Stat

JAN 0 9 1998

ENGINEERING DATA TRANSMITTAL

Page 1 of 1

1. EDT 601921

2. To: (Receiving Organization) NHC Process Development	3. From: (Originating Organization) NHC Process Chemistry	4. Related EDT No.: NA
5. Proj./Prog./Dept./Div.: TWRS	6. Design Authority/ Design Agent/Cog. Engr.: D. L. Herting	7. Purchase Order No.: NA
8. Originator Remarks: Approval/release of attached Test Plan is requested.		9. Equip./Component No.: NA
		10. System/Bldg./Facility: 222-S Laboratory
11. Receiver Remarks: 11A. Design Baseline Document? [] Yes [X] No		12. Major Assm. Dwg. No.: NA
		13. Permit/Permit Application No.: NA
		14. Required Response Date: December 31, 1997

15. DATA TRANSMITTED

(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev. No.	(E) Title or Description of Data Transmitted	(F) Approval Designator	(G) Reason for Transmittal	(H) Originator Disposition	(I) Receiver Disposition
1	HNF-1863		0	Test Plan for Tank 241-AN-104 Dilution Studies	NA	1,3	1	

16. KEY

Approval Designator (F)	Reason for Transmittal (G)		Disposition (H) & (I)	
E, S, Q, D or N/A (see WHC-CM-3-5, Sec.12.7)	1. Approval 2. Release 3. Information	4. Review 5. Post-Review 6. Dist. (Receipt Acknow. Required)	1. Approved 2. Approved w/comment 3. Disapproved w/comment	4. Reviewed no/comment 5. Reviewed w/comment 6. Receipt acknowledged

17. SIGNATURE/DISTRIBUTION
(See Approval Designator for required signatures)

(G) Reason	(H) Disp.	(J) Name	(K) Signature	(L) Date	(M) MSIN	(G) Reason	(H) Disp.	(J) Name	(K) Signature	(L) Date	(M) MSIN
		Design Authority				1	1	P. J. Certa	PJCerta	1/8/98	H5-61
		Design Agent				3		B. B. Peters			H5-03
1	1	Cog.Eng. D. L. Herting	DL Herting	12/23/97	T6-07	3		J. S. Garfield			H5-49
1	1	Cog. Mgr. J. R. Jewett	JR Jewett	12/31/97	T6-07	3		R. A. Kirkbride			H5-27
		QA				3		K. D. Wiemers			K6-51
		Safety				3		J. Y. Bourges			T6-07
		Env.				3		J. F. O'Rourke			T6-07

18.	19.	20.	21. DOE APPROVAL (if required) Ctrl. No.
D. L. Herting DL Herting 12/23/97 Signature of EDT Originator Date	P. J. Certa Paul J. Certa 1/8/98 Authorized Representative for Receiving Organization Date	J. R. Jewett JR Jewett 12/31/97 Design Authority/ Cognizant Manager Date	[] Approved [] Approved w/comments [] Disapproved w/comments

BD-7400-172-2 (05/96) GEF097

BD-7400-172-1 (07/91)

Test Plan for Tank 241-AN-104 Dilution Studies

D. L. Herting
Numatec Hanford Company, Richland, WA 99352
U.S. Department of Energy Contract DE-AC06-96RL13200

EDT/ECN: 601921 UC: 2030
Org Code: 8C510 Charge Code: D2DC4
B&R Code: EW3130010 Total Pages: 18

Key Words: dilution, dissolution, sodium salts, privatization, vitrification feed, retrieval

Abstract: Tank 241-AN-104 (104-AN) has been identified as the one of the first tanks to be retrieved for low level waste pretreatment and immobilization. Retrieval of the tank waste will require dilution. Laboratory tests are needed to determine the amount and type of dilution required for safe retrieval and transfer of feed and to re-dissolve major soluble sodium salts while not precipitating out other salts. The proposed laboratory tests are described in this document.

TRADEMARK DISCLAIMER. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof or its contractors or subcontractors.

Printed in the United States of America. To obtain copies of this document, contact: Document Control Services, P.O. Box 950, Mailstop H6-08, Richland WA 99352, Phone (509) 372-2420; Fax (509) 376-4989.

