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CEL WORKING PROCEDURES FOR WRAP 2A FORMULATION DEVELOPMENT TESTS	ECN No. N/A

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Page 1 of 381

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MASTER

CEL WORKING PROCEDURES FOR
WRAP 2A FORMULATION DEVELOPMENT TESTS

TABLE OF CONTENTS

Preparation of Grout Mixtures and Specimens	3
Viscosity	7
FANN Viscometer	7
Flow Cone	14
Bulk Density	18
Uncured Grout	18
28-day Cured Grout	21
Volume Percent Drainable Liquid	21
Time of Setting	24
Compressive Strength	27
ANSI 16.1 Leach Test	30
pH	34
pH of solids	34
pH of liquids	36
Thermocycling	37
90-Day Water Immersion	40
Toxicity Characteristic Leaching Procedure (TCLP)	41
References	47

LIST OF TABLES

Labeling Requirements for Compression Test Specimens	4
Containers Used for the Preparation of Each Batch of Cementitious Specimens	6
Fann Viscometer Data Example	12
Density of Water as a Function of Temperature (g/cm ³)	19
Total Elapsed Time at Leachant Changeout	32
Typical Schedule For Thermocycling Tests	38
Samples Scheduled for TCLP Extraction	41
TCLP Sample Maximum Holding Times	46
Feed Stream 1C Specimen Preparation Worksheet	I
Feed Stream 1B Specimen Preparation Worksheet	II
Feed Stream 1D-1 Specimen Preparation Worksheet	III
Feed Stream 1D-2 Specimen Preparation Worksheet	IV
Feed Stream 2A-1 Specimen Preparation Worksheet	V
Feed Stream 2A-2 Specimen Preparation Worksheet	VI
Feed Stream Type 7 Specimen Preparation Worksheet	VII

INTRODUCTION

This document provides the stepwise, procedural instructions for the Chemical Engineering Laboratory Personnel to prepare and test cementitious specimens, and supports the WRAP Module 2A Waste Immobilization Demonstration Test Plan for Cementitious Stabilization of Major Feedstreams, and the WRAP 2A Waste Form Qualification (WFQ) Plan, WHC-SD-W100-TP-002, REV. 0, for the WRAP module 2A facility.

The WRAP 2A facility will encapsulate retrieved, stored, and newly generated contact-handled mixed low level waste (MLLW) into relatively small 55-500 gal. cementitious forms at the Department of Energy's Hanford Site near Richland, Washington. The WFQ program will provide the technical basis supporting the process, and will demonstrate that the selected treatment process will produce final waste forms that will meet all applicable regulatory requirements and performance specifications.

Tests at the CEL involve mixing simulated (surrogate) MLLW with mixtures of either Portland or masonry cement, and fly ash. The mass ratios of cement, fly ash, waste, and water will be varied (using a statistically approach) in order to derive an optimum formulation for each type of waste. The surrogate feed streams that were prepared for use at the CEL were representative of the following waste types.

- 2A-1 High ammonium sulfate C-018H salts.
- 2A-2 No ammonium sulfate C-018 salts.
- 1B 183-H Basins 3 & 4 crystalline solids.
- 1C 183-H Basins 3 & 4 sludges.
- 1D-1 183-H basins 1 & 2 contaminated sandblast grit.
- 1D-2 183-H Basins 3 & 4 contaminated sandblast grit.
- 7 Ash from thermal treatment.

Simulated waste feed streams that will be used will be prepared by the Chemical Engineering Laboratory under; WRAP 2A Surrogate Make-Up Procedure, WHC-SD-W100-TP-008, REV. 0, prior to conducting the tests described by this test plan. These tests are listed below:

Photograph (Color, Texture, cracks, Irregularities)
Bulk Density, Cured/Uncured
Viscosity
Flowability
Curing time/Hardness
AS Cured/Compressive Strength
Leachability Index
Thermal Cycling/Compressive Strength
Water Immersion/Compressive Strength
Free Liquids
Toxic Characterization
Corrosivity (Leach pH)

This test plan does not contain a proceduralized safety section. Safety will be addressed by conducting and documenting a specific Job Hazard Analysis (JHA) for potentially hazardous activities. Deviations from these working procedures will be documented in the final test report, prepared after completion of the work.

Preparation of Grout Mixtures
and
Specimens

SCOPE

This procedure describes the mechanical mixing of dry solids blends¹ with simulated or actual waste solutions to form a grout slurry².

APPLICATION:

This procedure is applicable to both non-radioactive and low level activity grout mixtures.

MATERIALS AND APPARATUS:

1. Constant speed mixer with torque readout, a four liter stainless steel mixing beaker and a high efficiency paddle/impeller.
2. Graduated cylinder, 1000 mL.
3. Precision balance with a capacity of 3000 grams and readability of 0.1 gram.
4. 2" diam X 4" tall cement curing molds with press-on caps.
5. 3½" X 3" poly-jar with screw cap lid.
6. Liquid Scintillation Vial, 1" diam. X 2¾" tall
7. Plastic or glass tamping rod, ½" diam X 8"
8. Vortex mixer
9. Flat stainless steel spatula
10. Type "F" fly ash.
11. Portland Cement.
12. Simulated waste (2A-1, 2A-2, 1B, 1C, 1D-1, 1D-2, Type 7).
13. ANSI Primary Spike Solution (add 50.00g CsNO₃ to a tared 1.000 L volumetric flask and dilute to the calibration line with deionized H₂O. Record the final weight in the appropriate laboratory notebook)

PROCEDURE:

1. Tare weigh and label six (6) 2" x 4" grout curing specimen molds with the sample information as shown in table 1. Write the tare wt on the bottom of the specimen mold.

Table 1. Labeling Requirements for Compression Test Specimens

FEED TYPE	BATCH No.	TEST TYPE	ALIQOT NO.	DATE MM/DD/YY
2A1, 1D1 ..ETC	01,02,... ..18	A = CS AS-CURED B = CS IMMERS C = CS THERM	1 or 2	

example: 2C-3-B-1
04/18/94

2. Label one ANSI sample mold and six penetrometer molds with the appropriate feed type and batch No.

example: 2C-3
04/18/94
3. Maintain the grout laboratory room temperature for mixing between 20°C and 25°C.
4. Bring the temperature of the dry solids blend, waste solutions and the mixer to within the above temperature range at the time of mixing.
5. Assemble the mixing apparatus by Placing the mixing impeller into mixer chuck, placing the stainless steel mixing beaker onto it's stand and securing it to the mixer frame using the adjustable metal strap-type clamp.
6. Place the 4-Liter stainless steel beaker onto the electronic balance and tare the balance to obtain a zero reading. Leave the beaker on the balance.
7. Measure 50 ml of the ANSI Primary Spike Solution into a graduated cylinder, and transfer the contents of the graduated cylinder to the tared beaker. Record the weight of the spike solution used into the laboratory notebook.

8. Add deionized water to the beaker containing spike solution until the total mass of spike solution plus water equals the amount of water required by the formulation design (see appendix A). Use a plastic transfer pipet to transfer the last few grams of water. Set the beaker aside, near the working/mixing area.
9. Weigh out the appropriate amount of surrogate waste material into a plastic weighing boat/beaker and set it aside, near to the working/mixing area.
10. Weigh the calculated amount of the dry solids (fly ash and cement) blend into plastic weighing boats and set them aside, near to the working area.
11. Transfer the surrogate waste into the beaker containing the water/spike solution. to the stainless steel beaker. Turn the mixer on low speed (200 ± 5 rpm), and mix until the surrogate is well mixed (approximately 3.00 minutes).
12. Note the time and date in the laboratory notebook. Slowly add the Add the fly ash, then the cement to the beaker containing the surrogate/water mixture. Continue mixing for 60 seconds.
13. Increase the mixer speed from 200 rpm to 400 rpm. Continue mixing at 400 rpm for 60 additional seconds.
14. Sequentially measure and record the torque reading from the mixer controller at 400, 300, 200 and 100 rpm (use decreasing order). Pause between speed adjustments long enough to obtain a stable measurement.
15. Stop the mixer. Transfer and set aside a suitable amount (about 2000 ml) of slurry and perform density, Cone viscosity and fann viscosity measurements. This should take no more than 5 minutes. Upon completion of the density and viscosity tests. Transfer the slurry into the appropriately labeled molds/containers, as listed below in table 2. Fill the compressive strength molds first, followed by the ANSI mold, and the penetrometer molds.

Table 2. Containers Used for the Preparation of Each Batch of Cementitious Specimens for WRAP-2A Formulation Tests.

QUANTITY	VOLUME (ml)	DIMENSIONS (OD) Diam. X length	PURPOSE
2	206	2" X 4"	Normal Comp. Strength.
2	206	2" X 4"	Thermal-Cycle/Comp. Strength
2	206	2" X 4"	90-Day Immers/Comp. Strength
1	20	1" X 2½"	ANSI 16.1, 90 Day Leach
1	500	Stainless Dewar	Curing Exotherm (if reqd.)
5-6	500	3½" X 3" poly-jar	Penetrometer

16. Pour or scoop the cementitious slurry into each mold. If the slurry is thick, filleach mold half way, vortex, then fill the mold the rest of the way. Overfill each mold slightly. Tamp the slurry with a glass or plastic rod if necessary, and place the specimen onto a vortex mixer and vortex/vibrate the specimen for 15-25 seconds to eliminate/minimize air pockets. Blade-off the top of each specimen using the edge of a piece of stainless steel spatula. Wipe the outside of the mold off.
17. Weigh the specimen to the nearest 0.1g, and record the weight in a laboratory notebook.
18. Place caps onto each of the "compressive strength" molds, Being careful not to disturb the "trowelled" surface. place the molds into a 473 ml (16 fl. oz) glass jar and secure the lid. Label the container with the date and time of mixing for the batch (step 14).
19. Place the caps onto the ANSI and penetrometer specimens and label the containers with the date and time of mixing for the batch (step 14).
20. Clean/wash the mixing beaker and impeller and blot dry with clean paper towels.
21. Repeat steps 3 to 12 if another batch of grout slurry is required.

Determination of Viscosity
of Freshly Prepared Grout Mixtures
Using a FANN Viscometer

SCOPE:

This method describes the determination of viscosity of a grout mixture using a FANN viscometer.

APPLICATION:

This procedure is applicable to non-radioactive grout formulations only. This method will be used on grouts mixtures made from the following surrogate waste types:

Type 2A-1	High Ammonium Nitrate
Type 2A-2	No Ammonium Nitrate
Type 1B	183-H Basins 3&4 Crystalline Solids
Type 1C	183-H Basins 3&4 Sludge
Type 7	Incinerator Ash
Type 2C	Metal Sludge

APPARATUS and MATERIALS:

1. FANN viscometer and accessories set up with a R1-B1-F1 configuration for the Rotor-Bob-Spring Force.
2. Freshly prepared grout mixture.

