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ENGINEERING CHANGE NOTICE

0050795

1. ECN 653813

Proj. ECN

2. ECN Category (mark one)	3. Originator's Name, Organization, MSIN, and Telephone No.	4. USQ Required?	5. Date
Supplemental [] Direct Revision [X] Change ECN [] Temporary [] Standby [] Supersedure [] Cancel/Void []	Leela M. Sasaki, Data Assessment and Interpretation, R2-12, 373-1027	[] Yes [X] No	05/26/99
	6. Project Title/No./Work Order No. Tank 241-C-106	7. Bldg./Sys./Fac. No. 241-C-106	8. Approval Designator N/A
	9. Document Numbers Changed by this ECN (includes sheet no. and rev.) WHC-SD-WM-ER-615, Rev. 0-B	10. Related ECN No(s). ECNs: 612292, 649448	11. Related PO No. N/A

12a. Modification Work	12b. Work Package No.	12c. Modification Work Complete	12d. Restored to Original Condition (Temp. or Standby ECN only)
[] Yes (fill out Blk. 12b) [X] No (NA Blks. 12b, 12c, 12d)	N/A	N/A Design Authority/Cog. Engineer Signature & Date	N/A Design Authority/Cog. Engineer Signature & Date

13a. Description of Change

13b. Design Baseline Document? [] Yes [X] No

This ECN has been generated in order to update the document to reflect results of recent data/information evaluation.

Replace pages:

ES-1 through ES-4, ES-9, ES-10, ES-11, ES-12, 1-1, 1-2, 3-11 through 3-14, 4-1, 4-2, 4-23, 4-24, 5-35, 5-36, 6-1 through 6-4, and 7-1 through 7-8

JUN 1999 RECEIVED EDMC

14a. Justification (mark one)

Criteria Change	[X]	Design Improvement	[]	Environmental	[]	Facility Deactivation	[]
As-Found	[]	Facilitate Const	[]	Const. Error/Omission	[]	Design Error/Omission	[]

14b. Justification Details

A tank characterization report page change revision is required to reflect the results of recent evaluation of data/information pertaining to adequacy of tank sampling for safety screening purposes (Reynolds et al. 1999, Evaluation of Tank Data for Safety Screening, HNF-4217, Rev. 0, Lockheed Martin Hanford Corporation, Richland, Washington).

15. Distribution (include name, MSIN, and no. of copies)

See attached distribution.

RELEASE STAMP

DATE JUN 3 1999 HANFORD RELEASE STA: 4 ID: 58

ENGINEERING CHANGE NOTICE

1. ECN (use no. from pg. 1) ECN-653813

16. Design Verification Required	17. Cost Impact				18. Schedule Impact (days)	
	ENGINEERING		CONSTRUCTION			
[] Yes	Additional	[] $	Additional	[] $	Improvement	[]
[X] No	Savings	[] $	Savings	[] $	Delay	[]

19. Change Impact Review: Indicate the related documents (other than the engineering documents identified on Side 1) that will be affected by the change described in Block 13. Enter the affected document number in Block 20.

SDD/DD	[]	Seismic/Stress Analysis	[]	Tank Calibration Manual	[]
Functional Design Criteria	[]	Stress/Design Report	[]	Health Physics Procedure	[]
Operating Specification	[]	Interface Control Drawing	[]	Spares Multiple Unit Listing	[]
Criticality Specification	[]	Calibration Procedure	[]	Test Procedures/Specification	[]
Conceptual Design Report	[]	Installation Procedure	[]	Component Index	[]
Equipment Spec.	[]	Maintenance Procedure	[]	ASME Coded Item	[]
Const. Spec.	[]	Engineering Procedure	[]	Human Factor Consideration	[]
Procurement Spec.	[]	Operating Instruction	[]	Computer Software	[]
Vendor Information	[]	Operating Procedure	[]	Electric Circuit Schedule	[]
OM Manual	[]	Operational Safety Requirement	[]	ICRS Procedure	[]
FSAR/SAR	[]	IEFD Drawing	[]	Process Control Manual/Plan	[]
Safety Equipment List	[]	Cell Arrangement Drawing	[]	Process Flow Chart	[]
Radiation Work Permit	[]	Essential Material Specification	[]	Purchase Requisition	[]
Environmental Impact Statement	[]	Fac. Proc. Samp. Schedule	[]	Tickler File	[]
Environmental Report	[]	Inspection Plan	[]		[]
Environmental Permit	[]	Inventory Adjustment Request	[]		[]

20. Other Affected Documents: (NOTE: Documents listed below will not be revised by this ECN.) Signatures below indicate that the signing organization has been notified of other affected documents listed below.

Document Number/Revision	Document Number/Revision	Document Number Revision
N/A		

21. Approvals

	Signature	Date		Signature	Date
Design Authority			Design Agent		
Cog. Eng.	L.M. Sasaki L.M. Sasaki	6/3/99	PE		
Cog. Mgr.	K.M. Hall	6/3/99	QA		
QA			Safety		
Safety			Design		
Environ.			Environ.		
Other			Other		

DEPARTMENT OF ENERGY

Signature or a Control Number that tracks the Approval Signature

ADDITIONAL

Tank Characterization Report for Single-Shell Tank 241-C-106

Leela M. Sasaki
Lockheed Martin Hanford Corp., Richland, WA 99352
U.S. Department of Energy Contract 8023764-9-K001

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Abstract: N/A

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RECORD OF REVISION	(1) Document Number WHC-SD-WM-ER-615	Page 1

(2) Title
Tank Characterization Report for Single-Shell Tank 241-C-106

CHANGE CONTROL RECORD

(3) Revision	(4) Description of Change - Replace, Add, and Delete Pages	Authorized for Release (5) Cog. Engr.	(6) Cog. Mgr. Date
0	(7) Initially released 9/25/96 on EDT 617548.	R. D. Schreiber	J. G. Kristofzski
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		L M Sasaki	6/3/99

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EXECUTIVE SUMMARY

This tank characterization report summarizes information on the historical uses, current status, and sampling and analysis results of waste stored in single-shell underground tank 241-C-106. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order*, Milestone M-44-09 (Ecology et al. 1996).

