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16. KEY

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Test Procedures for Polyester-Immobilized Salt-containing Surrogate Mixed Wastes

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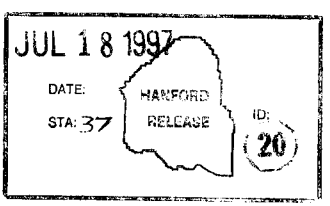
Key Words: Mixed Waste, Salt Waste, Simulant, Polyester, Vinyl Ester, Polymer, Encapsulation, Microencapsulation, Stabilization, Solidification, Test Procedures.

Abstract: This document provides the test procedures for the preparation, immobilization, and testing of surrogate mixed wastes. The solidified waste forms will be subjected to physical property tests, including a modified ANSI/ANS 16.1 leach test, TCLP (Toxicity Characteristic Leaching Procedure) and compressive strength. Waste loadings and waste volume increases will be documented.



James K. Bishop
Release Approval

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Test Procedures for Polyester-Immobilized Salt-containing Surrogate Mixed Wastes

1.0 INTRODUCTION

These test procedures are written to meet the procedural needs of the Test Plan for Immobilization of Salt-containing Surrogate Mixed Wastes Using Polyester Resins, HNF-SD-RE-TP-026 [Biyani, *et al.* 1997], and to ensure adequacy of conduct and collection of appropriate samples and data. This testing will demonstrate the use of four different polyester/vinyl ester resins in the solidification of surrogate liquid and dry wastes, similar to some mixed wastes generated by Department of Energy (DOE) operations.

The object of these procedures is to describe, stepwise, the preparation of final waste form specimens, and their subsequent physical property testing, including compressive strength and leachability tests.

This work is funded by the U.S. DOE Office of Science and Technology Mixed Waste Focus Area under Technical Task Plan (TTP) RL47MW42 *Microencapsulation of Salt Waste Using Polyester Resin* through Fluor Daniel Hanford, Inc.

2.0 DESCRIPTION OF SURROGATE WASTES TESTING

Three waste simulants, listed in Table 2.1, have been selected for testing. The Effluent Treatment Facility (ETF) liquid waste simulant, an aqueous solution containing primarily sulfate salts, is based on the evaporator bottoms stream from contaminated groundwater that is being treated at ETF [Scully 1997]. The two particulate surrogate wastes to be used in these tests are based on a table obtained from the Mixed Waste Focus Area [Beitel 1997].

All three waste surrogates will be prepared at the 222-SA Standards Laboratory in Hanford's 200 West Area and transported to the 306E Lab in the 300 Area, where the final waste forms will be prepared. The procedures used to make up these wastes, as well as the forms of the chemicals actually used, will be documented in the final report giving test results.

The polyester and vinyl ester resins to be used in these tests are manufactured by Ashland Chemical Co., Columbus, Ohio. The resin chosen for the aqueous waste encapsulation is Aropol™ WEP 662P[1], a water-extendible polyester resin. The solid-phase wastes will be encapsulated using the resins Aropol™ S2293, Aropol™ 7334, and Hetron® 922-L25. All resins, as obtained, will contain promoters to facilitate room-temperature curing.

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[1]Aropol™ is a trademark and Hetron®is a registered trademark of the Ashland Chemical Co., Columbus, OH.

Table 2.1: Composition of Mixed Waste Test Simulants

Fe_2O_3	0.0	12.8	6.0
$Al(OH)_3$	0.0	8.5	4.0
Na_3PO_4	0.0	4.3	2.0
$Mg(OH)_2$	1.2	8.5	4.0
MicroCel E	0.0	17.0	8.0
Portland cement (Type I-II)	0.0	4.3	2.0
H_2O	75.0	28.8	13.0
SiO_2	0.7	0.0	0.0
Na_2SO_4	12.7	0.0	0.0
$NaNO_3$	5.9	0.0	60.0
$CaSO_4$	3.8	5.0	0.0
NaCl	0.7	10.0	0.0
PbO	0.0	1,000	1,000
CrO_3	0.0	1,000	1,000
HgO	0.0	1,000	1,000
CdO	0.0	1,000	1,000
NiO	0.0	1,000	1,000
Trichloroethylene	0.0	1,000	1,000
$Sr(NO_3)_2$	0.0	1,000	1,000
$CsNO_3$	0.0	1,000	1,000
$Co(NO_3)_2$	0.0	1,000	1,000
	100.0	100.0	100.0
	1,500	5,000	5,000