THEORY

Flow properties of wellborn fluids³ (mud, displacement fluids, cements, slurries and heavy asphaltic oils) do not behave in a classical "Newtonian" manner. Newtonian fluids, such as oil, syrup, or water, exhibit a direct linear relationship between shear stress and shear rate⁴; that is, when shear stress is plotted against shear rate, the result will be a straight line going through the origin, with the slope of the line equal to the viscosity. Viscosity remains constant regardless of the shear rate, therefore only one point is required to define this curve.

For non-Newtonian fluids, this relationship is not linear. A plot of shear stress vs shear rate will result in a curved line, with the slope of that curve varying with shear rate and the line not necessarily intersecting with the origin. Unlike Newtonian fluids, the slope of a straight line drawn from the origin to any point on the shear-rate/shear-stress curve will not yield viscosity, but rather Apparent Viscosity (μ), which varies with shear rate. Viscosity is defined as the slope of the linear portion of this shear-rate/shear-stress curve, and is constant within a defined range of shear rates.

Viscosity (η) is defined as shear stress (τ) divided by shear rate (γ), (eqn. 1)

$$\eta = \frac{\tau}{\gamma} \quad (\text{eqn. 1})$$

Where: τ = Shear stress in Dyne/cm²
 η = Viscosity in poise (1 poise = 100 cP)
 γ = Shear rate in sec⁻¹

On the Fann viscometer, The shear stress (τ) is measured as a deflection (0-300°) of a torsion spring⁵. It is possible, therefore, to compute a shear stress (τ) from the dial reading (ϕ) with the appropriate instrument constants. Shear stress is defined as the torsion constant of the spring times the product of the deflection angle and the shear stress constant for the effective bob surface area (eqn. 2).

$$\tau = k_1 k_2 \phi \quad (\text{eqn. 2})$$

Where: k_1 = The torsion constant in Dyne-cm/degree deflection.
 k_2 = The shear stress constant for the effective bob surface, cm⁻³.
 ϕ = The angle of deflection.
 τ = Shear Stress, Dyne/cm².

Shear rate is defined as the product of the shear rate constant (a function of the rotor and bob used) and the rate of revolution of the outer cylinder (eqn. 3)

$$\gamma = k_3 N \quad (\text{eqn. 3})$$

Where: k_3 = The shear rate constant, sec⁻¹ per rpm.
 N = The rate of revolution of the outer cylinder, rpm
 γ = Shear rate, sec⁻¹

Non Newtonian fluid behavior can be described by one of two mathematical rheological models⁶: the Bingham Plastic model or the more complex Power-Law model. The power-Law model has been found to more accurately model the behavior of cement slurries, and therefore will be used in the WRAP-2A testing. The equation for the Power-Law model is defined as:

$$\tau = K(\gamma)^n \quad (\text{eqn. 4})$$

Where: n = The measure of non-Newtonian flow behavior index, unitless
 K = The Grout's fluid consistency index in $\text{Dyne} \cdot \text{sec}^{-1}/\text{cm}^2$
 γ = Shear Rate in sec^{-1}
 τ = Shear Stress in Dyne/cm^2

Taking the logarithm of both sides of the equation yields:

$$\text{Log } (\tau) = n \text{ Log } (\gamma) + \text{Log } K \quad (\text{eqn. 5})$$

Which takes the form of the equation of a line:

$$Y = mX + b \quad (\text{eqn. 6})$$

A plot of $\log(\text{shear stress})$ vs $\log(\text{shear rate})$ will result in a straight line, with a slope of n , and an intercept of K .

For a power-law fluid, apparent viscosity (μ) at any specific shear rate is given by

$$\mu = K(\gamma)^{n-1} \quad (\text{eqn. 7})$$

Where: K = The consistency index; intercept of $\log(\tau)$ vs $\log(\gamma)$ curve, in $\text{Dyne} \cdot \text{Sec}^n/\text{cm}^2$.
 n = The behavior index; slope of $\log(\tau)$ vs $\log(\gamma)$ curve, unitless.
 μ = The apparent viscosity in Poise
 γ = Shear rate, sec^{-1}

PROCEDURE:

1. Clean the rotor and bob of the viscometer. Examine for dents, abrasion, or other damage. If damaged replace the damaged part with one in good mechanical condition.
2. Clean the sample cup of the viscometer. The sample cup has a line at the proper level to hold 350 mL.
3. Wipe dry the sample cup, bob, and rotor of the viscometer.
4. Attach the bob to the bob shaft and the rotor to its socket. Twist the bob around the shaft bearing gently to tighten.
5. Fill the sample cup with the freshly prepared grout mixture to the proper level (350 ml).
6. Place the sample cup on the viscometer platform/stage.
7. Lock the cup to the platform/stage.
8. Raise the cup up to the scribed line on the rotor sleeve by raising the platform using the locking knob.
9. Connect the viscometer to the proper power source (115V).
10. Turn motor on, select the desired speed and read the shear stress value ϕ (1° Fann ≈ 1 lb/100 ft²) from the dial located on the viscometer head.
11. Repeat step 10 for four different speeds (600, 300, 200, 100) of the viscometer (in that order). Complete step 11 within 1.5 minutes. Record the value of the shear stress for each speed into table and record into the appropriate laboratory notebook.

CALCULATIONS

1. Calculate the shear rate, γ , for each speed using equation 8. Appendix B contains the applicable apparatus constants for the R1-B1-F1 configuration.

$$\gamma = k_3 N \quad (\text{eqn. 8})$$

$$\gamma = (1.7023) (N) \quad \text{sec}^{-1}$$

Where: k_3 = The shear rate constant, 1.7023 sec⁻¹ per rpm.

N = The rate of revolution of the outer cylinder, rpm

γ = Shear rate, sec⁻¹

2. Convert the dial readings obtained from steps 10 and 11 (approximate shear stress) to actual shear stress using equation 9. Appendix B contains the applicable apparatus constants for the R1-B1-F1 configuration.

$$\begin{aligned}\tau &= k_1 k_2 \phi & (\text{eqn. 9}) \\ \tau &= (386) (0.01323) \phi \\ \tau &= 5.107 \phi \text{ Dynes/cm}^2\end{aligned}$$

Where: k_1 = The torsion constant; 386 Dyne-cm/degree deflection.
 k_2 = The shear stress constant for the effective bob surface;
0.01323 cm^{-3} .
 ϕ = The angle of deflection.
 τ = Shear Stress, Dyne/cm^2 .

3. Construct a log-log plot of shear stress vs. shear rate. This plot should be linear. Fit a linear line through the data using a least squares, linear regression curve-fitting program. The measure of consistency (K) is the y intercept of the line. The non-Newtonian behavior (n) is the slope of the line. K has the units of $\text{Dyne}\cdot\text{sec}^n/\text{cm}^2$ and n is unitless.
4. Record the values of K and n from step 3.
5. Calculate the apparent viscosity, μ , of the grout mixture expressed as centipoise (cP) using equation 10 (centipoise = poise X 100).

$$\mu = K \gamma^{(n-1)} \quad (\text{eqn. 10})$$

Where: n = The measure of non-Newtonian flow behavior index.
K = The Grout's fluid consistency index, $\text{Dyne}\cdot\text{Sec}^n/\text{cm}^2$
 μ = Apparent viscosity, Poise
 γ = Shear Rate, sec^{-1}

EXAMPLE

Table 3. Fann Viscometer Data Example

N (rpm)	ϕ ($^{\circ}$ Fann)	γ (sec^{-1})*	τ (Dyne/cm 2)*
600	62	1021	316.6
300	41	511	209.4
200	32	340	163.4
100	24	170	122.6

* - Calculated for an R1-B1-F1; rotor-bob-force(spring) configuration.

REGRESSION OUTPUT

Constant	0.8825208
Std Err of Y Est	0.0205579
R Squared	0.9907608
No. Observations	4
Degrees of Freedom	2
X Coefficient(s)	0.533664
Std Err of Coef.	0.036441

From the log shear stress versus log shear rate curve (Figure 1),

$$\begin{aligned} \text{Log}(K) &= 1.3157453 & (\text{eqn. 11}) \\ 10^{\text{Log}(K)} &= 10^{1.3157453} \\ K &= 20.6893 \text{ Dyne}\cdot\text{sec}^n/\text{cm}^2 \end{aligned}$$

Apparent viscosity (μ) at 300 rpm from equation (7)

$$\begin{aligned} \mu &= K\dot{\gamma}^{n-1} & (\text{eqn. 12}) \\ \mu &= (20.6893) (511)^{(0.355-1)} \\ \mu &= 0.37052 \text{ Poise} \\ &= 37.1 \text{ centipoise (cP)} \end{aligned}$$

Determination of Viscosity of Grout by Flow Cone

SCOPE

The time of efflux of a specified volume of grout from a standardized flow cone is measured and the viscosity is determined by comparison with the time of efflux of water.

APPLICATION

This method can be used to determine the coefficient of viscosity and/or the "fluidity" of grout mixtures containing small aggregates. Grout mixtures subjected to this method should not contain particles larger than those capable of passing through a No. 8 (2.36 mm) sieve⁷.

THEORY

Typically, the coefficient of viscosity of liquids can be determined by the time of flow of a given volume of liquid through a vertical capillary tube under the influence of gravity. It can be shown⁸ that for a given total volume of liquid:

$$\frac{\eta}{\rho} = B \Delta t \quad (\text{eqn. 13})$$

Where: η = Coefficient of viscosity.

ρ = Density of the liquid.

B = Apparatus constant that must be determined through calibration with a liquid of known viscosity.

Δt = Time required for the first break in continuous flow of liquid from the funnel.

APPARATUS

1. Flow Cone, with dimensions as shown in figure 1. The body and discharge tube shall be made of stainless steel.
2. Receiving Container, Capacity of 2,000 ml min.
3. Ring Stand, capable of supporting a loaded flow cone in a steady vertical position over the receiving container.
4. Carpenter's level.
5. Stop Watch, least reading of not more than 0.2 seconds.

6. Thermometer

Figure 2. Dimensions of Flow Cone Used for Cement Flowability Measurements

PROCEDURE

1. Allow all materials to come to ambient temperature($23 \pm 2^{\circ}\text{C}$). Record the temperature in a laboratory notebook.
2. Mix the cement per mixing procedure.
3. Determine the density of the grout slurry using the appropriate volumetric method.
4. Mount the flow cone in such a manner that is free from vibration. Level the top.
5. Close the discharge tube by placing a finger against the lower end.
6. Introduce $1725 \pm 5\text{ml}$ of water into the cone.
7. Start the stopwatch and simultaneously remove finger from the bottom of the discharge to allow liquid to drain.
8. Stop the stopwatch at the first break in the continuous flow of water. Record the time as $\Delta t_{\text{H}_2\text{O}}$.
9. Moisten the inside of the flow cone by filling the cone with water and, 1 minute before introducing the grout sample, allow the water to drain from the cone.
10. Close the outlet of the discharge tube with a finger or stopper.
11. Introduce $1725 \pm 5\text{ml}$ grout to the cone.
12. Start the stopwatch and simultaneously remove finger from the bottom of the discharge to allow liquid to drain.
13. Stop the stopwatch at the first break in the continuous flow of cement. Record the time as Δt_{CRT} .
14. Look into the cone; if light is visible, the time indicated by the stopwatch is the efflux time of the grout. If light is not visible, the flow cone is not applicable for grout of this consistency.

