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Director

87-e09

WA-22-0030

STATE OF WASHINGTON

DEPARTMENT OF ECOLOGY

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MEMORANDUM
August 20, 1987

TO: Lynn Singleton

FROM: Dick Huntamer, Chemist

SUBJECT: Semivolatile and Resin Acid/Guaiacol Analysis of Grays Harbor Effluent Samples

Two sets of samples collected April 30 and May 4, 1987 from the Grays Harbor area were submitted to the laboratory on May 1 and May 5, 1987 respectively, for priority pollutant (semivolatiles, volatiles, pesticides/PCB's, metals) and resin acid/quaiacol analyses. The laboratory received only two days notification for the first sample set #187451-55 and less than one week notification for the second set #197475-79. Besides failing to notify the laboratory two weeks in advance for routine analysis requests, special analytical techniques and methods were required for the resin acid/quaiacol compounds, and consequently the short notification period compromised the analysis of these compounds.

The primary reason the laboratory was able to analyze for resin acids and quaiacols was that serendipitiously some of the compounds had been ordered earlier for another investigation and had just arrived when Grays Harbor was sampled. Unfortunately, due to the time frame and requirements to extract the samples within seven days from time of collection, the extraction procedures used was not optimized for the resin acids/quaiacols, but was the normal EPA Method 625 priority pollutant extraction using dichloromethane rather than the procedure given in NCASI[1] Technical Bulletin Number 501.

The extraction scheme used for the Grays Harbor samples is given in Figure #1. The major differences in the extraction methods were the use of dichloromethane in place of diethylether and diazomethane for derivitization. The selection of this procedure resulted from the desire to obtain priority pollutant compounds as well as the resin acids/quaiacols from the same sample. Since more notice was given for the second sample collection, the laboratory requested that additional samples (1/2 gallon) be collected and pH adjusted to 10. These samples were extracted by the same procedure as the priority pollutants using dichloromethane in order to provide comparison to the previous sample set and check on the efficiency of preservation at pH 10. The short sample lead-time and lack of some chemical reagents along with the desire to keep the differences in procedures to a minimum between the unpreserved and preserved (pH10) samples prevented the use of diethylether extraction given in NCASI Technical Bulletin 501.

1 NCASI National Council of the Paper Industry for Air and Stream Improvement, Incorporated.

Analyses results are given on the attached tables for the semivolatiles, Table 1 gives the surrogate spike recoveries. All surrogate spikes were within the normal QA/QC recovery limits established by EPA for the Contract Laboratory Program (CLP). Semivolatile analyses results are in the attached lab analysis reports for both sample collection, along with a list of tentatively identified compounds and their estimated concentrations. A second list of tentatively identified compounds is found after the resin acid/quaiacols, Table 2. It gives the tentative compounds found in the methylated fraction, some of which would not normally chromatograph without derivitizing the carboxylic acid group, e.g. resin and fatty acids.

During the verification and calculation of the semivolatiles analyses, it was discovered that the extraction scheme used failed to give complete separation of the neutral organics from the acids. Consequently upon analysis, considerable amounts (up to 50%) of the neutral compounds were found in the methylated acid fraction and had to be accounted for in the analyses. This was done by searching the methylated acid fraction for the neutral compounds then adding them to the underivatized semivolatile fraction. Due to the small amounts of semivolatiles found in the samples this was not a major problem but the surrogate spike compounds in the samples and the matrix spikes had to be corrected for the incomplete neutral/base extraction. The corrected values are given in Table 1 for the surrogate spike recoveries and in the attached Tables for the semivolatile matrix spikes 187455Y and 187455YJ.

In the future, in order to eliminate this problem, it is best to take separate resin acid and semivolatile samples and extract and analyze them separately. This creates more work and results in higher analytical costs but would eliminate the problem with incomplete extractions. The NCASI bulletin 343 also discussed this problem which results from the resin and fatty acids, partitioning into the organic solvent at pH12 and pH2 following EPA 625 protocols. This results in both acid and neutral-base fractions needing analyses. Their recommendation was also to collect and analyze separate samples.

The results for the resin acids/quaiacols analyses are given in Table 2. and results for the laboratory blanks and matrix spikes in Table 2a. 2-Naphthoic acid was used as a surrogate spike since it was the only compound available on short notice which structurally and chemically was related to the resin acids. The recommended surrogate compound was o-methylpodocarpic acid which has to be prepared by the saponification of methyl-o-methyl podocarpate. However, this compound was not available so 2-naphthoic acid was substituted. Recoveries of 2-naphthoic acid were good - 84.9 $\pm$ 26.7% and would be 89.1 $\pm$ 20.9% if the two low recoveries for sample 197476 (pH10) and matrix spike 197477Y are eliminated. Matrix spike 197477Y had low recoveries on almost all of the compounds except retene, which was high. The reason for the erratic

recoveries on the pH10 adjusted matrix spikes compared to the regular matrix spikes 157455Y and YJ cannot be explained since both groups would have been made acidic prior to extraction. Comparison between the pH10 preserved samples and the normal pH samples for 197475 and 197476 is somewhat ambiguous. Most of the values are in close agreement to each other but 4,5,6 trichloroguaiacol spans a large difference between the two, 91 versus 16 ug/L, because of higher detection limits on 197476 and a lack of compounds in 197477 it can not be determined if the large discrepancies on 197475 is an anomoly or a trend.

The spike recoveries also point out a problem in the analysis of levopimaric acid and neoabictic acid. In both spikes, but particularly in 187455Y and YJ, these compounds are conspicuous by their absence. Apparently they degrade rapidly during or after extraction and may not be measurable without some sample extract and preservation techniques.

As can be seen from the proceeding discussion, the resin acid/guaiacol analyses was not performed under optimum conditions given the time frame the laboratory had to operate under. Despite this, some usable results were obtained but should be qualified since optimum extraction and preservation techniques were not utilized. Future work in this area should incorporate separate resin acid quaiacol sample and analyses and follow the NCASII 501 Method as close as possible. Semivolatiles should be extracted and analyzed separately.

References:

NCASI Technical Bulletin Number 343, "Experience with the Analyses of EPA's Organic Priority Pollutants and Compounds Charateristic of Pulp Mill Effluents." Stream Improvement - Technical Bulletin Number 343, March 23, 1981.

NCASI Technical Bulletin Number 501, "Procedures for the Analyses of Resin and Fatty Acids in Pulp Mill Effluents." Technical Bulletin Number 501, August 20, 1986.

