about 1 to 1.3 kg_f/cm^2 means that Equation 3 is valid only for measurements above this range. It is seen that Equation 2 for the homogeneous strength simulants and Equation 3 for the heterogeneous simulants cross at about 1.6 kg_f/cm^2 where the predicted shear strength is about 70 kPa.

$$\text{Shear strength, kPa, heterogeneous} = -48.7 + 73.4 \times \text{UCS, kg}_f / \text{cm}^2 \qquad \text{(Equation 3)}$$

Because of the problem of unrealistic predicted shear strength at low UCS values for heterogeneous materials, mathematical fit of the joint homogeneous and heterogeneous simulant UCS and shear strength data was considered (Onishi et al. 2011). However, this approach was rejected because of the disparate natures and penetrometer responses of the two simulant material types.

Onishi and colleagues (2011) favor employing the correlation for heterogeneous materials (Equation 3) to actual sludge, which they judge to be heterogeneous. Accordingly, Equation 3 is recommended, where possible, in evaluating the actual sludge UCS values in the present testing. However, given the unrealistic shear strength values predicted by Equation 3 at low UCS, the homogeneous sludge relation given in Equation 2 is recommended in the present study for UCS values below the 1.6 kg_f/cm^2 crossing point for Equations 2 and 3. Comparison of the correlations for the homogeneous and heterogeneous simulants is shown in Figure 2.3 (taken from Figure F.7 of Onishi et al. 2011).

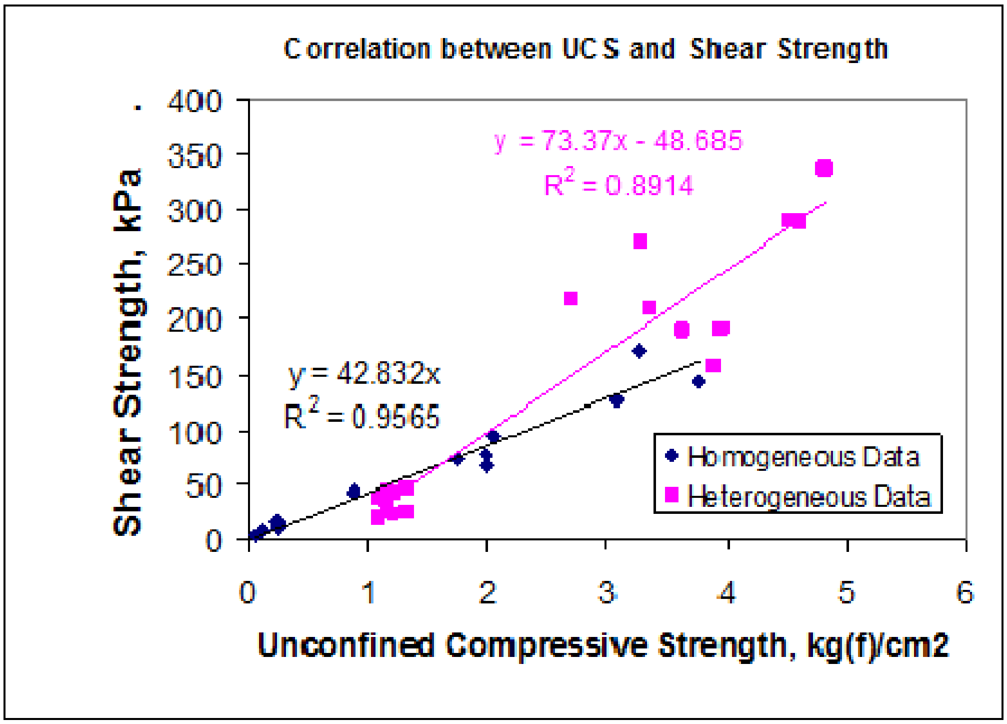


Figure 2.3. Correlations of Homogeneous and Heterogeneous Simulants Between UCS and Shear Strength

2.1.3 Penetrometer Calibration

In accordance with the TI and soil science practice (Bradford 1986), the penetrometer was calibrated by measurement of its readings as a function of its spring loading. The spring loading was done by pressing the penetrometer downward onto the platform of a calibrated analytical balance and noting the penetrometer and balance readings over the range of the penetrometer scale. The penetrometer UCS readings recorded at balance loadings ranging from 0 to 7000 grams (force) were compared with the expected penetrometer readings at those same force loadings to determine the penetrometer measurement bias.

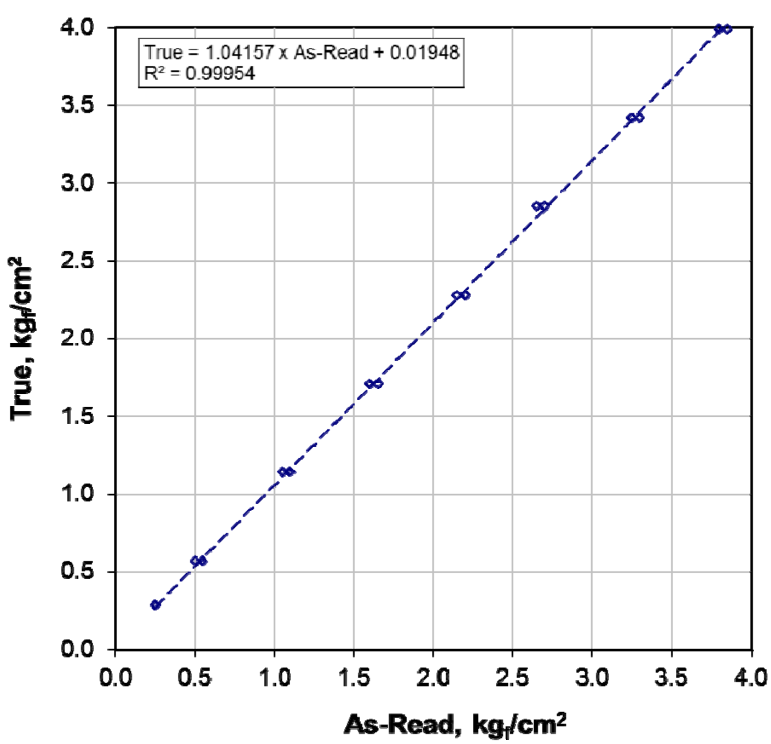
The expected UCS penetrometer reading response, in kg_f/cm^2, as a function of load, kg_f, is shown in Equation 4:

$$\text{UCS reading, } kg_f/cm^2 = 0.5695 \times \text{Load, } kg_f \qquad \text{(Equation 4)}$$

where kg_f is kg of force for the load the penetrometer places on the balance. The 0.5695 factor was derived based on evaluations provided in the Soiltest penetrometer description (Appendix A; see also Table 3.5 of Delegard et al. 2005). Thus, a 7.00 kg_f vertical load on the balance should compress the penetrometer by 0.5695 × 7.00 kg_f = 3.99 or ~4.0 kg_f/cm^2. The expected and observed penetrometer readings were compared to provide the penetrometer calibration so that as-read UCS values obtained from the penetrometer used in the sludge material measurements could be corrected, as determined from the calibration, to obtain the true UCS values. The calibration is expected to be consistent with related vendor literature on penetrometer precision, which is given as ±5%.