CALCULATIONS

1. The apparatus constant (B) is derived by solving equation 13 for B.

$$B = \frac{\eta_{H_2O}}{\rho_{H_2O} \Delta t_{H_2O}} \quad (\text{eqn.14})$$

Where: ρ_{H_2O} = Density of water at 25°C; 0.9970 g cm⁻³
 η_{H_2O} = Coefficient of Viscosity of water at 25°C; 0.8937 cP
 Δt_{H_2O} = Measured efflux time of the water ± 0.2 Seconds.

2. Equation 13 is then solved for η by substitution of B from equation 14.

$$\eta_{cmt} = \frac{\rho_{cmt} \eta_{H_2O} \Delta t_{cmt}}{\rho_{H_2O} \Delta t_{H_2O}} \quad (\text{eqn.15})$$

Where: ρ_{H_2O} = Density of water at 25°C; 0.9970 g cm⁻³
 ρ_{CRT} = Measured density of cement at 25°C
 η_{H_2O} = Coefficient of Viscosity of water at 25°C; 0.8937 cP
 Δt_{H_2O} = Measured efflux time of the water ± 0.2 Seconds.
 Δt_{CRT} = Measured efflux time of the cement ± 0.2 Seconds.
 η_{CRT} = Coefficient of Viscosity of cement at 25°C; cP

Determination of Bulk Density of Freshly-Made, Uncured Grout
By gravimetric methods

SCOPE

The densities of various different mixtures of synthetic WRAP-2A waste, cement, fly ash and water a grout slurry will be determined by measuring the weight of a specific volume of grout at ambient temperature and pressure.

APPLICATION

This method is applicable for cementitious slurries and liquids including grouts made from the following WRAP-2A feed streams:

Type 2A-1	High Ammonium Nitrate
Type 2A-2	No Ammonium Nitrate
Type 1B	183-H Basins 3&4 Crystalline Solids
Type 1C	183-H Basins 3&4 Sludge
Type 1D-1	183-H Basins 1&2 Sludge/Sandblast Grit
Type 1D-2	183-H Basins 3&4 Sludge/Sandblast Grit
Type 7	Incinerator Ash
Type 2C	Metal Sludge

The slurries must be measured within 5 minutes after mixing.

EQUIPMENT

1. 2" Diameter X 4" high Cylindrical Specimen Molds
2. Analytical Balance, Digital, readable to the nearest 0.001g minimum.
3. Analytical Balance, Digital, readable to the nearest 0.1g
4. Plastic Transfer Pipet
5. Stainless Steel Spatula
6. Tamping Rod, $\frac{1}{8}$ " to $\frac{1}{4}$ " diameter and 6-7" long, plastic, stainless steel or plastic.
7. Vortex Mixer

PROCEDURE

1. Obtain a four 2" X 4" (206 ml nominal) specimen molds.
2. Allow a 1 liter container of deionized water to come to ambient temperature for one hour. Measure the temperature of the water to the nearest 0.1°C.
3. Take one mold, place it onto the three-place balance and zero the balance. Leave the specimen mold on the balance.

4. Fill the specimen mold with deionized water. If the meniscus of the water extends above the top rim of the container, remove the excess water using a plastic transfer pipet until the meniscus is nominally level with the rim. Be careful not to spill water onto the outside of the container or onto the balance pan. Record the "Mass_{H₂O}" to the nearest 0.001g in a laboratory notebook.
5. Calculate the volume of the cylinder mold (in cm³) using equation 16 and table 4.

$$Volume = \frac{Mass_{H_2O}}{density_{H_2O}} \quad (eqn.16)$$

Where:

Mass_{H₂O} = Mass of H₂O contained in the specimen mold, in g.
Density_{H₂O} = Density of water as derived from table 1., in g/cm³

Table 4. Density of Water as a Function of Temperature (g/cm³)

°C	Tenths of a Degree									
	.0	.1	.2	.3	.4	.5	.6	.7	.8	.9
21	0.997992	970	948	926	904	882	860	837	815	792
22	0.997770	747	724	701	678	655	632	608	585	561
23	0.997538	514	490	466	442	418	394	369	345	320
24	0.997296	271	246	221	196	171	146	120	095	069
25	0.997044	018	992	967	941	914	888	862	836	809
26	0.996783	756	729	703	676	649	621	594	567	540

6. Calculate the average volume of a specimen container. Record this volume in the laboratory notebook
7. Obtain the required number (6 per batch/formulation; 108 per feed stream) 2" X 4" specimen molds and affix the appropriate sample label with tape.
8. Tare weigh each of the labeled specimen molds to the nearest 0.1g and write the tare onto the bottom of the mold using a paint marker. Record the Mold Wt._{Tare} in a laboratory notebook. Do not include the weight of the cap.

9. Mix surrogate waste and cement according to the specimen preparation procedure.
10. Transfer the slurry into the specimen mold. Fill the mold to approximately $\frac{1}{2}$ full and vortex it on a vortex mixer for about 15-20 seconds to remove any entrapped air. Take care not to get any cement onto the outside of the specimen mold. Wipe off any cement that spilled onto the outside.
11. Add more cement slurry if necessary to (over) fill the mold and repeat step 10.
12. "Trowel" or blade off the top of the mold so that the cement is flush with the rim.
13. Place the filled specimen mold onto the 1 place electronic balance and weigh to the nearest 0.1g (remove the plastic protective skirt if one was used). Record the "Mold Wt._{full}" in a laboratory notebook. Do not include the weight of the cap.
14. Calculate the "wet" density of the cement (g/cm^3) slurry using equation 17.

$$\text{Density} = \frac{(\text{MoldWt.}_{\text{Full}} - \text{MoldWt.}_{\text{Tare}})}{\text{Volume}_{\text{mold}}} \quad (\text{eqn. 17})$$

Where:

- Mold Wt._{Full} = Total mass of the specimen mold + cement, without lid, in grams.
Mold Wt._{Tare} = Tare weight of the specimen mold without lid, in grams.
Volume_{CRT} = The calculated volume of the cylindrical specimen (eqn. 16), in cm^3 .

Determination of Density, Percent Drainable Liquid/Water
and Drainable Liquid pH for 28-Day Cured Grouts

SCOPE:

This method is used to determine the 28-day cured grout bulk density, the volume percent drainable liquid/water and the pH of drainable liquid at the surface of 28-day cured grouts.

APPLICATION:

This procedure applies for the determination of drainable liquid/water 28-day cured grouts prepared from various blends of simulated WRAP-2A feed types.

THEORY

The curing of grout gives rise almost always leads to some extent of cementitious volume shrinkage. This shrinkage sometimes forces excess liquid (containing salts associated with the cement) out of the cement. Several factors affect the amount of shrinkage of grout; settling, chemical bonding, etc.

MATERIALS

1. Plastic specimen mold, 2" diam. X 4" tall, ~206 mL.
2. Graduated cylinders 10 mL with 0.1 mL graduations.
3. Plastic disposable pipets 5 mL, graduated, Serological or Mohr type.
4. Freshly prepared grout mixture.

PROCEDURE:

1. Introduce the freshly prepared grout mixture into a 2" diameter X 4" high (~206 mL) plastic specimen mold that has been previously calibrated.
2. Cap the plastic mold tightly and tape around the cap to prevent the evaporation of bleed water.
3. Allow the sample to cure for 28 days inside of a sealed secondary container at ambient temperature.
4. After 28 days, pipet any drainable liquid/water at the surface of the grout into a the graduated pipet and record the volume of liquid to the nearest 0.05 ml (depending on the type of pipet used, it may be necessary to suck the total volume of the liquid up into the graduated portion of the pipet in order to obtain an accurate measurement).
5. Dispense the liquid into a 15-30 ml plastic beaker and screen/measure the pH using pH (litmus) paper by transferring a small drop of liquid onto pH = 0-14

range paper. DO NOT DIP THE PAPER INTO THE LIQUID.

6. Remeasure the pH using litmus paper with a more narrow pH range. Choose paper that is appropriate for the acidity range of the sample. Record the pH to the nearest ± 0.5 pH unit.
7. Remove the 28-day cured specimen from the cylinder mold and measure its length and diameter to the nearest 0.1 millimeters using a dial caliper. Divide diameter and height values by 10 to convert them to centimeter units and record them in the laboratory notebook.
8. Place the cement specimen onto an electronic balance and measure its weight in grams to the nearest 0.1g. Record the weight.

CALCULATIONS

1. Calculate the cured volume of the cylindrical cementitious specimen in cm^3 using equation 18.

$$\text{Volume}_{\text{CMT}} = \pi \left(\frac{D}{2} \right)^2 H \quad (\text{eqn. 18})$$

Where:

D= Measured diameter of cured cylindrical specimen, in cm.

H= Height of the cured cylindrical specimen, in cm.

$\pi = 3.14159$

2. Calculate the volume percent drainable liquid/water using equation 19.

$$\% \text{FreeLiquid} = \frac{\text{Volume}_{\text{H}_2\text{O}}}{(\text{Volume}_{\text{H}_2\text{O}} + \text{Volume}_{\text{CMT}})} \times (100) \quad (\text{eqn. 19})$$

Where:

$\text{Volume}_{\text{H}_2\text{O}}$ = The volume of liquid/water, in milliliters.

$\text{Volume}_{\text{CMT}}$ = The volume of the cured cylindrical specimen (eqn. 18), in cm^3 .

3. Calculate the dry density of the cement specimen in g/cm^3 using equation 20.

$$\rho_{\text{DRY}} = \frac{\text{Mass}_{\text{CMT}}}{\text{Volume}_{\text{CMT}}} \quad (\text{eqn.20})$$

Where:

Mass_{CMT} = The mass of the cured cylindrical specimen, in grams.
 $\text{Volume}_{\text{CMT}}$ = The measured volume of the cured cylindrical specimen
(eqn.18), in cm^3 .

Time of Setting of Cement-Based Grouts
by Penetration Resistance

SCOPE

This method⁹ describes the determination of the time of setting of cement-based grouts by means of penetration resistance.

APPLICATION

This procedure is applicable to nonradioactive grout formulations.

