

Pacific Northwest National Laboratory

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Organic Speciation of AX-102, BX-104, C-104, C-201, and C-202 Tank Wastes

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Introduction

This report describes the work performed during FY 1998 by Pacific Northwest National Laboratory to identify organic components in Hanford waste tank samples to support resolution of the organic tank safety issue. The major focus during FY 1998 was the analysis of actual tank wastes under Hanford Analytical Services Quality Assurance (HASQARD) compliance.

The organic analysis methods that were optimized during this task are illustrated by their application to samples listed below.

AX-102
C-104 c162s2UH
C-202

BX-104
C-201

where c= core number, s = segment number, and UH = upper half. The samples from C-201 and C-202 were grab samples. The sample from Tank AX-102 was an auger sample. Table 1 lists the samples received by Pacific Northwest National Laboratory (PNNL) for analyses, the jar/vial numbers, and associated laboratory identification numbers from the chain of custody.

Table 1. Samples Shipped to PNNL with Associated ID Numbers

Tank	Jar/Vial	Core	S	Sample ID
AX-102	13480			S97M000169
C-104	13440	162	2	S97M000170
BX-104				
C-201	13619			S97T002248
C-202	13673			S97T000038

Samples from Tanks AX-102, C-104, BX-104, C-201, and C-202 were analyzed for organic constituents. Samples were analyzed using derivatization gas chromatography/flame ionization detection (GC/FID) for chelators and chelator fragments, ion chromatography (IC) for low-molecular-weight organic acids, and ion-pair chromatography (IPC) for chelators. The major components identified include low-molecular-weight acids (e.g., oxalic acid), chelators (e.g. EDTA) and chelator fragments.

Results

The concentrations of the analytes are summarized in Table 2. The concentrations for oxalate, glycolate, formate, and citrate were determined by ion chromatography (IC). The concentrations for EDTA, NTA, ED3A, and HEDTA were determined using GC/FID. The concentrations for s-EDDA were determined using IPC, and the values for EDTA and HEDTA were corroborated using IPC.

Table 2. Summary of Data from Analyses of Received Samples. Concentrations are listed in mg C/g Sample.

Component	Tank				
	AX-102	C-104	BX-104	C-201	C-202
Ox	8.51	2.41	1.19	7.94	6.82
Glycolate	5.73	5.33 (2)	2.83	2.22	2.52
Formate	3.51	1.13 (3)	0.43	0.14	0.20
IDA	1.41	0.22 (9)	nd	nd	nd
CA	3.43	nd	4.17	nd	nd
NTA	0.41	nd	nd	nd	nd
ED3A	0.002	0.001	nd	nd	nd
EDTA	3.09	nd	0.02	nd	nd
HEDTA	1.26	nd	nd	nd	nd
s-EDDA	0.26	nd	nd	nd	nd
Ox	oxalate				
IDA	iminodiacetic acid				
CA	citric acid				
NTA	nitrilotriacetic acid				
ED3A	ethylene				
EDTA	ethylenediaminetetraacetic acid				
HEDTA	N-(2-hydroxyethyl)ethylenediaminetriacetic acid				
s-EDDA	symmetric ethylenediaminediacetic acid				
nd	not detected				
()	relative percent difference. Calculated for duplicate samples				

The following discussion details the quality (QA) results of the blank, blank spike, blank-spike duplicate, matrix spike, and matrix-spike duplicate as a function of the analytical technique.

Ion-Pair Chromatography

The process blank and duplicate samples were spiked with EDTA, NTA, oxalate, and formate. Ion-pair chromatography was used to quantitate EDTA and NTA. The QA results for IPC analyses are shown in Table 3. The average percent recovery was 109% for EDTA and 0%

for NTA. It is apparent that NTA was omitted from the spiking solution for the process blank spike and duplicate.

Table 3. Results of IPC Analyses of the Process Blank, Blank Spike, and Blank-Spike Duplicate (mg/mL of processed sample solution)

Component	Process Blank	Blank Spike	Blank-Spike Duplicate
NTA	nd	nd	nd
EDTA	nd	0.054	0.055

Table 4 shows the results from IPC analyses of the matrix spike and matrix-spike duplicate. The average percent recovery was 81% for EDTA and 47% for NTA.

Table 4. IPC Results of Matrix Spike and Matrix-Spike Duplicate

Component	Matrix Spike	Matrix Spike Duplicate
NTA	0.03	0.02
EDTA	0.22	0.18

Table 5 lists the results from analyses of C-104, C-104 duplicate, matrix spike, and matrix-spike duplicate.

Table 5. Results of Analyses of C-104, Duplicate, Matrix Spike, and Matrix-Spike Duplicate. Concentrations are in mg C/g Sample.

Component	C-104	C-104 Dup	Matrix Spike	Matrix-Spike Dup
Ox	0.10	0.35	0.59	0.55
Glycolate	5.3	5.4	6.5	7.6
Formate	1.13	1.16	17.8	21.8
NTA	nd	nd	0.52	0.38
EDTA	nd	nd	4.32	4.76
Ox	oxalate			
NTA	nitrilotriacetic acid			
EDTA	ethylenediaminetetraacetic acid			
nd	not detected			

Ion Chromatography

The results of analyses of the process blank, blank spike, blank-spike duplicate, matrix spike, and matrix-spike duplicate using IC are shown in Table 6. The percent recoveries for the blank spike and duplicate were 75% and 94% for oxalate and formate, respectively. The average percent recoveries for the matrix spike and duplicate were 46% and 50% for oxalate and formate, respectively.

Table 6. Results of IC Analyses of Process Blank, Blank Spike, Blank-Spike Duplicate, Matrix Spike, and Matrix-Spike Duplicate

Sample	Oxalate	Formate
Process Blank	nd	nd
Blank Spike	0.04	0.05
Blank Spike Dup	0.04	0.04
Matrix Spike	0.77	0.63
Matrix-Spike Dup	0.59	0.56

Gas Chromatography/Flame Ionization Detection

The results from analyses of the process blank, blank spike, blank-spike duplicate, matrix spike, and matrix-spike duplicate are listed in Table 7. The average percent recovery for EDTA for the blank spike and duplicate was 98%. The average percent recovery for NTA was 68%. The average percent recovery for the matrix spike and duplicate was 69% and 51% for EDTA and NTA, respectively.

Table 7. Results of GC/FID Analyses of Process Blank, Blank Spike, Blank-Spike Duplicate, Matrix Spike, and Matrix-Spike Duplicate. Concentrations are in mg C/g Sample.

Sample	EDTA	NTA
Process blank	nd	nd
Blank Spike	0.05	0.05
Blank-Spike Dup	0.05	0.04
Matrix Spike	0.16	0.02
Matrix-Spike Dup	0.15	0.02