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Tank 48H Phase 1 Initial Testing: As-Received Characterization of HTF-48-25-17

E. J. Craig

September 2025

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Tank 48H Phase 1 Initial Testing Using Sodium Permanganate to Decompose Tetraphenylborate: As- Received Characterization of HTF-48-25-17

E. J. Craig

September 2025

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EXECUTIVE SUMMARY

This report provides results from a detailed characterization study of Tank 48H sample HTF-48-25-17 performed by the Savannah River National Laboratory (SRNL). The slurry sample was retrieved from Savannah River Site (SRS) Tank 48H on February 25, 2025 and received at the SRNL shielded cells on 02/25/2026. This effort completes the first task of a multi-phase approach, requested by Savannah River Mission Completion (SRMC), to study tetraphenylborate (TPB) decomposition in Tank 48H using sodium permanganate with radioactive waste.

A summary of key results from HTF-48-25-17 include:

- The average densities of the HTF-48-25-17 for slurry was 1.28 g/mL and for filtrate was 1.27 g/mL.
- The average weight percent total dried solids was 27.4 %, for dissolved solids was 26.1 %, and for insoluble solids was 1.77%.
- Cs-137 was analyzed in both the supernate and slurry. The result for Cs-137 in supernate was 2.13E+07 dpm/mL and the result for Cs-137 in slurry was 5.34E+08 dpm/g.
- High-Performance Liquid Chromatography (HPLC) was used to measure tetraphenylborate (TPB) and degradation products in the supernate and slurry. Phenol, TPB, and biphenyl were detected in the slurry. TPB was detected at 1.82E+04 mg/L in the slurry. Phenol was the only degradation product detected in the supernate. Volatile Organic Analysis (VOA) was used to measure volatile organic species. Benzene was detected and reported at 104 mg/L.

A comparison of results from the physical, chemical, and radionuclide characterizations in this report with results from previous samples is also provided.

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LIST OF ABBREVIATIONS

ATFC	Analytical and Tank Farm Characterization
AR	Aqua Regia
DMA	Direct Mercury Analysis
DOE	Department of Energy
DWPF	Defense Waste Processing Facility
HCl	Hydrochloric Acid
HDPE	High Density Polyethylene
HPLC	High-Performance Liquid Chromatography
IC	Ion Chromatography
ICP-ES	Inductively-Coupled Plasma-Emission Spectroscopy
ICP-MS	Inductively-Coupled Plasma-Mass Spectroscopy
ITP	In Tank Precipitation
MST	Monosodium Titanate
NaTPB	Sodium Tetraphenylborate
NM	Nuclear Measurements
PHA	Pulse Height Analysis
PF	Peroxide Fusion
SRMC	Savannah River Mission Completion
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
SS	Stainless Steel
SVOA	Semivolatile Organic Analysis
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon
TPB	Tetraphenylborate
TTQAP	Task Technical Quality Assurance Plan
TTR	Task Technical Report
VOA	Volatile Organic Analysis
wt%	Weight Percent

1.0 Introduction

Tank 48H at the Savannah River Site (SRS) currently holds approximately 270,000 gallons of legacy material comprised of decontaminated salt solution, approximately 20,000 kilograms of tetraphenylborate (TPB) solids, 3,400 kilograms of sludge solids, and 1,800 kilograms of monosodium titanate (MST). Tank 48H was the main reaction tank for the In-Tank Precipitation (ITP) process where sodium tetraphenylborate (NaTPB) was added to precipitate cesium and potassium as TPB salts. In addition to NaTPB, MST was added to bind strontium and actinides from the salt solution. The original plan called for processing the TPB slurry in the ITP Facility, the Late Waste Facility, and the Defense Waste Processing Facility (DWPF). That process included concentrating the TPB, washing the non-radioactive salts to reduce the nitrite concentration, decomposing the TPB to benzene, and separating the benzene from the aqueous waste.¹ The unexpected decomposition of the TPB in Tank 48 caused excessive benzene to be released that exceeded flammability limits established for the tank. Any future processing by ITP was ceased by the Department of Energy (DOE) in 1998.²

Recently, the Savannah River National Laboratory (SRNL) performed scoping tests using sodium permanganate to oxidize and decompose the TPB at room temperature and 40 °C.³ In addition, tests at 40°C were performed at pH 6, 10, and 11 and showed a destruction of TPB of >90% is possible after a reaction time of two weeks.⁴ Characterization for physical and chemical composition and radionuclides in support of the disposition of Tank 48H material was last conducted in 2022.¹

Savannah River Mission Completion (SRMC) has issued a Technical Task Request (TTR) which outlines the first of three phases for research and development activities to study TPB decomposition using sodium permanganate.⁵ SRNL issued a Task Technical and Quality Assurance Plan (TTQAP) in response.⁶ The initial phase is dedicated to a detailed characterization of Tank 48H slurry and the identification of key flowsheet parameters via testing using radioactive and simulated slurries. This report covers the detailed characterization of Tank 48H slurry.

2.0 Experimental Procedure

2.1 Sample Information

On February 25, 2025, SRMC retrieved sample HTF-48-25-17 from Tank 48H Riser B2 after a 24 hour, four-pump mixing campaign. The sample was acquired from Tank 48H using a 3L stainless steel (SS) sampler as soon as possible after pump shutdown. At the time of retrieval, the tank liquid level was 74.4 inches and the supernate temperature was 23 °C. Photographs of HTF-48-25-17 being retrieved at Tank 48H and the as-received in the SRNL Shielded Cells are provided in Figure 2-1.