The penetrometer calibration was performed the day before measurements commenced in the hot cell. The calibration data and plot, Figure 2.1, show that the true penetrometer UCS values are about 4% greater than the as-read values, the intercept is small and positive (i.e., the spring has a slight pre-load), and the linearity is excellent ($R^2 > 0.999$). The conversion of the as-read penetrometer UCS values to the true values based on the calibration measurements is shown in Figure 2.4 and Equation 5.

$$\text{True UCS reading, } kg_f/cm^2 = \text{As-Read UCS reading, } kg_f \times 1.042 + 0.019 \qquad \text{(Equation 5)}$$



Unconfined Comp. Strength, kg_f/cm^2	
True	As-Read
0.00	0.00
0.00	0.00
0.00	0.00
0.28	0.25
0.28	0.25
0.28	0.25
0.57	0.50
0.57	0.55
0.57	0.55
1.14	1.10
1.14	1.05
1.14	1.10
1.71	1.60
1.71	1.60
1.71	1.65
2.28	2.15
2.28	2.20
2.28	2.20
2.85	2.70
2.85	2.65
2.85	2.70
3.42	3.30
3.42	3.25
3.42	3.25
3.99	3.80
3.99	3.80
3.99	3.85

Figure 2.4. Penetrometer Calibration

A 1-inch diameter adapter foot can be attached to the penetrometer to measure cohesive materials with shear strengths <1 kPa. With the adapter foot attached, the UCS value obtained directly from the penetrometer must be divided by 16 to obtain the true UCS. However, sludge UCS values below the lower meaningful range of the ¼-inch diameter probe (~0.25 kg_f/cm^2, or about 12 kPa shear strength) are not of concern in sludge handling as they indicate materials sufficiently weak to be readily slurried and moved. Therefore, the adaptor foot was not used in the present measurements.

2.2 Sludge Samples

The sludge samples examined in the present testing were collected in the period 1995-2003 and have been retained since collection by the RPL in glass jars with plastic screw caps. Most of them have been held in archive in the HLRF and a few in the SAL. These samples have remained undisturbed for long periods in the intervening time. However, sub-sampling and the creation of sample composites for test purposes or to consolidate like materials also has occurred (Delegard et al. 2011).

Aging processes taking place in the 31°C to 38°C (average ~34°C; Delegard et al. 2005) hot cell storage with imperfect cap closure allowed some samples to dry completely before water additions could be made during routine sludge maintenance activities. The imperfect closures also meant that all samples were exposed to air. As a result, chemically reduced uranium compounds present in the sludge such as uraninite (uranium dioxide; UO_2) were oxidized by atmospheric oxygen to convert to hexavalent uranium compounds such as metaschoepite, $UO_3 \cdot 2H_2O$ (Delegard et al. 2011). At the same time, any uranium metal that might have been present in the initial sludge samples had opportunity to react with liquid water or water vapor during storage to form uraninite. Because the samples were not further characterized in the current testing, the extents of the uranium metal and uraninite oxidation reactions are unknown. However, characterization by X-ray diffractometry (XRD) of nine sludge samples in 2007 showed that the uranium was present only as U(VI) phases even though various uraninites (UO_2, U_4O_9, and U_3O_7) were found in initial characterizations for three of these samples (Delegard et al. 2011). Based on these prior observations, it is expected that any uranium in the archived sludge samples measured for sludge strength was present in the form of U(VI) phases.

The 23 sludge samples identified for penetrometer measurement in the TI are listed in Table 2.1. The sludge candidates included seven key archive samples that have been collected and maintained for future process testing and 16 other samples identified in test planning. Once the sample archives were accessed in the HLRF and measurements begun, three additional samples were found as potentially being of interest and were examined. These additional three samples are listed at the bottom of Table 2.1.

Table 2.1. Archive Sludge Materials Examined for Penetrometer Measurement

Sludge Sample	Sludge Origin*	Container Vol., mL	Solids Vol., mL, / Depth, cm	Condition	Measured? Y/N; Meas. Validity & No.
Key Archive Samples					
KC-2/3 Comp	Canisters w/ highly and moderately damaged fuel	1000	250 / 3.0	Wet; 500 mL total vol.	Y; valid 3×
96-05	Single closed-bottom canister w/ high damaged fuel	500	120 / 3.0	Wet; 300 mL total vol.	Y; valid 3×
96-13 KE Comp A	Comp. single canisters w/ good to very poor fuel	500	160 / 4.0	Wet; 450 mL total vol.	Y; valid 3×
KE NLOP #2	KE North Loadout Pit top-to-bottom composite	500	250 / 6.0	Wet; 400 mL total vol.	Y; valid 3×
KC-4-2	Floor between open canisters w/ high damaged fuel	500	200 / 5.0	Wet; 380 mL total vol.	Y; valid 4×
FE-5 Comp 1	Weasel Pit and South Loadout Pit	500	250 / 6.0	Wet; 325 mL total vol.	Y; valid 2×
KE Floc Comp	Broad floor and canister composite	500	120 / 3.0	Wet; 250 mL total vol.	Y; valid 3×
Other Samples Identified in Planning					
96-13 Solids Grad	Sngl. can./poor fuel; 28-mo stl.; Delegard et al. 2005	250	140 / 5.5	Wet; 160 mL total vol.; chunk	N; too loose
96-13 SSOL	Single canister w/ poor fuel	500	100 / 2.5	Wet; 160 mL total vol.	Y; depth unknown 3×
KC-4 Whole	Floor between open canisters w/ high damaged fuel	500	120 / 3.0	Wet; 250 mL total vol.	Y; valid 3×
KE Pit (1995)	Weasel Pit composite	500	70 / 1.5	Wet; 120 mL total vol.	Y; valid 4×
KE NLOP #1	KE North Loadout Pit top-to-bottom composite.	500	150 / 3.5	Wet; 450 mL total vol.	Y; valid 2×
KE NLOP #3	KE North Loadout Pit top-to-bottom composite.	500	100 / 2.5	Wet; 300 mL total vol.	Y; valid 1×
FE-3 Comp 1	KE North Loadout Pit	500	120 / 3.0	Dry cracked	Y; depth unknown 2×
KC-4 P250	Floor btwn. open can. w/ high damaged fuel; >250 µm	125	60 / 3.5	Wet; 80 mL total vol.	Y; valid 3×
Test 4 Residual	Hydrothermal Test 4; Delegard et al. 2007	250	50 / 2.0	Dry cake	N; cracked
Test 5 Residue	Hydrothermal Test 5; Delegard et al. 2007	250	Small	Dry granules	N; rubble
96-23 Grad 2	KW canister w/ mod. dmgd. fuel; Makenas et al. 1998	500	50 / 0.5	Wet; 450 mL total vol.	N; too thin
KC-1 M500	Canister w/ highly damaged fuel; <500 µm	500	50 / 0.5	Wet; 100 mL total vol.	N; too thin
SNF Comp Stl Study	Floor/can sludge/fuel; 28-mo stl.; Delegard et al. 2005	250	50 / 2.0	Dry cake	Y; depth not met 3×
Test 3 Residue	Hydrothermal Test 3; Delegard et al. 2007	250	50 / 2.0	Dry cake	N; cracked
SNF + Comp Fines 60G	"SNF Comp"; floor/can sldg./fuel; Poloski et al. 2002	125	15 / 0.5	Dry powder	N; powder
KES 17B	Split from Weasel Pit KES-Q-17; Makenas et al. 1996	125	30 / 1.0	Dry powder	N; powder
Additional Samples Found During Measurement Activities					
SNF Floor 60L PG	Gas Gen III, Test 11; Schmidt et al. 2003	250	– / 0.6	Dry rubble cake; est. 10 pieces	Y; thin cake broke 1×
KE Container Comp Floc	Canister and Weasel Pit composite	500	~30 / 0.5	Dry thin layer	N; too thin
96-13 Settling Study	Single canister w/ poor fuel	250	50 / 2.0	Dry rubble	N; rubble

