

✓
CORR-860398

387107

FINAL REPORT
ON THE SAMPLING AND ANALYSIS OF SEDIMENT CORES
FROM THE L-AREA OIL AND CHEMICAL BASIN

8



Research Planning Institute, Inc.

Prepared For:

E.I. Du Pont de Nemours Company, Inc.
Savannah River Plant
Aiken, South Carolina 29802

**FINAL REPORT
ON THE SAMPLING AND ANALYSIS OF SEDIMENT CORES
FROM THE L-AREA OIL AND CHEMICAL BASIN**

Prepared By:

Research Planning Institute, Inc.
925 Gervais Street
Columbia, South Carolina 29201

RPI/R/85/8/5-7

August 1985

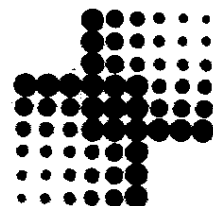


TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION	i
VIBRACORE AND SAMPLING	1
SAMPLE PREPARATION AND LABORATORY PROCEDURES	5
On-Site Procedures	5
Preparation of Samples for Off-Site Analysis	6
DETERMINATION OF RADIOACTIVE NUCLIDES BY GAMMA-RAY SPECTROSCOPY IN THE FIELD LABORATORY	13
APPENDIX I - SAMPLE LOG	
APPENDIX II - SUBSAMPLE FIELD NOTES	
APPENDIX III - SEDIMENT CHARACTERIZATION	
APPENDIX IV - FIELD LABORATORY NOTES FOR PERCENT MOISTURE SUBSAMPLE CALCUALTIONS	
APPENDIX V - FIELD LABORATORY NOTES FOR DETERMINATION OF SUBSAMPLE WET WEIGHTS	
APPENDIX VI - FIELD LABORATORY NOTES FOR CALCULATIONS OF SUB-SAMPLE DRY WEIGHTS	
APPENDIX VII - FIELD LABORATORY NOTES FOR PREPARATION OF DRIED SUBSAMPLES	
APPENDIX VIII - FATHOMETER TRACES	
APPENDIX IX - LAB NOTES/COPIES OF GC CHROMATOGRAMS	
APPENDIX X - STANDARD OPERATING PROCEDURE, LAB NOTES AND COMMENTS, FOR GREASE AND OIL EXTRACTION; AND GENERAL ANALYSIS COMMENTS	
APPENDIX XI - ANALYTICAL RESULTS "LABORATORY AND FIELD"	

INTRODUCTION

Nine vibracores were collected in the L-Area oil and chemical basin (904-83G) during late March and early April 1985. These cores were collected for analysis of the sludge on the basin floor and the underlying sediment. Several different field and laboratory analyses were performed on each three inch segment of all the cores. These included: 1) Sediment characterization; 2) Percent moisture; 3) Dry weight; 4) Spectral gamma analysis; 5) Gross alpha and beta analysis. Detailed chemical analysis were measured on selected intervals of 2 cores (LBC-5 and 6) for complete chemical characterization of the sediments. This sampling program was conducted to provide information so that a closure plan for the basin could be developed.

This report describes the methods employed during the project and provide a hard copy of the analytical results from the sample analyses. Included in the appendices are copies of all field and laboratory notes taken during the project and copies of the gas chromatograms for the petroleum hydrocarbon analysis. All chemical results were also submitted on a 5-inch floppy disk.

VIBRACORING AND CORE SAMPLING

Nine vibracores were taken in the L-Area oil and chemical basin (904-83G) in March and April 1985. Figure 1 shows both the location of the cores and the total depth of a given boring. This figure also shows the location of several fathometer traces run across the basin.

Prior to coring activities at the study site the perimeter of the basin was surveyed (Fig. 1). The elevation difference between the berm top and the water surface in the basin was also surveyed. The elevation difference between the concrete pad at test well 904-83G and the water level in the basin was 2.48 meters on 20 March 1985. The depth of water in the basin is shown on the fathometer traces. The fathometer transducer was positioned one foot beneath the boat and from the traces (Appendix VIII) it was determined that the basin had a maximum of 4 feet of water.

After the initial surveys were completed, the vibracoring of the basin floor was initiated. The vibracorer is a small, portable, gasoline-operated coring device. A strong vibration is developed by a cam-loaded shaft driven by a gas engine and is then transmitted to the core barrel. A three inch-diameter aluminum core barrel was used. To collect the samples, the vibracoring was conducted from a modified john boat (Fig. 2). When penetration of sediments was difficult for the standard vibracore, such as in de-watered silt and clay, a combination vibracorer/piston corer technique was employed.

A 2 inch-diameter thin gauged steel core barrel was employed when the vibracorer/piston core technique was used. This vibracorer/piston core technique was only used for borings LBC-2 and LBC-6 (Fig. 1). Using this technique, the initial boring made with the 3 inch-diameter core barrel was removed and the hole was re-entered with the 2 inch-diameter core barrel and sampling was continued. The core samples were pulled from the basin floor utilizing a large tri-pod and com-a-long.

Immediately after extraction of the core the core barrel was cut into 3-inch sections (0-3", 3-6"), etc. Each 3-inch sub-section of core barrel was split and the sample was removed intact and placed either in plastic containers (for sludge samples) or plastic bags (for clayey sediments). The samples were scanned on-site by personnel from Health Protection for radioactivity levels, labeled, and transported to a field laboratory. Cross-contamination of samples in the field was prevented by using new

L-AREA OIL AND CHEMICAL BASIN

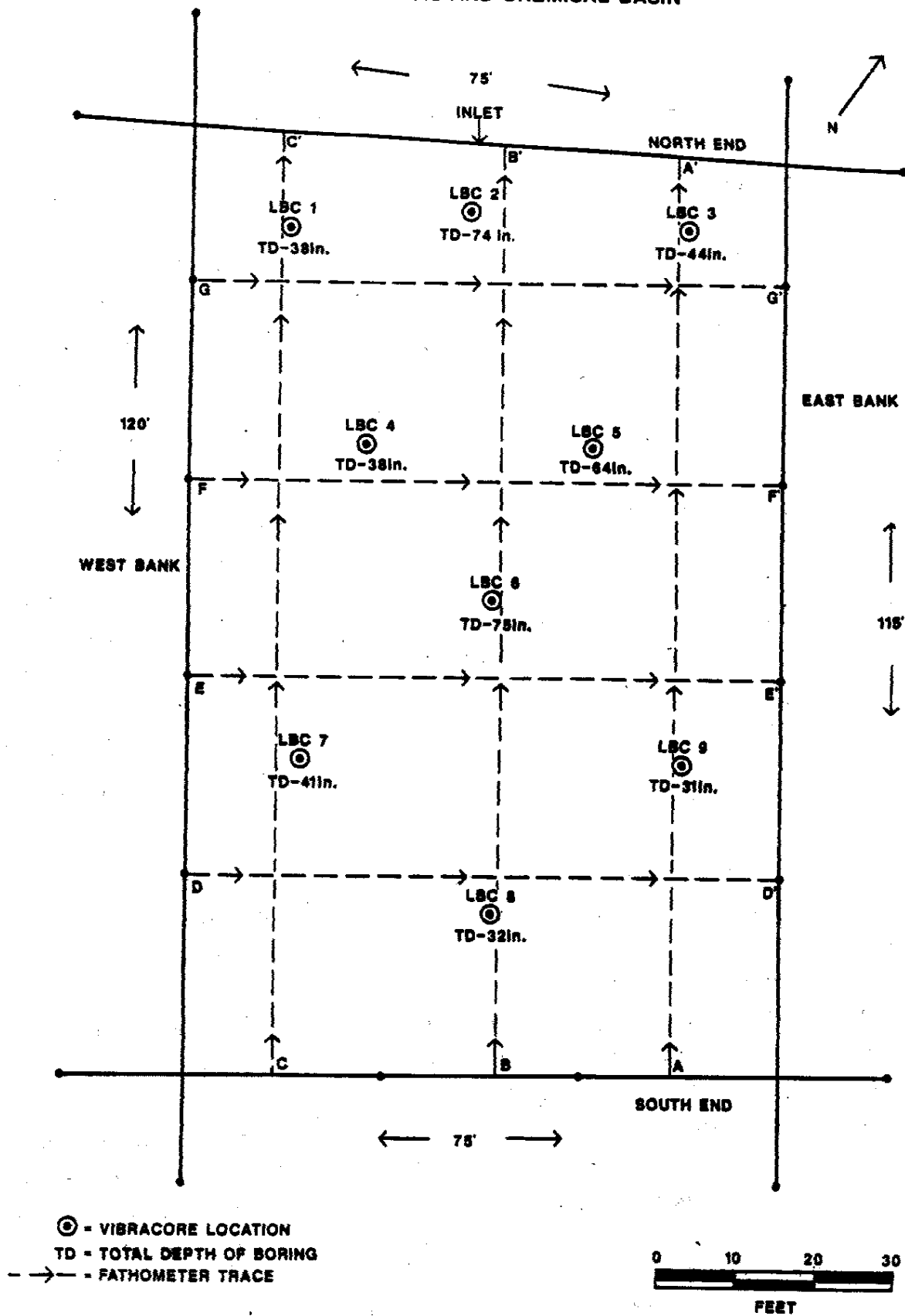


FIGURE 1. Location map of vibracores and fathometer traces.

BOAT SPECIFICATIONS
MAKE: SEA NYMPH-TRAVELER
MODEL #: JV-1544
CAPACITY: 450 POUNDS
MATERIALS: 72 GAUGE ALUMINUM
LENGTH: 15 FT.
WIDTH: 62 IN.
FREEBOARD HEIGHT: 20 IN.

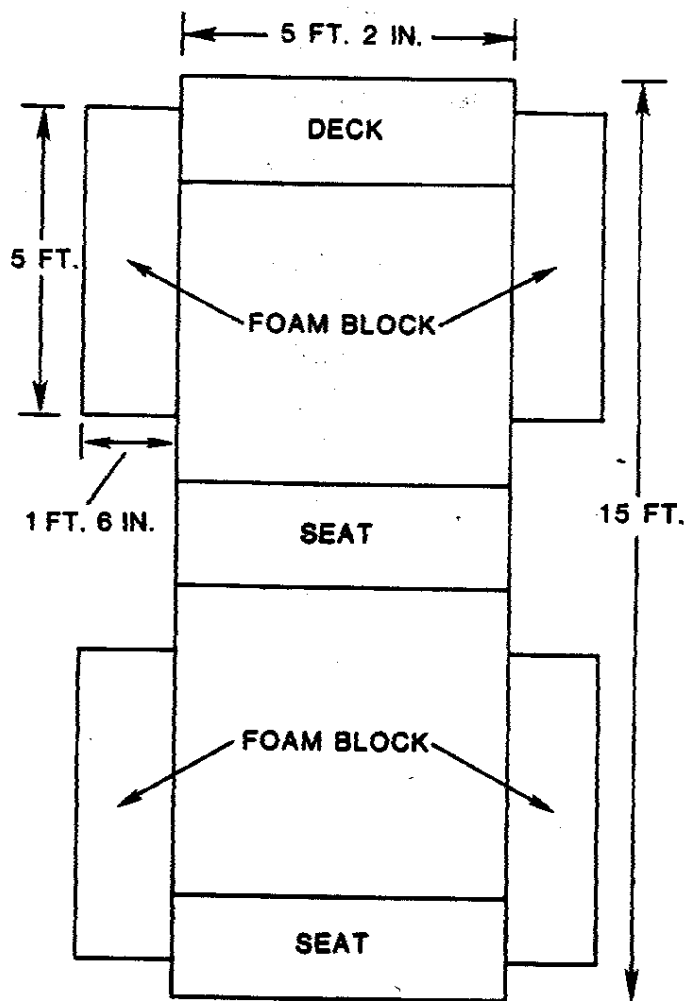


Figure 2. Drawing of the boat used while coring the L-Area oil and chemical basin. Foam blocks were cut and attached to the sides of the boat to increase the stability while coring.

cores barrels for each core. There was no means of contamination in this manner. A technician was on-site during sampling and was responsible for sample handling and labeling after they were taken. A field log was maintained during sample splitting (Appendix II), in which the date and time of core extraction and the field measurements for gross radioactivity were recorded.

The depth of each core was as follows:

<u>Core</u>	<u>Depth (Inches)</u>
LBC-1	38
LBC-2	77
LBC-3	44
LBC-4	38
LCB-5	64
LBC-6	75
LBC-7	42
LBC-8	32
LBC-9	31

SAMPLE PREPARATION AND LABORATORY PROCEDURES

ON-SITE PROCEDURES

At the field laboratory in the 700 area, the samples were logged in by sample ID and date in the lab notebook (Appendix I). A total of 133 subsamples were prepared in the field laboratory for analysis of percent moisture and more detailed on-site radioactivity measurements. All intervals of cores 2-9 were prepared and analyzed on-site; only interval 9-15 and 33-38 inches of LBC-1 were prepared and analyzed on-site.

The sludge samples were homogenized within the sample container by stirring and subsamples for on-site measurements were removed from the mixture. Samples of the underlying clayey sediments (usually beginning at or around the 9-12" interval) were scraped on all sides to remove any possible contamination from the coring process. Representative subsamples of each interval were collected in vertical slices through the core. During the sample preparation, a detailed visual sedimentological description was made of grain-size distribution, grain shape, sediment color, bedding and general physical characteristics. The subsamples were split into two unequal fractions: 1) a fraction of 50-100 grams (g) wet weight for spectral gamma activity measurement, and 2) a fraction of 1-10 g wet weight for determination of percent moisture and measurement of gross alpha and beta activity.

Vials for counting of the field spectral gamma activity were prelabeled and tared, with the weight recorded in the lab notebook. The sample was placed inside the vials and a pestal was used to pack the samples into a constant geometry and height of 4 centimeters (cm). The vials were reweighed, the weight recorded, and the total wet weight of the sample calculated (Appendix V).

The second split was weighed in a tared aluminum dish, dried overnight at 50°C and reweighed to determine percent moisture (Appendices IV and VII). The entire dried sample was then pulverized and an approximately 1 g sample was weighed out and placed in a counting planchet. These samples were transported to the on-site counting laboratory set up by E. Lang of the Geology Department of the University of South Carolina.

All remaining sample fractions were stored wet, in their original containers in the laboratory refrigerator until those to be analyzed further were selected.

Core LBC-6 was selected for detailed chemical characterization by an off-site laboratory (Envirodyne Engineers). Samples selected for detailed analysis included every interval from 0-43 inches, then every other interval from 48-75 inches. The original analytical plan called for triplicate analyses of all samples. However, for some intervals, there was inadequate sample quantity to perform triplicate analysis. For the sludge samples, there was not enough sample quantity to measure the entire suite of parameters to the specified detection levels. Therefore, selected intervals of core LBC-5 were used to supplement sample needed for certain parameters. Intervals 0-3 and 3-6 inches from LBC-5 were used for petroleum hydrocarbon and oil and grease measurements. Intervals 6-9 and 9-14 inches from LBC-5 were used for all inorganic, TOC, and COD measurements. Single analyses of all parameters were performed on LBC-6 intervals 48-51, 54-57, 60-63, 66-69, and 72-75 inches. Duplicate analyses were performed on LBC-6 intervals 12-15 and 15-18 inches. Therefore, various parameters were determined on a total of 23 samples.

PREPARATION OF SAMPLES FOR OFF-SITE ANALYSIS

Prior to shipment to the off-site laboratory selected sediment samples were processed in a portable laboratory provided by Du Pont. Sample intervals from the upper 15" of core holes LBC-5 and LBC-6 consisted of a dark oily sludge and were limited in solid material. Due to their extremely wet nature, it was decided to proceed these sample intervals in the controlled conditions of the off-site laboratory to minimize sample loss during processing. Those sample intervals forwarded for processing included LBC-5 (0"-3", 3"-6", 6"-9", 9"-14") and LBC #6 (0"-3", 3"-6", 6"-9", 9"-12", 12"-15", 15"-18"). Sample intervals processed onsite in the portable laboratory included LBC-6 (18"-21", 21"-24", 24"-27", 27"-30", 30"-33", 33"-36", 36"-39", 39"-43", 48"-51", 54"-57", 60"-63", 66"-69", 72"-75").

Sediments sealed in the plastic vials (from the field spectral gamma analysis) were dedicated for the petroleum derivative and EP Toxicity analyses. Samples contained in the plastic dishes were placed in labeled polyethylene trays and set out for air drying. The air drying of sediment samples was necessary to facilitate analytical procedures in the off-site laboratory. Drying time varied according to interstitial sample moisture and environmental conditions within the laboratory but averaged approximately 4-5

days. When full dried to the touch, samples were ground into a fine powder by use of a mortar and pestle. This pulverization process was accomplished under a hood to prevent the contamination of lab facilities or personnel. The resultant powder was then sieved through a number twenty mesh stainless steel screen. Residual soil clumps retained in the sieve were reground and resieved to ensure maximum sample retrieval. The ground and sieved sediments were sealed in appropriately labeled 250 ml amber glass jars and stored in a freezer until shipment off-site. The glass sample jars and plastic vials were prepared for shipment by first wrapping them in bubble-pack and then sealing them in zip-lock plastic bags. Preservation of samples during shipping off-site was accomplished by using Coleman coolers and blue ice. All transfers of sample custody to Du Pont prior to shipment off-site were documented by chain of custody records.

Quality assurance program for field laboratory off-site shipping included:

- A) Use of sample tracking log to track samples from date of processing to date of transfer of custody.
- B) Thorough cleaning of processing equipment with alconox, acetone and distilled water rinses between samples to prevent cross contamination.
- C) Use of fresh gloves between samples to prevent cross contamination.
- D) Use of proper labeling of sample containers (project number, sampling site, etc.) to ensure proper sample identification.
- E) Preservation of wet and processed samples at or below four degrees Centigrade prior to shipment off-site.
- F) Sample containers securely wrapped in bubble pack and zip-lock plastic bags to prevent breakage or spillage during shipment.
- G) Use of chain of custody sheets to document all transfers of custody.

The following tests and methods were performed:

<u>Test</u>	<u>Method</u>
As	Hydride, EPA 206.3
Se	Hydride, EPA 270.3
Hg	Manual Cold Vapor, EPA 245.1
Sb	Flameless AA, EPA 204.2
U, Pb, Cr, Zn	
Be, Ni, Ba, Cd	Plasma, EPA 200.7
Ag	Flameless AA, EPA 272.2
TOC	EPA 415.2
COD	EPA 410.1
CN	EPA 335.2
SiO ₂	NaOH Fusion*
Al ₂ O ₃	NaOH Fusion*
Fe ₂ O ₃	NaOH Fusion*
MgO	Plasma, EPA 200.7
CaO	Plasma, EPA 200.7
Na ₂ O	Plasma, EPA 200.7
K ₂ O	Flame AA, EPA 258.1
TiO ₂	Plasma, EPA 200.7
P ₂ O ₅	EPA 365.1
MnO	Flame AA, EPA 243.1

In addition to the basin samples, NBS standard soils or NBS traceable check standards were analysed. Aqueous NBS standards were used for EP Toxicity. The results are shown in Table 1. There were several parameters which showed large differences between the reported "true" values and the measured values, with antimony (Sb) and uranium of greatest concern. The digestion method used for Sb was selected because of the limited sample size and was not the optimum procedure, which required a larger sample. The standard was analyzed in the same size and manner as the samples. The recovery rates are poor, ranging from 5-17 percent, but it was not possible to use these results to make corrections to the analytical results because of matrix differences. Therefore, the absolute values for Sb are questionable but the trend clearly shows decreases to relatively low levels.

TABLE 1. Results of analyses for NBS standards or NBS traceable check standard.

EP Toxicity Metals Standards									
	As	Ba	Cd	Cr	Pb	Hg	Se	Ag	
Standard Value (mg/l)	8.0	0.5	3.0	30.0	30.0	1.5	8.0	10.0	
Measurement	1 5.0 2 4.0 3	0.53 0.545	3.1 4.5	23.0 32.0	32.0 28.0	1.56 1.83 1.65	5.0 4.0	9.3 9.0	
Check Standard Value	5.0 ppb	0.25 ppm	0.25 ppm	0.25 ppm	0.25 ppm		3.0 ppb	2.0 ppb	
Measurement	1 4.54 2 5.17 3 5.24 4	0.239 0.233 0.238 0.270	0.252 0.263 0.246	0.221 0.214	0.221 0.244 0.250		3.15 2.94	1.93 2.02	6
Standard True Value (µg/g)	51.0	66.0	0.025	10.2	29,600				
Measurement	1 7.23 2 8.46 3 2.69 4		57.0 52.1	0.021 0.021 0.22 0.021	5.03 14.84 14.81		27,335 27,185 27,217		

TABLE 1. Continued.

	Pb	Hg	Ni	Se*	Ag
Standard True Value (µg/g)	714.0	1.1	45.8	3.0	**
Measurement					
1	647.5	0.75	37.36	4.036	1.59
2	642.0	0.95	37.88	2.773	1.65
3	599.8	0.82	37.31	2.936	1.87
4		0.86		4.266	1.70
	U	Zn	TOC	COD	Cn
Standard True Value (µg/g)	1.11	1,720	2,000	100	50
Measurement					
1	19.6	1,741	2,008	112	48
2	18.4	1,753	2,016	112	50
3		1,421	2,016	98	35
					48
	Al	Fe	Mg	Ca	Na
Standard True Value (µg/g)	22,600	113,000	7,400	29,000	5,400
Measurement					
1	3,529	77,238	6,058	26,445	978.0
2	3,560	78,223	6,004	26,860	989.5
3	5,705	80,975	6,500	26,845	
4	6,358	80,662	6,489	26,948	

TABLE 1. Continued.

	K	Tj*	P*	Mn	Si*
Standard True Value (µg/g)	1,260	0.250	0.6	785.0	
Measurement					
1	627.9	0.214	0.601	638.7	
2	630.2	0.212	0.606	634.9	
3	629.6	0.226	0.601	640.6	
4	633.9	0.230	0.601	636.6	

* - NBS standard not available; NBS traceable check standards where possible.

** - True value not known.

The performance for U with NBS standard was also particularly poor and of concern. These results are for a large sample (5g) concentrated to 5 ml which was a second run to improve the detection levels with the plasma method. With these large sample volumes, aluminum and iron positively interfere with no way to make background corrections. The initial runs, which had a detection limit of 20 ppm, did not have this problem of background corrections.

DETERMINATION OF RADIOACTIVE NUCLIDES BY GAMMA-RAY SPECTROSCOPY IN THE FIELD LABORATORY

This method was used to measure gamma ray emitting nuclides present in all intervals of each core. The expected precision for a single determination varied with the nuclide composition of the sample. The range of sensitivity for this technique varied from 0.001 to 50 microcuries (μCi) per sample.

An aliquot of the sample was transferred to a polyethylene 40 dram round vial and the radionuclides present were determined using a lithium-drifted, germanium detector and a computer-based, multi-channel analyzer. The radionuclides present in the sample were determined by gamma-ray spectroscopic techniques.

The apparatus used included:

- 1) Counting Vials - Polyethylene - 40 dram round with lids.
- 2) Gamma Ray Detector - Ge(Li) semiconductor detector with associated electronics.
- 3) Multichannel Analyzer - Computer-based Nuclear Data 76 system with associated peripherals. This system has built-in peak extraction and peak identification programs.

The following calibration procedure was used:

- 1) A clean 40 dram round filled with water to the 4 cm mark was placed directly on the Ge(Li) detector, which is protected by a piece of mylar film. The background spectrum was recorded.
- 2) The Ge(Li) detector was calibrated using NBS and secondary standards in the sample configuration. The standard sample was used to generate a calibration curve for the source position of the detector system.
- 3) The calibration was checked to assure that the results were within the control limits for the system.

Each sample was counted for 20 minutes. All calculations were performed by the computer using an extraction and peak identification procedure. The background was determined to be unimportant in all regions of interest. The results of the peak identification analyses ($\mu\text{Ci}/\text{sample}$) were divided by the dry weight (Appendix VI) of the sample.

The following equation was used to calculate the activity for each nuclide detected:

$$\text{Activity} = \frac{A}{E \times R \times 2.22 \times 10^6 \times T \times W}$$

where Activity = gamma activity of the sample for each particular nuclide in $\mu\text{Ci}/\text{gram}$.

A = Area under the gamma-ray peak known to belong to the nuclide in total counts corrected for background.

E = Efficiency of the detector at that energy in counts/disintegration (c/d).

R = Fraction of decays leading to that particular gamma-ray for that nuclide in gammas/disintegration.

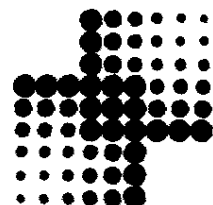
T = Counting time in minutes.

2.2×10^6 = Factor to convert disintegration per minute to μCi .

W = Weight of sample in grams.

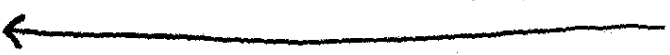
Normal propagation-of-error techniques were used to determine the 2 sigma error associated with each measurement.

APPENDIX I
SAMPLE LOG



Brought into Lab
3-27-85 11:15am

Sample #	Date Pulled	Date % mois	Date counted	Date charc.	Dated Dried	Date Processed	Date Packed	Date shipped
LBC 1								
0'-0.25'	3-27-85	3-28-85	3-29-85	3-28-85	3-29-85	3-29-85		
3-6"								
6-9								
9-15								
15-18								
18-21								
21-24								
24-27								
30-33								
33-38"		3-27-85	3-27-85	3-27-85	3-29-85	3-29-85		





Brought into lab
4-1-85 4:00pm

Sample no	Date Pulled	Date % mois	Date counted	Date charc.	Date Duad	Date Processed	Date Packed	Date Shipped
-----------	-------------	-------------	--------------	-------------	-----------	----------------	-------------	--------------

LBC 2

0-3"

3-6"

6-9

9-11

11-17

17-20

20-23

23-26

26-29

29-32

32-35

35-38

38-41

41-44

44-47

47-50

50-53

53-56

56-59

59-62

62-65

65-68

68-71

71-74

4-1-85

4-1-85

4-2-85

4-1-85

4-2-85

4-2-85

4-2-85

4-2-85

4-3-85

4-3-85

→

↑

↑

↑

↑

→

→

→

→

→

Brought into lab
3-26-85 3:30 PM

Sample #	Date Pulled	Date 86 mois	Date collected	Date charc.	Date Dried	Date Processed	Date packed	Date shipped
LBC 3	3-26-85	3-28-85	3-28-85	3-28-85	3-29-85	3-20-85		
D-3 "								
3-6								
6-9								
9-11								
11-15								
15-18								
18-21								
21-24								
24-27								
27-30								
30-33								
33-36								
36-39								
39-41								
41-44								

Brought into lab
3-26-85 3:30 pm

Sample #

Date pulled

Date o/o mois.

Date counted

Date charc.

Date Orid

Date processed

Date packed

Date shipped

LBC 4

3-26-85

3-27-85

3-27-85

3-27-85

3-28-85

3-28-85

0-3
3-6
6-9
9-15
15-18
18-21
21-24
24-27
27-30
30-33
33-38

←

←

←

←

←

←



Brought into lab
3-26-85 3:30pm

Sample #

Date
processed

Date
mailed

Date
copied

Date
charc.

Date
dried

Date
processed

Date
packed

Date
shipped

LBC 5

3-26-85

3-27-85

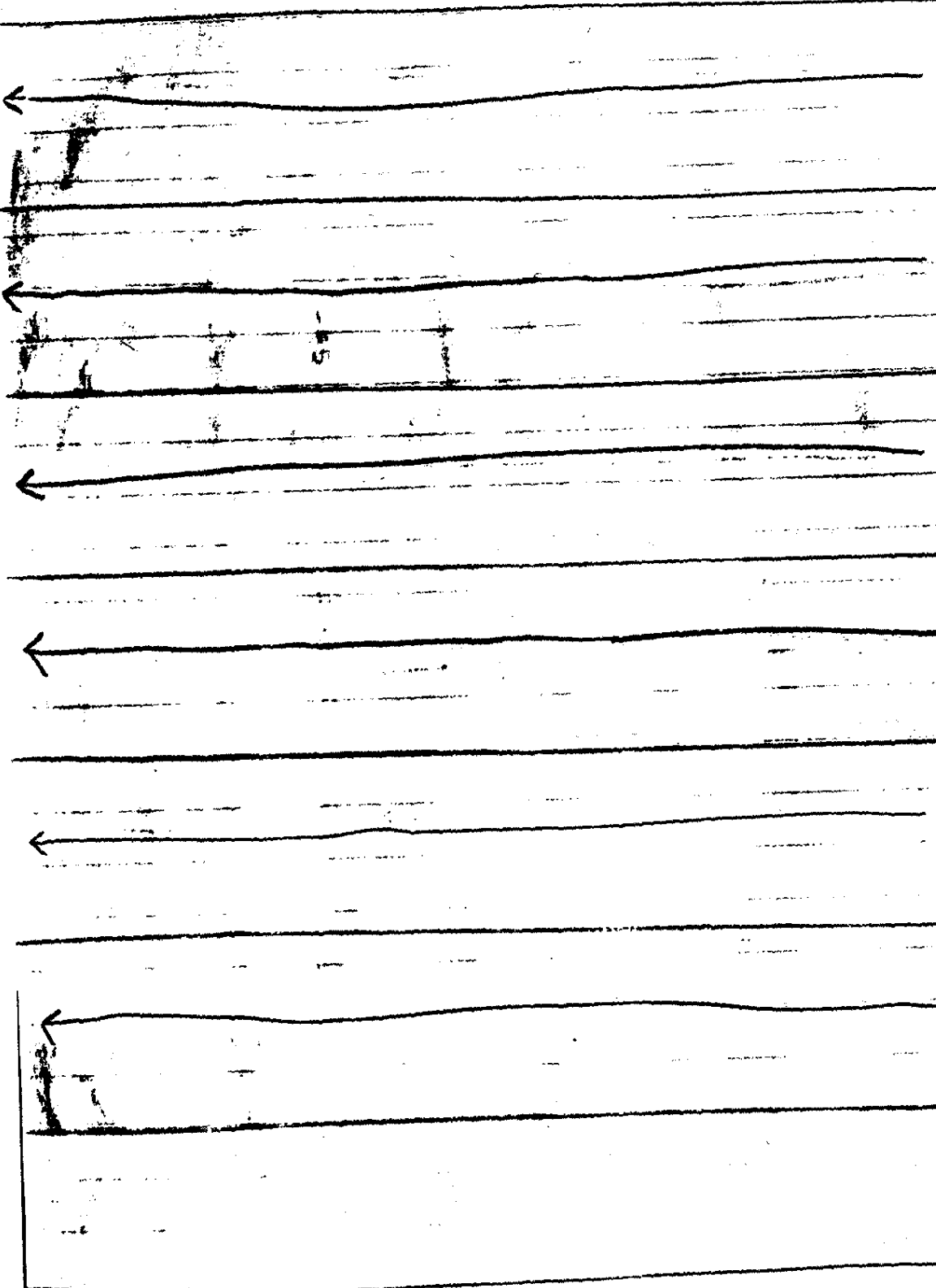
3-28-85

3-27-85

3-28-85

3-28-85

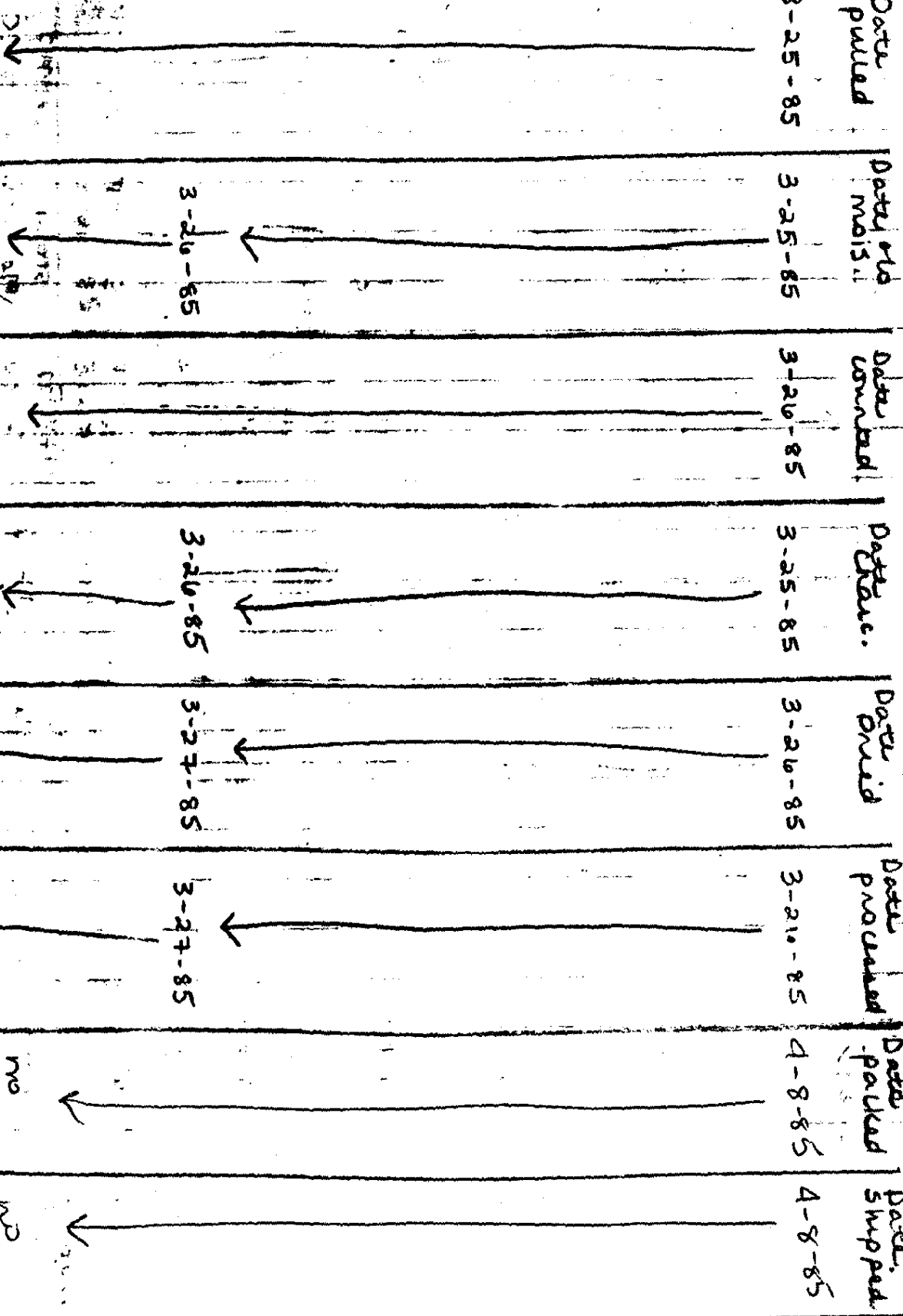
- 0-3
- 3-6
- 6-9
- 9-14
- 14-17
- 17-20
- 20-23
- 23-26
- 26-29
- 29-32
- 32-35
- 35-38
- 38-41
- 41-44
- 44-47
- 47-50
- 50-53
- 53-56
- 56-59



Brought into lab
3-25-85 3pm

LBC 6

Sample #	Date pulled	Date into mois.	Date counted	Date offered	Date drived	Date processed	Date packed	Date shipped
LBC 6	3-25-85	3-25-85	3-26-85	3-25-85	3-26-85	3-27-85	4-8-85	4-8-85
0-3"								
3-6"								
6-9"								
9-12"								
12-15"								
15-18"								
18-21"								
21-24"								
24-27"								
27-30"								
30-33"								
33-36"								
36-39"								
39-43"								



Brought into lab
3-25-85 3pm

UBC 7

Sample #	Date pulled	Date of mo's	Date received	Date chow	Date fed (1/2 min)	Date processed	Date packed	Date shipped
-BC 7								
01-0.25	3-25-85	3-25-85	3-26-85	3-25-85	3-27-85	3-27-85		
3-6"								
6-10"								
10-12"								
12-15"								
15-18"		3-26-85		3-26-85	3-28-85	3-28-85		
18-21"								
21-24"								
24-27"								
27-30"								
30-33"								
33-36"		3-27-85	3-27-85	3-27-85				
36-39"								
39-42"								

LBCL

Brought

into lab

4-2-85

3:45pm

Sample #

Date pulled

Date of mois.