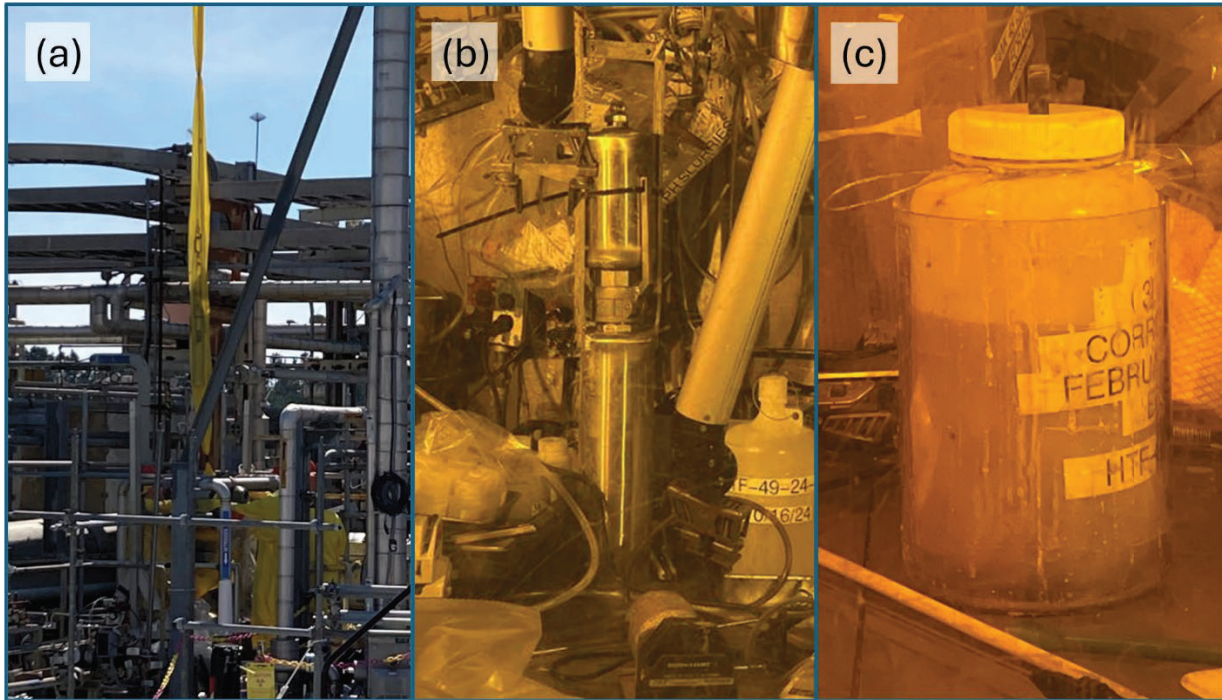


Figure 2-1 Photographs of (a) sample HTF-48-25-17 being retrieved from Tank 48H Riser B2, (b) the 3L sampler in the SRNL shielded cells, and (c) the 4L HDPE bottle with Tank 48H slurry.

The slurry sample was transferred from the 3L SS sampler into a 4L High Density Polyethylene (HDPE) bottle in the SRNL shielded cells (Figure 2-1). After the bulk of the slurry was transferred, the 3L metal sampler was rinsed with the slurry's supernate to ensure all solids were transferred into the HDPE bottle. Key observations from transferring the slurry from the metal sampler to the HDPE bottle are provided in Section 3.1. HTF-48-25-17 was mixed with an overhead mixer for 40 minutes prior to retrieving a 400 mL subsample (Figure 3-1). This 400 mL subsample was then used to complete the characterization analyses provided in this report.

2.2 Sample Analysis

Table 2-1 shows the preparation methods utilized for each analysis performed by the Analytical and Tank Farm Characterization (ATFC) and the Nuclear Measurements (NM) groups at SRNL. Weight % solids (wt%), and density measurements were performed in the Shielded Cells.

Table 2-1. Sample Preparations Performed for Each Analysis

Preparation	Slurry/Filtrate	Analysis
Aqueous Supernate Dilution	Filtrate	ICP-ES, ICP-MS, TIC/TOC, IC ANIONS, VOA, SVOA, TBASE/OH/OTHER BASE, GAMMA SCAN, GAMMA SCAN NO CS, SR-90, U_233_234_235_236, TC-99, GROSS ALPHA
Water Diluted Slurry Filtered	Slurry	IC ANIONS
Water Diluted Slurry Not Filtered	Slurry	TBASE/OH/OTHER BASE, VOA, TIC/TOC
Aqua Regia (AR)	Slurry	ICP-ES, ICP-MS, AA_DMA_TOTAL MERCURY, TC-99
Peroxide Fusion (PF)	Slurry	ICP-ES, ICP-MS, PU_242_244, PU_238_241, GAMMA SCAN, GAMMA SCAN NO CS, SR-90, U_233_234_235_236, GROSS ALPHA
Methylene Chloride Extraction	Slurry	SVOA
Mercury Dilution	Filtrate	AA_DMA_TOTAL MERCURY, INORGANIC MERCURY, PURGEABLE MERCURY, VOA (Dimethyl Mercury), ETHYL MERCURY, METHYL MERCURY
Americium and Curium	Slurry	AM_CM

2.2.1 Densities and Weight Percent Solids

Slurry densities and supernate densities were obtained in duplicate using 10 mL volumetric flasks and an analytical balance. The supernate was obtained by filtering a portion of the slurry through a 0.45 μm nylon filter. Filtrate, instead of supernate, was used to ensure no insoluble solids would be accounted for in the supernate density measurements.

Weight percent total and dissolved solids measurements were performed in quadruplicate. Aliquots of slurry and filtrate were dried to a constant weight at 115 $^{\circ}\text{C}$.⁷ Filtrate was obtained by filtering a portion of the slurry through a 0.45 μm nylon filter. Filtrate, instead of supernate, was used in weight percent solids to ensure no insoluble solids would be accounted for in the dissolved solids measurements. Weight percent insoluble and soluble solids were calculated from the total and dissolved solids measurement.

2.2.2 Aqueous Supernate Dilutions

Aqueous supernate dilutions were submitted to SRNL-ATFC for total inorganic carbon/total organic carbon (TIC/TOC), Total Base/OH/Other Base excluding CO_3^{2-} , ion chromatography (IC) anions, inductively coupled plasma - emission spectroscopy (ICP-ES), inductively coupled plasma - mass spectrometry (ICP-MS), gamma scan, Cs-removed gamma scan, volatile organic analysis (VOA), semi-volatile organic analysis (SVOA) and alpha counting. Isotopes of uranium were determined from purified aliquots analyzed by ICP-MS through the U233/U234/U235/U236 method. Aliquots of the dissolution were purified with diamyl amyolphosphonate based solid phase extraction.⁸ The supernate

was obtained by filtering a portion of the slurry through a 0.45 μm nylon filter. Filtrate, instead of supernate, was used for the supernate analytical results to ensure no insoluble solids would be accounted for in the measurements. One set of filtrate aliquots were diluted in duplicate by $\sim 10\text{x}$ with water. The purpose of dilution was to reduce personnel radiation exposure.