Release Approval 1/9/98 Date

DATE: STA: 4 HANFORD RELEASE ID: 3 JAN 09 1998

Release Stamp

Approved for Public Release

CONTENTS

1.0 INTRODUCTION 1

2.0 BACKGROUND 3

3.0 DESCRIPTION OF TEST 5
3.1 Tank Composite Sample Preparation 5
3.2 Dilution Testing With Whole Tank Composite 5
3.2.1 Dilution, settling, and chemical analyses 6
3.2.2 Viscosity 8
3.3 Dilution Testing With Settled Solids 8
3.3.1 Settle, decant, and dilute supernatant liquid 8
3.3.2 Dilution of settled solids. 9
3.3.3 Temperature effect 9
3.3.4 Rheology 9
3.3.5 Particle size distribution and microscopy 10

4.0 DATA INTERPRETATION 11
4.1 Physical Data 11
4.2 Chemical Data 13
4.3 Quality Assurance 13

5.0 SAFETY 14

6.0 WASTE HANDLING 14

7.0 SCHEDULE AND DELIVERABLES 14

8.0 REFERENCES 15

TABLES

Table 1. Comparison of Waste Compositions in Tanks 104-AN and 105-AN . . . 2

Table 2. Comparison of Analytical Results for Supernatant Liquid and Settled Solids Samples 4

Table 3. Core Segment Samples Available for Whole-Tank Composite 6

Table 4. Correspondence of Dilutions Tests for Tanks 104-AN and 105-AN . . 12

ACRONYMS AND ABBREVIATIONS

104-AN	Tank 241-AN-104
105-AN	Tank 241-AN-105
AT	total alpha
DSSF	double shell slurry feed
G	gravity
g	gram
gal	gallon
GEA	gamma energy analysis
IC	ion chromatography
ICP	inductively coupled plasma spectroscopy
kg	kilogram
L	liter
Liq	liquid sample
mL	milliliter
PLM	polarized light microscopy
RGS	retained gas sample
rpm	revolutions per minute
SEM/EDS	scanning electron microscope/energy dispersive x-ray spectroscopy
Sol	solid sample
SpG	specific gravity (or density)
SSol	settled solids
Sup	supernatant liquid
SupD	diluted supernatant liquid
TCR	Tank Characterization Report
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TOC	total organic carbon
wt%	weight percent
WTC	whole-tank composite
µCi	microcurie
µg	microgram

1.0 INTRODUCTION

Tank 241-AN-104 (104-AN) has been identified as one of the first tanks to be for retrieved for low level waste pretreatment and immobilization. Retrieval of the tank waste will require dilution. Laboratory tests to determine the amount and type of dilution required for safe and efficient retrieval of the tank waste are described in this document. They are based on test requirements established by Process Development (Garfield 1997 and Certa 1998).

The waste in tank 104-AN is very similar in composition to that in tank 241-AN-105 (105-AN). See Table 1. Therefore, most of the conclusions from the testing of 105-AN waste during fiscal year 1997 (Herting 1997) should be applicable to the 104-AN waste. The tests proposed here for the 104-AN waste are designed (1) to verify the similarity to 105-AN waste in chemical and physical behavior, and (2) to provide additional information on dilution effects.

The 104-AN tests will be done with a whole-tank-composite sample prepared by combining appropriate amounts of drainable liquid and settled solids core segment samples from the two core samples that were taken in 1996 (Hu 1997). The tests performed on the composite sample will be aimed primarily at the alternative retrieval scenario in which the tank supernatant liquid is removed (with some in-line dilution) before diluent is added to the settled solids remaining in the tank. Corresponding changes in laboratory procedures relative to the 105-AN tests are detailed later in this test plan.

Table 1. Comparison of Waste Compositions in Tanks 104-AN and 105-AN

Analyte	Liquid Phase (g/L)[e]		Settled Solids (g/kg)[e]	
	105-AN[a]	104-AN[b]	105-AN[c]	104-AN[d]
Al	41.7	38.6	22.1	20.6
B	0.074	0.065	0.090	0.13
Ca	---	---	0.64	0.22
Cd	---	---	0.012	0.011
Fe	---	---	0.12	0.10
K	6.50	6.70	3.45	3.57
Mn	---	---	0.021	0.018
Mo	0.097	0.080	0.052	0.050
Na	247	256	183	192
Ni	---	---	0.041	0.065
P	0.4	0.88	2.5	1.4
S	0.94	1.2	2.5	3.8
Si	0.22	0.24	0.14	0.61
Cl^-	9.9	8.1	5.2	4.4
NO_2^-	122	125	64	63
NO_3^-	161	170	111	112
PO_4^{3-}	0.9	2.3	8.0	3.7
TIC	2.20	2.17	13.1	14.7
TOC	2.59	3.11	5.4	4.6
OH^-	58.6	70.9	24.1	37.2
H_2O	50.2 wt%	51.1 wt%	44.4 wt%	45.6 wt%
SpG	1.42	1.40	---	1.60
^{137}Cs	0.475 Ci/L	0.57 Ci/L	0.30 Ci/kg	0.35 Ci/kg