Tank 241-C-106 is the only tank on the High-Heat Load Watch List. Grab samples were obtained from the tank in 1996. As a result of the analyses performed on these samples, the supernate and upper 60 percent of the sludge in the tank do not pose any safety concerns in addition to the high-heat load issue based on the decision limits of the safety screening data quality objective (DQO) (Dukelow et al. 1995). The lower 40 percent of the sludge was not sampled. However, tank safety was evaluated and extensive safety documentation was prepared in preparation for sluicing. Chemical aspects are summarized in Reynolds (1997). Sluicing of the tank waste to mitigate the tank's high heat load began in 1998.

Tank 241-C-106 is one of 12, 100-series, single-shell, underground waste storage tanks located in the 200 East Area C Tank Farm on the Hanford Site. It is the last tank in a three-tank cascade series beginning with tanks 241-C-104 and 241-C-105. Tank 241-C-106 was constructed between 1943 and 1944 and was put into service in September 1947.

Tank 241-C-106 received its first waste in June 1947 -- metal waste from the cascade overflow of tank 241-C-105. The metal waste was sluiced for uranium recovery in the first

quarter of 1953, and the tank became the metal waste supernate blend tank. During the third quarter of 1954, tank 241-C-106 received uranium recovery waste. In the second quarter of 1957, waste from tank 241-C-106 was transferred to tanks 241-C-109 and 241-C-112 for ferrocyanide scavenging of radiocesium. From the second quarter of 1958 until the second quarter of 1960, tank 241-C-106 received cladding waste from the Plutonium Uranium Extraction (PUREX) facility. During the first quarter of 1965, the tank received decontamination waste. From the first quarter of 1969 until the second quarter of 1972, the tank received washed PUREX sludge; this sludge is thought to be the primary source of the heat-generating ^{90}Sr in the tank (Agnew et al. 1996). The tank received wastewater from the third quarter of 1971 until the second quarter of 1975. From the third quarter of 1974 until the second quarter of 1976, the tank received B-Plant low-level waste. From 1976 to 1978, the tank received B-Plant strontium recovery waste, supernate, and complexed and evaporator wastes from tank 241-A-102. Water is periodically added to the tank to cool the tank waste by evaporation. As of May 31, 1996, the tank waste was classified as non-complexed (Hanlon 1996). However, the 1996 grab analysis results exceeded the 100 nCi/g specification limit for classification as transuranic (TRU) waste (Fowler 1995).

A description and status of tank 241-C-106 are presented in Table ES-1 and Figure ES-1. The tank has an operating capacity of 2,006 kL (530 kgal) and as of May 31, 1996, contained approximately 867 kL (229 kgal) of waste. Of this volume, approximately 121 kL (32 kgal) was supernate and 746 kL (197 kgal) was sludge (Hanlon 1996). The sludge is estimated to contain approximately 182 kL (48 kgal) of drainable interstitial liquid. Waste

surface levels have remained relatively constant over the past three years, although the supernate surface level fluctuates somewhat as cooling water is added and evaporates.

Table ES-1. Description and Status of Tank 241-C-106

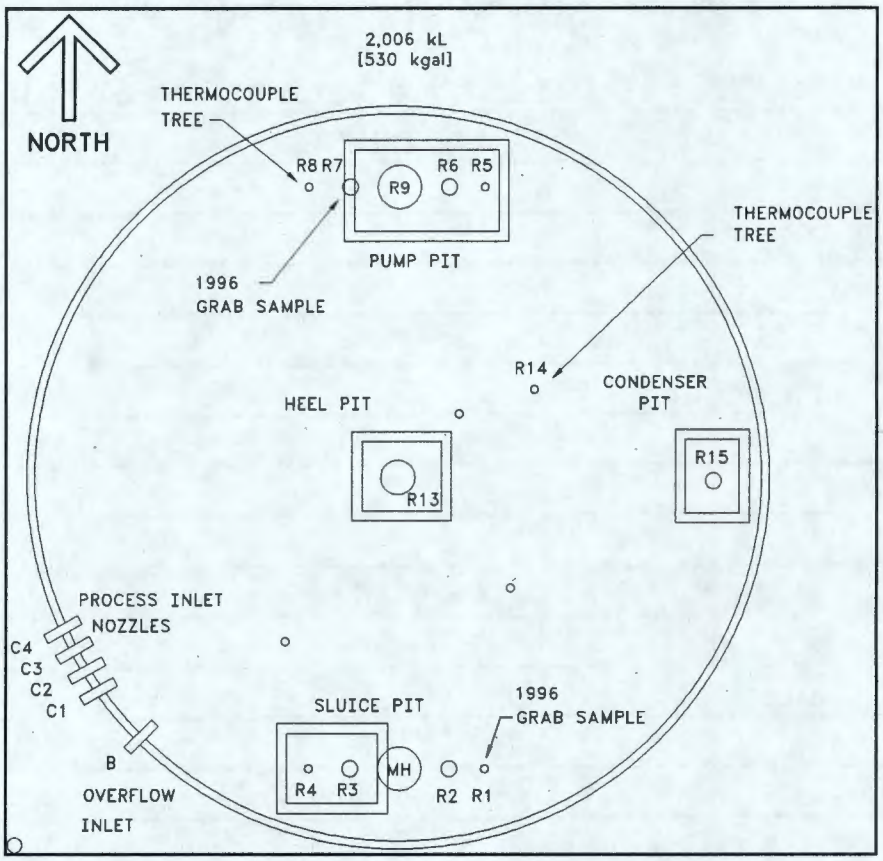
TANK DESCRIPTION	
Type	Single-shell
Constructed	1943 to 1944
In-service	1947
Diameter	23 m (75 ft)
Maximum operating depth	5.2 m (17 ft)
Capacity	2,006 kL (530 kgal)
Bottom shape	Dish
Ventilation	Active
TANK STATUS	
Waste classification	Non-complexed[1]
Total waste volume	867 kL (229 kgal)
Supernate volume	121 kL (32 kgal)
Sludge volume	746 kL (197 kgal)
Drainable interstitial liquid	182 kL (48 kgal)
Waste surface level (June 12, 1996)[2]	195 cm (76.6 in.)
Temperature (1982 to 1996)	7 °C (45 °F) to 98 °C (208 °F)
Integrity	Sound
Watch List	High-Heat Load
SAMPLING DATES	
Grab sample	February/March 1996
Tank headspace vapor samples	February 1994 and March 1996
SERVICE STATUS	
Interim stabilized	not completed
Partially isolated	August 1983
Declared Inactive	March 1979