2.2.3 Water Diluted Slurry

Filtered, water diluted slurry was prepared to obtain IC anions in slurry. This was performed by weighing out a set amount of slurry, diluting the sample with water by $\sim 10\text{x}$ to dissolve water soluble solids, and filtering the sample with a 0.45 μm Nylon syringe filter to prevent solids from being sent to the IC instrument.

Unfiltered, water diluted slurry was prepared to obtain free hydroxide, TIC/TOC and VOA. This was performed by weighing out a set amount of slurry and diluting the sample with water by $\sim 10\text{x}$ to dissolve water soluble solids.

2.2.4 Mercury Dilution

Mercury dilutions were performed to obtain the analyses for methyl- and ethyl- mercury, inorganic mercury, purgeable mercury (i.e. elemental mercury plus dimethylmercury), and elemental mercury. Inorganic mercury, purgeable mercury and elemental mercury were diluted $\sim 100\text{x}$ with purified water using a thick-walled glass volumetric flask with a Teflon stopper. The sample was then transferred to a 15 mL glass vial with little to no head space. Methyl- and ethyl- mercury were diluted $\sim 100\text{x}$ with purified water and high purity hydrochloric acid (HCl) (0.4% concentration) using a thick-walled glass volumetric flask with a Teflon stopper. The sample was then transferred to a 15 mL glass vial with little to no head space. These samples then exited the shielded cells and were refrigerated.⁹

2.2.5 Digestions

Slurry samples were digested by two methods, AR and PF, for analysis of the slurry solids. For the AR digestions, aliquots of slurry were mixed with aqua regia and heated for two hours in sealed Teflon pressure vessel in an oven at 115 $^{\circ}\text{C}$. The resulting liquids were diluted to 100 mL and submitted to SRNL-ATFC for ICP-ES, ICP-MS, and DMA analyses. For the PF digestions, aliquots of slurry were dried and fused at 675 $^{\circ}\text{C}$ with sodium peroxide and sodium hydroxide. The fused samples were then dissolved with nitric acid. The resulting liquids were diluted to 100 mL with water and analyzed for ICP-ES and alpha counting. Each digestion was performed in duplicate. See Procedure L16.1-ADS-2226 for aqua regia and L16.1-ADS-2502 for peroxide fusion in the SRNL-ATFC procedure manual for detail of the digestion techniques.^{10,11}

For the elements quantified by ICP-ES, results from two digestions were compared. In general, if the percent difference between the two results was 10% or less, the average is reported. If the percent difference between results was greater than 10%, one digestion was chosen. Those decisions are noted in the results section.

For the elements Ag, Be, Cd, Ce, Co, Cu, Li, Mo, P, S, Sb, Si, Sn, Ti, V, and Zn; ICP-ES results from AR digested samples are reported since these elements were not detected in the PF digested sample (AR digested samples typically have lower detection limits due to less dilution required at the instrument). For the elements not detected in either the PF or AR digestions, the lowest detection limit was reported. For K and Na, results from the ICP-ES of the AR digestions were used because these elements were detected in significant quantities in the PF reagent blank. The PF digestion method utilizes sodium peroxide, which, in addition to Na, likely contains K. The PF digestion method is performed in Zr crucibles, which contributes additional Zr to the analysis. For total Hg, results from the DMA analysis of the AR digestion were used. For Th, ICP-MS (mass 232) from the AR digestion was reported due to the lower detection limit instead of ICP-ES. For Al, and Ca, ICP-ES from the PF digestions were used because AR does not fully dissolve these elements.

Fission product elements Ru, Rh, and Pd were quantified by ICP-MS from the AR digestion using a methodology described by Bibler.¹² In summary, Ru is reported from the sum of masses 101, 102, and 104. Rh is taken from mass 103. Pd is determined from mass 105 and a factor derived from fission yields of other Pd isotopes.

Nd is also determined from ICP-MS from the AR digestion. Stable isotopes of Nd include masses 142 to 146, 148, and 150.¹³ However, there is a stable isotope of Ce at mass 142. Therefore, mass 142 is not used in the Nd quantification, which may lead to small under-reporting of Nd.

Isotopes of uranium in the slurry were determined from U233/U234/U235/U236 through ICP-MS on PF digested samples. Isotopes of plutonium were determined by PuTTA separation, alpha counting and ICP-MS of PF digested samples.

Cs-removed gamma scan method was used to determine Co-60, Ru-106, Sb-125, Ce-144, Eu-154, and Am-241 with a high purity coaxial germanium detector. Cs-137 was removed from the duplicate PF samples by using an ammonium phosphomolybdate resin. This allowed the remaining gamma photons at lower concentrations to be detected with lower detection limits. Cs-137 was determined through gamma pulse height analysis (PHA) on duplicate PF samples.

2.2.6 Am/Cm Preparation

This method was used to obtain Am-241, Am-242m, Am-243, Cm-242, Cm-243, Cm-244, Cm-245, Cm-247, Cf-249, and Cf-251. Peroxide fusion was performed on the slurry. The analytes were then extracted from the dissolution filtrate using a commercially available ion exchange resin (Eichrom[®] DGA). Samples were held in the Shielded Cells for 9 days to allow for the Y-90 to decay before submitting to NM. These samples were then purified further with a second RE resin extraction followed by an Eichrom[®] Ln resin extraction. These analytes were then analyzed by alpha and low-energy gamma counting techniques and by ICP-MS.⁸

2.3 Quality Assurance

This work was requested via a TTR and directed by a TTQAP.^{5,6} Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.¹⁴ The functional classification requested for this work is Safety Class. In accordance with Manual E7, Procedure 3.60, a technical review of the contents of this report has been performed to ensure consistency and accuracy. Per Manual E7, Procedure 2.60, a Design Verification, at a minimum, must be performed for work supporting a Safety Class functional classification.