[a] Tables B3-9 and B3-12 in TCR (Jo 1997)
[b] Tables B3-4 and B3-7 in TCR (Hu 1997)
[c] Tables B3-14 and B3-17 in TCR (Jo 1997)
[d] Tables B3-9 and B3-12 in TCR (Hu 1997)
[e] except as noted

2.0 BACKGROUND

Tank 104-AN currently contains approximately 4.0 million liters (1.1 million gal) of waste classified as double shell slurry feed (DSSF). Two core samples were taken in 1996. Each core was composed of 21 segments. Based on the core sample results, tank waste layer volumes were estimated (Hu 1997) as 2,100 L (5,500 gal) crust, 2.3 million L (600,000 gal) supernatant liquid, and 1.7 million L (449,000 gal) settled solids. The settled solids are also referred to as sludge, slurry, or wet saltcake in various documents.

The settled solids contain approximately 89% by weight interstitial liquid and 11% actual solid phase, based on the percent water analytical results. No specific analyses have been done on the actual solid phase, but some information can be gleaned by comparing the analyses of the settled solids layer with analyses of the supernatant liquid layer. This comparison is made in Table 2.

Components that are significantly higher in the settled solids than in the supernatant liquid include sodium, phosphate, sulfate, total inorganic carbon (TIC), and total organic carbon (TOC). The major solid phases, therefore, are likely to be sodium salts of carbonate, sulfate, phosphate, and oxalate; and double salts such as sodium fluoride sulfate, Na_3FSO_4, and sodium carbonate sulfate, $Na_6CO_3(SO_4)_2$. Typically, these double salts are less soluble than the individual salts, though only a little study has been done in this area for Hanford waste solutions.

One gram of settled solids contains less liquid than one gram of liquid, so components that are present only in the liquid phase (e.g. Al, K, nitrite, chloride, hydroxide) should have lower concentrations in the settled solids. The fact that the concentration of nitrate is nearly the same in both the liquid and the settled solids implies that there may be some $NaNO_3$ in the solid phase.

The solubilities of some of the sodium salts are known to be very temperature-dependent. Therefore, temperature is likely to be a key factor in the dilution/dissolution experiments. The solubility of sodium oxalate is very dependent on ionic strength, and is unlikely to dissolve very much at the dilution ratios used for these tests.

Table 2. Comparison of Analytical Results for Supernatant Liquid and Settled Solids Samples (averages of segment sample results)[1]

Component	Supernatant Liquid (μg/mL)	Supernatant Liquid (μg/g)[3]	Settled Solids (μg/g)
Density	1.40 g/mL	1.40 g/mL	1.60 g/mL
H_2O	51.1 wt%	51.1 wt%	45.6 wt%
Acetate	602	430	583
Al	38,600	27,600	20,600
Cl^-	8,090	5,780	4,420
Cr	336	240	1,650
Formate	983	702	777
K	6,700	4,800	3,570
NO_2^-	125,000	89,300	62,800
NO_3^-	170,000	121,000	112,000
Na	256,000	183,000	192,000
PO_4^{3-}	2,270	1,620	3,720
SO_4^{2-}	1,700	1,210	11,200
OH^- (2)	70,900	50,600	37,200
TIC[2]	2,170	1,550	14,700
TOC[2]	3,110	2,220	4,630

[1] from segment sample averages (Hu 1997)
[2] from composite sample averages (Hu 1997)
[3] previous column divided by density to convert units

3.0 DESCRIPTION OF TEST

3.1 Tank Composite Sample Preparation

All of the dilution/dissolution tests will begin with a whole-tank composite sample prepared from samples currently in the laboratory. Sample preparation and all of the testing will be done in hotcells at the 222-S Laboratory. The core samples shown in Table 3 are available for use in this study. The weights listed under the heading "Current Inventory" represent the total amount of material currently stored in the laboratory. The smaller weights under the heading "Available for Composite" represent the amount that TWRS Characterization is willing to release for the dilution study.