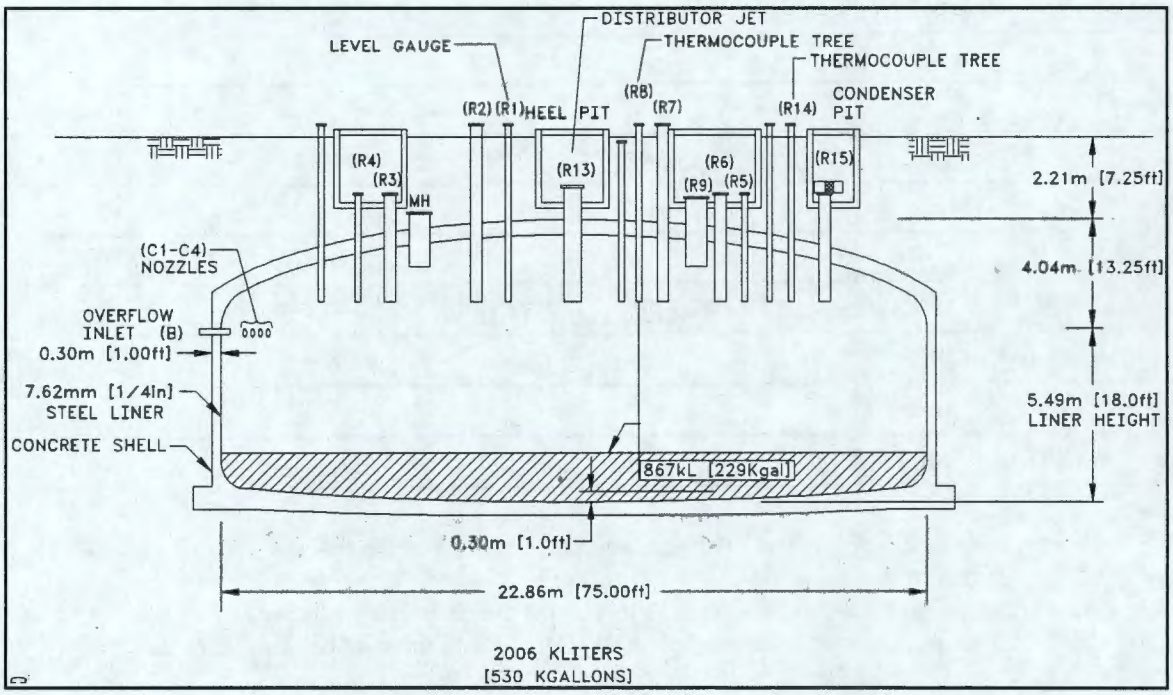
Note:

[1]Hanlon (1996). However, the 1996 grab sampling results indicate that the TRU content exceeds the 100 nCi/g limit in Fowler (1995).

[2]Referenced to an offset that is 30 cm (12 in.) above the centerline of the tank (i.e., add 30 cm to the above measurement to obtain the surface level referenced to the tank bottom centerline).

Figure ES-1. Profile of Tank 241-C-106.





trees ranged from 7 °C (45 °F) to 98 °C (208 °F) from 1982 to 1996. On June 12, 1996, temperatures ranged from 26 °C (78.8 °F) to 66.4 °C (151 °F). In general, tank temperature appears to be decreasing with time.

As required by the safety screening DQO, the flammability of the tank headspace gases was measured. Vapor samples taken from the tank headspace were measured for flammability by means of a combustible gas meter prior to grab sampling. Results of zero percent of the lower flammability limit (LFL) were measured for flammable gases in the tank headspace; these results are expected because tank 241-C-106 is actively ventilated. This result is well below the safety screening DQO decision threshold of 25 percent of the LFL. Samples of the tank headspace gases were also taken from the tank exhauster port in February 1994 and March 1996 and analyzed for flammable gases. Both sets of gas analyses showed the concentration of flammable species in the headspace to be less than 10 parts per million by volume (ppmv). During the 1996 vapor sampling event, hydrogen and methane were both measured at less than 10 ppmv. These results are far below the safety screening notification limits of 10,000 ppmv for hydrogen and 12,500 ppmv for methane (Buckley 1996).

Total alpha activity in the samples was determined in order to gauge the potential for criticality in the tank waste. The total alpha activity results for the grab samples were well below the safety screening limits. The highest supernate sample total alpha activity result was 1.03 μCi/mL; the safety screening limit for the supernate was 61.5 μCi/mL. The highest sludge subsample total alpha activity result was 4.38 μCi/g, and the corresponding upper limit to a one-sided 95 percent confidence interval on the mean was 16.7 μCi/g; the

safety screening limit for the sludge was 39.8 μCi/g. These results indicate that the potential for a criticality event is low. However, these results do exceed the 100 nCi/g specification limit for classification as TRU waste.

The TOC results were compared with the safety criteria limit of 3.0 weight percent (30,000 μg C/g) on a dry-weight basis (Turner et al. 1995). The overall mean dry-weight result for the supernate was 12,700 μg C/g, and for the sludge was 23,500 μg C/g. The upper limit to a one-sided, 95 percent confidence interval on the mean for the supernate was 29,200 μg C/g, and for the sludge was 29,300 μg C/g. The upper limit to the one-sided, 95 percent confidence interval on the mean dry-weight TOC values for the supernate and the sludge did not exceed the 30,000 μg C/g decision threshold.