DH/cm
Attachments

Figure 1. Extraction Scheme for Greys Harbor Semivolatiles, Resin Acids and Guaiacols

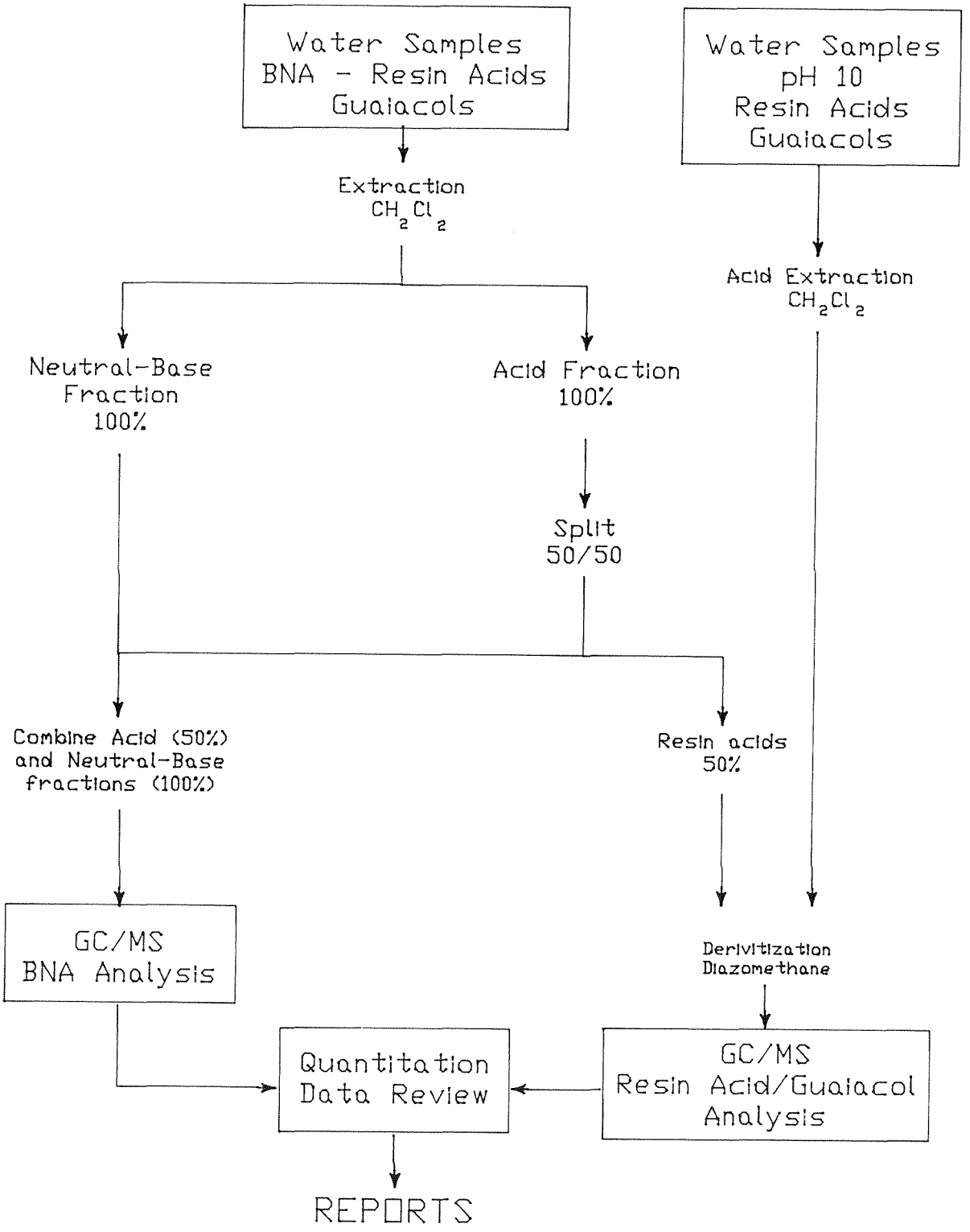


Table 1. Greys Harbor Semivolatile Surrogate Spike Recoveries.

Compound	Sample Identification 187451 %	187452 %	187453 %	187454 %	187455 %	187455Y %	187455YJ %	BN7127W %
2 fluorophenol	91	50	67	62	72	90	94	82
Phenol-d5	97	44	65	34	64	87	85	70
Nitrobenzene-d5	79	52	57	79	69	72	80	71
2-fluorobiphenyl	84	58	80	81	51	76	76	72
Pyrene-d10	103	66	86	81	99	84	89	85
Terphenyl-d14	65	68	110	79	88	91	103	109

Compound	Sample Identification 197475 %	197476 %	197477 %	197478 %	197479 %	BN7131W %	QA/QC Limits %
2 fluorophenol	58	53	55	<----Sample Lost---->	46	84	23-121
Phenol-d5	51	23	34		23	68	15-103
Nitrobenzene-d5	70	45	73		39	64	41-120
2 fluorobiphenyl	66	55	68		66	67	44-119
Pyrene-d10	71	63	90		88	80	--
Terphenyl-d14	43	70	104		116	103	38-128

DH/cm
8-10-87

*** Lab Analysis Report ***

==> Transaction #: 08111311 Laboratory: (WE) DOE, Manchester

Work Group: (68) B/N/Acid Scan

Instrument: (GCMS-E2) EPA2 GC/MS INCOS-5100 Capillary Colu

Method: (EP2-625) GC/MS Ext Scan

Chemist: (DDH) Huntamer, Dick DOE Hours Worked:

Project: DOE-541A GRAYS HARBOR EFFLUENTS, (ITT, WEYCO) Prg Ele#: 442

Prj Off: Unspecifed Analysis Due: 870430 Revised Due: 870501

*** Sample Records in Transaction ***

Parameter Form File: BNAFM1 Title: Organics - B/N/A Scan (FormsMaster1)

Seq#	Sample #	Date/Time	Description	Alternate Keys		
01	87187451	870430	WEYCO			
02	87187452	870430	ITT			
03	87187453	870430	ABERDEEN WTP			
04	87187454	870430	CONTROL			
05	87187455	870430	DUPLICATE			

Record Type: TRNIN1 Date Verified: 87/08/13 By: Covey, Pam DOE
Transaction Status: Verified Transaction...Ready to release.
*** Verified and Transferred to VERTRANS ***
Processed: 13-AUG-87 09:01:42 Status: V Batch: A (In CUR DB)

*** Lab Analysis Report ***

Transaction #: 08111311 (68) B/N/Acid Scan
Proj Code : DOE-541A GRAYS HARBOR EFFLUENTS, (ITT, WEYCO) PE # : 442

