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(Type of Report)

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DATE: May 30, 1986

TITLE: ANALYSIS OF LULING SAMPLES

AUTHORS: S. Gibson, S. Walker and R.W. Noble

ABSTRACT: Samples from Luling were received for the analysis of PCDOs, PCDFs, 2,3,7,8-TCDD, metals, acid and base/neutral extractables, pesticides and Aroclor formulations. This report gives the results of these analyses.

TECHNICAL APPROVAL:

P.L. Sherman
P.L. Sherman
Environmental Sciences Center
Senior Research Group Leader

APPROVED BY:

William M. Mees
W.M. Mees
Environmental Sciences Center
Quality Assurance Specialist

APPROVAL DATE:

5-30-86

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ANALYSIS REPORT

DATE: 22 May 1986

PROJECT NUMBER: 6405

LOG NUMBER: U-86-03-14-01

NOTEBOOK PAGE: 3353290

TO: L. Adams/ 1560

cc FILES/Mees
P. Sherman
R. Noble

Title: Analysis of Luling Samples

Procedure: See attached report

Abstract:

Samples from Luling were received for the analysis of PCDDs, PCDFs, 2,3,7,8-TCDD, metals, acid and base/neutral extractables, pesticides and Aroclor formulations. This report gives the results of these analyses.

Analysts: S. Gibson, S. Walker and R. W. Noble

ANALYST APPROVAL:

Roy W. Noble

Roy W. Noble

Senior Research Chemist

ANALYSIS REPORT FOR LULING SAMPLES

INTRODUCTION

Nine soils, two sediments and four wipe samples were received for selected analyses of polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), 2,3,7,8-tetrachloro dibenzo-p-dioxin (2,3,7,8-TCDD), selected metals, acid and base/neutral extractables, pesticides and Aroclor formulations. The metals, acid and base/neutral extractables, pesticides and Aroclor formulation analyses on samples #57 and #5 were performed by Environmental Testing and Certification Corporation (ETC) of Edison, NJ. The PCDD, PCDF and 2,3,7,8-TCDD analyses were performed in our laboratories. For internal purposes the samples were assigned Log number U-86-03-14-01. For quality assurance in the analyses we performed MRC Method #07-QAQC-R3 was followed. Briefly this method requires that for every set of samples of the same matrix type a replicate sample, a low spiked sample, a high spiked sample and a method blank must be analyzed. The reports for the analyses performed by ETC are attached at the end of this report.

PREPARATION AND ANALYSIS

Soil and sediment samples of 10 g were weighed and spiked with appropriate internal standards. Wipes were spiked with carbon-13 labeled 2,3,7,8-TCDD prior to extraction. Then 15 mL of methanol (all solvents are Burdick and Jackson "distilled in glass" grade) and 150 mL of hexane are added. The samples are then shaken for 3 hours on a wrist action shaker and the hexane is decanted off. The hexane extracts were washed with 40 mL of concentrated sulfuric acid (until clear), 20 mL of water and 40 mL of 1 N potassium hydroxide and 30 mL of water. The hexane was dried over sodium sulfate and concentrated to 3 mL.

The samples were then placed on the head of a mixed phase liquid chromatographic column consisting of (top to bottom) 2 g of silica, 4 g of silica plus 40% sulfuric acid, 1 g of silica, 2 g of silica plus 36% potassium hydroxide and 1 g of silica. The column was eluted with 50 mL of hexane which was subsequently concentrated to 3 mL. The hexane was then placed on the head of a column containing 2.5 g of Woelm basic alumina. This column was first eluted with 10 mL of 2% methylene chloride/hexane (v/v) mixture and this fraction discarded. An additional elution was made with 15 mL of a 50% methylene chloride/hexane (v/v) mixture to elute the PCDDs and PCDFs from the column.

For some of the samples it was necessary to conduct further cleanup using the EPA Option D technique in order to analyze for TCDDs. Option D involves placing the sample on top of a column containing 18% Carbowax C on Celite packed to a depth of 2 cm in

a 1.0 cm ID pipet. The column was then eluted with 1 mL of hexane, 1 mL of 1:1 cyclohexane/methylene chloride, 1 mL of 75:20:5 methylene chloride/methanol/benzene and finally 2 mL of toluene. The toluene fraction was collected to be concentrated.

All samples had 50 uL of dodecane containing tribromobiphenyl as an internal standard added to the eluant as a keeper and were then concentrated to 50 uL under a gentle stream of purified nitrogen and were submitted for GC/MS analysis. The GC/MS analysis was conducted on a Hewlett/Packard 5987 instrument. The mass spectrometer was operated in the selected ion monitoring mode analyzing for ions characteristic of the native (unlabeled) PCDDs, PCDFs and the internal standards.

RESULTS

The results of the analyses are given in Tables 1-5. Results are not corrected for recovery for the PCDD and PCDF analyses in Tables 1 and 3. Replicate analysis results for sample # 5 show a large variability due to the presence of shells in the sample causing homogeneity problems. Discussion with the customer determined that these results met their needs.

Table 1. PCDD RESULTS FOR SOIL AND SEDIMENT (in ppb)

Sample	C1-4	C1-5	C1-6	C1-7	C1-8
Background	ND	ND	ND	0.4	1.2
Soil #5	600	0.7	3.3	1.5	3.1
Soil #5 R	7500	ND	2.2	5.3	8.9
Soil #13	0.9	ND	3.6	30	190
Soil #27	2.1	ND	ND	0.4	1.5
Soil #35	0.7	ND	0.7	1.5	21
Soil #39	0.8	ND	0.8	1.6	18
Soil #51	ND	ND	0.4	0.6	1.5
Soil #51 H	10.1	19.1	11.6	11.3	11.9
% recovery	107%	170%	103%	91%	109%
spike level	9.4	11.4	10.9	11.8	9.5
Soil #57	5.2	ND	1.4	0.8	1.4
Soil #59	4.8	ND	4.0	ND	0.2
Sediment #1	ND	ND	3.0	38	160
Sediment #1 L	1.1	1.2	3.5	29	104
% recovery	110%	100%	-a	-a	-a
spike level	1.0	1.2	1.1	1.2	1.0
Sediment #2	18	0.3	8.0	89	420
BLANK	ND	ND	ND	ND	ND
MDL	0.5	0.5	0.5	0.5	0.5

MDL method detection limit

ND none detected at method detection limit

R replicate sample

L,H low and high spiked samples

a

spike recovery can not be calculated as sample is three times the spike level.

Table 2. REPEATED PCDD RESULTS FOR SEDIMENT # 2 (in ppb)

<u>Sample</u>	<u>C1-4</u>	<u>C1-5</u>	<u>C1-6</u>	<u>C1-7</u>	<u>C1-8</u>
Sediment # 2	15	ND	20	127	1000
Sediment # 2 R	13	ND	22	156	1500
BLANK	ND	ND	ND	ND	ND
<u>MDL</u>	0.5	0.5	0.5	0.5	0.5
MDL method detection limit					
ND none detected at method detection limit					
R replicate sample					

Table 3. PCDF RESULTS FOR SOIL AND SEDIMENT (in ppb)

Sample	C1-4	C1-5	C1-6	C1-7	C1-8
Background	ND	ND	ND	ND	ND
Soil #5	3.9	1.4	3.8	0.9	0.8
soil #5 R	4.9	3.5	5.4	1.2	1.1
Soil #13	0.2	2.8	4.2	0.9	2.4
soil #27	ND	ND	ND	ND	ND
Soil #35	ND	ND	<0.9	0.4	0.9
Soil #39	ND	ND	<0.6	0.4	1.0
Soil #51	ND	ND	ND	ND	ND
Soil #51 H	5.0	25	32	8.1	33
% recovery	37%	84%	64%	76%	93%
spike level	13.6	29.4	49.7	10.7	35.3
Soil #57	ND	ND	ND	ND	ND
Soil #59	ND	ND	ND	ND	ND
Sediment #1	0.2	<1.6	<1.9	0.6	2.4
Sediment #1 L	0.7	4.7	4.3	1.0	5.3
% recovery	46%	100%	82%	90%	81%
spike level	1.0	1.2	1.1	1.2	1.0
Sediment #2	<0.6	<8.2	<22	1.9	5.5
BLANK	ND	ND	ND	ND	ND
MDL	0.5	0.5	0.5	0.5	0.5

MDL method detection limit
 ND none detected at method detection limit
 R replicate sample
 L,H low and high spiked samples

Table 4. 2378-TCDD RESULTS FOR SOIL AND SEDIMENT (in ppb)

<u>Sample</u>	<u>2378-TCDD</u>	<u>ppb Added</u>
Background	ND	
Soil #5	510	
Soil #5 R	7000	
Soil #13	1.8	
Soil #27	1.6	
Soil #35	0.9	
Soil #39	0.7	
Soil #51	0.1	
Soil #51 H	8.2	9.4
Soil #57	5.2	
Soil #59	2.6	
Sediment #1	ND	
Sediment #1 L	1.0	1.0
Sediment #2	16.5	
BLANK	ND	
MDL	0.2	

MDL method detection limit

ND none detected at method detection limit

R replicate sample

L,H low and high spiked samples

Table 5. 2378-TCDD RESULTS FOR WIPES (in ng)

<u>Sample</u>	<u>2378-TCDD</u>	<u>ng Added</u>
Auger 1 Bit	ND	
Auger 1 Handle	ND	
Auger 2 Bit	3.4	
Auger 2 Handle	ND	
High Spike	44	48
Low Spike	4.3	4.8
Blank	ND	
MDL	0.3	
MDL method detection limit		
ND none detected at method detection limit		

Technical Report

for

MONSANTO COMPANY
800 N.LINDBERGH BLVD.
R3B
ST.LOUIS, MO 63167

Chain of Custody Data Required for ETC Data Management Summary Reports

L7194	MONSANTO COMPANY	MONCORPENG	5	060314				
ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed	Hours	


John J. Fitzgerald
Vice President
Research and Operations

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Introduction

This report contains the analytical results on your soil sample, 5 86/03/14. It is designed to include comprehensive data from the entire analytical process in order to satisfy the needs of various levels of review.