Based on the tank waste profile and layer densities reported in Section 2, the tank contains approximately 2.7 million kg of settled solids and 3.2 million kg of supernatant liquid. The whole-tank composite sample will be created by mixing the layer samples in the same ratio. Thus, approximately 900 g (560 mL) of settled solids samples from Table 3 will be mixed with 1100 g (780 mL) of supernatant liquid samples.

The waste composition is sufficiently homogeneous within each layer in the tank that using different sample weights from individual segments within a layer will not have any adverse effect on the composition of the composite sample. In addition, the previous tests with waste from tank 105-AN showed that there is a large range of acceptable dilution ratios. Therefore, virtually all of the "Available for Composite" sample can be used. The composite will be mixed and stored at the ambient hotcell temperature. All tests will be done with aliquots taken from the composite sample.

It is widely recognized that one of the most difficult problems in analytical chemistry is to obtain a uniform aliquot from a two-phase (solid/liquid) sample. To ensure the best possible uniformity, all aliquots will be taken by syringe sampling while the composite sample is being mixed. Duplicate tests performed on similar 105-AN samples (Herting 1997) showed that this sampling method gives acceptable reproducibility.

The composite sample will be divided into three 500 mL sample jars for ease in handling with hotcell manipulators. At least one of these jars will be marked with volumetric indicators. This jar will be used to perform an ambient temperature settling rate test on a larger scale than is possible with the 50 mL centrifuge cones that will be used for most of the tests.

3.2 Dilution Testing with Whole Tank Composite

The first tests will represent adding mixer pumps to the tank and mixing the waste before any dilution or transfer takes place. These tests will duplicate tests performed on the 105-AN samples, but the number of tests will be much smaller. Tests will be limited to one temperature (45 °C), which was the baseline temperature for the 105-AN sample tests.

Table 3. Core Segment Samples Available for Whole-Tank Composite

Core	Segment[1]	Parent Jar No.[2]	Current Inventory (g)	Available for Composite (g)
163	2	11188	295	100
163	3	RGS	375	150
163	4	11189	305	150
163	5	11523	240	50
163	6	11522	280	100
163	7	11192	275	50
163	8	11195	325	150
163	9	11196	305	150
163	10	11526	235	50
163	11	11551	290	150
163	15	RGS	375	200
163	17	RGS	225	100
163	19	RGS	375	250
163	21	RGS	225	100
164	18	RGS	375	250
			TOTAL	2,000

[1] Segments 2-11 represent supernatant liquid; 15-21 settled solids
[2] RGS = retained gas sample analysis samples

3.2.1 Dilution, settling, and chemical analyses

Two sample aliquots, 30 mL each, will be transferred from the whole-tank composite into 50 mL centrifuge cones. The cones will be weighed, and then placed in a constant temperature water bath held at 45 °C for at least 16 hours.

After the 16 hour equilibration period, water will be added to one of the cones to represent dilution to 50% by volume, i.e., the volume of water added will be one-half of the volume of sample in the cone before dilution. The dilution water will be equilibrated at the same temperature for the same length of time as the samples. No water will be added to the other cone, but it will be mixed and settled as described below.

As soon as the dilution water is added, both cones will be mixed with a vortex mixer. Then the cones will be returned to the water bath. At the end of each hour for five hours, the cones will be mixed again, and returned to the water bath. The cones will then be allowed to settle overnight in the water bath. The following day, the cones will again be mixed and settled three times for one hour each settling period. After the third and final mixing, readings of volume percent settled solids will be recorded as a function of time for the remainder of the work shift. Settling will be allowed to continue for a minimum of 48 hours after the final mixing.

After the final readings of volume percent settled solids, the cones will be centrifuged at approximately 2000 rpm (300 G force) for 20 minutes. Volumes of centrifuged solids will be recorded. Supernatant liquids will be decanted into sample vials, and the cones will be re-weighed to determine the weight percent centrifuged solids. One portion of each supernatant liquid will be analyzed for $\%H_2O$ (gravimetric method) and density (SpG). A second portion will be analyzed for the following:

IC (ion chromatography for F^-, Cl^-, NO_3^-, NO_2^-, PO_4^{3-}, SO_4^{2-}, and oxalate analyses)
TIC (total inorganic carbon)
TOC (total organic carbon)
ICP (inductively coupled plasma spectroscopy for metal ion analyses)
OH^- (free hydroxide)
GEA (gamma energy analysis)
AT (total alpha)
^{90}Sr and ^{99}Tc.