Sample Id:	87187451	87187452	87187453	87187454	87187455
Matrix:	Water-Tot	Water-Tot	Water-Tot	Water-Tot	Water-Tot
Units:	ug/l	ug/l	ug/l	ug/l	ug/l
% Slds:					
QA Code:					
Date Extract:	870507	870507	870507	870507	870507
Date Analyzd:	870522	870522	870522	870527	870527
1 Phenol	0.9U	3U	0.9U	2U	2U
2 bis(2-Chloroethyl)Ether	0.9U	0.7U	0.9U	2U	2U
3 2-Chlorophenol	0.9U	3U	0.9U	2U	2U
4 1,3-Dichlorobenzene	0.9U	3U	0.9U	2U	2U
5 1,4-Dichlorobenzene	0.9U	3U	0.2J	2U	0.2J
6 Benzyl Alcohol	0.9U	3U	0.9U	2U	2U
7 1,2-Dichlorobenzene	0.9U	3U	0.9U	2U	2U
8 2-Methylphenol	0.9U	3U	0.9U	2U	2U
9 bis(2-Chloroisopropyl)Ethe	0.9U	3U	0.9U	2U	2U
10 4-Methylphenol	47	0.5J	0.9U	2U	2U
11 N-Nitroso-di-n-Propylamine	0.9U	3U	0.9U	2U	2U
12 Hexachloroethane	0.9U	3U	0.9U	2U	2U
13 Nitrobenzene	0.9U	3U	0.9U	2U	2U
14 Isophorone	0.9U	3U	0.9U	2U	2U
15 2-Nitrophenol	0.9U	3U	0.9U	2U	2U
16 2,4-Dimethylphenol	0.9U	3U	0.9U	2U	2U
17 Benzoic acid	23U	17U	4U	9U	11U
18 bis(2-Chloroethoxy)Methane	0.9U	3U	0.9U	2U	2U
19 2,4-Dichlorophenol	2	3U	0.9U	2U	2U
20 1,2,4-Trichlorobenzene	0.9U	3U	0.9U	2U	2U
21 Napthalene	0.2J	3U	0.9U	2U	2U
22 4-Chloroaniline	0.9U	3U	0.9U	2U	2U
23 Hexachlorobutadiene	0.9U	3U	0.9U	2U	2U
24 4-Chloro-3-Methylphenol	0.9U	3U	0.9U	2U	2U
25 2-Methylnaphthalene	0.9U	3U	0.9U	2U	2U
26 Hexachlorocyclopentadiene	0.9U	3U	0.9U	2U	2U
27 2,4,6-Trichlorophenol	0.9U	0.9J	0.9U	2U	2U
28 2,4,5-Trichlorophenol	11	17U	4U	9U	11U
29 2-Chloronaphthalene	0.9U	3U	0.9U	2U	2U
30 2-Nitroaniline	4U	17U	4U	9U	11U
31 Dimethylphthalate	0.9U	3U	0.9U	2U	2U
32 Acenaphthylene	0.9U	3U	0.9U	2U	2U
33 3-Nitroaniline	4U	17U	4U	9U	11U
34 Acenaphthene	0.9U	3U	0.9U	2U	2U
35 2,4-Dinitrophenol	4U	17U	4U	9U	11U
36 4-Nitrophenol	4U	17U	4U	9U	11U
37 Dibenzofuran	0.9U	3U	0.9U	2U	2U
38 2,4-Dinitrotoluene	0.9U	3U	0.9U	2U	2U
39 2,6-Dinitrotoluene	0.9U	3U	0.9U	2U	2U
40 Diethylphthalate	0.9U	3U	0.9U	2U	2U
41 4-Chlorophenyl-phenylether	0.9U	3U	0.9U	2U	2U
42 Fluorene	0.9U	3U	0.9U	2U	2U
43 4-Nitroaniline	4U	17U	4U	9U	11U
44 4,6-Dinitro-2-methylphenol	4U	17U	4U	9U	11U
45 N-Nitrosodiphenylamine	0.9U	3U	0.9U	2U	2U
46 4-Bromophenyl-phenylether	0.9U	3U	0.9U	2U	2U
47 Hexachlorobenzene	0.9U	3U	0.9U	2U	2U
48 Pentachlorophenol	4U	17U	4U	9U	11U
49 Phenanthrene	0.9U	3U	0.9U	2U	2U
50 Anthracene	0.9U	3U	0.9U	2U	2U

J = estimated value
B = found in blank

*** Lab Analysis Report ***

Transaction #: 08111311 (68) B/N/Acid Scan
Proj Code : DOE-541A GRAYS HARBOR EFFLUENTS, (ITT, WEYCO) PE # : 442

Sample Id:	87187451	87187452	87187453	87187454	87187455
Matrix:	Water-Tot	Water-Tot	Water-Tot	Water-Tot	Water-Tot
Units:	ug/l	ug/l	ug/l	ug/l	ug/l
% Slds:					
QA Code:					
Date Extract:	870507	870507	870507	870507	870507
Date Analyzd:	870522	870522	870522	870527	870527
51 Di-n-Butylphthalate	0.9U	1J	0.3J	2U	0.2J
52 Fluoranthene	0.9U	3U	0.9U	2U	2U
53 Pyrene	0.9U	3U	0.9U	2U	2U
54 Butylbenzylphthalate	0.9U	3U	0.9U	2U	2U
55 3,3'-Dichlorobenzidine	2U	7U	2U	4U	4U
56 Benzo(a)anthracene	0.9U	3U	0.9U	2U	2U
57 bis(2-Ethylhexyl)Phthalate	0.9BU	3BU	0.5BJ	2BU	0.5BJ
58 Chrysene	0.9U	3U	0.9U	2U	2U
59 Di-n-Octyl Phthalate	0.9U	3U	0.9U	2U	2U
60 Benzo(b)fluoranthene	0.9U	3U	0.9U	2U	2U
61 Benzo(k)fluoranthene	0.9U	3U	0.9U	2U	2U
62 Benzo(a)pyrene	0.9U	3U	0.9U	2U	2U
63 Indeno(1,2,3-cd)pyrene	0.9U	3U	0.9U	2U	2U
64 Dibenz(a,h)anthracene	0.9U	3U	0.9U	2U	2U
65 Benzo(ghi)perylene	0.9U	3U	0.9U	2U	2U