LIMITATION

1. The temperature of the air in the vicinity of the mixing laboratory, the dry solids blend and molds shall be maintained between 20°C and 27.5°C.
2. If the specimens are not cured in closed containers, the relative humidity of the laboratory should not be less than 50%. If used, The curing box/cabinet shall be so constructed as to provide storage facilities for test specimens at a relative humidity of not less than 90%.

APPARATUS AND MATERIALS

1. ACME penetrometer equipped with a secondary "memory" needle that moves with the indicating needle to the indicated pressure, then remains at that pressure until reset.
2. Specimen molds (plastic cylinders with a diameter of 3½ inches and minimum height of at least three inches), with screw-on lids.
3. Vibrating table or Vortex mixer.
4. Freshly prepared grout mixture.
5. Dial Calipers, standard

PROCEDURE:

1. Fill the specimen molds with the freshly prepared grout mixture up to about 1/2 Inch below the top edge of the molds and to a depth of at least two inches.
2. Compact the mixture by placing the molds on a vibrating table and vibrating the system for at least one minute.
3. Cover the molds with caps that fit the molds.
4. Store and maintain the specimens at room temperature (23°C) and allow them to set for 24 hours.

5. After exactly 24 hours, remove any bleed water from the grout surface using a plastic pipet, and save it in a suitable, closeable container to prevent evaporation (this will be returned to the top of the specimen after the penetrometer test is complete).
6. Insert proctor needle of the appropriate sizes (measure diameter with a dial caliper), depending upon the state of hardening of the grout (start with 1.132 in diameter), into the penetration resistance apparatus and bring the bearing surface of the needle into contact with the grout surface. The needle should be positioned at least one inch from the edge of the container, and the clear distance between needle impressions shall be at least two diameters of the needle being used, but not less than $\frac{1}{2}$ inch. Re-zero the pressure gauge's "memory" needle.
7. Gradually and uniformly apply a vertical force downward on the apparatus lever arm until the needle penetrates the grout to a depth of one inch as indicated by the scribe mark. The time to reach the one inch scribe mark should be ten seconds. If previous penetration tests have been performed on the specimen,
8. Record the lb force (lb_f) required (as indicated by the "memory" needle on the pressure gauge, proctor needle size, and the time of application, measured as elapsed time after initial contact of the dry-solids grouting materials with the simulated waste solution.
9. If the sample is reuseable, return bleed water, if any, to the grout surface, place the screw cap onto the mold, and return the specimen to its storage location.
10. Repeat steps 5 through 9 on another 24-hour specimen, taking care to perform the penetration at a distance at least two diameters of the needle being used, but not less than 0.5 inch from any previous penetration.
11. Repeat steps 5 through 9 at elapsed times 48, 72 and 96 hours, or until a hardness of $500 lb_f/in^2$ is reached for each specimen.
12. Record the time, date, ACME readings and needle size for each grout penetration resistance test.

CALCULATIONS

1. Calculate the Penetration Resistance (PR) in pounds force per square inch (psi) as the force required to cause a one-inch depth of penetration of the needle divided by the area of the bearing face of the needle (eqn. 21).

$$PR (lb_f/inch^2) = \frac{(lb_f)}{\frac{\pi D^2}{4}} \quad (eqn. 21)$$

Where: D = The Diameter of the needle, in inches
lb_f = The gauge reading, in lb_f

2. Convert the penetration resistance (PR) units of lb_f/in² to value to neutons per square meter (N/m²) using equation 22, and record this value.

$$PR (N/m^2) = PR (lb_f/inch^2) \times 6.897 \times 10^3 \quad (eqn. 22)$$

Compressive Strength of Cement-based Grouts

SCOPE

This method describes the determination of the compressive strength of cement-based grouts^{10,11,12}.

APPLICATION

This procedure is applicable to non-radioactive WRAP-2A waste surrogates mixed with cementitious material. This procedure is specific for use with a Cal-Cert® compression testing machine.

APPARATUS AND MATERIALS

1. 2" X 4" (diameter X height) right circular cylindrical cement specimens, cured the same number of days (± 1 day) from the mixing date (minimum of 28 days).
2. Cal-Teck Inc.® CT-Series , 400,000 Lb. capacity concrete testing machine fitted with a 30,000 lb capacity load cell, spacers for 2" X 4" specimens, and conforming to ASTM C39 specifications. Machine must be calibrated annually to ASTM, Practices E 4 and E 74 requirements.
3. Miter saw, Craftsman®, equipped with a masonry blade.
4. Plastic bags, 2 gallon capacity
5. Masking tape.
6. Shop Vacuum.
7. $\frac{1}{4}$ " thick X 3" diameter circular neoprene spacer caps
14. Carefully place the specimen in the testing machine below the center of the upper bearing block.

PROCEDURE

1. Remove any drainable liquid (if any). Record the volume (to the nearest 0.05 ml) measure the pH with litmus paper to the nearest 0.5 pH unit.
2. Remove the cement specimen from the mold by cutting the plastic mold using a plastic cutting "hot knife" which is made to cut through plastic. Cut the bottom off, then cut vertically down the side.
3. Weigh the specimen to the nearest 0.1g. record the weight.
4. Carefully inspect the specimen for irregularities (air pockets, cracks, graduation in color/texture). Record the findings for each specimen.

5. Photograph (or camcord) each sample (more than one specimen can be in a photo frame to save time and film).
6. If the top of the sample is not perpendicular to the sides of the sample¹, or is not plane, cut the top cross sectional end of the specimen off (if necessary) with the miter saw to render it both flat and perpendicular. DO NOT CUT THE SAMPLE ANY SHORTER THAN 3.6" IN LENGTH. LENGTH TO DIAMETER RATIO MUST BE ≥ 1.8
7. Measure the diameter and length twice at, midpoints, to the nearest 0.01 in (0.2mm), rotating the specimen approximately 90° so that an average determination can be made. Use a caliper directly readable to 0.001" (standard) or 0.02mm (metric). Record the dimensions as a decimal format (0.875", not 7/8"). Convert standard values to metric by the following conversion (eqn 23.).

$$\text{Centimeters} = \text{inches} \times 2.540 \quad (\text{eqn.23})$$

8. Place the lower bearing block (hardened face up) and the 2" X 4" specimen spacer directly under the upper bearing block.
9. Wipe the bearing faces clean.
10. Cut a hole in a plastic bag the diameter of the spacer, and place the plastic bag over the spacer to collect fragments of specimen that fall off during compression. These fragments will be saved for TCLP analysis.
11. Carefully align the vertical axis of the specimen with the center of the upper block. Use the concentric rings on the lower bearing block (platten) as a guide.
12. Center a 1/2" thick neoprene spacer on top of the specimen. This spacer will serve to even out the load for specimens with a slightly uneven upper surface.
13. Turn on the compression machine power and ensure that the digital readout is set to kgs.

¹ Ensure that the top and bottom surfaces are perpendicular ($\Delta = 90^\circ$) to the sides by "squaring-up" the top/bottom of the specimen with the jaws of the caliper or using a steel "T" square (its long end oriented along the side of the specimen) and estimating the gap (if any) between the side of the specimen and the edge of the "T" square. Note any "non-perpendicular", greater than $> 1/16$ " gap between specimen side and "T" square) specimens.

14. Apply the load continuously and without shock, at a maximum rate of 28 kg to 71 kg/sec (63 Lb - 157 Lb/sec) for a 2" diameter cylindrical sample. sample). DO NOT ADJUST THE RATE OF MOVEMENT AT ANY TIME AFTER THE FIRST HALF OF THE ANTICIPATED LOADING HAS BEEN REACHED.
15. Apply the load until the specimen fails, and record the maximum load (L_{Max}) carried by the specimen during the test. Record the type of load and the appearance of the cement.
16. Collect 125 g of the broken specimen and set aside for TCLP if applicable.
17. Remove the plastic bag containing pieces of specimen and vacuum the work area.
18. Repeat steps 9-18 for each specimen.

CALCULATIONS

1. Calculate the cross sectional surface area of the specimen in square meters (eqn. 24).

$$Area = \frac{\pi D^2}{4} \times \frac{m^2}{10^4 cm^2} \quad (eqn. 24)$$

Where:

- $\pi = 3.1415$
 $D =$ The diameter of the specimen, in cm
 $Area =$ The cross sectional area of the sample in square meters.

2. Calculate the compressive strength of the specimen by dividing the maximum load by the cross-sectional area. Express the result (eqn. 25) in kilopascals (1 kPa = kN/m²).

$$CS = \frac{(L_{Max})(g_c)}{Area} \times \frac{kN}{1000N} \quad (eqn. 25)$$

Where:

- $L_{Max} =$ The maximum load applied to failure, in kg.
 $g_c =$ The gravitational constant (9.8 m/s²)
 $Area =$ The cross sectional area of the specimen, in m²
 $CS =$ The compressive strength, in kPa

ANSI 16.1 Leach Test

1.0 SCOPE

This procedure describes how to conduct the American Nuclear Society (ANSI) 16.1 short-term leach testing procedure¹³. This procedure is intended to provide controlled and easily attainable conditions for the comparative evaluation of waste retention in waste forms. Subjects covered by this procedure include sample preparation, calculation of leachate volume, preparation of leach vessels, sampling procedure, and description of related calculations.

This procedure does not deal with the reduction of data from the chemical analysis of leachates because this is covered in the source ANN 16.1 procedure. This procedure only describes the specific practices used in the Chemical engineering laboratory to accomplish the physical test requirements of ANN 16.1. Minor schedule changes have been incorporated into this procedure to accommodate the work week and to reduce analytical costs. This includes the requirement to start the initial leach on a Monday morning. These changes do not affect the necessary boundary conditions of the standard procedure.

Other modifications to the procedure include:

- °The elimination of the initial 30 sec rinse of the sample (p. 5).
- °Analyses of bottle rinses are not conducted (p. 6).
- °Filter media are discarded without being analyzed (p. 6).

2.0 DEFINITIONS

- 2.1 Trained Operator: Staff member trained in the conduct of this procedure. Training will be documented in Task files.
- 2.2 Leach Test: Experiment in which a sample of known surface area is submerged in a fluid to determine the rate at which various chemical species in the sample are dissolved.

3.0 EQUIPMENT

- 3.1.1 Caliper (Accuracy: ± 0.001 ")
- 3.1.2 Balance (Accuracy: ± 0.1 g)
- 3.1.3 Polypropylene Wide Mouth Bottles -Two bottles are required for each sample. Two bottles allow the sample to be placed in a cleaned bottle with fresh leachant while leachate from the other bottle is being sampled. Following sampling the bottle is cleaned in preparation for the next changeout according to the procedure below. Clean bottles prior to tests by filling with Micro® detergent diluted with de-ionized water (DIW) and allow to soak overnight. Rinse the bottles with DIW at least three times. Between leaching intervals, rinse used bottles

three times with DIW then fill the bottle with DIW above the normal leachate level and allow it to sit until the next bottle changeout. At changeout, pour this DIW out and refill with fresh DIW leachant.