During the preparation of the sludge samples for analysis, a second liquid phase was discovered and reported. This phase was designated a potential organic layer and constituted approximately 0.5 to 3 percent of the sludge volume. Reynolds (1997) reports that this layer consisted largely of bis(2-ethylhexyl)phosphate. When subjected to propagating reaction tests, bis(2-ethylhexyl)phosphate did not produce a propagating exothermic reaction.

In summary, the requirements of the safety screening DQO were met for the supernate and upper 60 percent of the sludge in tank 241-C-106, tank safety was further evaluated in preparation for sluicing, and sluicing of the tank waste began in 1998 to reduce the heat load in the tank. No further sampling of tank 241-C-106 is required for the purpose of safety screening (Reynolds et al. 1999).

The waste compatibility evaluation revealed several issues that may affect waste management decisions:

- Exotherm-to-endotherm ratios as determined by DSC were less than the decision threshold value of one for all samples portions (except one that failed the quality control requirements).
- Significant amounts of organic carbon, predominately in the form of oxalate, were found in the tank 241-C-106 waste -- 2,910 μg C/mL (wet-weight) in the supernate, and 20,000 μg C/mL (wet-weight) in the sludge. Therefore, the waste in the tank may need to be segregated from non-complexant waste types when transferred to the double-shell tank system.
- The waste compatibility criticality decision threshold value was 0.800 μCi/mL of $^{239/240}$Pu. The overall mean $^{239/240}$Pu value for the supernate was 0.745 μCi/mL, nearly equalling the threshold value. The overall mean $^{239/240}$Pu value for the sludge was 1.67 μCi/mL, which exceeded the threshold value. The overall mean TRU content of the waste was found to be 0.648 μCi/g for the supernate and 1.08 μCi/g for the sludge; both values exceed the 0.1 μCi/g TRU limit in the DQO.
- The overall supernate mean pH of 10.2 and the caustic demand results of Herting (1996) indicate that the waste is very caustic deficient.

- The heat-load estimates for the tank ranged from 32,200 to 38,700 W (110,000 to 132,000 Btu/hr) (Bander 1993a; Ogden et al. 1996). The decision threshold for the receiver tank, 241-AY-102, is 205,000 W (700,000 Btu/hr). These estimates are based on temperature profiles of the tank waste and assume non-uniform distribution of the heat-generating radionuclides in the waste. Therefore, these estimates differ from those provided earlier which are based on the ^{90}Sr and ^{137}Cs activities in the waste and assume uniform distribution of the radionuclides.
- Waste from tank 241-C-106 exceeds the 100 nCi/g segregation limit, and could be designated as TRU waste.

1.0 INTRODUCTION

This tank characterization report summarizes the information on the historical uses, current status, and sampling and analysis results of waste stored in single-shell tank 241-C-106. The tank was grab sampled in 1996 to satisfy the requirements of the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995), the *Data Quality Objectives for Tank Farms Waste Compatibility Program* (Fowler 1995), and *Tank 241-C-106 Grab Sample — Technical Letter of Instruction* (Cash and Babad 1996). In addition, the tank was vapor sampled in 1996 and 1994 in support of Osborne et al. (1994). This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order*, Milestone M-44-09 (Ecology et al. 1996).

Tank 241-C-106 is on the High-Heat Load Watch List. It has been removed from service and is partially isolated. Water is periodically added to the tank for cooling purposes; however, waste removals or additions that substantially affect the tank inventory are unlikely until retrieval activities commence. The concentration estimates reported in this document reflect the composition of the waste based on the most current data.

1.1 PURPOSE

This report summarizes information about the use and contents of tank 241-C-106. When possible, this information will be used to assess issues associated with safety, operations, environmental, and process activities.

1.2 SCOPE

Fifteen grab samples, consisting of both supernate and sludge material, were obtained in February and March 1996. Of these fifteen samples, two supernate and five sludge samples were analyzed in accordance with the requirements of the safety screening and waste compatibility data quality objectives (DQOs) and the technical letter of instruction. The main objectives of the sampling event were four-fold (Schreiber 1996a):

- To verify the High-Heat Load Watch List tank status and identify any additional safety issues associated with tank 241-C-106;
- To ensure that no compatibility issues arise regarding the mixing of tank 241-C-106 waste with tank 241-AY-102 waste during retrieval;
- To spot check the ^{137}Cs content in solution to verify dose estimates during retrieval;

- To measure the ^{90}Sr and transuranic (TRU) content to determine whether these chemical constituents are present in significant quantities in the topmost sludge layer in the tank.

The primary grab sample analyses included the following: differential scanning calorimetry (DSC) to evaluate fuel content and energetics; thermogravimetric analysis (TGA) and gravimetry to determine moisture content; total alpha activity, $^{239/240}Pu$ analysis, and density measurements by centrifugation to evaluate criticality potential; ion chromatography (IC) to determine anion concentrations; inductively coupled plasma/atomic emission spectrometry (ICP/AES) to determine metal concentrations; gamma energy analysis to obtain radiochemical (^{137}Cs) activities; persulfate oxidation and furnace combustion to determine the total organic carbon (TOC) and total inorganic carbon (TIC) concentrations; a visual inspection to determine the presence of a separable organic layer; and separation and beta counting to determine ^{90}Sr content.

The remaining samples obtained from tank 241-C-106 were mainly used for non-routine analyses related to the retrieval of tank 241-C-106 sludge for transfer to tank 241-AY-102. The details and results of these analyses are discussed in *Chemical and Chemically Related Considerations Associated with Sluicing Tank C-106 to Tank AY-102* (Reynolds 1997).

In addition to the February/March 1996 grab sampling event, vapor sampling events of the tank 241-C-106 headspace gases were performed in February 1994 and March 1996. The March 1996 event obtained one vapor sample from the tank exhaust to assess the flammability of the tank exhaust gases. In February 1994, a complete in-tank headspace gas and vapor characterization was performed in accordance with the *Data Quality Objectives for Generic In-Tank Health and Safety Vapor Issues Resolution* (Osborne et al. 1994). During this event, organic, flammable, and inorganic vapors were analyzed. The 1994 results are summarized in this tank characterization report, and the full details may be found in *Tank 241-C-106 Headspace Gas and Vapor Characterization Results for Samples Collected in February 1994* (Huckaby and Bratzel 1995).