Notes: MicroCel E is a registered trademark of the Celite Corporation, Lompoc, CA

2.1 PARTICULATE WASTES

Polyester microencapsulation of *particulate waste* involves a simple mixing process (see Figure 1) in which a batch of resin is first mixed with an initiator, e.g. methyl ethyl ketone peroxide (MEKP), in a mixing bowl. A predetermined amount of waste is then blended into the resin. When the resin has coated the waste particles, the contents of the bowls are transferred to sample molds and allowed to cure. The resin polymerization reaction takes several minutes to gel and harden the waste-resin mixture. The gel time can be varied by adjusting the amount of initiator added.

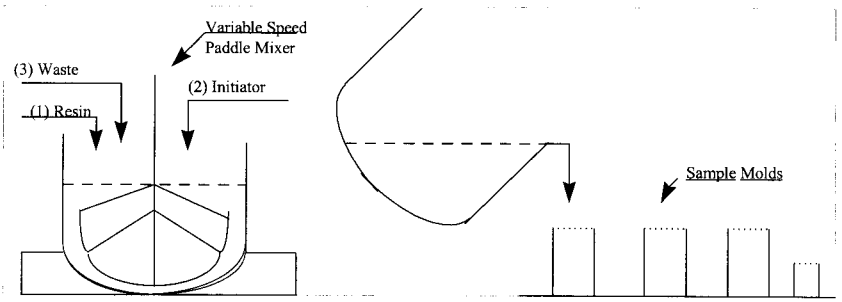

Figure 1: Laboratory Polyester Microencapsulation Flow Diagram

2.2 AQUEOUS WASTES

For encapsulating *aqueous wastes*, a batch of the WEP 662P water-extendible resin is taken in a blender and mixing is started. The waste is added in a steady stream to the resin and a water-in-polyester emulsion is formed due to the high-shear mixing. After all the waste has been added to the resin and a stable emulsion is formed, the initiator is added and evenly dispersed. The initiated emulsion is then poured into molds and allowed to cure.

2.3 EQUIPMENT AND FACILITIES

Equipment and materials needed for preparing the samples and subsequent compressive strength and leach testing is specified under the relevant sections below. The waste surrogates are being prepared in the 222-SA Standards Laboratory in Hanford's 200 West Area. The polyester-immobilized specimens will be prepared in the 306E laboratory. Compressive strength evaluation, and modified ANSI/ANS 16.1 leach tests will also be done in 306E. The TCLP test and leachate analyses will be performed at the Special Analytical Services (SAS) Laboratory in the 600 Area.

3.0 TEST PROCEDURE

The test methodology will be to prepare waste form specimens in twenty separate batches as laid out in the Test Plan [Biyani *et al* 1997] and specified in the batch preparation data sheet in Appendix A. The main variables under investigation are resin type, waste to resin loading, and initiator concentration required for proper resin hardening. In addition to physical appearance evaluation, the test specimens will be subjected to compressive strength measurement [ASTM 1993], the TCLP test [EPA 1992], and a modified ANSI/ANS 16.1 static leach test [ANS 1986].

3.1 SPECIMEN PREPARATION AND DOCUMENTATION

The final waste form specimens will be prepared as outlined in Sections 2.1 and 2.2. In conformance with ALARA principles, opening of resin, initiator, and surrogate waste containers, and waste specimen preparation will be performed in a laboratory hood that has been checked for adequate air flow. If deemed necessary by an industrial hygienist, personnel directly handling the resins will be monitored for exposure to styrene vapors being formed at exposed resin surfaces. A suitable device for representative air sampling and capture of styrene vapors in the vicinity of the hood will be provided and evaluated by the industrial hygienist.