The results obtained from your sample are presented in tabular format immediately following this introduction. Quality assurance data is tabulated along with the appropriate sample results for verification. Depending on the analyses ordered, the quality assurance data may include results from blank, spiked blank, spiked sample (i.e. matrix spike) and replicate samples as well as results from surrogate compound analyses. Quality assurance data for verification of proper instrument performance is also included where appropriate. The report appendices include the chain of custody record for your sample and, where appropriate, the gas chromatograms and mass spectra.

The procedures used in the analysis of the sample are described in this report's methodology section. All analytical procedures within our laboratory are performed within a strictly enforced Quality Assurance Protocol. A description of this Protocol is included in the report.

Results

Sample results, and associated quality assurance data, are always tabulated in one or more of this report's Quantitative Results Tables. The format of each table varies with the class of analysis.

Priority Pollutants

The priority pollutant compounds and elements are listed with their NPDES (National Pollution Discharge Elimination System) numbers, and the Method Detection Limit (MDL) published in the Federal Register. When a compound or element is present below its published MDL it is reported as BMDL (Below Method Detection Limit). When a compound or element is not present at any detectable concentration it is reported as ND (Not Detected). MDL's for non-aqueous matrices are based on USEPA published MDL's but are adjusted as per sample weight. Matrix spike and replicate analyses, where included, were performed on samples randomly chosen within each quality assurance batch and are therefore not necessarily spikes and replicates of this report's sample. Surrogate compound recovery data and instrument calibration data are included in the Method Performance Data Tables.

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA

Acid Compounds - GC/MS Analysis Data (ORO2)

Chain of Custody Data Required for ETC Data Management Summary Reports

L7194 MONSANTO COMPANY

MONSANTO S

868316

ETC Sample No

(Owner)

Facility

Sample Date

Date

Time

Operator

NPDES Number	Compound	Results		GC Duplicate		GC Blank and Spiked Blank			GC Matrix Spike		
		Sample Concentration ug/kg	ND ug/kg	First ug/kg	Second ug/kg	Blank Data ug/kg	Concen Added ug/kg	% Recov	Unspiked Sample ug/kg	Concen Added ug/kg	% Recov
1A	2-Chlorophenol	ND	110	ND	ND	ND	0	-	ND	3200	84
2A	4-Dichlorophenol	283	80	ND	ND	ND	0	-	ND	3200	88
3A	4-Dimethylphenol	ND	80	ND	ND	ND	0	-	ND	3200	73
4A	6-Dinitro-2-cresol	ND	80	ND	ND	ND	0	-	ND	3200	48
5A	2,4-Dinitrophenol	ND	1400	ND	ND	ND	0	-	ND	3200	36
6A	2-Nitrophenol	ND	120	ND	ND	ND	0	-	ND	3200	84
7A	4-Nitrophenol	ND	80	ND	ND	ND	0	-	ND	3200	85
8A	p-Chloro-m-cresol	ND	180	ND	ND	ND	0	-	ND	3200	82
9A	Pentachlorophenol	127	120	ND	ND	ND	0	-	ND	3200	81
10A	Phenol	ND	80	ND	ND	ND	0	-	ND	3200	80
11A	2,4,6-Trichlorophenol	6600	90	ND	ND	ND	0	-	ND	3200	86

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA
BASE/NEUTRAL COMPOUNDS - GC/MS ANALYSIS DATA (OR03)

Chain of Custody Data Required for ETC Data Management Summary Reports					
L7194	MONSANTO COMPANY	MONCORPENE S	060314		
ETC Sample No.	Company	Facility	Sample Point	Date	Time

NPLIS Number	Compound	Results		GC Replicate		GC Blank and Spiked Blank			GC Matrix Spike		
		Sample Concn. ug/kg	MDL ug/kg	First ug/kg	Second ug/kg	Blank Data ug/kg	Concen Added ug/kg	% Recov	Unspiked Sample ug/kg	Concen Added ug/kg	% Recov
10	Acenaphthene	NO	63	666	666	666	0	-	NO	3200	77
20	Acenaphthylene	NO	128	666	666	666	0	-	NO	3200	74
30	Anthracene	NO	63	666	666	666	0	-	NO	3200	77
40	Benizidine	NO	1500	666	666	666	0	-	NO	3200	0
60	Benzo(a)anthracene	NO	260	666	666	666	0	-	NO	3200	77
65	Benzo(b)pyrene	NO	83	666	666	666	0	-	NO	3200	75
70	Benzo(b)fluoranthene	NO	330	666	666	666	0	-	NO	3200	79
80	Benzo(g,h)perylene	NO	128	666	666	666	0	-	NO	3200	73
90	Benzo(k)fluoranthene	NO	128	666	666	666	0	-	NO	3200	70
100	bis(2-Chloroethyl)ethane	NO	180	666	666	666	0	-	NO	3200	86
110	bis(2-Chloroethyl) ether	NO	190	666	666	666	0	-	NO	3200	70
120	bis(2-Chloroisopropyl) ether	NO	190	666	666	666	0	-	NO	3200	86
130	bis(2-(4-hydroxy)phenyl) ether	NO	330	666	666	666	0	-	NO	3200	72
140	4-Bromophenyl phenyl ether	NO	63	666	666	666	0	-	NO	3200	100
150	Butyl benzyl phthalate	NO	320	666	666	666	0	-	NO	3200	88
160	2-Chlorophthalate	NO	63	666	666	666	0	-	NO	3200	78
170	4-Chlorophenyl phenyl ether	NO	128	666	666	666	0	-	NO	3200	78
180	Chrysene	NO	63	666	666	666	0	-	NO	3200	78
190	Dibenz(a,h)anthracene	NO	320	666	666	666	0	-	NO	3200	77
200	1,2-Dichlorobenzene	NO	63	666	666	666	0	-	NO	3200	75
210	1,4-Dichlorobenzene	NO	63	666	666	666	0	-	NO	3200	77
220	1,4-Dichlorobenzene	NO	128	666	666	666	0	-	NO	3200	8
230	3,3'-Dichlorobenzidine	NO	63	666	666	666	0	-	NO	3200	76
240	Diethyl phthalate	NO	320	666	666	666	0	-	NO	3200	67
250	Diethyl phthalate	NO	320	666	666	666	0	-	NO	3200	64
260	Di-n-butyl phthalate	NO	320	666	666	666	0	-	NO	3200	61
270	2,4-Dinitrophenol	NO	190	666	666	666	0	-	NO	3200	82
280	2,6-Dinitrophenol	NO	63	666	666	666	0	-	NO	3200	103
290	Di-n-octyl phthalate	NO	320	666	666	666	0	-	NO	3200	76
300	1,2-Diphenylhydrazine	NO	320	666	666	666	0	-	NO	3200	76
310	Fluoranthene	NO	63	666	666	666	0	-	NO	3200	76
320	Fluorene	NO	63	666	666	666	0	-	NO	3200	76

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TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA
BASE/NEUTRAL COMPOUNDS - GC/MS ANALYSIS DATA (OR03)

Chain of Custody Data Required for ETC Data Management Summary Reports

L7184 MONSANTO COMPANY MONCORPEN 6 860314
ETC Sample No. Company Facility Sample Point Date Time Analyst

NPDES Number	Compound	Results		QC Replicate		QC Blank and Spiked Blank			QC Matrix Spike		
		Sample Concentration ug/kg	MDL ug/kg	First ug/kg	Second ug/kg	Blank Data ug/kg	Concns Added ug/kg	R Factor	Unspiked Sample ug/kg	Concns Added ug/kg	% Recover
338	Hexachlorobenzene	ND	63	ND	ND	ND	0	-	ND	3200	68
348	Hexachlorobutadiene	ND	30	ND	ND	ND	0	-	ND	3200	79
350	Hexachlorocyclopentadiene	ND	330	ND	ND	ND	0	-	ND	0	-
360	Hexachlorocyclohexene	ND	63	ND	ND	ND	0	-	ND	3200	79
370	Indeno[1,2,3-c]dipyrrene	ND	160	ND	ND	ND	0	-	ND	0	-
380	Isophorone	ND	73	ND	ND	ND	0	-	ND	3200	81
390	Naphthalene	ND	63	ND	ND	ND	0	-	ND	3200	77
400	Nitrobenzene	ND	63	ND	ND	ND	0	-	ND	3200	76
410	N-Nitrosodimethylamine	ND	330	ND	ND	ND	0	-	ND	0	-
420	N-Nitrosodi-n-propylamine	ND	330	ND	ND	ND	0	-	ND	3200	85
430	N-Nitrosodiphenylamine	ND	63	ND	ND	ND	0	-	ND	3200	66
440	Phenanthrene	ND	160	ND	ND	ND	0	-	ND	3200	82
450	Pyrene	ND	63	ND	ND	ND	0	-	ND	3200	96
460	1,2,4-Trichlorobenzene	ND	63	ND	ND	ND	0	-	ND	3200	77

