

February 2, 1983

TECHNICAL REPORT

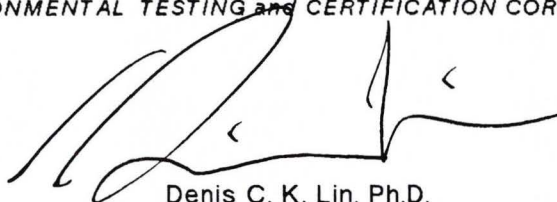
for

**E. I. DuPont
Seneca Works
7700 W. DuPont Rd.
Morris, IL 60450**

Chain of Custody Data Required for ETC Data Management Summary Reports

B7466	EI DuPont	SENECA WKS	BARKSDALE TNX				
ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours	

ENVIRONMENTAL TESTING and CERTIFICATION CORPORATION



Denis C. K. Lin, Ph.D.
Vice President
Research and Operations

ETC ENVIRONMENTAL TESTING and CERTIFICATION CORPORATION

DENIS C.K. LIN, Ph.D.

*Vice President,
Research and Operations*

February 2, 1983

Mr. Bruce Lawrence
E. I. DuPont
Seneca Works
7700 W. DuPont Rd.
Morris, IL 60450

Dear Mr. Lawrence:

We are pleased to submit the attached reports in response to your testing requirements. The data were acquired by my staff and we are confident that the results are of the highest quality.

If you have any questions regarding your report, we encourage you to contact Deb Holton, or Diane Foster in our Client Service organization (201/225-5600). They will coordinate your inquiries with the appropriate laboratory personnel. You are also invited to visit with Henry Beal, Esq. or Michael Bonchonsky, Esq., if you have any questions regarding the regulatory or the legal aspects of your project. Your account executives along with our Client Service organization are also available to assist you in defining the requirements for future testing programs.

If we can be of further service to your organization in the future, please contact us.

Sincerely,



Denis C. K. Lin, Ph.D.

DCKL:rp
Attachments

cc: T. M. Dearth, E. I. DuPont

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INTRODUCTION

This report contains the analytical results on your soil sample submitted on January 10, 1983. It is designed to satisfy the needs of your people at various levels in your organization.

The results we obtained on your sample are presented in a tabular format immediately after this introduction. Included with the sample results, the quality assurance data on your specific sample are tabulated to verify the validity of the results obtained. The quality assurance data include those obtained on the surrogates, the blank, the spiked blank, the replicate and the spiked sample (commonly known as matrix spike). Also presented in the quality assurance data report is the verification of the proper functioning of the instruments used. The gas chromatograms and/or mass spectra generated in the analysis of your sample are included in the Appendix of this report. The chain of custody record for your sample is included at the end of this report.

The established methods we used in the analysis of your sample are described in the Methodology section after the Results. In the analysis we followed a rigidly controlled Quality Assurance Protocol. This Protocol is described after the Methodology section.

We hope our report format is useful in assisting you to obtain pertinent information on your sample.

RESULTS

The results obtained on your sample and the accompanying quality data are listed in Table 1. The compounds of interest are listed with their NPDES (National Pollution Discharge Elimination System) number and Method Detection Limit (MDL) published in the Federal Register, December 3, 1979. When a compound is detected below its MDL it is reported in Table 1 as BMDL (Below Method Detection Limit). When a compound is searched for and cannot be found it is reported as ND (Not Detected). The quality assurance data contain results obtained on the Method Blank, Spiked Blank, Replicate, and Matrix Spike Analyses. However, since the Replicate and Matrix Spike analyses were performed on samples randomly chosen in a sample batch, your specific sample may not be the selected one.

The data on the recovery of the surrogates in your sample and the certification of the GC/MS systems used in the analysis of your sample are listed in Table 2.

The Chain-of-Custody Record on your sample is also included at the end of this Report.