The data sheet to be completed when preparing each resin-waste batch is given in Appendix A. A second data sheet, also in Appendix A, shall be used to record results from specimen evaluations and testing. As stated in Section 2.3, the TCLP test [EPA 1992] will be performed at the SAS Laboratory using their in-house procedures. The procedure to be used for the compressive strength tests and the ANSI/ANS 16.1 leach tests are detailed below.

3.2 COMPRESSIVE STRENGTH MEASUREMENT

Only those waste form specimens that are properly solidified with no free liquids shall be tested for compressive strength. The method [Franz, *et al.* 1994 and ASTM 1993] used for determining the compressive strength of both the aqueous and solid phase waste form samples is outlined below:

3.2.1 Apparatus and Materials

1. 50 mm diameter x 100 mm high right circular cylindrical test specimens, cured for a period of at least two days from the mixing date.
2. Model CT-40K-DLC compression testing machine manufactured by Cal-Teck Inc., Clackamas, Oregon, with capacity of 17,800 kg (40,000 lb), spacers for 50 mm x 100 mm specimens, and conforming to ASTM C39 - 93a specifications.
3. Plastic bags, 4-liter capacity.

4. Masking tape.
5. Humboldt®[2] heating pot for melting sulfur capping compound.
6. Cylindrical capping fixture.

3.2.2 Specimens

1. It is expected that the test specimens will be easily removed from the plastic mold by lightly tapping on the sides of the mold. If this fails to release the specimens, wear leather gloves and use the "Thermo Schneider 20 ZTS" hot knife to score the outside of the mold vertically and the bottom to allow removal of the sample.

2. Weigh the specimen to the nearest 0.1 g.

3. Carefully inspect the specimen for irregularities (air cavities, cracks, gradation in color/texture). Record the findings for each specimen.

4. Plane the ends of the test specimens to 0.050 mm (0.002 in.) by grinding,filing, or sawing. If this is not readily achievable, the specimens shall be capped as described in Step 6.

5. At midpoints, measure the diameter and length, two readings each, approximately 90° apart, to the nearest 0.03 mm, so that an average determination can be made. Use a caliper directly readable to 0.001 in. or 0.03 mm. Record the dimensions. These readings are also to be used for density calculations.

6. Cap the specimen ends with sulfur capping compound.
Safety Note: This step requires handling a molten sulfur based compound, which may result in burns if it comes in contact with the skin. Exercise utmost care when transferring this material.

In the laboratory hood, melt approximately 1 kg of Cylcap sulfur capping compound in the Humboldt® pot (at 115-120°C setting). Put on heat resistant gloves and, using a metal dipping ladle, carefully pour approximately 20 to 30 mL of molten sulfur into the cavity of the cylinder capper. Mount the test specimen to be capped in the vertical cylinder capper. After 10 to 15 seconds, the sulfur will solidify, and the specimen may be removed by tapping the base of the capper lightly with a hammer. Repeat capping procedure on the opposite end of this specimen as well as for the other specimens.

[2]Humboldt® is a registered Trademark of the Humbolt Mfg. Co., Norridge IL.

7. Place the lower bearing block and the 50 mm x 100 mm specimen spacer directly under the upper bearing block.

8. Wipe the bearing block faces clean.

9. Place the sample inside a plastic bag and seal the bag with tape to collect fragments of specimen that fall off during compression. These fragments will be saved for the Toxicity Characteristic Leaching Procedure (TCLP) test.

10. Carefully place the specimen in the testing machine below the center of the upper bearing clock ensuring that the plastic bag is not folded over the upper and lower surfaces of the specimen.

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 (platen) as a guide.

12. Secure the latch on the the wire mesh protective shield of the compression tester to protect personnel from any fragments which may eject when the speciment fails.

13. Turn on the compression machine power and ensure that the digital readout is set to kg.

14. Apply the load continuously and without shock, at a rate of 28 kg to 71 kg/sec (63-157 lb/sec) for a 50 mm diameter cylindrical sample. Do not adjust the rate of movement once loading is started.