Date counted

Date chare.

Date (mo) died

Date produced

Date packed

Date shipped

42-45

4-2-85

4-2-85

4-3-85

4-2-85

4-3-85

4-4-85

4-8-85

4-8-85

45-48"

48-51

51-54

54-57

57-60

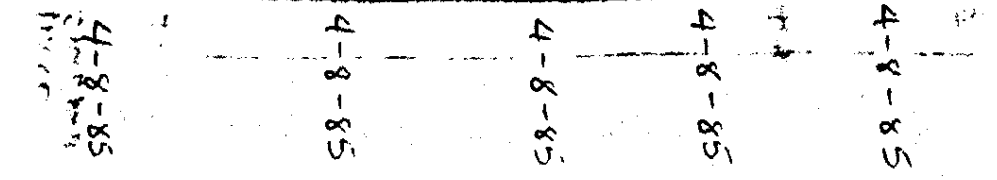
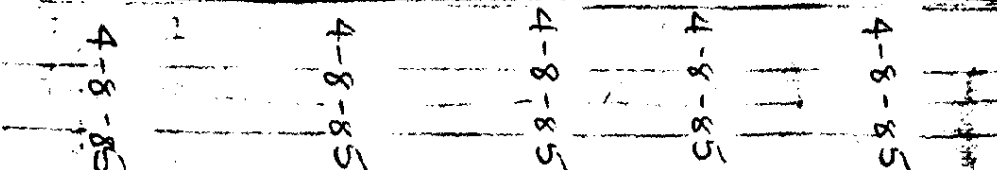
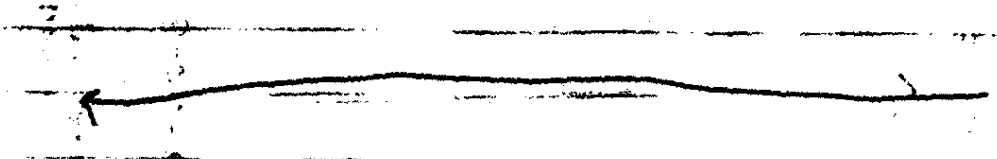
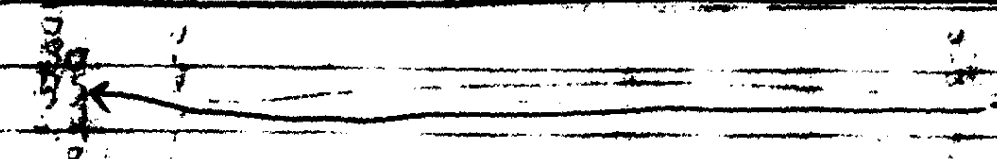
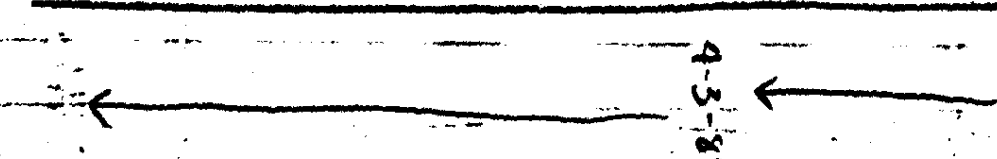
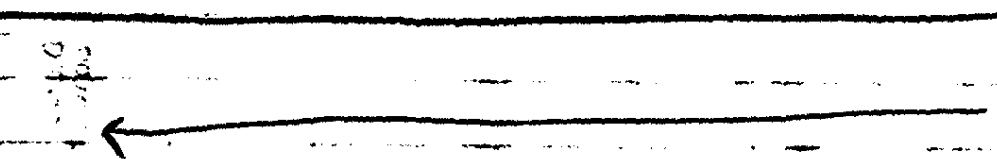
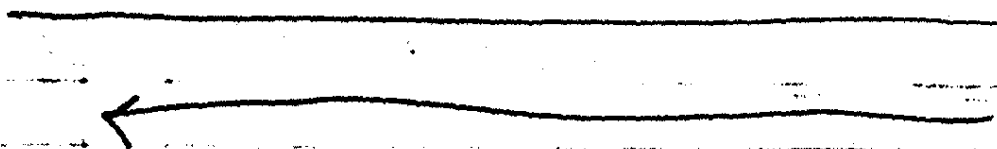
60-63

63-66

66-69

69-72

72-75



LBC 8

Brought into lab
10:15 pm (3-20-85)

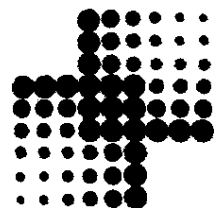
Sample #	Date pulled	Date olo mois.	Date counted	Date Charl.	Date (mois) Bred	Date Processed	Date packed	Date shipped
LBC 8								
0"-3"	3-20-85	3-21-85	3-21-85	3-22-85	3-22-85	3-22-85		
3"-8"			3-21-85	3-22-85				
8"-11"			3-21-85	3-22-85				
11"-14"			3-21-85	3-22-85				
14"-17"				3-22-85				
17"-20"				3-22-85				
20"-23"				3-22-85				
23"-26"				3-21-85				
26"-29"				3-21-85				
29"-32"				3-21-85				

Brought into lab
at 2:15 pm (3-21-85)

LBC-9

Sample #	Date punched	Date to mois.	Date counted	Date chase.	Date (mois) Driver	Date processed	Date packed	Date shipped
LBC 9								
0"-3"	3-21-85	3-21-85	3-22-85	3-21-85	3-22-85	3-22-85		
3"-6"	3-21-85	3-21-85		3-21-85				
8-12"	3-21-85			3-21-85				
12-15"	3-21-85			3-21-85				
15-18"	3-21-85			3-21-85				
18-21"	3-21-85			3-21-85				
21-24"				3-22-85				
24-27"				3-22-85				
27-31"				3-22-85				

APPENDIX II
SUBSAMPLE FIELD NOTES



Sub-Sample Field Notes

Field notes

pulled 3-26-85

2:30pm

LBC 3

0-3" sludge 300 c/m BS < 100

3-6" sludge 400 c/m

6-9" sludge 1000 c/m

9-11" sludge 2300 c/m
3000 c/m

11-15" sludge ends at 13"
1800 c/m

15-18" Red clay - little sludge

18-21" Red clay 400 c/m

21-24" Red clay 400 c/m

24-27" Red clay 250 c/m

27-30" Red clay 200 c/m

30-33" Red clay 200 c/m

33-36" Red clay 200 c/m

36-39" Red clay 300 c/m

39-44" Red/yellow clay 200 c/m

LBC 2

0-3" sludge 600 c/m

3-27-85
9:30am

3-6" sludge 800 c/m

6-9" sludge 1000 c/m

9-15" Hot spot. sludge to 12" 16,000 c/m

15-18" clay 1500 c/m

18-21" red clay 350 c/m

21-24" red clay 500 c/m

24-27" red clay 300 c/m

27-30" red clay 200 c/m

30-33" red clay 200 c/m

33-38" red clay 200 c/m

Field notes

pulled 4-1-85

2pm

LBC-2

0-3"	Sludge	200 c/m	2100 B.S.
3-6"	sludge	200 c/m	
6-9"	sludge	1000 c/m	
9-11"	sludge	1500 c/m	
11-13"	sludge ^{to 14"}	Hot spot. 3500 c/m	
17-20"	Red clay	300 c/m	
20-22"	Red clay	200 c/m	
23-26"	Red + grey clay	200 c/m	
26-29"	Red + grey clay	200 c/m	
29-32"	Red clay	200 c/m	
32-35"	Red clay	200 c/m	
35-38"	Red + grey clay	200 c/m	
38-41"	Red + grey clay	100 c/m	
41-44"	grey clay	200 c/m	
44-47.5"	Red, y + g. clay	200 c/m	
47-50"	Red + grey	100 c/m	
50-53"	grey clay	100 c/m	
53-56"	red clay	100 c/m	
56-59"	Red + y. clay	100 c/m	
59-62"	Red, y + grey clay	100 c/m	
62-65"	Red + grey clay	200 c/m	
65-68"	Red clay	200 c/m	
68-71"	Red clay	200 c/m	
71-74"	Red clay	200 c/m	

field notes — pulled 3-20-85. noon

LBC 4 at 0-3" sludge 500 c/m 4100 Br

3-6" sludge 1500 c/m

6-9" sludge 4000 c/m
4000 c/m

9-15" sludge only 10" sludge - rest clay

9-15" (b) Clay 700 c/m

also 32 ns 15-18" Clay-red + grey 400 c/m

18-21" red s.c. 200 c/m

21-24" red s.c. 300 c/m

24-27" red s.c. 200 c/m

27-30" At 29" turns to grey s.c. 200 c/m

30-33" grey + white s.c. 150 c/m

33-38" red s.c. 150 c/m

LBC 5 0-3" sludge 250 c/m 4180 Br 47-50" red s.c. 400 c/m

pulled 3-26-85 1pm 3-6" sludge 700 c/m 50-53" red s.c. 300 c/m

6-9" sludge 2000 c/m 53-56" dense red clay 250 c/m

9-12" sludge 2000 c/m 56-59" red clay 200 c/m

14-17" sludge 1400 c/m 59-64" red + yellow clay 200 c/m

17-20" sludge 1200 c/m

20-23" sludge 500 c/m

23-26" sludge 400 c/m
sludge 800 c/m
1/2 clay

26-29" At 28" turns from sludge to clay

29-32" sludge/clay 1500 c/m

32-35" At 35" hit sludge again 1200 c/m

35-38" sludge pocket 1000 c/m

38-41" s. clay 400 c/m

41-44" grey s.c. 300 c/m

44-47" red s.c. 250 c/m

Field notes

LBC 42" →

pulled 4-2-85

42-45: red clay 100 c/m

45-48: red clay 100 c/m

48-51: red clay 100 c/m

51-54: red clay 100 c/m

54-57: red clay 100 c/m

57-60: red clay 100 c/m

60-63: red clay 100 c/m

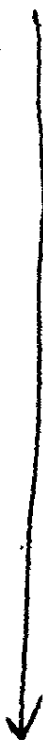
63-66: red clay 100 c/m

66-69: red clay 100 c/m

69-72: red clay 100 c/m

72-75: red clay 100 c/m

2100 B8



field note pulled
3-25-85 1pm

LBC 6 0-3" 2900 cpm B.S. sludge BKg 8
3-6" 800 cpm sludge
6-9" 2500 cpm sludge
9-12" 3000 cpm sludge
12-15" 200 cpm s.c. red
15-18" 1800 cpm little bit of red sl. / sludge
18-21" sandy 500 cpm
21-24" 300 cpm red s.c.
24-27" At 27" a thin sludge layer - 400 cpm + red color
27-30" natural fractures in sample w/ seepages - brown sludge
30-33" 300 cpm br + grey color.
33-36" 200 cpm Red, s.c. yellow
36-39" 200 cpm Red, s.c. upward sweeping beds
39-43" 150 cpm Red, yellow, grey color. s.c.

3-25-85 1:30 pm

LBC 7 0-3" 250 cpm sludge 4100 8.
3-6" 1200 cpm sludge
6-10" 3500 cpm sludge
10"-12" 800 cpm Brown sludge
12-15" 600 cpm contact zone
15-18" 400 cpm brown
18-21" 350 cpm brown
21-24" 300 cpm grey + red
24-27" 250 cpm grey, yellow + red color
27-30" 200 cpm red
30-33"
33-36"
36-39" red 39-42 red

LBC 8

pulled 3-20-85

Field notes

300 ct/min

A 0'-3"

Black sludge

B 3'-8"

Hot spot

5000 c/min

C 3'-8"

1000 cpm

D 8"-11"

Black sludge 1/2 contact zone

700 ct/min

E 11"-14"

Blue + red clay

500 cpm

* make 2 vials full blue, 1 red

F 14"-17"

dense clay

200 cpm

G 17"-20"

bluish, red dense clay

By 0

* make 3 vials - 1 red, 1 y, 1 blue

H 20"-23"

mostly grey clay

250 cpm

I 23"-26"

dense clay

< B 0

J 26"-29"

dense clay

< B 0

K 29"-32"

y, w, r clay

< B 0

into law 6:15 pm 3-20-85

LBC 9

pulled 3-21-55 field note

0-3' clay

< 100 BR 100 c/m

3-6' silty clay

< 100 BR 1000 c/m

9 sections

6-12' clay

< 100

★ Ho spot

12-15'

dense clay < 100 BR 300 c/m

15-18'

dense clay < 100 BR 300 c/m

18-21'

dense clay < 100 BR 300 c/m

21-24'

dense clay < 100 BR 300 c/m

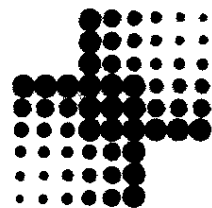
24-27'

" " < 100 BR 300 c/m

27-31'

" " < 100 BR 300 c/m

APPENDIX III
SEDIMENT CHARACTERIZATION



SEDIMENT CHARACTERIZATION

GRAIN SIZE SCALE

(sediment sizes in millimeters, after Folk 1974)

Clay - Less than 0.004 mm

Silt - Between 0.004 mm and 0.06 mm

Sand - Very Fine - Between 0.06 mm and 0.1 mm
Fine - Between 0.1 mm and 0.25 mm
Medium - Between 0.25 mm and 0.5 mm
Coarse - Between 0.5 mm and 1.0 mm

SEDIMENT CHARACTERIZATION

LBC 1

9-15"

Hot spot - sludge to 12" then clayey sand.
Sludge - liquid, dark colored, round grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
silt		(20%)
sand	very fine	(30%)
	fine	(10%)
	medium	(5%)

33-38"

Dense, compact, gray and red in color, sandy clay, does not crumble easily.

<u>Grain Size</u>		<u>Distribution</u>	
clay		(50%)	
sand	very fine	(10%)	Subangular sand grains, quartz rich
	fine	(10%)	
	medium	(20%)	
	coarse	(10%)	

LBC 2

0-3"

Sludge. Very little clay, mostly liquid, dark-colored round grains.

<u>Grain Size</u>		<u>Distribution</u>	
clay		(10%)	Sand-silt, clay-mixture
silt		(40%)	
sand	very fine	(30%)	
	fine	(20%)	

3-6"

Sludge. Liquid, dark-colored round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(25%)
sand	very fine	(30%)
	fine	(10%)
	medium	(5%)

LBC 2

6-9" Sludge. Liquid, dark-colored round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(25%)
sand	very fine	(30%)
	fine	(10%)
	medium	(5%)

9-11" Sludge. Liquid, dark-colored round grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
silt		(20%)
sand	very fine	(30%)
	fine	(10%)
	medium	(5%)

11-17" Sludge to 14". Clayey sand to 17". Hot spot, clear-sub-rounded sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
silt		(15%)
sand	very fine	(25%)
	fine	(15%)
	medium	(10%)

17-20" Clayey sand $\frac{1}{2}$ red, $\frac{1}{2}$ gray. Clear-sub-rounded sand grains.

<u>Grain Size</u>		<u>Distribution</u>	
clay		(45%)	Crumbles easily, abundant quartz.
sand	very fine	(10%)	
	fine	(15%)	
	medium	(30%)	

20-23" Clayey sand $\frac{1}{2}$ red, $\frac{1}{2}$ gray. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>	
clay		(45%)	Crumbles easily
sand	very fine	(5%)	
	fine	(15%)	
	medium	(30%)	
	coarse	(5%)	

LBC 2

23-26"

Predominately gray (w/red) clayey sand (in middle of core, a section of 85 percent clay).

<u>Grain Size</u>		<u>Distribution</u>	
clay sand		(45%)	Clear, sub-angular sand grains
	very fine	(5%)	
	fine	(15%)	
	medium	(10%)	
	coarse	(10%)	

26-29"

Predominately gray (w/red) clayey sand in middle, small section of dense compact clay.

<u>Grain Size</u>		<u>Distribution</u>	
clay sand		(45%)	Clear, sub-angular sand, does not crumble easily
	very fine	(5%)	
	fine	(15%)	
	medium	(25%)	
	coarse	(10%)	

29-32"

Clayey sand $\frac{1}{2}$ red, $\frac{1}{2}$ gray, very well-sorted, clear sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>	
clay sand		(45%)	Red sediments are a little bit clay- ier than gray sedi- ments.
	very fine	(5%)	
	fine	(15%)	
	medium	(25%)	
	coarse	(10%)	

32-35"

Gray and red clayey sand. Gray sediment are noticeably more sandy in parts than red sediments.

<u>Grain Size</u>		<u>Distribution</u>	
clay sand		(40%)	Clear, sub-angular sand grains.
	very fine	(5%)	
	fine	(15%)	
	medium	(30%)	
	coarse	(10%)	

LBC 2

35-38" Predominately red clayey sand with a little bit of gray color (which swirls).

<u>Grain Size</u>		<u>Distribution</u>	
clay sand	very fine	(45%)	Clear, sub-angular sand.
	fine	(5%)	
	medium	(15%)	
	coarse	(20%)	
		(15%)	

38-41" Gray and red in colored clayey sand. Compact, dense sample. Does not crumble easily.

clay sand	very fine	(45%)	Clear, sub-angular sand grains.
	fine	(5%)	
	medium	(15%)	
	coarse	(20%)	
		(15%)	

41-44" Gray and red clayey sand compact, dense.

<u>Grain Size</u>		<u>Distribution</u>	
clay sand	very fine	(45%)	Clear, sub-angular sand. No bedding visible in the sandy clay.
	fine	(5%)	
	medium	(15%)	
	coarse	(20%)	
		(15%)	

44-47" Gray and red clayey sand, some yellow. Compact, dense sample.

<u>Grain Size</u>		<u>Distribution</u>	
clay sand	very fine	(45%)	Clear, sub-angular grains. Cross bedded sandy clay. clayey sand
	fine	(5%)	
	medium	(15%)	
	coarse	(20%)	
		(15%)	

47-50" Dark to light gray and red colored sandy clay. Dark gray with vertical bedding (rooting?) located in the middle of core.

<u>Grain Size</u>		<u>Distribution</u>	
clay		(50%)	(Beginning of 2" diameter core barrel.) Clear, sub-angular sand grains, compact dense. Does not crumble easily.
sand	very fine	(5%)	
	fine	(15%)	
	medium	(20%)	
	coarse	(10%)	

LBC 2

50-53" Gray, red and some mustard-yellow colored sandy clay.

<u>Grain Size</u>		<u>Distribution</u>	
clay sand		(50%)	Dense, compact sample clear, sub-angular sand grains
	very fine	(5%)	
	fine	(15%)	
	medium	(20%)	
	coarse	(10%)	

53-56" Red sandy clay. Dense, compact. Clear sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>	
clay sand		(50%)	Does not crumble easily.
	very fine	(5%)	
	fine	(15%)	
	medium	(20%)	
	coarse	(10%)	

56-59" Red sandy clay with some gray. Dense, compact. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>	
clay sand		(50%)	Crumbles a little easier than 53-56".
	very fine	(5%)	
	fine	(15%)	
	medium	(25%)	
	coarse	(10%)	

59-62" Gray and mustard yellow sandy clay. Dense, clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>	
clay sand		(50%)	Some vertical bedding (root- ing?).
	very fine	(5%)	
	fine	(15%)	
	medium	(25%)	
	coarse	(5%)	

62-65" Horizontally bedded which sweeps up on one side of the core. Red and gray sandy clay, clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>	
clay sand		(50%)	
	very fine	(5%)	
	fine	(15%)	
	medium	(25%)	
	coarse	(5%)	

LBC 2

65-68" Gray sandy clay with dark gray swirls in middle of core.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(55%)
	very fine	(5%)
	fine	(15%)
	medium	(25%)
	coarse	(5%)

68-71" Red, dark gray to light gray sandy clay. Vertically bedded (rooting?). Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(55%)
	very fine	(5%)
	fine	(15%)
	medium	(20%)
	coarse	(5%)

71-74" Red and gray clayey sand. Dense and compact. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(40%)
	very fine	(15%)
	fine	(25%)
	medium	(15%)
	coarse	(5%)

LBC 3

0-3" Sludge. Dark green/gray color. Dark colored (stained) round sand grains, liquid, very few lumps

<u>Grain Size</u>		<u>Distribution</u>
clay silt sand		(30%)
		(35%)
	very fine	(25%)
	fine	(10%)

LBC 3

3-6" Sludge. Dark gray color. Liquid, a few lumps. Dark colored (stained) round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(30%)
sand	very fine	(25%)
	fine	(10%)
	medium	(5%)

6-9" Sludge. Dark gray color. Liquid, thicker than core interval 3-6".

<u>Grain Size</u>		<u>Distribution</u>	
clay		(30%)	Dark-colored, round sand grains.
silt		(20%)	
sand	very fine	(25%)	
	fine	(15%)	
	medium	(10%)	

9-11" Dark gray sludge, many lumps, moist, pliable.

<u>Grain Size</u>		<u>Distribution</u>	
clay		(40%)	Dark-colored, round sand grains. Hot spot
silt		(5%)	
sand	very fine	(25%)	
	fine	(15%)	
	medium	(10%)	
	coarse	(5%)	

11-15" Dark gray sludge, dark colored round sand grains. Moist, pliable, a few lumps.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
sand	very fine	(15%)
	fine	(20%)
	medium	(15%)
	coarse	(10%)

15-18" Gray and red sandy clay, $\frac{1}{2}$ sludge, $\frac{1}{2}$ clay. Pliable, moist clear, round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
sand	very fine	(10%)
	fine	(15%)
	medium	(25%)

LBC 3

18-21" Gray, with some red and yellow, sandy clay. Clear, sub-rounded sand grains, compact, dense sample.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
sand	very fine	(10%)
	fine	(15%)
	medium	(25%)

21-24" Red, with some blue and yellow sandy clay. Clear, sub-round sand grains, compact, dense clay.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
sand	very fine	(10%)
	fine	(15%)
	medium	(25%)

24-27" Red, gray and yellow colored clayey sand. Crumbles easily. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
sand	very fine	(5%)
	fine	(15%)
	medium	(25%)
	coarse	(5%)

27-30" Red with gray streaks (1 cm wide) in middle of core clayey sand. Crumbles easily. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
sand	very fine	(5%)
	fine	(20%)
	medium	(35%)
	coarse	(5%)

30-33" Red and blue with some yellow. Clayey sand. Clear, sub-angular sand grains. Crumbles easily.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
sand	very fine	(5%)
	fine	(20%)
	medium	(35%)
	coarse	(5%)

LBC 3

33-36" Red with gray swirls in middle of core. Clayey sand. Crumbles easily. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(35%)
	very fine	(5%)
	fine	(20%)
	medium	(35%)
	coarse	(5%)

36-39" Red, gray and yellow clayey sand. Crumbles easily. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(35%)
	very fine	(5%)
	fine	(20%)
	medium	(30%)
	coarse	(10%)

39-44" Gray, yellow and red clayey sand. Crumbles easily. Clear, sub-angular sand grains, crumbles easily.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(35%)
	very fine	(5%)
	fine	(20%)
	medium	(30%)
	coarse	(10%)

LBC 4

0-3" Sludge - green/gray color, liquid. Sand-silt-clay mixture. Dark colored round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay silt sand		(30%)
		(40%)
	very fine	(15%)
	fine	(10%)
	medium	(5%)

LBC 4

3-6" Sludge - dark gray color, liquid. Sand-silt-clay mixture. Dark colored, round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
silt		(35%)
sand	very fine	(15%)
	fine	(10%)
	medium	(5%)

6-9" Sludge - dark gray color. Sand-silt-clay mixture. Dark colored, round sand grains. Thicker sample than 3-6".

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
silt		(30%)
sand	very fine	(15%)
	fine	(10%)
	medium	(5%)

9-15" Brown sludge and gray sandy clay. Separated into 2 splits. Hot spot. Clear, sub-round sand grains, sludge to 10". Moist pliable.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
silt		(10%)
sand	very fine	(15%)
	fine	(10%)
	medium	(15%)

15-18" Red and gray sandy clay, no bedding visible. Clear, sub-angular sand grains; sticky.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(10%)
	fine	(15%)
	medium	(20%)

18-21" Gray and red mottled sandy clay. No bedding visible. Clear, sub-angular sand grains; sticky dense sample.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(10%)
	fine	(15%)
	medium	(20%)

LBC 4

21-24"

Predominantly red, with some gray in middle of core, sandy clay. Clear, sub-angular sand grains; dense sample.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(10%)
	fine	(15%)
	medium	(20%)

24-27"

Red; gray streak at bottom of core, sandy clay. Clear, sub-angular sand grains; dense sample.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(5%)
	fine	(15%)
	medium	(25%)

27-30"

Gray sandy clay; small circle of red sandy clay at bottom of core, some of the gray sandy clay is tinged yellow. Clear, sub-angular grains, dense sample. Clay does not crumble easily.

<u>Grain Size</u>		<u>Distribution</u>
clay		(60%)
sand	very fine	(5%)
	fine	(15%)
	medium	(20%)

30-33"

Red at top, gray at bottom, sandy clay. Slightly bedded in middle of core. Clear, sub-angular sand grains. Does not crumble easily. Dense sample.

<u>Grain Size</u>		<u>Distribution</u>
clay		(70%)
sand	very fine	(5%)
	fine	(10%)
	medium	(15%)

33-38"

Red sand clay, thin gray streaks on top $\frac{1}{2}$ of core. Clear, sub-angular sand grains. Very dense and compact sample.

<u>Grain Size</u>		<u>Distribution</u>
clay		(60%)
sand	very fine	(5%)
	fine	(10%)
	medium	(25%)

LBC 4

3-6" Sludge - dark gray color, liquid. Sand-silt-clay mixture. Dark colored, round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
silt		(35%)
sand	very fine	(15%)
	fine	(10%)
	medium	(5%)

6-9" Sludge - dark gray color. Sand-silt-clay mixture. Dark colored, round sand grains. Thicker sample than 3-6".

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
silt		(30%)
sand	very fine	(15%)
	fine	(10%)
	medium	(5%)

9-15" Brown sludge and gray sandy clay. Separated into 2 splits. Hot spot. Clear, sub-round sand grains, sludge to 10". Moist pliable.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
silt		(10%)
sand	very fine	(15%)
	fine	(10%)
	medium	(15%)

15-18" Red and gray sandy clay, no bedding visible. Clear, sub-angular sand grains; sticky.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(10%)
	fine	(15%)
	medium	(20%)

18-21" Gray and red mottled sandy clay. No bedding visible. Clear, sub-angular sand grains; sticky dense sample.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(10%)
	fine	(15%)
	medium	(20%)

LBC 5

0-3" Sludge - dark gray/green color. Liquid-no lumps, dark-colored round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(40%)
sand	very fine	(15%)
	fine	(10%)
	medium	(5%)

3-6" Sludge - dark gray color. Dark colored, round sand grains. Liquid with a few lumps.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(40%)
sand	very fine	(15%)
	fine	(10%)
	medium	(5%)

6-9" Sludge - dark gray color. Lumpy and much thicker than 3-6". Moist, dark-colored round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
silt		(30%)
sand	very fine	(15%)
	fine	(10%)
	medium	(5%)

9-14" Dark gray color, two pints of sludge. Dark-colored, liquid - very few lumps. Hot spot.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(40%)
sand	very fine	(15%)
	fine	(10%)
	medium	(5%)

LBC 5

14-17" Gray sludge. Very few lumps. Liquid, dark colored, round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(45%)
sand	very fine	(15%)
	fine	(10%)

17-20" Gray sludge. Thick consistency. Dark colored round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
silt		(20%)
sand	very fine	(25%)
	fine	(20%)

20-23" Sludge/clayey sand contact, 50/50 mixture. Brown and gray color. Clear, sub-round sand grains. Moist and pliable.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
silt		(10%)
sand	very fine	(20%)
	fine	(20%)
	medium	(10%)

23-26" Sample is $\frac{1}{2}$ sludge, $\frac{1}{2}$ clay. Sludge is brown. Clayey sand is red, blue and yellow. Clear, sub-round sand grains. Moist and pliable.