The remaining fraction of each supernatant liquid sample will be stored in the water bath for at least one month, and will be observed periodically to see whether any solids have formed. If solids do form, they will be characterized for physical behavior (e.g. gelatinous or rapid-settling). They will be examined by polarized light microscopy (PLM) to attempt identification based on crystal habit and optical properties. They may also be analyzed by whatever chemical analysis methods are deemed appropriate by the chemist-in-charge.

The centrifuged solids in each cone will be weighed, and then transferred to a sample vial for the following analyses:

$\%H_2O$ by thermogravimetric analysis (TGA)
direct solid sample for TIC, TOC
water digest for IC and OH^-
acid digest for ICP, GEA, AT, ^{90}Sr and ^{99}Tc.

3.2.2 Viscosity

Rheology measurements (shear stress vs. shear rate) will be made on three samples as a function of temperature over the range ambient to 65°C. The first sample will be a fresh aliquot of the undiluted whole-tank composite sample. The second sample will be another whole-tank composite sample diluted to 50% by volume with water. The third sample will be the supernatant liquid from an undiluted whole-tank-composite sample.

If the viscosity of the supernatant liquid sample is above 5 cP at any temperature, then a fourth sample will be tested. The fourth sample will be the supernatant liquid diluted 20% by volume with water.

The viscometer used for these measurements will consist of a Haake Rotovisco® RV20 with a CV20 Sensor System. A Mooney-Ewart type measuring system will be used in conjunction with the CV20 for the rheological measurements. This coaxial cylinder system consists of a beaker and cylinder each with a cone-shaped bottom face. The torque on the inner cylinder is measured as a function of the speed of the rotating beaker.

3.3 Dilution Testing with Settled Solids

The next series of tests will represent removing the supernatant liquid from the tank with in-line dilution, then adding mixer pumps and dilution water to the tank to dissolve the sodium salts in the settled solids.

3.3.1 Settle, decant and dilute supernatant liquid

Eight sample aliquots, 45 mL each, will be transferred from the whole-tank composite into 50 mL centrifuge cones. The cones will be weighed, and then placed in a constant temperature water bath at 45 °C for at least 72 hours.

The next step will be to decant the supernatant liquid from each of the eight centrifuge cones. All of the decanted liquid will be collected in a single jar or bottle. After the liquid is mixed (by stirring), it will be re-divided into nine 15 mL centrifuge cones, 10 mL per cone. Density of the liquid will be measured from the volume and weight of liquid in each cone. The remaining liquid will be used for viscosity measurement (Section 3.3.4).

Three of the liquid samples will be diluted 10% by volume with water, and another three will be diluted 20% by volume. The other three samples will remain undiluted. New densities will be measured for the six diluted samples.

One undiluted sample, one 10%-diluted sample, and one 20%-diluted sample will be held at ambient temperature (approximately 25 °C) for a minimum of one week. Three corresponding samples will be held at 45 °C, and the three remaining samples at 65 °C for the same time period. All samples will be observed for possible formation of precipitates. If any precipitates form, the volume and weight of solids will be measured. The solids will be examined with a polarized light microscope to attempt identification of the solid phase. Additional chemical analyses of the solids may be requested by the chemist-in-charge.

3.3.2 Dilution of settled solids

The volume and weight of settled solids remaining after decanting the supernatant liquids will be recorded. Then water will be added to each cone to represent dilution to 0, 40, 80, or 120% of the settled solids volume, each dilution made in duplicate. Before the dilutions are made, the water will be equilibrated at the same temperature for the same length of time as the samples.

The remainder of the testing will follow the procedure described for the whole-tank composite samples described in Section 3.2. The supernatant liquid decanted after centrifuging the samples will be divided. One portion of each liquid will be stored in the water bath for at least one month to watch for precipitation of solids. The remainder of each liquid and the centrifuged solids will be analyzed as described in Section 3.2.1.

3.3.3 Temperature effect

A subset of the settled-solids dilution series of tests (Sections 3.3.1 and 3.3.2) will be repeated at higher and lower temperatures. The subset will consist of the 80% dilution samples at 65°C and at 25°C. Analytical requirements will be the same as described in Section 3.2.1.

