ANALYSIS OF VOLATILE HEADSPACE GASES
SAMPLED BY CRYOGENIC TRAPS FROM
WESTINGHOUSE HANFORD COMPANY
TANK 242-C- 112 MARCH 1992

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SUMMARY

In this report, results are given from gas chromatography/mass spectrometry (GC/MS) analyses of the headspace samples obtained by using cryogenic traps from Westinghouse Hanford Company (WHC) Tank 112-C during the month of March, 1992. Cryogenic samples were received and logged into Battelle Pacific Northwest Laboratories notebook #BNW54426, and all subsequent information was recorded in this notebook. Samples were analyzed as received with no sample preparation. Analyses included direct GC/MS for volatile/semi-volatile components, and direct GC/MS for ammonia. Purge and trap GC/MS analysis was not done in this study. The cryogenic vapor trap system was designed by WHC and is not discussed in this report. In addition, aliquots were sent to Karl Pool, Pacific Northwest Laboratory, for hydrogen cyanide analysis by ion chromatography, and the results are reported here. All concentrations are reported for the methanol extract solutions. To calculate concentrations in the headspace, the cryo-sampling air volume and the methanol rinse volume must be obtained from the cryo-sampling personnel at WHC. Triplicate analyses were done on all samples, and the average concentrations and standard deviations are reported. One significant result was that no ammonia was detected.

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INTRODUCTION

In this report, results are given from gas chromatography/mass spectrometry (GC/MS) analyses of the headspace samples obtained by using cryogenic traps from Westinghouse Hanford Company (WHC) Tank 242-C-112 (Tank 112-C) during the month of March, 1992. This work was performed by Pacific Northwest Laboratory (PNL)^(a) for WHC using headspace samples provided by WHC.

Analyses included direct GC/MS for volatile/semi-volatile components and direct GC/MS for ammonia. Purge-and-trap GC/MS analysis was not done in this study. The cryogenic vapor trap system was designed at WHC and is not discussed in this report. In addition, aliquots were sent to Karl Pool, PNL, for hydrogen cyanide analysis, and the results are reported here. All concentrations are reported for the methanol extract solutions. To calculate concentrations in the headspace, the cryo-sampling air volume and the methanol rinse volume must be obtained from the cryo-sampling personnel at WHC. Triplicate analyses were done on all samples, and the average concentrations and standard deviations are reported.

(a) Pacific Northwest Laboratory is operated for the U. S. Department of Energy by Battelle Memorial Institute under Contract DE-ACO6-76RLO 1830.

SAMPLE HANDLING AND PREPARATION

The cryogenic samples were received in 40-mL glass vials with Teflon®-lined caps from WHC during March 1992. Samples were stored at 4°C +/- 2°C before analysis. Samples were logged into Battelle Pacific Northwest Laboratory notebook (#BNW54426). All subsequent information was recorded in this notebook. Samples were analyzed as received with no sample preparation.

ANALYSIS METHOD

Analysis of the cryogenic traps by GC/MS was achieved using procedure PNL-ALO-334. The instrument used for analysis was a Hewlett-Packard, model 5890/5970 (Simi Valley, California), (PNL property #WB37047). Analyses were achieved using the direct injection GC/MS method. Different GC conditions were used for volatile/semivolatile analyses and for the ammonia analyses. Tentative identification of unknowns was accomplished using mass spectral interpretation and comparison of the mass spectra with the standard spectra from the Environmental Protection Agency/National Institute of Standard Technology (EPA/NIST) library of mass spectra.

HYDROGEN CYANIDE ANALYSIS

Five-mL aliquots were delivered to Karl Pool (PNL) for hydrogen cyanide analysis by ion chromatography (IC). Samples were stored at 4°C until analysis. Quality Assurance (QA) plan MCS-033 was followed at PNL MA-70 impact level II. Results are reported in this letter report.

GAS GRAB SAMPLES

Two 150-mL stainless steel canisters representing gas grab samples from WHC Tank 112-C were received from WHC for direct GC/MS analysis. The analysis method required direct gas sample/standard injection into the GC/MS instrument. The oven temperature profile consisted of -40°C for 0.5 min followed by 16°/min to 240°C. The GC oven was then held at 240°C for 7 min. to elute potential semivolatiles such as normal parafin hydrocarbon (NPH).