3.0 Results and Discussion

3.1 Sample Transfer

Multiple strategies were employed to transfer material from the SS sampler to the 4L HDPE bottle. Initial efforts to transfer slurry utilized a peristaltic pump, but a large amount (approximately 600 mL) of slurry remained in the SS sampler even after rinsing with supernate. The peristaltic pump was ultimately abandoned after several attempts with different pump heads, tubes and dip legs (max diameter 0.31 inches). A ladle and metal spatula were used to transfer the remaining material into the 4L HDPE bottle. Photographs of the transfer process are shown in Figure 3-1. A portion of the “soft crystal” solids can be seen in the right photo in a weigh boat being added to the HDPE bottle.

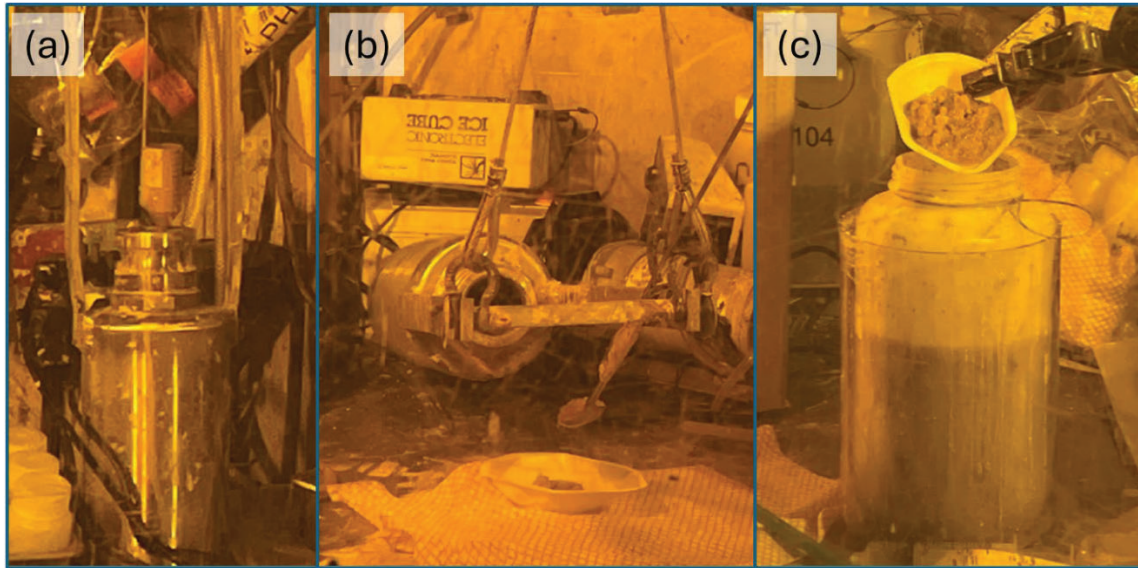


Figure 3-1 Photographs of HTF-48-25-17 (a) being ladled out of the metal sampler, (b) being scooped out of the metal sampler with a metal spoon and (c) a portion of the “soft crystal” solids in weigh boat being transferred into a HDPE bottle.

3.2 Density and Weight Percent Solids

Presented in Table 3-1 are the results for density and weight percent solids of the Tank 48H Subsample.

Table 3-1. Densities and Weight Percent Solids

Method	Average	RSD, n*
Slurry Density (g/mL)	1.28	0.43, 3
Filtrate Density (g/mL)	1.27	0.50, 3
Wt% Total Dried Solids (Slurry Basis)	27.4	1.94, 3
Wt% Dissolved Solids (Supernate Basis)	26.1	0.32, 3
Wt% Insoluble Solids (Slurry Basis)	1.77	1.81
Wt% Soluble Solids (Slurry Basis)	25.6	NA

*RSD=relative standard deviation with number of measurements (n). NA=not applicable because result is calculated.

Error propagation of the total solids (slurry basis) and soluble solids (filtrate basis) yielded a 95% confidence interval for the insoluble solids of approximately ± 0.3 weight % for both sets performed. The confidence interval is derived using the inverse Student’s t-distribution which requires probability and degrees of freedom, partial derivative of the weight percent of insoluble solids with respect to total solids, partial derivative of the weight percent of insoluble solids with respect to soluble solids, standard error of the weight percent total solids, and standard error of the weight percent soluble solids. Uncertainty for the insoluble solids does not account for variability in the sampling. Contributing factors to larger 95% confidence interval values include standard deviation of results (incorporated in standard error), number of replicates (incorporated in degrees of freedom), and average weight percent of soluble solids (incorporated in partial derivative of weight percent insoluble solids with respect to total solids and soluble solids). A higher standard deviation of results, larger amount of weight percent soluble solids, and fewer number of replicates can lead to a larger 95% confidence interval value.

3.3 Supernate Analytical Results

The result for mercury in supernate is provided in Table 3-2, the supernate anions is provided in Table 3-3, the results from SVOA and VOA in supernate are provided in Table 3-4, the results for the HPLC measurements of tetraphenylborate and degradation products are provided in Table 3-5, and the results for the elemental composition and radionuclides in supernate are provided in Table 3-6 and Table 3-7.

Table 3-2. Mercury Results in Supernate

Analyte	Units	Result	%RSD	One Sigma Uncertainty (%)
Elemental Mercury	mg/L	<1.12E+02	NA	NA
Inorganic Mercury	mg/L	<1.12E+01	NA	NA
Purgeable Mercury	mg/L	<1.12E+01	NA	NA
Ethyl Mercury	mg/L	<1.18E+02	NA	NA
Methyl Mercury	mg/L	<1.18E+01	NA	NA

Table 3-3. Supernate Anions

Analyte	Analytical Method	Units	Results	%RSD	One Sigma Uncertainty (%)
Bromide	IC	mg/L	<6.65E+02	NA	NA
Chloride	IC	mg/L	7.36E+02	1.19	10
Fluoride	IC	mg/L	<1.33E+02	NA	NA
Formate	IC	mg/L	2.71E+02	1.83	10
Nitrate	IC	mg/L	1.07E+04	0.72	10
Nitrite	IC	mg/L	2.95E+04	1.00	10
Oxalate	IC	mg/L	9.48E+02	2.77	10
Phosphate	IC	mg/L	3.96E+02	0.83	10
Sulfate	IC	mg/L	2.21E+02	0.68	10
Carbonate	TIC	mg/L	1.07E+05	0.59	7
Free OH	Titration	M	1.48E+00	7.65	10
Total Organic Carbon	TOC	mg Carbon/L	2.26E+03	2.72	7

< Results are not the average. They are the lowest detection limit recorded. NA = Not Applicable

Table 3-4 indicated that only VOA analyte detected was toluene, 7.75E+00 mg/L. All other VOA analytes in the supernate were below the detection limit of <13 mg/L and all SVOA analytes in the supernate were below the detection limit of <2.6 mg/L. Table 3-5 indicates only phenol was detected in the supernate. Biphenyl, 2PB, 3PB, and TPB were lower than the detection limits in the HPLC method.