<u>Grain Size</u>		<u>Distribution</u>
clay		(45%)
silt		(5%)
sand	very fine	(15%)
	fine	(15%)
	medium	(20%)

26-29" Clayey sand with a little sludge. Red and blue in color. Crumbles easily. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
sand	very fine	(15%)
	fine	(20%)
	medium	(30%)

LBC 5

29-32" Sludge and clayey sand. Pliable, crumbles easily. Clear sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
sand	very fine	(15%)
	fine	(20%)
	medium	(30%)

32-35" At 35" brown sludge. Clayey sand $\frac{1}{2}$ red and blue. Very pliable. Clear, sub-round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
silt		(5%)
sand	very fine	(15%)
	fine	(20%)
	medium	(25%)

35-38" Sludge pocket. Brown. Moist, pliable. Clear, sub-round sand grains. Lumpy-small amounts of red sandy clay.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
silt		(10%)
sand	very fine	(15%)
	fine	(20%)
	medium	(5%)

38-41" Red and blue sandy clay. Some sludge. Dense, compact sample; clear, sub-angular sand grains. Does not crumble easily.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(10%)
	fine	(15%)
	medium	(20%)

41-44" Gray and red sandy clay; dense, compact sample. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(10%)
	fine	(15%)
	medium	(20%)

LBC 5

44-47" Gray and red sandy clay, pliable, moist, some sludge.
Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(55%)
	very fine	(10%)
	fine	(15%)
	medium	(20%)

47-50" Red sandy clay, very pliable. Clear, sub-angular sand grains.
Moist.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(55%)
	very fine	(10%)
	fine	(15%)
	medium	(20%)

50-53" Red and gray sandy clay, compact, dense sample. Subangular sand
grains. Quartz rich.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(55%)
	very fine	(10%)
	fine	(15%)
	medium	(15%)
	coarse	(5%)

53-56" Clayey sand . Some gray color. Crumbles easily. Sub-angular,
clear sand grains, quartz rich.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(45%)
	very fine	(10%)
	fine	(10%)
	medium	(20%)
	coarse	(15%)

56-59" Red and gray clayey sand. Crumbles easily.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(45%)
	very fine	(10%)
	fine	(10%)
	medium	(20%)
	coarse	(15%)

LBC 5

59-64"

Gray with a red streak at top and bottom of sample, clayey sand. Crumbles easily. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(45%)
sand	very fine	(10%)
	fine	(15%)
	medium	(25%)
	coarse	(5%)

LBC 6

0-3"

Sludge. Gray color, mostly a liquid. Silt-sand-clay mixture. Dark-colored round, smooth grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
silt		(30%)
sand	very fine	(20%)
	fine	(10%)

3-6"

Sludge. Dark gray, thick liquid. Sand-silt-clay mixture. Predominantly very fine grained, dark-colored (stained) sand.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
silt		(25%)
sand	very fine	(30%)
	fine	(5%)

6-9"

Sludge. Dark gray color. Paste-like consistency. Liquid sand-silt-clay mixture.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(25%)
sand	very fine	(20%)
	fine	(15%)
	medium	(10%)

LBC 6

9-12" Sludge. Thicker than 6-9" section. Liquid, dark-colored sand grains. Sand-silt-clay mixture.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(10%)
sand	very fine	(25%)
	fine	(20%)
	medium	(15%)

12-15" Brown colored sludge. Thick liquid. Smooth, round grains. Sand-silt-clay mixture.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
silt		(10%)
sand	very fine	(15%)
	fine	(15%)
	medium	(20%)

15-18" Brown sludge, liquid. Sand-silt-clay mixture, sorted, smooth, round sand grains. Some red sandy clay.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
silt		(5%)
sand	very fine	(10%)
	fine	(10%)
	medium	(35%)

18-21" Contact zone - sludge to red and gray sandy clay/predominantly sandy clay.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
silt		(5%)
sand	very fine	(5%)
	fine	(10%)
	medium	(25%)

21-24" Gray and red sandy clay. No bedding, Moist sample. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(5%)
	fine	(10%)
	medium	(30%)

LBC 6

24-27" Yellow and gray sandy clay. At 27" a thin layer of brown sludge. Clear, sub-angular sand grains. No bedding.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(5%)
	fine	(10%)
	medium	(30%)

27-30" Orange/red and gray colored sandy clay. Sludge at 27". Moist sample, clear, sub-angular sand grains. No bedding.

<u>Grain Size</u>		<u>Distribution</u>
clay		(60%)
sand	very fine	(5%)
	fine	(10%)
	medium	(20%)

30-33" Red, yellow and gray color, sandy clay. Crumbly, clear, sub-angular sand grains. No bedding visible.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
sand	very fine	(5%)
	fine	(15%)
	medium	(30%)

33-36" Red and gray colored wavy bedded, sandy clay. Crumbly. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
sand	very fine	(5%)
	fine	(15%)
	medium	(30%)

36-39" Red and yellow sandy clay, banded. Crumbly; clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
sand	very fine	(5%)
	fine	(15%)
	medium	(25%)
	coarse	(5%)

LBC 6

39-42" Gray, yellow and red colored sandy clay. Red and yellow thin bands for an inch. Crumbly. Very clear sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(50%)
	very fine	(5%)
	fine	(10%)
	medium	(25%)
	coarse	(10%)

42-45" Gray and red colored clayed sand. Crumbles easily. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(45%)
	very fine	(10%)
	fine	(15%)
	medium	(25%)
	coarse	(5%)

45-48" Red clayey sand. Crumbles easily. Clear, sub-angular grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(40%)
	very fine	(10%)
	fine	(15%)
	medium	(25%)
	coarse	(10%)

48-51" Red and gray colored, clayey sand. Crumbles fairly easily. Clear, sub-angular sand grains. No bedding visible.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(40%)
	very fine	(10%)
	fine	(15%)
	medium	(20%)
	coarse	(15%)

51-54" Red colored, clayey sand, crumbles easily. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(40%)
	very fine	(10%)
	fine	(15%)
	medium	(20%)
	coarse	(15%)

LBC 6

54-57: Red colored, clayey sand. Crumbles easily; clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(30%)
	very fine	(10%)
	fine	(15%)
	medium	(20%)
	coarse	(25%)

57-60" Red colored clayey sand, crumbles easily. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(25%)
	very fine	(10%)
	fine	(15%)
	medium	(20%)
	coarse	(30%)

60-63" Red colored clayey sand. Crumbles easily. Compact sample, clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(25%)
	very fine	(10%)
	fine	(15%)
	medium	(20%)
	coarse	(30%)

63-66: Clayey sand, red; crumbles easily. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(25%)
	very fine	(10%)
	fine	(15%)
	medium	(20%)
	coarse	(30%)

66-69" Yellow, clayey sand. Thin red beds run horizontally. Clear sub-angular sand grains. Compact.

<u>Grain Size</u>		<u>Distribution</u>
clay sand		(20%)
	very fine	(10%)
	fine	(15%)
	medium	(20%)
	coarse	(35%)

LBC 6

69-72" Red and yellow horizontal banding, clayey sand. Compact, crumbles easily. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(20%)
sand	very fine	(10%)
	fine	(15%)
	medium	(20%)
	coarse	(35%)

72-75" Red colored clayey sand. Crumbles easily; compact. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(15%)
sand	very fine	(10%)
	fine	(15%)
	medium	(20%)
	coarse	(40%)

LBC 7

0-3" Sludge - dark green color. Sand-silt-clay mixture. Sample mostly liquid. Very fine, round dark sand fraction.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(40%)
sand	very fine	(25%)
	fine	(5%)

3-6" Sludge - dark green color. Sand-silt-clay mixture. Liquid, dark-colored round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(40%)
sand	very fine	(20%)
	fine	(5%)
	medium	(5%)

LBC 6

6-10" Dark green sludge. Sand-silt-clay mixture.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
silt		(35%)
sand	very fine	(15%)
	fine	(5%)
	medium	(5%)

LBC 7

~~8-12"~~ Brown liquid sludge. Dark colored, round, sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(40%)
sand	very fine	(15%)
	fine	(10%)
	medium	(5%)

12-15" Chocolate brown sludge. Sand-silt-clay mixture. Clear, sub-rounded sand fraction, moist.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
silt		(25%)
sand	very fine	(10%)
	fine	(15%)
	medium	(10%)

15-18" Sludge/clayey sand contact, gray and red. Clear, sub-rounded sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(45%)
silt		(10%)
sand	very fine	(10%)
	fine	(20%)
	medium	(15%)

LBC 7

18-21" Brown and gray sludge mixed in with clayey sand. Clear, sub-rounded sand grains. Very moist, pliable, sticky.

<u>Grain Size</u>		<u>Distribution</u>
clay		(45%)
silt		(10%)
sand	very fine	(10%)
	fine	(20%)
	medium	(15%)

21-24" Red and gray sandy clay. No sludge. Clear, sub-angular sand grains. Crumbly. No bedding visible.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(5%)
	fine	(15%)
	medium	(25%)

24-27" Gray with red streaks sandy clay, No bedding present. Clear, sub-angular sand grains, crumbly.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
sand	very fine	(5%)
	fine	(15%)
	medium	(25%)
	coarse	(5%)

27-30" Gray sandy clay - red streak on one side, and top and bottom of sample. Dense. Crumbly, clear, sub-angular sand fraction.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(5%)
	fine	(20%)
	medium	(20%)

30-33" Red sandy clay with gray swirls. Crumbly, dense sample; clear, sub-angular sand fraction.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(5%)
	fine	(25%)
	medium	(15%)

LBC 7

33-36" Sandy clay $\frac{1}{2}$ red, $\frac{1}{2}$ gray-interbedded vertically in middle of core. Crumbly, clear, sub-angular sand grains. Dense sample.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
sand	very fine	(5%)
	fine	(15%)
	medium	(25%)

36-39" Red sandy clay with gray swirls in top half of core. Crumbly, clear, sub-angular sand grains. Dense sample.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
sand	very fine	(5%)
	fine	(15%)
	medium	(30%)

39-42" Red sandy clay. Several thin gray streaks (2" long) on the side of the sample. Dense sample. Clear, sub-angular sand grains. Crumbly.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
sand	very fine	(10%)
	fine	(15%)
	medium	(25%)

LBC 8

0-3" Olive green sludge; liquid. Silt-clay-sand mixture.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(40%)
sand	very fine	(20%)
	fine	(10%)

3-8" Hot spot. Liquid sludge. Thicker consistency than 0-3" section. Sand-silt-clay mixture. Clear, round sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(20%)
silt		(35%)
sand	very fine	(20%)
	fine	(15%)

LBC 8

8-11" Chocolate brown sludge; liquid. Feels gritty. Clear, sub-angular grains. Sand-silt-clay mixture.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(5%)
sand	very fine	(25%)
	fine	(20%)
	medium	(15%)
	coarse	(5%)

11-14" Brown and gray sludge, thick liquid. Sand-silt-clay mixture. Clear, sub-angular sand grains. Smooth and sticky.

<u>Grain Size</u>		<u>Distribution</u>
clay		(35%)
silt		(5%)
sand	very fine	(15%)
	fine	(20%)
	medium	(15%)
	coarse	(10%)

14-17" Predominantly white/gray clayey sand. Horizontal bands between yellow, white/gray bands. Crumbly dry sample. Clear, sub-angular grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
sand	very fine	(10%)
	fine	(15%)
	medium	(20%)
	coarse	(15%)

17-20" Red to gray/yellow colored clayey sand. Clear, sub-angular grains. Moist, crumbly.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
sand	very fine	(5%)
	fine	(10%)
	medium	(30%)
	coarse	(15%)

LBC 8

20-23" Gray and yellow (and red - not predominate color) clayey sand with some horizontal bedding. Clear, sub-angular grains. Moist, crumbly.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
sand	very fine	(5%)
	fine	(10%)
	medium	(30%)
	coarse	(15%)

23-26" Gray and yellow colored clayey sand with bands of red, yellow and gray, sand clay. Clear, sub-angular sand grains. Moist.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
sand	very fine	(5%)
	fine	(10%)
	medium	(30%)
	coarse	(15%)

26-29" Alternating bands of gray and yellow (some white color) clayey sand. Dense, compact sample. Clear sand grains, sub-angular, quartz rich.

<u>Grain Size</u>		<u>Distribution</u>
clay		(45%)
silt		(5%)
sand	fine	(5%)
	medium	(15%)
	coarse	(30%)

29-32" Sand clay with bands of gray and white/light yellow with swirls of dark red, (3 cm high by 5 cm long). Dense, compact sample. Sand grains are clear and sub-angular.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
silt		(5%)
sand	fine	(5%)
	medium	(15%)
	coarse	(25%)

LBC 9

0-3" Dark gray sludge. Liquid, smooth, not gritty. Sand-silt-clay mixture.

<u>Grain Size</u>		<u>Distribution</u>
clay		(30%)
silt		(35%)
sand	very fine	(20%)
	fine	(10%)
	medium	(5%)

3-6" Medium dark gray color sludge. Liquid, smooth, lumpy. Dark colored sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(45%)
silt		(40%)
sand	very fine	(10%)
	fine	(5%)

6-12" Hot spot. Contact zone of sludge to clay. Sludge medium gray color. Smooth and sticky, gray color with little bit of red and yellow clay.

<u>Grain Size</u>		<u>Distribution</u>
clay		(95%)
silt		(5%)

12-15" Light gray sandy clay. Horizontal sections colored red, yellow. Clear sand grains; sub-angular.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
silt		(5%)
sand	very fine	(5%)
	fine	(5%)
	medium	(10%)
	coarse	(20%)

15-18" Gray and yellow colored sandy clay. Smooth, clear, sub-angular sand grains. Dense, compact sample.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
silt		(5%)
sand	fine	(5%)
	medium	(15%)
	coarse	(20%)

LBC 9

18-21" Gray and yellow colored sandy clay. Horizontal streak (2"wide) of red. Dense, compact sample. Clear, sub-angular sand grains.

<u>Grain Size</u>		<u>Distribution</u>
clay		(55%)
silt		(5%)
sand	fine	(5%)
	medium	(15%)
	coarse	(20%)

21-24" Gray and yellow sandy clay. 2" wide vertical red streak. Clear, sub-round sand grains. Dense, compact sample. Crumbles easily.

<u>Grain Size</u>		<u>Distribution</u>
clay		(50%)
silt		(5%)
sand	fine	(5%)
	medium	(10%)
	coarse	(30%)

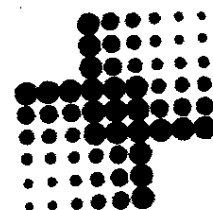
24-27" Clayey sand with swirls of red or gray between yellow/white swirls. Predominate color - yellow. Compact, dense sample.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
silt		(5%)
sand	very fine	(5%)
	fine	(5%)
	medium	(15%)
	coarse	(30%)

27-31" Clayey sand with bands colored red, brown, yellow and orange. A 3" wide 1" high red section on one side of core. Very clear sand grains, sub-round. Dense, compact sample, crumbles.

<u>Grain Size</u>		<u>Distribution</u>
clay		(40%)
silt		(5%)
sand	very fine	(5%)
	fine	(5%)
	medium	(15%)
	coarse	(30%)

APPENDIX IV
COPIES OF FIELD LABORATORY NOTES
FOR CALCULATION OF PERCENT MOISTURE
DETERMINATION OF ALL SUBSAMPLES



% moisture

sample #	Dish wt.	Wet (Dish + sample)	Dry (Dish + sample)	Dry sample wt.	% mois.
LBC 3					
0-3"	1.00g	11.01 ^(10.01)	2.78	1.78	82%
3-6	0.99g	11.06 ^(10.07)	2.88	1.89	81%
6-9	1.00g	11.10 ^(10.10)	4.32	3.32	67%
9-11	1.00g	11.18 ^(10.18)	5.15	4.15	59%
11-15	0.99g	11.52 ^(10.53)	6.87	5.88	44%
15-18	0.99g	6.06 ^(5.07)	4.75	3.76	26%
18-21	0.99g	6.27 ^(5.28)	5.85	4.36	17%
21-24	1.00g	6.09 ^(5.09)	5.16g	4.16	18%
24-27	1.00g	6.11 ^(5.11)	5.19	4.19	18%
27-30	0.99g	6.22 ^(5.23)	5.32	4.33	17%
30-33	1.00g	6.11 ^(5.11)	5.30	4.30	16%
33-36	0.99g	6.12 ^(5.13)	5.30	4.31	16%
36-39	0.89g	6.01 ^(5.02)	5.20	4.21	16%
39-44"	0.99g	6.11 ^(5.12)	5.31	4.32	16%
LBC 1					
0-3"					
3-6					
6-9					
9-15	0.98	11.24 ^(10.26)	5.02	4.04	60%
15-18					
18-21					
21-24					
24-27					
27-30					
30-33					
33-38"	0.99g	6.01 ^(5.02)	5.14	4.15	17%

% moisture

4-2-85	Sample #	Dish WT.	Wet wt. (Dish + Sample)	Dry (Dish + Sample)	Dry Sample WT.	% mois.
LBC 2	0-3	0.99g	11.04 ^(10.05)	1.80	0.81g	92%
	3-6	1.03g	11.12 ^(10.09)	2.62	1.59	84%
	6-9	1.01g	11.03 ^(10.02)	2.93	1.82	82%
	9-11	1.00	11.18 ^(10.18)	3.08	2.08	80%
	11-17	1.00g	6.55g ^(5.55)	3.33	2.33	58%
	17-20	0.99g	5.99g ^(5.0)	5.14	4.15	17%
	20-23	0.99g	6.06 ^(5.07)	5.21	4.22	17%
	23-26	0.99g	6.10 ^(5.11)	5.12	4.13	19%
	26-29	0.99	5.99g ^(5.0)	5.08	4.09	18%
	29-32	1.00	6.08 ^(5.08)	5.20	4.20	17%
	32-35	1.00	6.07 ^(5.07)	5.23	4.23	17%
	35-38	1.00	6.04 ^(5.04)	5.16	4.16	17%
	38-41	0.99	6.08 ^(5.09)	5.25	4.26	16%
	41-44	1.01	6.03 ^(5.02)	5.25	4.24	16%
	44-47	0.99	6.05 ^(5.06)	5.38	4.39	15%
	47-50	1.01	6.01 ^(5.0)	5.40	4.40	16%
	50-53	1.00	6.10 ^(5.1)	5.32	4.32	15%
	53-56	1.01	6.02 ^(5.01)	5.09	4.08	19%
	56-59	1.00	6.02 ^(5.02)	5.23	4.23	16%
	59-62	0.99	6.06 ^(5.07)	5.29	4.29	15%
	62-65	0.98	6.12 ^(5.14)	5.37	4.39	15%
	65-68	0.99	6.05 ^(5.06)	5.24	4.25	16%
	68-71	0.99	6.00 ^(5.01)	5.34	4.35	13%
	71-74	0.99	6.03 ^(5.04)	5.57	4.58	9%

mo moisture

Sample #	Dish wt	wet (Dish + Sample)	Dry (Dish + Sample)	Dry sample wt.	mo mois
LBC 4 0-3"	1.01g	6.06g ^(5.05)	1.83g	0.82g	84%
3-6	1.00g	6.08g ^(5.08)	2.04g	1.04	80%
6-9	1.00g	6.11g ^(5.11)	2.21g	1.21	76%
S 9-15	1.01g	6.01g ^(5.0)	3.02g	2.01	60%
C 9-15 ^②	0.99g	6.35g ^(5.36)	4.94	3.95	26%
15-18	1.00g	6.11g ^(5.11)	5.07g	4.07	20%
18-21	1.00g	6.10g ^(5.10)	5.11g	4.11	19%
21-24	1.01g	6.03g ^(5.03)	5.14g	4.13	18%
24-27	1.00g	6.03g ^(5.03)	5.20	4.20	17%
27-30	0.99g	6.02g ^(5.03)	5.65	4.06	19%
30-33	1.00g	6.05g ^(5.05)	5.24	4.24	16%
33-38	1.00g	6.03g ^(5.03)	5.20	4.20	17%
LBC 5 0-3"	0.99g	11.02g ^(10.03)	2.36g	1.37g	86%
3-6	1.01g	11.05g ^(10.04)	3.05g	2.04	80%
6-9	1.00g	11.03g ^(10.03)	3.98g	2.98	71%
9-14	1.02g	11.02g ^(10.0)	3.09	2.07	79%
14-17	1.01g	11.01g ^(10.0)	4.33	3.32	67%
17-20	1.01g	11.02g ^(10.02)	6.01	5.00	50%
20-23	1.01g	6.26g ^(5.25)	4.67	3.66	30%
23-26	1.00g	6.01g ^(5.01)	4.59	3.59	28%
26-29	1.01g	6.01g ^(5.0)	4.91	3.90	22%
29-32	1.02g	6.08g ^(5.06)	4.77	3.75	26%
32-35	1.01g	6.13g ^(5.12)	4.60	3.65	28%
35-38	1.00g	6.12g ^(5.12)	3.72	2.72	47%
38-41	1.00g	6.13g ^(5.12)	4.90	3.90	24%
41-44	1.00g	6.10 ^(5.10)	5.25	4.25	17%
44-47	1.00g	6.02g ^(5.02)	5.11	4.11	100%

0% moisture

sample#	Wet (Dish + wt)	Wet (Dish + S)	Dry (Dish + S)	Dry Sample wt	% mois
365 47-50"	0.99g	6.11g ^(5.12)	4.94	3.95	23%
50-53"	0.99g	6.09g ^(5.1)	5.18	4.19	18%
53-56"	0.99g	6.08g ^(5.06)	5.36	4.37	14%
56-59"	1.00g	6.06g ^(5.06)	5.34	4.34	14%
59-64"	1.00	6.16g ^(5.16)	5.45	4.45	14%

LBC 7 39-42"

1.01g

6.41g^(5.40)

5.53g

4.52

16%

% moisture

Sample #	Dish Wt.	Wet (Dish + Sample)	Dry (Dish + Sample)	Dry Sample Wt	% mois
0-3"	0.98g	11.06g ^(10.08)	12.22g	21.24g	87%
3-6"	1.01g	11.05g ^(10.04)	2.43g	1.42	86%
6-9"	0.99g	6.02g ^(5.03)	2.25g	1.26	75%
9-12	0.97g	6.24g ^(5.27)	2.37g	1.40	74%
12-15	0.99g	6.01g ^(5.02)	3.53g	2.54	49%
15-18	0.99g	5.99g ^(5.0)	3.70g	2.71	46%
18-21	0.98g	5.98g ^(5.0)	4.96g	3.98	20%
21-24	0.96g	6.32g ^(5.36)	5.45g	4.49	16%
24-27	1.00g	6.38g ^(5.38)	5.18g	4.18g	22%
27-30	1.01g	6.05g ^(5.04)	5.98g	3.97g	21%
30-33	1.01g	6.06g ^(5.05)	5.29g	4.28g	15%
33-36	1.01g	6.10g ^(5.09)	5.31g	4.30	15%
36-39	1.01g	6.10g ^(5.09)	5.40g	4.39	14%
39-43"	0.98g	5.99g ^(5.0)	5.25g	4.36	13%

LBC 7

0-3"	1.01g	11.03g ^(10.02)	2.37g	1.34g	86%
3-6"	1.01g	11.18g ^(10.17)	2.94g	1.95	80%
6-10	1.01g	11.29g ^(10.29)	3.64g	2.15	79%
10-12	1.01g	11.11g ^(10.10)	4.31g	3.30	67%
12-15	1.01g	11.54g ^(10.53)	6.79g	5.78	45%
15-18	1.00g	6.21g ^(5.21)	4.61g	3.61	31%
18-21	1.00g	6.27g ^(5.27)	5.04g	4.04	25%
21-24	1.00g	6.08g ^(5.08)	5.13g	4.13	19%
24-27	1.00g	6.06g ^(5.06)	5.22g	4.22	17%
27-30	1.01g	6.05g ^(5.04)	5.07g	4.06	19%
30-33	1.02g	6.01g ^(5.01)	5.21g	4.19	16%
33-36	1.02g	6.10g ^(5.08)	5.27g	4.25	16%
36-39	1.01g	6.14g ^(5.14)	5.24g	4.24	16%

39-42

nt'd)

C 6

MOISTURE

sample #	Dish wt.	wet (Dish + sample)	Dry (Dish + sample)	Dry sample wt.	% moisture
42-45"	0.99	6.12 ^(5.13)	5.20	4.21	18%
45-48"	0.99	6.01 ^(5.02)	5.24	4.25	15%
48-51"	0.99	6.00 ^(5.07)	5.36	4.37	14%
51-54"	0.99	6.08 ^(5.09)	5.40	4.41	13%
54-57"	1.01	6.14 ^(5.13)	5.47	4.46	13%
57-60"	1.00	6.00 ^(5.0)	5.40	4.40	12%
60-63"	1.00	6.08 ^(5.02)	5.48	4.48	12%
63-66"	1.00	6.02 ^(5.02)	5.32	4.32	14%
66-69"	1.00	6.08 ^(5.01)	5.41	4.41	13%
69-72"	0.99	6.00 ^(5.01)	5.42	4.43	12%
72-75"	0.99	6.12 ^(5.13)	5.54	4.55	11%

olo moisture

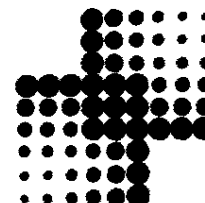
(5.3) wet-dry
5 wet

	Sample #	Dish wt	Wet (Dish + sample)	Dry (Dish + sample)	Dry sample wet	o/o mois
LBC 8	0-3"	1.01	6.31g (5.30)	1.87g	0.86g	84%
	3-8"	1.0g	6.26 (5.26)	2.18g	1.18g	78%
	8"-11"	1.01g	6.27 (5.26)	4.98g	3.17g	25%
Blue	11"-14"	1.02	6.24 (5.24)	5.08g	4.06g	23%
Red	11"-14"	1.02	6.54 (5.52)	5.43g	4.41g	20%
	14"-17"	0.98	7.83 (6.85)	6.46g	5.42g	20%
Red	17"-20"	1.05	6.44 (5.39)	5.50	4.45g	17%
Yellow	17"-20"	1.05	6.33 (5.28)	5.47	4.42g	16%
Blue	17"-20"	1.05	6.38 (5.33)	5.35g	4.3g	19%
	20"-23"	1.01	6.26 (5.25)	5.43g	4.42g	16%
	23"-26"	1.0g	6.56 (5.50)	5.74g	4.74g	15%
	26"-29"	0.98	6.14g (5.16)	5.38g	4.4g	15%
	29"-32"	0.97	6.01g (5.00)	5.30g	4.33g	14%

LBC 9	0-3"	0.99g	6.10g (5.11)	1.47g	0.68g	83%
	3-6"	1.01g	6.01g (5.0)	2.05g	1.04g	79%
	6-12"	1.00g	6.03g (5.03)	4.04g	3.24g	36%
	12-15"	1.01g	6.11g (5.1)	5.03	4.08g	20%
	15-18"	0.99g	6.35g (5.36)	5.45	4.46	17%
	18-21"	0.99g	6.22g (5.23)	5.40	4.41	16%
	21-24"	0.99g	5.99g (5.0)	5.10g	4.11g	18%
	24-27"	0.98	6.09g (5.11)	5.34	4.36g	15%
	27-31"	0.98	6.45 (5.44)	5.45	4.47g	17%

APPENDIX V

**FIELD LABORATORY NOTES FOR DETERMINATION OF WET
WEIGHT OF SUBSAMPLES USED IN THE ON-SITE
SPECTRAL GAMMA ACTIVITY DETERMINATION**



Spectral Gamma Analysis

	<u>sample #</u>	<u>vial wt.</u>	<u>vial + sample wt.</u>	<u>sample wt.</u>
<u>LBC3</u>	0-3"	20.77g	91.79g	71.02
	3-6	20.60g	92.82g	72.22
	6-9	20.72g	97.11g	76.39
	9-11	20.61g	101.24g	80.63
	11-15	20.62g	114.55g	93.93
	15-18	20.61g	130.68g	110.07
	18-21	20.58g	121.93g	101.35
	21-24	20.65g	121.77g	101.12
	24-27	20.79	117.67g	96.88
	27-30	20.71	121.43g	100.72
	30-33	20.64	119.10g	98.46
	33-36	20.62	118.64	98.02
	36-39	20.76g	122.43g	101.67
	39-44	20.63g	118.85g	98.22
<u>LBC4</u>	0-3			
	3-6			
	6-9			
	9-15	20.72g	107.07g	86.35
	15-18			
	18-21			
	21-24			
	24-27			
	27-30			
	30-33			
	33-38	21.01g	127.98g	106.97

LBC2

<u>Sample #</u>	<u>Vial wt.</u>	<u>Vial + S.</u>	<u>Sample wt.</u>
0-3	20.62g	87.74g	67.12g
3-6	20.67	86.87	66.20
6-9	20.59	90.29	69.70
9-11	20.62	91.04	70.42
11-17	20.58	106.41	85.83
17-20	20.72	128.38	107.66
20-23	20.70	123.79	103.09
23-26	20.70	124.18	103.48
26-29	20.72	121.38	100.66
29-32	20.71	113.35	92.64
32-35	20.85	116.34	95.49
35-38	20.70	119.06	98.36
38-41	20.74	114.78	94.04
41-44	20.64	117.00	96.36
44-47	20.63	107.79	87.16
47-50	20.74	115.56g	94.81
50-53	20.61	116.43	95.82
53-56	20.60	121.08	100.48
56-59	20.62	116.44	95.82
59-62	20.61	119.59	98.98
62-65	20.63	113.72	93.09
65-68	20.63	113.91	93.26
68-71	20.72	114.45	93.73
71-74	20.76	103.92g	83.16

	<u>Sample #</u>	<u>vial wti</u>	<u>vial + s. wt.</u>	<u>sample wt.</u>
--	-----------------	-----------------	----------------------	-------------------

LBC 4

	0-3	20.96g	91.40g	70.64g
	3-6	21.00g	92.31g	71.31
	6-9	21.03g	96.04g	75.01
S	9-15	21.03g	108.51g	87.48
C	9-15	20.94g	131.66g	110.72
	15-18	21.04g	134.33g	113.29
	18-21	20.98g	133.02g	112.04
	21-24	21.02g	136.94g	115.92
	24-27	21.00g	125.42g	104.42
	27-30	20.96g	123.24g	102.28
	30-33	20.96g	120.15g	99.19
	33-38	21.02	130.64g	109.62g

LBC 5

	<u>0-3</u>	<u>20.61g</u>	<u>89.55</u>	<u>48.94</u>	<u>vial</u>	<u>v+s</u>	<u>s. wt</u>
--	------------	---------------	--------------	--------------	-------------	------------	--------------

	3-6	20.70	92.28	71.58	47-50	20.71	117.42
--	-----	-------	-------	-------	-------	-------	--------

	6-9	20.63	95.36	74.73	50-53	20.71	104.49
--	-----	-------	-------	-------	-------	-------	--------

	9-14	20.64	92.81	72.17	53-56	20.61	95.51
--	------	-------	-------	-------	-------	-------	-------

	14-17	20.61	103.23	82.62	56-59	20.63	97.33
--	-------	-------	--------	-------	-------	-------	-------

	17-20	20.63	117.14g	96.51	59-64	20.58	95.12
--	-------	-------	---------	-------	-------	-------	-------

	20-23	20.63	131.44	110.81	64-67	20.61	115.12
--	-------	-------	--------	--------	-------	-------	--------

	23-26	20.62	128.13	107.51	67-70	20.61	115.12
--	-------	-------	--------	--------	-------	-------	--------

	26-29	20.74	138.17	117.68	70-73	20.61	115.12
--	-------	-------	--------	--------	-------	-------	--------

	29-32	20.61g	131.25	110.64	73-76	20.61	115.12
--	-------	--------	--------	--------	-------	-------	--------

	32-35	20.73	123.67	102.94	76-79	20.61	115.12
--	-------	-------	--------	--------	-------	-------	--------

	35-38	20.73	122.39	101.66	79-82	20.61	115.12
--	-------	-------	--------	--------	-------	-------	--------

	38-41	20.61	136.18	115.57	82-85	20.61	115.12
--	-------	-------	--------	--------	-------	-------	--------

	41-44	20.70	120.28	99.58	85-88	20.61	115.12
--	-------	-------	--------	-------	-------	-------	--------

	44-47	20.64	118.08	107.44	88-91	20.61	115.12
--	-------	-------	--------	--------	-------	-------	--------

Sample # Vial wt. Vial + S Sample

LBC 6

3-25-85
3pm - 5:30 pm

0-3"	21.05	92.88g	71.83g
3-6"	20.99g	92.95	71.96g
6-9"	20.94g	101.28g	80.34g
9-12"	20.98g	98.84g	77.86g
12-15"	20.98g	112.94g	91.96g
15-18"	20.99g	116.21g	95.22g
18-21"	20.86g	134.63	113.77g
21-24"	20.93g	114.22	93.29g
24-27"	20.86g	135.65g	114.79g
27-30"	20.97g	137.96g	116.99g
30-33"	20.77g	118.35g	97.58g
33-36"	20.61g	117.19g	96.58g
36-39"	20.71g	118.65g	97.94
39-42"	20.90g	109.57g	88.67

LBC 7

3-25/26

0-3"	21.05g	90.81g	69.76g	39-42" 20.97g	117.11g
3-6"	20.85g	91.08g	70.83g	25-27" 20.96g	119.19g
6-9"	21.02g	95.48g	74.39g	18-21" 20.58g	139.94g
9-12"	20.89g	93.88g	73.0g	12-15" 20.98g	113.34g
12-15"	20.98g	113.34g	92.36g	6-9" 21.02g	95.48g
15-18"	20.57g	130.03g	109.46	3-6" 20.85g	91.08g
18-21"	20.58g	139.94g	119.36	0-3" 21.05g	90.81g
21-24"	20.73g	120.93g	100.29	21-24" 20.73g	120.93g
24-27"	20.95g	122.98g	102.03g	15-18" 20.57g	130.03g
27-30"	21.03g	113.87g	92.84	9-12" 20.89g	93.88g
30-33"	20.95g	116.19g	96.04	3-6" 20.85g	91.08g
33-36"	20.75g	118.78g	98.03	0-3" 21.05g	90.81g
36-39"	20.88g	121.82g	101.82g	3-6" 20.85g	91.08g

<u>BLC 6</u> (cont'd)	<u>vial</u>	<u>vial + S.</u>	<u>sample</u>
42-45	20.72g	114.55g	93.83
45-48	20.72	122.40	101.68
48-51	20.64	121.34	100.70g
51-54	20.64	121.49	100.85
54-57	20.78	115.38	94.60
57-60	20.76	112.58	91.82
60-63	20.77	114.15	93.38
63-66	20.70	117.11	96.41
66-69	20.60	111.68	91.08
69-72	20.71	114.88	94.17
72-75	20.61	93.79g	73.18