3.1.2 Sample Analysis (February/March 1996 Grab Samples)

This section summarizes the analytical methods used to analyze the samples after the sample handling and preparation described in Section 3.1.1. The analyses were performed in support of the safety screening and waste compatibility DQOs and a letter of instruction from the Tank Waste Remediation Systems Safety Program Office, as described in Section 3.1 and Table 3-1.

Table 3-5 summarizes the analytical procedures performed and the procedure numbers used on the samples. Appendix B charts the breakdown of the grab samples and the analyses performed on the prepared samples. The weight percent of water was determined using TGA and gravimetry. The fuel content or energetics of the waste was determined by DSC. Metals were determined using ICP; the solid samples were prepared by acid digestion. Anions were determined using IC; solid samples were prepared using a water leach, and liquid samples were analyzed directly. The TOC was determined using either furnace oxidation or hot persulfate oxidation followed by coulometric titration. The TIC was determined by sulfamic acid/sulfuric acid evolution of carbon dioxide with coulometric titration detection. Sample pH was determined using a glass pH electrode. For solid samples, pH was determined in a water leach of the solid sample. Ammonium was determined with an ammonium-selective electrode and the method of standard additions.

Radiochemical determinations were performed on direct mounts or chemical separations of liquid samples. The sludge samples were dissolved by using an alkali metal fusion (potassium or sodium hydroxide) procedure followed by dissolution with dilute hydrochloric acid, and an aliquot of the dissolved sludge was then mounted or separated for counting. Total alpha activity was determined using an alpha proportional counter. ^{90}Sr was determined using a strontium carbonate carrier precipitation followed by beta counting. ^{99}Tc was determined by extracting the ^{99}Tc into an organic extractant followed by liquid scintillation counting. ^{137}Cs was determined by gamma energy analysis. $^{239/240}Pu$ and ^{241}Am were determined by ion-exchange separation followed by total alpha counting and alpha energy analysis.

In addition to the standard analytical determinations tabulated in Table 3-5, Schreiber (1996a) and Crawford (1996a) required several other analyses be performed on the tank 241-C-106 samples. The potential organic layers from samples 6C-96-7 and 6C-96-13 and methylene chloride extracts of the sludges from samples 6C-96-8 and 6C-96-14 were sent to PNNL for further analysis by GC/MS and TOC. The results are reported in Reynolds (1997).

Viscosity measurements were performed on the supernate and sludge from samples 6C-96-8 and 6C-96-14. Particle size distributions were determined on only the sludge from samples 6C-96-8 and 6C-96-14 (O'Rourke 1996). The majority of the viscosity results are completed and presented in Esch (1996) and are not discussed in this tank characterization report. The remaining viscosity analyses are pending and the results will be reported at a later date.

Table 3-5. Tank 241-C-106 February/March 1996 Grab Sample Analytical Procedures.[1] (2 sheets)

Analysis	Matrix	Preparation Procedure/Revision	Analysis Procedure/Revision
Energetics by DSC	Liquid/solid	n/a	LA-514-113 / B-1, C-1 LA-514-114 / B-1
% water by TGA	Liquid/solid	n/a	LA-514-114 / C-1 LA-560-112 / B-1
Specific gravity	Liquid	n/a	LA-510-112 / C-3
% water by gravimetry	Liquid/solid	n/a	LA-564-101 / F-1
pH	Liquid solid	n/a	LA-212-106 / A-0 LA-212-105 / A-0
Particle size	Solid	n/a	LT-519-101 / A-1
Viscosity	Liquid/solid	n/a	LT-519-115 / A-0
Ammonium	Liquid	n/a	LA-631-001 / B-2
IC anions	Liquid solid	n/a LA-504-101 / E-0[2]	LA-533-105 / D-1
ICP metals	Liquid solid	n/a LA-505-159 / D-0[3]	LA-505-151 / D-3 LA-505-161 / B-0
Total organic carbon	Liquid (furnace) Liquid/solid (persulfate)	n/a	LA-344-105 / C-0 LA-342-100 / C-0, D-0
Total inorganic carbon	Liquid (acid) solid (acid persulfate)	n/a	LA-622-102 / C-0 LA-342-100 / C-0, D-0
Total alpha activity	Liquid solid	n/a LA-549-141 / F-0[4]	LA-508-101 / D-2
^{90}Sr	Liquid solid	n/a LA-549-141 / F-0[4]	LA-220-101 / D-1
^{99}Tc	Liquid solid	n/a LA-549-141 / F-0[4]	LA-438-101 / D-2
^{137}Cs	Liquid solid	n/a LA-549-141 / F-0[4]	LA-548-121 / D-1, E-0

Table 3-5. Tank 241-C-106 February/March 1996 Grab Sample Analytical Procedures.[1] (2 sheets)

Analysis	Matrix	Preparation Procedure/Revision	Analysis Procedure/Revision
^{241}Am	Liquid solid	n/a LA-549-141 / F-0[4]	LA-953-103 / A-5, B-0
$^{239/240}Pu$	Liquid solid	n/a LA-549-141 / F-0[4]	LA-943-128 / A-1
NPH/TBP	Liquid	methylene chloride extraction	LA-523-437 / B-0
Semivolatile organics	Liquid	methylene chloride extraction	LA-523-406 / A-0

Notes:

n/a = not applicable
NPH = normal paraffin hydrocarbon
TBP = tributyl phosphate

[1]Esch (1996)

[2]Water digest procedure.

[3]Acid digest procedure.

[4]Fusion digest procedure.

Caustic demand was measured for the sludge from samples 6C-96-8 and 6C-96-14 (Herting 1996). Caustic demand was determined by mixing sludge samples with known volumes of 2.78 mole/L sodium hydroxide for 24 hours, centrifuging the mixtures for two hours, and measuring the free hydroxide remaining in supernate using 222-S Laboratory procedure LA-211-102, Rev. C-0. The total inorganic carbon and ICP metals in the supernates were also measured.