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA
Base/Neutral Compounds - GC/MS Analysis Data (QR03)

Chain of Custody Data Required for ETC Data Management Summary Reports						
B7466	EI DuPont	SENECA WKS	BARKSDALE TNX			Elapsed
ETC Sample No.	Company	Facility	Sample Point	Date	Time	Hours

NPDES Number	Compound	Results		QC Replicate		QC Blank and Spiked Blank			QC Matrix Spike		
		Sample Concn. ug/kg	MDL ug/kg*	First ug/kg	Second ug/kg	Blank Data ug/ml	Concn. Added ug/ml	** % Recov	Unspiked Extract ug/ml	Concn. Added ug/ml	% Recov
1B	Acenaphthene	-	-	-	-	-	-	-	-	-	-
2B	Acenaphthylene	-	-	-	-	-	-	-	-	-	-
3B	Anthracene	-	-	-	-	-	-	-	-	-	-
4B	Benzidine	-	-	-	-	-	-	-	-	-	-
5B	Benzo(a)anthracene	-	-	-	-	-	-	-	-	-	-
6B	Benzo(a)pyrene	-	-	-	-	-	-	-	-	-	-
7B	Benzo(b)fluoranthene	-	-	-	-	-	-	-	-	-	-
8B	Benzo(ghi)perylene	-	-	-	-	-	-	-	-	-	-
9B	Benzo(k)fluoranthene	-	-	-	-	-	-	-	-	-	-
10B	bis(2-Chloroethoxy)methane	-	-	-	-	-	-	-	-	-	-
11B	bis(2-Chloroethyl) ether	-	-	-	-	-	-	-	-	-	-
12B	bis(2-Chloroisopropyl) ether	-	-	-	-	-	-	-	-	-	-
13B	bis(2-Ethylhexyl)phthalate	-	-	-	-	-	-	-	-	-	-
14B	4-Bromophenyl phenyl ether	-	-	-	-	-	-	-	-	-	-
15B	Butyl benzyl phthalate	-	-	-	-	-	-	-	-	-	-
16B	2-Chloronaphthalene	-	-	-	-	-	-	-	-	-	-
17B	4-Chlorophenyl phenyl ether	-	-	-	-	-	-	-	-	-	-
18B	Chrysene	-	-	-	-	-	-	-	-	-	-
19B	Dibenzo(a,h)anthracene	-	-	-	-	-	-	-	-	-	-
20B	1,2-Dichlorobenzene	-	-	-	-	-	-	-	-	-	-
21B	1,3-Dichlorobenzene	-	-	-	-	-	-	-	-	-	-
22B	1,4-Dichlorobenzene	-	-	-	-	-	-	-	-	-	-
23B	3,3'-Dichlorobenzidine	-	-	-	-	-	-	-	-	-	-
24B	Diethyl phthalate	-	-	-	-	-	-	-	-	-	-
25B	Dimethyl phthalate	-	-	-	-	-	-	-	-	-	-
26B	Di-n-butyl phthalate	-	-	-	-	-	-	-	-	-	-
27B	2,4-Dinitrotoluene	ND	3330	ND	ND	ND	0	-	ND	100	51
28B	2,6-Dinitrotoluene	-	-	-	-	-	-	-	-	-	-
29B	Di-n-octyl phthalate	-	-	-	-	-	-	-	-	-	-
30B	1,2-Diphenylhydrazine	-	-	-	-	-	-	-	-	-	-
31B	Fluoranthene	-	-	-	-	-	-	-	-	-	-

* ETC established Method Detection Limit for this particular sample.

** Reagent Blank. Spiked Blank cannot be performed for this sample.

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA
Base/Neutral Compounds - GC/MS Analysis Data (QR03)

Chain of Custody Data Required for ETC Data Management Summary Reports						
B7466	EI DuPont	SENECA WKS BARKSDALE TNX				Elapsed
ETC Sample No.	Company	Facility	Sample Point	Date	Time	Hours