Petri Dish

<u>LBC 6</u>	<u>Dish</u>	<u>Dish + S.</u>	<u>sample</u>
42-45	3.15	4.15	1.00
45-48	3.09	4.09	1.00
48-51	3.12	4.12	1.00
51-54	3.18	4.18	1.00
54-57	3.18	4.18	1.00
57-60	3.08	4.08	1.00
60-63	3.14	4.14	1.00
63-66	3.15	4.15	1.00
66-69	3.07	4.07	1.00
69-72	3.25	4.25	1.00
72-75	3.26	4.26	1.00

			<u>vial</u>	<u>vial + s</u>	<u>Sample</u>
started 9am 3-21-85	LBC 8	0"-3"	20.96	101.25	80.29g
		3"-8"	21.00	100.08	79.08
		8"-11"	21.03	115.34	94.31
	Blue	11"-14"	21.07	141.20	120.13
	Rd	11"-14"	20.90	136.54	115.64g
		14"-17"	20.93	127.52	106.59
	R	17"-20"	20.95	127.49	106.54
	Y	17"-20"	20.95	118.95	97.0g
	B	17"-20"	21.04	126.47	105.43g
		20"-23"	21.04	129.65	108.61g
		23"-26"	20.93	134.04	113.11
		26"-29"	20.93g	135.74	114.81g
		29"-32"	20.94g	131.43	110.49

			<u>vial</u>	<u>vial + s</u>	<u>Sample</u>
started 2:45 pm 3-21-85	LBC 9	0-3"	21.03g	87.84g	66.81g
		3-6"	21.02g	93.84g	72.82g
		6-12"	21.01g	119.88g	98.82
		12-15"	21.02g	130.54g	109.52
		15"-18"	21.01	125.51g	104.50g
		18"-21"	21.00g	128.10g	107.10g
		21"-24"	21.02g	131.62g	110.60g
		24"-27"	20.97g	122.88g	101.91g
		27"-31"	21.04g	122.36g	101.32g

APPENDIX VI

**FIELD LABORATORY NOTES FOR CALCULATION OF DRY
WEIGHTS OF SUBSAMPLES USED IN THE ON-SITE
SPECTRAL GAMMA ACTIVITY DETERMINATIONS**



DRY WEIGHT

	<u>Sample #</u>	<u>% mois</u>	<u>1- % mois</u>	<u>wet (sample only) Vial wt</u>	<u>Dry weight</u>
1362					
0-3	→	92%	0.08	67.12	5.36g
-6		84%	0.16	66.20	10.59g
1-9		82%	0.18	69.70	12.54
1-11		80%	0.20	70.42	14.08
-17		58%	0.42	85.53	35.92
17-20		17%	0.83	107.66	89.35
20-23		17%	0.83	103.09	85.56
23-26		19%	0.81	103.48	83.81
26-29		18%	0.82	100.66	82.54
29-32		17%	0.83	92.64	76.89
32-35		17%	0.83	95.49	79.25
35-38		17%	0.83	98.36	81.63
38-41		16%	0.84	94.04	78.99
41-44		16%	0.84	96.36	80.94
44-47		13%	0.87	87.16	75.82
47-50		16%	0.84	94.81	79.64
50-53		15%	0.85	95.82	81.44
53-56		14%	0.81	100.98	81.39
56-59		16%	0.84	95.82	80.48
59-62		15%	0.85	98.98	84.13
62-65		15%	0.85	93.09	79.72
65-68		16%	0.84	93.26	78.83
68-71		13%	0.87	93.73	81.54
71-74		9%	0.91	83.16	75.67

<u>LBC 1</u>	9-15"	60%	0.40	86.35	34.54g
	33-38"	17%	0.83	106.97	88.78g

Sample #	% mois	1- % mois	Wet sample wt (in vial)	Dry wt
----------	--------	-----------	-------------------------	--------

<u>LBC 4</u>	0-3"	84%	0.14	70.64g	11.30g
	3-6	80%	0.20	71.31	14.26
	6-9	76%	0.24	75.01	18.00g
	9-15	60%	0.40	87.48	34.99
	9-15	26%	0.74	110.72	81.93
	15-18	20%	0.80	113.29	90.63
	18-21	19%	0.81	112.04	90.75
	21-24	18%	0.82	115.92	95.05
	24-27	17%	0.83	114.42	86.66
	27-30	14%	0.81	102.28	82.84
	30-33	16%	0.84	99.19	83.31
	33-38	17%	0.83	109.62	90.98g

<u>LBC 3</u>	0-3"	82%	0.18	71.02	12.85g
	3-6	81%	0.19	72.22	13.76
	6-9	77%	0.23	76.39	15.52
	9-11	59%	0.41	70.63	21.41
	11-15	44%	0.56	73.93	32.00
	15-18	26%	0.74	110.07	31.54
	18-21	19%	0.83	101.35	83.21
	21-24	18%	0.82	101.12	81.71
	24-27	18%	0.82	96.88	72.72
	27-30	17%	0.83	100.72	83.51
	30-33	16%	0.84	98.46	82.90
	33-36	16%	0.84	98.02	82.71
	36-39	16%	0.84	101.63	85.46
	39-44	16%	0.84	97.85	83.50

	Sample	% mois	1-% mois	Wet sample wt (in vial)	Dry wt
LBC 5	0-3	86%	0.14	68.94	9.65 g
	3-6	80%	0.2	71.58	14.31
	6-9	71%	0.29	74.73	21.67
	9-14	79%	0.21	72.17	15.15
	14-17	67%	0.33	82.62	27.26
	17-20	50%	0.50	96.51	48.25
	20-23	30%	0.70	110.81	73.56
	23-26	28%	0.72	107.31	73.40
	26-29	22%	0.78	117.48	91.79
	29-32	26%	0.74	110.69	81.87
	32-35	28%	0.72	102.94	74.11
	35-38	47%	0.53	101.66	53.87
	38-41	24%	0.76	115.57	83.83
	41-44	17%	0.83	99.51	73.65
	44-47	1%	0.82	107.74	81.10
	47-50	22%	0.77	113.51	90.41
	50-53	13%	0.82	124.7	105.68
	53-56	24%	0.86	95.51	72.3
	56-59	14%	0.86	93.13	73.72
	59-64	24%	0.86	95.12	77.10

DRY WEIGHTLBC 6
Cont'dSample #% mois1- % mois

(in vial)

Wet
Sample
wt (g)Dry
wt (g)

42-45"

18%

0.82

93.83 g

76.94g

45-48

15%

0.85

101.68

86.42g

48-51

14%

0.86

100.70

86.60

51-54

13%

0.87

100.85

87.73

54-57

13%

0.87

94.60

82.30

57-60

12%

0.88

91.82

80.80

60-63

12%

0.88

93.38

82.17

63-66

14%

0.86

96.41

82.91

66-69

13%

0.87

91.08

79.23

69-72

12%

0.88

94.17

82.86

72-75

11%

0.89

73.18g

65.13g

LBC 7

39-42"

16%

0.84

96.19g

80.79g

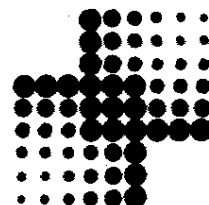
	Sample #	% mois	1- % m.	Vial wet wt.	Dry wt.
<u>LBC 6</u>	0-3"	87%	0.13	71.83	9.33g
	3-6	86%	0.14	71.96	10.07g
	6-9	75%	0.25	80.34	20.05
	9-12	74%	0.26	77.86	20.24
	12-15	49%	0.51	91.96	46.89
	15-18	46%	0.54	95.22	51.41
	18-21	20%	0.80	113.77	91.01
	21-24	16%	0.84	93.29	78.36
	24-27	22%	0.78	114.79	89.53
	27-30	21%	0.79	116.99	92.32g
	30-33	15%	0.85	97.58	82.94
	33-36	15%	0.85	96.58	82.69
	36-39	14%	0.86	97.94	84.22
	39-43	13%	0.87	88.67	73.14
<u>LBC 7</u>	0-3	86%	0.14	89.76g	15.75g
	3-6	80%	0.20	70.83	20.11g
	6-9	79%	0.21	74.39	15.67
	9-12	67%	0.33	73.0	14.09
	12-15	45%	0.55	92.36	50.79
	15-18	31%	0.69	109.96	75.82
3-28-84	18-21	23%	0.77	119.36	59.90
	21-24	19%	0.81	100.20	53.22
	24-27	17%	0.83	102.03	54.68
	27-30	19%	0.81	102.84	55.20
	30-33	16%	0.84	96.04	50.62
	33-36	15%	0.84	118.03	52.34
	36-39	14%	0.84	118.03	52.34

DRY WEIGHT

	SL. no.	% mois.	1 - % mois.	vial wet wt.	Dry wt.
82.8	0-3	84%	0.16	80.29g	12.84 g
	3-6	78%	0.22	79.08	17.39
	6-11	25%	0.75	94.31	70.73
	11-14	23%	0.77	120.13	92.50
	14-17	20%	0.80	115.64	92.51
	17-20	20%	0.80	106.59	85.27
	20-23	17%	0.83	106.54	88.42
	23-26	16%	0.84	97.09	81.48
	26-29	19%	0.81	105.43g	85.39
	29-32	16%	0.84	108.61g	91.23
	32-35	15%	0.85	113.11g	96.14
	35-38	15%	0.85	114.81	97.58
	38-41	14%	0.86	110.49g	95.02

LBG 9	0-3	87%	0.13	66.81g	8.68 g
	3-6	79%	0.21	72.82g	15.29g
	6-12	36%	0.64	98.87	63.27
	12-15	20%	0.80	109.52	87.61
	15-18	17%	0.83	104.50	86.73
	18-21	16%	0.84	107.10	89.96
	21-24	18%	0.82	110.60	90.69
	24-27	15%	0.85	101.91	86.62
	27-31	18%	0.82	101.32	83.08

APPENDIX VII
FIELD LABORATORY NOTES FOR THE PREPARATION OF
DRIED SUBSAMPLES FOR THE ON-SITE GROSS ALPHA
AND BETA ACTIVITY DETERMINATION



GAUSS Alpha and Beta Analysis

wt. ☐ Petri Dish ☐ Dish ☐ Sample Wt. ☐ Sample wt.

LBC 3	Petri Dish	Dish	Sample Wt.	Sample wt.
0-3"	3.19	4.19	1.00g	
3-6	3.18	4.18	1.00g	
6-9	3.16	4.16	1.00g	
9-11	3.16	4.16	1.00g	
11-15	3.16	4.16	1.00g	
15-18	3.07	4.07	1.00g	
18-21	3.13	4.13	1.00g	
21-24	3.12	4.12	1.00g	
24-27	3.07	4.07	1.00g	
27-30	3.14	4.14	1.00	
30-33	3.16	4.16	1.00	
33-36	3.09	4.09	1.00	
36-39	3.12	4.12	1.00	
39-44	3.16	4.16	1.00	

LBC 1

0-3"			
3-6			
6-9			
9-15	3.18	4.18	1.00
15-18			
18-21			
21-24			
24-27			
27-30			
30-33			
33-38	3.17	4.17	1.00

LBC 2	Perm Dish	Dish + sample	Sample
0-30	3.18	3.86g	0.68g
3-6	3.08	4.08	1.00g
6-9	3.09	4.09	1.00g
9-11	3.15	4.15	1.00g
11-17	3.15	4.15	1.00
17-20	3.07	4.07	1.00
20-23	3.09	4.09	1.00
23-26	3.16	4.16	1.00
26-29	3.13	4.13	1.00
29-32	3.19	4.19	1.00
32-35	3.18	4.18	1.00
35-38	3.18	4.18	1.00
38-41	3.19	4.19	1.00
41-44	3.09	4.09	1.00
44-47	3.19	4.19	1.00
47-50	3.15	4.15	1.00
50-53	3.20	4.20	1.00
53-56	3.07	4.07	1.00
56-59	3.12	4.12	1.00
59-62	3.18	4.18	1.00
62-65	3.19	4.19	1.00
65-68	3.17	4.17	1.00
68-71	3.15	4.15	1.00
71-74	3.10	4.10	1.00

Petri Dish
 LBC 4 c.c. Dish
 Dish + Sample
 sample wt.

0-3	3.18g	3.90g	1.00g
3-6	3.06g	3.90g	1.00g
6-9	3.09g	4.09g	1.00g
9-15	3.13g	4.13g	1.00g
15-18	3.09g	4.08g	1.00g
18-21	3.15g	4.15g	1.00g
21-24	3.08g	4.08g	1.00g
24-27	3.20g	4.20g	1.00g
27-30	3.19g	4.19g	1.00g
30-33	3.12g	4.12g	1.00g
33-38	3.08g	4.08g	1.00g

	Dish	D+S	S
LBC5 Q-3	3.18	4.18	1.00g
3-6	3.18	4.18	1.00
6-9	3.17	4.17	1.00
9-14	3.17	4.17	1.00
14-17	3.07	4.07	1.00
17-20	3.15	4.15	1.00
20-23	3.08	4.08	1.00
23-26	3.14	4.14	1.00
26-29	3.15	4.15	1.00
29-32	3.17	4.17	1.00
32-35	3.17	4.17	1.00
35-38	3.17	4.17	1.00
38-41	3.15	4.15	1.00
41-44	3.08	4.08	1.00
44-47	3.18	4.18	1.00

1 gm into Petri Dish

LBC u Petri Dish Dish + sample sample 3-26-85
8-9:30am

0-3	3.06g	4.06g	1.00g
3-6	3.13g	4.13g	1.00g
6-9	3.15g	4.15g	1.00g
9-12	3.17g	4.17g	1.00g
12-15	3.17g	4.17g	1.00g
15-18	3.19g	4.19g	1.00g
18-21	3.16g	4.16g	1.00g
21-24	3.15g	4.15g	1.00g
24-27	3.16g	4.16g	1.00g
27-30	3.08g	4.08g	1.00g
30-33	3.16g	4.16g	1.00g
33-36	3.15g	4.15g	1.00g
36-39	3.16g	4.16g	1.00g
39-42	3.17g	4.17g	1.00g

3-27-85 1:30pm

LBC f 0-3 3.19g 4.19g 1.00g 3-27-85 2pm

3-6	3.09g	4.09g	1.00g
6-10	3.15g	4.15g	1.00g
10-12	3.12g	4.12g	1.00g
12-15	3.13g	4.13g	1.00g
15-18	3.18g	4.18g	1.00g
18-21	3.16g	4.16g	1.00g
21-24	3.16g	4.16g	1.00g
24-27	3.13g	4.13g	1.00g
27-30	3.09g	4.09g	1.00g
30-33	3.19g	4.19g	1.00g
33-36	3.18g	4.18g	1.00g
36-39	3.13g	4.13g	1.00g

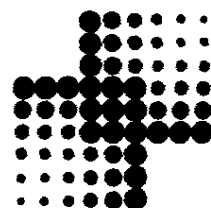
3-28-85 9am

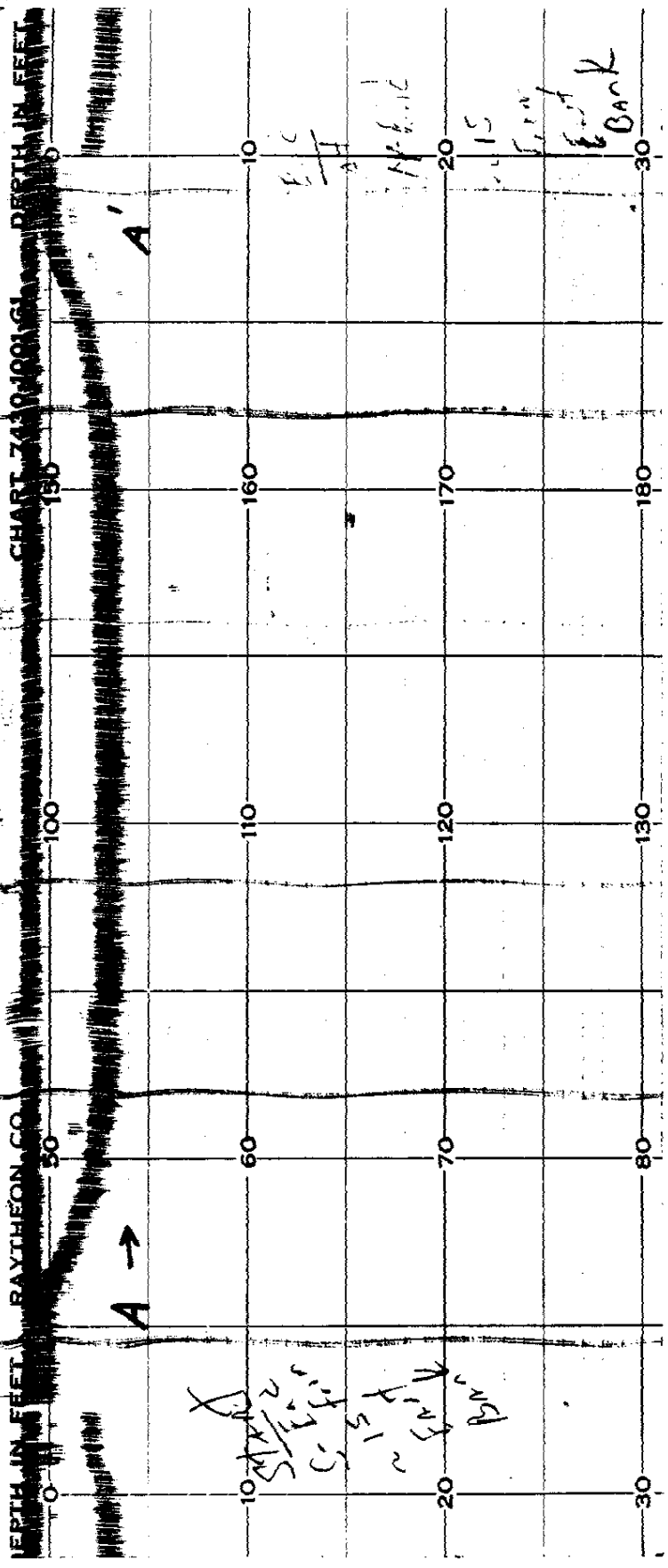
lgm Into Petri Dish

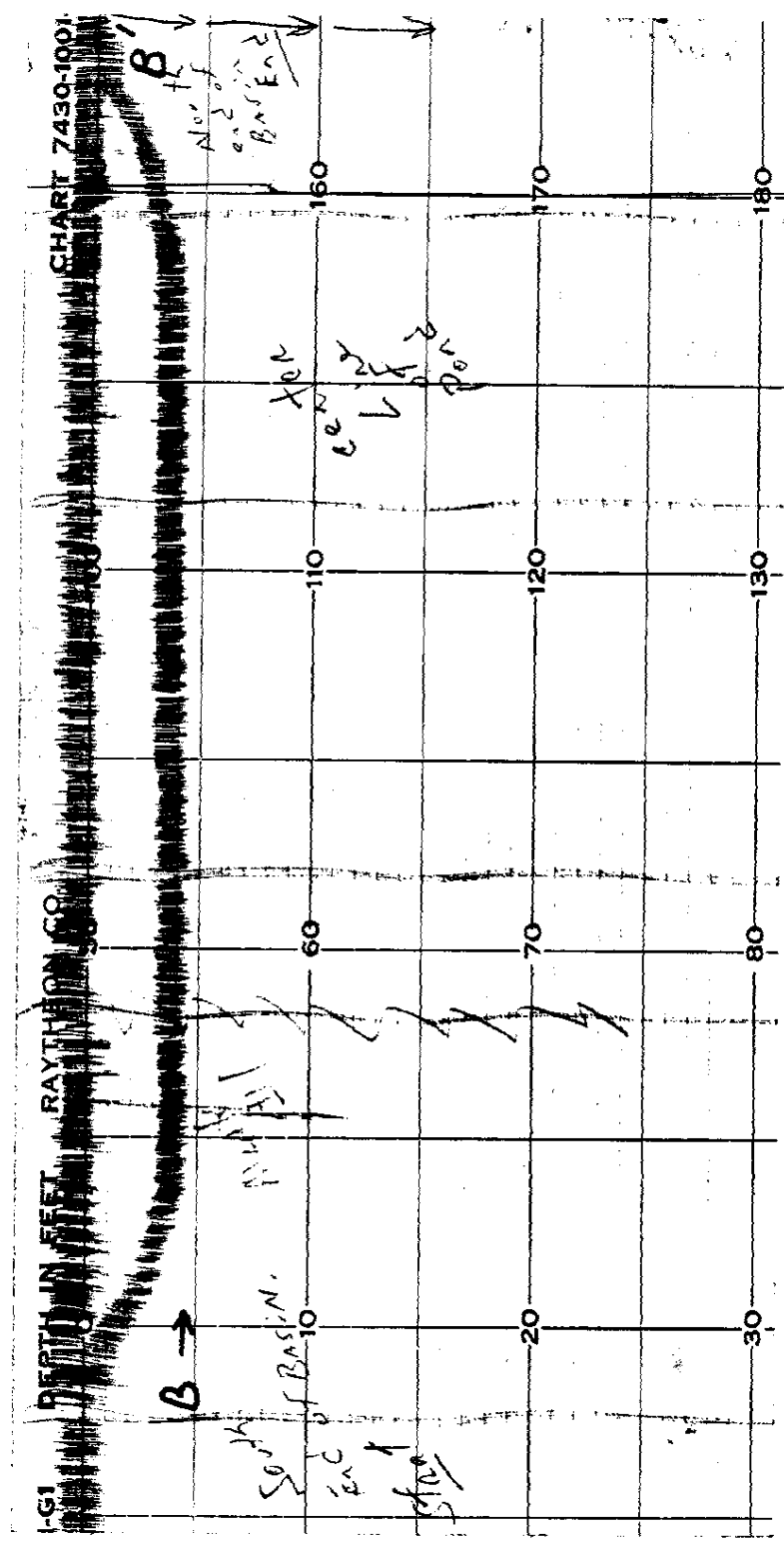
<u>LBC8</u>	<u>Petri Dish</u>	<u>Dish + sample</u>	<u>Sample</u>	3-22-85 9:30am - 11:30am
0-3"	3.12g	3.89g	0.770g	
3-18"	3.08g	4.03g	0.95g	
8-11"	3.08g	4.08g	1.00g	
11-14" B	3.18g	4.18g	1.00g	
14-14" R	3.19g	4.19g	1.00g	
14-17"	3.11g	4.16g	1.00g	
17-20" R	3.14g	4.14g	1.00g	
20-20" Y	3.15g	4.15g	1.00g	
20-20" B	3.08g	4.08g	1.00g	
20-23"	3.14g	4.14g	1.00g	
23-26"	3.15g	4.15g	1.00g	
26-29"	3.16g	4.16g	1.00g	
29-32"	3.08g	4.08g	1.00g	

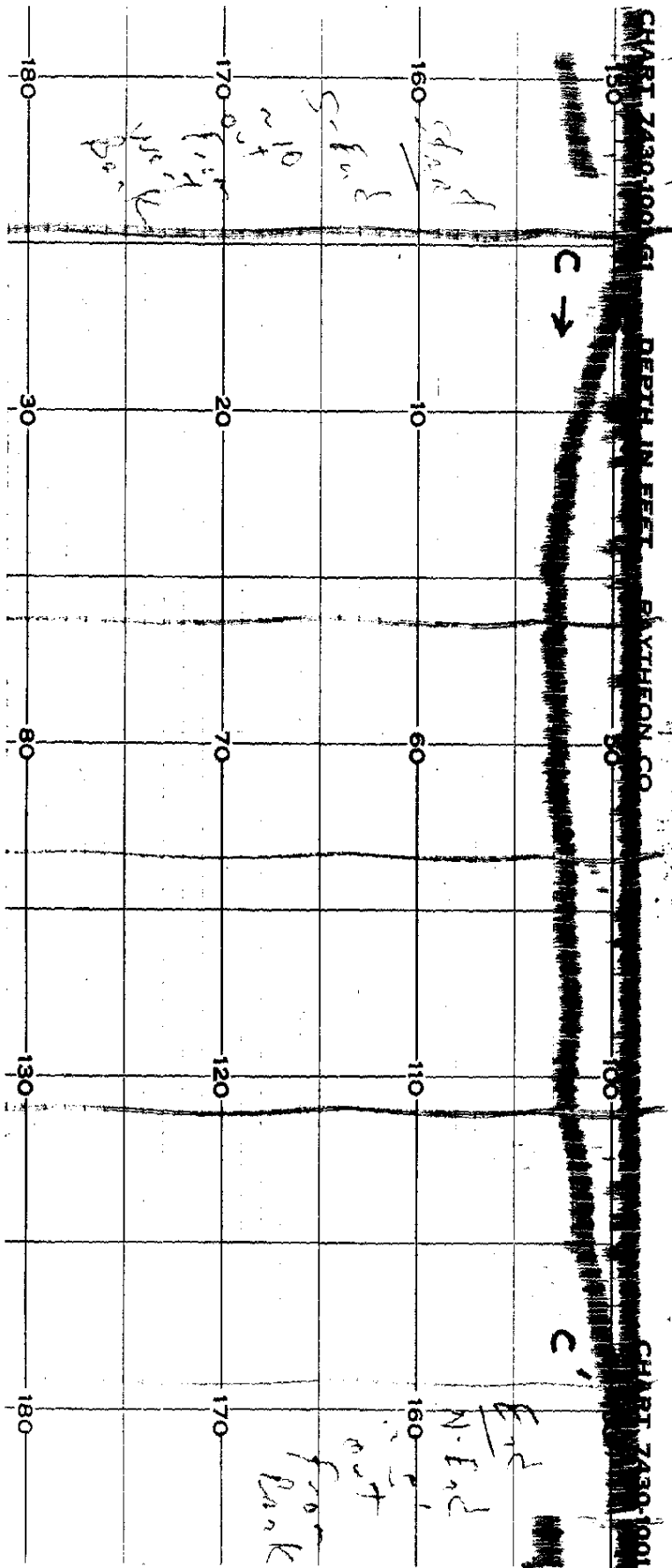
<u>LBC9</u>	<u>Petri Dish</u>	<u>Dish + sample</u>	<u>Sample</u>	3-22-85 noon -
0-3"	3.08g	3.70g	0.62g	
3-6"	3.12g	4.06g	0.89g	
6-12"	3.18g	4.18g	1.00g	
12-15"	3.19g	4.19g	1.00g	
15-18"	3.10g	4.10g	1.00g	
18-21"	3.20g	4.20g	1.00g	
21-24"	3.19g	4.19g	1.00g	
24-27"	3.14g	4.14g	1.00g	
27-31"	3.09g	4.09g	1.00g	