* See Delegard et al. (2011) and documents referenced therein for further descriptions of most samples and references cited in this table for others.

In the end, 26 sludge samples were examined as potential candidates for strength measurement. These samples were selected based on the expectation that they might have sufficient quantity (>½ inch or ~1 cm depth) so that valid measurements could be made using the soil penetrometer. However, of these 26, eight were either dry powders, rubble, or cake or had sample depths too shallow to attempt a valid measurement (Test 4 Residual, Test 5 Residue, 96-23 Grad 2, KC-1 M500, Test 3 Residue, SNF + Comp Fines 60G, KES 17B, KE Container Comp Floc, and 96-13 Settling Study). One of the 26 (96-13 Solids Grad from the 28-month settling study) was a solid cylinder held only loosely in the jar. The sludge cylinder for this sample could not be held sufficiently steady to allow a measurement to be made. Measurements of another two samples were judged to be invalid because the probe penetration depth could not be seen due to sample turbidity or jar wall smearing (96-13 SSOL and FE-3 Comp 1). Note also that sample 96-13 SSOL had been dry since at least 2007 (Delegard et al. 2011) but had had water added during routine sludge maintenance activities on 30 August 2011, about three weeks before the sludge strength measurements made 20-22 September 2011. In one case (SNF Comp Stl Study) the measurement was invalid because the sample was dry and the penetration depth was inadequate (<¼ inch) even when the penetrometer was compressed to near its maximum capacity. Finally, a dry ~¾-inch cake fragment (from SNF Floor 60L PG) about ¼-inch thick was too thin for a valid measurement and fractured when the measurement was attempted.

The remaining 12 samples were present in their storage jars as moist or water-immersed solids of sufficient depth for valid measurements. The solids in these 12 samples largely had been maintained in the wetted condition throughout storage. However, KC-4 Whole was prepared in 2007 as a composite of five samples, one of which (~14% by volume) had been dry before blending (Delegard et al. 2011). Sample KC-4-2 also had been dry and had been blended with water in 2007 (Delegard et al. 2011). Two of the samples were measured in quadruplicate, seven in triplicate, two in duplicate, and one had a single measurement.

The compositions of the sludges whose strengths were measured, whether the measurements ultimately were valid or invalid, are shown in Table 2.2. The composition of sample KC-4-2 is taken to be the same as that of KC-4 (Delegard et al. 2011). However, no composition data are available for sample KC-4 P250. This is the >250 µm particle size fraction for wet-sieved KC-4 sludge. It constituted only 16 wt% (dry basis) of the entire KC-4 sludge (Bredt et al. 1999). Therefore, the composition for KC-4 sludge given in Table 2.2 is only broadly indicative of the composition of the KC-4 P250 fraction. The KE NLOP sludge composition given in Table 2.2 represents the composition of the KE NLOP #1, #2, and #3 samples measured by the penetrometer as each of these three were split from the same mother composite KE NLOP material.

The SNF Floor 60L PG sample was the residue from Test 11 of the Gas Generation Series III experiments (Schmidt et al. 2003). This material was prepared from a composite sludge from the KE Basin floor (KC Floor Comp; sludge samples KC-4 and KC-5; Delegard et al. 2011) and crushed irradiated fuel particles. The sludge and fuel particle mixture had been reacted to 25% extinction of the contained uranium metal in gas generation testing and further complete reaction of the irradiated uranium metal to form UO_2 is presumed to have occurred in the intervening years. The elemental composition of the SNF Floor 60L PG sample is derived based on the weights of the KC Floor Comp sludge (as-settled wet basis) and crushed fuel used in the testing. Based on parallel measurements conducted in the Series III tests (Schmidt et al. 2003), the crushed fuel was assumed to be 93 wt% uranium metal in the present analysis and in prior tests (Delegard et al. 2005). In addition, and as postulated in prior tests (Delegard et al. 2005), the ^{154}Eu, ^{241}Am, ^{238}Pu, and ^{239,240}Pu radionuclide contributions from the irradiated fuel per mass of uranium are presumed to be the same as found in the accompanying sludge because of the low solubility of these elements and their capture in the preponderant low-solubility uranium phases. However, the uranium-based concentrations of ^{60}Co and ^{137}Cs in the sludge and fuel likely are not the same because of the high water solubility of cobalt and cesium and the confounding effects of basin water ion exchange purification and losses of ion exchange media to the basin sludge. Therefore no estimates of ^{60}Co and ^{137}Cs concentrations are provided. As noted, these same rationales were used to determine element and radionuclide concentrations in the SNF Comp Stl Study sample (Delegard et al. 2005). The SNF + Comp Fines 60G sample was prepared from the same source as the SNF Comp Stl Study material and its preparation is described by Poloski and colleagues (2002).

The dry-basis composition material balances shown in Table 2.2 are based on assignment of the elements to the compounds $Al(OH)_3$, $CaCO_3$, $Fe(OH)_3$, $MgCO_3$, Na_2O, SiO_2, and $UO_{2.63}\cdot H_2O$. The compounds $Al(OH)_3$, $CaCO_3$, and SiO_2 have been observed in genuine sludge. The compound $Fe(OH)_3$ generally is X-ray indifferent but represents the likely state of the wet iron hydroxide solids present in sludge even though Fe_2O_3 and FeOOH have been observed by XRD. The compound $MgCO_3$ is assigned based on its chemical similarity to $CaCO_3$; Mg is too scarce to have a phase identifiable by XRD. The hypothetical compound Na_2O represents the stoichiometry of sodium as oxide within more complex sludge oxide minerals. The hypothetical compound $UO_{2.63}\cdot H_2O$ represents a 50:50 (moles of U basis) mixture of $UO_{2.25}$ and $UO_3\cdot 2H_2O$, the uranium phases most frequently observed in sludge (see Schmidt and Delegard 2003). Substitution of fully oxidized $UO_3\cdot 2H_2O$ for the 50:50 $UO_{2.25}$ and $UO_3\cdot 2H_2O$ mix would increase the uranium phase fraction by ~8%, relative to the uranium fraction for the 50:50 mix, reflecting the difference in the $UO_{2.63}\cdot H_2O$ and $UO_3\cdot 2H_2O$ formula weights, and increase the total compound mass balance by the same amount.