15. Apply the load until the specimen fails and record the maximum load (L_{max}) carried by the specimen during the test. Record the appearance of the fragments, noting how the specimen fractured, e.g., conically, or columnar [ASTM 1993].

16. Remove the plastic bag containing pieces of specimen and clean the work area with a brush.

17. Separate the Cylcap material from the sample. If required for this batch, collect 250 g of the fractured specimen and set aside for the TCLP test. Collect all test debris in a plastic bag labeled with the batch number and place in the satellite accumulation area. Such identification will facilitate waste segregation and proper characterization, resulting in waste minimization.

18. Repeat Steps 1-17 for each specimen.

CALCULATIONS

1. Calculate the cross-sectional area of the specimen in square meters (Eqn. 1).

$$Area = \frac{\pi D^2}{4} \times 10^{-4} \qquad \text{Eqn (1)}$$

Where: π = 3.1416
D = The diameter of the specimen, in centimeters.
Area = The cross-sectional area of the sample in m^2.

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

$$CS = \frac{L_{max}}{Area} \times 9.81 \times 10^{-3} \qquad \text{Eqn.(2)}$$

Where:
L_{max} = The maximum load applied to failure, in $kg \cdot m/sec^2$.
Area = The cross-sectional area of the specimen, in m^2.
CS = Compressive strength, in kPa.

3.3 TCLP TEST SPECIMEN PREPARATION

The TCLP, designed to determine the mobility of both organic and inorganic analytes present in solid wastes, is described in considerable detail as Method 1311, *Toxicity Characteristic Leaching Procedure* [EPA 1992]. This test will be performed by the SAS Laboratory according to their internal procedure [Smith 1995]. Described below is the procedure for preparing size-reduced specimens per Method 1311 as applicable to the polyester-immobilized high-chloride and high-nitrate waste samples. Samples from Batches 1 through 6 (see Batch Preparation Data Sheet in Appendix A) will thus be prepared for TCLP testing. The ETF liquid waste simulant samples do not contain the toxicity characteristic metals and will not undergo TCLP testing.

The leachates from these tests will be analyzed for toxicity characteristic metals: lead, chromium, cadium, mercury, and trichloroethylene.

3.3.1 Summary of TCLP Method

The solidified test specimen is reduced in size so as to pass through a 9.5 mm standard sieve and extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid used is a function of the alkalinity of the solid phase of the waste. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

3.3.2 Specimen Size-Reduction

Determination of whether the solid portion of the waste requires particle size reduction (Section 7.1.3 of [EPA 1992]):

> Particle size reduction is required if the sample is greater than 1 cm in its narrowest dimension (i.e., is not capable of passing through a 9.5 mm [0.375 in.] standard sieve). If the particle size is larger than described above, prepare the solid portion of the waste for extraction by crushing or cutting the waste to a surface area, or particle size, as described above. Sieving of the waste is not recommended due to the possibility that the volatile trichloroethylene may be lost. The use of an appropriately graduated ruler is recommended. It is expected that sample fragments from the compressive strength test will be usable for the TCLP test with little further size-reduction required.

3.4 MODIFIED ANSI/ANS 16.1 STATIC LEACH TEST

The procedure given below, which serves as a basis for indexing radionuclide release from waste forms, is derived from the *American National Standard Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure* [ANS 1986].

This procedure describes how to conduct a modified ANSI/ANS-16.1 short-term leach testing procedure. 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 ANSI/ANS 16.1 procedure. This procedure describes only the specific practices used for those tests to accomplish the physical test requirements of ANSI/ANS 16.1. Minor schedule changes have been incorporated into this procedure to accommodate the work week and reduce analytical costs. This includes the requirement to start the initial leach on a Monday morning. The total duration of the test remains three months.

Modifications to the procedure include:

- A modified schedule of leachate changeout (see Table 3.3).
- Analysis of bottle rinses are not conducted.
- Filter media are discarded without being analyzed.

3.4.1 Equipment

- Calipers: (Accuracy: ±0.03 mm)

- Laboratory Balance: (Accuracy: ±0.1 g); ensure calibration is current.

