Removal of Organic Compounds From the "Contaminants of Concern" List for Tank Farm Vadose Zone Characterization

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

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Reviews of many historical tank farm documents indicate that the tank farm operations personnel were well aware of the hazards associated with the boiling waste tanks and, for safety reasons, were careful to not intentionally add organics to these tanks. Thus, assuming that any organics were added to tanks SX-107, SX-108, or SX-109 is unreasonable. In addition, had any been added, the organics likely would not have survived long enough to have entered the vadose zone during a subsequent leak event.

3.0 TANK WASTE TRANSFER RECORDS

Records of waste transfers into and out of Hanford Site high-level waste tanks up to 1994 are documented in the *Waste Status and Transactions Record Summary* (WSTRS, Rev. 2) (Agnew et al. 1995). The WSTRS, Rev. 2, also provides a record of the waste type and origin of the incoming waste. According to the WSTRS, Rev. 2, tanks SX-107 through SX-109 received only REDOX high-level waste. Because this waste stream contained few, if any, organic compounds, a reasonable assumption is that no organics were lost from these tanks during leak events.

The waste transfer records in the WSTRS, Rev. 2, were combined with waste stream compositions derived from process flow sheets to develop the Hanford Defined Waste (HDW) Model (Agnew et al. 1997). The HDW model was used to provide a current (as of 1994) tank-by-tank waste composition and waste inventory and to predict the composition and inventory of 24 SSTs suspected or confirmed as having leaked waste into the vadose zone. This modeling effort again indicates that no organic compounds are present in tanks SX-107, -108, and -109. Because the waste did not contain organics, pursuing organic analyses of soil samples collected near or under these tanks is unnecessary.

During 1971 and 1972, a number of tanks in the SX farm received waste materials that undoubtedly contained organic compounds. According to transfer records in WSTRS, Rev. 2, 8 of the 15 SX tanks received "RIX" waste in 1971 and 1972. The RIX waste stream came from processing REDOX high-level waste in the B Plant to remove ^{137}Cs and/or ^{90}Sr . This type of B Plant waste is known to contain significant quantities of organic complexing agents. One other tank, SX-103, stored organic wash waste (OWW) from 1971 through 1975. These nine tanks also received evaporator bottoms (EB). The composition of the EB waste reflects the composition of the supernatant liquid sent to the evaporator; these compositions currently are unknown. Thus, nine SX tanks likely contained some organic residue that could potentially result in organics being detected in the analysis of tank headspace gases for volatile organic compounds. The six tanks in the SX farm that did not receive waste streams containing organic compounds are SX-107, -108, -109, -112, -113, and -115. However, as discussed in detail in Section 4, the presence of organic compounds in the tank waste does not necessarily indicate that the organic compounds will be found in soil samples contaminated with that waste when those samples are analyzed using EPA GC-MS methods.

4.0 ORGANIC ANALYSES OF TANK T-106 CORE SAMPLES

In 1973, 435,321 L (115,000 gal) of organic-rich tank waste was lost from tank T-106. This is the largest documented leak from a Hanford Site tank storing high-level waste (Hanlon 1999). This leak event was extensively investigated (Routson et al. 1979 and references therein). In 1993, a borehole was drilled near tank T-106 through the most highly contaminated zone (Freeman-Pollard 1994). Extensive chemical characterization of cores from this borehole provided a wealth of information about the movement of chemicals and radionuclides in Hanford Site soils (Jones et al. 1998). The analytical data generated during the 1993 effort are included in Appendix 1. This appendix also includes the figures from the body of the report showing selected radionuclides and chemicals as a function of depth.