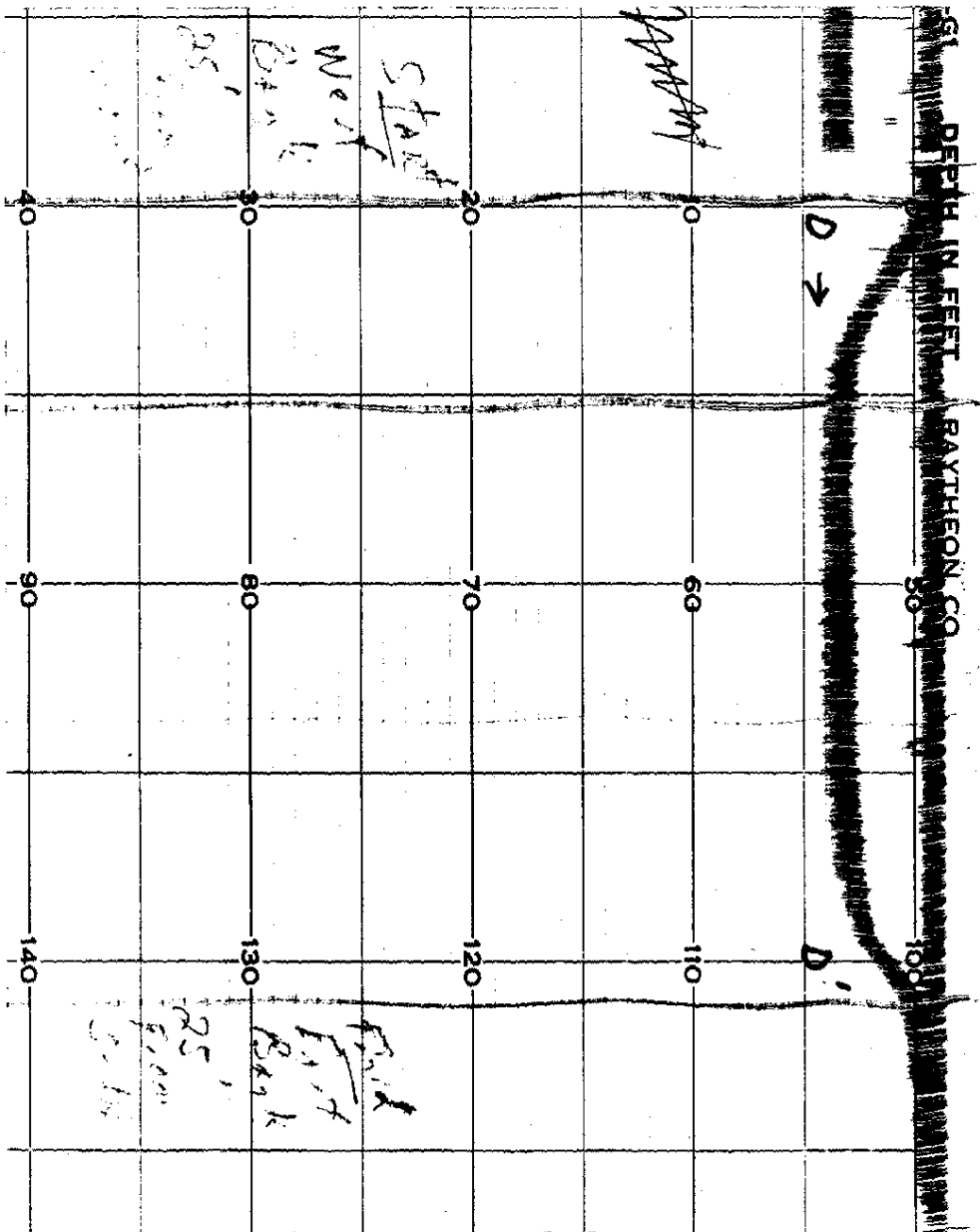
APPENDIX VIII
FATHOMETER TRACES

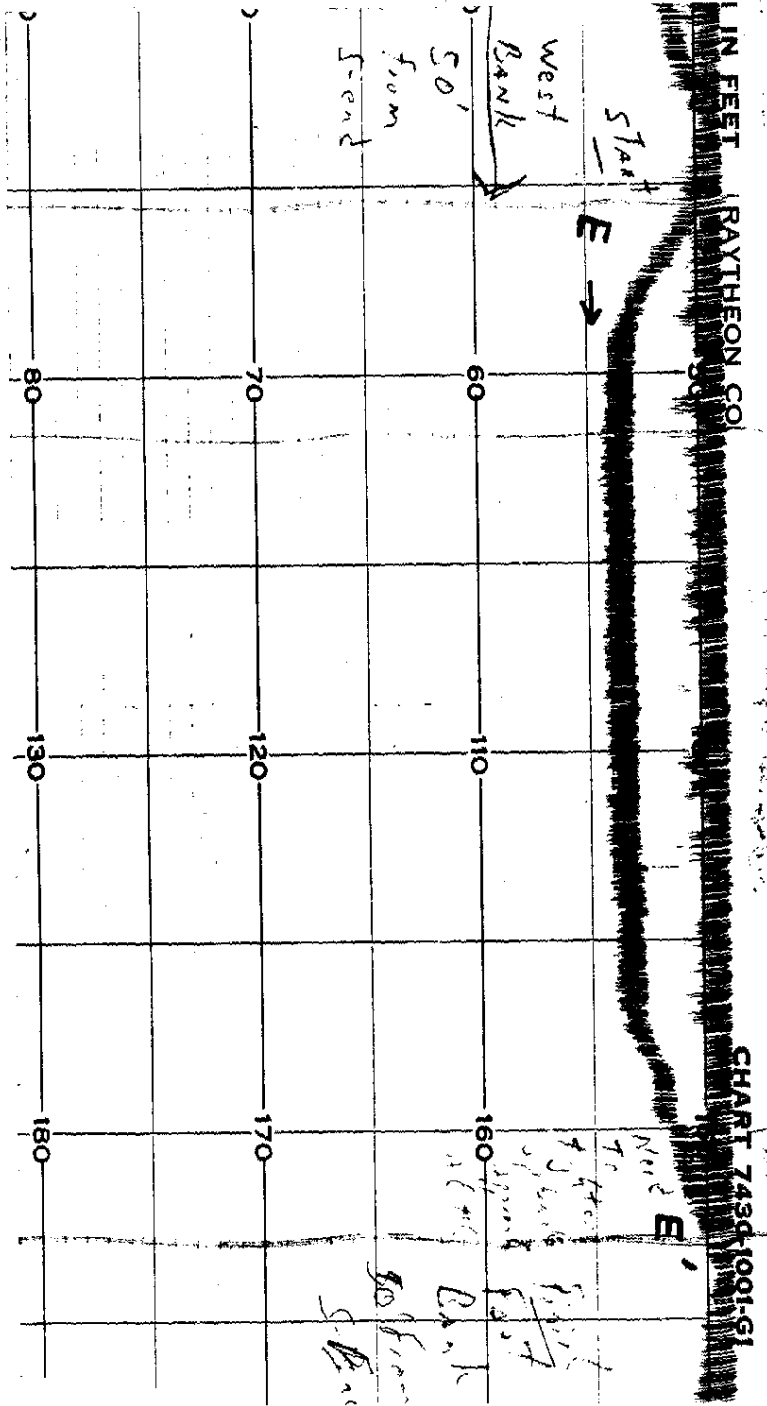


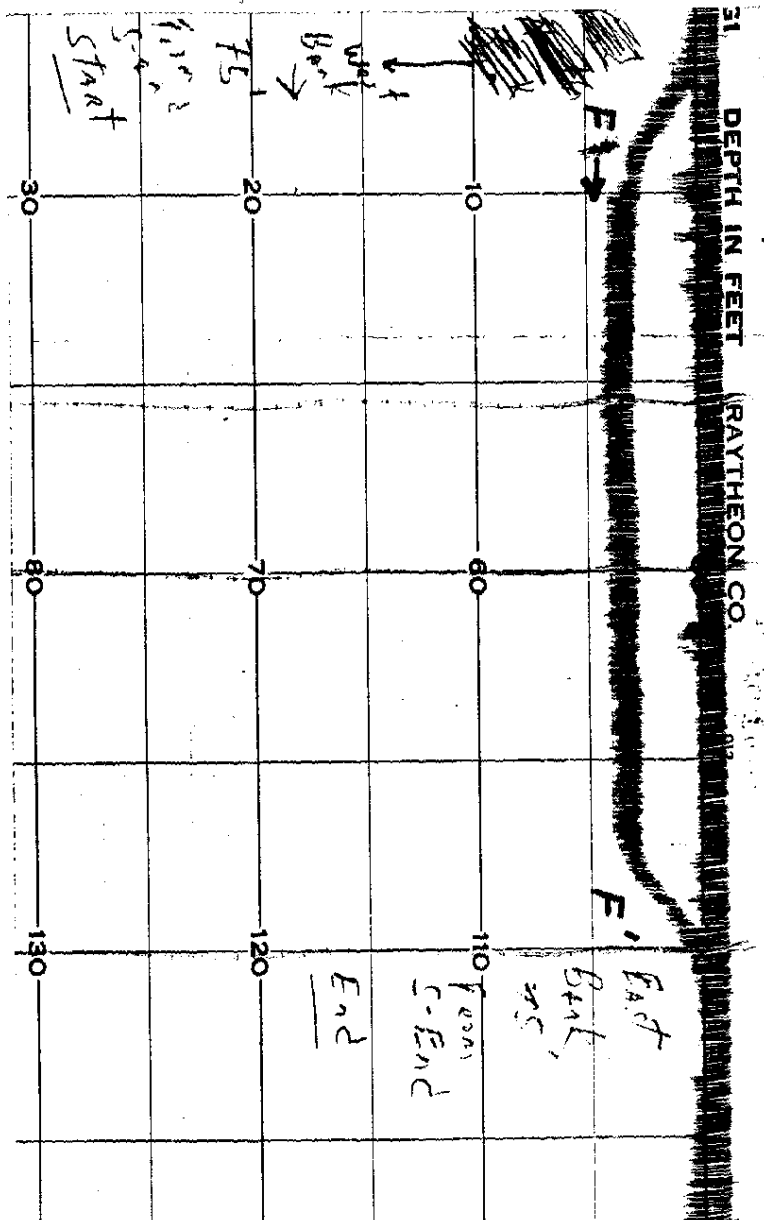


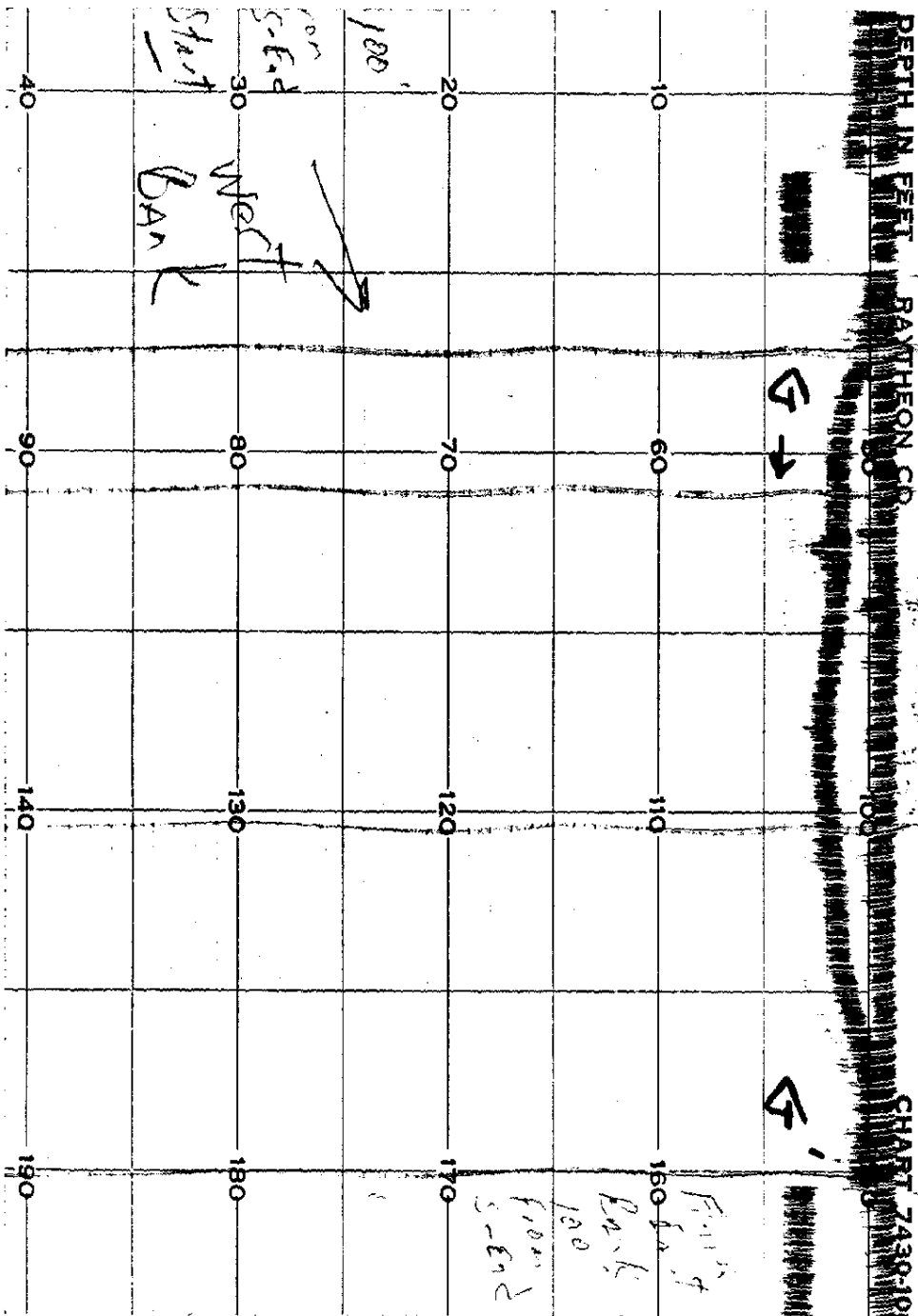




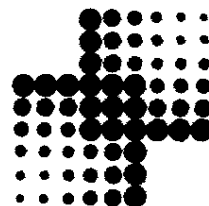








APPENDIX IX
LAB NOTES/COPIES OF GC
CHROMATOGRAMS FOR ALL RE-RUNS
AND FOR PHC ANALYSIS



8-19-85 2086-00110 Dupont Petroleum Hydrocarbons/soil
 Returns (see pg 134-136)

GC#2 Col # 324

Operator <u>DOK</u>	Date <u>8/14/85</u>
Column	Detector <u>FID</u>
Length <u>30 m</u>	Range
Dia. <u>0.150 mm</u>	Atten. <u>294</u>
Liquid Phase <u>SPB-5</u>	Flow Rates. ml/min.
Wt. % <u>1 μm film thickness</u>	Hydrogen <u>30</u> Air <u>300</u>
Support	Scavenge
Mesh	Split
Carrier Gas <u>He</u>	Temperature, °C
Rotameter	Det. <u>250</u> Inj. <u>225</u>
Inlet Press <u>3.5</u> psig	Column Initial <u>35-40</u>
Rate <u> </u> ml/min	Final <u>300</u>
CHART SPEED <u>0.50 cm/min</u>	Rate <u>8</u>
SAMPLE <u>soil</u>	Solvent <u>CH₂Cl</u>
Size <u>4 μ</u>	Concn.

Extraction Procedure: 10g of sample was extracted (Soxhlet) with 250 ml Methylene chloride. The extract was concentrated to 10ml in methylene chloride.

The extract concentrated were analyzed by GC/FID @ the above conditions. Injection volume was 4 μ l in order to achieve maximum sensitivity.

✓) Blank CH₂Cl₂

Pks at 3.16, 4.43, 5.40 will not be included in Standard response calculation.

✓) Naphtha 264 μ g/ml # 1661-1

<u>R.T.</u>	<u>Total Response #2</u>	<u>#14</u>	<u>#26</u>	<u>#35</u>
5.71-13.68	305895	361781	286504	299069

<u>Response</u>	<u># of peaks</u>
313312	42

David D. Kornegay 8-19-85
 JAW 8-20-85

8-19-85 2086-110 Dupont Pot. Hydrocarbons/soil Reruns cont'd

Detection Limit Calculation

$$\frac{(42)(500)}{313312} \times 264 \mu\text{g/ml} \times \frac{10 \text{ ml/kg}}{10^8} \times \frac{24}{24} = 4.42 \mu\text{g/g}$$

* 8/19/85

✓ 3) M. Blank E.S. # 3913 Ext'd 6/3/85

✓ 4) 76110/18-21

✓ 5) 76111/21-24

✓ 6) 76112/24-27

✓ 7) 76113/27-30

✓ 8) 76114/30-33

✓ 9) 76115/33-36

✓ 10) 76116/36-39

all cmpds < DL

✓ 11) 76117/39-43

✓ 12) 76118/48-51

all cmpds < DL

* ✓ 13) Blank CH_2Cl_2 ✓ 14) Naphtha 264 $\mu\text{g/ml}$ # 1661-1

✓ 15) 76119/54-57

✓ 16) 76120/60-63

✓ 17) 76121/66-69

✓ 18) 76122/72-75

all cmpds < DL

✓ 19) M. Blank E.S. # 3918 Extracted 6-5-85

all cmpds < DL

Jan J. Kornegay 8-19-85
 SHN 8-20-85

8-19-85 2086-110 Dupont Pet. Hydrocarbons/soil Runs contd.

- ✓20) 76110 A / 18-21
 ✓21) 76110 B / "
 ✓22) 76111 A / 21-24
 ✓23) 76111 B / "
 ✓24) 76112 A / 24-27
- } all cmpds < DL

✓25) Blank CH_2Cl_2

✓26) Naphtha 264 $\mu\text{g/ml}$ #1661-1

- ✓27) 76112 B / 24-27
 ✓28) 76113 A / 27-30
 ✓29) 76113 B / "
 ✓30) 76114 A / 30-33
 ✓31) 76114 B / "
 ✓32) 76115 A / 33-36
 ✓33) 76115 B / "
- } all cmpds < DL

✓34) Blank CH_2Cl_2

✓35) Naphtha 264 $\mu\text{g/ml}$ #1661-1

DOK 8/19/85
~~Temp 1 46.8°C~~

David O. Koneguy 8/19/85
 JAW 8-20-85

8-19-85 2086-110 Dupont Pet Hydrocarbons/soil
Reruns

Conditions as on pg. 157. Temp 1 = 40°C.
Run date 8/16/85. DOK.

✓ 1) Blank CH_2Cl_2
Peaks at 2.89, 5.04 will not be included in
standard response calculation.

✓ 2) Naphtha 264 $\mu\text{g}/\text{ml}$ #1661-1

RT	Total Response #2	# 14	# 26
5.30-13.05	270566/40 pks	253429/31 pks	273560/ 301828 /39 pks

<u>Response</u>	<u># of pks</u>
265852	39

$$\left(\frac{39 \times 500}{265852} \right) \times \left(\frac{264 \mu\text{g}}{\text{ml}} \right) \times \left(\frac{10 \text{ ml}}{10 \text{ g}} \right) \times \left(\frac{2 \text{ ml}}{2 \text{ ml}} \right) = 4.8 \mu\text{g/g}$$

DL

✓ 3) 76116 A / 36-39"

✓ 4) 76116 B / "

✓ 5) 76117 A / 39-43"

✓ 6) 76117 B / "

✓ 7) M. Blank ES, # 3912 Extracted 6-10-85

✓ 8) 76110 / 18-21"

✓ 9) 76111 / 21-24"

✓ 10) 76112 / 24-27"

✓ 11) 76113 / 27-30"

✓ 12) 76114 / 30-33"

all compds < DL

✓ 13) Blank CH_2Cl_2

✓ 14) Std Naphtha 264 $\mu\text{g}/\text{ml}$ #1661-1

David O'Haregoy 8-20-85
SRW 8-20-85

8-20-85 2086-110 Dupont Pet. Hydrocarbons /soils reruns cont'd.

✓15) 76115/33-36"

✓16) 76116/36-39"

✓17) 76117/39-43"

✓18) 76119/54-57"

✓19) 76120/60-63"

✓20) 76121/66-69"

✓21) M. Blank E.S. # 3933 Extracted 6-20-85

} all cmpds < DL

✓22) 76949/76959

✓23) 76950/76960

✓24) 76955/76965

✓25) Blank CH_2Cl_2

} Pet. Hydrocarbons < DL

Samples contain high boiling cmpds that are not petroleum hydrocarbons.

✓26) Naphtha 264 $\mu\text{g/ml}$ #1661-1

✓27) 76956/76966

✓28) 76957/76967

✓29) 76958/76968

✓30) 76957/76967 DUP.

✓31) 76958/76968 DUP

} Pet. Hydrocarbons < DL

Samples contain high boiling cmpds that are not pet. hydrocarbons.

✓32) Blank CH_2Cl_2

lost air (tank depleted) flame out.

✓33) Naphtha 264 $\mu\text{g/ml}$ #1661-1 do not use.

A. J. Okuney 8/20/85
SPW 8-20-85

13.64

1682

0.001

DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
 TEMP1 400 35 35
 TIME1 4.00
 RATE 8.00
 TEMP2 400 300
 TIME2 20.00
 INJ TEMP 400 225 225
 FID TEMP 400 250 250
 AUX TEMP 400 225 225

CHT SPD 0.50
 ZERO 15.0
 ATTN 2+ 4
 FID SGNL +B
 SLP SENS 0.10
 AREA REJ 1000
 FLOW A 0.0 28.0
 FLOW B 0.0 43.7

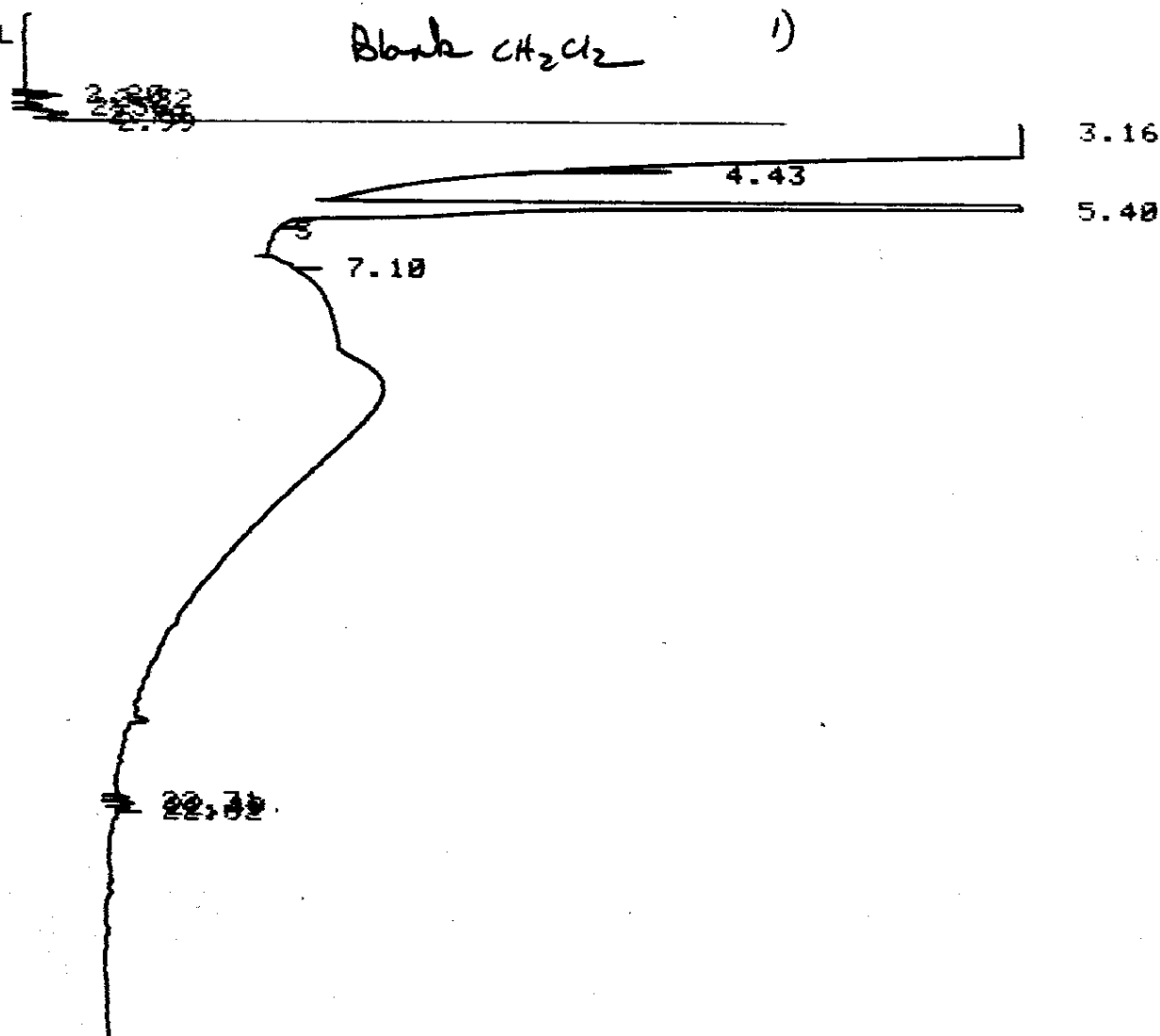
0.50 VLV/EXT- 1
 20.00 STOP

DELETE MIN? 2 0 0

STOP MIN? 3 0 0

CHANGE RUN 0 0

START VL



8/14/85

2086-110

Dupont

Pet. Hydrocarbons/soil
 Reruns

Col #324

DOK

4ul inj attn = 2x4

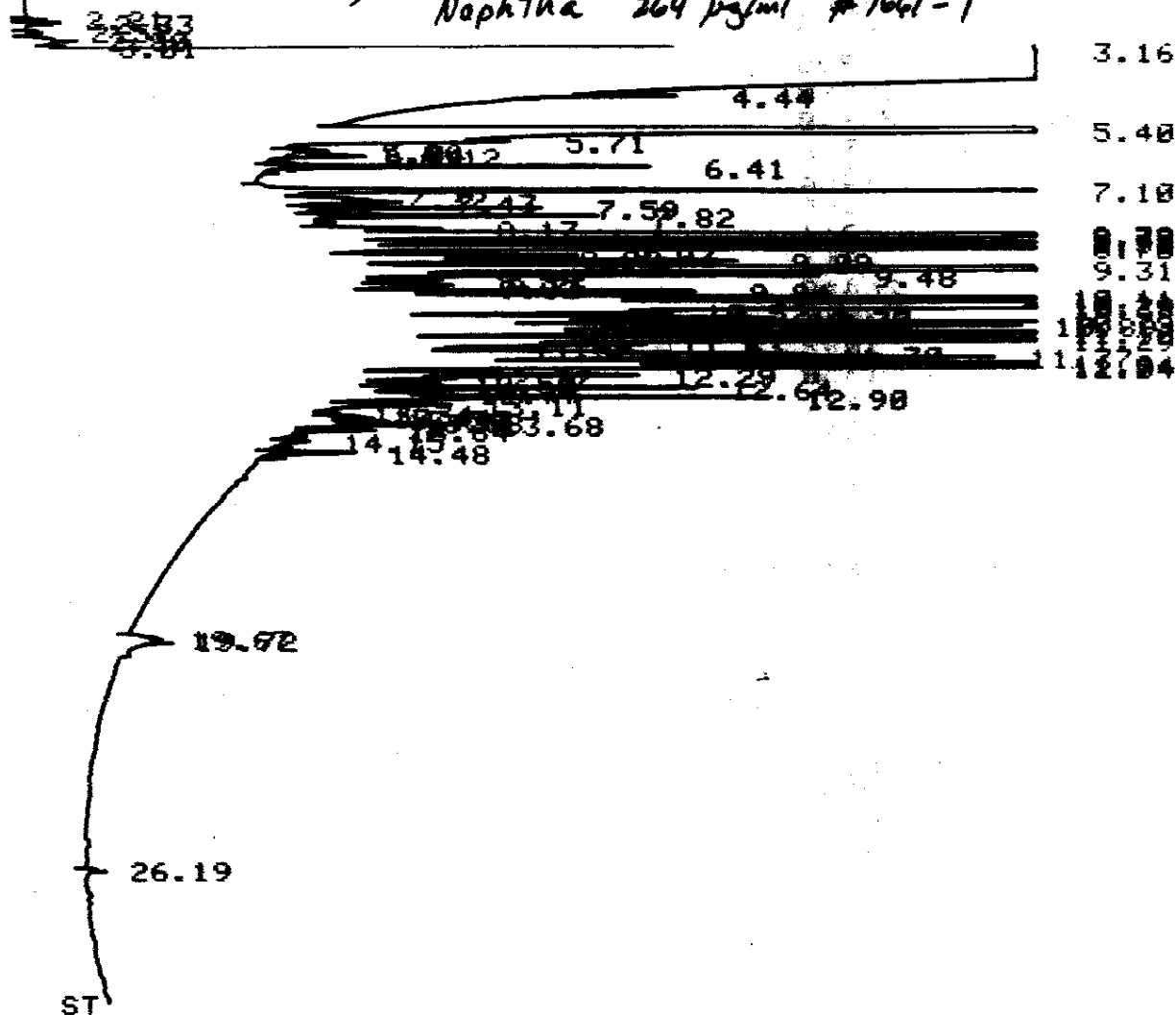
HP RUN # 1
BOTTLE 1
AREA %

RT	AREA	AREA %
3.16	154600000	99.871
5.40	199600	0.129

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VL

2) Nophta 264 µg/ml #1661-1



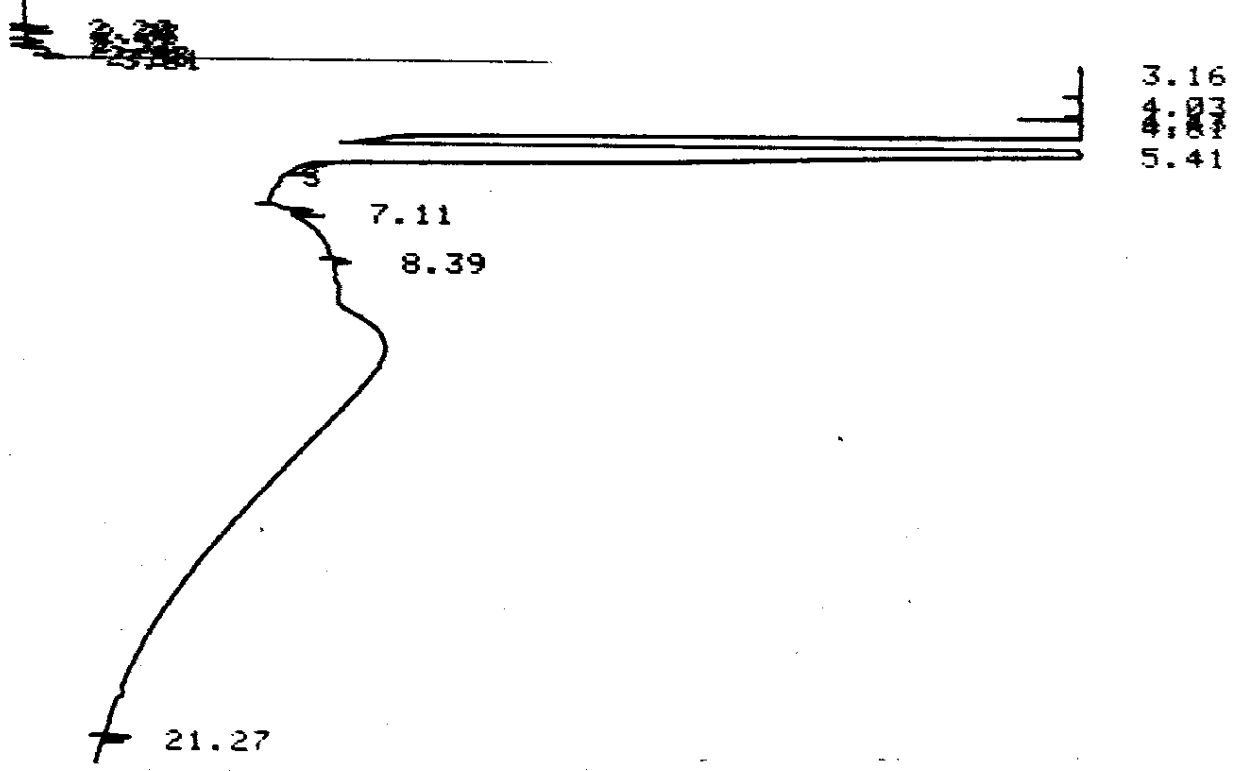
HP RUN # 2
BOTTLE 2
AREA %

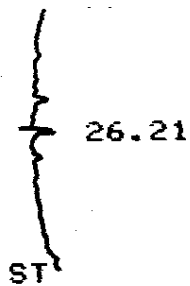
RT	AREA	AREA %
3.16	154400000	99.675
5.40	197300	0.127
5.71	2294	0.001
6.41	2593	0.002
7.10	9098	0.006
7.43	1329	0.001
7.59	1731	0.001
7.82	1994	0.001

8.32	9146	0.006
8.39	6750	0.004
8.55	7204	0.005
8.70	14330	0.009
8.93	1899	0.001
8.98	1141	0.001
9.09	3716	0.002
9.31	38520	0.025
9.48	3804	0.002
9.59	1424	0.001
9.76	1181	0.001
9.94	3002	0.002
10.11	11880	0.008
10.29	19770	0.013
10.38	15070	0.010
10.52	3008	0.002
10.70	5025	0.003
10.79	5664	0.004
10.89	5177	0.003
10.97	3305	0.002
11.10	19510	0.013
11.29	35240	0.023
11.43	3712	0.002
11.55	1362	0.001
11.70	5166	0.003
11.77	7214	0.005
11.94	7914	0.005
12.04	26410	0.017
12.29	3747	0.002
12.37	1082	0.001
12.50	1350	0.001
12.64	3431	0.002
12.77	1244	0.001
12.90	4416	0.003
13.11	1538	0.001
13.68	1420	0.001

DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
 START VL
 M. Blanc ES. # 3913 Exp'd 6-3-85





HP RUN # 3
BOTTLE 3
AREA %

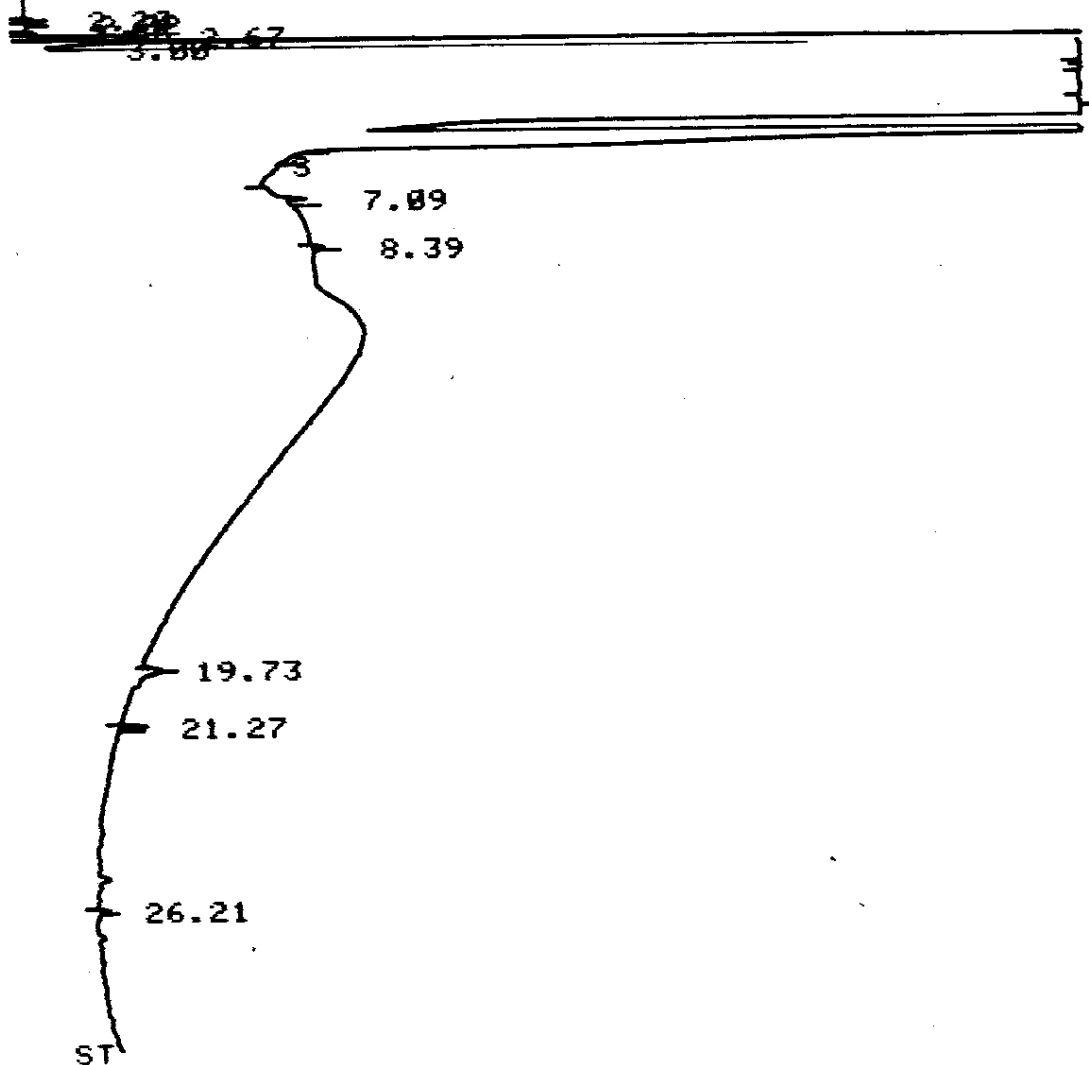
RT	AREA	AREA %
3.16	148400000	95.391
4.03	5200000	3.343
4.43	12760	0.008
4.61	1502000	0.965
5.41	455800	0.293

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VL

4) 76110

18-21"



4.43
5.41
6.43
7.43
8.43
9.43
10.43
11.43
12.43
13.43
14.43
15.43
16.43
17.43
18.43
19.43
20.43
21.43
22.43
23.43
24.43
25.43
26.43
27.43
28.43
29.43
30.43
31.43
32.43
33.43
34.43
35.43
36.43
37.43
38.43
39.43
40.43
41.43
42.43
43.43
44.43
45.43
46.43
47.43
48.43
49.43
50.43
51.43
52.43
53.43
54.43
55.43
56.43
57.43
58.43
59.43
60.43
61.43
62.43
63.43
64.43
65.43
66.43
67.43
68.43
69.43
70.43
71.43
72.43
73.43
74.43
75.43
76.43
77.43
78.43
79.43
80.43
81.43
82.43
83.43
84.43
85.43
86.43
87.43
88.43
89.43
90.43
91.43
92.43
93.43
94.43
95.43
96.43
97.43
98.43
99.43
100.43

HP RUN # 4
BOTTLE 4

AREA %

RT	AREA	AREA %
2.82	16850	0.007
3.13	131400000	57.673
3.65	292300	0.128
3.76	689000	0.302
4.19	87200000	38.273
4.66	7856000	3.448
5.40	382200	0.168