- 3.1.4 Nylon Monofilament - Tie monofilament into a loop then slip loop around the sample and tighten the knot securely so that the sample may be suspended from a hole in the cap of the leaching bottle.
- 3.1.5 Scintillation vials - Rinse each vial, at least once, with a small amount of filtered leachate before filling it with an aliquot of the filtered leachate for analysis (see 3.1.6).
- 3.1.6 Syringe and 0.45 μm syringe filters. - Label one syringe for each sample leached. This syringe will be rinsed and reused throughout the test for filtering leachates from this sample only. In preparation for sampling, each syringe should be rinsed well with the leachate to be filtered as follows: Pour leachate into the syringe and replace the plunger. Shake the syringe to make sure that the plunger tip is rinsed. Squeeze out the remaining rinse leachate. Remove the plunger and place a filter on end of the syringe. Pour leachate into the syringe, replace the plunger and rinse the filter and sample vials (see 3.1.5) by filtering a minimum of 5 mL of leachate into each of the sample vials. Syringes should be rinsed with DIW and allowed to dry between intervals.
- 3.1.7 pH Meter (Follow Instruction Manual). Calibrate using pH 7 and 10 buffered standards, record calibration slope and temperature (see Exhibit 1).
- 3.1.8 Thermometer (Accuracy: $\pm 0.2^{\circ}\text{C}$)
- 3.1.9 Cured cementitious waste surrogate specimen, cut to 2.4 cm X 3.0 cm right circular cylinders (diam. X length).

4.0 PROCEDURE

4.1 PRECAUTIONS

- 4.1.1 Leachate solution pH can approach 12.5. Appropriate eye protection must be worn when processing samples.
- 4.1.2 Avoid exposure to skin, and sample contamination by wearing gloves.
- 4.1.3 Do not dispose of leachate down the drain unless it is non-hazardous, i. e. $\text{pH} \leq 12.5$ and contains no regulated toxic chemicals above permissible limits (reference TCLP procedure).

4.2 INSTRUCTIONS

- 4.2.1 Record all data onto data sheets and paste them into the appropriate laboratory notebook for the feed type under examination. Ensure that all balances are in calibration. Record measurements and the results of calculations as they are called out in the procedure below.
- 4.2.2 Cut samples into right circular cylinders. Lightly wipe the samples with a soft-bristled brush to remove any loose material that may be left on the samples.
- 4.2.3 Measure diameters and lengths twice, rotating samples approximately 90° so that average determinations can be made.
- 4.2.4 Calculate the amount of leachant by determining the surface area of the sample and multiplying by 10. Calculation of surface area is found in section 4.3
- 4.2.5 Fill a leach bottle with the calculated volume of DIW.
- 4.2.6 Suspend the sample near the center of the DIW-filled leaching bottle using monofilament threaded through a hole in the lid. Record the test start time.
- 4.2.7 The leachant will be changed out as described in the "Elapsed Time" table below. When a changeout period is near the replacement bottle should be rinsed and filled with fresh DIW leachant. Changeout is accomplished simply by removing the lid, with the suspended sample, from the first bottle and transferring it to the bottle containing the fresh leachant.

Table 5. Total Elapsed Time at Leachant Changeout

Leach	Modified ANSI 16.1	
Interval	Schedule	Schedule
int 0	0 h	30 s
int 1	7 h	2 h
int 2	1 d	7 h
int 3	2 d	1 d
int 4	4 d	2 d
int 5	7 d	3 d
int 6	14 d	4 d
int 7	28 d	5 d
int 8	56 d	19 d
int 9	91 d	47 d
int 10	----	90 d

- 4.2.8 Measure pH of the leachate by pouring it directly into a pH measuring cup. Rinse the electrode with the sample in the cup and then dump it out. Repeat this rinse two more times. Refill the cup and place a small stirring bar in the cup and record the pH (to the nearest 0.1 unit) when it has reached its maximum stable reading.
- 4.2.9 Rinse the syringe, syringe filter, and sample vials as described in 3.1.6. Fill the vials with filtered leachate and label the vial with traceable identification.
- 4.2.10 Submit samples for analysis and retain at least one sample as an archive.
- 4.2.11 Record the temperature of the solutions to the nearest 1°C.

4.3 CALCULATIONS

- 4.3.1 Surface Area = $2(\pi r^2) + 2\pi rh$
- 4.3.2 Volume = $\pi r^2 h$
where: r = radius of sample, cm
h = height of sample, cm

4.4 RECORDING OF DATA AND RESULTS

- 4.4.1 Whenever possible, data should be recorded in a controlled laboratory notebook. Make entries so that analytical records for leachate analyses are traceable. Data are input to a spreadsheet template "ANSIDAT" for reducing data. The calculations performed by this spreadsheet will be verified by hand calculation.

pH of Solids by Electrometric Method

SCOPE

The pH of solids is measured by USEPA method 9045, Soil pH. The solid sample is mixed with deionized water, and the pH of the slurry is then measured with a pH meter^{14,15}.

APPLICATION

This procedure will be used on cured grout products, and on ungrouted, surrogate waste to determine the corrosivity of the material.

MATERIALS

1. pH meter with means for temperature compensation.
2. Combination electrode.
3. 30-50 ml plastic beaker
4. Squeeze bottle filled with deionized water
5. Secondary Standard Buffer salts: pH = 4.00; pH = 7.00; pH = 10.00
6. Magnetic stirrer
7. Teflon or glass coated stir bar

PROCEDURE

1. Assemble the pH electrode/pH metering system according to the manufacturer's operating manual.
2. Choose two buffers to for calibrating the pH system that will cover the expected pH range of the sample being measured, and are a minimum of three pH units apart (Alternatively, one can pre-mix the solid and water and place a drop of the liquid from the mixture onto litmus paper to determine the approximate pH of the solution/slurry.).
3. Calibrate the electrode for the two buffers chosen in step 2, following the pH probe/meter manufacturer's operating instructions. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer value. Note the temperature of the buffer solution. record the slope of the calibration.
3. Weigh out duplicate, 20.0 gram aliquots of solid into individual plastic beakers, add 20.0 grams of deionized water to each and stir the suspensions for 30 minutes.
4. Allow the slurries to come to room temperature.
5. Adjust the electrode into its holder so that upon lowering into a sample, the electrode will be immersed just below the suspension.
6. Record the pH and temperature of the sample and its duplicate.

7. Rinse the pH probe and thermometer/thermocouple.
8. Repeat steps 3-6 for each sample. Perform calibration checks every 20 measurements or every hour. Recalibrate as necessary.

pH of liquids by Electrometric Method

SCOPE

The pH of liquids is measured by USEPA method 9040, pH Electrometric Measurement. The pH of an aqueous sample is measured with a pH probe/meter^{14,16}.

APPLICATION

This procedure will be used on aqueous solutions resulting from 90-day water leaches, and ANSI 16.1 Test leaches of cured cementitious specimens.

MATERIALS

1. pH meter with means for temperature compensation. (a low sodium error electrode is suggested at pH levels > 10.)
2. Combination electrode.
3. 30-50 ml plastic beaker
4. Squeeze bottle filled with deionized water
5. Secondary Standard Buffer salts: pH = 4.00; pH = 7.00; pH = 10.00
6. Magnetic stirrer
7. Teflon or glass coated stir bar

PROCEDURE

1. Assemble the pH electrode/pH metering system according to the manufacturer's operating manual.
2. Choose two buffers to for calibrating the pH system that will cover the expected pH range of the sample being measured, and are a minimum of three pH units apart (Alternatively, one can pre-mix the solid and water and place a drop of the liquid from the mixture onto litmus paper to determine the approximate pH of the solution/slurry.).
3. Calibrate the electrode for the two buffers chosen in step 2, following the pH probe/meter manufacturer's operating instructions. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer value. Note the temperature of the buffer solution. Record the slope of the calibration.
4. Place about 10 ml of the liquid sample into a clean plastic beaker, ensuring that the sensing portion of the electrode is immersed. Give adequate clearance for the stir bar to rotate.
5. Record the pH and temperature of the sample.
6. Rinse the pH probe and thermometer/thermocouple
7. Repeat steps 4-5 for each sample. Perform interim calibration checks every 20 measurements or every hour. Recalibrate as necessary.

Thermocycling of Cementitious Specimens

SCOPE

This method covers the thermal cycling procedure and apparatus used to test cementitious waste forms for evaluation of thermal degradation. The thermocycle test¹⁷ covers conditions which simulate environmental extremes to which waste forms may be subjected, either in shipping or use.

APPLICATION

This procedure is based on ASTM B 553, Sections 5.4.1 through 5.4.4. Solidified waste samples are exposed to 30 cycles of temperature extremes, from 60°C (140°F) to -40°C (-40°F), equilibrating for one hour at each temperature. Two, 4-hour cycles will be completed each day, allowing the specimens to equilibrate 16 hours at ambient temperature before initiating another set of cycles. Samples will be cycled during normal business hours, and will remain at ambient temperature during off hours and weekends. Wastes subject to this test must have cured for 28 ± 1 days, and must be removed from their curing molds. Each cycle will be scheduled as follows:

Table 6. Typical Schedule For Thermocycling Tests.

Day	60°C	22°C	40°C	22°C	60°C	22°C	-40°C	22°C
1. Mon.	1	1	1	1	1	1	1	17
2. Tues.	1	1	1	1	1	1	1	17
3. Wed.	1	1	1	1	1	1	1	17
4. Thur.	1	1	1	1	1	1	1	17
5. Fri.	1	1	1	1	1	1	1	17
6. Sat.	---	---	---	---	---	---	---	24
7. Sun.	---	---	---	---	---	---	---	24
8. Mon.	1	1	1	1	1	1	1	17
9. Tues.	1	1	1	1	1	1	1	17
10. Wed.	1	1	1	1	1	1	1	17
11. Thur.	1	1	1	1	1	1	1	17
12. Fri.	1	1	1	1	1	1	1	17
13. Sat.	---	---	---	---	---	---	---	24
14. Sun.	---	---	---	---	---	---	---	24
15. Mon.	1	1	1	1	1	1	1	17
16. Tues.	1	1	1	1	1	1	1	17
17. Wed.	1	1	1	1	1	1	1	17
18. Thur.	1	1	1	1	1	1	1	17
19. Fri.	1	1	1	1	1	1	1	17

APPARATUS

1. Circulating air heating chamber (oven), insulated and controlled to maintain a preset temperature $\pm 3^{\circ}\text{C}$, and equipped with temperature readout accurate $\pm 1^{\circ}\text{C}$.
2. Cooling chamber (freezer), insulated and controlled to maintain a preset temperature $\pm 3^{\circ}\text{C}$, and equipped with temperature readout accurate $\pm 1^{\circ}\text{C}$.