*** Lab Analysis Report ***

==> Transaction #: 08111323 Laboratory: (WE) DOE, Manchester

Work Group: (68) B/N/Acid Scan

Instrument: (GCMS-E2) EPA2 GC/MS INCOS-5100 Capillary Colu

Method: (EP2-625) GC/MS Ext Scan

Chemist: (DDH) Huntamer, Dick DOE Hours Worked:

Project: DOE-541B GRAYS HARBOR EFFLUENTS Prg Ele#: 442

Prj Off: Unspecifed Analysis Due: 870505 Revised Due:

*** Sample Records in Transaction ***

Parameter Form File: BNAFM1 Title: Organics - B/N/A Scan (FormsMaster1)

Seq#	Sample #	Date/Time	Description	Alternate Keys		
01	87197475	870504	WEYCO			
02	87197476	870504	ITT			
03	87197477	870504	ABERDEEN			
04	87197479	870504	BLANK			

Record Type: TRNIN1 Date Verified: 87/08/13 By: Covey, Pam DOE
Transaction Status: Verified Transaction...Ready to release.
*** Verified and Transferred to VERTRANS ***
Processed: 13-AUG-87 08:58:58 Status: V Batch: A (In CUR DB)

*** Lab Analysis Report ***

Transaction #: 08111323 (68) B/N/Acid Scan
Proj Code : DOE-541B GRAYS HARBOR EFFLUENTS PE # : 442

Sample Id:	87197475	87197476	87197477	87197479
Matrix:	Water-Tot	Water-Tot	Water-Tot	Water-Tot
Units:	ug/l	ug/l	ug/l	ug/l
% Slds:				
QA Code:				
Date Extract:	870507	870507	870507	870507
Date Analyzd:	870608	870608	870527	870527
1 Phenol	0.7J	9U	2U	2U
2 bis(2-Chloroethyl)Ether	4U	0.9J	2U	2U
3 2-Chlorophenol	4U	9U	2U	2U
4 1,3-Dichlorobenzene	4U	9U	2U	2U
5 1,4-Dichlorobenzene	4U	9U	0.3J	2U
6 Benzyl Alcohol	4U	9U	2U	2U
7 1,2-Dichlorobenzene	4U	9U	0.3J	2U
8 2-Methylphenol	4U	9U	2U	2U
9 bis(2-Chloroisopropyl)Ethe	4U	9U	2U	2U
10 4-Methylphenol	35	9U	2U	2U
11 N-Nitroso-di-n-Propylamine	4U	9U	2U	2U
12 Hexachloroethane	4U	9U	2U	2U
13 Nitrobenzene	4U	9U	2U	2U
14 Isophorone	4U	9U	2U	2U
15 2-Nitrophenol	4U	9U	2U	2U
16 2,4-Dimethylphenol	4U	9U	2U	2U
17 Benzoic acid	3J	43U	11U	8U
18 bis(2-Chloroethoxy)Methane	4U	9U	2U	2U
19 2,4-Dichlorophenol	3J	9U	2U	2U
20 1,2,4-Trichlorobenzene	4U	9U	2U	2U
21 Napthalene	0.3J	9U	2U	2U
22 4-Chloroaniline	4U	9U	2U	2U
23 Hexachlorobutadiene	4U	9U	2U	2U
24 4-Chloro-3-Methylphenol	4U	9U	2U	2U
25 2-Methylnaphthalene	4U	9U	2U	2U
26 Hexachlorocyclopentadiene	4U	9U	2U	2U
27 2,4,6-Trichlorophenol	11	9U	2U	2U
28 2,4,5-Trichlorophenol	18U	43U	11U	8U
29 2-Chloronaphthalene	4U	9U	2U	2U
30 2-Nitroaniline	18U	43U	11U	8U
31 Dimethylphthalate	4U	9U	2U	2U
32 Acenaphthylene	4U	9U	2U	2U
33 3-Nitroaniline	18U	43U	11U	8U
34 Acenaphthene	4U	9U	2U	2U
35 2,4-Dinitrophenol	18U	43U	11U	8U
36 4-Nitrophenol	18U	43U	11U	8U
37 Dibenzofuran	4U	9U	2U	2U
38 2,4-Dinitrotoluene	4U	9U	2U	2U
39 2,6-Dinitrotoluene	4U	9U	2U	2U
0 Diethylphthalate	4U	9U	2U	2U
1 4-Chlorophenyl-phenylether	4U	9U	2U	2U
2 Fluorene	4U	9U	2U	2U
3 4-Nitroaniline	18U	43U	11U	8U
4 4,6-Dinitro-2-methylphenol	18U	43U	11U	8U
5 N-Nitrosodiphenylamine	4U	9U	2U	2U
6 4-Bromophenyl-phenylether	4U	9U	2U	2U
7 Hexachlorobenzene	4U	9U	2U	2U
8 Pentachlorophenol	18U	43U	11U	8U
9 Phenanthrene	4U	9U	2U	2U
0 Anthracene	4U	9U	2U	2U

*** Lab Analysis Report ***

Transaction #: 08111323 (68) B/N/Acid Scan
Proj Code : DOE-541B GRAYS HARBOR EFFLUENTS PE # : 442

Sample Id:	87197475	87197476	87197477	87197479
Matrix:	Water-Tot	Water-Tot	Water-Tot	Water-Tot
Units:	ug/l	ug/l	ug/l	ug/l
% Slds:				
QA Code:				
Date Extract:	870507	870507	870507	870507
Date Analyzd:	870608	870608	870527	870527
51 Di-n-Butylphthalate	4U	9U	0.3J	0.2J
52 Fluoranthene	4U	9U	2U	2U
53 Pyrene	4U	9U	2U	2U
54 Butylbenzylphthalate	4U	9U	2U	2U
55 3,3'-Dichlorobenzidine	7U	17U	4U	3U
56 Benzo(a)anthracene	4U	9U	2U	2U
57 bis(2-Ethylhexyl)Phthalate	4BU	9BU	0.6BJ	0.2BJ
58 Chrysene	4U	9U	2U	2U
59 Di-n-Octyl Phthalate	4U	9U	2U	0.7J
60 Benzo(b)fluoranthene	4U	9U	2U	2U
61 Benzo(k)fluoranthene	4U	9U	2U	2U
62 Benzo(a)pyrene	4U	9U	2U	2U
63 Indeno(1,2,3-cd)pyrene	4U	9U	2U	2U
64 Dibenz(a,h)anthracene	4U	9U	2U	2U
65 Benzo(ghi)perylene	4U	9U	2U	2U