ND = Not Detected

Chain of Custody Data Required for ETC Data Management Summary Reports

NPDES Number	Compound	Result	
		SAMPLE CONCENTRATION ug/kg	NOL ug/kg
1M	Antimony	ND	14000
7K	Arsenic	8000	1000
9K	Beryllium	600	100
4H	Cadmium	M-DL	450
5H	Chromium	25000	1900
6H	Copper	27000	2500
7H	Lead	32000	6200
8H	Mercury	700	100
9H	Nickel	31000	1000
10H	Selenium	ND	500
11H	Silver	ND	1000
12H	Iodine	ND	500
13K	Zinc	15000	700
	Calcium	107000000	12000
	Iron	134000000	17000
	Aluminum	69400000	13000
	Manganese	611000	400
	Boron	110000	500
	Cobalt	4500	6100
	Magnesium	8060000	9200
	Sodium	375000	22000
	Tin	M-DL	6500
	Vanadium	17000	2300
	Potassium	1340000	4000

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TABLE 2: METHOD PERFORMANCE DATA
Surrogate Recovery Soil - GC/MS Data (QR20)

Chain of Custody Data Required for ETC Data Management Summary Reports

LT194	MONSANTO COMPANY	MONCORPENG 5	86631	0
ETC Date: 4/20	Company	Facility	Sample Point	Date Time Sampled

Compound	Amount Added	% Recovery	Control Limits B	
			Lower	Upper
VOLATILE FRACTION				
Toluene-D8	-	-	50	100
o-Bromofluorobenzene	-	-	50	100
1,2-Dichloroethane-D4	-	-	50	100
ACID FRACTION				
Phenol-D5	100	86	20	100
2-Fluorophenol	100	90	20	100
2,6-Di-Bromophenol	100	100	10	100
BASE/NEUTRAL FRACTION				
Nitrobenzene-D5	50	76	20	100
2-Fluorobiphenyl	50	62	20	100
Terphenyl-D10	50	49	20	150

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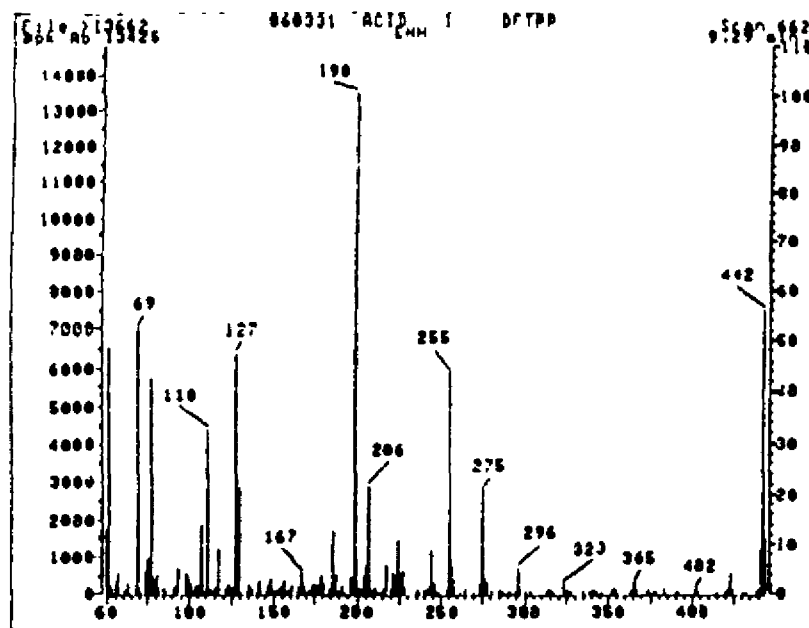


TABLE 2: METHOD PERFORMANCE DATA (QR22)

GC/MS Tuning Data - Decafluorotriphenylphosphine (DFTPP) for Acids Analysis

m/z	Ion Abundance Criteria	% Relative Abundance Base Peak	% Relative Abundance Appropriate Peak	Status
51	30-60% of mass 198	48.32	48.32	Ok
68	Less than 2% of mass 69	.95	1.81	Uk
69	(reference only)	52.22	52.22	Ok
70	Less than 2% of mass 69	.23	.43	Ok
127	40-60% of mass 198	47.25	47.25	Ok
197	Less than 1% of mass 198	0.00	0.00	Uk
198	Base peak, 100% relative abundance	100.00	100.00	Ok
199	5-9% of mass 198	6.94	6.94	Uk
275	10-30% of mass 198	20.47	20.47	Uk
365	Greater than 1% of mass 198	2.33	2.33	Uk
441	0-100% of mass 443	9.10	82.74	Ok
442	Greater than 40% of mass 198	55.89	55.89	Ok
443	17-23% of mass 442	10.99	19.67	Ok

Injection Date: 03/31/86
Injection Time: 14:59
Run No: 13662
Spectrum No: 662

Analyst: J. Smith
Processor: A. C. Smith
QC Batch: QA 4747
Samples: L7195, L7249, L7194, L7248, L7196, L7315, L7321, L7322

4/1/86

MONS 220955

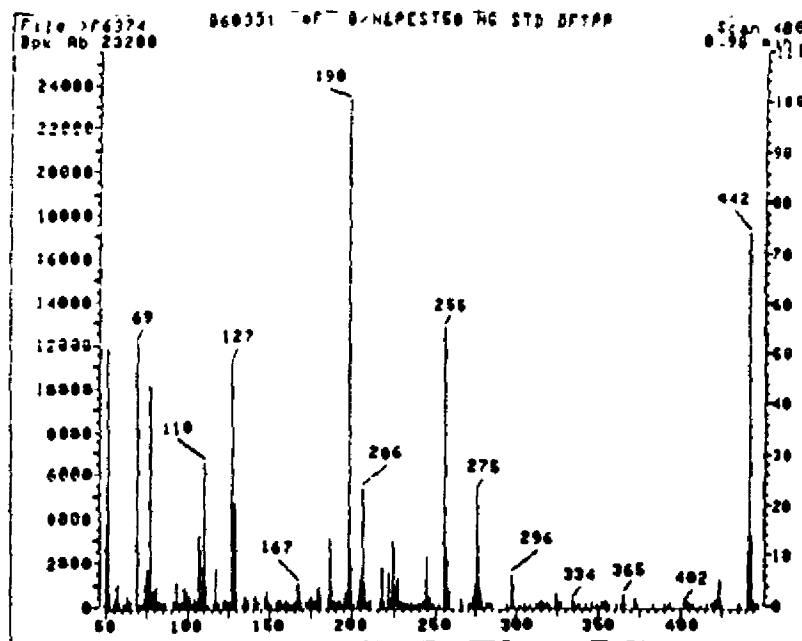


TABLE 21 METHOD PERFORMANCE DATA (QR23)

GC/MS Tuning Data - Decafluorotriphenylphosphine (DFIPP) for Base/Neutral Analysis

m/z	Ion Abundance Criteria	% Relative Abundance Base Peak	% Relative Abundance Appropriate Peak	Status
51	30-60% of mass 198	50.74	50.74	OK
68	Less than 2% of mass 69	0.00	0.00	OK
69	(reference only)	52.21	52.21	OK
70	Less than 2% of mass 69	.26	.50	OK
127	40-60% of mass 198	48.40	48.40	OK
197	Less than 1% of mass 198	0.00	0.00	OK
198	Base peak, 100% relative abundance	100.00	100.00	OK
199	5-9% of mass 198	7.32	7.32	OK
275	10-30% of mass 198	22.40	22.40	OK
365	Greater than 1% of mass 198	2.43	2.43	OK
441	0-100% of mass 443	10.56	76.75	OK
442	Greater than 40% of mass 198	74.07	74.07	OK
443	17-23% of mass 442	13.75	18.57	OK

Injection Date: 04/01/86

Injection Time: 00:41

Run No: >F6374

Spectrum No: 400

Analyst: *And. Munion*

Processor: *John Carpenter*

QC Batch: *604747*

Samples: *L4248-L4249, L7194-L7195, L6314-L6315, L6321-L6322*

VS
4/5/86

MONS 220956

Methodology for GC/MS Analysis of Priority Pollutant Compounds

The methods employed in the GC/MS analysis for priority pollutants are established EPA methods. Rigid compliance with the instrument parameters and performance criteria of the published methods was achieved. In some cases the precise amounts of sample used and the sample handling procedures vary with the complexity of the sample matrix. Qualitative identification of the priority pollutants was performed using the relative retention times, the relative abundance of three characteristic ions and the abundance ratios. The entire mass spectrum was reviewed to confirm each identification. Quantitative analysis of detected compounds was performed by using a response factor generated by a major characteristic ion of the specific compound and an internal standard.

Compounds, in addition to those on the priority pollutant list, were identified through a computer-aided search of the NBS-EPA spectra library. After review the identifications are included in a separate tabulation and labelled "tentatively identified".