The waste lost in this leak event was produced in the B Plant as part of the isotope recovery program. Although the major isotope recovery programs at the B Plant involved removing ^{137}Cs and ^{90}Sr from Plutonium-Uranium Extraction (PUREX) process high-level waste streams, at certain times other radionuclides and other waste types were targeted. Appendix 2 contains an overview of the various separations chemistries. Although many organic compounds were used in the separations processes, most were aminocarboxylic- or phosphate-based organic acids. Interestingly, the EPA GC-MS methods generally do not detect these classes of organic compounds. Although analytical methods exist for detecting these compounds in Hanford Site tank waste matrices, they are used only in research efforts. For example, Toste and coworkers (1987) were able to identify approximately 75 percent of the organic compounds contributing to the 44 g/L total organic carbon (TOC) in the organic-rich complex tank waste sample already on hand. The sample likely was similar to the waste lost at tank T-106. Toste and coworkers used several analytical techniques, including derivatization of hydrophilic organics followed by analyses using a GC-flame ionization detector, GC-MS, GC-Fourier-transform infrared spectroscopy, and high-pressure liquid chromatography, to identify these compounds. Most of the organic compounds identified in this effort were aminopolycarboxylic acids and their degradation products.

The waste material lost from tank T-106 in 1973 originated in the B Plant and moved through several tanks before reaching tank T-106. The HDW model was used to estimate the composition of the waste lost from tank T-106. The compositional estimate for the organic compounds tracked by the HDW model are listed in Table 1. The estimated quantities of organics lost during the leak event also are provided in Table 1.

Based on this assessment, an estimated 1.6 metric tons of organic carbon were lost in the 435,321 L (115,000 gal) of waste leaked from tank T-106. The initial investigation estimated that 1700 m³ of soil were contaminated by this leak. If the organic carbon was uniformly distributed through the mass of contaminated soil with a specific gravity of 1.6 (typical for the Hanford Site soil), the TOC would be approximately 600 mg/kg (or ppm). In all likelihood, organic compounds lost to the vadose zone would partition much like other chemicals and radionuclides. (For an example, see Figures A1-1 through A1-17 in Appendix 1.) Thus, one would expect to see leaked organic compounds concentrated in specific geologic layers. Table A1-5 in Appendix 1 reports TOC results for 14 core samples at depths ranging from 4 m (13 ft) to 50 m (163 ft). TOC values of 14,000 mg/kg were reported at a depth of 20 m (67 ft), 9700 mg/kg at 23 m (74 ft), 2900 mg/kg at 29 m (96 ft), and 4800 mg/kg at 31 m (103 ft). Other

TOC values were in the 100 mg/kg to 500 mg/kg range. However, the TOC data set contains many gaps.

Considerable caution is required in interpreting soil TOC results because organic carbon is a natural component of soils. For example, high TOC values could reflect a buried soil horizon rather than tank waste. Quantitative interpretation of soil TOC data would require data from nearby uncontaminated soils. Nevertheless, these TOC data suggest regions that could be evaluated using other organic analysis techniques. The high TOC region from 20 m to 23 m (67 to 74 ft) coincides with high levels of radioactivity and chemicals. The high TOC values around 30 m (100 ft) fall in the Plio-Pleistocene unit. A number of tank waste components exhibit peaks in this unit (see figures in Appendix 1). Clearly, routine analysis of TOC in all tank farm vadose zone samples would lead to a far better understanding of the potential extent of organic contamination coming from tank waste leaks.

In addition to the TOC analyses discussed earlier, an extensive number of soil samples were submitted for analyses of organics using EPA protocols. Twenty-eight soil samples and 22 "sampling-protocol" blanks were submitted for analysis of volatile organic compounds using GC-MS techniques following regulatory-driven procedures. The GC-MS method for volatile organics provides quantitative analyses for 28 regulated, low-boiling-point, organic compounds plus tentative identification for many other organic compounds. Twenty-eight soil samples and 4 "sampling protocol" blanks were submitted for analysis of semivolatile organic compounds using GC-MS techniques following regulatory-driven procedures. The GC-MS method for semivolatile organics provides quantitative analysis for 67 regulated, moderate-boiling-point, organic compounds, plus tentative identification for many others. The volatile and semivolatile organic analyses data contained no gaps.

Analytical results for volatile and semi-volatile organics and TOC are compiled in Tables A1-3, A1-4, A1-5, and A1-7 in Appendix 1. The volatile organic analysis and TOC results are summarized in Table 2. The semi-volatile organic analysis results are summarized in Table 3. The "tentatively identified compounds" organic analysis results are summarized in Table 4.