*** Lab Analysis Report ***

==> Transaction #: 08111407 Laboratory: (WE) DOE, Manchester

Work Group: (68) B/N/Acid Scan

Instrument: (GCMS-E2) EPA2 GC/MS INCOS-5100 Capillary Colu

Method: (EP2-625) GC/MS Ext Scan

Chemist: (DDH) Huntamer, Dick DOE Hours Worked:

Project: DOE-541B GRAYS HARBOR EFFLUENTS Prg Ele#: 442

Prj Off: Unspecifed Analysis Due: 870505 Revised Due:

*** Sample Records in Transaction ***

Parameter Form File: BNAFM1 Title: Organics - B/N/A Scan (FormsMaster1)

Seq#	Sample #	Date/Time	Description	Alternate Keys		
01	87197478	870504	CONTROL			

Record Type: TRNIN1 Date Verified: 87/08/13 By: Covey, Pam DOE
Transaction Status: Verified Transaction...Ready to release.
*** Verified and Transferred to VERTRANS ***
Processed: 13-AUG-87 08:58:58 Status: V Batch: A (In CUR DB)

*** Lab Analysis Report ***

Transaction #: 08111407 (68) B/N/Acid Scan
Proj Code : DOE-541B GRAYS HARBOR EFFLUENTS PE # : 442

Sample Id:	87197478
Matrix:	Water-Tot
Units:	ug/l
% Slds:	
QA Code:	
Date Extract:	870507
Date Analyzd:	870608
1 Phenol	LAC
2 bis(2-Chloroethyl)Ether	LAC
3 2-Chlorophenol	LAC
4 1,3-Dichlorobenzene	LAC
5 1,4-Dichlorobenzene	LAC
6 Benzyl Alcohol	LAC
7 1,2-Dichlorobenzene	LAC
8 2-Methylphenol	LAC
9 bis(2-Chloroisopropyl)Ethe	LAC
10 4-Methylphenol	LAC
11 N-Nitroso-di-n-Propylamine	LAC
12 Hexachloroethane	LAC
13 Nitrobenzene	LAC
14 Isophorone	LAC
15 2-Nitrophenol	LAC
16 2,4-Dimethylphenol	LAC
17 Benzoic acid	LAC
18 bis(2-Chloroethoxy)Methane	LAC
19 2,4-Dichlorophenol	LAC
20 1,2,4-Trichlorobenzene	LAC
21 Napthalene	LAC
22 4-Chloroaniline	LAC
23 Hexachlorobutadiene	LAC
24 4-Chloro-3-Methylphenol	LAC
25 2-Methylnaphthalene	LAC
26 Hexachlorocyclopentadiene	LAC
27 2,4,6-Trichlorophenol	LAC
28 2,4,5-Trichlorophenol	LAC
29 2-Chloronaphthalene	LAC
30 2-Nitroaniline	LAC
31 Dimethylphthalate	LAC
32 Acenaphthylene	LAC
33 3-Nitroaniline	LAC
34 Acenaphthene	LAC
35 2,4-Dinitrophenol	LAC
36 4-Nitrophenol	LAC
37 Dibenzofuran	LAC
38 2,4-Dinitrotoluene	LAC
39 2,6-Dinitrotoluene	LAC
40 Diethylphthalate	LAC
41 4-Chlorophenyl-phenylether	LAC
42 Fluorene	LAC
43 4-Nitroaniline	LAC
44 4,6-Dinitro-2-methylphenol	LAC
45 N-Nitrosodiphenylamine	LAC
46 4-Bromophenyl-phenylether	LAC
47 Hexachlorobenzene	LAC
48 Pentachlorophenol	LAC
49 Phenanthrene	LAC
50 Anthracene	LAC

*** Lab Analysis Report ***

Transaction #: 08111407 (68) B/N/Acid Scan
Proj Code : DOE-541B GRAYS HARBOR EFFLUENTS PE # : 442

Sample Id:	87197478
Matrix:	Water-Tot
Units:	ug/l
% Slds:	
QA Code:	
Date Extract:	870507
Date Analyzd:	870608
51 Di-n-Butylphthalate	LAC
52 Fluoranthene	LAC
53 Pyrene	LAC
54 Butylbenzylphthalate	LAC
55 3,3'-Dichlorobenzidine	LAC
56 Benzo(a)anthracene	LAC
57 bis(2-Ethylhexyl)Phthalate	LAC
58 Chrysene	LAC
59 Di-n-Octyl Phthalate	LAC
60 Benzo(b)fluoranthene	LAC
61 Benzo(k)fluoranthene	LAC
62 Benzo(a)pyrene	LAC
63 Indeno(1,2,3-cd)pyrene	LAC
64 Dibenz(a,h)anthracene	LAC
65 Benzo(ghi)perylene	LAC

Laboratory Name: MANCHESTER LAB
Case No: DOE-451A

Sample Number
187455YJ

Matrix Spike

ORGANICS ANALYSIS DATA SHEET
(Page 2)

SEMIVOLATILE COMPOUNDS

Concentration: LOW
Date Extracted/Prepared: 05/07/87
Date Analyzed: 05/27/87
Conc/Dil Factor: 1.
Percent Moisture: (Decanted) _____

GPC Cleanup ___ Yes _X_ No
Separatory Funnel Extraction _X_ Yes
Continuous Liquid-Liquid Extraction ___ Yes

CAS Number		%	Total
108-95-2	Phenol	81	81
111-44-4	bis(2-Chloroethyl)Ether	(43)	86
95-57-8	2-Chlorophenol	93	93
541-73-1	1,3-Dichlorobenzene	(30)	63
106-46-7	1,4-Dichlorobenzene	(29)	61
100-51-6	Benzyl Alcohol	10	U
95-50-1	1,2-Dichlorobenzene	(28)	59
95-48-7	2-Methylphenol	98	98
39638-32-9	bis(2-Chloroisopropyl)Ether	(42)	82
106-44-5	4-Methylphenol	95	95
621-64-7	N-Nitroso-Di-n-Propylamine	(45)	93
67-72-1	Hexachloroethane	(23)	44
98-95-3	Nitrobenzene	(45)	91
78-59-1	Isophorone	(49)	91
88-75-5	2-Nitrophenol	92	92
105-67-9	2,4-Dimethylphenol	80	80
65-85-0	Benzoic Acid	110	110
111-91-1	bis(2-Chloroethoxy)Methane	(43)	85
120-83-2	2,4-Dichlorophenol	92	92
120-82-1	1,2,4-Trichlorobenzene	(28)	54
91-20-3	Naphthalene	(32)	83
106-47-8	4-Chloroaniline	190	190
87-68-3	Hexachlorobutadiene	(25)	58
59-50-7	4-Chloro-3-Methylphenol	100	100
91-57-6	2-Methylnaphthalene	(32)	63
77-47-4	Hexachlorocyclopentadiene	(15)	24
88-06-2	2,4,6-Trichlorophenol	110	110
95-95-4	2,4,5-Trichlorophenol	120	120
91-58-7	2-Chloronaphthalene	(38)	69
88-74-4	2-Nitroaniline	(50)	78
131-11-3	Dimethyl Phthalate	(47)	91
208-96-8	Acenaphthylene	(42)	80
99-09-2	3-Nitroaniline	(42)	
83-32-9	Acenaphthene	(38)	75
51-28-5	2,4-Dinitrophenol	100	100
100-02-7	4-Nitrophenol	110	110