The material balance shortfall for FE-3 is because of the presence of organic ion exchange resin, OIER. The OIER is comprised largely of organic polymers which do not dissolve in the acid digestion done for this sample. All other sample analyses except KE Pit (acid digest) are based on fusion digests. Fusion digestion solubilizes silicates whereas acid digestion does not. For KE Pit, the acid-insoluble residue was assumed to be SiO_2 (see footnotes to Table 3 of Delegard et al. 2011).

Table 2.2. Chemical and Radiochemical Compositions of Penetrometer-Measured Archive Sludges

Sludge	96-05	96-13 SSOL	96-13 KE Comp A	FE-3	FE-5	KC-2/3	KC-4 Whole	KE Floc Comp	KE NLOP	KE Pit	SNF Comp Stl Study	SNF Floor 60L PG
Dry Basis												
Element	**Concentration, Wt%**											
Al	1.32	1.45	2.08	0.987	2.66	5.16	6.82	7.70	3.93	3.34	4.62	6.83
Ca	0.0779	0.0698	0.0751	0.820	1.2	0.134	1.04	0.945	0.937	1.21	0.234	0.413
Fe	0.698	0.281	0.880	3.37	30.6	1.84	24.3	24.2	6.83	36.4	5.76	11.3
Mg	0.221	0.19	0.194	0.076	0.146	0.0462	0.33	0.230	0.122	0.194	0.0800	0.143
Na	0.0416	0.043	0.0395	0.025	BDL	0.24	0.36	0.365	BDL	0.0732	0.177	0.207
Si	NR	NR	NR	0.154	0.330	0.752	4.91	3.57	36.3	8.00	1.61	3.04
U	58.5	74.0	52.1	1.72	5.32	59.0	16.6	10.3	2.51	7.99	60.2	44.3
Compound	79.4	97.9	73.9	13.1	77.1	94.8	101.7	92.6	108.0	110.3	103.8	107.3
Radionuc.	**Concentration, µCi/g**											
^{60}Co	0.892	BDL	1.27	0.339	0.875	0.441	1.08	1.02	0.280	1.59	See text	See text
^{137}Cs	1140	648	748	18.8	170	860	1680	783	34.6	412	See text	See text
^{154}Eu	18.6	9.12	11.3	0.425	0.985	8.14	2.6	1.68	0.542	2.41	~9.1	~7.3
^{238}Pu	16.2	BDL	36.6	0.918	2.06	16.2	4.91	3.22	0.280	1.37	~17	~13
$^{239,240}Pu$	153	110	197	5.96	13.1	114	39.2	23.9	9.00	19.4	~128	~101
^{241}Am	133	72.0	90.3	4.92	10.4	90.5	29.2	18.9	7.82	14.6	~104	~84
As-Received or Previously-Measured Settled Sludge Basis												
El. / H_2O	**Concentration, Wt%**											
Al	0.706	NR	1.22	0.472	1.56	3.04	3.83	2.53	0.562	2.03	NR	NR
Ca	0.0417	NR	0.0439	0.392	0.704	0.0791	0.584	0.310	0.134	0.737	NR	NR
Fe	0.373	NR	0.515	1.61	18.0	1.09	13.7	7.92	0.977	22.1	NR	NR
Mg	0.118	NR	0.114	0.036	0.0857	0.0273	0.185	0.0755	0.0174	0.118	NR	NR
Na	0.0223	NR	0.0231	0.0121	BDL	0.142	0.202	0.120	BDL	0.0445	NR	NR
Si	NR	NR	NR	0.0734	0.194	0.444	2.76	1.17	5.19	4.86	NR	NR
U	31.3	NR	30.5	0.820	3.12	34.8	9.33	3.37	0.359	4.85	NR	NR
H_2O	46.5	NR	41.5	52.2	41.3	41.0	43.8	67.2	85.7	39.3	NR	NR
Radionuc.	**Concentration, µCi/g**											
^{60}Co	0.477	NR	0.743	0.162	0.514	0.260	0.607	0.334	0.0400	0.962	NR	NR
^{137}Cs	610	NR	438	8.96	100	507	944	257	4.95	250	NR	NR
^{154}Eu	9.95	NR	6.59	0.203	0.578	4.80	1.46	0.552	0.0775	1.46	NR	NR
^{238}Pu	8.67	NR	21.4	0.439	1.21	9.56	2.76	1.06	0.0400	0.832	NR	NR
$^{239,240}Pu$	81.9	NR	115	2.85	7.69	67.3	22.0	7.84	1.29	11.8	NR	NR
^{241}Am	71.2	NR	52.9	2.35	6.10	53.4	16.4	6.20	1.12	8.89	NR	NR
Reference	1	1	1	2	1	1	1	1	1	1	3	4

BDL – below detection limit. NR – not reported. References: 1 – Delegard et al. 2011; 2 – Bryan et al. 2004; 3 – Delegard et al. 2005; 4 – Schmidt et al. 2003

3.0 Results and Discussion

The uncorrected and calibration-corrected UCS values observed in the sludge penetrometer measurements are shown in Table 3.1 with the approximate settling times since their prior thorough mixings (i.e., disregarding incidental jostling during inventory and routine hot cell activities). Views of the sample KE Pit measurement are shown in Figure 3.1 and of KC-4 P250 measurement in Figure 3.2.