Reporting requirements associated with the EPA GC-MS protocols are complex and involve numerous quality flags depicted through various "letter" qualifiers. Nondetected targeted compounds are reported as numerical values representing the contractually required quantitation limit (RQL) followed by the letter "U." Targeted organic compounds detected at levels above the RQL and meeting all quality assurance/quality control (QA/QC) requirements are reported as numerical values with no letter flag. Targeted organic compounds detected at levels below the RQL are reported with a "J" flag. Analytical data for targeted organic compounds are flagged with "B" and "BJ" designators when targeted organic compounds are found in any laboratory QA/QC samples analyzed with the samples. The flags are defined in the glossary in Appendix 1.

The organic analysis data summaries in Tables 2 through 4 are separated as "non-flagged" data, "J flagged" data, and "B and BJ flagged" data. By separating the data in this manner, one can quickly understand the relationships between the detected organic compounds reported in the soil samples and the detected organic compounds in the QA/QC samples.

As shown in Table 2, three non-flagged volatile organic results were reported for the 28 samples submitted for analyses. Acetone, in the 10 mg/kg to 20 mg/kg range, was reported in two samples and methylene chloride, at 11 mg/kg was reported in a third sample. Acetone, methylene chloride, and toluene frequently were reported in the J flagged data at 10 mg/kg or less. These compounds were reported at comparable levels in the B and BJ flagged data. A reasonable interpretation is that all nonflagged, J flagged, and B/BJ flagged data reported for these 28 samples reflect nothing more than analytical laboratory contamination. Analytical laboratory contamination at these levels is extremely common for the compounds reported (e.g., acetone, toluene, and methylene chloride).

As shown in Table 3, only one nonflagged data point was reported for the 28 samples submitted for semivolatile organic analyses—di-n-butylphthalate was reported at 420 mg/kg. In fact, the only class of compounds reported was the “phthalates.” Phthalates were frequently reported in both J flagged and B/BJ flagged data. Phthalates are commonly used plasticizers and are found as contaminants in any material contacting “plastic.” A reasonable interpretation is that all the nonflagged, J flagged, and B/BJ flagged semivolatile data reported for these 28 samples reflect nothing more than analytical laboratory contamination. Analytical laboratory contamination at these levels of phthalates is extremely common (EPA 1996).

By definition, all the data for tentatively identified compounds are reported as flagged. As shown in Table 4, unknown hydrocarbons, unknown alkanes, and unknown ketones were commonly reported at essentially all depths. Although some values reported were in excess of a milligram per kilogram (part per million), QA/QC sample contamination was extensive. Thus, little can be gleaned from these data.

Apparently, a component of the 1993 tank T-106 leak characterization activities was focused on determining potential RCRA-regulated organic compounds in the soil column near the tank. These efforts failed to detect anything. Such results should discourage future soils analysis following these protocols unless a strong case can be made that useful information would be developed. For example, analyses of vadose zone samples collected near the carbon tetrachloride plume in the 200 West Area may indicate commingling of cribbed waste with tank waste.

A reasonable conclusion is that resources could be better used in other soil characterization activities. For example, the limited soil TOC values reported for a small number of core samples (also subjected to GC-MS protocols) taken near tank T-106 suggest that a large quantity of organics may be just below the bottom of the tank. Had more core samples been submitted for TOC analysis, the sample results could have been more conclusive.

5.0 CONCLUSIONS

Based on this assessment, the analysis of tank farm vadose zone samples for organic compounds using EPA GC-MS methods will not provide any useful information, and therefore should not be performed. The environment within the boiling-waste tanks (including tanks in S, SX, A, and AX farms) would likely have destroyed any organic compounds added to them. The waste transfer records indicate that organics were not added to the boiling-waste tanks.