PROCEDURE

1. Turn on the oven. Set the temperature to 60°C (140°F). Monitor with a thermocouple probe through the external air vent.
2. Turn on the freezer. Set the temperature to -40°C (-40°F)
3. Ensure chambers are at their set temperatures.
4. Place the samples into the heating chamber. Expose the specimens for 1 hour.
5. Remove the specimens from the oven and allow them to return to ambient temperature ($22 \pm 3^{\circ}\text{C}$).
6. Place the specimens into the cooling chamber. Expose the specimens for 1 hour.
7. Remove the specimens from the cooling chamber and let them return to ambient temperature ($22 \pm 3^{\circ}\text{C}$).
8. This constitutes one complete thermal cycle.
9. Repeat steps 5-9 for a total of 30 cycles (perform two per day).
10. Perform compressive strength test on specimen after 30 thermocycles.

90-Day Water Immersion

SCOPE

This method covers the Water Submersion procedure and apparatus used to test cementitious waste forms for evaluation of hydrological degradation. The Immersion test covers conditions which simulate environmental elements to which waste forms may be subjected, either in shipping, storage or use.

APPLICATION

Solidified waste samples are submersed in deionized water for a period of 90 days, after which time they are removed from the water and examined for compressive strength. Wastes subject to this test must have cured for a minimum of 28 days, and must be removed from their curing molds.

APPARATUS

1. Deionized water
2. 2" X 4" (diam X height) cementitious waste specimen
3. 500 ml (16 oz) glass jar with screw-cap lid.
4. Graduated cylinder, 250 ml

PROCEDURE

1. Obtain and label a 500 ml (16 oz) glass jar for each specimen (re-use the outer jar that was used during the curing step). The label should show the sample identification and the date of immersion.
2. Remove the cement specimen from its plastic curing container and place it into the glass, 500 ml jar.
3. Measure 250 milliliters of water with a graduated cylinder and transfer the water to the glass jar.
4. Screw the lid onto the jar and seal around the lid with either parafilm or teflon tape.
5. Immersed specimen for 90 ± 2 days, then remove the specimen from the jar, blot it dry with a paper towel. Immediately (90 ± 2 days from beginning of leach) perform the compressive strength test on the specimen.
6. Measure the pH of the water leachate in the jar with a pH electrode. Record this value.

TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

1.0 SCOPE AND APPLICATION

- 1.1 The TCLP¹⁸ is designed to determine the mobility of inorganic analytes present in solid wastes as defined in section(s) 7.1.1 through 7.1.4 of USEPA SW-846 Method 1311, TOXICITY CHARACTERISTIC LEACHING PROCEDURE. The following simulated waste streams Samples will grouted with cement, cured, crushed and analyzed for TCLP metals.

Table 7. Samples Scheduled for TCLP Extraction.

Stream 2A1	Stream 2A2	Stream 1B	Stream 1C	Stream 1D1	Stream 1D2	Stream 7
2A1-03-A-1	2A2-03-A-1	1B-03-A-1	1C-03-A-1	1D1-03-A-1	1D2-03-A-1	1D2-03-A-1
2A1-05-A-1	2A2-05-A-1	1B-05-A-1	1C-05-A-1	1D1-05-A-1	1D2-05-A-1	1D2-05-A-1
2A1-07-A-1	2A2-07-A-1	1B-07-A-1	1C-07-A-1	1D1-07-A-1	1D2-07-A-1	1D2-07-A-1
2A1-08-A-1	2A2-08-A-1	1B-08-A-1	1C-08-A-1	1D1-08-A-1	1D2-08-A-1	1D2-08-A-1
2A1-09-A-1	2A2-09-A-1	1B-09-A-1	1C-09-A-1	1D1-09-A-1	1D2-09-A-1	1D2-09-A-1
Matrix Spike	Matrix Spike	Matrix Spike	Matrix Spike	Matrix Spike	Matrix Spike	Matrix Spike

- 1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.
- 1.3 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

2.0 SUMMARY OF METHOD

- 2.1 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatile analytes (see Table 1 for a list of volatile compounds). Following extraction, the liquid extract is separated

from the solid phase by filtration through a 0.6 to 0.8 μ m glass fiber filter.

3.0 INTERFERENCES

- 3.1 Potential interferences that may be encountered during analysis are discussed in the analytical method used for the specific analyte.

4.0 APPARATUS AND MATERIALS

- 4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion (see Figure 1) at 30 ± 2 rpm.
- 4.2 Borosilicate Glass Extraction Bottle - with sufficient capacity to hold the sample and the extraction fluid, designed to fit the agitation apparatus. Headspace is allowed in this vessel.
- 4.3. Filter Holder - capable of supporting a 47 mm glass fiber filter. Must have a minimum internal volume of 300 mL and able to withstand the pressure needed to accomplish separation (~50 - 70 psi).
- 4.4 Filters - Borosilicate glass fiber, no binder, effective pore size of 0.6 to 0.8 μ m. Filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended).
- 4.5 pH Meter - accurate to ± 0.05 units at 25°C.
- 4.6 Laboratory Balance - accurate to within ± 0.01 grams.
- 4.7 Beaker or Erlenmeyer flask, glass, 500 mL.
- 4.8 Watchglass, appropriate diameter to cover beaker or Erlenmeyer flask.
- 4.9 Magnetic stirrer.

5.0 REAGENTS

- 5.1 ACS Reagent grade chemicals shall be used in all tests. Unless otherwise indicated.
- 5.2 Deionized Reagent Water, 17 Mohm.
- 5.3 Hydrochloric acid (1N), HCl, made from ACS reagent-grade.
- 5.4 Nitric acid (1N), HNO₃, made from ACS reagent grade.
- 5.5 Sodium hydroxide (1N), NaOH, made from ACS reagent grade.
- 5.6 Glacial acetic acid, CH₃COOH, ACS reagent grade.
- 5.7 Extraction fluid.
 - 5.7.1 Extraction fluid # 1 - Add 5.7 mL glacial CH₃COOH to 500 mL of reagent water, add 64.3 mL of 1N NaOH, and dilute to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 + 0.05.
 - 5.7.2 Extraction fluid # 2: Dilute 5.7 mL glacial CH₃COOH with reagent water to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 + 0.05.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

- 6.1 Preservatives shall not be added to samples before extraction.
- 6.2 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Section 7.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. See Section 8.0 (QA requirements) for acceptable sample and extract holding times.

7.0 PROCEDURE

- 7.1 Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include:
 - (1) Determination of whether the solid portion of the waste requires particle size reduction (Section 7.1.3).
 - (2) Determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 7.1.4).

7.1.1 Particle size reduction is required if the sample doesn't have a surface area per gram of material equal to or greater than 3.1 cm²/g, or is smaller than 1 cm in its narrowest dimension (i.e., is not capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above.

7.1.4 Determination of appropriate extraction fluid:

7.1.4.1 Weigh out 5.0 g of the solid phase of the size-reduced sample and transfer it to a 500 mL beaker or Erlenmeyer flask.

7.1.4.2 Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1.

7.1.4.3 If the pH from Section 7.1.4.2 is >5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watchglass, heat to 50°C, and hold at 50°C for 10 minutes.

7.1.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2.

7.2 18 Hour Extraction

7.2.1 Prepare a 120 grams of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.1.3.

7.2.2 Transfer 100.0g of the solid material into an extractor bottle.

Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon coated sieve should be used to avoid contamination of the sample.

- 7.2.2 Determine the amount of extraction fluid to add to the extractor vessel as follows:

Weight of extraction fluid = 20 x Weight of waste

- 7.2.3 Slowly add the appropriate extraction fluid to the extractor vessel, replace the cap and seal it with either parafilm or Teflon tape.

- 7.2.4 Secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours at $22 \pm 3^\circ\text{C}$.

- 7.2.5 Relieve pressure buildup in the extraction bottle after a periods of 15, 30, and 60 minutes. Vent into a hood.

7.3 Filtration Following 18 hr. Extraction.

- 7.3.1 Assemble the filter holder and filter following the manufacturer's directions. Secure the filter onto the support screen.

- 7.3.2 Gradually apply vacuum or gentle pressure of 1-10 psi until air moves through the filter. Separate the material into its liquid and solid phases. If necessary, slowly increase in 10 psi increments at approximately 2 minute intervals to a maximum of 50 psi. The glass fiber filter may be changed, if necessary, to facilitate filtration.

7.4 Extract Preparation Following Extract Filtration

- 7.4.1 The pH of the extract is measured and recorded using a pH meter.

- 7.4.2 Aliquot 100 ml of the extract into a 150 ml beaker and add 100 μL of TCLP Matrix Spike Solution to the appropriate samples. Stir for 5 min.

- 7.4.3 Acidify the extract² (metal analysis preservation only) to a pH < 2 using 0.1N nitric acid.

- 7.4.4 Aliquot 100 ml of the preserved extract into clean ICHEM® bottles, and ship to the appropriate laboratory for Acid Digestion, followed by TCLP metals analyses by ICP/AA or ICPMS.

²

If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible.

8.0 QUALITY CONTROL

- 8.1 Prepare one acetic acid blank (using the same extraction fluid as used for the samples) for every 20 extractions that have been conducted in an extraction vessel.
- 8.2 The matrix spike shall be performed for each waste type (2A1, 2A2, 1C, 1B, 1D1, 1D2, and Type 7).
- 8.2.1 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.
- 8.2.2 Matrix spikes should be added such that the metals are at a concentration of 5 times the Method Detection Limit (MDL). The MDL is 10 times the instrument detection limit (IDL) (assuming a 1:10 dilution factor) due to the acid digestion step. The net result is that the concentration of metals in the spiked sample should be 50 IDL. Assuming the IDL = 100 $\mu\text{g/L}$ (ppb), The concentration of metals in the spiked sample should be 5000 $\mu\text{g/L}$ (ppb) or 5 $\mu\text{g/ml}$ (ppm).
- 8.2.3 The Matrix spike solution will become diluted by a factor of 1000 upon addition of 100 μl spike to 100 ml sample. The concentration of metals in the Spike Solution, therefore, should be prepared at a concentration of 5000 $\mu\text{g/ml}$.
- 8.3 Samples must undergo TCLP extraction within the following time periods:

Table 8. TCLP Sample Maximum Holding Times (days)

	From: Field Collection TO: TCLP Extraction	From: TCLP Extraction TO: Analysis	Total Elapsed Time
ICP (Ag, Ba, Cd, Cr, Pb)	180	180	360
AA (As, Se)	180	180	360
AA (Hg)	28	28	56

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

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WHC-SD-W100-TP-011 REV. 0
APPENDIX A