Table 3.1. Archive Sludge Material Penetrometer Measurement Results

Sludge Sample	Measurement Validity	Solids Depth, cm	Condition / Settling Time – Reference*	Penetrometer Reading, UCS, kg_f/cm^2 Uncorrected / Corrected			
Key Archive Samples							
KC-2/3 Comp	valid	3.0	Wet/ 30 mo. – 1	0.50/0.54	0.15/0.18	0.10/0.12	
96-05	valid	3.0	Wet/ 52 mo. – 2	0.30/0.33	0.25/0.28	0.15/0.18	
96-13 KE Comp A	valid	4.0	Wet/ 52 mo. – 2	0.50/0.54	0.15/0.18	0.25/0.28	
KE NLOP #2	valid	6.0	Wet/ 30 mo. – 1	0.25/0.28	0.25/0.28	0.50/0.54	
KC-4-2	valid	5.0	Wet/ 52 mo. – 2	0.35/0.38	0.50/0.54	0.55/0.59	0.40/0.44
FE-5 Comp 1	valid	6.0	Wet/ 42 mo. – 3	0.25/0.28	0.25/0.28		
KE Floc Comp	valid	3.0	Wet/ 30 mo. – 1	0.30.0.33	0.25.0.28	0.30/0.33	
Other Samples Identified in Planning							
96-13 SSOL	unknown depth	2.5	Wet/ 179 mo. – 4	1.70/1.79	1.00/1.06	0.75/0.80	
KC-4 Whole	valid	3.0	Wet/ 30 mo. – 1	0.50/0.54	0.30/0.33	0.40/0.44	
KE Pit (1995)	valid	1.5	Wet/ 52 mo. – 2	1.90/2.00	2.30/2.42	1.65/1.74	2.40/2.52
KE NLOP #1	valid	3.5	Wet/ 52 mo. – 2	0.25/0.28	0.25/0.28		
KE NLOP #3	valid	2.5	Wet/ 90 mo. – 2, 5	0.25/0.28			
FE-3 Comp 1	unknown depth	3.0	Dry cracked/ 134 mo. – 6	1.20/1.27	1.00/1.06		
KC-4 P250	valid	3.5	Wet/ 146 mo. – 7	3.00/3.14	1.60/1.69	2.85/2.99	
SNF Comp Stl Study	depth not met	2.0	Dry cake/ 84 mo. – 8	>4.30/4.50	>3.75/3.93	>4.30/4.50	
Additional Samples Found During Measurement Activities							
SNF Floor 60L PG	thin cake broke	0.6	Dry cake/ 126 mo. – 9	1.25/1.32			

* Settling times until the UCS measurements were made in September 2011 are shown.
The references to estimate starting dates for settling are:
1 – Sinkov et al. 2010
2 – Delegard et al. 2011
3 – Delegard et al. 2008
4 – Makenas et al. 1997
5 – Mellinger et al. 2004
6 – Bryan et al. 2004
7 – Bredt et al. 1999
8 – Delegard et al. 2005
9 – Schmidt et al. 2003

The shear strengths were derived using Equations 2 and 3 based on the calibration-corrected UCS values given in Table 3.1. The resulting shear strengths are provided in Table 3.2. It is seen in Table 3.1 that most UCS values are <1.6 kg_f/cm^2 and thus must be fit using Equation 2 for homogeneous materials. Only samples KE Pit and KC-4 P250 had UCS values consistently greater than 1.6 kg_f/cm^2. Therefore, the UCS values for these samples were converted to shear strength values using Equation 3 for heterogeneous materials.

Table 3.2. Archive Sludge Shear Strength Measurement Results

Sludge Sample	Shear Strengths, kPa, Based on							
	Equation 2 for Homogeneous Materials				Equation 3 for Heterogeneous Materials			
Key Archive Samples								
KC-2/3 Comp	**23**	**8**	**5**		-9	-36	-40	
96-05	**14**	**12**	**8**		-24	-28	-36	
96-13 KE Comp A	**23**	**8**	**12**		-9	-36	-28	
KE NLOP #2	**12**	**12**	**23**		-28	-28	-9	
KC-4-2	**16**	**23**	**25**	**19**	-21	-9	-5	-17
FE-5 Comp 1	**12**	**12**			-28	-28		
KE Floc Comp	**14**	**12**	**14**		-24	-28	-24	
Other Samples Identified in Planning								
96-13 SSOL	77*	**45***	**34***		**83***	29*	10*	
KC-4 Whole	**23**	**14**	**19**		-9	-24	-17	
KE Pit (1995)	86	103	74	108	**98**	**129**	**79**	**136**
KE NLOP #1	**12**	**12**			-28	-28		
KE NLOP #3	**12**				-28			
FE-3 Comp 1	**54***	**45***			44*	29*		
KC-4 P250	135	72	128		**182**	**75**	**171**	
SNF Comp Stl Study	>193*	>168*	>193*		**>281***	**>239***	**>281***	
Additional Samples Found During Measurement Activities								
SNF Floor 60L PG	**57***				48*			

Shear strength values in **bold** are recommended over the values in normal font. Values obtained using Equation 3, for heterogeneous materials, are preferred if the UCS measured by the penetrometer is >1.6 kg_f/cm^2. For materials with penetrometer readings of UCS <1.6 kg_f/cm^2, values obtained using Equation 2 for homogeneous materials are preferred.

* Measurements invalid; see Table 3.1.

Figure 3.1. Pocket Penetrometer Measurement of Sludge Sample KE Pit

Upper left – KE Pit sample jar; upper middle – ¼”×¼” penetrometer tip approaching sludge surface; upper right through 3rd row right – penetrometer progressively entering sludge for fourth measurement; lower left – four indentations left by penetrometer tip, note cracking between measurement indentations; lower right – unconfined compressive strength reading 2.40 kg_f/cm^2 (equivalent to 108 kPa shear strength for homogeneous sludge based on the penetrometer calibration in Equation 2 or 136 kPa based on Equation 3 for heterogeneous sludge).

Views of the measurement of sample KE Pit are shown in Figure 3.1; views of KC-4 P250 measurement are shown in Figure 3.2.