CAS Number		%	Total
132-64-9	Dibenzofuran	(41)	81
121-14-2	2,4-Dinitrotoluene	(46)	71
606-20-2	2,6-Dinitrotoluene	(47)	80
84-66-2	Diethylphthalate	(46)	87
7005-72-3	4-Chlorophenyl-phenylether	(42)	80
86-73-7	Fluorene	(42)	82
100-10-6	4-Nitroaniline	50	U
534-52-1	4,6-Dinitro-2-Methylphenol	99	
86-30-6	N-Nitrosodiphenylamine (1)	10	U
101-55-3	4-Bromophenyl-phenylether	(47)	89
118-74-1	Hexachlorobenzene	(48)	93
87-86-5	Pentachlorophenol	250	high
85-01-8	Phenanthrene	(46)	88
120-12-7	Anthracene	(45)	10
84-74-2	Di-n-Butylphthalate	(48)	9[illegible]
206-44-0	Fluoranthene	(49)	87
129-00-0	Pyrene	(35)	79
85-68-7	Butylbenzylphthalate	57	107
91-94-1	3,3'-Dichlorobenzidine	20	U
56-55-3	Benzo(a)Anthracene	(53)	94
117-81-7	bis(2-Ethylhexyl)Phthalate	(50)	86
218-01-9	Chrysene	(51)	94
117-84-0	Di-n-Octyl Phthalate	(44)	84
205-99-2	Benzo(b)Fluoranthene	48	
207-08-9	Benzo(k)Fluoranthene	48	
50-32-8	Benzo(a)Pyrene	(48)	59
193-39-5	Indeno(1,2,3-cd)Pyrene	(48)	54
53-70-3	Dibenz(a,h)Anthracene	(47)	79
191-24-2	Benzo(g,h,i)Perylene	(48)	64
	Nitrobenzene-d5		
	2-Fluorobiphenyl		
	Terphenyl-d14		See Table 1.
	Pyrene-d10		
	Phenol-d5		
	2-Fluorophenyl		

(1) - Cannot be separated from diphenylamine

Form I

Total % recovered is given on right hand side
( ) indicates partial recovery - neutral fraction only

Laboratory Name: MANCHESTER LAB
Case No: DOE-451A

Sample Number
187455Y

ORGANICS ANALYSIS DATA SHEET
(Page 2)

Matrix Spike Duplicate

SEMIVOLATILE COMPOUNDS

Concentration: LOW
Date Extracted/Prepared: 05/07/87
Date Analyzed: 05/27/87
Conc/Dil Factor: 1.
Percent Moisture: (Decanted) _____

GPC Cleanup ___ Yes _X_ No
Separatory Funnel Extraction _X_ Yes
Continuous Liquid-Liquid Extraction ___ Yes

CAS Number		%	Total	CAS Number		%	Total
108-95-2	Phenol	87	87	132-64-9	Dibenzofuran	(41)	81
111-44-4	bis(2-Chloroethyl)Ether	(40)	88	121-14-2	2,4-Dinitrotoluene	(46)	50
95-57-8	2-Chlorophenol	85	85	606-20-2	2,6-Dinitrotoluene	(47)	65
541-73-1	1,3-Dichlorobenzene	(25)	51	84-66-2	Diethylphthalate	(46)	91
106-46-7	1,4-Dichlorobenzene	(24)	49	7005-72-3	4-Chlorophenyl-phenylether	(43)	82
100-51-6	Benzyl Alcohol	10	U	86-73-7	Fluorene	(44)	84
95-50-1	1,2-Dichlorobenzene	(26)	52	100-10-6	4-Nitroaniline	50	U
95-48-7	2-Methylphenol	87	87	534-52-1	4,6-Dinitro-2-Methylphenol	110	110
39638-32-9	bis(2-Chloroisopropyl)Ether	(38)	79	86-30-6	N-Nitrosodiphenylamine (1)	10	U
106-44-5	4-Methylphenol	87	87	101-55-3	4-Bromophenyl-phenylether	(46)	85
621-64-7	N-Nitroso-Di-n-Propylamine	(42)	82	118-74-1	Hexachlorobenzene	(52)	100
67-72-1	Hexachloroethane	(21)	40	87-86-5	Pentachlorophenol	210	210 hi
98-95-3	Nitrobenzene	(46)	78	85-01-8	Phenanthrene	(49)	88
78-59-1	Isophorone	(47)	87	120-12-7	Anthracene	(41)	75
88-75-5	2-Nitrophenol	92	92	84-74-2	Di-n-Butylphthalate	(52)	96
105-67-9	2,4-Dimethylphenol	83	83	206-44-0	Fluoranthene	(53)	87
65-85-0	Benzoic Acid	110	110	129-00-0	Pyrene	(50)	92
111-91-1	bis(2-Chloroethoxy)Methane	(45)	88	85-68-7	Butylbenzylphthalate	(54)	103
120-83-2	2,4-Dichlorophenol	94	94	91-94-1	3,3'-Dichlorobenzidine	20	U
120-82-1	1,2,4-Trichlorobenzene	(28)	53	56-55-3	Benzo(a)Anthracene	(51)	89
91-20-3	Naphthalene	(32)	63	117-81-7	bis(2-Ethylhexyl)Phthalate	53	B
106-47-8	4-Chloroaniline	200	200	218-01-9	Chrysene	(48)	90
87-68-3	Hexachlorobutadiene	(23)	44	117-84-0	Di-n-Octyl Phthalate	(46)	84
59-50-7	4-Chloro-3-Methylphenol	100	100 high	205-99-2	Benzo(b)Fluoranthene	48	48
91-57-6	2-Methylnaphthalene	(32)	65	207-08-9	Benzo(k)Fluoranthene	48	48
77-47-4	Hexachlorocyclopentadiene	(13)	15	50-32-8	Benzo(a)Pyrene	(45)	53
88-06-2	2,4,6-Trichlorophenol	100	100	193-39-5	Indeno(1,2,3-cd)Pyrene	46	46
95-95-4	2,4,5-Trichlorophenol	120	120	53-70-3	Dibenz(a,h)Anthracene	(46)	73
91-58-7	2-Chloronaphthalene	(37)	67	191-24-2	Benzo(g,h,i)Perylene	(47)	60
88-74-4	2-Nitroaniline	50	U		Nitrobenzene-d5		
131-11-3	Dimethyl Phthalate	(47)	91		2-Fluorobiphenyl		
208-96-8	Acenaphthylene	(44)	77		Terphenyl-d14		See Table 1.
99-09-2	3-Nitroaniline	(42)	68		Pyrene-d10		
83-32-9	Acenaphthene	(37)	72		Phenol-d5		
51-28-5	2,4-Dinitrophenol	100	100		2-Fluorophenyl		
100-02-7	4-Nitrophenol	120	120 high				