- 1-liter wide-mouthed high-density polyethylene bottles : Two bottles are required for each sample to allow the sample to be placed in a clean bottle with fresh leachant, while leachate from the other beaker is being sampled. Rinse the bottles three times with de-ionized water (DW) before use.

- Leachate sample vials: High-density polyethylene 125-mL vials will be used. 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.

- 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:
  (1) Pour leachate into the syringe and replace the plunger.
  (2) Shake the syringe to make sure that the plunger tip is rinsed.
  (3) Squeeze out the remaining rinse leachate.
  (4) Remove the plunger and place a filter on the end of the syringe.
  (5) Pour leachate into the syringe, replace the plunger and rinse the filter and sample vials by filtering a minimum of 5 mL of leachate into each of the sample vials. Syringes should be rinsed with DW and allowed to dry between intervals.

- pH meter calibrated using pH 4 and 7 buffered standards. Record calibration slope and temperature (follow instruction manual) in logbook.

3.4.2 Procedure

Note the following **safety precautions**:

1. Leachate solution can be corrosive. Safety glasses with side shields must be worn when processing samples.

2. Nitrile gloves must be worn to prevent skin contamination by exposure to samples.

3. After sampling, collect all excess leachate in a labeled polyethylene carboy. *Do not dispose of leachate down the drain.*

The instructions below are to be followed for the test procedure:

1. Record all data in the Sample Evaluation Datasheet and laboratory notebook. Record measurements and the results of calculations as they are called out in the procedure below.

2. Lightly wipe the samples with a soft-bristled brush to remove any loose material that may be left on the samples while removing the samples from the molds.

3. Weigh the sample and record the weight to nearest 0.1 g.

4. Measure diameters and lengths, two readings each, approximately 90° apart, so that average determinations can be made.

5. 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 3.4.3.

6. Fill a leach bottle with the calculated volume of DW.

7. Position the sample near the center of the DW-filled leaching bottle, supported by a wire mesh screen stand placed at the bottom of the beaker. Record the test start time.

8. The leachant will be changed out as described in Table 3.1. When a changeout period is near, the replacement bottle should be rinsed and filled with fresh DW leachant. Changeout is accomplished simply be removing the sample, from the first bottle and transferring it to the bottle containing the fresh leachant.

9. 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 replace it. 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 stable reading.

10. Rinse the syringe, syringe filter, and sample vials as described in Section 3.6. Fill the vials with filtered leachate and labeled by sample number. Sample numbers will be [Batch] - [Sample] - A - [interval from Table 3.1] e.g., 3-4-A-6 indicates the seventh day modified ANS 16.1 leach sample from the fourth sample of Batch 3.

Table 3.1: Total Elapsed time at Leachant Changeout

0	30 sec	30 sec
1	2 hr	7 hr
2	7 hr	1 day
3	1 day	2 day
4	2 day	3 day
5	3 day	4 day
6	4 day	7 day
7	5 day	14 day
8	19 day	28 day
9	47 day	56 day
10	90 day	91 day

3.4.3 Calculations

(A) Surface Area = $2\pi r(r+h)$ [=] cm^2

(B) Volume = $\pi r^2 h$ [=] cm^3

Where: r = radius of sample, cm
h = height of sample, cm

3.4.4 Recording of Data and Results

Record all data in a controlled laboratory notebook. Make entries so that analytical records for leachate analyses are traceable.

4.0 WASTE MANAGEMENT

An important parameter of efficient waste management in this project is *proper segregation* of the various types of wastes expected to be generated.

Wastes, including used paper towels and gloves, and spent samples generated during the high-chloride and high-nitrate wastes testing may be characteristic wastes [WDOE 1994] because they

contain hazardous metals and organics. These wastes shall be segregated from the wastes generated during testing of the ETF surrogate waste that do not contain characteristic contaminants. Characterization of the wastes accumulated will be easily performed with laboratory leach test results and package/container inventory sheets.