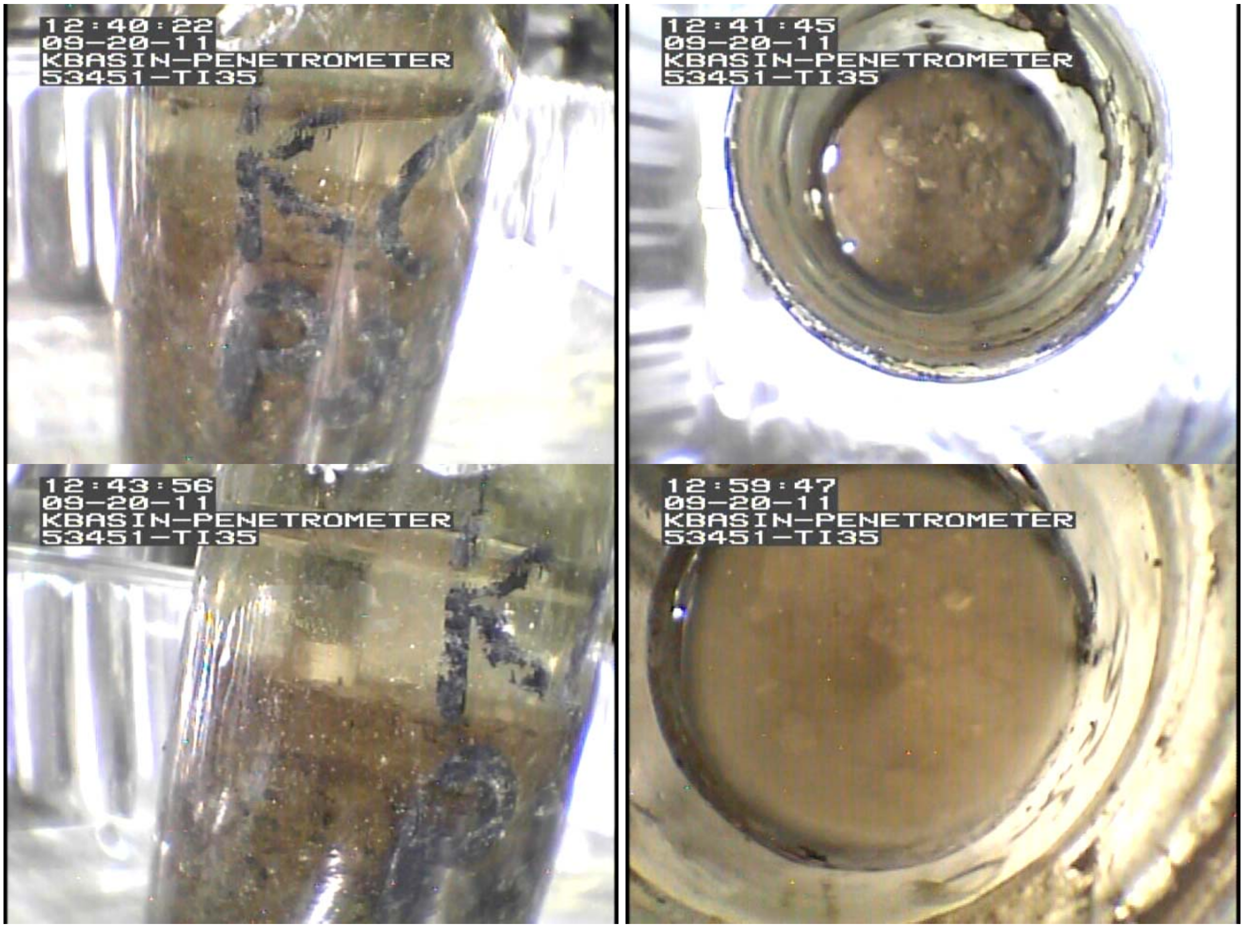


Figure 3.2. Pocket Penetrometer Measurement of Sludge Sample KC-4 P250

Upper left – KC-4 P250 sample jar; upper right – top view of KC-4 P250 surface before measurement; lower left – ¼”×¼” penetrometer tip being pressed into sludge surface; lower right – sludge surface after measurement.

The average UCS and shear strengths, the standard deviations of the UCS measurements and shear strength estimates at 1 sigma (σ), and the consistency descriptions of the measured sludges are shown in Table 3.3. Based on the consistency descriptions provided by Clayton et al. (1995) and the British Standard (1999), it is seen that the application of Equation 3 for heterogeneous material to the archive sludges with UCS values <1.6 kg_f/cm^2 would have been inaccurate, indicating negative shear strength. Appropriately, Equation 2 for homogeneous materials was used to determine the shear strengths for these ten sludges (KC-2/3 Comp, 96-05, 96-13 KE Comp A, the three KE NLOP samples, KC-4-2, FE-5 Comp 1, KE Floc Comp, and KC-4 Whole). Their consistencies are accurately described as “molded by light finger pressure” (i.e., soft) or “exudes between fingers when squeezed in hand” (i.e., very soft) in accord with the 11 to 21 kPa shear strengths found by Equation 2.

The UCS and shear strengths of all of the key archive samples are low, with individual consistencies being described in Table 3.3 as very soft (0.02 to 0.25 kg_f/cm^2) or soft (0.25 to 0.50 kg_f/cm^2) according to

Corps of Engineers definitions (Corps of Engineers 1994). Three of the remaining samples giving valid UCS readings (KC-4 Whole and KE NLOP #1 and #3) also were very soft to soft in consistency in individual tests. On average, these materials were soft. Of the samples providing valid UCS measurements, only KE Pit and KC-4 P250 had UCS values consistently above 1.6 kg_f/cm^2. Their consistencies are described as stiff (1.0 to 2.0 kg_f/cm^2) to very stiff (2.0 to 4.0 kg_f/cm^2). Very similar consistency descriptions are made based on shear strength (Clayton et al. 1995; British Standard 1999).

Table 3.3. Strengths of Archive Sludges Estimated Based on Valid Penetrometer Measurements

Sludge Sample (* key archive samples)	UCS, kg_f/cm^2, Average and Standard Deviation (±1σ)	Shear Strength, kPa, Average and Standard Deviation (values in bold are recommended)		Consistency	
		Equation 2, Homogeneous	Equation 3, Heterogeneous	UCS – Corps of Engineers[(a)]	Shear Strength – British Standard[(b)]
KC-2/3 Comp*	0.28 ± 0.23	**12 ± 10**	-28 ± 17	Soft	Very soft
96-05*	0.26 ± 0.08	**11 ± 3**	-29 ± 6	Soft	Very soft
96-13 KE Comp A*	0.33 ± 0.19	**14 ± 8**	-24 ± 14	Soft	Very soft
KE NLOP #2*	0.37 ± 0.15	**16 ± 6**	-22 ± 11	Soft	Very soft
KC-4-2*	0.49 ± 0.10	**21 ± 4**	-13 ± 7	Soft	Soft
FE-5 Comp 1*	0.28 ± 0.00	**12 ± 0**	-28 ± 0	Soft	Very soft
KE Floc Comp*	0.31 ± 0.03	**13 ± 1**	-26 ± 2	Soft	Very soft
KC-4 Whole	0.44 ± 0.10	**19 ± 4**	-17 ± 8	Soft	Very soft
KE Pit (1995)	2.17 ± 0.36	93 ± 16	**110 ± 27**	Very stiff	Stiff
KE NLOP #1	0.28 ± 0.00	**12 ± 0**	-28 ± 0	Soft	Very soft
KE NLOP #3	0.28 ± —	**12 ± —**	-28 ± —	Soft	Very soft
KC-4 P250	2.61 ± 0.80	112 ± 34	**143 ± 59**	Very stiff	Stiff

(a) Consistency descriptions obtained from the Corps of Engineers (1994) and from "Consistency/strength of clay mixtures" (Solum 2005) are based on UCS.

- Fluid mud (UCS <0.02 kg/cm^2).
- Very Soft (UCS 0.02-0.25 kg/cm^2) – Easily penetrated several inches by thumb. Exudes between fingers and thumb when squeezed.
- Soft (UCS 0.25-0.5 kg/cm^2) – Easily penetrated one inch by thumb. Molded by light finger pressure.
- Medium (UCS 0.5-1.0 kg/cm^2) – Can be penetrated ¼" by thumb with moderate effort. Molded by strong finger pressure.
- Stiff (UCS 1.0-2.0 kg/cm^2) – Indented about ¼" by thumb but penetrated only with great effort.
- Very stiff (UCS 2.0-4.0 kg/cm^2) – Readily indented by thumb nail.
- Hard (UCS >4.0 kg/cm^2) – Difficult to indent by thumb nail.

(b) 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. The consistency descriptions for the sludge are based on the recommended shear strength values given in **bold**.

- 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*.