Table 1. Feed Stream 1C Specimen Preparation Worksheet

Total Batch 5000
Amount

Order	Ident.	Masonry Cement	Fly Ash	Waste	Grit	Water	Total
1	1C-01	1697.4	712.9	1425.0	0.0	1164.7	5000.0
2	1C-02	2373.5	0.0	1558.4	0.0	1068.1	5000.0
3	1C-03	1000.5	999.0	2000.0	0.0	1000.6	5000.1
4	1C-04	1700.0	1450.0	1000.0	0.0	850.0	5000.0
5	1C-05	1075.0	1075.0	2000.0	0.0	850.0	5000.0
6	1C-06	2154.5	876.0	1000.0	0.0	969.6	5000.1
7	1C-07	1685.0	0.0	2000.0	0.0	1315.0	5000.0
8	1C-08	2057.0	17.50	2000.0	0.0	925.6	5000.1
9	1C-09	1000.5	999.0	2000.0	0.0	1000.6	5000.1
10	1C-10	1333.5	1333.5	1000.0	0.0	1333.4	5000.4
11	1C-11	1769.5	616.0	1400.0	0.0	1214.7	5000.2
12	1C-12	2154.5	876.0	1000.0	0.0	969.6	5000.1
13	1C-13	2350.0	0.0	1000.0	0.0	1650.0	5000.0
14	1C-14	1390.5	1390.5	1368.6	0.0	850.0	4999.6
15	1C-15	1650.0	0.0	1700.0	0.0	1650.0	5000.0
16	1C-16	1650.0	700.0	1000.0	0.0	1650.0	5000.0
17	1C-17	2758.5	0.0	1000.0	0.0	1241.4	4999.9
18	1C-18	1700.0	1450.0	1000.0	0.0	850.0	5000.0
	Total	31499.9	12495.4	25452.0	0.0	20553.0	90000.3

WHC-SD-W100-TP-011 REV. 0
APPENDIX A

Table 4. Feed Stream 1B Specimen Preparation Worksheet

Total Batch 5000
Amount

Order	Ident.	Cement	Fly Ash	Waste	Grit	Water	Total
1	1B-01	1697.4	712.9	1425.0	0.0	1164.7	5000.0
2	1B-02	2373.5	0.0	1558.4	0.0	1068.1	5000.0
3	1B-03	1000.5	999.0	2000.0	0.0	1000.6	5000.1
4	1B-04	1700.0	1450.0	1000.0	0.0	850.0	5000.0
5	1B-05	1075.0	1075.0	2000.0	0.0	850.0	5000.0
6	1B-06	2154.5	876.0	1000.0	0.0	969.6	5000.1
7	1B-07	1685.0	0.0	2000.0	0.0	1315.0	5000.0
8	1B-08	2057.0	17.50	2000.0	0.0	925.6	5000.1
9	1B-09	1000.5	999.0	2000.0	0.0	1000.6	5000.1
10	1B-10	1333.5	1333.5	1000.0	0.0	1333.4	5000.4
11	1B-11	1769.5	616.0	1400.0	0.0	1214.7	5000.2
12	1B-12	2154.5	876.0	1000.0	0.0	969.6	5000.1
13	1B-13	2350.0	0.0	1000.0	0.0	1650.0	5000.0
14	1B-14	1390.5	1390.5	1368.6	0.0	850.0	4999.6
15	1B-15	1650.0	0.0	1700.0	0.0	1650.0	5000.0
16	1B-16	1650.0	700.0	1000.0	0.0	1650.0	5000.0
17	1B-17	2758.5	0.0	1000.0	0.0	1241.4	4999.9
18	1B-18	1700.0	1450.0	1000.0	0.0	850.0	5000.0
	Total	31499.9	12495.4	25452.0	0.0	20553.0	90000.3

WHC-SD-W100-TP-011 REV. 0
APPENDIX A

Table 5. Feed Stream 1D-1 Specimen Preparation Worksheet

Total Batch 5000
Amount

Order	Ident.	Cement	Fly Ash	Waste	Grit	Water	Total
1	1D1-01	1360.5	421.9	232.5	2092.1	892.9	4999.8
2	1D1-02	687.5	687.4	331.6	2984.4	309.4	5000.3
3	1D1-03	333.5	333.3	400.0	3600.0	333.4	5000.2
4	1D1-04	1000.0	1000.0	200.0	1800.0	1000.0	5000.0
5	1D1-05	2069.0	0.0	200.0	1800.0	931.1	5000.1
6	1D1-06	1203.0	1050.0	220.6	1985.0	541.4	4999.9
7	1D1-07	1250.0	500.0	200.0	1800.0	1250.0	5000.0
8	1D1-08	997.9	431.0	287.8	2590.3	693.0	5000.0
9	1D1-09	1365.0	0.0	302.1	2718.9	614.2	5000.2
10	1D1-10	639.5	639.3	308.2	2773.8	639.3	5000.1
11	1D1-11	847.5	0.0	330.6	2975.0	847.4	5000.4
12	1D1-12	1628.0	639.8	200.0	1800.0	732.5	5000.3
13	1D1-13	687.5	687.4	331.6	2984.4	309.4	5000.3
14	1D1-14	689.5	0.0	400.0	3600.0	310.4	4999.9
15	1D1-15	1000.0	1000.0	200.0	1800.0	1000.0	5000.0
16	1D1-16	1761.5	0.0	200.0	1800.0	1238.6	5000.1
17	1D1-17	1250.0	0.0	250.0	2250.0	1250.0	5000.0
18	1D1-18	1203.0	1050.0	220.6	1985.0	541.4	4999.9
	Total	19972.9	8440.0	4815.4	43338.7	13433.9	90000.9

WHC-SD-W100-TP-011 REV. 0
APPENDIX A

Table 6. Feed Stream 1D-2 Specimen Preparation Worksheet

Total Batch 5000
Amount

Order	Ident.	Cement	Fly Ash	Waste	Grit	Water	Total
1	1D2-01	1360.5	421.9	232.5	2092.1	892.9	4999.8
2	1D2-02	687.5	687.4	331.6	2984.4	309.4	5000.3
3	1D2-03	333.5	333.3	400.0	3600.0	333.4	5000.2
4	1D2-04	1000.0	1000.0	200.0	1800.0	1000.0	5000.0
5	1D2-05	2069.0	0.0	200.0	1800.0	931.1	5000.1
6	1D2-06	1203.0	1050.0	220.6	1985.0	541.4	4999.9
7	1D2-07	1250.0	500.0	200.0	1800.0	1250.0	5000.0
8	1D2-08	997.9	431.0	287.8	2590.3	693.0	5000.0
9	1D2-09	1365.0	0.0	302.1	2718.9	614.2	5000.2
10	1D2-10	639.5	639.3	308.2	2773.8	639.3	5000.1
11	1D2-11	847.5	0.0	330.6	2975.0	847.4	5000.4
12	1D2-12	1628.0	639.8	200.0	1800.0	732.5	5000.3
13	1D2-13	687.5	687.4	331.6	2984.4	309.4	5000.3
14	1D2-14	689.5	0.0	400.0	3600.0	310.4	4999.9
15	1D2-15	1000.0	1000.0	200.0	1800.0	1000.0	5000.0
16	1D2-16	1761.5	0.0	200.0	1800.0	1238.6	5000.1
17	1D2-17	1250.0	0.0	250.0	2250.0	1250.0	5000.0
18	1D2-18	1203.0	1050.0	220.6	1985.0	541.4	4999.9
	Total	19972.9	8440.0	4815.4	43338.7	13433.9	90000.9

WHC-SD-W100-TP-011 REV. 0
APPENDIX A

Table 7. Feed Stream 2A-1 Specimen Preparation Worksheet

Total Batch 5000
Amount

Order	Ident.	Cement	Fly Ash	Waste	Grit	Water	Total
1	2A1-01	1697.4	712.9	1425.0	0.0	1164.7	5000.0
2	2A1-02	2373.5	0.0	1558.4	0.0	1068.1	5000.0
3	2A1-03	1000.5	999.0	2000.0	0.0	1000.6	5000.1
4	2A1-04	1700.0	1450.0	1000.0	0.0	850.0	5000.0
5	2A1-05	1075.0	1075.0	2000.0	0.0	850.0	5000.0
6	2A1-06	2154.5	876.0	1000.0	0.0	969.6	5000.1
7	2A1-07	1685.0	0.0	2000.0	0.0	1315.0	5000.0
8	2A1-08	2057.0	17.50	2000.0	0.0	925.6	5000.1
9	2A1-09	1000.5	999.0	2000.0	0.0	1000.6	5000.1
10	2A1-10	1333.5	1333.5	1000.0	0.0	1333.4	5000.4
11	2A1-11	1769.5	616.0	1400.0	0.0	1214.7	5000.2
12	2A1-12	2154.5	876.0	1000.0	0.0	969.6	5000.1
13	2A1-13	2350.0	0.0	1000.0	0.0	1650.0	5000.0
14	2A1-14	1390.5	1390.5	1368.6	0.0	850.0	4999.6
15	2A1-15	1650.0	0.0	1700.0	0.0	1650.0	5000.0
16	2A1-16	1650.0	700.0	1000.0	0.0	1650.0	5000.0
17	2A1-17	2758.5	0.0	1000.0	0.0	1241.4	4999.9
18	2A1-18	1700.0	1450.0	1000.0	0.0	850.0	5000.0
	Total	31499.9	12495.4	25452.0	0.0	20553.0	90000.3

WHC-SD-W100-TP-011 REV. 0
APPENDIX A

Table 8. Feed Stream 2A-2 Specimen Preparation Worksheet

Total Batch 5000
Amount

Order	Ident.	Cement	Fly Ash	Waste	Grit	Water	Total
1	2A2-01	1697.4	712.9	1425.0	0.0	1164.7	5000.0
2	2A2-02	2373.5	0.0	1558.4	0.0	1068.1	5000.0
3	2A2-03	1000.5	999.0	2000.0	0.0	1000.6	5000.1
4	2A2-04	1700.0	1450.0	1000.0	0.0	850.0	5000.0
5	2A2-05	1075.0	1075.0	2000.0	0.0	850.0	5000.0
6	2A2-06	2154.5	876.0	1000.0	0.0	969.6	5000.1
7	2A2-07	1685.0	0.0	2000.0	0.0	1315.0	5000.0
8	2A2-08	2057.0	17.50	2000.0	0.0	925.6	5000.1
9	2A2-09	1000.5	999.0	2000.0	0.0	1000.6	5000.1
10	2A2-10	1333.5	1333.5	1000.0	0.0	1333.4	5000.4
11	2A2-11	1769.5	616.0	1400.0	0.0	1214.7	5000.2
12	2A2-12	2154.5	876.0	1000.0	0.0	969.6	5000.1
13	2A2-13	2350.0	0.0	1000.0	0.0	1650.0	5000.0
14	2A2-14	1390.5	1390.5	1368.6	0.0	850.0	4999.6
15	2A2-15	1650.0	0.0	1700.0	0.0	1650.0	5000.0
16	2A2-16	1650.0	700.0	1000.0	0.0	1650.0	5000.0
17	2A2-17	2758.5	0.0	1000.0	0.0	1241.4	4999.9
18	2A2-18	1700.0	1450.0	1000.0	0.0	850.0	5000.0
	Total	31499.9	12495.4	25452.0	0.0	20553.0	90000.3