Acid, Base/Neutral and Pesticide Priority Pollutant Compounds

For the analysis of the Acid, Base/Neutral and Pesticide priority pollutants in a soil matrix, a modification of EPA Method 625 was used. The method can be summarized as follows: A 30 gm semi-wet soil sample is Soxhlet extracted with a 1:1 mixture of acetone and hexane. The acetone is thermally stripped. The remaining hexane extract is diluted to 200 ml with methylene chloride and twice extracted with an aqueous NaOH solution followed by an extraction with deionized water. The methylene chloride extract is dried and concentrated to a 1 ml final volume. This concentrate is injected into a GC/MS instrument configured for the analysis of Base/Neutral priority pollutant compounds. The remaining aqueous extracts are combined and the pH adjusted to a value less than 2. The aqueous phase is serially extracted with 3 aliquots of methylene chloride. The methylene chloride extracts are dried, concentrated to a 1 ml final volume. The concentrate is injected into a GC/MS instrument configured for the analysis of acid extractable priority pollutant compounds.

Methodology for Analysis of Metals

AQUEOUS

The determination of metals in aqueous samples is performed according to the methods published by EPA in "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020 March 1983 and the Federal Register, October 26 1983. Arsenic, selenium and thallium are determined by furnace AA; silver, aluminum, barium, beryllium, boron, cadmium, calcium, chromium, copper, cobalt, iron, magnesium, manganese, molybdenum, nickel, lead, sodium, antimony, tin, titanium, vanadium, and zinc are determined by ICP emission spectrometry, except where lower levels of detection are required in these cases (e.g. lead in groundwater monitoring samples) furnace AA is used. All furnace AA parameters are run by method of standard additions. The determination of mercury is performed by cold vapor AA.

EP TOXICITY

The determination of metals in aqueous EP Toxicity leachates is performed according to the methods published by EPA in "Test Methods for Evaluating Solid Waste" EPA SW-846 revised April 1984 and the Federal Register, Oct. 26 1983, 1979. Silver, arsenic, barium, cadmium, chromium, lead and selenium are determined by ICP emission spectrometry. Mercury is determined using cold vapor AA. For leachates that are organic in nature, the analyses are performed according to the methods described under OIL/SLUDGE below.

SOIL/SEDIMENT

The determination of silver, beryllium, cadmium, chromium, copper, nickel, antimony, lead and zinc in sediment samples is performed according to methods published by EPA in "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue", EPA 600/4-81-055, October 1980. Mercury is determined according to the sediment method published by EPA in "Method for Chemical Analysis of Water and Wastes", EPA 600/4-79-020 March 1983. Arsenic, selenium and thallium are determined by furnace AA using nitric acid in a closed decomposition vessel for sample digestion.

OIL/SLUDGE

The determination of silver, aluminum, boron, barium, beryllium, calcium, cadmium, copper, chromium, cobalt, iron, magnesium, manganese, molybdenum, sodium, nickel, lead, antimony, tin, titanium, vanadium, and zinc in sludge/petroleum-based samples is performed by ICP emission spectrometry using a magnesium nitrate dry ashing digestion technique. Arsenic, selenium and thallium are determined by furnace AA using nitric acid in a closed decomposition vessel for sample digestion. Mercury is determined by cold vapor AA using the same digestion technique.

Summary of Quality Assurance/Quality Control Procedures (QA/QC)

ETC bases its quality assurance protocols on the following government guidelines:

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EPA-600/4-79-019, March 1979,

National Enforcement Investigation Center Policies, and Procedures manual, EPA-330/9/79/001-R, October 1979,

the recommended guidelines for EPA Methods 624 and 625 (Federal Register December 3, 1979, updated on October 26, 1984),

"Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples," EPA 600/8-80-038, June 1980,

"Determination of 2,3,7,8-TCDD in Soil and Sediment" EPA, Region VII, Kansas City, September 1983,

Organic Analysis: Multi-media, Multi-Concentration-IFB WA84-A267, and

Dioxin Analysis: Soil/Sediment Matrix, Multi-Concentration, Selected Ion Monitoring with Jar Extraction Procedure-IFB WA84-A002

However, we have modified our protocols to provide a higher level of QA/QC than the guidelines require. For example, we analyze a higher than required number of quality control samples and we pay especially careful attention to the certification of the "reference standard" compounds we use in analysis. Below are listed the key QA/QC elements for the methods we used:

Analysis of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry

- Each batch of 13 samples consists of 9 customer samples (at a maximum), one blank sample, one spiked blank, one spiked sample and one replicate sample. This amounts to a 30% quality control factor.
- Three surrogate compounds are added to each sample in the batch of 13.
- A blind quality control sample is introduced to the laboratory for analysis on a weekly basis.
- Each GC/MS is checked and retuned, if necessary, at the beginning of each day to ensure that its performance on bromofluorobenzene (BFB) meets the EPA criteria.
- A calibration curve for quantitation is prepared using a mixture of Volatile Organic Priority Pollutant "standards" at a minimum of 3 different concentrations and using a mixture of 3 internal standards at a constant concentration.
- The calibration curve is verified with a mixture of priority pollutant standards every day. If the response factors vary greater than 25%, the instrument must be recalibrated.

Analysis of Organic Compounds Extracted in Acid or Base/Neutral Solutions by Gas Chromatography/Mass Spectrometry

- Each batch of 20 samples consists of 16 customer samples (at a maximum), one blank sample, one spiked blank (for water matrices), one sample spiked with the priority pollutant standard mixture and a duplicate customer sample. This amounts to a 20% quality control factor.

ETC ENVIRONMENTAL TESTING and CERTIFICATION

- Three surrogate compounds are added to each sample in the batch for Base/Neutral analysis
- Three surrogate compounds are added to each sample in the batch for Acid analysis
- A blind quality control sample is introduced to the laboratory for analysis on a weekly basis
- Each GC/MS is checked and retuned, if necessary, at the beginning of each day to ensure that its performance on decafluorotriphenylphosphine (DFTPP) meets the EPA criteria
- A calibration curve for quantitation is prepared using a mixture of standards composed of either the Organic Acid or Base/Neutral Extractable Compounds at a minimum of 3 concentrations and using five internal standards for quantitation

Analysis of Metals

All Samples

- New standards are prepared for each batch of samples
- Normal calibration is performed using a blank sample and four standards that have been through the sample preparation procedure. A regression analysis is used to construct the calibration curve
- All EP Toxicity samples and all samples determined by furnace atomic absorption are calculated by the "method of additions"
- For each sample analysis that requires the use of the "method of additions" technique a three point calibration is performed using U.S. EPA "Methods for Chemical Analysis of Water and Wastes, 1979". Results are obtained using linear regression analysis. Any regression with a coefficient of correlation below 0.990 is considered suspect necessitating review of calibration date or sample re-analysis
- In constructing the normal calibration curves the lowest concentration levels we use are values greater than or equal to 5 times the Instrumental Detection Limit (IDL)
- All calibration standards are analyzed in duplicate, at a minimum
- Independent reference standards are used to check the accuracy of calibration standards
- A check standard is analyzed every ten samples to validate the normal calibration curve
- One customer sample out of every ten is analyzed in triplicate

Homogeneous Samples (except for Mercury analysis)

Samples are analyzed in batches of 30 or less. For batches in which the sample matrices are homogeneous, the QC program is a minimum of 25% and consists of analyzing

- 3 sets of triplicate analyses
- 2 Replicate spikes
- 1 independent reference standard,
- 4 Calibration standards (processed using the sample preparation method)
- 4 Calibration standards (without sample preparation), and
- 1 Reagent Blank

ETC ENVIRONMENTAL TESTING and CERTIFICATION

Heterogeneous Samples (except for Mercury analysis)

Samples are analyzed in batches of 30 or less. For batches in which the sample matrices are heterogeneous, the QC program is a minimum of 35% and consists of analyzing

- 3 sets of triplicate analyses
- 2 Replicate spikes,
- 1 Replicate independent reference standards,
- 4 Calibration standards (processed using the sample preparation method),
- 1 Procedural Blank,
- 4 Calibration standards (without sample preparation), and
- 1 Reagent Blank

Analysis of Mercury

To analyze samples for mercury we group them by matrix in batches of 30 or less. Our QC program is a minimum of 30% and consists of analyzing

- each of the 30 customer samples in duplicate,
- 3 sets of triplicate analyses,
- 2 Replicate spikes,
- 2 Replicate independent reference standards,
- 10 Calibration standards (processed using the sample preparation method), and
- 2 Procedural Blanks

Analysis of Pesticides, Herbicides and PCB's by Gas Chromatography

Pesticide, herbicide and PCB samples are grouped in batches of 16 customer samples or less according to the type of analysis to be performed. The QC program for each of these three types of analyses is a minimum of 20% and consists of analyzing

- 1 procedural blank sample (a reagent blank is analyzed in the case of non-water matrices),
- 1 spiked blank sample (the spiked blank is eliminated in the case of non-water matrices),
- 1 replicate sample,
- 1 replicate spiked sample, and
- 1 known reference QC sample for at least each 100 samples analyzed

The instrument is calibrated each run with three standards, and checked every 10 samples

Analysis of Cyanides, Phenols, Fluoride, Chloride, Nitrate and Nitrite

- All parameters are analyzed using a Technicon Autoanalyzer II GT
- 3 calibration standards are analyzed at the beginning and end of each batch