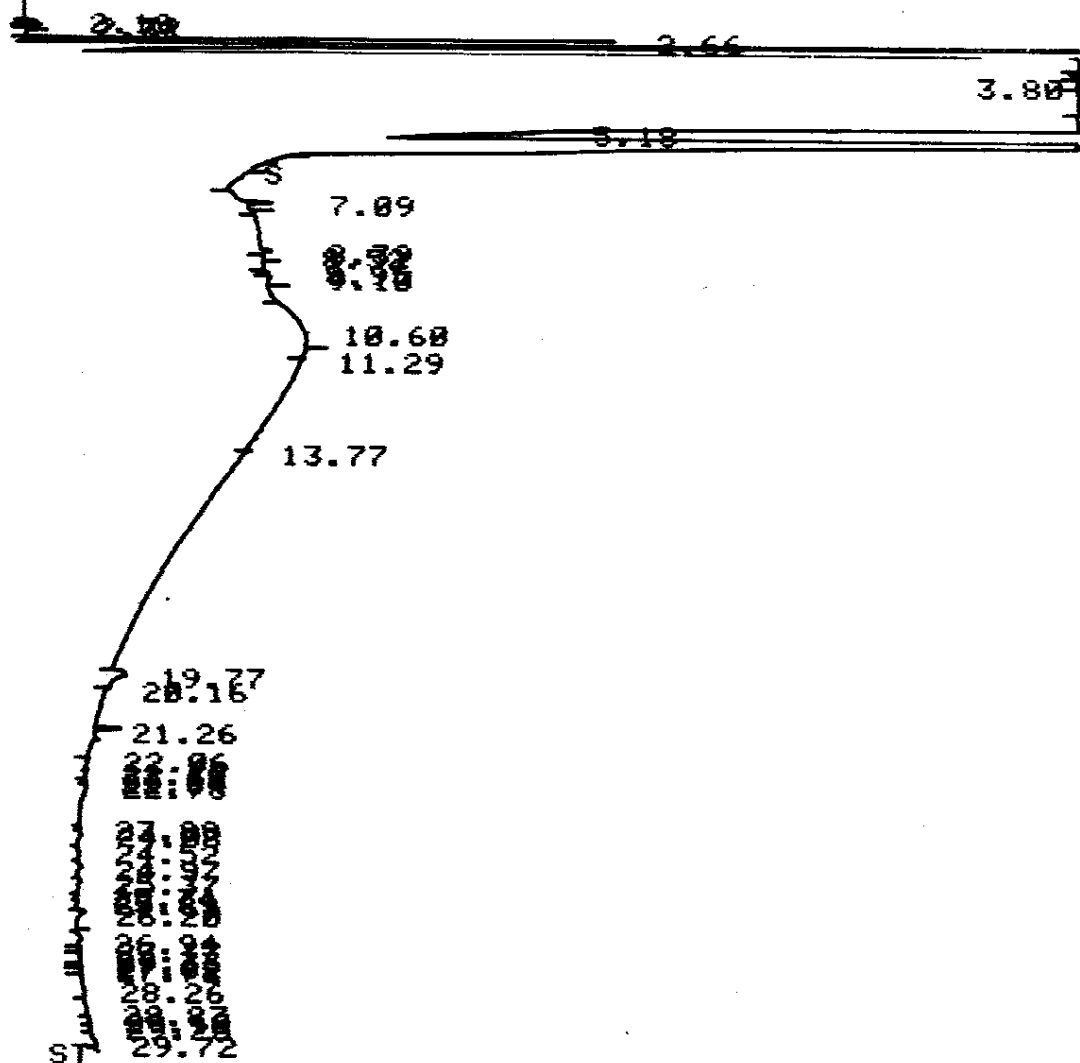
DIL FACTOR: 1.0000 E+ 0

ATTN 2↑
START VL

4

5) 76111

21-24'



4.00
4.40
5.42

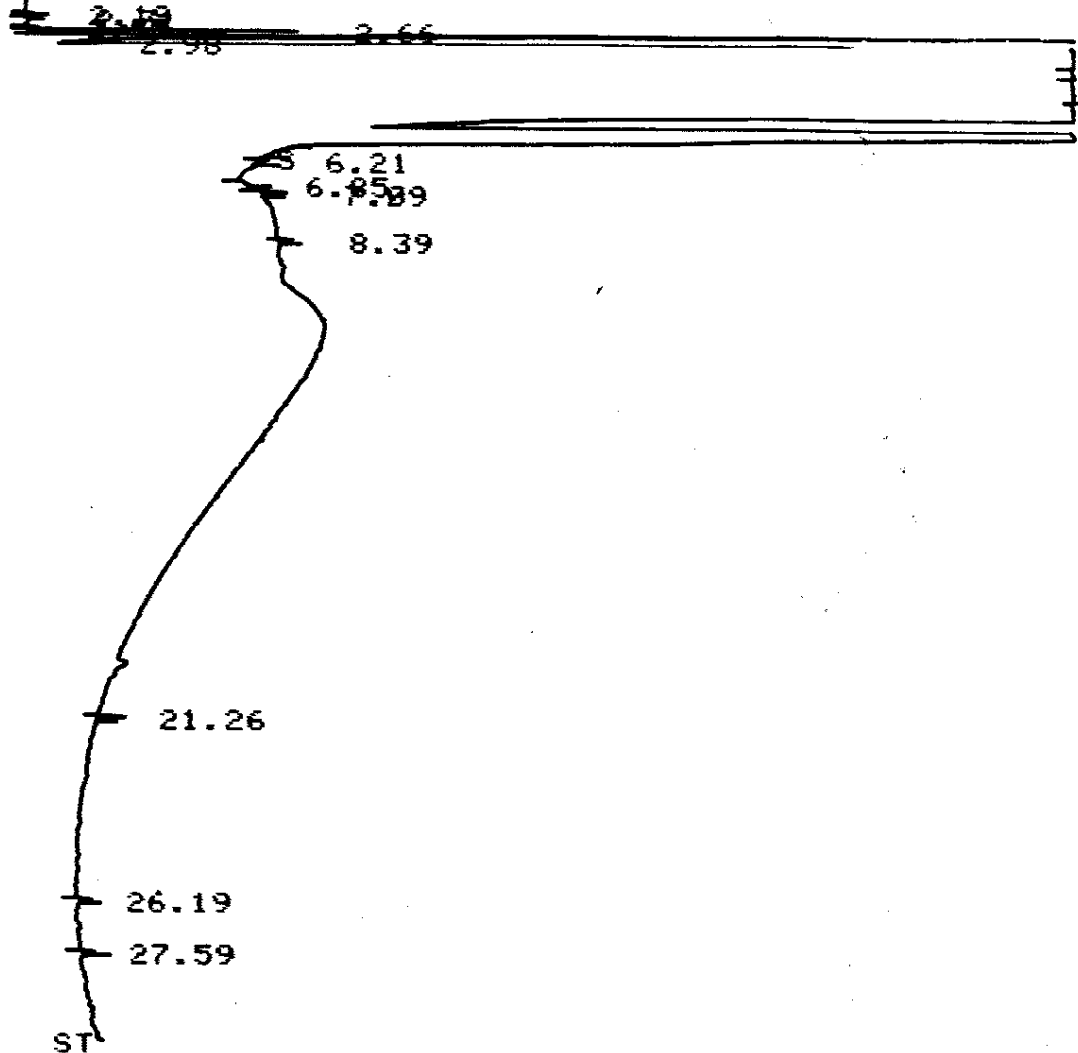
HP RUN # 5
BOTTLE 5
AREA %

RT	AREA	AREA %
2.66	2089	0.000
2.81	74420	0.017
3.07	73440000	17.190
3.43	31020	0.007
3.52	124700	0.029
3.80	3769000	0.882
4.40	318800000	74.620
4.70	704000000	17.177

ATTN 21 4
START YLI

6) 76112

24-27



THE UNIVERSITY OF

HP RUN # 6
BOTTLE 6
AREA %

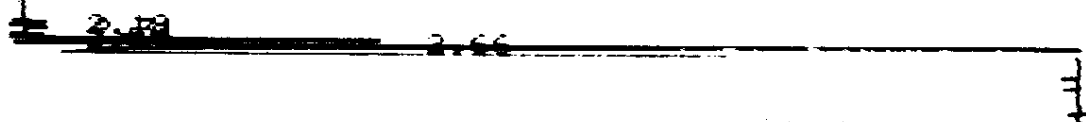
RT	AREA	AREA %
2.81	35700	0.811
3.10	106300000	32.718
3.79	2110000	0.649
4.31	196400000	60.450
4.73	19510000	6.005
5.41	539300	0.166

DIL FACTOR: 1.0000 E+ 0

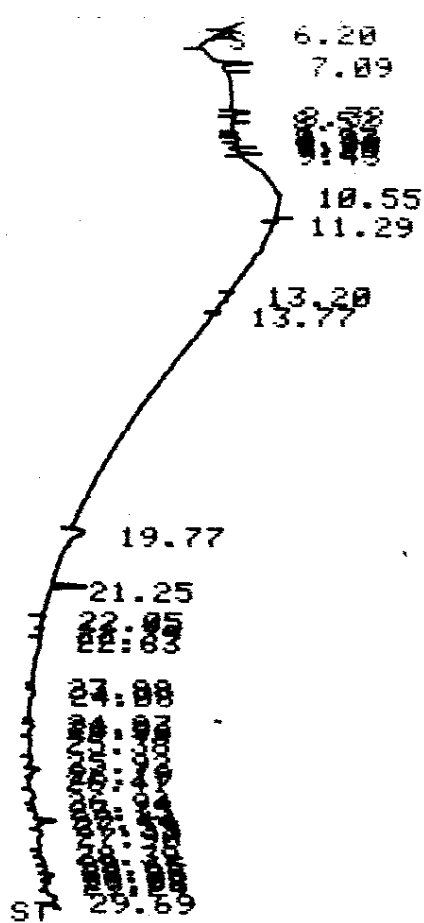
ATTN 21 4
START VL

7) 76113

27-30."



10-11-12

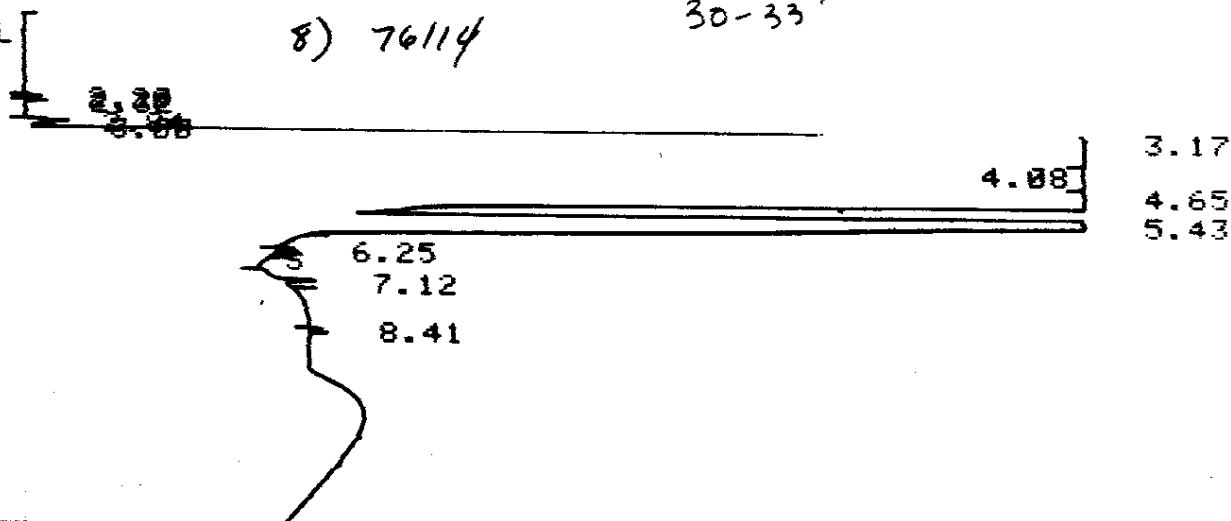


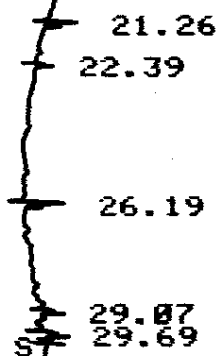
HP RUN # 7
BOTTLE 7
AREA %

RT	AREA	AREA %
2.66	1268	0.000
2.81	47150	0.013
3.10	105200000	29.092
3.80	2470000	0.683
4.34	230700000	63.797
4.74	22620000	6.255
5.42	573700	0.159
10.55	1954	0.001

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VL





HP RUN # 8
BOTTLE 8
AREA %

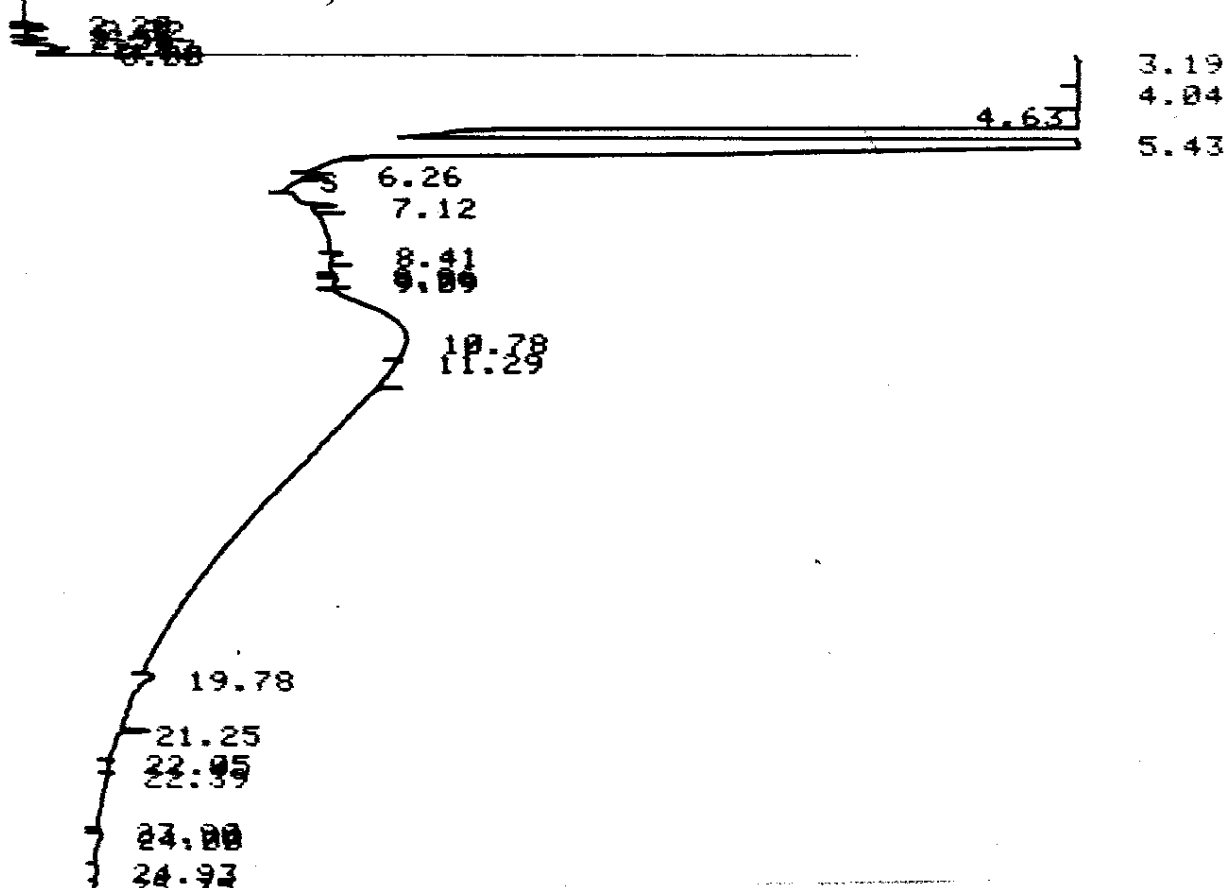
RT	AREA	AREA %
3.17	158200000	88.662
4.08	16310000	9.141
4.65	3291000	1.844
5.43	630000	0.353

DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
START VL

9) 76115

33-36"



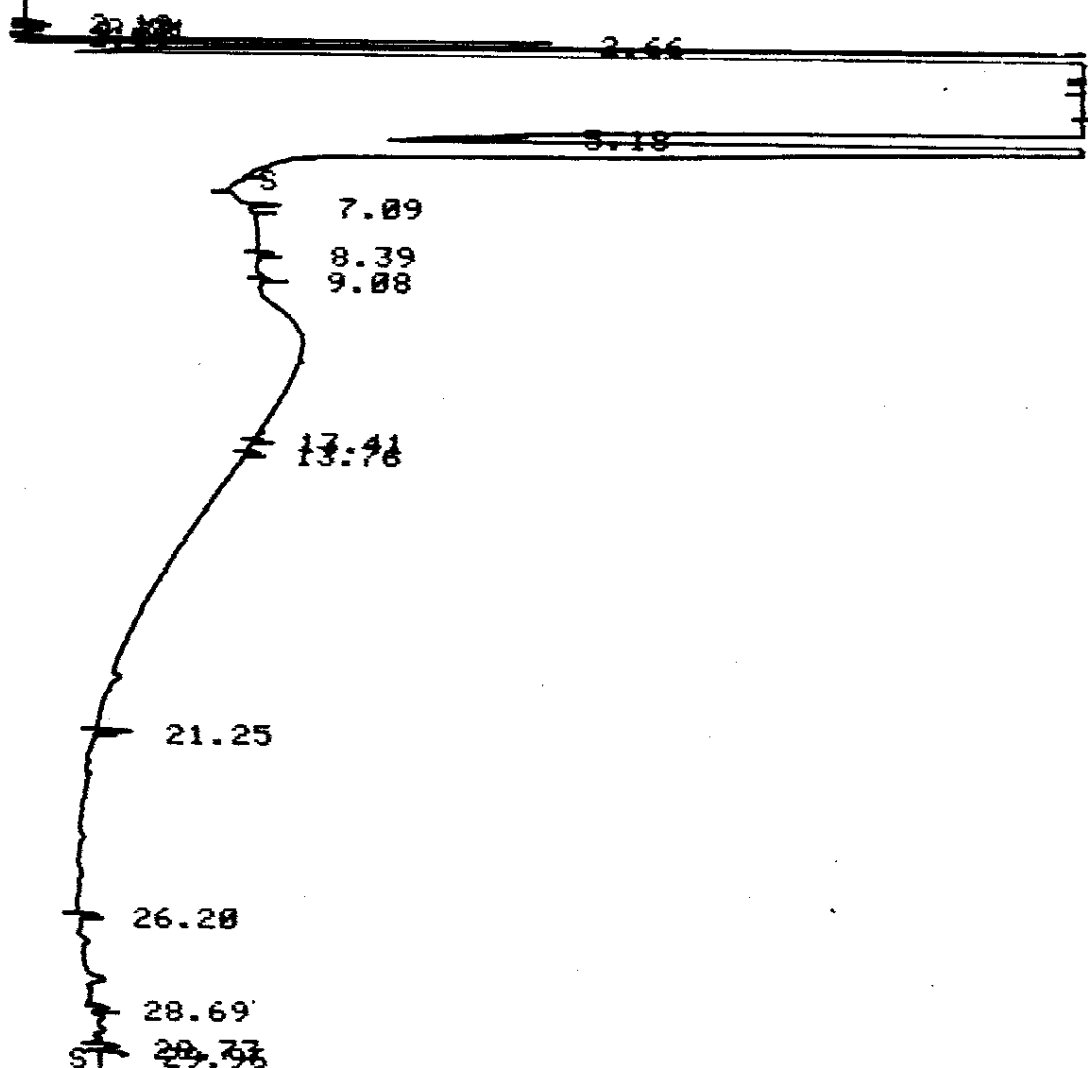
HP RUN # 9
BOTTLE 9
AREA %

RT	AREA	AREA %
3.19	163800000	94.570
4.04	6972000	4.025
4.63	1903000	1.099
5.43	521300	0.301
10.78	7464	0.004
11.29	1435	0.001

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VLF

10) 76116 36-39"



000000-7-4

000000-6-1

```

HP RUN # 10
BOTTLE 10
AREA %

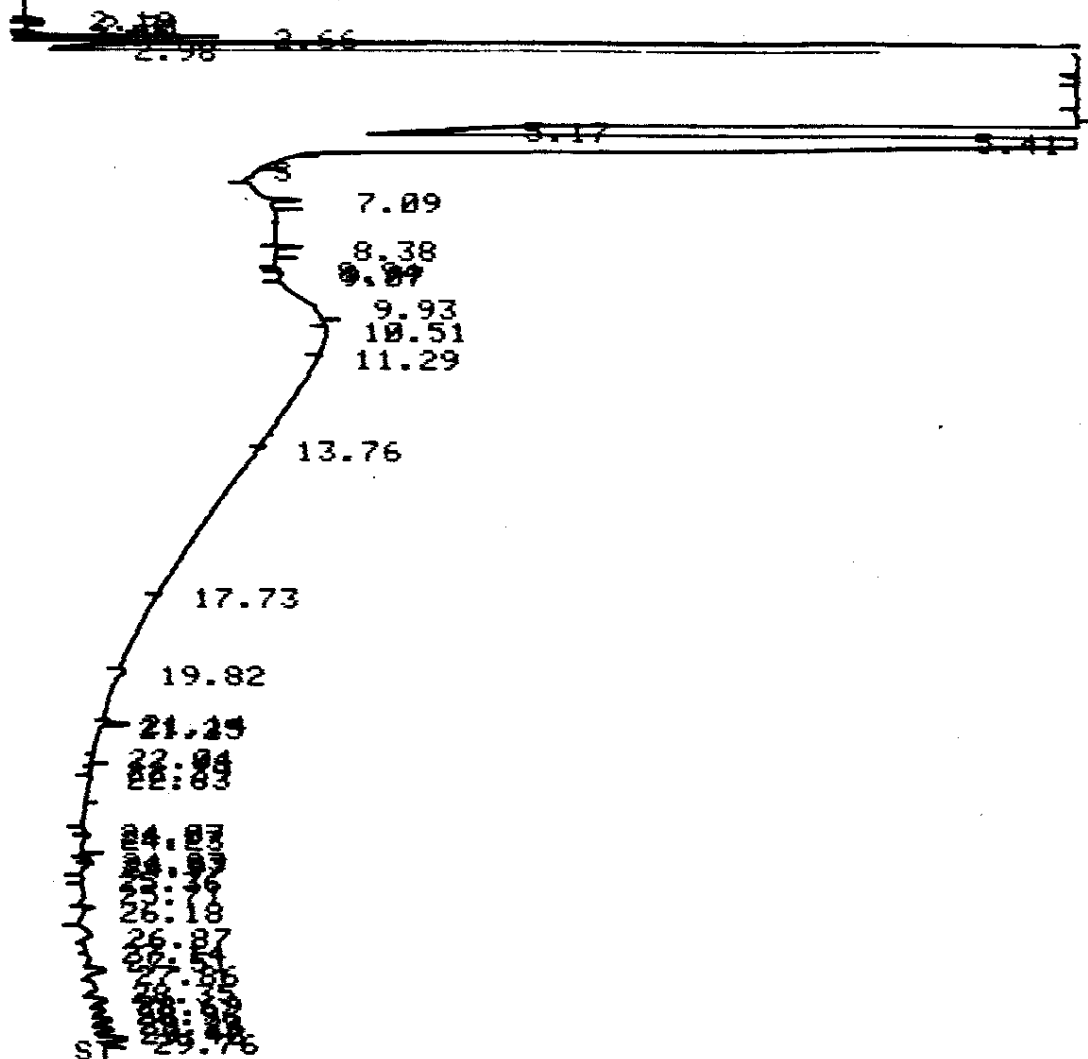
```

DIL FACTOR: 1.0000 E+ 0

4

11) 76117

39-43'



06-08-20
07-09-20

RT	AREA	AREA %
2.81	25230	0.009
3.13	125400000	45.948
3.64	172700	0.063
3.78	1198000	0.439
4.25	132000000	48.366
4.70	13530000	4.958
5.41	585200	0.214
11.29	4001	0.001
13.76	2125	0.001

ATTN 2↑
START

4

VL

12) 76118

48-51
~~8807~~

3.18

4.05

4.43

5.42

6.24

7.18

8.32

10.34

11.29

19.81

21.25

22.85

24.87

25.73

26.19

26.88

27.08

27.27

27.46

27.65

27.84

28.03

28.22

28.41

28.60

28.79

28.98

29.17

HP RUN # 12
BOTTLE 12
AREA %

RT	AREA	AREA %
3.18	156200000	91.862
4.05	10700000	6.293
4.43	19130	0.011
4.63	2523000	1.484
5.42	592400	0.348
10.34	2976	0.002

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑
START

4

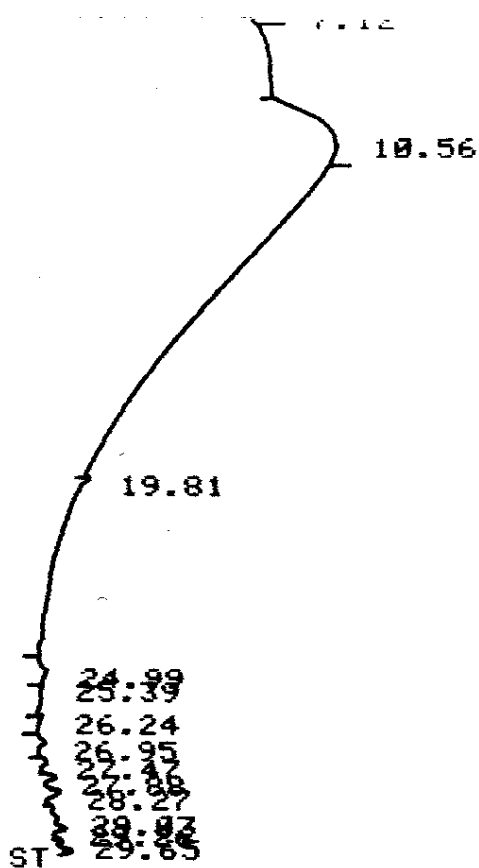
VL

13) Blank CH₂Cl₂

3.18

4.45

5.42



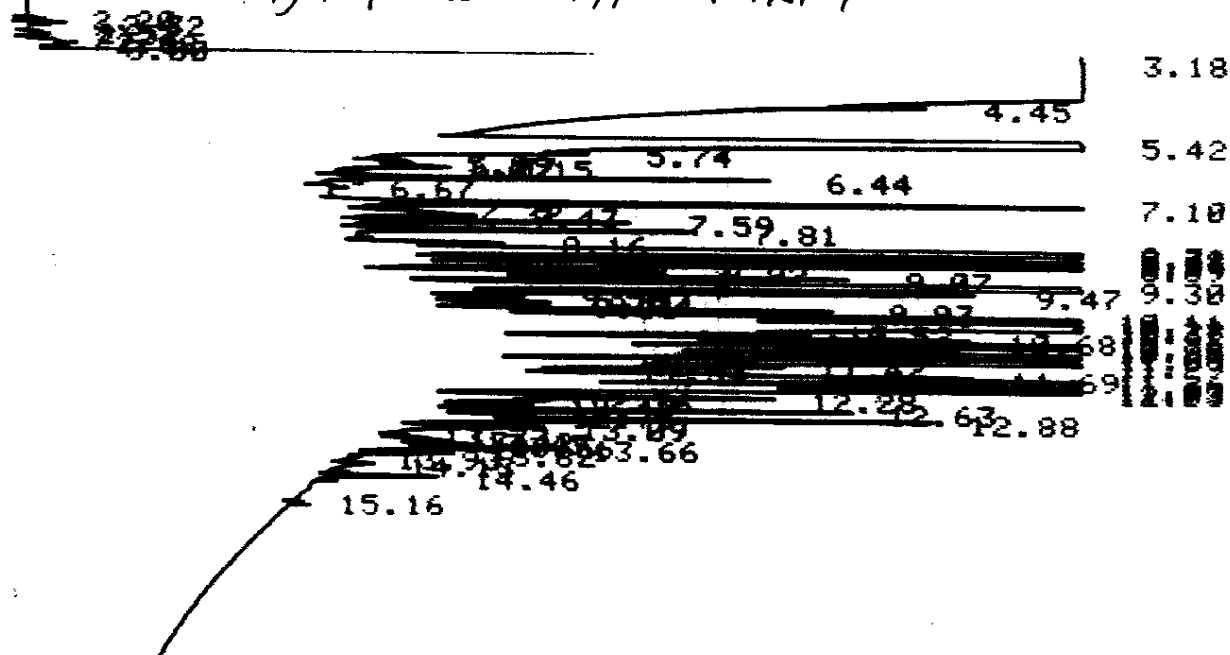
HP RUN # 13
BOTTLE 13
AREA %

RT	AREA	AREA %
3.18	158700000	99.871
5.42	200700	0.126
10.56	4065	0.003

DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
START VL

14) Naphtha 264 ppm #1461-1



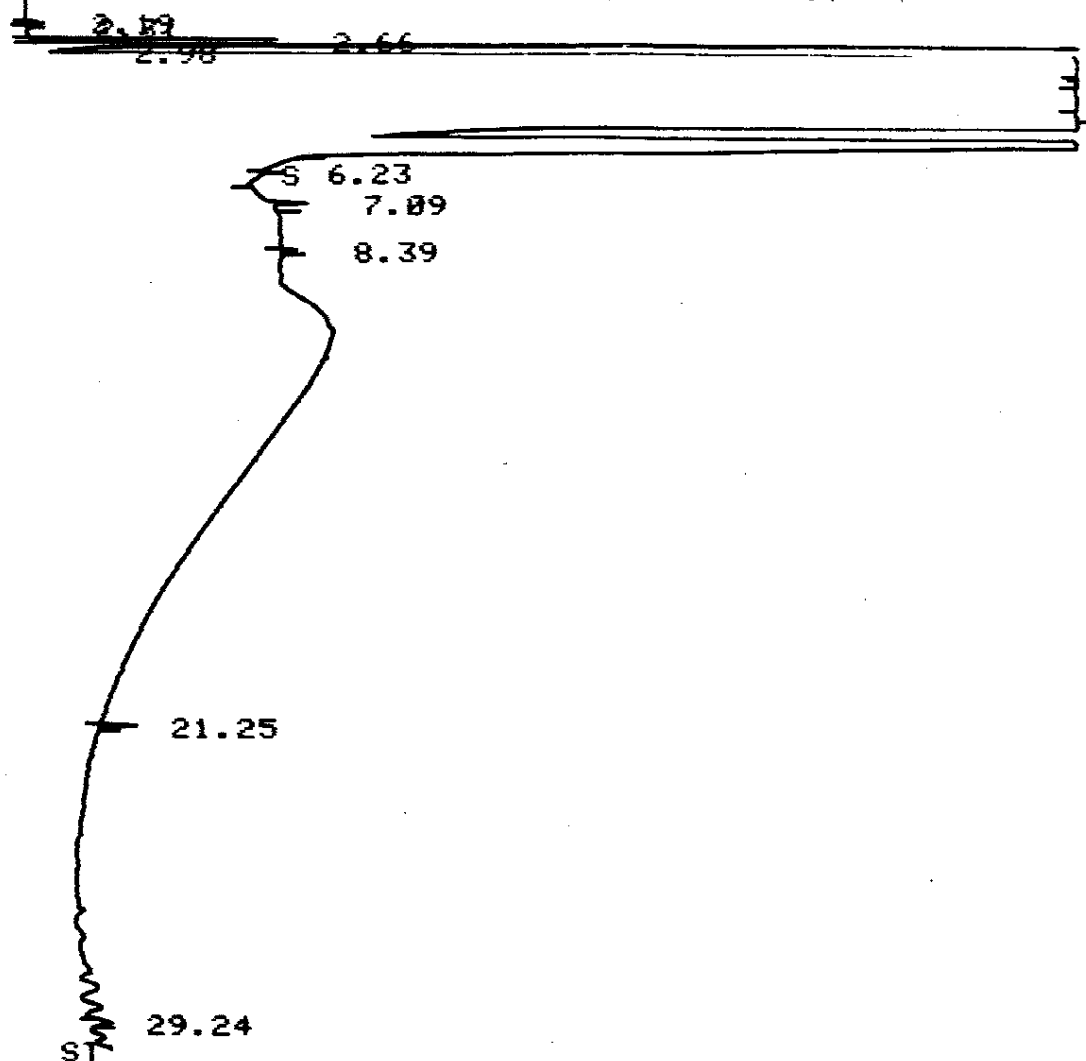
ST

HP RUN # 14
BOTTLE 14
AREA %

RT	AREA	AREA %
3.18	171400000	99.662
5.42	220000	0.128
5.74	1889	0.001
6.44	2982	0.002
7.10	10150	0.006
7.43	1450	0.001
7.59	1936	0.001
7.81	2394	0.001
8.16	1231	0.001
8.31	10390	0.006
8.38	7672	0.004
8.54	8092	0.005
8.69	16160	0.009
8.92	3357	0.002
9.07	4127	0.002
9.30	43900	0.026
9.47	4406	0.003
9.58	1954	0.001
9.74	1717	0.001
9.81	1292	0.001
9.93	3913	0.002
10.11	14360	0.008
10.28	23120	0.013
10.37	17710	0.010
10.52	3780	0.002
10.68	6211	0.004
10.78	6882	0.004
10.88	6269	0.004
10.95	4014	0.002
11.09	23030	0.013
11.29	40900	0.024
11.42	4661	0.003
11.54	1816	0.001
11.69	6384	0.004
11.76	8688	0.005
11.93	9496	0.006
12.03	30800	0.018
12.28	4876	0.003
12.36	1495	0.001
12.49	1973	0.001
12.63	4402	0.003
12.76	1775	0.001
12.88	5628	0.003
13.09	2503	0.001
13.66	1916	0.001