Leachates will be collected in a separate container, characterized, and disposed of appropriately [WDOE 1994]. All potentially hazardous wastes will be segregated and accumulated in Satellite Accumulation Areas until they are determined to be non-hazardous. Lab personnel will be instructed in the proper use of the satellite accumulation containers. Hazardous wastes will be disposed of in accordance with WAC-173-303 [WDOE 1994].

5.0 QUALITY ASSURANCE

The Hanford Site *Quality Assurance Manual* (WHC-CM-4-2 *Quality Assurance Manual*) will govern performance of the work described in this test plan. The quality assurance (QA) manual provides the Hanford Site implementation of 10 CFR 830.120 and DOE order 5700.6C.

The quality assurance plan for the chemical analysis of sample leachates is the *Special Analytical Support Quality Assurance Plan* [NHC 1997]. This plan implements the *Hanford Analytical Services Quality Assurance Requirements Document* (DOE 1996), which in turn is the implementation of DOE order 5700.6C [DOE 1991] for laboratory analytical work.

The batch preparation and sample evaluation data sheets in Appendix A contain additional implementation of the *Quality Assurance Manual*. A QA representative will occasionally observe batch preparation and document any oversight observations on the data sheets. This will provide independent verification that test procedures are being executed as stated herein.

6.0 CHANGE PROCEDURE

As in any experimental test conduct, the possibility of procedural change exists. Should such a change be mandated to ensure adequate performance of the work and safe conduct of operations, the operating personnel may institute the required change with approval of the lead engineer or scientist. The latter shall notify the appropriate safety and health support personnel.

Such a change will be documented via an engineering change notice at test completion for configuration control when necessary; all such changes will be entered into appropriate logbooks and reported at test completion.

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APPENDIX A: Data Sheets

Batch Preparation Data Sheet

Date_____

Initials_____

Preparation for Run _____

1. Equipment Setup
   1a. Set Mixer Speed (Target 30 rpm, ± 5rpm):
   _____, _____, _____, _____, _____
   rpm = avg(2 Last) = _____ rpm
   1b. Thread thermocouple through cap of first mold, sheath with Al foil, with stop 5 cm from mold bottom. T/C placed = _____ data channel.
   1c. Don PPE: vinyl gloves, labcoat, safety glasses with sideshields.
   1d. Prepare 4 molds (3x 200mL, 1 x 50mL) by marking as [run number]-[mold number] e.g. (1-1, 1-2, 1-3, 1-4 [50mL mold]), and set aside.
2. Resin: Place mixer bowl on scale, tare, add appropriate resin at mass on table. Record actual mass as:
   R = _____ g resin (±0.1).
3. Initiator: Calculate required initiator (I) as:

$$I = \frac{wt\%}{100} \times \frac{[R,g]}{1.1\ g/mL} = \_\_\_\_\_\ mL\ (\pm 0.1)$$

Load pipette and prepare for addition. Set aside.

4. Waste: Tare 1L beaker. Calculate and add to beaker Waste (W) as:

$$W = \frac{Load \times (R + I \ast 1.1)}{100 - Load} = \_\_\_\_\_\ g\ (\pm 0.1)$$

Run	Resin		Waste Load		Initiator
	Type	Mass (g)	Type	Wt%	Wt%
1	S	443	Cl	50	1.25
2	A	389	Cl	50	1.25
3	H	443	Cl	50	1.25
4	S	443	NO3	50	1.25
5	A	389	NO3	50	1.25
6	H	443	NO3	50	1.25
7	S	355	Cl	60	1.25
8	A	311	Cl	60	1.25
9	H	355	Cl	60	1.25
10	S	355	NO3	60	1.25
11	A	311	NO3	60	1.25
12	H	355	NO3	60	1.25
13	S	266	Cl	70	1.25
14	A	233	Cl	70	1.25
15	H	266	Cl	70	1.25
16	S	266	NO3	70	1.25
17	A	233	NO3	70	1.25
18	H	266	NO3	70	1.25
19	W	342	ETF	60	1.5
20	W	238	ETF	70	1.5

Resin Types: S [=] S2293, A [=] Aropol 7334, H [=] Hetron 922-L25, W [=] Aropol WEP 662P

Waste Types: Cl [=] high chloride, NO3 [=] high nitrate, ETF [=] ETF Liquid

For mixing activities: Do not attempt to dislodge any settled or unmixed material from sides or bottom of bowl without turning off mixer. For batches 19 and 20: A high-speed blender will be used for mixing. Only speed setting (not rpm) will be recorded. Reverse order of Steps 5 and 6; a stable waste-in-polyester emulsion must be formed before the initiator is added.