ETC ENVIRONMENTAL TESTING and CERTIFICATION

- Each batch (up to 80 samples) consists of analyzing one blank, one spiked blank, one duplicate and spiked sample every 20 samples, and an EPA known reference sample

Analysis of Total Organic Carbon (TOC)

TOC samples are analyzed on a daily basis with the number of samples analyzed per day dependent on the request for duplicate or quadruplicate analyses. The quality control program is designed to maintain the appropriate amount of QC and consists of the following elements:

- Daily instrument calibration
- One blank
- Standard recalibration every 10 samples
- Spiked samples at a low and high level
- Every sample is run in duplicate at a minimum

Analysis of Total Organic Halide (TOX)

- Blank reagent water for absolute carbon background must contain less than 5 ug/l of halide (as chloride)
- Using a trichlorophenol standard, the mean adsorption efficiency must be within +/- 15% of the standard value
- Calibration standards are run every 10 samples.
- Every sample is run in duplicate at a minimum

Analysis of 2,3,7,8-TCDD (Dioxin) by GC/MS (SIM)

- Each sample is dosed with a known quantity of $^{13}C_{12}$ -2,3,7,8-TCDD as internal standard and $^{37}Cl_4$ -TCDD as surrogate standard. The action limits for surrogate standard results is +/- 40% of the true value. Samples showing surrogate standard results outside of these limits are reextracted and reanalyzed.
- Two laboratory "method blanks" are run along with each set of 24 or fewer samples. The method blank is also dosed with the internal standard and surrogate standard.
- At least one per set of 24 samples is run in duplicate to determine intralaboratory precision.
- Qualitative Requirements: The following are met in order to confirm the presence of native 2,3,7,8-TCDD:
 - a. Isomer specificity must be demonstrated initially and verified once per 8-hour work shift. The verification consists of injecting a mixture containing TCDD isomers which elute close to 2,3,7,8-TCDD. The 2,3,7,8-TCDD must be separated from interfering isomers, with no more than 25% valley relative to the 2,3,7,8-TCDD peak.
 - b. The 320/322 ratio is within the range of 0.67 to 0.87.
 - c. Ions 320, 322, and 257 are all present and maximize together; the signal to mean noise ratio must be 2.5 to 1 or better for all 3 ions.
 - d. The retention time is equal (within 3 seconds) to the retention time for the isotopically labeled 2,3,7,8-TCDD.
 - e. At least one of the positives can be confirmed by obtaining partial scan spectra from mass 150 to mass 350. The partial scan guidelines are as follows:

ETC ENVIRONMENTAL TESTING and CERTIFICATION

the 320/324 ratio should be 158 ± 0.16

the 257/259 ratio should be 103 ± 0.10

the 194/196 ratio should be 154 ± 0.15

- One sample is spiked with native 2,3,7,8-TCDD at a level of 10 PPB (for soil) for each set of 24 or fewer samples
- In cases where no native 2,3,7,8-TCDD is detected, the actual detection limit is estimated and reported based on a signal to noise ratio of 2.5 to 1 at ions 320 and 322
- For each sample, the internal standard is present with at least a 10 to 1 signal to noise ratio for both mass 332 and mass 334. Also, the internal standard 332/334 ratio must be within the range of 0.67 to 0.87

Subcontractor QA/QC

Each subcontractor is required to maintain an appropriate level of quality control. To insure this, each subcontractor is required to submit to ETC the quality control data for all analyses it performs. This data is kept on file at ETC. In general, the amount of quality control required is one duplicate sample with one spiked sample for every ten analyses.

Chain-of-Custody

The chain-of-custody procedure is part of our quality assurance protocol. We believe our chain-of-custody record fully complies with the legal requirements of federal, state and local government agencies and of the courts of law. The record covers:

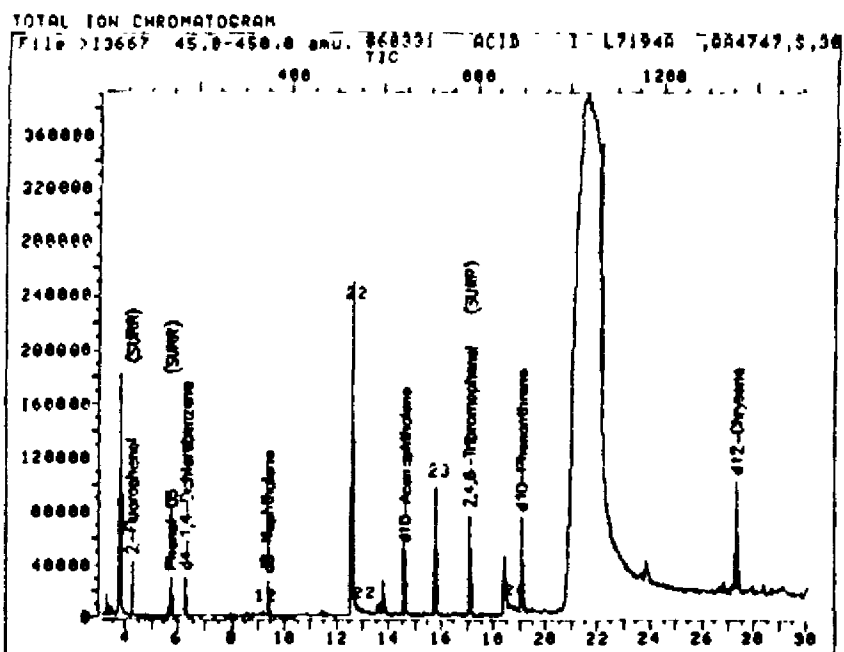
- labeling of sample bottles, packing the Sample Shuttle and transferring the Shuttle under seal to the custody of a shipper,
- outgoing shipping manifests,
- the chain-of-custody form completed by the person(s) breaking the Shuttle seal, taking the sample, resealing the Shuttle and transferring custody to a shipper,
- incoming shipping manifests,
- breaking the Shuttle's reseat,
- storing each labeled sample bottle in a secured area,
- disposition of each sample to an analyst or technician, and
- the use of the sample in each bottle in a testing procedure appropriate to the intended purpose of the sample.

The records show for each link in this process:

- the person with custody, and
- the time and date each person accepted or relinquished custody

Appendix A
Mass Spectral Data
for
Quantitated Compounds

- 1) A total ion chromatogram for each sample analyzed by a GC/MS instrument
- 2) A Quant report used by the analyst to determine qualitative and quantitative results of the compounds present
- 3) A mass spectrum and a reference spectrum for each priority pollutant compound detected in the sample



Data File: >1366711U7
Name: 860331 ACID 1
Misc: L7194A ,0A4747,S,30.0Y,1

BTL#12

Id File: IACID11US
Title: PP/ACID IDFILE
Last Calibration: 860331 14:52

Operator ID: JH2180
Quent Time: 860331 21:36
Injected at: 860331 21:03

MONS 220965

QUANT REPORT

Operator ID: JH2180
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 Date File: ^I366711U7
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 Misc: L7194A ,QA4747,S,30.09,1

Quant Rev: 5 Quant Time: 860331 21:36
 Injected at: 860331 21:03
 Dilution Factor: 1.00

BTLE12

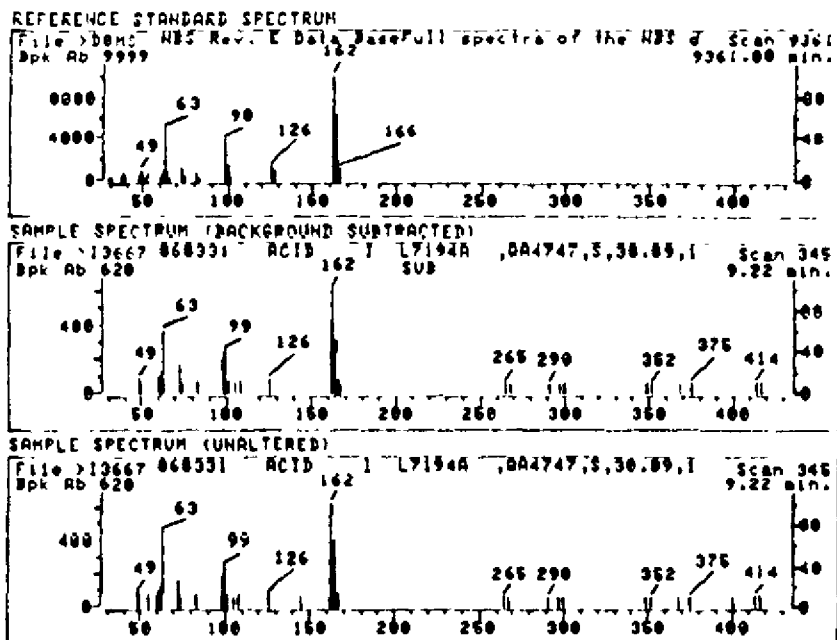
ID File: IACID11US
 Title: PP/ACID ID FILE
 Last Calibration: 860331 14:52