DIL FACTOR: 1.0000 E+ 0

54-57 "



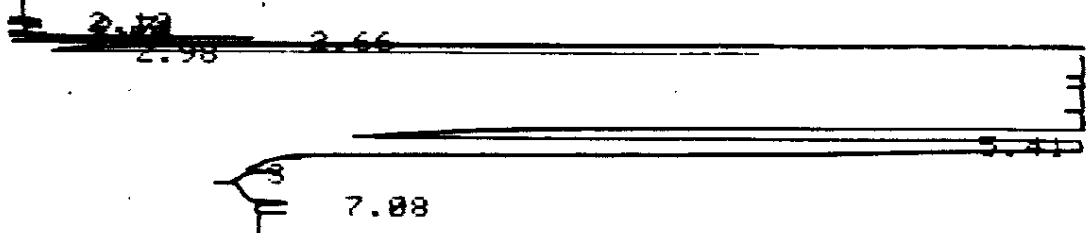
10-15-50
 10-15-50
 10-15-50

```
HP RUN # 15
BOTTLE 15
AREA %
```

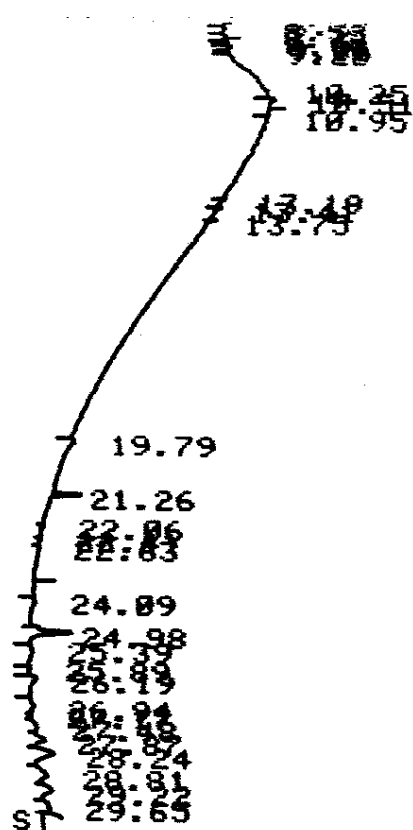
RT	AREA	AREA %
2.81	29850	0.011
3.14	132000000	50.069
3.64	188400	0.071
3.77	1187000	0.450
4.23	119200000	45.214
4.68	10530000	3.994
5.41	500900	0.190

DIL FACTOR: 1.0000 E+ 0

60-63'



11-11-11



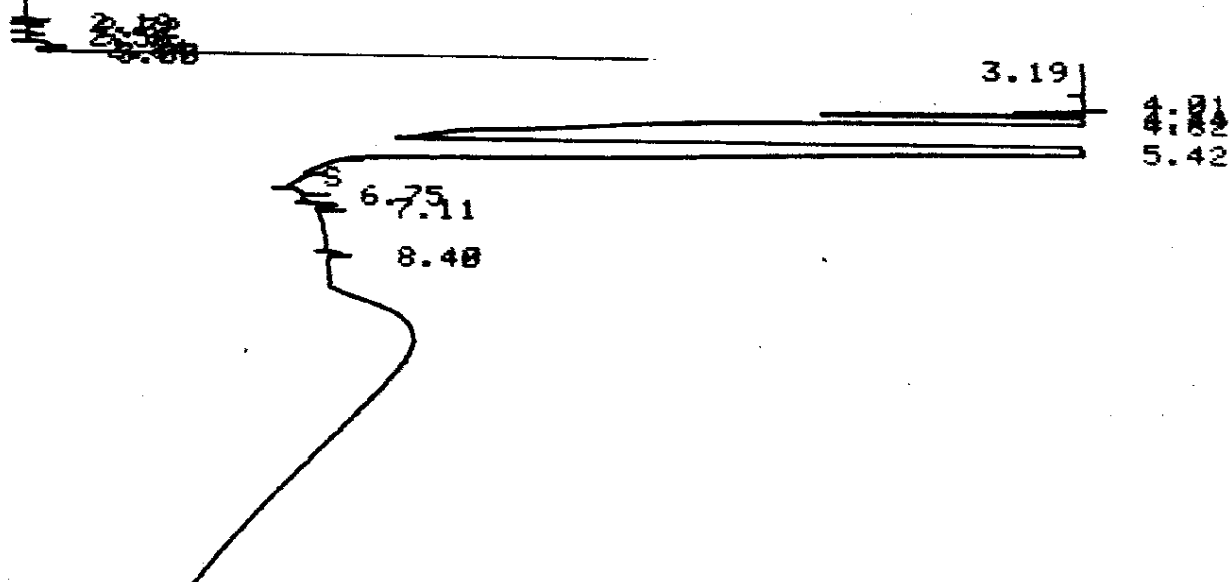
HP RUN # 16
BOTTLE 16
AREA %

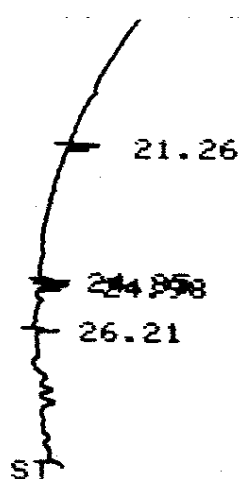
RT	AREA	AREA %
2.81	29470	0.011
3.11	112900000	40.823
3.77	1585000	0.573
4.26	147100000	53.189
4.70	14400000	5.207
5.41	546000	0.197
10.95	1251	0.000

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VL

17) 76121 66-69"





HP RUN # 17
BOTTLE 17
AREA %

RT	AREA	AREA %
3.19	163900000	99.307
4.01	574400	0.348
4.44	1745	0.001
4.62	133200	0.081
5.42	434600	0.263

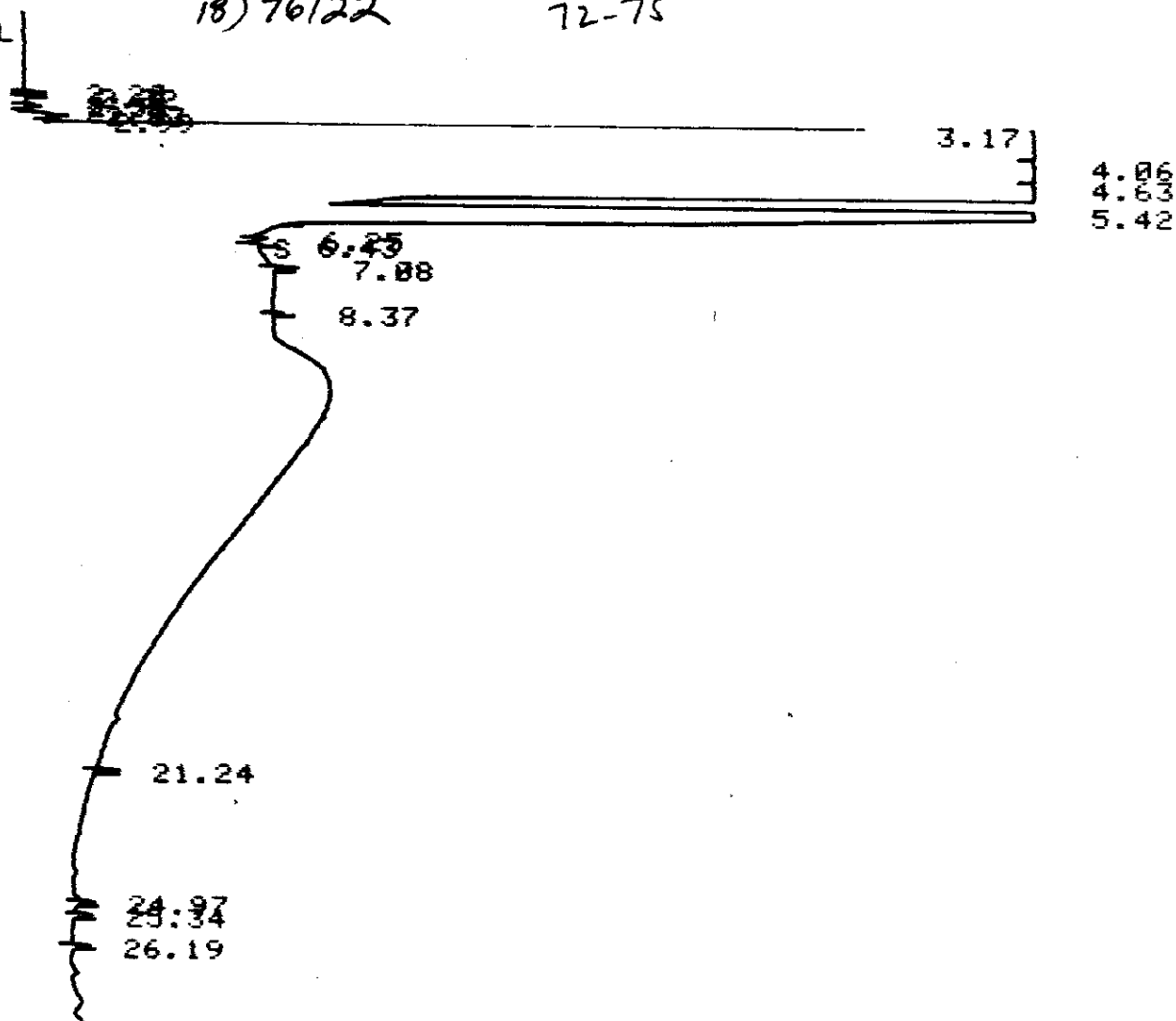
DIL FACTOR: 1.0000 E+ 0

ATTN 2↑
START VL

4

18) 76122

72-75"



ST

HP RUN # 18
BOTTLE 18
AREA %

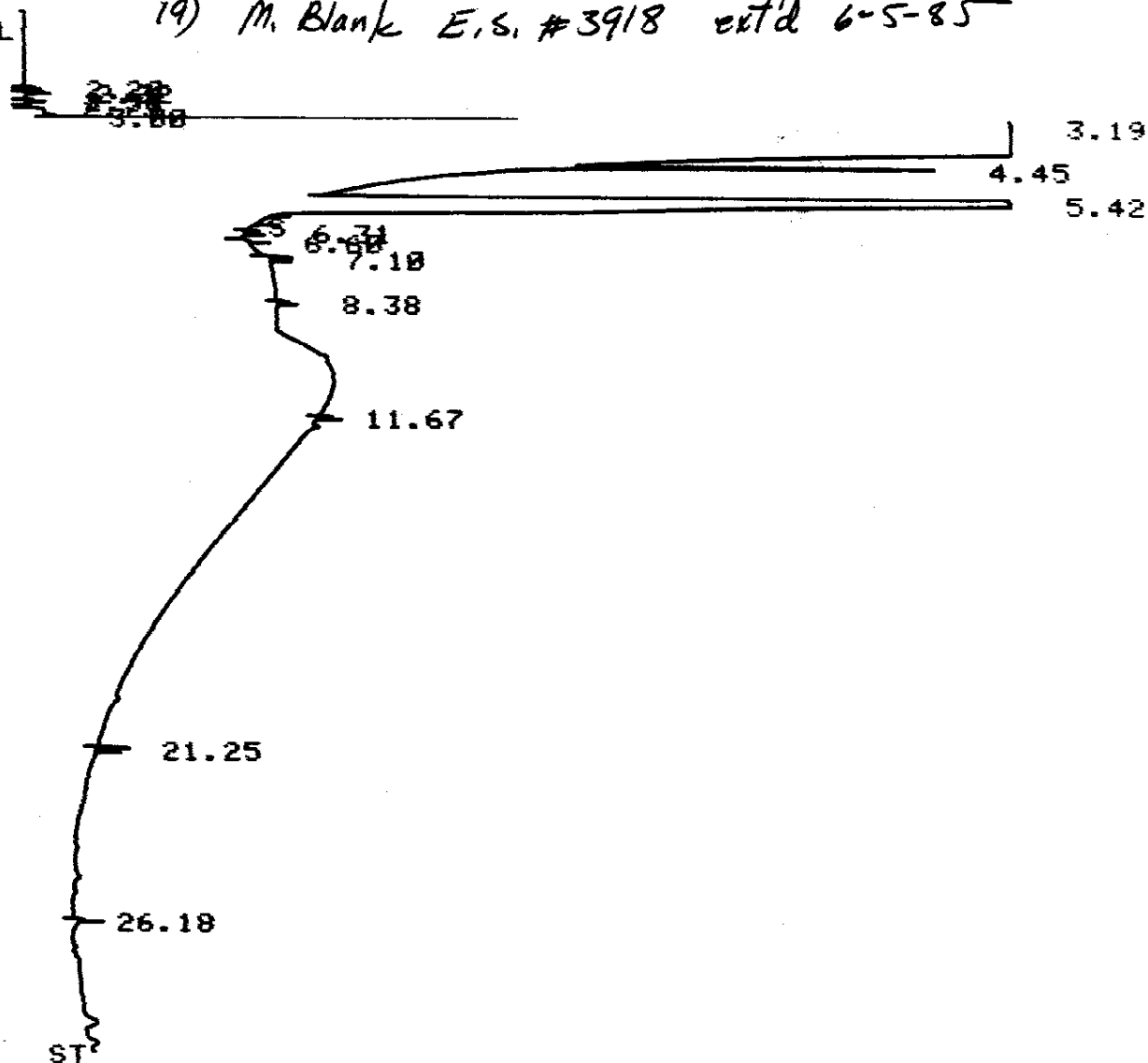
RT	AREA	AREA %
3.17	151100000	89.142
4.06	14780000	8.720
4.63	313600	1.850
5.42	488300	0.288

DIL FACTOR: 1.0000 E+ 0

ATTN 2+
START VL

4

19) M. Blank E.S. #3918 ext'd 6-5-85



HP RUN # 19
BOTTLE 19
AREA %

RT	AREA	AREA %
3.19	160600000	99.747
4.45	2146	0.001
5.42	405900	0.252

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VL

20) 76110 A

18-21"

2.32
2.32
2.32
2.32

3.18

4.45

5.42

6.24
6.24
7.11

8.48

21.26

26.20

27.60

ST

HP RUN # 20
BOTTLE 20
AREA %

RT	AREA	AREA %
3.18	157000000	99.783
4.45	1681	0.001
5.42	340100	0.216

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VL

21) 76110 B

18-21"

2.32
2.32
2.32
2.32

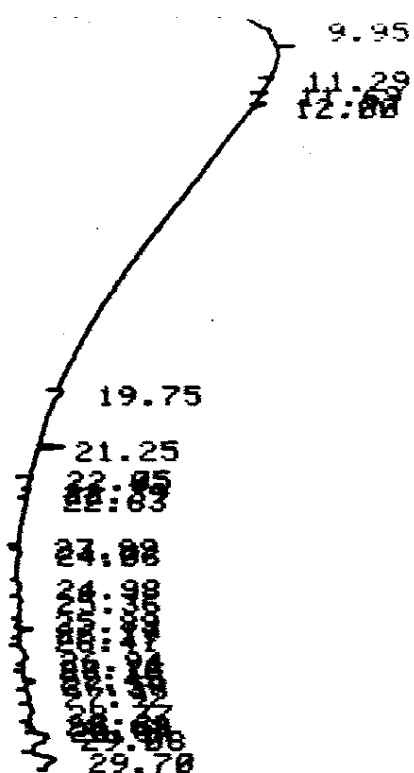
3.17

4.44

5.41

6.22
7.009

8.38

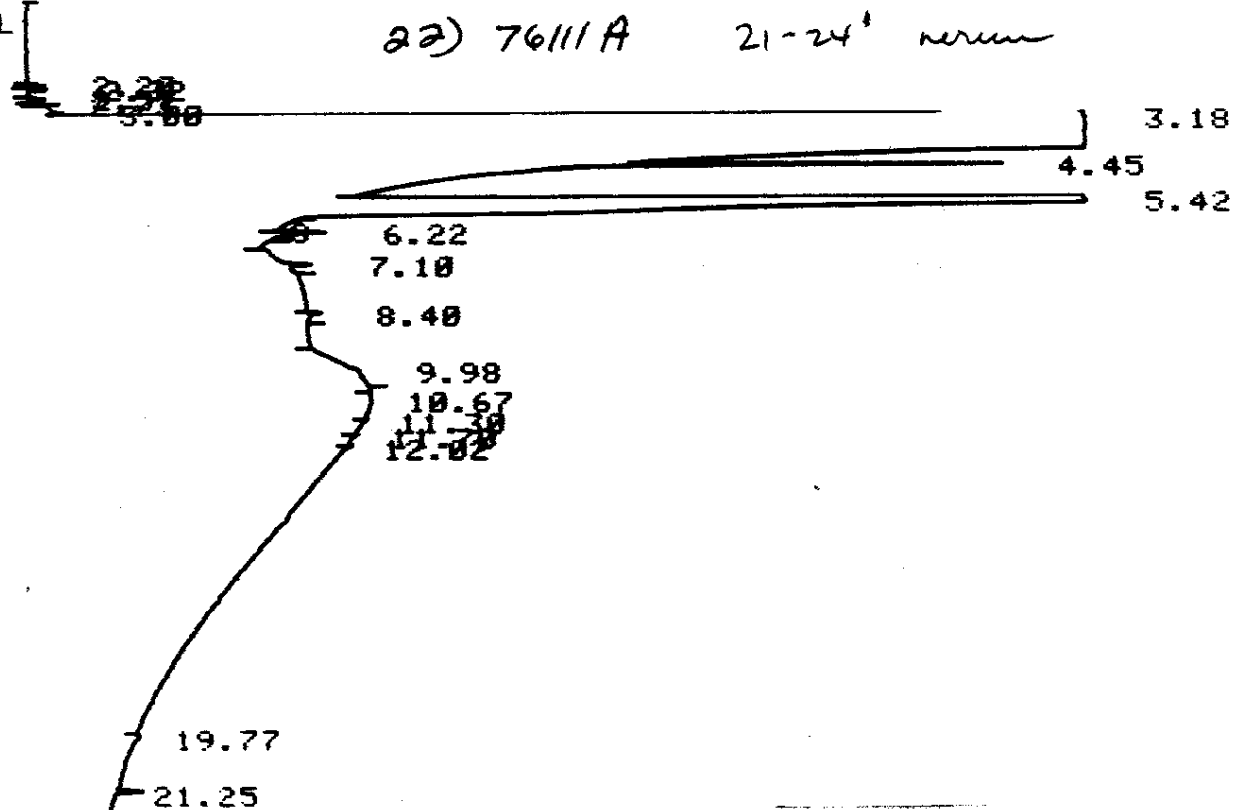


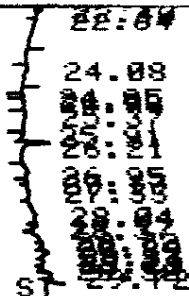
HP RUN # 21
BOTTLE 21
AREA %

RT	AREA	AREA %
3.17	149500000	99.761
4.44	1781	0.001
5.41	356500	0.238

DIL FACTOR: 1.0000 E+ 0

ATTN 21 4
START VL



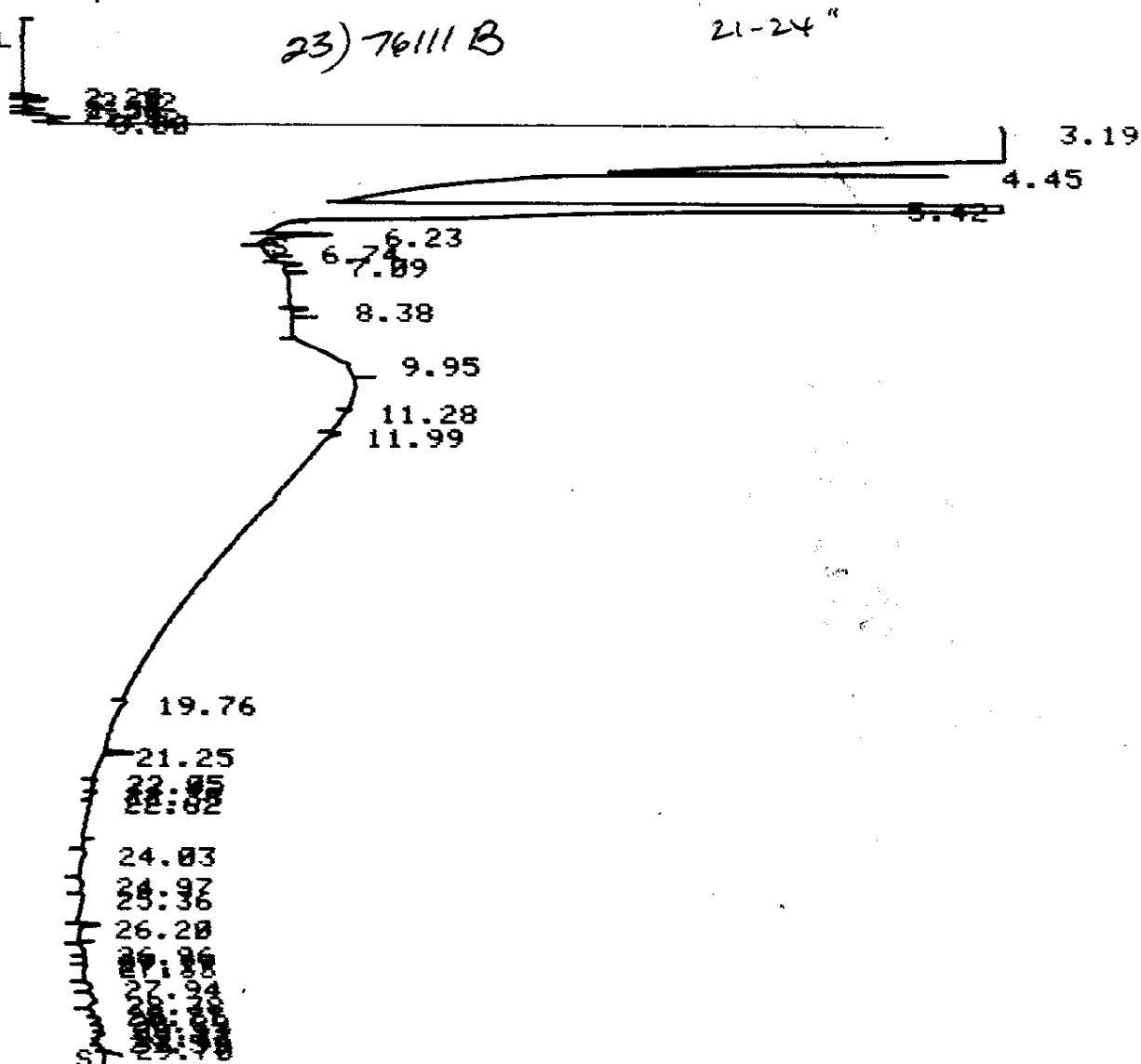


HP RUN # 22
BOTTLE 22
AREA %

RT	AREA	AREA %
3.18	162100000	99.753
4.45	2035	0.001
5.42	399600	0.246

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VL



HP RUN # 23
BOTTLE 23
AREA %

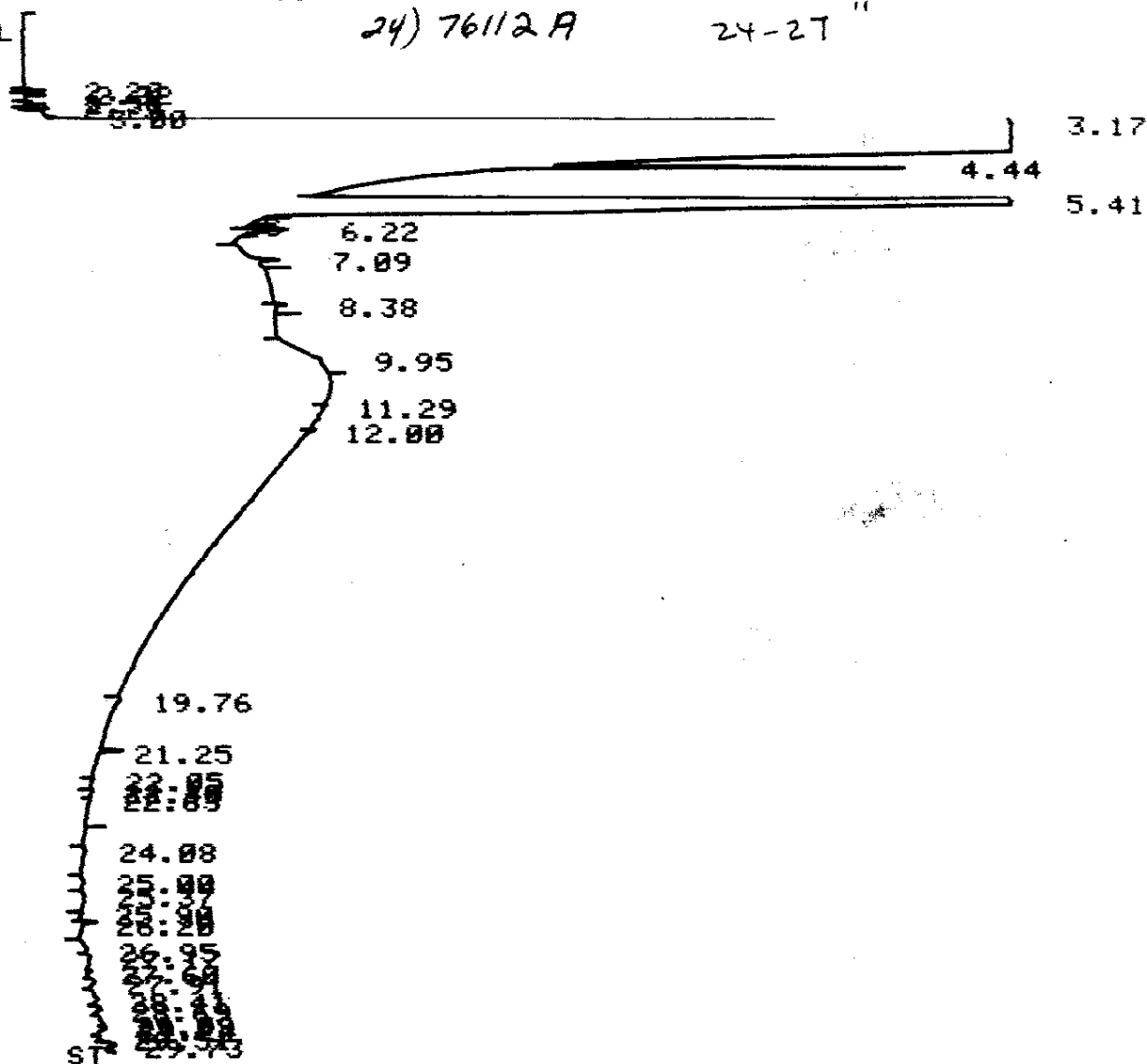
RT	AREA	AREA %
3.19	167400000	99.766
4.45	1992	0.001
5.42	389900	0.232

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
TEMP1 400 35 65
START VL

24) 76112 A

24-27 "