Compatibility studies of tank 241-C-106 sludge with tank 241-AY-102 waste were performed and included several tests (Crawford 1996a, Crawford 1996b). Foaming-upon-agitation tests were performed on supernate from samples 6C-96-8 and 6C-96-14. The mixing and settling behavior of tank 241-C-106 sludge (samples 6C-96-8 and 6C-96-14) with tank 241-AY-102 supernate was investigated. The investigations included measuring the volume percent settled solids of the mixture, the pH and density of supernate before and after mixing, and the weight percent water (by TGA), energetics (by DSC), and the density of the sludge layer after mixing.

The compatibility studies were repeated with two mixtures of tank 241-C-106 and tank 241-AY-102 sludges. One mixture consisted of four parts of tank 241-C-106 sludge from sample 6C-96-8 mixed with one part tank 241-AY-102 sludge; the other mixture was four parts sludge from sample 6C-96-14 to one part tank 241-AY-102 sludge. Finally, the sedimentation characteristics of the waste mixtures in tanks 241-C-106 and 241-AY-102 were determined by centrifugation. Reynolds (1997) reports the results of these studies.

3.2 DESCRIPTION OF VAPOR SAMPLING EVENTS

This section describes the vapor sampling events associated with tank 241-C-106. The sampling events (from March 1996 and February 1994) are discussed in reverse chronological order.

3.2.1 Description of the March 1996 Vapor Sampling Event

Matheson (1996) reports the March 1996 vapor sampling event and the associated sample results, and Section 4.4.2 summarizes these results. Two sets of gas samples were collected for hydrogen analysis in March 1996 from Tank 241-C-106 to address continuing in-tank hydrogen generation issues related to potential flammability concerns and to compare these data with results from the 1994 headspace hydrogen generation data.

The samples were drawn from the tank exhauster port and collected using evacuated Whitey[1] stainless-steel sample bottles and SUMMA[2] gas collection canisters. The tank 241-C-106 exhauster flow rate the day before sampling was 68.64 m^3/min (2,424 ft^3/min). The dry-bulb temperature in the exhaust pipe was 16 °C (61 °F). At the time the samples were collected, the ambient air temperature was 14 °C (58 °F), the humidity was near 100%, and the ambient air pressure was 99.9 kPa (749 Torr).

3.2.2 Description of the February 1994 Vapor Sampling Event

Huckaby and Bratzel (1995) report the collection of vapor samples during February 1994 and report the results for this sampling event; Section 4.4.3 summarizes these results. Tank 241-C-106 headspace gas and vapor samples were collected and analyzed in accordance with *Data Quality Objectives for Generic In-Tank Health and Safety Vapor Issues Resolution* (Osborne et al. 1994). This DQO directed the collection and analysis of headspace vapor samples to help determine the potential risks of fugitive emissions to tank farm workers.

[1]Whitey is a registered trademark of Whitey Company, Highland Heights, Ohio.

[2]SUMMA is a trademark of Molectrics, Inc., Cleveland, Ohio.

4.0 ANALYTICAL RESULTS

4.1 OVERVIEW

This section summarizes the analytical results associated with the grab samples taken in February and March 1996 from tank 241-C-106. Reynolds (1997) presents much of the non-routine analytical data from the February/March samples in addition to brief summaries of the key analytical results. Pertinent findings from that report will be reproduced here. However, Reynolds (1997) also presents detailed discussions of the sample analysis for organic content and the compatibility and settling studies with tank 241-AY-102 supernate and sludge. Those results are not discussed in this tank characterization report.

This report does include the data that supported the applicable data quality objectives. The February/March 1996 grab samples were taken and analyzed to satisfy the requirements of the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995), *Data Quality Objectives for Tank Farms Waste Compatibility Program*, (Fowler 1995), and *Tank 241-C-106 Grab Sample — Technical Letter of Instruction* (Cash and Babad 1996). The sampling and analyses were performed according to the *Tank 241-C-106 Grab Sampling and Analysis Plan* (Schreiber 1996a) and the *Sample Preparation of Tank 241-C-106 Grab Samples and Testing for Compatibility with Tank 241-AY-102 Supernate* test plan (Crawford 1996a).

Table 4-1 summarizes the data covered in this report and shows the table locations for each set of analytical results. Data from the analysis of all 1996 grab samples were reported in Esch (1996) and can also be found in the Tank Characterization Database[1].

In addition to the results of the 1996 grab sampling event, this section summarizes the analyses and the results of the March 1996 and February 1994 vapor sampling events from tank 241-C-106. Data from the 1996 vapor sample analyses can be found in Matheson (1996). Results of the 1994 sampling event are discussed in Huckaby and Bratzel (1995). The 1996 vapor samples were taken from the tank 241-C-106 exhauster port to address continuing in-tank hydrogen generation issues related to potential fuel flammability concerns. The governing document for this event was *Tank C-106 Flammable Vapor Sampling and Analysis Plan* (Homi 1996). The February 1994 full-vapor characterization event was performed in accordance with *Data Quality Objectives for Generic In-Tank Health and Safety Vapor Issues Resolution* (Osborne et al. 1994).

[1]The Tank Characterization Database can be found on the Internet at website http://twins.pnl.gov:8001/refmain.htm.

Table 4-1. Analytical Data Tables.

Analysis	Table Location
1996 grab sample chemical data summary -- Supernate -- Sludge -- Total tank	 Table 4-2 Table 4-3 Table 4-4
1996 grab sample thermogravimetric analysis results	Table 4-5
1996 grab sample differential scanning calorimetry results	Table 4-6
1996 vapor sample data -- Flammability testing -- Exhaust gas hydrogen testing	 Table 4-7 Table 4-8
1994 vapor sample data -- Inorganic compounds -- Organic compounds	 Table 4-9 Table 4-10
1996 grab sample comprehensive analytical data	Appendix A
1996 grab sample breakdown flowcharts	Appendix B
Statistical analysis	Appendix C

4.2 1996 GRAB SAMPLE CHEMICAL DATA SUMMARY

Tables 4-2, 4-3, and 4-4 list the analytes that are found in the supernate, the sludge, and the total tank, respectively. Samples were identified as supernate or sludge using visual observations. For each analyte, the following information is provided: the restricted estimated maximum likelihood (REML) mean, the REML-derived relative standard deviation (RSD) of the mean, and the projected inventory in kilograms or curies.