As shown in Table 3.3, only two of the twelve sludges that had valid measurements and measurement conditions, KE Pit and KC-4 P250, showed significant UCS and shear strength. The shear strengths, 110 and 140 kPa for KE Pit and KC-4 P250, respectively, are greater than observed for any K Basin sludge in prior testing except for sludge that had been processed at 185°C under hydrothermal conditions.

The shear strength of the KC-4 P250 sludge had been measured in 1999, shortly after it was split from the KC-4 mother sample and after being allowed to remain undisturbed for about two weeks (see Table 10 and associated text of Bredt et al. 1999). Shear strengths for KC-4 P250 were 3600 and 2000 Pa (3.6 and 2.0 kPa), considerably lower than the ~140 kPa shear strength derived from penetrometer measurements in the present testing. No prior strength measurements of KE Pit sludge are available.

The KE Pit sludge was created in 1998 as a composite of five sludge samples taken from the KE Basin Weasel Pit (Carlson et al. 1998). The five individual samples (KES-P-16, Q-17, R-18, S-19, and T-20) were collected in 1995 (Makenas et al. 1996) and had air-dried before the composite was prepared. The dry solids were separately dry-sieved to remove most of the accompanying OIER, the sieved fractions combined, and then distilled and deionized water was added to reconstitute the dry sludge to a composite wet settled sludge.

It is seen that the KE Pit sludge, at 36.4 wt%, dry basis, has the highest iron concentration of any tested sludge while the KC-4 Whole sludge has the second-highest iron concentration, 24.3 wt%, dry basis. However, the exact composition of KC-4 P250, taken from KC-4 Whole, is unknown. As a result, ascribing the high strengths of the KE Pit and KC-4 P250 sludges to commonalities in their compositions is tenuous. It is also noteworthy that the strengths measured for KC-4 Whole and KC-4-2 are low even though they, like the strong KC-4 P250 fraction, are derived entirely from KC-4 origins. Like KC-4 Whole and KC-4-2, the KE Floc Comp sludge also has relatively high iron concentration (24.2 wt%) and low strength.

A further attempt was made to estimate the relative iron concentration in the KC-4 sludge sieve fractions based on particle size and color. The iron oxide and hydroxide materials found in sludge generally are extremely fine and are unlikely to be retained in the P250 (>250 μm) sieve fraction constituting the KC-4 P250 material. The appearance of the P250 fraction during its separation from the bulk KC-4 sludge does not show the red-brown color characteristic of iron corrosion products (see Figure **3.3**; taken from Figure 9 of Bredt et al. 1999). However, the color of the sludge retained on the screens, which constitute the P250 fraction, is similar to that of the sludge passing the screens (the M250 fraction), thus suggesting that the KC-4 P250 sludge iron concentration may be similar to, or even greater than, that of the parent KC-4 sludge.

The high strengths of the KE Pit and the KC-4 P250 samples also might be attributed to lengthy settling times. The KE Pit sample had been left undisturbed for 52 months before it was measured while the KC-4 P250 sample had been left undisturbed for 146 months before measurement. While these are comparatively long times, they are not markedly different than the 30 to 90 months that the ten weaker samples had been left undisturbed.

In the end, no correlation could be found between the high strengths found for the KE Pit and KC-4 P250 sludge samples and their sludge compositions or their settling times.

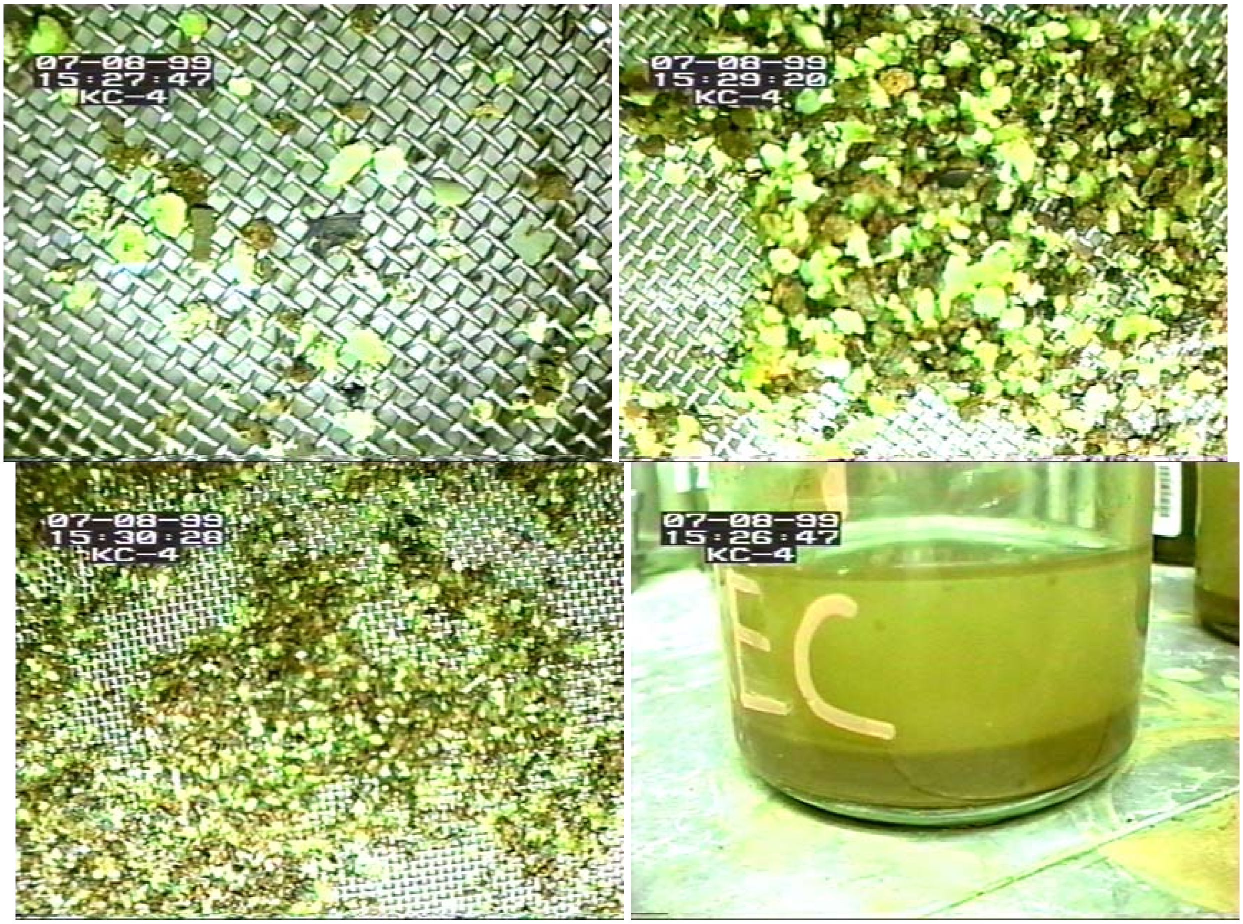


Figure 3.3. Sludge Particles Retained on 12-Mesh (top left; 1400 µm), 32-Mesh (top right; 500 µm), and 60-Mesh (lower left; 250 µm) Screens and Passing (lower right; <250 µm) in KC-4 Sludge Wet Sieving. (Sludge retained on the sieves constituted KC-4 P250; that passing the sieves constituted KC-4 M250.)