WHC-SD-W100-TP-011 REV. 0
APPENDIX A

Table 9. Feed Stream Type 7 Specimen Preparation Worksheet

Total Batch 5000
Amount

Order	Ident.	Cement	Fly Ash	Waste	Grit	Water	Total
1	Type 7-01	1697.405	712.9	1425.0	0.0	1164.7	5000.0
2	Type 7-02	2373.5	0.0	1558.4	0.0	1068.1	5000.0
3	Type 7-03	1000.5	999.0	2000.0	0.0	1000.6	5000.1
4	Type 7-04	1700	1450.0	1000.0	0.0	850.0	5000.0
5	Type 7-05	1075	1075.0	2000.0	0.0	850.0	5000.0
6	Type 7-06	2154.5	876.0	1000.0	0.0	969.6	5000.1
7	Type 7-07	1685	0.0	2000.0	0.0	1315.0	5000.0
8	Type 7-08	2057	17.50	2000.0	0.0	925.6	5000.1
9	Type 7-09	1000.5	999.0	2000.0	0.0	1000.6	5000.1
10	Type 7-10	1333.5	1333.5	1000.0	0.0	1333.4	5000.4
11	Type 7-11	1769.5	616.0	1400.0	0.0	1214.7	5000.2
12	Type 7-12	2154.5	876.0	1000.0	0.0	969.6	5000.1
13	Type 7-13	2350	0.0	1000.0	0.0	1650.0	5000.0
14	Type 7-14	1390.5	1390.5	1368.6	0.0	850.0	4999.6
15	Type 7-15	1650	0.0	1700.0	0.0	1650.0	5000.0
16	Type 7-16	1650	700.0	1000.0	0.0	1650.0	5000.0
17	Type 7-17	2758.5	0.0	1000.0	0.0	1241.4	4999.9
18	Type 7-18	1700	1450.0	1000.0	0.0	850.0	5000.0
	Total	31499.9	12495.4	25452.0	0.0	20553.0	90000.3

Viscosity Computation

Viscosity can be computed in several ways:

1. In terms of an overall instrument constant

$$\eta = K \frac{\theta}{N}$$

where K is the overall instrument constant

$$\left(\frac{\text{dyne sec}}{\text{cm}^2} \right) \left(\frac{\text{rpm}}{\text{degree deflection}} \right)$$

θ is the torsion spring factor

θ is the Fann Viscometer reading

N is the rate of revolution of the outer cylinder

η will be in centipoise

2. In terms of three instrument constants for torsion spring, bob surface, and shear gap.

$$\eta = \frac{k_1 k_2}{k_3} (100) \frac{\theta}{N}$$

where k_1 is the torsion constant in dyne-cm/degree deflection

k_2 is the shear stress constant for the effective bob surface, cm^2

k_3 is the shear rate constant, sec^{-1} per rpm

100 is a conversion factor

1 poise = 100 cP

η will be in centipoise

3. In terms of shear stress divided by shear rate

$$\eta = \frac{\tau}{\dot{\gamma}}$$

where τ is the shear stress in dynes/cm²

$\tau = k_1 k_2 \theta$

$\dot{\gamma}$ = shear rate in sec^{-1}

$\dot{\gamma} = k_3 N$

η will be in poise

1 poise = 100 cP

Torsion Springs

TORSION SPRING ASSEMBLY	TORSION SPRING CONSTANT k_1 (dyne-cm/deg. defl)	MAXIMUM SHEAR STRESS WITH B1 BOB (dynes/cm ²)	COLOR CODE
F0.2	77.2	0.2	307 Green
F0.5	193	0.5	768 Yellow
F1	386	1	1,533 Blue
F2	772	2	3,068 Red
F3	1,158	3	4,600 Purple
F4	1,544	4	6,132 White
F5	1,930	5	7,665 Black
F10	3,860	10	15,330 Orange

Viscosity Measurements

Viscosity measurements of a fluid (dotted rhogram) with different instruments: one-point; two-point; multiple points; continuous flow curve.

Conversion Factors

SI UNITS	SYMBOL	UNIT	ABBV.	CONVERSION
Shear Stress	τ	Pascal 1 Pascal = 1 Newton/meter ²	Pa	1 Pa = 10 dynes/cm ²
Shear Rate	$\dot{\gamma}$	Reciprocal Second	s ⁻¹	1 s ⁻¹ (no change)
Viscosity	η	Pascal Second or Mill Pascal Second	Pa · s mPa · s	1 Pa · s = 10 poise 1 mPa · s = 1 cP
OILFIELD UNITS (R1 Rotor, B1 Bob and F1 Torsion Spring)				
Shear Stress (exact)	τ	dyne/cm ²	—	1" Fann = 5.111 dynes/cm ²
Shear Stress (exact)	τ	lb/100 ft ²	—	1" Fann = 1,085 lb/100 ft ²
Shear Stress (approx.)	τ	lb/100 ft ²	—	1" Fann = 1 lb/100 ft ²
Shear Rate	$\dot{\gamma}$	Reciprocal Second	1/sec	1/sec = 1.7023 N
Viscosity	η	centipoise	cP	$\eta = \frac{5.111}{1.7023} (100) = 300 \frac{\text{cP}}{\text{N}}$
Effective Viscosity	η_e	centipoise	cP	$\eta_e = 300 \frac{\text{cP}}{\text{N}}$
Plastic Viscosity	PV	centipoise	cP	PV = $\eta_{sp} - \eta_{sp0}$
Yield Point	YP	lb/100 ft ²	—	YP = $\eta_{sp0} - PV$

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WHC-SD-W100-TP-011, REV. 0
APPENDIX B

MEASURING RANGE

Measuring Range for FANN Direct Indicating Viscometers

ROTOR-BOB	R1 B1	R2 B1	R3 B1	R1 B2	R1 B3	R1 B4
BASIC DATA						
Rotor Radius, R_r , cm	1.8415	1.7588	2.5868	1.8415	1.8415	1.8415
Bob Radius, R_b , cm	1.7245	1.7245	1.7245	1.2276	0.8622	0.8622
Bob Height, L , cm	3.800	3.800	3.800	3.800	3.800	1.900
Shear Gap in Annulus, cm	0.1170	0.0343	0.8621	0.6139	0.9793	0.9793
Radius Ratio, R_r/R_b	0.9365	0.9905	0.667	0.668	0.468	0.468
Maximum Use Temperature, °C	93	93	93	93	93	93
Minimum Use Temperature, °C	0	0	0	0	0	0
Overall Instrument Constant, K	300.0	94.18	1355	2672	7620	15,200
Standard F1 Torsion Spring $\eta = K/B/N$						
SHEAR STRESS RANGE						
Shear Stress Constant for Effective Bob Surface k_r , cm ²	0.01323	0.01323	0.01323	0.0261	0.0529	0.106
Shear Stress Range, dynes/cm ² $\tau = k_r k$						
F0.2 $\theta = 1^\circ$	1.02	1.02	1.02	2.01	4.1	8.2
F0.2 $\theta = 300^\circ$	307	307	307	605	1225	2450
F0.5 $\theta = 1^\circ$	2.56	2.56	2.56	5.04	10.2	20.4
F0.5 $\theta = 300^\circ$	766	766	766	1510	3060	6140
F1.0 $\theta = 1^\circ$	5.11	5.11	5.11	10.1	20.4	40.9
F1.0 $\theta = 300^\circ$	1533	1533	1533	3022	6125	12,300
F2.0 $\theta = 1^\circ$	10.22	10.22	10.22	20.1	40.8	81.8
F2.0 $\theta = 300^\circ$	3066	3066	3066	6044	12,250	24,500
F3.0 $\theta = 1^\circ$	15.3	15.3	15.3	30.2	61.3	123
F3.0 $\theta = 300^\circ$	4600	4600	4600	9067	18,400	36,800
F4.0 $\theta = 1^\circ$	20.4	20.4	20.4	40.3	81.7	164
F4.0 $\theta = 300^\circ$	6132	6132	6132	12,090	24,500	49,100
F5.0 $\theta = 1^\circ$	25.6	25.6	25.6	50.4	102	205
F5.0 $\theta = 300^\circ$	7665	7665	7665	15,100	30,600	61,400
F10 $\theta = 1^\circ$	51.1	51.1	51.1	100.7	204	409
F10 $\theta = 300^\circ$	15,330	15,330	15,330	30,200	61,200	123,000
SHEAR RATE RANGE						
Shear Rate Constant k_r , sec ⁻¹ per rpm	1.7023	5.4225	0.377	0.377	0.268	0.268
Shear Rate range, sec ⁻¹ $\dot{\gamma} = k_r N$						
N = 0.9 rpm	1.5	4.9	0.4	0.4	0.24	0.24
N = 1.8 rpm	3.1	9.8	0.7	0.7	0.48	0.48
N = 3 rpm	5.1	16.3	1.1	1.1	0.80	0.80
N = 6 rpm	10.2	32.5	2.3	2.3	1.61	1.61
N = 30 rpm	51.1	163	11.3	11.3	8.0	8.0
N = 60 rpm	102	325	22.6	22.6	16.1	16.1
N = 90 rpm	153	488	33.9	33.9	24.1	24.1
N = 100 rpm	170	542	37.7	37.7	26.8	26.8
N = 180 rpm	306	976	67.9	67.9	48.2	48.2
N = 200 rpm	340	1084	75.4	75.4	53.6	53.6
N = 300 rpm	511	1627	113	113	80.4	80.4
N = 600 rpm	1021	3254	226	226	161	161
VISCOSITY RANGE IN CENTIPOISE⁽¹⁾						
Minimum Viscosity ⁽²⁾						
All models, 600 rpm maximum	0.5 ⁽³⁾	0.5 ⁽³⁾	2.3	4.5	12.7	25
Maximum Viscosity ⁽⁴⁾						
For Model 34A & HC34A, 300 rpm minimum	300	94	1,350	2,700	7,620	15,000
For Model 35A & 35SA, 3 rpm minimum	30,000	9,400	135,000	270,000	762,000	1,500,000
For Model 35A/SR12 & 35SA/SR12, 0.9 rpm minimum	100,000	31,400	400,000	890,000	2,550,000	5,000,000
Notes:						
(1) Computed for standard torsion spring $G = 1.1$ For other torsion springs multiply viscosity range by f factor.						
(2) Minimum viscosity is computed for minimum shear stress and maximum shear rate.						
(3) For practical purposes the minimum viscosity is limited to 0.5 cP because of Taylor Vortices.						
(4) Maximum viscosity is computed for maximum shear stress and minimum shear rate.						