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12. Description of Change

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**45-DAY SAFETY SCREEN RESULTS FOR TANK 241-C-103
PUSH-MODE CORE SAMPLES 63 AND 66.**

This is the final 45-Day report for the fiscal year 1994/1995 Tank 241-C-103 (C-103) push-mode characterization effort. It supersedes an earlier 45-Day report (Reference 1) which transmitted the safety-screen data for segment 1 of core 63.

Included are copies of the differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) scans as requested in References 2 and 3. Also included are copies of any immediate notification documentation. Other pertinent documentation will be included in the C-103 216-day report.

Summary

Two core samples from tank C-103, obtained by the push-mode core sampling method, were received, extruded, and analyzed by the 222-S Laboratories in accordance with References 2 (core 63) or 3 (core 66). Drainable liquids were analyzed at the segment level for a separable organic layer, energetics by DSC, and percent water by TGA. Sludge samples were analyzed at the half-segment level by DSC, TGA, and for total alpha. Notification limits were exceeded for the following: (1) presence of a separable organic layer in the drainable liquid of segment 1, cores 63 and 66, and segment 2 of core 63; (2) DSC exotherm greater than 481 J/g on the lower half of segment 3 and drainable liquid of segment 4 of core 63; and (3) percent water less than 17% on the upper half of segment 4 of core 63.

Sample Receipt and ExtrusionC-103 Core 63, Segment 1

Segment 1 of core 63 was removed from riser 2 of tank C-103 on 10/28/94, received at the 222-S Laboratory on 10/31/94, and extruded on 11/04/94. Approximately 5 mL of liner liquid was collected prior to extrusion, but was not analyzed. Segment 1 consisted entirely of drainable liquid yielding 225.8 g, or approximately 220 mL. Percent recovery based on an expected sample length of 9.25 inches was estimated at 71%. An immediate notification was made due to a separable, presumably organic, layer of dark brown liquid floating on top of a light brown, presumably aqueous, layer. The volume of organic liquid measured roughly 20 mL and it was separated from the aqueous layer. A subsample of the aqueous layer was submitted for safety screening analysis.

C-103 Core 63, Segment 2

Segment 2 was removed from the tank on 1/17/95, shipped to the 222-S Laboratory on 1/19/95, and received on the same day. Extrusion took place on 1/23/95. A small amount (2 mL) of liner liquid was collected and there were no problems with the extrusion. Collected were 173.62 g, or approximately 150 mL, of dark brown, drainable liquid and 160.30 g of dark brown, moist sludge. The percent recovery was an estimated 95%. An immediate notification was made since the drainable liquid appeared to separate into two layers. The sludge was divided into half-segment portions. Radiation levels for this waste were

higher than those seen on typical tank waste samples, therefore separate aliquots were taken for direct analysis and fusion dissolution in an effort to reduce sample sizes and dose rates. Samples from the upper and lower half were submitted for analysis.

C-103 Core 63, Segment 3

The third segment was removed from C-103 on 1/17/95. It was sent to the laboratory on 1/19/95 and received the same day. Roughly 4 mL of liner liquid was collected prior to extrusion on 1/24/95. The sample consisted entirely of soft, dark brown sludge, which was partitioned into upper and lower halves. The lower half portion appeared to be slightly drier and there was some pitting on the surface of the lower half subsample. The yield was 405.6 g, giving an estimated sample recovery of 100%. For each half segment subsample, separate aliquots were taken for direct analysis and fusion dissolution.

C-103 Core 63, Segment 4

The final segment from core 63 was received by the 222-S Laboratory on 1/19/95. It was removed from C-103 on 1/18/95, and shipped the following day. Approximately 7 g of liner liquid was collected before extrusion on 1/24/95. Collected after extrusion was 73.06 g (roughly 60 mL) of dark brown, drainable liquid followed by 236.60 g of moist, sludge from the upper half of the segment. Although the outside of the sludge was dark brown, the interior of the sample was cream-colored and appeared to be somewhat drier than the dark material. Recovery was estimated at 84%. Two subsamples of the drainable liquid were removed for analysis. A subsample of the sludge was removed for direct analysis and fusion digestion.

C-103 Core 66, Segment 1

There were no problems during the extrusion of the first segment of core 66, which was taken from C-103 on 2/6/95. It was received at 222-S on 2/9/95 and extruded on 2/27/95. Collected before extrusion was 11.85 mL of liner liquid. The sampler contained 195.55 g (approximately 200 mL) of drainable liquid resulting in an estimated 100% recovery based on the expected 12.5 inch sample length. An immediate notification was made due to a separable, presumably organic, layer of dark brown liquid floating on top of a light brown, presumably aqueous, layer. The volume of organic liquid measured roughly 25 mL and it was separated from the aqueous layer. A subsample of the aqueous layer was submitted for safety screening analysis.

C-103 Core 66, Segment 2

Segment 2 was collected from the field on 2/6/95. It was shipped and received on 2/9/95. No liner liquid was observed prior to extrusion on 2/27/95. The sampler contained 330.36 g (approximately 310 mL) of light brown, drainable liquid, which was collected in two jars. The sampler efficiency was estimated at 100%. In order to comply with the ALARA (As Low As Reasonably Achievable) principle, two subsamples of the drainable liquid from one of the parent sample jars were removed for analysis in an effort to reduce radiation dose rates.