3.3.4 Rheology

Rheology measurements (shear stress vs. shear rate) will be made on three samples as a function of temperature over the range ambient to 65°C. The first sample will be the undiluted settled solids sample. The second sample will be the settled solids sample diluted to 80% by volume with water, relative to the volume of settled solids after decanting the supernatant liquid. The third sample will be the supernatant liquid from the 80%-diluted sample.

3.3.5 Particle size distribution and microscopy

Particle size distribution analysis will be performed on one sample of undiluted solids and one sample of solids remaining after 80% dilution of the settled solids. Polarized light microscopy (PLM) will be performed on the same two samples to identify changes in morphology caused by dilution. Analysis of the solids by scanning electron microscopy with energy dispersive x-ray spectroscoy (SEM/EDS) may be performed in addition to or instead of the PLM, depending on availability and state of repair of the SEM/EDS instrument.

4.0 DATA INTERPRETATION

4.1 Physical Data

Volumes and weights of settled and centrifuged solids will be the primary measurements used to evaluate dilution parameters. All of the dilution parameters match those used for tests performed last fiscal year on waste from tank 105-AN. Therefore, direct comparison of the results for the two tank wastes will be possible. Table 4 shows the correspondence.

Dilution with NaOH solution will not be performed on the 104-AN waste samples because the NaOH concentration is higher in 104-AN waste than in 105-AN waste. Therefore, it is very unlikely that NaOH solution would be used for dilution of the 104-AN waste.

Temperature may have a complex effect on the volume of solids. Increasing temperature is expected to cause increasing solubility of sodium sulfate, phosphate, and nitrate, but cause decreasing solubility of sodium carbonate.

Rheology measurements are needed to assess pump characteristics that will be required for mixing and transferring the waste. These measurements will be provided as a function of temperature for the following samples:

undiluted whole-tank composite sample
whole-tank composite sample diluted 50% by volume with water
undiluted settled solids sample with the supernatant liquid decanted
settled solids sample diluted 80% by volume
undiluted tank waste supernatant liquid
supernatant liquid from 80%-diluted settled solids sample.

In all of the dilution tests, decanted centrifuged liquid samples will be stored for at least one month to determine whether solids form after dilution. Any solids that form will be evaluated for physical properties (e.g. gel formation), and analyzed by PLM and chemical analyses if deemed appropriate.

Table 4. Correspondence of Dilution Tests for Tanks 104-AN and 105-AN

Sample[1]	Diluent	Temp, °C	Test[2]	Dilution, Vol%, 105-AN	Dilution, Vol%, 104-AN
WTC	H_2O	45	Sol/Liq	0-25-50-75[3]	0-50
WTC	2M NaOH	45	Sol/Liq	25-50-75	none
WTC	H_2O	25	Sol/Liq	0-25-50-75	none
WTC	H_2O	65	Sol/Liq	0-25-50	none
SSol	H_2O	45	Sol/Liq	0-40-80-120	0-40-80-120[3]
SSol	2M NaOH	45	Sol/Liq	40-80-120	none
SSol	H_2O	25	Sol/Liq	40-80-120	80[3]
SSol	H_2O	65	Sol/Liq	0-40-80	80[3]
SSol	H_2O	25/45	Kinetics	50	none
WTC	H_2O	25-65	Rheology	0-50	0-50-Sup[4]
SSol	H_2O	25-65	Rheology	0-80	0-80-SupD[5]
WTC	H_2O	45	Particle Size	0-50	none
SSol	H_2O	45	Particle Size	none	0-80
SSol	H_2O	45	PLM/SEM/EDS	none	0-80

[1] WTC = whole-tank composite sample; SSol = settled solids sample
[2] Sol/Liq = chemical and physical analysis of solid/liquid phases
[3] tests performed in duplicate
[4] Sup = supernatant liquid from undiluted sample
[5] SupD = supernatant liquid from 80%-diluted sample

4.2 Chemical Data

Chemical analyses of centrifuged liquid and solid phases will be used to develop an understanding of the physical data. With analyses of both phases, it will be possible to calculate the contribution of the interstitial liquid to the centrifuged solids to provide a composition of the true solid phase. Knowledge of which solid phases are present as a function of dilution ratio should provide the information needed to explain the physical data.