NPDES Number	Compound	Results		QC Replicate		QC Blank and Spiked Blank			QC Matrix Spike		
		Sample Concn. ug/kg	MDL ug/kg*	First ug/kg	Second ug/kg	Blank Data ug/ml	Concn. Added ug/ml	** % Recov	Unspiked Extract ug/ml	Concn. Added ug/ml	% Recov
32B	Fluorene	-	-	-	-	-	-	-	-	-	-
33B	Hexachlorobenzene	-	-	-	-	-	-	-	-	-	-
34B	Hexachlorobutadiene	-	-	-	-	-	-	-	-	-	-
35B	Hexachlorocyclopentadiene	-	-	-	-	-	-	-	-	-	-
36B	Hexachloroethane	-	-	-	-	-	-	-	-	-	-
37B	Indeno(1,2,3-c,d)pyrene	-	-	-	-	-	-	-	-	-	-
38B	Isophorone	-	-	-	-	-	-	-	-	-	-
39B	Naphthalene	-	-	-	-	-	-	-	-	-	-
40B	Nitrobenzene	ND	3330	ND	ND	ND	0	-	ND	100	84
41B	N-Nitrosodimethylamine	-	-	-	-	-	-	-	-	-	-
42B	N-Nitrosodi-n-propylamine	-	-	-	-	-	-	-	-	-	-
43B	N-Nitrosodiphenylamine	-	-	-	-	-	-	-	-	-	-
44B	Phenanthrene	-	-	-	-	-	-	-	-	-	-
45B	Pyrene	-	-	-	-	-	-	-	-	-	-
46B	1,2,4-Trichlorobenzene	-	-	-	-	-	-	-	-	-	-
	2,3,7,8-TCDD	-	-	-	-	-	-	-	-	-	-
	2-Nitrotoluene	ND	3330	ND	ND	ND	0	-	ND	100	67
	2,4,6-Trinitrotoluene	ND	3330	ND	ND	ND	0	-	ND	500	7

* ETC established Method Detection Limit for this particular sample.

** Reagent Blank. Spiked Blank cannot be performed for this sample.

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

Chain of Custody Data Required for ETC Data Management Summary Reports

B7466

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
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Compound	Amount Added ug	% Recovery	Control Limits *	
			Lower	Upper
<i>VOLATILE FRACTION</i>				
Bromochloromethane	-	-	79	127
Benzene, d ₆	-	-	63	122
Fluorobenzene	-	-	74	122
1,4-Dichlorobutane	-	-	75	117
Pentafluorobenzene	-	-	58	124
Ethylbenzene, d ₁₀	-	-	78	114
<i>ACID FRACTION</i>				
2-Fluorophenol	-	-	20	86
Pentafluorophenol	-	-	37	127
<i>BASE/NEUTRAL FRACTION</i>				
2-Fluorobiphenyl	80	85	62	122
1-Fluoronaphthalene	80	74	64	104
Nitrobenzene, d ₅	80	65	58	105

* Three Standard Deviations About the Mean.

TABLE 2: METHOD PERFORMANCE DATA

GC/MS Tuning Data – Decafluorotriphenylphospine (DFTPP) for Base/Neutrals Analysis (QR23)

Chain of Custody Data Required for ETC Data Management Summary Reports

B7466

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
----------------	---------	----------	--------------	------	------	---------------

m/z	Ion Abundance Criteria	Abundance (% Base Peak)
51	30-60% of mass 198	58
68	Less than 2% of mass 69	<2
70	Less than 2% of mass 69	<2
127	40-60% of mass 198	40
197	Less than 1% of mass 198	<1
198	Base peak, 100% relative abundance	100
199	5-9% of mass 198	5
275	10-30% of mass 198	25
365	Greater than 1% of mass 198	2
441	Less than mass 443	14
442	Greater than 40% of mass 198	77
443	17-23% of mass 442	15

Date: 012783
Run No: 7610
Spectrum No: 123
Analyst: K. Weiner

METHODOLOGY

The methods employed in the analysis of your sample are established EPA methods for priority pollutants. Combined gas chromatography and mass spectrometry (GC/MS) was used in the analysis of the organic compounds.

For the analysis of the Base/Neutral priority pollutants, EPA Method 625 (Federal Register, December 3, 1979; page 69540) was used. The method can be summarized as follows: A 30 gram semi-wet soil sample is soxhlet extracted with 1:1 acetone and hexane. The acetone is thermally stripped. The remaining extract is diluted to 200 ml with methylene chloride and extracted twice with NaOH solution and once with reagent free water. The methylene chloride fraction is dried, concentrated, and analyzed for base neutral compounds by Method 625.

Qualitative identification of the priority pollutants was performed initially using the relative retention times, the relative abundance of three characteristic ions and their ratios. The entire mass spectrum was reviewed before an identification was recorded. Quantitative analysis was performed using an internal standard with a single characteristic ion.

QUALITY ASSURANCE PROTOCOL

The quality assurance protocol followed in the analysis of your sample is based on the "Handbook for Analytical 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; and the recommended guidelines for EPA Method 625.