The means and uncertainties reported are derived from statistical analysis of variance (ANOVA) methods (see Section 5.3 and Appendix C). When more than half of the subsamples had detected results, the mean was reported as a detected value. When more than half the subsamples had non-detected results, a less-than (<) sign was placed preceding the mean. In both situations, non-detected results were used in the mean calculation by using the detection limit for the assay as the mean concentration. Since the detection limit is the highest value possible for a non-detect result, these mean concentration results may be

The DSC results are reported on a wet-weight basis. The safety screening DQO, however, requires that the exothermic reactions be evaluated on a dry-weight basis in order to make a decision about tank safety. The dry-weight value is obtained from the wet-weight value by dividing the reported exothermic value for a subsample by the solid fraction of the subsample (that is, one minus the fractional percent water value for that subsample). The largest mean exotherm observed for the subsamples was -681.15 J/g (dry) for the potential organic layer from sample 6C-96-13. It is important to note, though, that the potential organic layer represents 0.5 to 3 percent of the total tank volume and was only discovered after vigorous centrifugation and sample treatment (i.e., it is not representative of the tank waste matrix).

4.3.3 Separable Organic Layer

Prior to breakdown, each grab sample was visually inspected for the presence of a separable organic layer. Separable organic layers were not observed in any of the raw samples taken from tank 241-C-106. However, upon centrifugation of the sludge material at tank temperature (approximately 55 °C [130 °F]), a separable phase was found (Schreiber 1996b through 1996e). The separable phase was identified as a potential organic layer. This layer was estimated to be approximately 0.5 to 3 percent of the total sample volume, and its color was described as very dark reddish-brown. Analysts at PNNL determined that the potential organic layer was composed primarily of sodium bis(2-ethylhexyl)phosphate, an extracting agent used in B-Plant as part of the ^{90}Sr separation process. Minor amounts of tributyl phosphate, normal paraffin hydrocarbons, and the trans-esterification products of tributyl phosphate were also found in the samples. Reynolds (1997) presents further information regarding these and other analyses performed on the potential organic layer samples.

4.4 TANK HEADSPACE VAPOR DATA SUMMARY

This section summarizes the data resulting from three different tank vapor sampling and analysis events. Section 4.4.1 describes the results from the tank headspace vapor field screening conducted in association with the February/March 1996 grab samples. Sections 4.4.2 and 4.4.3 outline the results from the March 1996 and February 1994 tank exhauster vapor samples.

4.4.1 February/March 1996 Tank Headspace Vapor Field Screening Data

The safety screening DQO threshold decision value for flammable gas screening in the tank headspace is 25 percent of the LFL (Dukelow et al. 1995). Prior to obtaining the grab samples, tank headspace vapors were field screened using a combustible gas meter and an organic vapor meter. Table 4-7 presents these results. The result of zero percent of the LFL in the tank headspace did not exceed the safety screening threshold value of 25 percent of the LFL. This result is expected because tank 241-C-106 is actively ventilated.

Table 4-7. Flammability Testing Vapor Survey Results for Tank 241-C-106.

Riser	Location	Measurement	Result
1	Tank headspace[1]	Flammable vapor concentration as percent of LFL	0%
		Volume percent oxygen	20.7-20.9%
		Total organic vapor	0 ppm
		Ammonia gas	0 ppm
1	Exhauster vent	Flammable vapor concentration as percent of LFL	0%
		Volume percent oxygen	20.8-20.9%
		Total organic vapor	0-1.1 ppm
		Ammonia gas	<5 ppm
1	Breathing zone	Flammable vapor concentration as percent of LFL	0%
		Volume percent oxygen	20.9%
		Total organic vapor	0 ppm
		Ammonia gas	0 ppm
7	Tank headspace[1]	Flammable vapor concentration as percent of LFL	0%
		Volume percent oxygen	20.9%
		Total organic vapor	0 ppm
		Ammonia gas	<5 ppm
7	Exhauster vent	Flammable vapor concentration as percent of LFL	0%
		Volume percent oxygen	21.0%
		Total organic vapor	0 ppm
		Ammonia gas	2 ppm
7	Breathing zone	Total organic vapor	0 ppm
		Ammonia gas	<5 ppm

Note:

[1]Samples were obtained 6.1 m (20 ft) below the top of the riser.

Operations issues are based on the policy of segregating TRU and complexant wastes, avoiding excess heat in the tanks, and ensuring pumpability of the source waste to the receiving tank. The total concentration of TRU elements was calculated by converting the values to a per-weight basis from the per-volume basis by dividing the analytical result for each radionuclide from each phase by the mean density for that phase, then summing the per-weight results. The total was then compared to the 0.1 μCi/g standard for segregating TRU waste from non-TRU. The results showed the concentration of TRU elements exceeded the limit of 0.1 μCi/g. Finally, the heat load estimate for the tank based on the 1996 grab samples was 5,130 W (17,500 Btu/hr); this estimate is undoubtedly biased low because the lower 40 percent of the tank sludge was not sampled. Reynolds (1997) provides a best estimate heat load of 36,000 W (123,000 Btu/hr) which is based on thermomodeling and evaporation rates in 1994 (Ogden et al. 1996) and decayed to 1996. This heat load estimate does not exceed the waste compatibility heat load decision threshold of 205,000 W (700,000 Btu/hr) for tank 241-AY-102.