As a first approximation, the assumption will be made that all of the water in the centrifuged solids can be attributed to interstitial liquid. If the calculations and/or PLM results suggest that the true solid phase contains some salts that are highly hydrated [e.g. $Na_7F(PO_4)_2 \cdot 19H_2O$], then an iterative calculation may be required to distribute the water in the centrifuged solids between the true solid phase and the interstitial liquid phase.

Analytical determinations will be the same as those for the 105-AN samples done earlier, except that:

(1) Water digest will added to the analysis of the centrifuged solids so that the solids can be analyzed by IC and OH^- for water-soluble anions. This will give a more direct indication than in the 105-AN study of whether nitrate and other IC species are present in the solid phase.

(2) The list of analytes from the ICP that will be reported will be longer, and include the "less-than" elements that were not reported for the 105-AN samples. This will allow more of the Privatization envelope specifications (analyte to sodium mole ratios) to be evaluated.

4.3 Quality Assurance

Based on the requirements in "Review and Approval of Documents", HNF-PRO-233, this test plan is assigned Approval Designator N/A. Approval signatures are required from the author, immediate manager, and customer.

All testing will follow the "Process Chemistry & Statistics Quality Assurance Plan", WHC-SD-CP-QAPP-018, Rev. 0 (Meznarich 1996). Specific laboratory instructions will be reviewed and approved by Process Chemistry management, 222-S Radiological Control, 222-S Environmental, and 222-S Industial Safety prior to initiation of testing. The instructions will be recorded in a controlled laboratory notebook before work begins; observations and data will be recorded as the work is done.

5.0 SAFETY

All of the safety requirements that apply to this test plan are described in "Development of Instrumentation, Methods and Performance of Process Testing", Hanford Analytical Services Laboratory Operating Procedure LO-140-100, Rev. B-0, released July 8, 1997.

6.0 WASTE HANDLING

Three waste streams are expected to be generated from this test program. Stream #1 is the waste resulting from analysis of samples. Each waste generated from these routine analyses will be handled in accordance with the instructions in the procedures for the respective analyses.

Stream #2 is the used and left-over tank waste material. This material, expected to be less than 1 kg, will be discarded to the 219-S Building tanks via a hotcell drain, per procedure LO-100-107, "Cubicle Housekeeping, Waste Disposal, and Management".

Stream #3 is the solid hotcell waste consisting of used centrifuge cones, used sample vials, and used plastic syringes. This waste will be rinsed with water, loaded into waste cans, removed from the hotcell, and disposed of according to LO-100-151.

Other than the waste streams just described, there will be no accumulating of hazardous waste. Therefore, no Waste Stream Fact Sheet or Waste Compatibility Assessment documentation will be required.

7.0 SCHEDULE AND DELIVERABLES

This test plan is scheduled to be issued by December 31, 1997. Management approval for the specific laboratory instructions will be obtained by January 15, 1998. Laboratory work will commence by January 20, 1998.

A complete report of the test results will be issued in the form of a draft supporting document by May 20, 1998. Informal interim status reports will be issued to the customer via cc:Mail on a weekly basis.

8.0 REFERENCES

Certa, P. J. (1998), *Data Quality Objectives for TWRS Privatization Phase I: Confirm Tank T is an Appropriate Feed Source for Low-Activity Waste Feed Batch X*, PSDQO-01, HNF-1796, Rev. 0 (DRAFT).

Garfield, J. S. (1997), *Tank 241-AN-105 Dilution/Dissolution Test Requirements*, Numatec Hanford Corporation Internal Memo 8C451-97-004, March 27.

Herting, D. L. (1997), *Results of Dilution Studies with Waste from Tank 241-AN-105*, HNF-SD-WM-DTR-046, Rev. 0, Numatec Hanford Corporation, Richland, Washington.

Hu, T. A. (1997), L. W. Shelton and T. L. Welsh, *Tank Characterization Report for Double-Shell Tank 241-AN-104*, HNF-SD-WM-ER-690, Rev. 0, Lockheed Martin Hanford Corporation, Richland, Washington.

Jo, J. (1997), L. W. Shelton, T. L. Welsh and J. Stroup, *Tank Characterization Report for Double-Shell Tank 241-AN-105*, HNF-SD-WM-ER-678, Rev. 0, Lockheed Martin Hanford Corporation, Richland, Washington.