Our protocol calls for a higher percentage of quality assurance samples than the requirements of Method 625. The key QA elements for the analysis of priority pollutants are summarized as follows:

Method 625

- In every block of 20 samples extracted, there are 16 field samples, one blank, one spiked blank, one sample spiked with the priority pollutant standard mixture and a duplicate field sample.
- Five surrogate compounds are added to each sample in the block of 20.
- Blind quality control samples are included in field samples at a minimum of one every hundred samples.
- The GC/MS is checked and retuned, if necessary, every eight hours to ensure its performance on decafluorotriphenylphosphine (DFTPP) meets the EPA criteria.
- GC performance criteria as specified in Method 625 are met before analysis starts.
- A calibration curve for quantification is prepared using a minimum of 3 concentrations of a standard mixture of the priority pollutants of interest and 2,2'-difluorobiphenyl as internal standard.
- The calibration curve is verified with a standard priority pollutant mixture every eight hours.
- Results meet the acceptance criteria given in Method 625.

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 seal, taking the sample, resealing the Shuttle and transferring custody to a shipper;
- incoming shipping manifests;
- breaking the Shuttle's reseal;
- 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 record shows for each link in this process:

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

REPORT APPENDICES

The following appendices provide the support analytical data associated with your sample analyses. They are arranged as follows:

Appendix A

- 1) Reconstructed total ion chromatogram of GC/MS analyses of your sample.
- 2) The individual mass spectra of all priority pollutant compounds which have been identified as being present in your sample.
- 3) The individual mass spectra of the corresponding compounds as obtained from the standards.

Appendix B

- 1) The mass spectra of the calibration compounds, 4-bromofluorobenzene (BFB) and decafluorotriphenylphosphine (DFTPP), as obtained on the date of sample analyses.

Appendix C [for plus 10 and 50% internal standard option]

- 1) Spectra of tentatively identified compounds, with their differences.