	Compound	R.T.	Score	Area	Conc	Units	q
1)	*d4-1,4-Dichlorobenzene	6.27	179	12846	40.00	UG/ML	98
3)	2-Fluorophenol (SURR)	4.19	62	21599	90.20	UG/ML	98
8)	Phenol-D5 (SURR)	5.72	148	24474	86.32	UG/ML	98
11)	*d8-Naphthalene	9.38	354	28886	40.00	UG/ML	98
12)	2,4-Dichlorophenol	9.22	345	1723	8.51	UG/ML	98
17)	*d10-Acenaphthylene	14.62	649	20642	40.00	UG/ML	98
22)	2,4,6-Trichlorophenol	12.60	535	115838	509.95	UG/ML	98
22)	2,4,6-Trichlorophenol	12.99	557	1797	7.91	UG/ML	98
23)	2,3,4,6-Tetrachlorophenol	15.00	715	25954	167.31	UG/ML	98
24)	*d10-Phenanthrene	19.07	899	80950	40.00	UG/ML	98
25)	2,4,6-Tribromophenol (SURR)	17.06	786	22399	99.76	UG/ML	98
26)	Pentachlorophenol	18.79	883	984	3.67	UG/ML	98
28)	*d12-Chrysene	27.34	1362	92208	40.00	UG/ML	101

* Compound is IS10

MS Data

MONS 220966



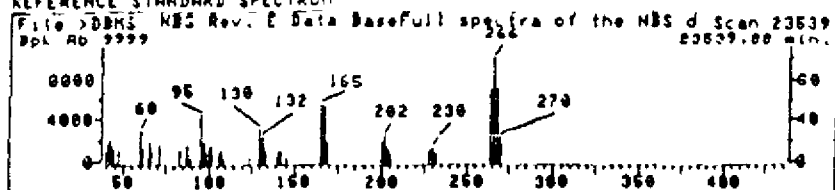
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Misc: L7194A ,QA4747,S,30.09,1
Quant Time: 860331 21:36
Injected at: 860331 21:03

BIL 812

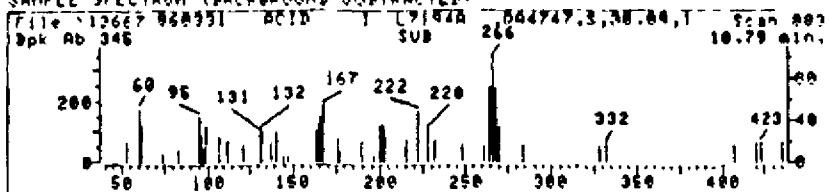
Compound No: 12
Compound Name: 2,4-Dichlorophenol
Scan Number: 345
Retention Time: 9.22 min.
Area: 1723
Concentration: 0.51 UG/ML
q-value: 90

MONS 220967

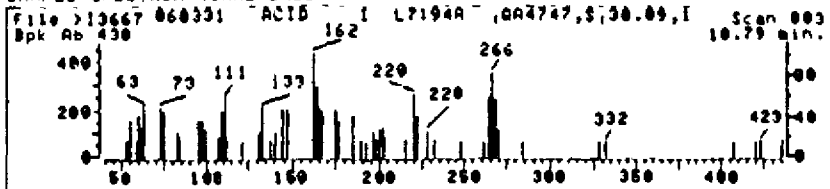
REFERENCE STANDARD SPECTRUM



SAMPLE SPECTRUM (BACKGROUND SUBTRACTED)



SAMPLE SPECTRUM (UNALTERED)



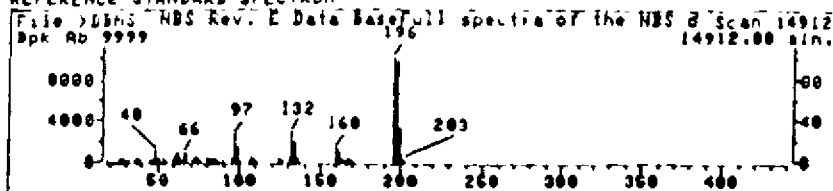
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Quant Time: 860331 21:36
Injected at: 860331 21:03

E1L012

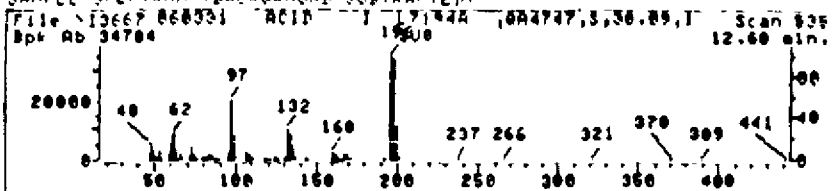
Compound No: 26
Compound Name: Pentachlorophenol
Scan Number: 803
Retention Time: 10.79 min.
Area: 984
Concentration: 3.67 UG/ML
q-value: 89

MONS 220968

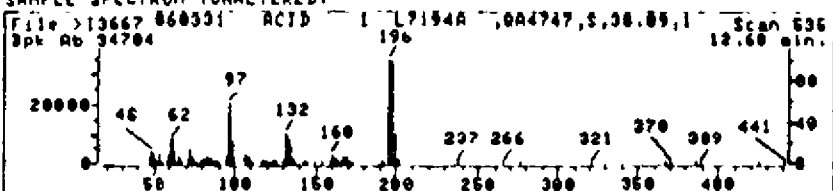
REFERENCE STANDARD SPECTRUM



SAMPLE SPECTRUM (BACKGROUND SUBTRACTED)



SAMPLE SPECTRUM (UNALTERED)

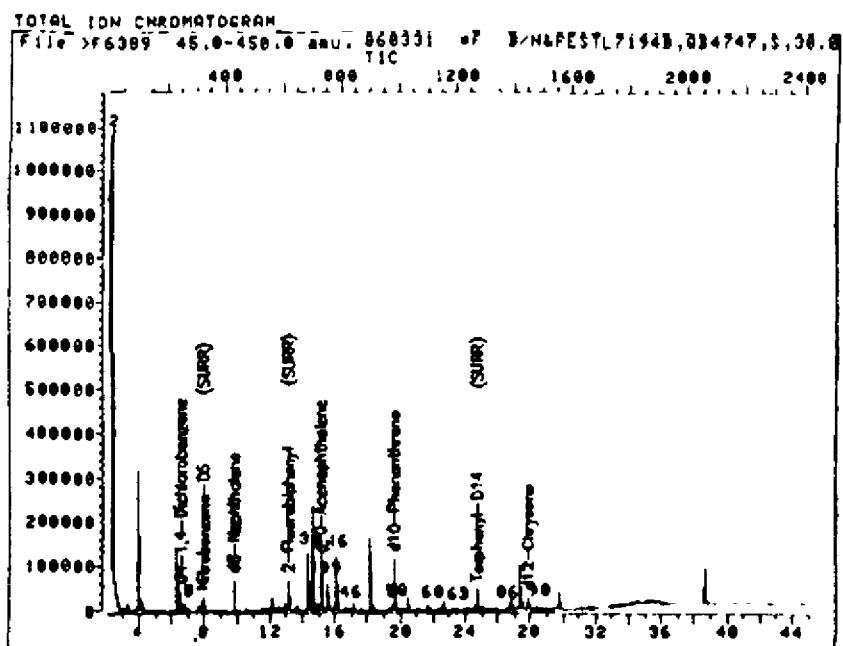


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Misc: L7194A ,QA4747,S,30.09,1
Quant Time: 860331 21:36
Injected at: 860331 21:03

B1L812

Compound No: 22
Compound Name: 2,4,6-Trichlorophenol
Scan Number: 535
Retention Time: 12.60 min.
Area: 115838
Concentration: 509.95 UG/ML
q-value: 97

MONS 220969



Data File: >F638911U2
Name: 860331 #F B/N&PEST
Misc: L7194B,QB4747,3,30.09,1

BTL#23

Id File: FBNP11U5
Title: B/N&PEST ID FILE FOR F 860326
Last Calibration: 860401 12:03

Operator ID: RA0157
Quant Time: 860401 19:32
Injected at: 860401 18:44

MONS 220970

QUANT REPORT

Operator ID: RA0157
 Output File: ^F638911AD
 Data File: >F638911U2
 Name: B60331 SF B/N&PEST
 Misc: L7194B,QB4747,S,30.09,1

Quant Rev: 5 Quant Time: 860401 19:32
 Injected at: 860401 18:44
 Dilution Factor: 1.00

BTL#23

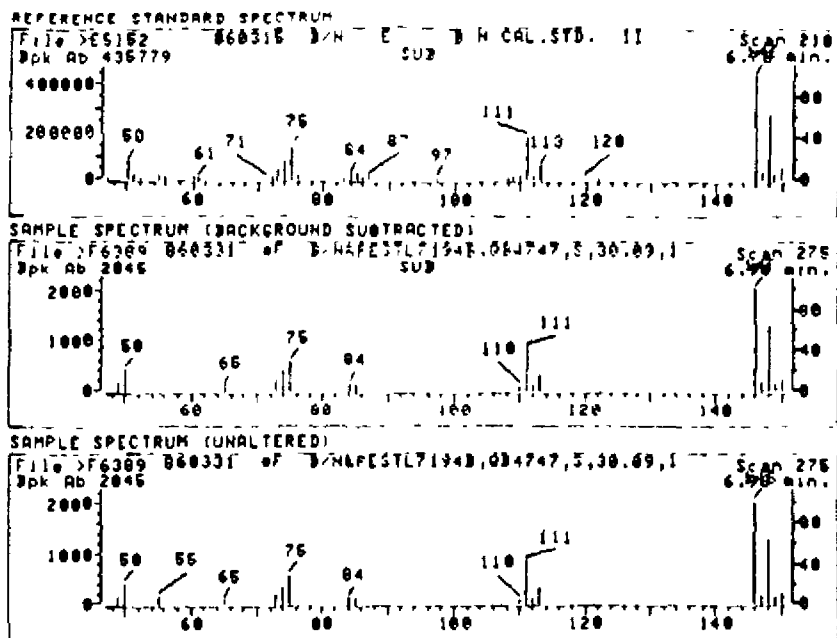
ID File: FBNP11US
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 Last Calibration: 860401 12:03