Table 3-4. Chemical Concentrations for Semi-Volatile Organic Compounds and Volatile Organic Compounds in Supernate

Analyte	Analytical Method	Units	Results	%RSD	One Sigma Uncertainty (%)
Toluene	VOA	mg/L	7.75E+00	5.45	10

Table 3-5. HPLC Measurements of Tetraphenylborate and Degradation Products in Supernate

Analyte	Results (mg/L)	%RSD	One Sigma Uncertainty (%)
Phenol	5.61E+02	0.31	10
2PB	<101	NA	NA
3PB	<101	NA	NA
TPB	<101	NA	NA
Biphenyl	<101	NA	NA

NA = Not Applicable.

Table 3-6. Elemental Composition of Supernate

Element	Analytical Method	Results (mg/L)	% RSD	One Sigma Uncertainty (%)
Ag	ES	<9.31E-02	NA	NA
Al	ES	2.91E+03	1.94	5.14
B	ES	5.92E+02	1.27	5.00
Ba	ES	<7.98E-02	NA	NA
Be	ES	<3.99E-02	NA	NA
Ca	ES	<2.17E+00	NA	NA
Cd	ES	<6.65E-02	NA	NA
Ce	ES	<9.31E-01	NA	NA
Co	ES	<2.26E-01	NA	NA
Cr	ES	4.91E+01	1.15	5.00
Cu	ES	<1.04E+00	NA	NA
Fe	ES	<1.65E+00	NA	NA
Gd	MS	<1.32E-02	NA	NA
Hg	DMA	<1.12E+02	NA	NA
K	ES	5.34E+02	3.35	5.09
La	ES	<9.31E-02	NA	NA
Li	ES	<1.64E+00	NA	NA
Mg	ES	<1.06E-01	NA	NA
Mn	ES	<6.12E-01	NA	NA
Mo	ES	6.82E+00	1.38	5.01
Na	ES	1.38E+05	0.68	5.03
Nd	MS	<7.95E-02	NA	NA
Ni	ES	<3.06E-01	NA	NA
P	ES	2.24E+02	1.26	5.00
Pd	MS	<1.10E-01	NA	NA
Rh	MS	1.27E-01	0.00	10.0
Ru	MS	2.89E-01	2.75	10.0
S	ES	2.96E+02	1.59	5.02
Sb	ES	<9.31E-01	NA	NA
Si	ES	<1.23E+01	NA	NA
Sn	ES	<3.99E+00	NA	NA
Sr	ES	<3.99E-02	NA	NA
Th	ES	<1.26E+00	NA	NA
Ti	ES	<4.39E-01	NA	NA
V	ES	<9.31E-01	NA	NA
Zn	ES	1.52E+01	1.24	5.01
Zr	ES	<9.31E-02	NA	NA

< Results are not the average. They are the lowest detection limit recorded. NA = Not Applicable

Table 3-7. Radionuclides in Supernate

Analyte	Analytical Method	Results (dpm/mL)	%RSD	One Sigma Uncertainty (%)
Cs-133	MS	4.30E-01 (mg/L)*	2.45	10.0
Cs-137	Gamma Scan	2.13E+07	0.29	5.00
Ba-137m	Calculated from Cs-137	2.02E+07	NA	NA
Co-60	Gamma Scan Cs removed	<3.44E+02	NA	NA
Ru-106	Gamma Scan Cs removed	<2.08E+03	NA	NA
Sb-125	Gamma Scan Cs removed	<1.15E+03	NA	NA
Sb-126	Gamma Scan Cs removed	4.77E+02	5.31	13.9
Sn-126	Gamma Scan Cs removed	4.77E+02	5.31	13.9
Ce-144	Gamma Scan Cs removed	<2.37E+03	NA	NA
Eu-154	Gamma Scan Cs removed	<7.33E+02	NA	NA
Am-241	Gamma Scan Cs removed	<1.49E+03	NA	NA
Sr-90	Sr 90	<5.33E+04	NA	MDA
Y-90	Secular Equilibrium	<5.33E+04	NA	NA
Tc-99	Tc 99	5.11E+04	7.84	7.97
U-233	U 233 234 235 236	4.32E+02	0.92	17.3
U-234	U 233 234 235 236	1.58E+03	1.18	17.3
U-235	U 233 234 235 236	1.14E+00	0.56	17.3
U-236	U 233 234 235 236	7.23E+00	0.96	17.3
U-238	U 233 234 235 236	1.23E+00	1.62	10.0
Total Alpha	Liquid Scintillation	<6.33E+04	NA	NA

*dpm/mL does not apply as Cs-133 is stable. < Results are not the average. They are the lowest detection limit recorded. NA = Not Applicable. MDA = Minimum Detectable Activity

3.4 Slurry Analytical Results

The results for the slurry anions are provided in Table 3-8, chemical concentrations for SVOA and VOA compounds in slurry are provided in Table 3-9, HPLC measurements of tetraphenylborate and degradation products are provided in Table 3-10, and elemental composition and radionuclides in slurry are provided in Table 3-11 and Table 3-12.

Table 3-8. Slurry Anions

Analyte	Analytical Method	Units	Results	%RSD	One Sigma Uncertainty (%)
Bromide	IC	mg/L	<6.37E+02	NA	NA
Chloride	IC	mg/L	6.67E+02	1.37	10
Fluoride	IC	mg/L	<1.27E+02	NA	NA
Formate	IC	mg/L	2.53E+02	0.00	10
Nitrate	IC	mg/L	9.78E+03	1.59	10
Nitrite	IC	mg/L	2.79E+04	0.76	10
Oxalate	IC	mg/L	1.59E+03	3.11	10
Phosphate	IC	mg/L	4.23E+02	14.4	10
Sulfate	IC	mg/L	1.85E+02	NA*	10
Carbonate	TIC	mg/L	8.56E+04	0.03	7
Free OH	Titration	M	1.65E+00	2.16	10
Total Organic Carbon	TOC	mg Carbon/L	1.09E+04	68.3	7

< Results are not the average. They are the lowest detection limit recorded. NA = Not Applicable

*Reported as a single result as the duplicate sample was below detection limit.