QA/QC

The QA/Quality Control (QA/QC) procedures in the analytical procedure were followed. Instrument blanks, tune/mass calibration, internal standards, calibration, and calibration check samples were all met. This report does not address the efficiency of the cryogenic traps.

DATA

The GC/MS raw data were archived on to a magnetic storage tape, located in lab 8B of the 329 building, for permanent storage. Hard copy output data that include several pages of ion chromatograms, mass spectra, calibration/quantitation data, and library-searched spectra are stored in the GC/MS file cabinet located in room 2 of the 329 Building. The present report includes the final quantitation tables and pertinent information. Data are also maintained in Laboratory Record Book (LRB) #BNW-54426. Table 1 shows the inventory of samples received from WHC.

TABLE 1. Sample Inventory of Vapor Samples From WHC Tank 112-C

<u>Date Received</u>	<u>Sample ID</u>	<u>COC#</u>	<u>Description</u>
3/10/92	C112/7S2/1B	2277B	Cryo extract sample
3/10/92	C112/7S2/2B	2277B	Cryo extract sample
3/10/92	C112/7S1/SB	2277B	Cryo extract sample
3/12/92	C112/7M1/1B	2278B	Cryo extract sample
3/12/92	C112/7M1/2B	2278B	Cryo extract sample
3/12/92	C112/7M1/SB	2278B	Cryo extract sample
3/18/92	C112/7L2/1B	2293B	Cryo extract sample
3/18/92	C112/7L2/2B	2293B	Cryo extract sample
3/18/92	C112/7L2/SB	2293B	Cryo extract sample
3/18/92	C112/7L3/1B	2293B	Cryo extract sample
3/18/92	C112/7L3/2B	2293B	Cryo extract sample
3/18/92	C112/7LS/SB	2293B	Cryo extract sample
3/18/92	C112/2S1/1B	2293B	Cryo extract sample
3/18/92	C112/2S1/2B	2293B	Cryo extract sample
3/18/92	C112/2S1/SB	2293B	Cryo extract sample
3/18/92	BLIND	2293B	WHC Blind
3/20/92	C112/2M1/1B	2279B	Cryo extract sample
3/20/92	C112/2M1/2B	2279B	Cryo extract sample
3/20/92	C112/2M1/SB	2279B	Cryo extract sample
3/20/92	C112/2L1/1B	2295B	Cryo extract sample
3/20/92	C112/2L1/2B	2295B	Cryo extract sample
3/20/92	C112/2L1/SB	2295B	Cryo extract sample
3/17/92	C112/7L3/R Grab		Gas grab sample
3/18/92	C112/7L1/R Grab		Gas grab sample

COC# = Chain of Custody receipt number

RESULTS

Triplicate analyses were done on all samples, and the average concentrations and standard deviations are reported. All concentrations are reported in mg/L for the methanol extract solutions.

AMMONIA AND HYDROGEN CYANIDE

As seen in Table 2, ammonia (NH_3) was not detected in the cryogenic methanol extract samples at the detection limit of the instrument (3 mg/L). Table 2 also shows the results for hydrogen cyanide (HCN) analysis by IC. Trace amounts of HCN were detected in two samples at slightly above the detection limit (1 $\mu\text{g/L}$).

GAS GRAB SAMPLES

A trace amount of methanol (3 $\mu\text{g/L}$) was detected using direct GC/MS analysis of the two gas grab samples. No other organics were detected above the detection limit of the instrument (2 $\mu\text{g/L}$). Table 3 shows the results of the gas grab samples.