	Compound	R.T.	Score	Area	Conc	Units	q
1)	*d4-1,4-Dichlorobenzene	6.50	249	19112	40.00	UG/ML	97
2)	N-Nitrosodimethylamine	2.27	10	3043	8.28	UG/ML	100
7)	1,3-Dichlorobenzene	6.96	275	5071	7.69	UG/ML	96
8)	1,4-Dichlorobenzene	6.96	275	5071	7.69	UG/ML	96
9)	1,2-Dichlorobenzene	6.96	275	5071	7.98	UG/ML	95
12)	Nitrobenzene-D5 (SURR)	7.92	329	25944	37.90	UG/ML	87
18)	*d8-Naphthalene	9.76	433	84812	40.00	UG/ML	93
19)	2-Fluorobiphenyl (SURR)	13.13	623	63515	31.10	UG/ML	98
33)	*d10-Acenaphthalene	15.08	733	49813	40.00	UG/ML	96
39)	Dimethyl phthalate	14.30	689	61432	27.86	UG/ML	63
39)	Dimethyl phthalate	15.54	759	3990	1.76	UG/ML	60
41)	2,6-Dinitrotoluene	15.88	733	6658	11.79	UG/ML	68
41)	2,6-Dinitrotoluene	15.54	759	2480	4.25	UG/ML	46
45)	2,4-Dinitrotoluene	16.84	787	20652	24.74	UG/ML	55
46)	Diethyl phthalate	16.82	831	1236	.53	UG/ML	89
54)	*d10-Phenanthrene	19.59	987	145557	40.00	UG/ML	96
58)	Phenanthrene	19.66	991	2431	.73	UG/ML	96
59)	Anthracene	19.66	991	2431	.71	UG/ML	93
60)	Di-n-butyl phthalate	21.87	1116	7194	2.00	UG/ML	95
61)	Fluoranthene	23.36	1200	2953	1.63	UG/ML	85
63)	Pyrene	23.36	1200	2953	1.76	UG/ML	95
72)	*d12-Chrysene	27.84	1452	33477	40.00	UG/ML	100
84)	Terphenyl-D14 (SURR)	24.70	1275	49962	24.51	UG/ML	91
86)	Butyl benzyl phthalate	26.45	1374	1765	2.91	UG/ML	88
90)	bis(2-Ethylhexyl)phthalate	28.42	1485	6247	8.75	UG/ML	98

* Compound is ISTD

SLAMP
4-2-8

MONS 220971



Date File: >F6389::U2
 Name: 860331 #F B/N&PEST
 Misc: L7194B,QB4747,S,30.09,1
 Quant Time: 860401 19:32
 Injected at: 860401 18:44

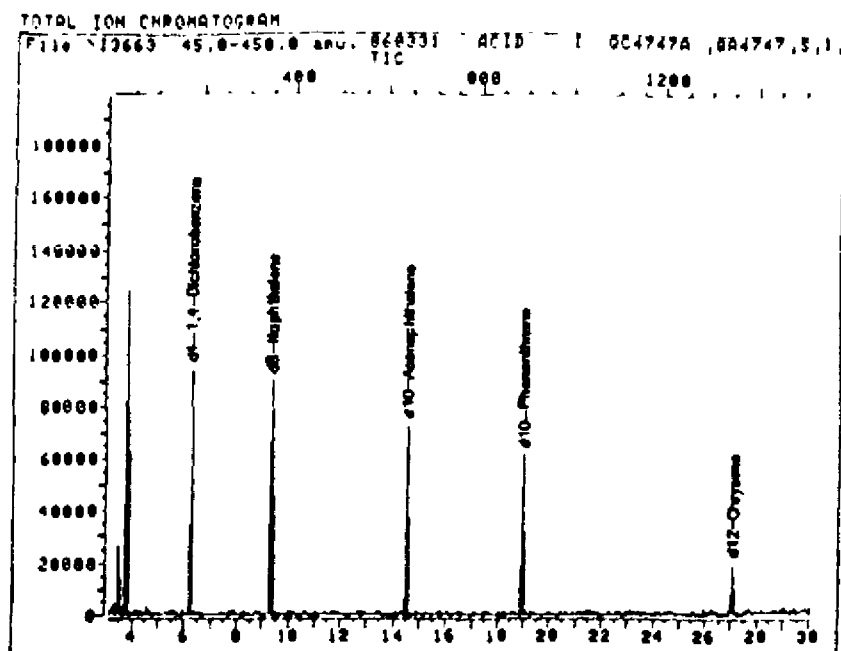
BTL423

Compound No: 9
 Compound Name: 1,2-Dichlorobenzene
 Scan Number: 275
 Retention Time: 6.96 min.
 Area: 5071
 Concentration: 7.98 UG/ML
 q-value: 95

MONS 220972

Appendix C1
GC/MS Subsidiary Data

MONS 220973



Date Filed: 13663:107
 Name: 860331 ACID 1
 Misc: QC4747A ,QA4747,S,1,1

FILE 8

Id File: 1ACID:1US
 Title: PP/ACID IDFILE
 Last Calibration: 860331 14:52

Operator ID: JH2180
 Quant Time: 860331 16:52
 Injected at: 860331 16:19

MONS 220974

QUANT REPORT

Operator ID: JH2180
 Output File: ^1366311AQ
 Data File: >1366311U7
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 Misc: QC4747A ,QA4747,S,1,1

Quant Rev: 5 Quant Time: 860331 16:52
 Injected at: 860331 16:19
 Dilution Factor: 1.00

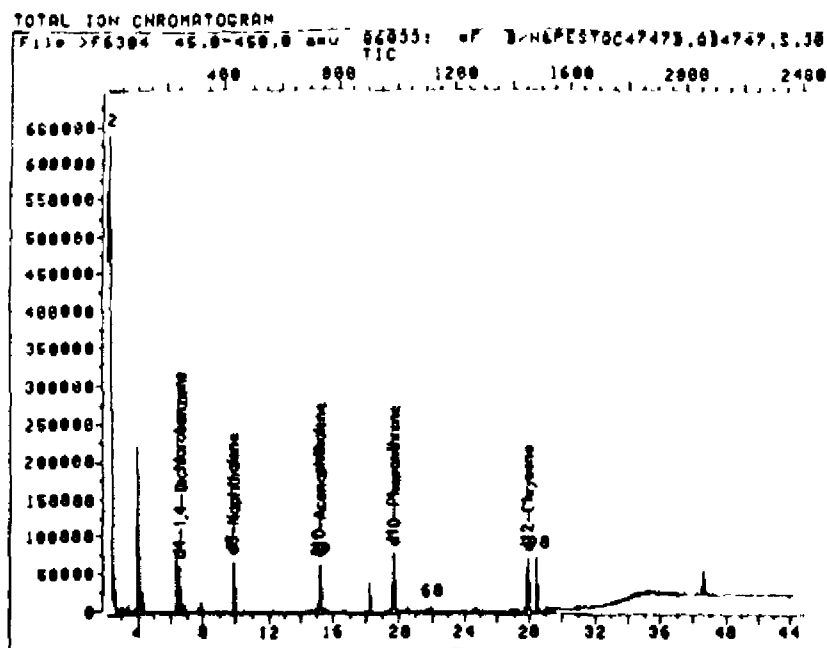
BTLE 8

ID File: IACID11US
 Title: PP/ACID 10FILE
 Last Calibration: 860331 14:52

Compound	R.T.	Scan#	Area	Conc	Units
1) *d4-1,4-Dichlorobenzene	6.17	175	39403	40.00	UG/ML
11) *d8-Naphthalene	9.26	349	90499	40.00	UG/ML
17) *d10-Acenaphthelene	14.46	642	34953	40.00	UG/ML
24) *d10-Phenanthrene	18.92	893	60065	40.00	UG/ML
28) *d12-Chrysene	27.02	1349	18515	40.00	UG/ML

* Compound is ISTD

MONS 220975



Date File: >F6384:1U2
 Name: 860331 #F B/N&PEST
 Misc: QC4747B,QB4747,S,30,1

BTL#18

Id File: FBNP:1US
 Title: B/N&PEST ID FILE FOR F 850326
 Last Calibration: 860401 02:06

Operator ID: KU0784
 Quent Time: 860401 09:56
 Injected at: 860401 09:08

MONS 220976

QUANT REPORT

Operator ID: KU0786
 Output File: ^F6384::AQ
 Data File: >F6384::U2
 Name: 860331 #F B/N&PEST
 Misc: QC4747B,QB4747,S,30,1

Quant Rev: 5 Quant Time: 860401 09:56
 Injected at: 860401 09:08
 Dilution Factor: 1.00

BTL#18

ID File: FBNP:US
 Title: B/N&PEST ID FILE FOR F 850326
 Last Calibration: 860401 02:06