4.0 Conclusions

The UCS of archive K Basin sludge samples and sludge blends were measured using a pocket penetrometer modified for hot cell use. Based on known correlations, the UCS values measured by the pocket penetrometer were converted to shear strengths. Twenty-six sludge samples were either suggested in the TI as potential candidates for sludge strength measurement or were examined in the course of accessing the samples identified in the TI. For various reasons, including sample dryness and inability to view penetration depth, valid UCS measurements could only be made for twelve samples. These materials included all seven of the key archive samples. The sludges for which valid measurements were made were present in their storage jars as moist or water-immersed solids at least ½-inch deep. Two of the samples were measured in quadruplicate, seven in triplicate, two in duplicate, and one had a single measurement. The UCS measurement reproducibility generally was 30%, relative, for these multiple determinations.

Ten of the twelve samples, including all seven key archive samples, were relatively weak, having UCS values between 0.26 and 0.49 kg_f/cm^2, with consistencies described as “soft”. The shear strengths, ranging from 11 kPa to 21 kPa, were determined based on the published shear strength correlation to UCS for homogeneous materials (Onishi et al. 2011). The consistencies according to the corresponding shear strength values were similarly described as “very soft” to “soft”.

Two of the twelve samples, KE Pit and KC-4 P250, were strong with respective UCS values of about 2.2 and 2.6 kg_f/cm^2. These UCS values correspond to shear strengths of about 110 kPa and 140 kPa, respectively, based on the experimentally-determined correlation for heterogeneous materials (Onishi et al. 2011). The UCS values for both materials correspond to “very stiff” consistency, described qualitatively as “readily indented by thumb nail”. The shear strength values for both materials correspond to “stiff” consistency described as “can be indented by thumb”.

Although both KE Pit and, likely, KC-4 P250 have relatively high iron concentrations, assignment of their high strength to this factor cannot be made with confidence. This is because sludge samples KC-4 Whole, KC-4-2, and KE Floc Comp, which also have relatively high iron concentrations, have low UCS and shear strengths. The high strengths of the KE Pit and the KC-4 P250 samples also might be attributed to their prolonged settling times of 52 months and 146 months, respectively, before measurement. While these are comparatively long times, they are not markedly different than the 30 to 90 months that the ten weaker samples had been left undisturbed. In the end, no reason could be found to explain the much greater strengths of the KE Pit and KC-4 P250 sludge materials compared with the other measured archive sludge materials. The observed UCS and shear strengths were greater than observed in any prior testing of K Basin sludge except sludge that had been processed at 185°C under hydrothermal conditions.

The KE Basin sludge samples that were measured for UCS are composites of distinct sludge types collected prior to consolidation of the KE Basin sludge into underwater engineered containers SCS-CON-240, -250, -260, and -230 in the KW Basin. Consolidation of these KE Basin sludge types into these engineered containers has resulted in the mixing of KE Basin floor, pit and some canister sludge. This consolidation and mixing could affect the magnitude of strength changes with time.

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Appendix A

Soiltest Penetrometer Information

MODEL CL-700A

POCKET PENETROMETER

03/84

Technical Data

SOILTEST®

Soiltest, Inc. • 86 Albrecht Drive • P.O. Box 8004
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1. GENERAL

The Soiltest CL-700A Pocket Penetrometer was originally developed by Soiltest, Inc. for use by field engineers in checking visual classifications of soils. Soiltest compiled data on several thousand unconfined compressive strength tests of silty clays and clayey soils against the penetrometer readings to develop the penetrometer scale. A close relationship exists between the reading and the soil type. Penetrometer principle and accuracy have been proven by thousands of penetrometers in years of use throughout the world. Many organizations now supply all field and laboratory engineers with Soiltest Pocket Penetrometers to insure uniform soil classification; although the penetrometer does not replace field and laboratory testing analysis.

Several features have been incorporated into the design of the Soiltest Pocket Penetrometer: lightweight construction, a polished and ground steel loading piston, and a direct reading scale which has been permanently etched into the piston barrel. The scale covers the range of 0.25 to 4.5 tons/sq. ft. and kg/cm^2 with 0.25 tons/sq. ft. (kg/cm^2) divisions.

The calibrated spring has been heat treated and plated for rust resistance. Light and compact, the complete assembly weighs only seven ounces (198 grams); diameter is 3/4 inches and the length is 6 3/8 inches. Includes a canvas carrying case with handy belt loop.

The CL-700A has a spring constant of 12 $\pm$ 0.25 pounds/inch. One ton/sq. ft. interval on the scale is equivalent to 8 mm. Therefore, a compressive force of 3.78 pounds on the foot is required to read 1 ton/sq. ft. The equivalent of 3.78 pounds on .049 sq. inch (1/4 inch diameer foot) is 5.58 tons/sq. ft.

Why does the penetrometer need this very high force to read 1 ton/sq. ft.? The penetrometer reading is taken by pushing its foot into the material to a depth of 1/4 inch. The conical surface area of the material being sheared is twice the area of the foot, and the material under the foot is more compacted when the foot penetrates 1/4 inch. This accounts for the large value of compressive force.

2. OPERATION

2.1 Move the red ring up to the lowest reading on the scale. When the ring is in its proper position, it should rest against the lower edge of the instrument handle.

2.2 Grip the knurled portion of the handle and push the piston with steady pressure into the soil up to the calibration groove (machined on the piston about 1/4 inch from the end).

2.3 Read unconfined compressive strength directly in tons per sq. ft. or kilograms per sq. cm. on the low-load side of the red ring. (Indicator ring automatically holds its position after piston is released.)

3. SPECIAL NOTES

3.1 Select test spots with smooth surface.

3.2 Hold penetrometer piston at right angles to the surface of material being tested.

3.3 For minimizing errors, (a) take several readings; (b) discard those readings which differ considerably from the majority of readings, and (c) take the average of the remaining readings.

4. ACCESSORIES

4.1 CL-701 Penetrometer Adapter foot

The CL-701 Penetrometer adapter foot is an accessory for the CL-700A pocket penetrometer when it is used for measuring the compressive strength of very soft materials. The adapter foot has a diameter of 1 inch compared to the 1/4-inch diameter of the pocket penetrometer piston. The effective area of the piston is increased 16 times when the adapter foot is attached. Therefore, the reading in tons per square foot (or kilograms per sq. cm.) on the low-load side of the red ring should be divided by 16 to get the correct unconfined compressive strength of the material.

In order to use the CL-701 on the CL-700A, (a) fix the adapter foot on the piston of the CL-700A by inserting the piston all the way into the adapter foot collar and then tightening the screw, and (b) grip the knurled portion of the handle of CL-700A and push the piston with steady pressure into the soil (test material) up to the full thickness (1/4 inch) of the adapter foot.

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