5. Initiator Addition: With resin in mixer bowl, and mixer on at ~30rpm, add initiator over 3-4 minutes. Start time for initiator addition _____.
6. Waste Addition: With resin and initiator in bowl, and mixer on at ~30 rpm, add waste over 7-9 minutes. Stop mixer. With spatula, collect excess mixture from mixer blades in bowl, scrape bottom and sides of bowl, briefly homogenizing with body of mixture.
7. Sample pour: Dispense the waste mixture to the four molds, filling to ~3mm from top.
8. Waste Cleanup: Follow the principles outlined in **Section 4.0.** Pour bowl residuals into 1-gallon can/carton. Clean bowl and blades with paper towels wetted with Dibasic ester (DBE) solvent. Place toweling and residuals in a plastic bag labeled with the batch number and the number of paper towels. Weigh the bag of waste material. Tie the end of the bag off and place the bag in the appropriate satellite accumulation container. Enter the information on the Satellite Accumulation Logsheet. To calculate the wt.% of test material deduct the weight of the bag and the paper towels from the total weight.
9. Sample closure and storage: Seal samples with caps, placing the thermocouple fitted cap into the first sample. Place sealed samples into insulated ice chest and verify data logger operation for temperature capture.
10. QA comments:_____

_____

Sample Evaluation Data Sheet

Date_____
Initials_____

Evaluations for Run _____

1. Exotherm Observation: Sample _____ Datachannel _____
   Acquire initiator addition time from Batch Preparation sheet and exotherm data from datalogger and calculate time to peak temperature. T_{ref} should be the mean sample storage chest temperature over the period from placement through peak.

t_0 =	Date/hh/mm	t_{maxT} =	Date/hh/mm	Δt =	min
T_{ref} =	°C	T_{max} =	°C		

2. Sample Observation:
   Remove the samples from the sample insulated ice chest, remove caps and thermocouple. Observe sample conditions, if solidified, remove from mold, weigh and complete observations for liquids, cracks, bubbles, or other deformations. Record observations by sample number:

Sample (batch-number)	Mass (g) [±0.1g]	Observations
-1		
-2		
-3		
-4		

Following observation, place each sample in individually marked plastic bags by sample number.

3. Compressive Strength:
   a. Select sample [Batch#]-2 ([Batch#]-3 if second is not available): Sample:_____
   b. Conduct Compressive Strength procedure per **Section 3.2**, record observations, and calculate density of sample.

Sample Dimensions (±0.003 cm)				Sample Mass (g) (±0.1 g)		Compressive Strength (kPa)	
L_1 =	cm	D_1 =	cm	mass =	g	CS =	kPa
L_2 =	cm	D_2 =	cm	Sample Density $\rho = 4 \cdot mass/(\pi \cdot L_{avg} \cdot D_{avg}^2)$		Failure Mode:	
L_{avg} =	cm	D_{avg} =	cm	ρ =	g/mL		

4. TCLP: For Batches (1, 2, 3, 4, 5, and 6)
   Prepare 100 g of crushed sample from Step 3 by TCLP procedure per [Smith 1995]
   Record Sample Number: _____. Record Extraction Fluid: Extractant (1) or Extractant (2).
5. Modified ANS 16.1 Leach testing: For Batches (1, 3, 4, 6, 7, 9, 10, 12, 13, 15, 16, 18, and 19) dependent upon sample availability.
   a. Select and record the fourth sample:_____
   b. Conduct modified ANS 16.1 leach procedure per **Section 3.4.** Upon availability, accumulate sample and analytical data in logbook.
6. QA comments:_____

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