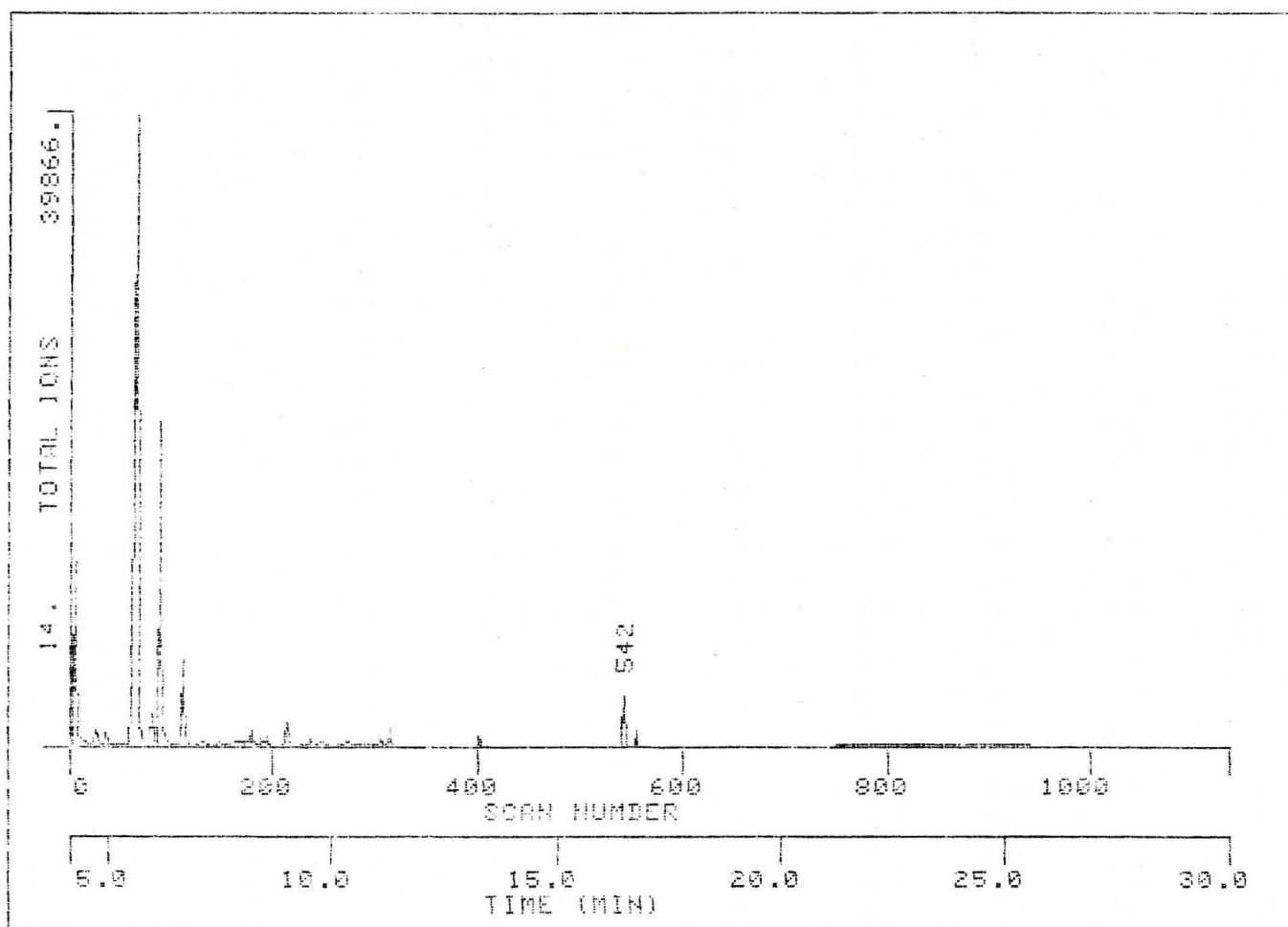
Appendix D

- 1) Subcontractor's report.

These data are provided to present a complete report on your sample and to provide the data on which analytical decisions have been based. We hope this will aid you in your own analysis of the data.