Table 3-9 indicates that toluene and benzene were detected with VOA. Benzene was double the ~50 mg/L value reported in the 2012 characterization report.¹⁵ All other VOA analytes in the slurry were below detection limit of <1 mg/L. Table 3-10 indicates that phenol and TPB were detected with HPLC. 2PB and 3PB were lower than the detection limit in the HPLC method. Biphenyl was measured using both SVOA and HPLC, but concentrations were outside the desired working range of the SVOA method. The HPLC method is designed for the relatively high concentrations of biphenyl and is considered to provide a more precise measurement.

Table 3-9. Chemical Concentrations in Slurry for Semi-Volatile Organic Compounds and Volatile Organic Compounds

Analyte	Analytical Method	Results (mg/L)	%RSD	One Sigma Uncertainty (%)
Toluene	VOA	1.27E+01	2.16	10
Benzene	VOA	1.04E+02	15.7	10
Biphenyl	SVOA	4.85E+02	20.4	10

All other VOA analytes in the were <1 mg/L. All SVOA analytes in the supernate were <0.2 mg/L.

Table 3-10. HPLC Measurements of Tetraphenylborate and Degradation Products in Slurry

Analyte	Results (mg/L)	%RSD	One Sigma Uncertainty (%)
Phenol	7.62E+02	7.02	10
TPB	1.82E+04	1.97	10
2PB	<102	NA	NA
3PB	<102	NA	NA
Biphenyl	1.04E+03	18.7	10

NA = Not Applicable.

Table 3-11. Elemental Composition of Slurry

Element	Analytical Method	Results (mg/L)	% RSD	One Sigma Uncertainty (%)
Ag	AR, ES	<3.75E-01	NA	NA
Al	PF, ES	3.10E+03	1.94	5.00
B	AR/PF, ES	1.21E+03	1.27	5.00
Ba	AR, ES	5.95E+00	2.74	5.00
Be	AR, ES	<1.61E-01	NA	NA
Ca	PF, ES	5.21E+02	14.0	5.01
Cd	AR, ES	<3.20E-01	NA	NA
Ce	AR, ES	<2.15E+00	NA	NA
Co	AR, ES	<4.86E-01	NA	NA
Cr	AR, ES	6.25E+01	3.15	5.00
Cu	AR, ES	<1.43E+01	NA	NA
Fe	AR, ES	1.08E+02	4.38	5.04
Gd	AR, ES	<5.31E-01	NA	NA
Hg	DMA	2.61E+01	1.39	10.0
K	AR, ES	2.76E+03	3.35	5.03
La	AR, ES	<2.09E-01	NA	NA
Li	AR, ES	<3.81E+00	NA	NA
Mg	AR, ES	2.67E+01	6.09	5.03
Mn	AR, ES	1.03E+01	2.82	5.02
Mo	AR, ES	7.15E+00	3.08	5.06
Na	AR, ES	1.38E+05	0.84	5.02
Nd	AR, MS	<3.68E-01	NA	NA
Ni	AR, ES	<4.85E+00	NA	NA
P	AR, ES	2.23E+02	2.08	5.01
Pd	AR, MS	<5.09E-01	NA	NA
Rh	AR, MS	1.66E-01	1.09	10.0
Ru	AR, MS	<5.67E-01*	NA	NA
S	AR, ES	3.03E+02	3.05	5.10
Sb	AR, ES	<2.16E+00	NA	NA
Si	AR, ES	1.41E+02	4.09	5.04
Sn	AR, ES	<9.32E+00	NA	NA
Sr	AR, ES	<1.06E+00	NA	NA
Th	AR, MS	<3.10E-01	NA	NA
Ti	AR/PF, ES	9.55E+02	2.75	NA
V	AR, ES	<2.18E+00	NA	NA
Zn	AR/PF, ES	1.70E+01	1.24	5.00
Zr	AR, ES	<1.08E+00	NA	NA

< Results are not the average. They are the lowest detection limit recorded. NA = Not Applicable
*Mass 104 was below detection limit when Ru was calculated. Therefore, the Average result is <.

Table 3-12. Radionuclides in Slurry

Analyte	Analytical Method	Results (dpm/g)	%RSD	One Sigma Uncertainty (%)
Cs-133	MS	1.44E+01 (mg/L)*	1.89	10.0
Cs-135	Calculated from MS ¹⁶	2.75E+04	NA	NA
Cs-137	Gamma Scan	5.34E+08	2.48	5.00
Ba-137m	Calculated from Cs-137	5.05E+08	NA	NA
Co-60	Gamma Scan Cs Removed	<1.67E+03	NA	NA
Ru-106	Gamma Scan Cs Removed	<9.20E+03	NA	NA
Sb-125	Gamma Scan Cs Removed	<5.37E+03	NA	NA
Ce-144	Gamma Scan Cs Removed	<1.12E+04	NA	NA
Eu-154	Gamma Scan Cs Removed	<3.47E+03	NA	NA
Am-241	Am/Cm	<1.13E+02	NA	NA
Am-242m	Am/Cm	<3.54E+00	NA	NA
Am-243	Am/Cm	<5.34E-01	NA	NA
Cm-242	Am/Cm	<2.93E+00	NA	NA
Cm-243	Am/Cm	<9.48E-01	NA	NA
Cm-244	Am/Cm	<3.17E+02	NA	NA
Cm-245	Am/Cm	<7.78E-01	NA	NA
Cm-247	Am/Cm	<2.39E-01	NA	NA
Cf-249	Am/Cm	<2.67E-01	NA	NA
Cf-251	Am/Cm	<8.39E-01	NA	NA
Sr-90	Sr 90	<6.39E+05	NA	NA
Y-90	Secular Equilibrium	<6.39E+05	NA	NA
Tc-99	Tc 99	4.02E+04	9.50	9.29
U-233	U 233 234 235 236	4.32E+02	0.92	17.3
U-234	U 233 234 235 236	1.58E+03	1.18	17.3
U-235	U 233 234 235 236	1.14E+00	0.56	17.3
U-236	U 233 234 235 236	7.23E+00	0.96	17.3
U-238	U 233 234 235 236	1.23E+00	1.62	10.0
Pu-239	PU 242 244, PU 238 241	<1.54E+03	NA	NA
Pu-240	PU 242 244, PU 238 241	<1.54E+03	NA	NA
Pu-242	PU 242 244, PU 238 241	<9.77E+01	NA	NA
Pu-244	PU 242 244, PU 238 241	<4.54E-01	NA	NA
Pu-241	PU 242 244, PU 238 241	<1.14E+04	NA	NA
Pu-238	PU 242 244, PU 238 241	1.90E+05	35.0	5.66
Total Alpha	Liquid Scintillation	<3.19E+05	NA	NA