Compound	R.T.	Scan#	Area	Conc	Units	q
1) *d4-1,4-Dichlorobenzene	6.53	249	24624	40.00	UG/ML	9
2) N-Nitrosodimethylemine	8.29	10	4420	10.46	UG/ML	10
18) *d8-Naphthelene	9.77	432	89791	40.00	UG/ML	9
33) *d10-Acenaphthelene	15.08	732	41534	40.00	UG/ML	9
39) Dimethyl phthalate	15.88	732	11917	6.30	UG/ML	6
54) *d10-Phenanthrene	19.58	986	119392	40.00	UG/ML	9
60) Di-n-butyl phthalate	21.89	1116	9574	3.24	UG/ML	9
72) *d12-Chrysene	27.82	1451	98321	40.00	UG/ML	10
90) bis(2-Ethylhexyl)phthalate	28.41	1484	53616	25.56	UG/ML	9

no PCBs found.
 • Compound is 1STD

Handwritten:
 4-7-86

MONS 220977

Appendix E

Chain-of Custody Forms

- 1) A field Chain-of-Custody form (CC1) is included for all samples shipped by ETC shuttle
- 2) An in-house sample Chain-of Custody form is included for all samples not shipped by ETC shuttle
- 3) Any additional Chain-of-Custody material provided by a client or by a client's sampling agent is also included.
- 4) A subcontractor's Chain-of-Custody form is included for any analytical work not performed within ETC's laboratory.
- 5) Analysis and Extraction Custody forms are included for the period the sample was in ETC's possession

MONS 220978

CHAIN OF CUSTODY

Company: Monsanto Job No. _____

Address _____

Attention: _____

Sample Description:

CUSTOMER ID	DESCRIPTION	ETC #
Sample # 5	2 - 500g (non Etc)	L7194
Sample # 57	↓	L7195

Sample(s) Relinquished by: Ram International

Time: 1:15 Date: 3/2/86

Sample(s) Received by: B. Basso

Time: 1:15 Date: 3/2/86

CHAIN OF CUSTODY RECORD

MRC JOB NUMBER 6405
 P.O. CONTRACT NUMBER RQ14054X
 PLANT NAME OR CODE _____

CUSTOMER REPRESENTATIVE _____
 MRC CONTRACT MANAGER Roy W. Noll
 MRC TEAM LEADER Roy W. Noll

(Specific information on samples is available on attached "Sample Identification Log")

Signatures		Date	Time	Remarks
Team Leader Verification of Collected Samples: <u>Roy W. Noll</u>		<u>3/14/86</u>	<u>1000</u>	
Relinquished by: <u>Roy W. Noll</u>	Received by: _____	<u>3/17/86</u>	<u>0900</u>	<u>Relinquished for shipment to ETC</u>
Relinquished by: _____	Received by: _____			
Logged-in-by: _____				
Received for Analysis by: _____				
Method of Shipment: <u>Air Express</u>		Number of Containers: <u>1</u>		
Special Instructions (Sample preservation, security, delivery to, etc.)				
<u>Sample #5 (2 bottles)</u>				
<u>Sample #57 (2 bottles)</u>				

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Sample Number	Log Link	Sample Weight (gm)	Extract Vol (ml)		Comments
			BN	ACID	
+ L4248	13976	30.50	1.0	1.0	PP/SP/ACID
+ L4249	↓	30.33	1.0	1.0	↓
L7194	14156	30.09	1.0	1.0	PP/ACID
L7195	↓	31.99	1.0	1.0	↓
★ L6314	14137	30.00	5.0	5.0	FOR/ACID FOR/ACID SPEC
L6315	↓	30.16	1.0	1.0	↓
★ L6321	↓	31.50	5.0	5.0	↓
★ L6322	↓	31.52	5.0	5.0	↓
QC 4747	—	—	1.0	1.0	
L4248	B	30.51	1.0	1.0	
L4249	A	31.13	1.0	1.0	

QC Batch # 4747

Analysis 3-27 (See comments)

Matrix Soil

Turnaround Normal

Date 3/26/96

Extraction Method

Rep Funnel —

Continuous —

Boxhiet ✓

Other —

COMMENTS

+ L4248/49 - Refer to QC 4720 - samples redone on this batch to label mixup.

Log 14137: SPERSP - These are soil samp

★ L6314, L6321, L6322 had bad bad emul in the BN Fraction Recovery may be affected.

FRACTION	SPIKE		
	Amt (ml)	Conc	Lot #
BN	1.0	100	12683
ACID	1.0	100	12340
Drac 1g 1260	1.0	100	12649
AST	1.0	100/200	12830
FOR/BN	1.0	100	12526
FOR/ACID	1.0	100	12522
Beni - VOA			

SURROGATE		
Amt (ml)	Conc	Lot #
1.0	BN: 50 ACID: 100	12846

Set-up: Pamela Williams 3/27/96
BN Conc.: 1/4 Acid Conc.: 1/4
ETC FORM 8110

UPD/Supervisor: Mary Gordon 3/31/96
Spike/Surr. Verified: Quinn Rembert 3/27/96

LABORATORY CHRONICLE: GC-MS Department

DATE 3/3/86 SHIFT
FRACTION Acid
INSTRUMENT F
TUNE FILE MIF601
SEQUENCE FILE JTHI, JTHI
METHOD FILE A-45
ID FILE IAJ
ANALYST(S) J. H. H.
SUPERVISOR [Signature]
BATCH # 00477

STANDARD	CONC PPM	LOT NO	LOT VOL
DETPP	25		
SUP STD	400		
Acid Cal Std I	60	13577	
Acid Cal Std II	100	12576	
Acid Cal Std III	200	12874	
FRP/acid I	60	12535	
FRP/acid II	100	12524	
FRP/acid III	200	12523	

PLEASE PRINT

CURRENT C506 STATUS		STANDARDS UPDATED	
ACO	3/3	DATE	3/3/1
WIP		BY	3/4/1

NAME	DATA FILE	HL INJ	ALS #	OIL	TAPE #	SPECIALS (WRITE A TYPE)	PLUS Y/N
DFTTP	2I3655	2	—		I00437		
Acid cal std III	I3656		1				
Acid cal std II	I3657		2				
Acid cal std I	I3658		3				
FOR/Acid I; 100ppm	I3659		4				
FOR/Acid II; 100ppm	I3660		5				
FOR/Acid III; 100ppm	I3661		6				
DFTTP	I3662		7				
QC4747A	I3663		8				SA Y
L7195A	I3664		9				
L4249A	I3665		10				SA Y
L4249AR	I3666		11				
L7194A	I3667		12				
L4249AS	I3668		13				
L4248A	I3669		14				SA Y
L6314A	I3670		15				
L6315A	I3671		16				
L6321A	I3672		17				
L6322A	I3673		18				

ETC / OAH #102

MONS 220982

ETC ENVIRONMENTAL
TESTING AND CERTIFICATION

LABORATORY CHRONICLE: GC-MS Department

F-1/2

DATE 4/1/86 SHIFT NIGHT
FRACTION B/N
INSTRUMENT F
TUNE FILE NTFC01
SEQUENCE FILE KV3
METHOD FILE 3NPF 12.10
ID FILE 3NPF 12.10
ANALYST(S) W. J. [unclear]
SUPERVISOR John [unclear]
BATCH # Q34723 (plate) 4.10 E)
Q34747

STANDARD	CONC PPM	LOT NO	LOT VOL
Pest. V	150	12851	
3 N D	250	12850	
2 N F	130	12845	
3 N E	60	12846	
Carbaryl	350	12837	
Carbaryl	100	12838	
Carbaryl	60	12839	
3 N F	25	12842	
12.10	450	12841	

(PLEASE INITIAL)

CURRENT C605 STATUS	STANDARDS UPDATED
ACC <u>WJ</u>	DATE <u>4/1/86</u>
WIP	BY <u>WJ</u>

NAME	DATA FILE	UL INJ	ALS S	DIL	TAPE #	SPECIALS (WRITE A TYPE)	PLUS Y/N
2FTPP - 50N6 SR	F6366				100437		
Carbaryl 300ppm	F6367		1				
Carbaryl 100ppm	F6368		2				
Carbaryl 60ppm	F6369		3				
B/N 12.10 100ppm	F6370		4				
B/N 12.10 60ppm	F6371		5				
B/N 12.10 30ppm	F6372		6				
B/N 12.10 15ppm	F6373		7				
2FTPP - 50N6 SR	F6374		8				
L 3281 B	F6375		9			Q34723 DUBIA	
L 6781 B	F6376		10			2812 1/10	
L 6317 B	F6377		11				
L 6318 B	F6378		12				
L 6220 B	F6379		13				
L 5813 B	F6380		14				
L 5109 B	F6381		15				
L 5110 B	F6382		16				
DCM	F6383		17				
Q 647473	F6384		18			TRACE	S3 Y
L 4249 B	F6385		19				S3 Y
L 4249 B	F6386		20				
2FTPP - 50N6 SR	F6387		21				
B/N 12.10 100ppm	F6388		22			AF 260	
L 7194 B	F6389		23				
L 7195 B	F6390		24				

ETC FORM 1103

MONS 220983

LABORATORY CHRONICLE: Metals Department

Samples L7194 , L7195

	Chemist	Date
Hg Prep	<u>Steven C. McPue</u>	<u>4/24/86</u>
AA/ICAP Prep	<u>Joan Komarek</u>	<u>4/22/86</u>

Lab Supervisor Lidia Wkianoy Date 4/25/86