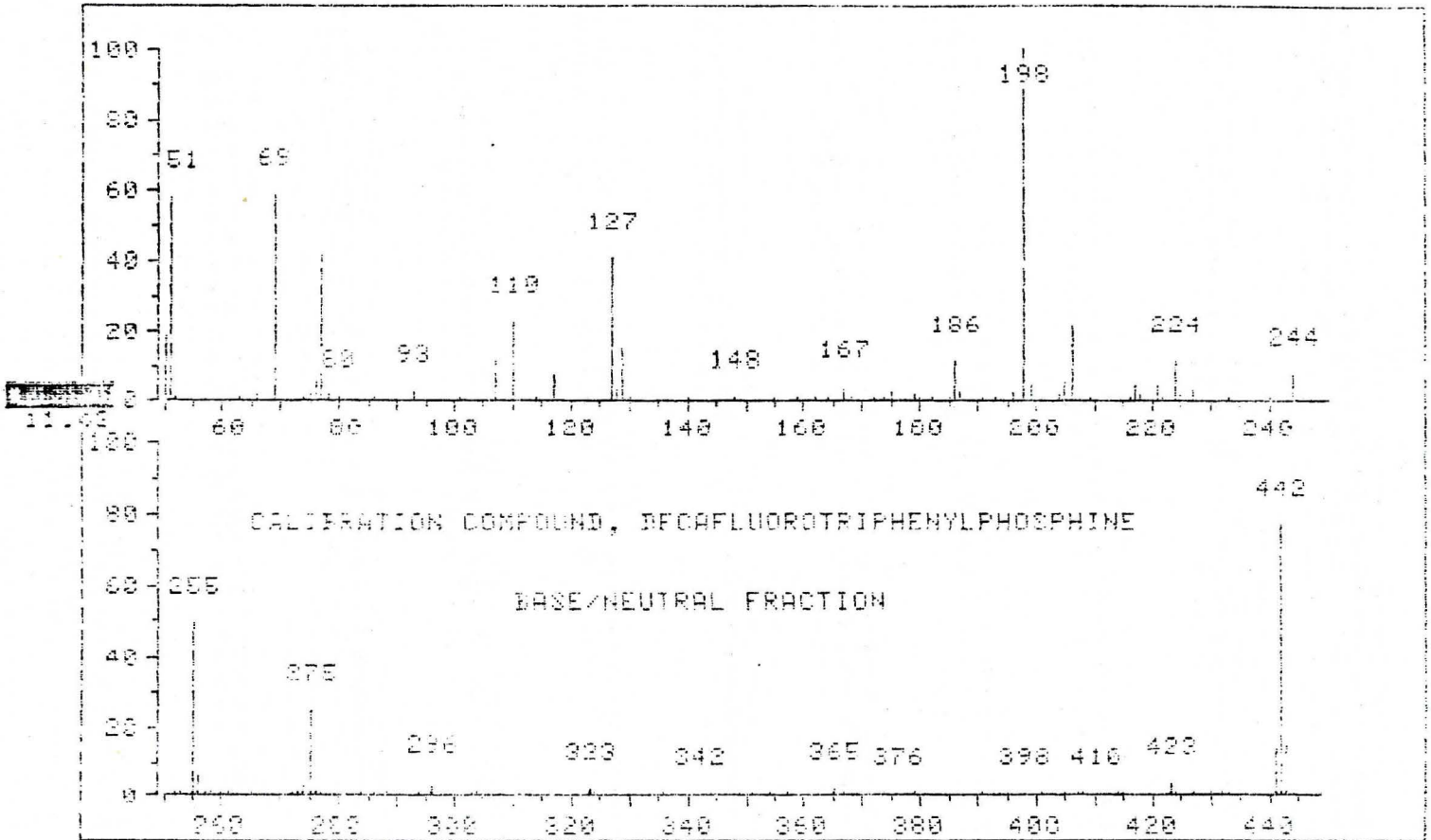
APPENDIX A

TOTAL ION CHROMATOGRAM



MS Data File FRN: 7641
Name: B7466B, B/N FRACTION, 830127, F, DIL 10X
Misc Data: B7466B ETL#30 D7641
Last R.T.: 30.0 minutes

APPENDIX B



DFTRP EPA CRITERION VERIFIED

DPN: 7611
SPECTRUM: 1

Mass	Rel. Abund.	Criterion
51	57.975	30-60% MASS 198
68	0	< 2% MASS 69
69	53.6906	
70	0	< 2% MASS 69
127	40.4705	40-60% MASS 198
197	0	< 1% MASS 198
198	100	BASE PEAK
199	5.725	5-9% MASS 198
275	24.7456	10-30% MASS 198
345	2.35313	> 1% MASS 198
441	13.7	< MASS 443
442	77.0969	> 40% MASS 198
443	15.0312	17-23% MASS 442

NOTE: '**' indicates out of range!

PRESS (RETURN) TO RETURN...

CHAIN-OF-CUSTODY RECORD

CHAIN OF CUSTODY

Company: E. I. DUPONT Job No. _____

Address _____

Attention: _____

Sample Description:

MCD at room temp. approx 100gr of soil
sample in plastic bag.

<u>Cust</u>	<u>ETC#</u>
Barkdale TNX area composite	B7466

Sample(s) Relinquished by: En D

Time: 4⁰⁰p Date: 1/10/83

Sample(s) Received by: J. Capozzi

Time: 4⁰⁰p Date: 1/10/83

LABORATORY CHAIN-OF-CUSTODY

ETC Sample Number(s) 37466

Relinquished By J. Papozzy

Received By Ben [Signature] Date 11/11/83 Time 10:24am

Relinquished By _____

Received By _____ Date _____ Time _____

Relinquished By _____

Received By _____ Date _____ Time _____

Relinquished By _____

Received By _____ Date _____ Time _____

Relinquished By _____

Received By _____ Date _____ Time _____

LABORATORY CHAIN-OF-CUSTODY CHRONICLE

ETC Sample Number(s) B7466

Sample Preparation For:	Analyst	Date
<u>Base/Neutral/PCB's & Pesticides</u>	<u>Lidya Whiawer</u>	<u>1/14/83</u>
Acids	_____	_____
Metals	_____	_____
Others _____	_____	_____
Others _____	_____	_____
Others _____	_____	_____
Others _____	_____	_____

Sample Analysis For:	Analyst	Date
<u>Base/Neutral/PCB's & Pesticides</u>	<u>Don Wainer / Harold H. Speer</u>	<u>1/27/83</u>
Acids	_____	_____
VOA/Purgeables	_____	_____
Metals	_____	_____
Others _____	_____	_____
Others _____	_____	_____
Others _____	_____	_____
Others _____	_____	_____
Others _____	_____	_____
Others _____	_____	_____

Verified By Bob Anderson