(1) - Cannot be separated from diphenylamine

Form I

Total % recovered is given on right hand side
( ) indicates partial recovery neutral fraction only.

TENTATIVELY IDENTIFIED COMPOUNDS

BNA

PROJECT: Grey's Harbor COMPILED BY: [illegible] DATE: 8/5/87

LABORATORY: Manchester REVIEWED BY: _____ DATE: _____

CAS #	Compound Name	Sample No.	187451	187452	187453	187454	187455	BN7127W	BN7131W		
		Hit Ratio									
		Conc units	µg/L								
1768-31-6	2-Propanone 1,1,1,3,3-pentachloro-		69.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.		
97-61-0	2-methyl hexanoic or pentanoic acid or related compd.		7.4	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.		
4536-23-6	2 methyl hexanoic acid or related compd.		12.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.		
632-21-3	1,1,3,3 Tetrachloro-2-propanone or related compd		12.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.		
1453-06-1	Benzenebutanoic acid, 2,5 dimethyl		66.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.		
57-10-3	Hexadecanoic Acid		230.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.		
110-12-3	5 methyl-2-hexanone		140.	120	110	120	120	320 ~~N.D.~~ gne	33 ~~N.D.~~ gne		
78-51-3	Ethanol, 2 butoxy, phosphate (3:1)		N.D.	N.D.	14.	N.D.	7.5	N.D.	N.D.		
119-65-3	Isoquinoline		N.D.	N.D.	15.	N.D.	13.	ND ~~320~~ gne	ND ~~33.~~ gne		
3056-60-8	3-ethyl, phenol, acetate		present	ND	ND	ND	ND	ND	ND		
11											
12											
13											
14											
15											

From Roy's Mac

Aug 1986

TENTATIVELY IDENTIFIED COMPOUNDS

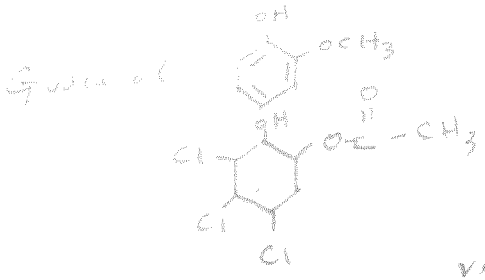
BNA

PROJECT: Grey's Harbor COMPILED BY: [illegible] DATE: 8/5/87

LABORATORY: Manchester REVIEWED BY: _____ DATE: _____

		Sample No.	1974 75	1974 76	1974 77	1974 78	1979 79	BN 7127	BN 7131		
CAS #	Compound Name	Hit Ratio									
		Conc units	µg/L								
1708-31-6	2-Propanone, 1,1,1,3,3-Pentachloro-		44.	N.D.	N.D.		N.D.	N.D.	N.D.		
620-02-0	2 Furancarboxaldehyde, 5-methyl-		30.	N.D.	N.D	S	N.D.	N.D.	N.D.		
108-94-1	Cyclohexanone		2.7	N.D.	N.D.	A M	N.D.	N.D.	N.D.		
98-01-1	2-Furancarboxaldehyde		890.	N.D.	N.D.	P L	N.D	N.D	N.D.		
1453-06-1	Benzene butanoic acid, 2,5 dimethyl-		82.	N.D.	N.D.	E	N.D.	N.D.	N.D.		
632-21-3	1,1,3,3 Tetrachloro-2-propanone		8.0	N.D.	N.D.	L	N.D.	N.D.	N.D.		
110-12-3	5 methyl-2-hexanone		14.	11.	16.	O S T	9.8	320.	33.		
8						I					
9											
10											
11											
12											
13											
14	Hydrocarbon Acids		Yes	—	—	—	—				
15	Hydrocarbons		Yes	—	—	—	—				
			✓	✓	✓	✓	✓	✓	✓		

From Roy's Mac
Aug 1986



veratrole = 1,2-Dimethoxy benzene
guaiacol = 2, methoxy phenol
← Veratrole is methyl derivative of guaiacol from use of diazomethane

Table 2. Results of Grays Harbor Resin Acid/Guaiacol Water Sample Analyses

CAS#	Compound	Units	SAMPLE ID 1874 - 51	52	53	54	55	SAMPLE ID 1974 - 75	75**	76	76**	77	77**	78	79
90-05-1	Guaiacol	ug/L	5u	5u	15u	5u	5u	4.0UJ	3.6UJ	5u	15u	5u	5u	SAMPLE LOST	5u
	4,5,6 Trichloro-guaiacol*	ug/L	46.	3.0UJ	15u	5u	5u	91.	16.	6.1	15u	5u	5u		5u
944-31-6	Tetrachloroguaiacol*	ug/L	15.	5.6	15u	5u	5u	19.	6.7	7.4	15u	5u	5u		5u
483-65-8	Retene	ug/L	5u	5u	15u	5u	5u	10u	3u	5u	15u	5u	5u		5u
1686-62-0	Isopimaric Acid*	ug/L	5u	5u	15u	5u	5u	10u	3u	5u	15u	5u	5u		5u
3513-69-7	Levopimaric Acid*	ug/L	10u	10u	30u	10u	10u	20u	3u	5u	30u	10u	10u		10u
1235-74-1	Dehydroabietic Acid*	ug/L	15.	1.90J	15u	5u	5u	18.	23	1.8UJ	15u	5u	5u		5u
127-25-3	Abietic Acid*	ug/L	5u	0.53UJ	15u	5u	5u	10u	3u	1.1UJ	15u	5u	5u		5u
3310-97-2	Neoabietic Acid*	ug/L	10u	10u	30u	10u	10u	20u	6u	10u	30u	10u	10u		10u
	Dichlorodehydro-abitic Acid*	ug/L	13.	8.2	15u	5u	5u	57	47	5.0	15u	5u	5u		5u
2459-25-8	2-Napthoic Acid* (Surrogate Spike)	%	97	88	93	86	89	86	87	86	17	109	94		88
	Sample pH	pH	2.9	6.3	7.2	6.7	7.2	2.8	10	6.4	10	7.4	10		--