*dpm/g does not apply as Cs-133 is stable. < Results are not the average. They are the lowest detection limit recorded. NA = Not Applicable

3.5 Comparison Data

Samples from Tank 48H have been analyzed over many years. Therefore, it would be wise to compare the current results with previous results to ensure data quality. However, several considerations apply. First, due to changes in the tank volume, care must be taken to either volume normalize the results or compare samples that came from comparable tank volumes. In this case, the tank volumes from comparison samples vary <10%, so a high-level comparison is still valid. Second, as water evaporates from the tank, it is replaced with solutions containing NaOH, and NaNO₂ for the purposes of corrosion control.¹⁷ Over time, the concentrations of nitrite and nitrate will increase. Free hydroxide also increases, but this is somewhat compromised by reaction of free hydroxide with atmospheric CO₂ to create carbonate. Finally, due to the chemical changes, salt precipitation and dissolution can be occurring. The effects of this are difficult to predict. Table 3-13 and Table 3-14 show comparison data for Tank 48H supernate and slurry results from various characterization reports, ranging from 2005 to 2022.

For the supernate data, the closest data set to this document is from the corrosion control sample from 2025 (HTF-48-25-17). The results (ICP-ES, Free OH, Cs-137, IC-Anions) from this corrosion control sample agree quite well with the current sample. For example, the common filtrate analyses (ICP-ES, Free OH, Cs-137, IC-Anions) differ by an average of 4%. When looking at the comprehensive 2015 sample, it is observed that the same analyte set also varies 4%, but there is more dispersion in the individual results.¹⁸ Notably, the 2014 sample has less sodium, because of fewer corrosion control additions, and more oxalate. Taken altogether, the current filtrate results are consistent with past results.

Table 3-13 Comparison Data of Tank 48H Supernate Results

	Units	2005 ¹⁹	2012 ¹⁵	2015 ¹⁸	2022 ¹	2025 ^{20*}	2025
Volume	gal	242,000	245,000	255,000	262,000	270,000	270,000
Density	g/mL	NA	1.20	1.23	1.28	1.26	1.27
Al	mg/L	2309	2050	2740	3370	2781	2910
B	mg/L	460	463	563	704	NA	592
Cr	mg/L	NA	42.1	53.8	63.2	NA	49.1
K	mg/L	267	286	448	<582	NA	534
Na	mg/L	87899	85800	106000	137000	137080	138000
Chloride	mg/L	172	187	703	827	703	736
Formate	mg/L	502	296	283	NA	257	271
Phosphate	mg/L	428	465	594	756	NA	396
Sulfate	mg/L	323	209	218	540	161	221
Nitrite	mg/L	23750	24500	28500	34029	29210	29500
Nitrate	mg/L	14250	11000	11500	13000	10230	10700
Oxalate	mg/L	1440	1590	1660	1540	1021	948
Free OH	M	1.34	1.02	1.73	1.69	1.72	1.48
Cs-137	dpm/mL	3.00E+07	2.57E+07	2.38E+07	2.42E+07	2.06E+07	2.13E+07
Phenol	mg/L	535	NA	275	43.8**	NA	561

NA = Not Applicable *Corrosion Control Sample **SVOA instrumentation used instead of HPLC

For the slurry data, the results (ICP-ES, Free OH, Cs-137, IC-Anions) are consistent but show more dispersion among individual results. The 2012 sample contains less free hydroxide compared to the 2022 and 2025 results, which can be attributed to the additions of the corrosion control solutions into the tank. Weight percent total solids have increased over the years due to the increase of soluble solids. Phenol results vary in the supernate and the slurry from the 2022 report. The discrepancy could be due to the type of instrumentation used for analyzing the data set and the conditions under which the Tank 48H sample was prepared. Biphenyl is a factor of three lower than the 2022 slurry, but trends with the 2005 and 2012 reported values.

Table 3-14 Comparison Data of Tank 48H Slurry Results

	Units	2005¹⁹	2012¹⁵	2022¹	2025
Volume	gal	242,000	245,000	262,000	270,000
Density	g/mL	1.17	1.22	1.26	1.28
Al	mg/L	2014	3110	3270	3098
B	mg/L	867	1070	676	1207
Cr	mg/L	70	74	56.1	62.6
K	mg/L	5155	3221	<582	2765
Na	mg/L	NA	NA	1.03E+05	1.38E+05
Free OH	M	NA	0.652	1.56	1.65
Biphenyl	mg/L	415	925	3043	1040
Phenol	mg/L	771	609	40.8*	762
TPB	mg/L	21000	19000	19700	18240
Total Dried Solids	%	20.2	23.2	25.8	27.4
Total Insoluble Solids	%	3.05	1.47	1.95	1.77
Cs-137	dpm/mL	7.53E+08	7.81E+08	7.04E+08	6.84E+08
Pu-238	dpm/mL	NA	3.13E+05	1.54E+05	2.43E+05

NA = Not Applicable *SVOA instrumentation used instead of HPLC

4.0 Conclusions and Recommendations

SRNL received approximately 3L of slurry (i.e., HTF-48-25-17) retrieved from Tank 48H on February 25, 2025. This report documents a detailed characterization of this sample and includes: supernate and slurry density, weight percent solids, chemical composition, and radionuclides. A comparison to results from analogous Tank 48H characterization studies performed in 2005, 2012, and 2022 is also provided.