Some Hanford Site waste tanks are known to have received large quantities of organic compounds, and some of these tanks are known to have leaked. However, even though large quantities of organic compounds were lost during the tank T-106 leak event in 1973 and considerable resources were dedicated to determining whether RCRA-regulated organic compounds were present in the resulting tank waste-contaminated soil, the data produced using EPA GC-MS methods provided no information on the "nature and extent" of tank waste contamination in the vadose zone. The EPA GC-MS method is not well suited to determining the classes of organic compounds generally lost during SST leaks. In addition, RCRA-regulated compounds for which the GC/MS methods were developed generally are not expected in tank waste lost during leaks. However, routine analysis of TOC for all tank farm vadose zone core samples is highly recommended.

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Table 1. Inventory of Organic Compounds Lost in Tank T-106 Leak.

Only incoming (T-107 +T-105) waste leaks.							
Analyte	Conc gMoles/L	L/gal	Conc gMoles/gal	g/gfw	Gallons lost	Grams lost	kg lost
Density (g/cc)	1.05 E+00						
wt.% H2O	6.42 E+01						
TOC wt.%C	3.55 E-01						1630
C6H5O73-	2.02 E-02	3.8	7.67 E-02	189	115,000	1.67 E+06	1.67 E+03
EDTA4-	3.12 E-04	3.8	1.18 E-03	292	115,000	3.98 E+04	3.98 E+01
HEDTA3-	6.23 E-04	3.8	2.37 E-03	278	115,000	7.57 E+04	7.57 E+01
glycolate-	4.36 E-02	3.8	1.66 E-01	76	115,000	1.45 E+06	1.45 E+03
acetate-	2.46 E-05	3.8	9.35 E-05	59	115,000	6.35 E+02	6.35 E-01
oxalate2-	1.18 E-09	3.8	4.48 E-09	88	115,000	4.54 E-02	4.54 E-05
DBP	1.10 E-02	3.8	4.17 E-02	104	115,000	4.99 E+05	4.99 E+02
butanol	1.10 E-02	3.8	4.17 E-02	74	115,000	3.55 E+05	3.55 E+02
						Total =	4.08 E+03

Table 2. Volatile Organic Analysis Data Summary. (3 sheets)

Depth of Sample	Unflagged Data?	Compounds Reported (ppb)	"J" Flagged Data?	Compounds Reported (ppb)	"B" & "BJ" Flagged Data	Compounds Reported (ppb)	TOC Analysis (mg/kg)
13	No		No		No		500
21.8	No		No		No		209
30	No		No		Yes	MetCl 2; Acet 15	176
36.4	No		Yes	MetCl 4, Tol 1	No		NA
38.9	No		Yes	MetCl, 4	No		NA
43.4	Yes	Acetone, 11	Yes		No		NA
46.1	No		Yes	Tol 1	No		NA
52.8	No		Yes	Acet 7, Tol 0.9	No		NA
52.8	No		Yes	Tol 1	No		NA
59.8	No		No		No		NA
67.1	No		No		No		14000
74	NA		NA		No		9730
82.7	No		No		No		674
T. Blank	No		Yes	MetCl 8, Tol 1, Xyl 2	No		
T. Blank	No		Yes	MetCl 1	No		
90.8	No		Yes	Tol 2	No		448
90.8	No		Yes	Tol 2	No		502
T. Blank	No		No		No		
95.8	No		Yes	Tol 7	No		2920

Table 2. Volatile Organic Analysis Data Summary. (3 sheets)

Depth of Sample	Unflagged Data?	Compounds Reported (ppb)	"J" Flagged Data?	Compounds Reported (ppb)	"B" & "BJ" Flagged Data	Compounds Reported (ppb)	TOC Analysis (mg/kg)
132.4	No		No		No		NA
155.4	No		No		Yes	Acet 12	NA
162.7	No		Yes	MetCl 4	No		97.2
162.7	No		Yes	Tol 1	Yes	Acet 13	NA
173.8	No		Yes	Tol 2	Yes	Acet 10	NA
179.6	No		No		No		NA
T. Blank	No		No		No		
T. Blank	No		Yes	Chloroform 2	Yes	MetCl 2	
T. Blank	No		No		No		
T. Blank	No		Yes	MetCl 5	No		
T. Blank	No		Yes	MetCl 9			