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6.0 CONCLUSIONS AND RECOMMENDATIONS

The waste in tank 241-C-106 has been evaluated according to the requirements listed in the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995), the *Data Quality Objectives for Tank Farms Waste Compatibility Program* (Fowler 1995), the *Tank 241-C-106 Grab Sample — Technical Letter of Instruction* (Cash and Babad 1996), the *Tank C-106 Flammable Vapor Sampling and Analysis Plan* (Homi 1996), and *Data Quality Objectives for Generic In-Tank Health and Safety Vapor Issues Resolution* (Osborne et al. 1994). As of April 30, 1996, tank 241-C-106 was the only tank listed on the High-Heat Load Watch List (Hanlon 1996). Results from the February/March 1996 grab sampling event, and the February 1994 and March 1996 vapor sampling events were used in the characterization of the tank waste.

The safety screening requirement to obtain two vertical profiles of the tank waste from widely spaced risers was met for the supernate and the upper 60 percent of the sludge. To assess tank safety, the safety screening DQO required analyses for energetics, weight percent water, density, total alpha activity, a visual inspection for a separable organics layer, and the flammability of gases in the tank headspace. To examine possible waste compatibility problems, the waste compatibility DQO required analyses for energetics, percent water, TIC, TOC, density, pH, selected metals, anions, and radionuclides. The grab sample analyses were performed at the Westinghouse Hanford Company 222-S Laboratory and at the Pacific Northwest National Laboratory. Because there is some question regarding how well the samples represent the depths at which the tank was sampled, there may be unknown biases in the analytical results.

The DSC results for two out of 32 samples or subsamples exceeded the safety screening exothermic threshold of -480 J/g on a dry-weight basis while six samples had upper limits to a one-sided 95 percent confidence interval on the mean that exceeded the threshold. However, the overall mean weight percent water (as determined by TGA) for the supernate was 80.1 with a lower limit to a one-sided 95 percent confidence interval on the mean of 75.2. For the sludge the overall mean weight percent water (TGA) was 36.3 with a lower limit to a one-sided 95 percent confidence interval on the mean of 24.0. Therefore, both percent water values were well above 17 weight percent, the minimum amount of moisture needed to prevent a propagating exothermic reaction (Turner et al. 1995).

The TOC results were compared with the organic DQO limit of 3.0 weight percent (30,000 μg C/g) on a dry weight basis (Turner et al. 1995). The overall mean result for the supernate was 12,700 μg C/g and was 23,500 μg C/g for the sludge. The upper limit to a one-sided 95 percent confidence interval on the mean for the supernate was 29,200 μg C/g, and the sludge was 29,300 μg C/g. The upper limit to the 95 percent confidence interval on the mean dry-weight TOC values for the supernate and the sludge did not exceed the 30,000 μg C/g decision threshold.

During the preparation of the sludge samples for analysis, a second liquid phase was discovered and reported. This phase was designated a potential organic layer and constituted approximately 0.5 to 3 percent of the tank volume. Reynolds (1997) reports that this layer consisted largely of bis(2-ethylhexyl)phosphate. When subjected to propagating reaction tests, bis(2-ethylhexyl)phosphate did not exhibit a propagating exothermic reaction.

All remaining requirements of the safety screening DQO were satisfied. The total alpha activity overall mean for the supernate was 1.03 μCi/mL with an upper limit to a one-sided 95 percent confidence interval on the mean of 1.79 μCi/mL. Both values were less than the decision threshold value of 61.5 μCi/mL. An overall mean total alpha value could not be calculated for the sludge, but the highest sludge subsample total alpha value was 4.38 μCi/g with an upper limit to a one-sided 95 percent confidence interval on the mean of 16.7 μCi/g; both values were less than the decision threshold value of 39.8 μCi/g.

Finally, the flammability of gases in the tank headspace was 0 percent of the lower flammability limit. The results of the vapor characterization indicated that there was no hazard posed from ammonia or organic vapors in the dome space or breathing zone of the tank.

The waste compatibility evaluation revealed other issues that may impact waste management decisions. Because significant amounts of organic carbon were found in the tank 241-C-106 waste, the waste in the tank may need to be segregated from non-complexant waste types when transferred to the double-shell tank system.

The waste compatibility criticality decision threshold value was 0.800 μCi/mL $^{239/240}$Pu. The overall mean $^{239/240}$Pu value for the supernate was 0.745 μCi/mL, nearly equalling the threshold value; the overall mean $^{239/240}$Pu value for the sludge was 1.67 μCi/mL and exceeded the threshold value. The overall mean TRU content of the waste was found to be 0.648 μCi/g for the supernate and 1.08 μCi/g for the sludge; both values exceed the 0.1 μCi/g TRU limit in the DQO.

The supernate overall mean pH of 10.2 and the caustic demand results of Herting (1996) indicate that the waste is very caustic deficient, which could lead to potential corrosion problems. However, prior to sluicing activities, caustic additions will be made to tank 241-AY-102. These caustic additions may mitigate the caustic deficiency of the waste.

Finally, the heat load estimates for the tank (Bander 1993a, Ogden et al. 1996) do not exceed the decision threshold of 205,000 W (700,000 Btu/hr) (Fowler 1995). Radionuclide data were used to calculate an estimate of 5,130 W (17,500 Btu/hr) for the tank heat load. In contrast, Bander (1993b) lists the value of 32,200 $\pm$ 5,900 W (110,000 $\pm$ 20,000 Btu/hr), Agnew et al. (1996) estimates a value of 35,600 W (12,100 Btu/hr), and Ogden et al. (1996) gives a value of 38,700 W (132,000 Btu/hr). The disparity between the values calculated from the February/ March 1996 grab samples and the other values most likely indicates that the bulk of the heat-generating radionuclides may reside in the lower 40 percent of the sludge. Evidence exists from the historical transfer records that the high ^{90}Sr is not located

in the hardpan layer; therefore, it is believed that the bulk of the radionuclides may reside in this layer. Additional information regarding the heat load for tank 241-C-106 may be found in Appendix E.

In addition to the sampling and analysis of the 1996 grab samples, the tank safety was further evaluated in preparation for sluicing. Chemical aspects are summarized in Reynolds (1997). Sluicing of the tank began in 1998, and no further sampling of tank 241-C-106 is required for the purpose of safety screening (Reynolds et al. 1999).

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