C-103 Core 66, Segment 3

Received at the laboratory on 2/9/95 was segment 3 of core 66. This segment was taken on 2/7/95 and shipped on 2/9/95. Before extrusion on 2/27/95, 4.85

g of liner liquid was collected. The sampler was found to contain both sludge and drainable liquid. The sampler efficiency was estimated at 95%. The dark brown drainable liquid was collected in two jars to give a recovery of 280.19 g (approximately 240 mL). Two subsamples from one of the parent sample jars were removed for analysis. Also collected from the upper half of the sampler was 43.66 g of soft, dark brown sludge. Separate aliquots of this material were taken for direct analysis and fusion dissolution.

C-103 Core 66, Segment 4

Segment 4 was removed from C-103 on 2/7/95. It was shipped to and received at the laboratory on 2/9/95. Extrusion took place on 2/27/95. The sampler valve was found to be closed, however 125.19 g of olive-green liner liquid was collected. The sampler contained both drainable liquid and sludge. The drainable liquid measured 89.80 g (roughly 85 mL) and was medium to dark brown in color. The moist, dark brown sludge was collected from the upper half of the sampler and measured 58.24 g. The estimated sampler efficiency was 80% based on an expected 11.65 inch sample. Two subsamples from the drainable liquid were submitted for analysis. Likewise, separate aliquots of the sludge material were taken for direct analysis and fusion dissolution.

C-103 Core 66, Field Blank

The field blank was created on 2/7/95, shipped to the laboratory on 2/9/95, and received on the same day. Extrusion took place on 2/24/95. There was no liner liquid observed. As expected, the sampler contained clear, colorless drainable liquid to yield 255.19 g that was collected in two jars. No safety screening analyses were performed on subsamples of the field blank.

Analytical Results

The safety screening analytical results are presented in Tables 1 and 2, which include the LabCore sample number. They also include the upper or lower action limits as defined in the References 2 and 3. The limit selected for immediate notification is highlighted in greybar. Column 2 of the tables indicates the sample preparation used, if any. As shown, analyses on fused samples are marked with "F".

DSC (Energetics Content)

Analyses by DSC were performed under a nitrogen atmosphere using procedure LA-514-113, Rev. B-1. No exotherms are observed for the sample or duplicate of the aqueous, drainable liquid, with the exception of the liquid from segment 4 of core 63 (sample S95T000052). The results in Table 1 indicate a high relative percent difference (RPD) between the sample and duplicate results for that sample. Not shown in the table, but provided with the raw data, is the result from a second duplicate, or third sample, that was run. The result of the third run is 46.2 J/g, consistent with the duplicate result. The sample result of 156.8 J/g exceeds the notification limit of 481 J/g when calculated on a dry-weight basis, and immediate notification was made. Exotherms were observed on sludge from segments 2 and 3 of core 63, while sludge from core 66 exhibited no exothermic behavior. The calculated dry-weight result on sample S95T000046 exceeds the notification limit of 481 J/g and immediate notification was made. A RPD of greater than 10% exists on samples S95T000045 and S95T000046. Re-runs were not performed due to the high

dose rates associated with these samples. Re-runs will be made at the customer's request.

TGA (Moisture Content)

Weight percent water by TGA was performed under a nitrogen atmosphere using procedure LA-560-112, Rev. A-2. Results on the drainable liquid samples are well above the notification limit of <17% water, being in the 80-90% range. Results on the sludge samples are also significantly above the <17% notification limit with the exception of sample S95T000048 from the upper half of segment 4. An immediate notification was made per Reference 2 since the sample result is less than 17%, although the duplicate result and a second duplicate result of 29.70% (not shown in Table 2) are above the limit. The third run was made due to the high relative percent difference (RPD) between the sample and duplicate and is in good agreement with the duplicate result. Samples S95T000046 and S95T000224 also demonstrated high RPD values. Additional runs were not made on these samples due to the high dose rate of the samples as mentioned above, and because the results were well above the notification limit. The high RPD values are indicative of sample heterogeneity at the 15-30 mg level, the sample size required for the TGA instrument used.

Total Alpha

Total alpha analyses were performed on fusion digestions of all sludge subsamples using procedure LA-508-101, Rev. D-2. All results are well below the notification limit of 41 uCi/g. Results on aliquots of segments 2 and 3 from core 63 and segment 4 from core 66 are in the 11 to 19 uCi/g range. The alpha activity in the remaining sludge samples is substantially lower. The average ranges from 1.73 to 5.85 uCi/g. A RPD of greater than 10% exists on samples S95T000057, S95T000226, and S95T000227, with the highest being 30.1%. Reruns of these samples were not requested due to the very high beta to alpha activity ratio in these samples. Large dilutions are necessary to reduce the beta activity to acceptable levels and, consequently, detection limits are high such that the alpha activity is only on the order of ten times the detection limit. The spike recovery on sample S95T000057 is slightly below the limit of 90%. Partial dissolution of the sample mount was observed, most likely due to the reaction of chloride in the sample with the nitric acid in the spiking solution, which lead to corrosion of the mount. The solids thus formed caused sample self-absorption and low spike recoveries.

References: (1) WHC-SD-WM-DP-080, Rev. 0, "45-Day Safety Screening for C-103 Push Mode Sample, Riser 2," dated December 13, 1994, Westinghouse Hanford Company, Richland, Washington 99352

(2) WHC-SD-WM-TP-207, Rev. 0, "Tank 241-C-103 Tank Characterization Plan," dated October 3, 1994, Westinghouse Hanford Company, Richland, WA 99352

(3) WHC-SD-WM-TP-207, Rev. 1, "Tank 241-C-103 Tank Characterization Plan," dated October 3, 1994, Westinghouse Hanford Company, Richland, WA 99352

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