TABLE 2. Ammonia and Hydrogen Cyanide Analyses Results of Cryogenic Samples

<u>Sample ID</u>	<u>Ammonia</u>	<u>HCN</u>
C112/7S2/1B	< 3 mg/L	< 1 µg/L
C112/7S2/2B	< 3 mg/L	< 1 µg/L
C112/7S2/SB	< 3 mg/L	< 1 µg/L
C112/7M1/1B	< 3 mg/L	1.4 µg/L
C112/7M1/2B	< 3 mg/L	< 1 µg/L
C112/7M1/SB	< 3 mg/L	< 1 µg/L
C112/7L2/1B	< 3 mg/L	< 1 µg/L
C112/7L2/2B	< 3 mg/L	< 1 µg/L
C112/7L2/SB	< 3 mg/L	< 1 µg/L
C112/7L3/1B	< 3 mg/L	< 1 µg/L
C112/7L3/2B	< 3 mg/L	< 1 µg/L
C112/7L3/SB	< 3 mg/L	< 1 µg/L
C112/2S1/1B	< 3 mg/L	< 1 µg/L
C112/2S1/2B	< 3 mg/L	< 1 µg/L
C112/2S1/SB	< 3 mg/L	< 1 µg/L
BLIND	< 3 mg/L	< 1 µg/L
C112/2M1/1B	< 3 mg/L	< 1 µg/L
C112/2M1/2B	< 3 mg/L	< 1 µg/L
C112/2M1/SB	< 3 mg/L	< 1 µg/L
C112/2L1/1B	< 3 mg/L	1.6 µg/L
C112/2L1/2B	< 3 mg/L	< 1 µg/L
C112/2L1/SB	< 3 mg/L	< 1 µg/L

HCN = Hydrogen cyanide, ammonia by GC/MS, HCN by IC

TABLE 3. GC/MS Analysis Results of Vapor Grab Samples From Tank 112-C

C11 2/7L3/R Grab	3 µg/L
C11 2/7L1/R Grab	3 µg/L

Triplicate analysis: All other organic components were below the detection limit of the instrument (2 µg/L in the headspace).

VOLATILE/SEMIVOLATILE ANALYSIS

Tables 4 through 25 show the GC/MS results for volatiles and semivolatile analyses. Identification and quantitation from authentic standards are reported in the upper half of the tables. Tentatively identified compounds are reported in the lower half of the tables. Concentration estimates for these components are made assuming the instrument response factor is 1.0 relative to an appropriate standard. Confirmation of tentatively identified compounds was made using a computer search of the NIST/Wiley library and/or mass spectral interpretation. Hexone (MIBK, methylisobutylketone, or 4-methyl-2-pentanone) was detected in the C112/7S2 samples. Figure 1 shows the mass spectra comparison of hexone in the cryogenic sample with that from the mass spectra library. Hexone was identified and quantitated from an authentic hexone standard. The hexone concentration is puzzling since the levels are similar for trap #1, trap #2, and the rinse solution. It is also strange that hexone was not found in the other samples. Possible explanations are field contamination or incorrect sampling technique. Since hexone is a common organic solvent found in Hanford tanks, it seems possible that some was present in the C112/7S2 samples. If the hexone was not from the tank, its source would be difficult to determine since Hexone was not being used in the PNL GC/MS laboratory where the analyses were performed.

TABLE 4. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/7S2/1B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	<u>Dev</u>
Acetone EI	5	2.6	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-
Hexone	16	8.6	133.10	110.80	133.50	125.80	10.61
Undecane EI	7	16.6	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-
* NPH			-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Ethanol	13	2.31	2713.67	2804.93	2699.40	2739.40	57.23
Ethyl Acetate	15	4.74	35.20	36.91	34.19	35.43	1.37
Alkane	17	14.88	-	1.17	1.08	1.12	0.06
Silane	19	20.75	2.38	1.91	1.99	2.10	0.25
Silane	20	22.75	2.92	2.36	2.87	2.72	0.31
Silane	21	25.13	-	-	3.57	3.57	-

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.

- Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 5. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/7S2/2B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	<u>Dev</u>
Acetone EI	5	2.6	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-
Hexone	16	8.6	137.20	147.30	151.70	145.40	6.07
Undecane EI	7	16.6	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-
* NPH			-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Ethanol	13	2.31	3051.15	2918.75	2844.67	2938.20	105.00
Ethyl Acetate	15	4.74	45.45	42.67	42.33	43.49	1.71
Alkane	17	14.88	-	1.34	1.15	1.24	0.13
Silane	18	18.51	4.30	4.31	4.28	4.30	0.02
Silane	19	20.75	10.66	10.93	11.04	10.88	0.20
Silane	20	22.75	13.53	13.19	13.16	13.29	0.20
Silane	21	25.13	11.79	11.03	11.09	11.30	0.42

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.

- Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 6. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/7S2/SB

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Acetone EI	5	2.6	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-
Hexone	16	8.6	145.30	147.70	144.90	145.97	1.24
Undecane EI	7	16.6	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-
* NPH			-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Ethanol	13	2.31	2675.86	2661.15	2576.55	2637.90	54.00
Ethyl Acetate	15	4.74	34.75	33.30	33.76	33.93	0.74
Alkane	17	14.88	1.22	1.17	1.08	1.16	0.07

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 7. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/7M1/1B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Acetone EI	5	2.6	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-
* NPH			-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Ethanol	13	2.31	23.00	23.00	20.00	22.00	2.00
Silane	20	22.75	2.54	1.27	1.87	1.90	0.64

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 8. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/7M1/2B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Acetone EI	5	2.6	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-
* NPH			-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Ethanol	13	2.31	21.00	21.00	20.00	21.00	0
Alkane	17	14.88	1.17	1.13	1.05	1.12	0.06
Silane	18	18.51	1.84	1.78	1.56	1.73	0.15
Silane	19	20.75	4.54	4.34	4.47	4.45	0.11
Silane	20	22.75	4.36	4.74	5.60	4.90	0.63
Silane	21	25.13	2.41	2.21	2.95	2.52	0.38

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.

- Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 9. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/7M2/SB

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Acetone EI	5	2.6	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-
* NPH			-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Ethanol	13	2.31	20.00	21.00	21.0	21.00	1.00
Alkane	17	14.88	1.19	1.14	1.15	1.16	0.03

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.

- Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 10. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/7L2/1B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Acetone EI	5	2.6	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-
* NPH			-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Ethanol	15	2.31	34.00	35.00	34.0	34.00	1.00
Alkane	17	14.88	-	1.07	1.05	1.06	0.02

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 11. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/7L2/2B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Acetone EI	5	2.6	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-
* NPH			-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Ethanol	13	2.31	33.93	35.74	35.13	34.94	0.92
Alkane	17	14.88	1.01	1.03	1.08	1.04	0.04
Silane	19	20.75	1.26	1.37	1.37	1.33	0.06
Silane	20	22.75	2.30	1.79	1.88	1.99	0.27

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 12. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/7L2/SB

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Acetone EI	5	2.6	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-
* NPH			-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Ethanol	13	2.31	34.00	36.00	36.00	35.00	1.00
Alkane	17	14.88	1.11	1.19	1.23	1.18	0.06

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.

- Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 13. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/7L3/1B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Acetone EI	5	2.6	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-
* NPH			-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Ethanol	13	2.31	13.15	12.74	12.45	12.78	0.35
Alkane	17	14.88	1.14	1.11	1.07	1.11	0.04

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.

- Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 14. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/7L3/2B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Acetone EI	5	2.6	-	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-	-
* NPH			-	-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Ethanol	13	2.31	12.97	12.67	12.67	12.77	0.17	
Alkane	17	14.88	1.03	-	1.20	1.11	0.12	
Silane	24	15.87	2.68	2.72	2.54	2.64	0.10	
Silane	18	18.51	3.87	3.69	3.51	3.69	0.18	
Silane	19	20.75	7.33	7.20	7.19	7.24	0.08	
Silane	20	22.75	7.46	8.46	7.03	7.65	0.74	
Silane	21	25.13	4.28	4.22	4.23	4.24	0.03	

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 15. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/7L3/SB

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Acetone EI	5	2.6	-	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-	-
* NPH			-	-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Ethanol	13	2.31	12.30	13.49	13.35	13.04	0.65	
Alkane	17	14.88	1.09	1.05	1.05	1.06	0.02	

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 16. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/2S1/1B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Acetone EI	5	2.6	-	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-	-
* NPH			-	-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Ethanol	13	2.31	17.83	18.41	18.21	18.15		0.30
Dichlorobenzene	23	15.62	6.43	6.45	6.33	6.40		0.07