MetCl = methylene chloride

Tol = toluene

Acet. = acetone

Table 3. Semi-Volatile Organic Analysis Summary. (2 sheets)

Depth of Sample	Unflagged Data?	Compounds Reported (ppb)	"J" Flagged Data?	Compounds Reported (ppb)	"B" & "BJ" Flagged Data	Compounds Reported (ppb)
13	No		No		No	
21.8	No		No		No	
30	No		no		No	
36.4	No		No		No	
38.9	No		Yes	Buytalphthlate 110	No	
39.8	No				No	
E. Blank	No				No	
43.4	No		Yes	Buytalphthlate 12	No	
46.1	Yes	Butylphthalate 420	Yes	Octylphthlate 19	No	
52.8	No		Yes	Etphth 34; Ociphth 13; Buphth 120	No	
52.8	No		Yes	Etphth 17; Ociphth 22; Buphth 24	No	
59.8	No		No		No	
67.1	No		No		No	
74	No		No		No	
82.7	No		No		No	
90.8	No		No		No	

Table 3. Semi-Volatile Organic Analysis Summary. (2 sheets)

Depth of Sample	Unflagged Data?	Compounds Reported (ppb)	"J" Flagged Data?	Compounds Reported (ppb)	"B" & "BJ" Flagged Data	Compounds Reported (ppb)
90.8	No		No		No	
95.8	No		Yes	Etphth 54	Yes	Butylphthlate 76
97.8	No		No		Yes	Butylphthlate 78
97.8	No		No		No	
101	No		Yes	Butylphthlate 110	No	
103	No		No		Yes	Butylphthlate 97
E. Blank	No		Yes	Diethylphthlate 4	No	
F. Blank	No		No		No	
F. Blank	No		No		No	
109.8	No		No		Yes	Butylphthlate 150
117.3	No		No		Yes	Butylphthlate 360
120.3	No		No		Yes	Butylphthlate 290
132.4	No		No		Yes	Butylphthlate 210
145.2	No		No		No	
155.4	No		No		Yes	Butylphthlate 190
162.7	No		No		Yes	Butylphthlate 290
173.8	No		No		Yes	Butylphthlate 230
179.6	No		No		Yes	Butylphthlate 150

FEED PREPARATION

SIX MAJOR STEPS in D2HEPA SOLVENT EXTRACTION PROCESS

- **Metathesis Feed Preparation**
- **Sr / Rare Earth Extraction into D2EHPA/TBP**
- **Strip of Strontium from Solvent**
- **Pm / Cerium Partitioning**
- **Strip of Cerium from Solvent**
- **Solvent Treatment**

Sr-90 AND RARE EARTH EXTRACTION FROM PAW

STRIPPING OF Sr-90 FROM D2EHPA

STRONTIUM PURIFICATION PROCESSES

- SOLVENT EXTRACTION**
- SULFATE PRECIPITATION**
- HYDROXIDE PRECIPITATION**
- RARE EARTH PRECIPITATION**
- ION EXCHANGE**
- BARIUM REMOVAL**

PARTITIONING of Ce FROM OTHER RARE EARTHS

D2EHPA SOLVENT WASH

CS-137 RECOVERY BY LINDE AW-500 ZEOLITE ION EXCHANGE

Cs-137 Recovery by Duolite ARC-359 Ion Exchange

Cs-137 RECOVERY USING PHOSPHOTUNGSTIC ACID (PTA)

Technetium Recovery From PSN Using Ion Exchange

SR PURIFICATION WITH D2EHPA EXTRACTION

Sr-90 PURIFICATION, STRIP AND CONCENTRATION

SUMMARY

- **PRIMARY REFERENCE : ISO -100 DEL
AUGUST 31, 1967**
- **KEN GASPER'S DRAFT BOOK CHAPTER**
- **OTHER PROCESSES**
 - **D2EHPA ACTINIDE RECOVERY**
 - **RARE EARTH PURIFICATION**
 - **RHODIUM AND PALLADIUM RECOVERY**
 - **WESF**

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