* Measured as the Methylester
** Sample Preserved at pH 10 at Time of Collection
Analyzed for Resin Acids/Guaiacols Only
DH/cm
8-10-87

Table 2a. Results of Grays Harbor Resin Acid/Guaiacol Laboratory Blanks and Matrix Spike Analyses

		LAB BLANKS			MATRIX SPIKES				
			SAMPLE ID 1874 -			SAMPLE ID 1874 -		1974 -	
CAS #	Compound	Units	27W	31W	Units	55Y	55YJ	77Y	77YJ
90-05-1	Guaiacol	ug/L	10u	10u	%	144	69	49	9
	4,5,6 Trichloroguaiacol*	ug/L	10u	10u	%	138	145	--	188
944-31-6	Tetrachloroguaiacol*	ug/L	10u	10u	%	121	106	1	149
4833-65-8	Retene	ug/L	10u	10u	%	90	95	183	117
1686-62-0	Isopimaric Acid*	ug/L	20u	20u	%	88	82	2	107
3513-69-7	Levopimaric Acid*	ug/L	20u	20u	%	--	--	13	--
1235-74-1	Dehydroabictic Acid*	ug/L	10u	10u	%	94	90	4	145
127-25-3	Abietic Acid*	ug/L	10u	10u	%	89	83	1	39
3310-97-2	Neoabietic Acid*	ug/L	20u	20u	%	1	0.1	2	0.1
	Dichlorodehydroabietic Acid*	ug/L	10u	10u	%	51	56	1	96
2459-25-8	2-Napthoic Acid* (surrogate spike)	%	90	85	%	93	95	13	118
	Sample pH	pH	--	--	pH	7.2	7.2	10	10

* Measured as the Methylester
** Sample Preserved at pH 10 at Time of Collection
Analyzed for Resin Acids/Guaiacols Only

DH/cm
8-10-87

TENTATIVELY IDENTIFIED COMPOUNDS

BNA - Methylated

PROJECT: Grays Harbor COMPILED BY: O. Huntamer DATE: 8/5/87

LABORATORY: Manchester REVIEWED BY: _____ DATE: _____

CAS #	Compound Name	Sample No.	1874 51	1874 52	1874 53	1874 54	1874 55	WBN7131W	WBN7127W		
		Hit Ratio									
		Conc units	J µg/L	J µg/L	J µg/L	J µg/L	J µg/L	J µg/L	J µg/L		
140-03-4	9-octadecanoic acid, 12-(acetyloxy)-, methyl ester		85.	83.	51.	23.	65.	ND	N.D.		
112-62-9	9-octadecanoic acid (z)-methyl ester		N.D.	N.D	N.D	N.D.	35.	N.D.	N.D.		
61566-34-5 (8571)	Benzacetic acid, α-methyl-4-(2-methyl propyl)-, methyl ester		N.D.	N.D	8.7	N.D.	N.D.	N.D.	N.D.		
2675-77-6	Benzene, 1,4-Dichloro-2,5-Dimethoxy-		N.D.	60	N.D	N.D.	N.D	N.D	N.D.		
944-61-6	Benzene, 1,2,3,4-Tetrachloro-5,6-Dimethoxy-		N.D	75.	N.D	N.D.	N.D.	N.D.	N.D.		
0											
11											
12											
13											
14											
15											

From Roy's Mac
Aug 1986

TENTATIVELY IDENTIFIED COMPOUNDS

BNA Methylated

PROJECT: Grey's Harbor COMPILED BY: D-Hartamer DATE: 5/5/87

LABORATORY: Manchester REVIEWED BY: _____ DATE: _____

	CAS #	Compound Name	Sample No.	1974 75	1974 76	1974 77	1974 78	1974 79		1974 75	1974 76	1974 78
			Hit Ratio							pH 5	pH 5	pH 5
			Conc units									
1	87-40-1	Benzene, dichloro-dimethoxy-		~~20.~~	5.1	N.D	SAMPLE	N.D		N.D	N.D	N.D
2	140-03-4	9-octadecanoic acid, 12-(acetyloxy)-, methyl ester (R-(Z))-		220.	N.D	29.		N.D		590	N.D	92
3	5129-64-6	Decanoic Acid, 8-methyl, Methyl ester		92.	N.D	N.D		N.D		N.D	N.D	N.D
4	13481-95-3	10 octadecanoic acid methyl ester		360.	N.D	N.D		N.D		740	N.D	N.D
5	5129-61-3	Heptadecanoic acid 16-methyl-, methyl ester		180.	N.D	N.D		N.D		410	N.D	71
6	2675-77-6	Benzene, 1,4-dichloro-2,5 dimethoxy-		N.D ~~10~~	10	N.D		N.D		N.D	N.D	N.D
7	79-34-5 (10636)	Ethane, 1,1,2,2-Tetrachloro		N.D	N.D	57.		14.		N.D	N.D	9.1
8	106-73-0 (6940)	Heptadecanoic acid methyl ester		N.D	N.D	N.D		N.D		240.	N.D	N.D
9	20185-59-1	Benzoic Acid, 4-(1-methylethyl)-, methyl ester		N.D	N.D	N.D		N.D		9.8	N.D	N.D
10	17904-29-9	Cyclohexanecarboxylic acid 4-(1,5-dimethyl-3-oxahexyl)-, methyl ester		N.D	N.D	N.D		N.D		990	N.D	N.D
11	80-40-0 (10694)	Benzenesulfonic acid, 4-methyl, ethyl ester		N.D	N.D	N.D		N.D		N.D	55	N.D
12	57186-69-3	Benzenesulfonamide N-4-dimethyl-N propyl		N.D	N.D	N.D		N.D		N.D	110	N.D
13	61566-34-5	Benzene acetic acid, x methyl-4-(2 methyl propyl)-, methyl ester		N.D	N.D	N.D		N.D		N.D	N.D	8.6
14	78-57-3	Ethanol, 2 Butoxy-phosphate (3:1)		N.D	N.D	N.D		N.D		N.D	N.D	260
15												

From Roy's Mac
Aug 1986