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 17. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/2S1/2B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Acetone EI	5	2.6	-	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-	-
* NPH			-	-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Ethanol	13	2.31	17.49	17.95	18.64	18.03		0.57
Dichlorobenzene	23	15.62	6.06	6.37	6.18	6.20		0.15
Silane	24	15.87	4.29	4.50	4.46	4.42		0.11
Silane	18	18.51	4.81	5.28	4.88	4.99		0.25
Silane	19	20.75	7.14	7.20	7.16	7.17		0.03
Silane	20	22.75	6.84	6.83	7.77	7.15		0.54
Silane	21	25.13	4.26	4.58	4.47	4.44		0.16

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 18. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/2S1/SB

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Acetone EI	5	2.6	-	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-	-
* NPH			-	-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Ethanol	13	2.31	18.15	17.80	18.67	18.21	0.44	
Dichlorobenzene	23	15.62	5.93	6.00	6.13	6.02	0.10	

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 19. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/BLIND

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Acetone EI	5	2.6	-	-	-	-	-	-
1-Butanol EI	6	5.2	-	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-	-
* NPH			-	-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Ethanol	13	2.31	21.74	16.85	17.80	18.80	2.59	
Dichlorobenzene	23	15.62	6.19	5.97	6.10	6.09	0.11	

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 20. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/2M1/1B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Acetone EI	5	2.6	-	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-	-
* NPH			-	-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Ethanol	13	2.31	27.81	30.10	27.72	28.54	1.35	
Dichloromethane	25	3.16	55.20	51.60	51.64	54.81	3.04	

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 21. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/2M1/2B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Acetone EI	5	2.6	-	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-	-
* NPH			-	-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Ethanol	13	2.31	29.45	28.47	27.52	28.48	0.97	
Dichloromethane	25	3.16	57.72	53.33	55.78	55.61	2.20	

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 22. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/2M1/SB

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Acetone EI	5	2.6	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-
* NPH			-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Ethanol	13	2.31	28.63	28.92	29.10	28.88	0.24
Dichloromethane	25	3.16	56.33	54.73	57.19	56.08	1.25

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 23. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/2L1/1B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Acetone EI	5	2.6	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-
* NPH			-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>	<u>Average Std</u>	
Ethanol	13	2.31	20.98	20.97	20.80	20.92	0.10
Dichlorobenzene	23	15.62	3.51	3.81	3.75	3.69	0.16
Silane	20	22.75	1.27	2.03	1.77	1.69	0.39

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.
 - Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 24. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/2L1/2B

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Acetone EI	5	2.6	-	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-	-
* NPH			-	-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Ethanol	13	2.31	22.23	21.88	20.92	21.68		0.68
Dichlorobenzene	23	15.62	4.30	3.95	4.10	4.11		0.17
Silane	19	20.75	1.85	1.70	1.69	1.75		0.09
Silane	20	22.75	3.75	3.06	3.07	3.29		0.39

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.

- Indicates that the target compound was not found in the sample at the detection limit of the analysis.

TABLE 25. Volatile/Semivolatile Concentration in the Cryogenic Methanol Extract of C112/2L1/SB

<u>Compound</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Acetone EI	5	2.6	-	-	-	-	-	-
1-Butanol EI	6	6.2	-	-	-	-	-	-
Undecane EI	7	16.6	-	-	-	-	-	-
Dodecane	8	18.1	-	-	-	-	-	-
Tridecane EI	9	19.5	-	-	-	-	-	-
Tetradecane EI	10	20.9	-	-	-	-	-	-
Pentadecane EI	11	22.1	-	-	-	-	-	-
* NPH			-	-	-	-	-	-

<u>Tentative Ident</u>	<u>Peak #</u>	<u>Ret. Time</u>	<u>Estimated Concentration (mg/L)</u>				<u>Average Std</u>	<u>Dev</u>
			<u>Rep 1</u>	<u>Rep 2</u>	<u>Rep 3</u>			
Ethanol	13	2.31	21.82	22.34	21.77	21.97		0.31
Dichlorobenzene	23	15.62	4.01	4.01	3.89	3.97		0.07

*NPH concentration is the sum of dodecane, tridecane, tetradecane, and pentadecane.

- Indicates that the target compound was not found in the sample at the detection limit of the analysis.

FIGURE 1. Mass Spectra Comparison of Hexone Detected In Cryogenic Extract Sample With That From The Mass Spectral Library Database

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