A summary of key results from HTF-48-25-17 includes:

- The average densities of the HTF-48-25-17 for slurry was 1.28 g/mL and for filtrate was 1.27 g/mL.
- The average weight percent solids for total dried solids was 27.4 %, for dissolved solids was 26.1 %, and for insoluble solids was 1.77%.
- Cs-137 was analyzed in both the supernate and slurry. The result for Cs-137 in supernate was 2.13E+07 dpm/mL and the result for Cs-137 in slurry was 5.34E+08 dpm/g.
- High-Performance Liquid Chromatography (HPLC) was used to measure tetraphenylborate (TPB) and degradation products in the supernate and slurry. Phenol (7.62E+02 mg/L), TPB (1.82E+04 mg/L), and biphenyl (1.04E+03 mg/L) were detected in the slurry. Phenol was the only degradation product detected in the supernate. Volatile Organic Analysis (VOA) was used to measure volatile organic species. Benzene was detected and reported at 104 mg/L.

During transfer of the slurry, it was observed that the material could not be transferred out by peristaltic pumping due to a large amount of solids remaining at the bottom of the metal sampler. It is recommended that any future work that involves Tank 48 a peristaltic pump is not used to pump the material.

5.0 Reference

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Appendix A. Mass Spectrometry results for supernate and slurry

Mass Spectroscopy Results for Supernate and Slurry					
	Supernate Duplicate 1 (mg/L)	Supernate Duplicate 2 (mg/L)		Slurry Duplicate 1 (µg/g)	Slurry Duplicate 2 (µg/g)
59	< 6.65E-02	< 6.59E-02		0.111	0.0963
84	< 1.33E-02	< 1.32E-02	<	4.79E-02	< 4.90E-02
85	0.134	0.140		2.18	2.11
86	< 1.33E-02	< 1.32E-02		0.0736	0.0778
87	0.219	0.233		3.73	3.64
88	< 6.65E-02	< 6.59E-02		0.571	0.632
89	< 1.33E-02	< 1.32E-02	<	4.79E-02	< 4.90E-02
90	< 1.33E-02	< 1.32E-02		0.259	0.275
91	< 1.33E-02	< 1.32E-02		0.109	0.108
92	0.223	0.227		0.361	0.348
93	< 1.33E-02	< 1.32E-02		0.179	0.16
94	0.141	0.145		0.287	0.286
95	1.689	1.741		1.52	1.48
96	0.261	0.270		0.333	0.33
97	1.556	1.609		1.32	1.29
98	1.742	1.794		1.59	1.55
99	1.208	1.241		1.08	1.05
100	1.609	1.662		1.44	1.4
101	0.115	0.121		0.109	0.104
102	0.101	0.106		0.0964	0.0925
103	0.127	0.127		0.129	0.131
104	0.067	0.068	<	2.39E-01	< 2.45E-01
105	< 6.65E-02	0.066	<	2.39E-01	< 2.45E-01
106	< 6.65E-02	< 6.59E-02	<	2.39E-01	< 2.45E-01
107	0.032	0.028	<	2.39E-01	< 2.45E-01
108	< 6.65E-02	< 6.59E-02	<	4.79E-02	< 4.90E-02
109	< 1.33E-02	< 1.32E-02	<	2.39E-01	< 2.45E-01
110	< 1.33E-02	< 1.32E-02	<	4.79E-02	< 4.90E-02
111	< 1.33E-02	< 1.32E-02	<	4.79E-02	< 4.90E-02
112	< 1.33E-02	< 1.32E-02	<	4.79E-02	< 4.90E-02
113	< 1.33E-02	< 1.32E-02	<	4.79E-02	< 4.90E-02
114	< 1.33E-02	< 1.32E-02	<	4.79E-02	< 4.90E-02
116	0.076	0.079		0.0762	0.0749

117	0.046	0.048	<	4.79E-02	<	4.90E-02
118	0.133	0.133		0.127		0.127
119	0.057	0.059		0.0746		0.0724
120	0.177	0.187		0.165		0.175
121	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
122	0.040	0.040	<	4.79E-02	<	4.90E-02
123	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
124	0.059	0.059		0.055		0.0557
125	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
126	0.065	0.067		0.0585		0.0527
128	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
130	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
133	0.423	0.438		11.4		11.1
134	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
135	0.05	0.05		1.36		1.31
136	< 1.33E-02	< 1.32E-02		0.115		0.101
137	0.13	0.13		5.88		5.74
138	0.050	0.044		1.1		0.995
139	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
140	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
141	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
142	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
143	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
144	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
145	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
146	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
147	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
148	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
149	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
150	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
151	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
152	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
153	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
154	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
155	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
156	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
157	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02

158	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
159	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
160	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
161	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
162	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
163	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
164	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
165	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
166	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
167	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
168	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
169	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
170	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
171	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
172	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
173	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
174	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
175	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
176	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
177	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
178	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
179	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
180	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
181	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
182	0.358	0.361		0.311		0.295
183	0.196	0.199		0.17		0.165
184	0.419	0.427		0.37		0.352
185	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
186	0.395	0.404		0.348		0.338
187	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
191	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
193	< 1.33E-02	< 1.32E-02	<	2.39E-01	<	2.45E-01
194	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
195	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
196	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
198	< 1.33E-02	< 1.32E-02		1.13		1.1
203	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02

204	0.014	0.013		0.561		0.548
205	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
206	0.214	0.216		0.803		0.772
207	0.189	0.197		0.713		0.707
208	0.467	0.475		1.77		1.72
230	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
232	< 1.33E-02	< 1.32E-02	<	2.39E-01	<	2.45E-01
233	0.020	0.019		0.0505		0.0492
234	0.112	0.113		0.277		0.271
235	0.229	0.231		0.584		0.575
236	0.051	0.050		0.125		0.123
237	0.051	0.050		0.183		0.18
238	1.623	1.662		3.81		3.76
239	< 6.65E-02	< 6.59E-02	<	2.39E-01	<	2.45E-01
240	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
241	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
242	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
243	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02
244	< 1.33E-02	< 1.32E-02	<	4.79E-02	<	4.90E-02

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