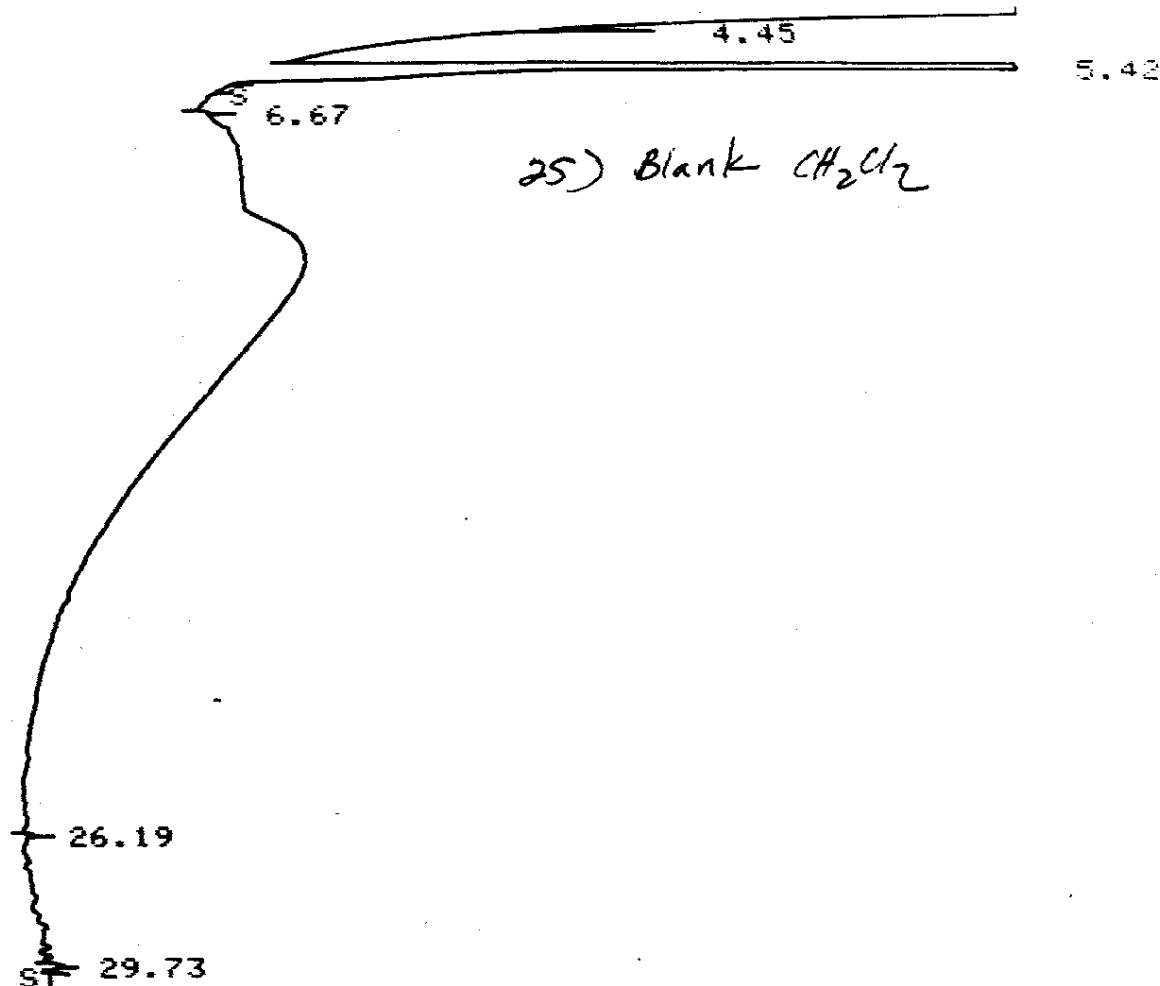
HP RUN # 24
BOTTLE 24
AREA %

RT	AREA	AREA %
3.17	153300000	99.742
4.44	2046	0.001
5.41	394000	0.256

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VL



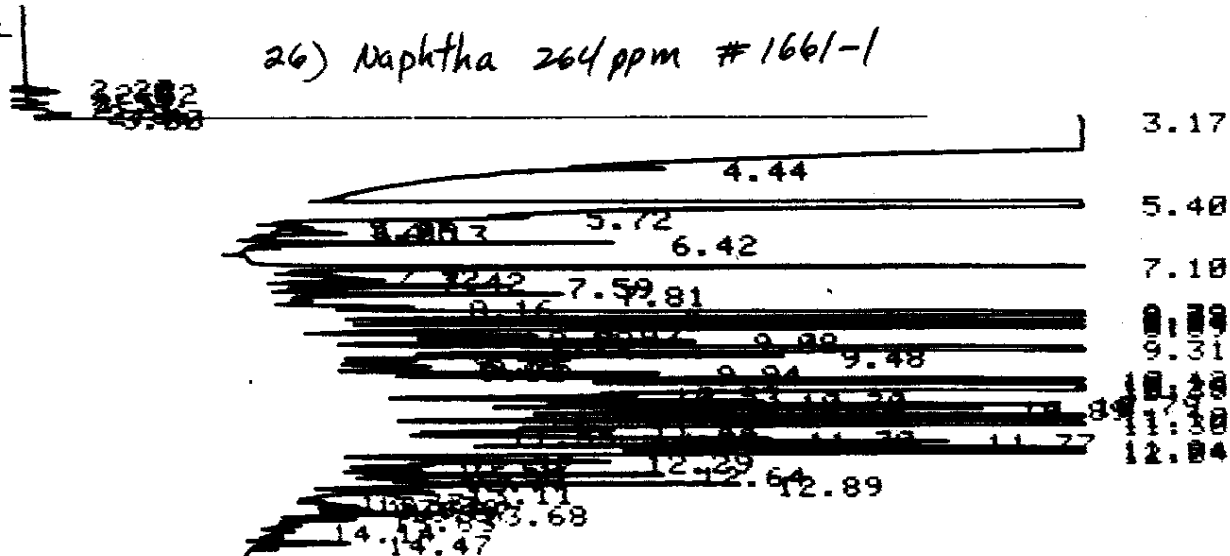


HP RUN # 25
BOTTLE 25
AREA %

RT	AREA	AREA %
3.19	161300000	99.873
5.42	205000	0.127

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VL



ST

HP RUN # 26
BOTTLE 26
AREA %

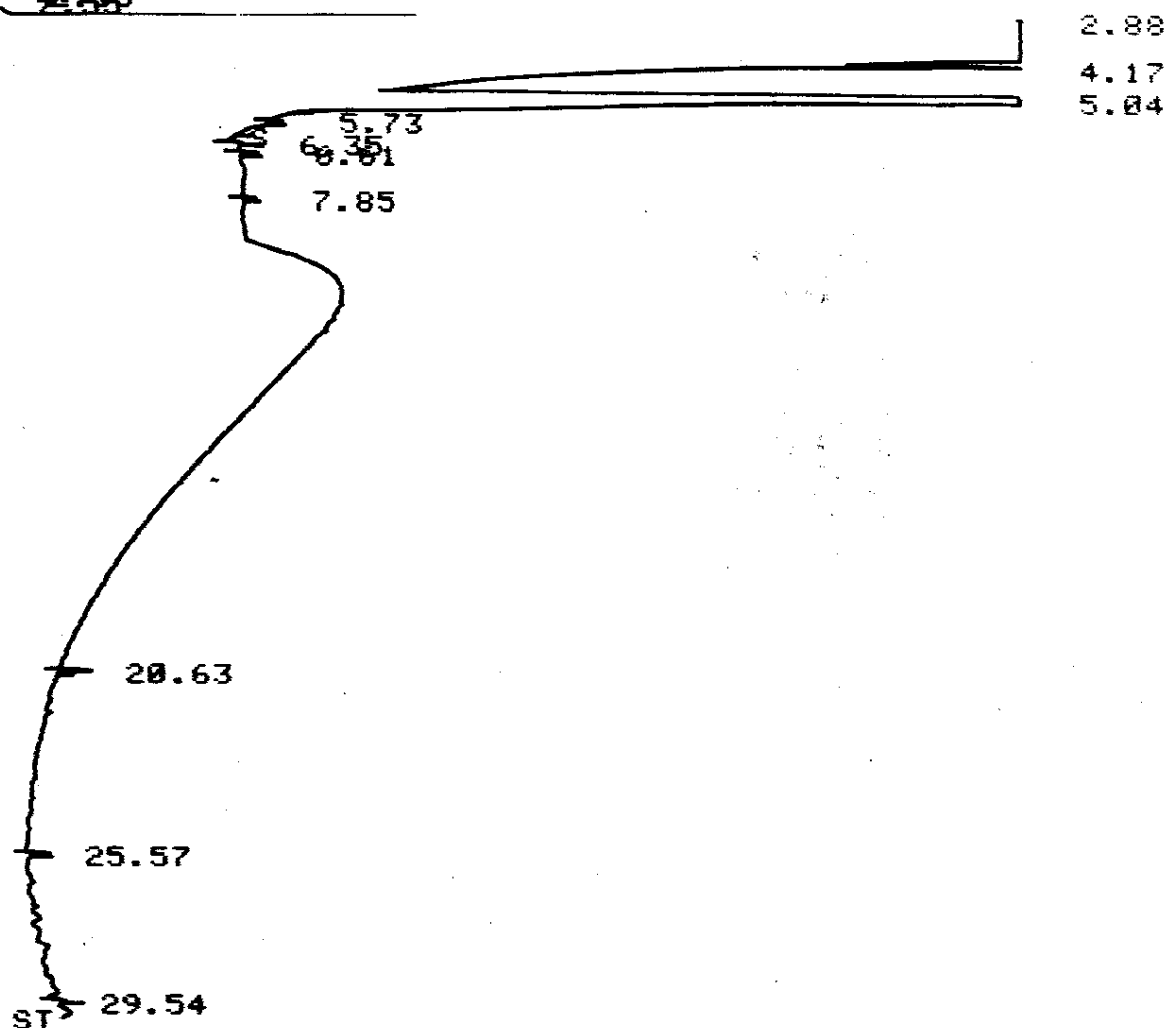
RT	AREA	AREA %
3.17	148200000	99.682
5.40	185900	0.125
5.72	2305	0.002
6.42	2416	0.002
7.10	8624	0.006
7.42	1240	0.001
7.59	1625	0.001
7.81	1829	0.001
8.32	8508	0.006
8.39	6269	0.004
8.54	6686	0.004
8.69	13340	0.009
8.93	1754	0.001
8.98	1019	0.001
9.08	3393	0.002
9.31	35870	0.024
9.48	3513	0.002
9.58	1400	0.001
9.75	1164	0.001
9.94	2877	0.002
10.12	11240	0.008
10.29	18360	0.012
10.38	14390	0.010
10.53	2828	0.002
10.70	4699	0.003
10.79	5370	0.004
10.89	4873	0.003
10.97	3080	0.002
11.10	18350	0.012
11.30	32990	0.022
11.44	3432	0.002
11.55	1309	0.001
11.70	4786	0.003
11.77	6900	0.005
11.94	7444	0.005
12.04	24760	0.017
12.29	3624	0.002
12.37	1061	0.001
12.50	1341	0.001
12.64	3318	0.002
12.77	1263	0.001
12.89	4274	0.003
13.11	1624	0.001
13.68	1356	0.001

ATTN 2↑ 4
 TEMP1 4 0 0
 START VL

27) 76112B

24-27"

22.25



HP RUN # 27
 BOTTLE 27
 AREA %

RT	AREA	AREA %
2.88	152000000	99.795
4.17	1556	0.001
5.04	310800	0.204

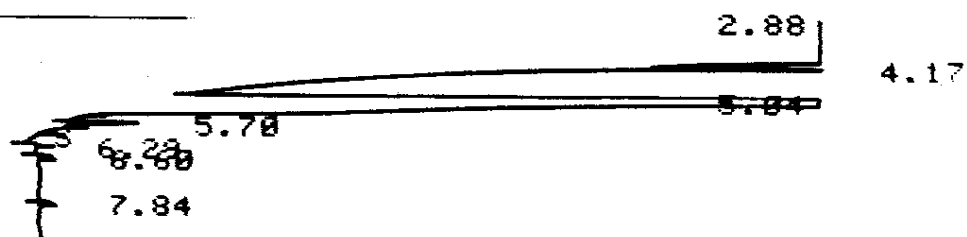
DIL FACTOR: 1.0000 E+ 0

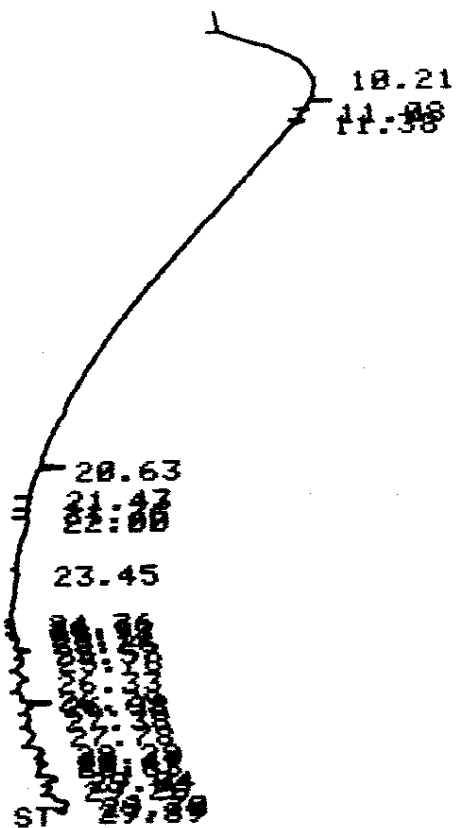
ATTN 2↑ 4
 START VL

28) 76113A

21-30"

22.25



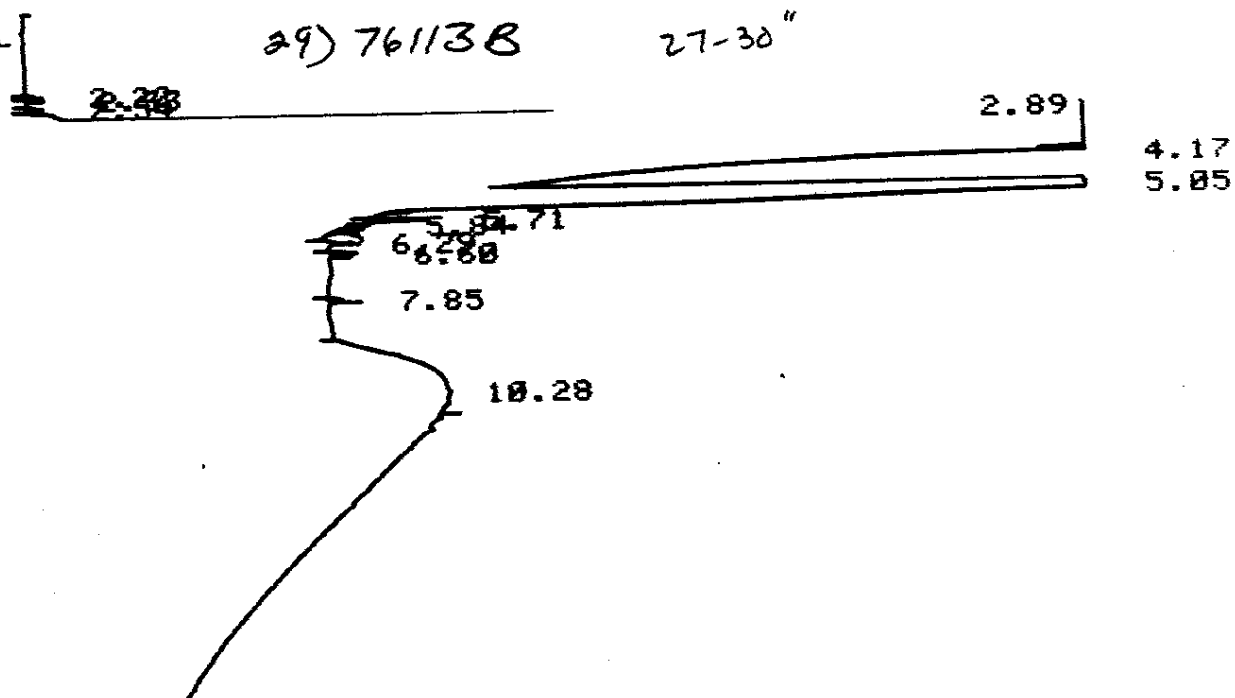


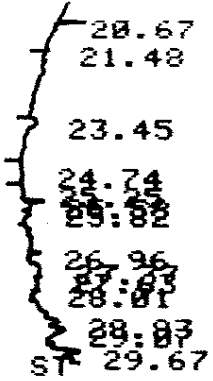
HP RUN # 28
BOTTLE 28
AREA %

RT	AREA	AREA %
2.88	153900000	99.821
4.17	1169	0.001
5.04	268200	0.174
10.21	5106	0.003
27.78	1005	0.001

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VL



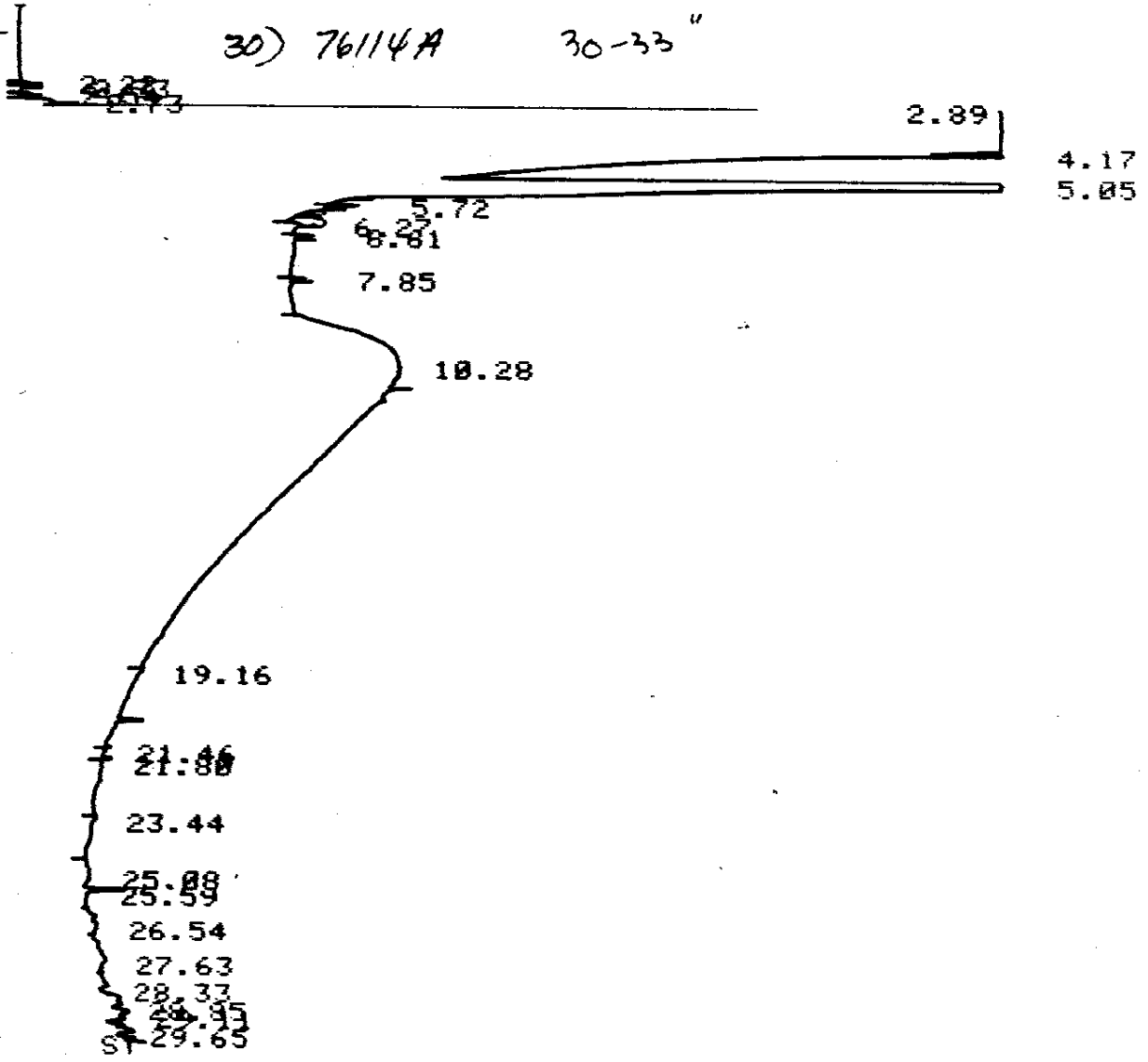


HP RUN # 29
BOTTLE 29
AREA %

RT	AREA	AREA %
2.89	156200000	99.718
4.17	2085	0.001
5.05	431800	0.276
10.28	7614	0.005

DIL FACTOR: 1.0000 E+ 0

ATTN 21 4
START VL

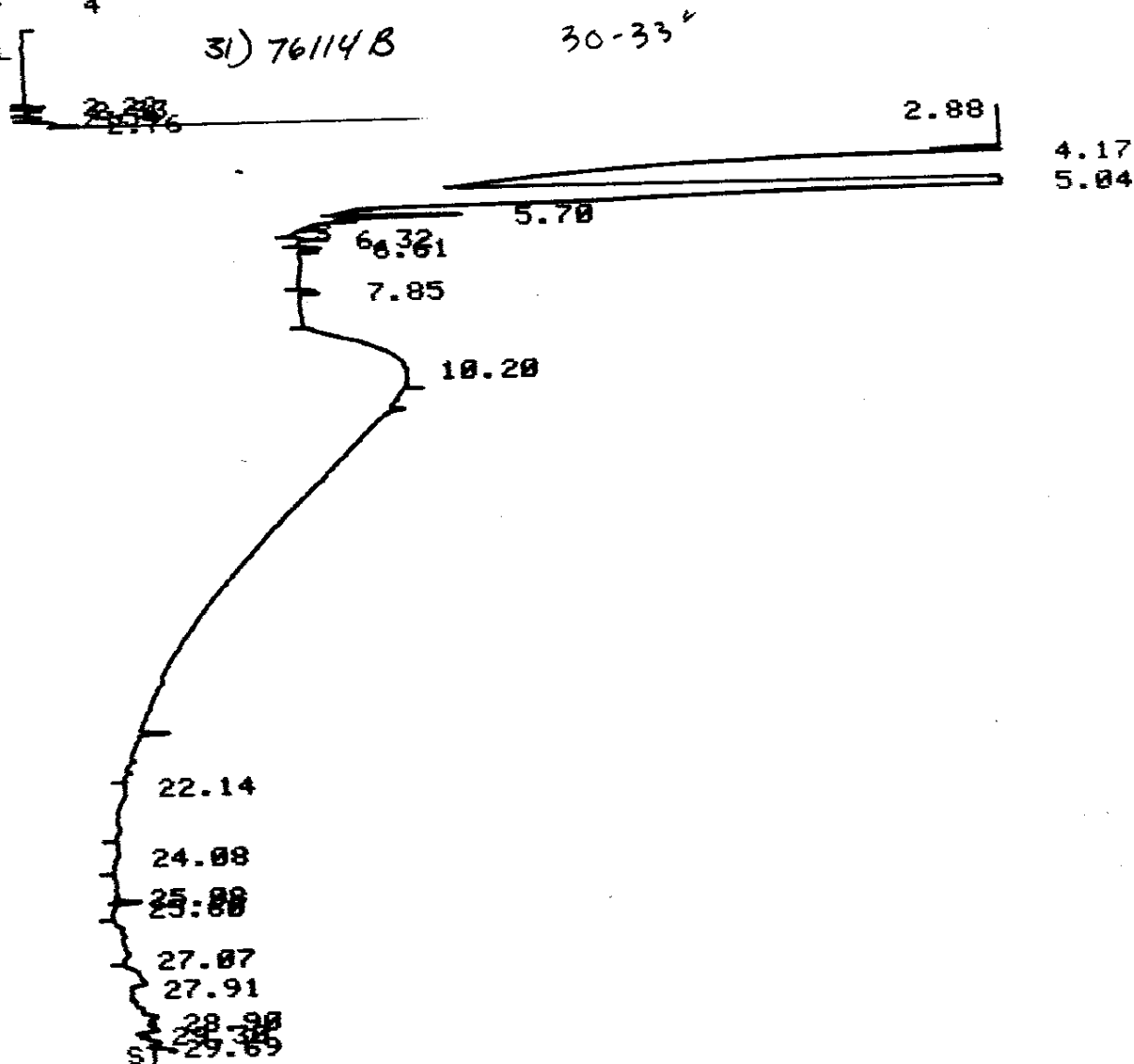


HP RUN # 30
BOTTLE 30
AREA %

RT	AREA	AREA %
2.89	152900000	99.770
4.17	1620	0.001
5.05	341000	0.223
10.20	8156	0.005
27.63	1513	0.001

DIL FACTOR: 1.0000 E+ 0

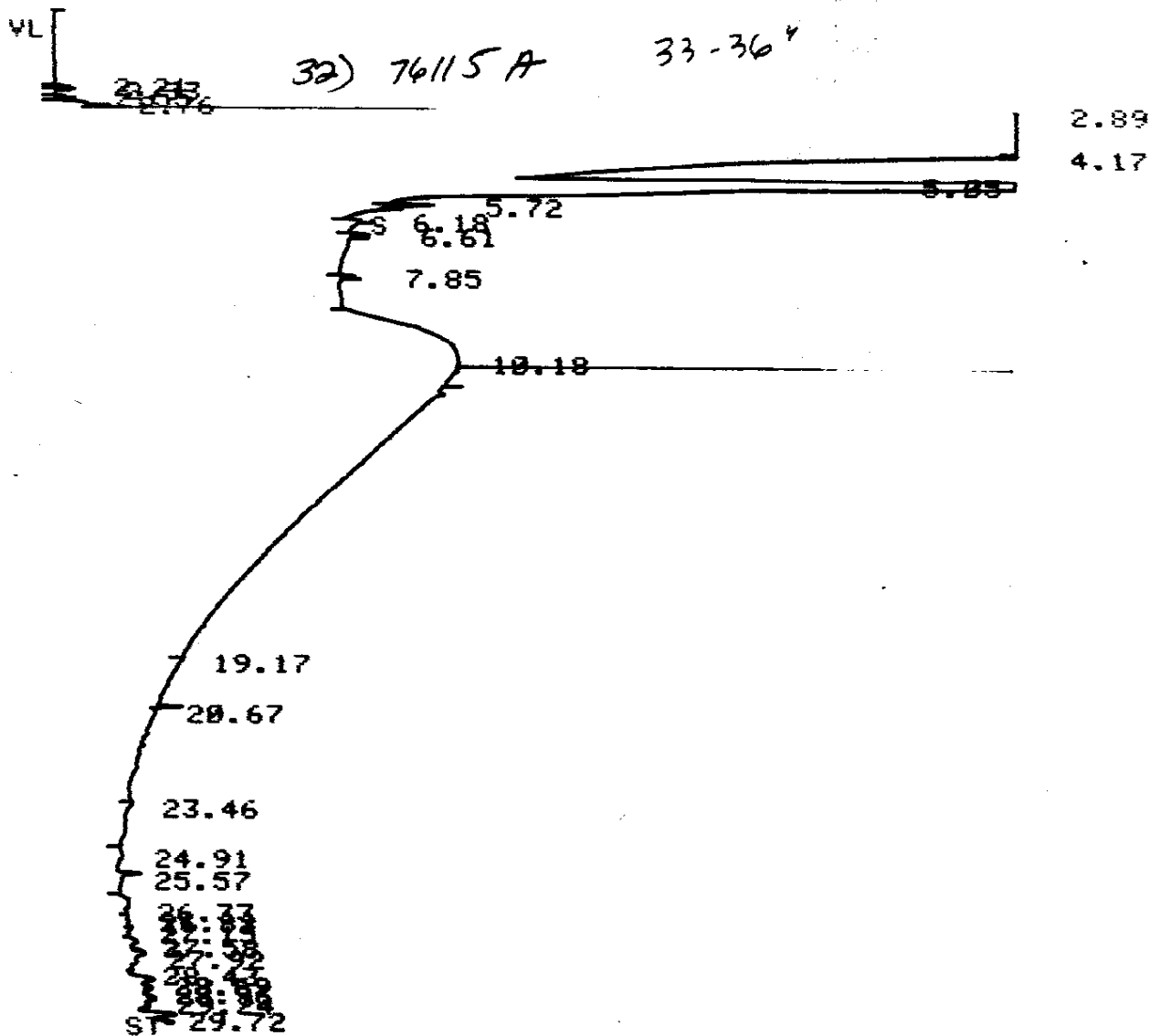
ATTN 2↑ 4
START VL



HP RUN # 31
BOTTLE 31
AREA %

RT	AREA	AREA %
2.88	154900000	99.729
4.17	1922	0.001
5.04	411000	0.265
10.20	5707	0.004
27.91	1092	0.001

ATTN 2↑ 4
 OPTN # 1 @
 ID: 2 @
 OPTN # 3 ESCAPE
 OPTN # 2 @
 (M-D-Y) DATE: 8 - 15 - 85
 (H.M.S) TIME: 16 . 29 . 00 @
 START



HP RUN # 32
 ID: 2
 AREA %

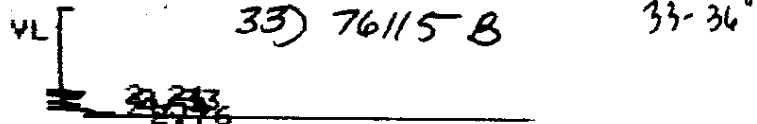
AUG/15/85
 BOTTLE 32

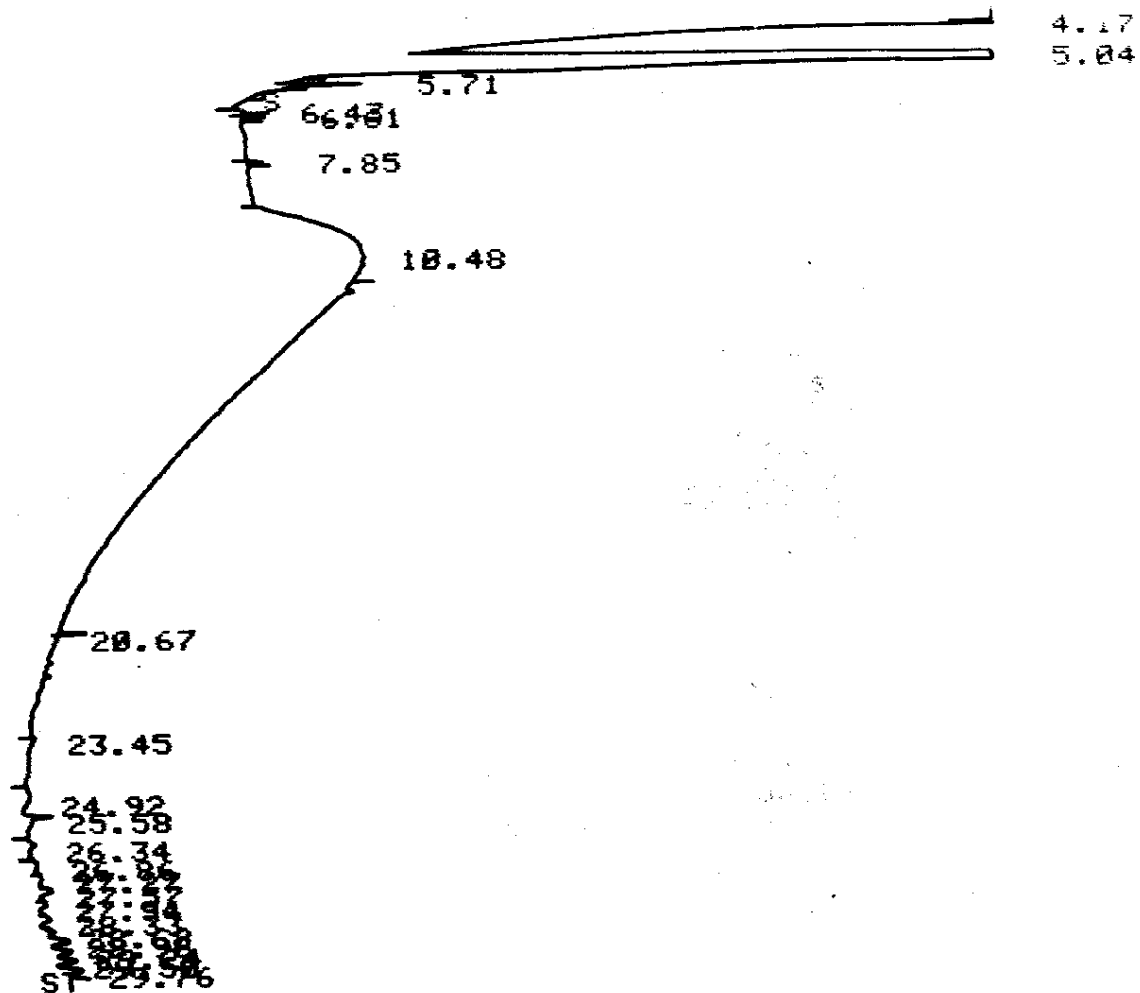
TIME 16:44:08

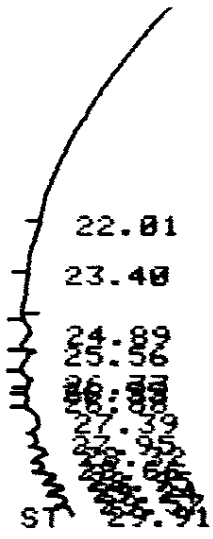
RT	AREA	AREA %
2.89	161100000	99.700
4.17	1762	0.001
5.05	373900	0.231
10.18	107800	0.067
27.92	1490	0.001

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
 START







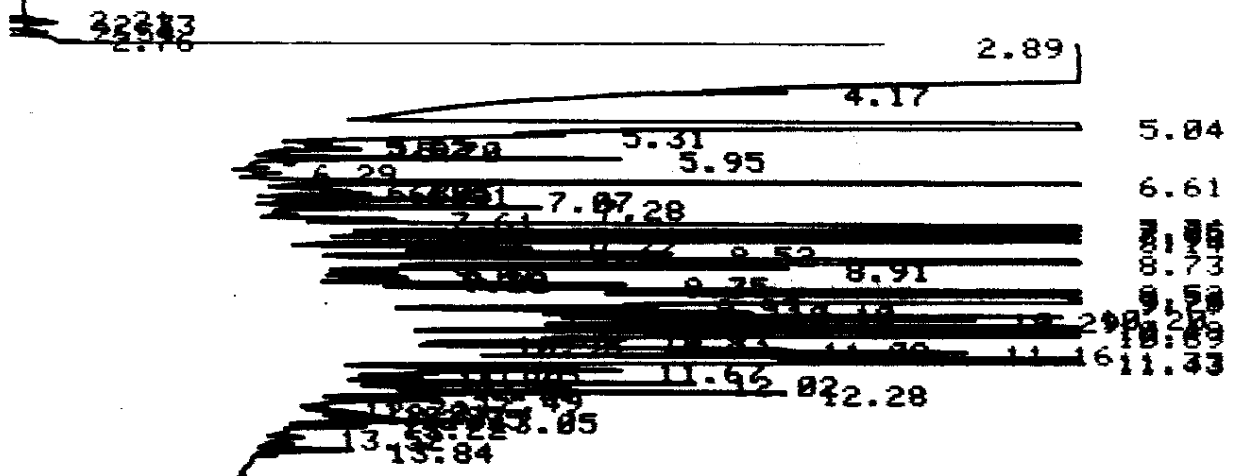
HP RUN # 34 AUG/15/85 TIME 18:19:09
ID:2 BOTTLE 34
AREA %

RT	AREA	AREA %
2.89	160100000	99.868
5.04	201800	0.126
10.10	8880	0.006
27.39	1195	0.001

DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
START VL

35) Naphtha 264 ppm #1661-1



ST

HP RUN # 35
ID:2
AREA %

AUG/15/85
BOTTLE 35

TIME 19:06:34

RT	AREA	AREA %
2.89	1408000000	99.664
5.04	175900	0.125
5.31	3597	0.003
5.95	2367	0.002
6.61	8110	0.006
7.07	1512	0.001
7.28	1792	0.001
7.76	8812	0.006
7.85	5950	0.004
7.98	6798	0.005
8.14	13310	0.009
8.37	2838	0.002
8.52	3439	0.002
8.73	36140	0.026
8.91	3698	0.003
9.01	1369	0.001
9.18	1092	0.001
9.35	2984	0.002
9.52	11360	0.008
9.70	19210	0.014
9.79	14510	0.010
9.93	3030	0.002
10.10	5081	0.004
10.20	5577	0.004
10.29	5119	0.004
10.37	3405	0.002
10.49	19110	0.014
10.69	34410	0.024
10.83	3866	0.003
10.95	1490	0.001
11.09	5320	0.004
11.16	7286	0.005
11.33	7812	0.006
11.43	26070	0.018
11.67	4164	0.003
11.75	1276	0.001
11.88	1725	0.001
12.02	3800	0.003
12.15	1536	0.001
12.28	4923	0.003
12.49	2363	0.002
13.05	1735	0.001
13.22	1083	0.001

41
DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
TEMP1 3 0 0

TEMP1 400 30 36
TEMP1 4 0 0

TEMP1 400 40 40
TIME1 4.00
RATE 8.00
TEMP2 400 300
TIME2 20.00
INJ TEMP 400 225 225
FID TEMP 400 250 250
AUX TEMP 400 225 225

CHT SPD 0.50
ZERO 15.0
ATTN 2+ 4
FID SGNL +8
SLP SENS 0.10
AREA REJ 1000
FLOW A 0.0 28.0
FLOW B 0.0 43.8

0.50 VLV/EXT- 1
30.00 STOP

CHANGE RUN 1 @
OPTN #1
ID: 2
ESCAPE
OPTN #3
INJ/BTL, STROKE: 1.2
START

VL

D Blank CH₂Cl₂

32.232
21.5167

2.89
4.17
5.84
6.27

10.20

25.59
26.27
26.80
27.50
28.34
28.70
29.23
ST 29.85

8/16/85
2086-00110
Dupont

Pet. Hydrocarbons/soi
Reruns

DOK
Col # 324

Attn 24
4 µl inj vol.

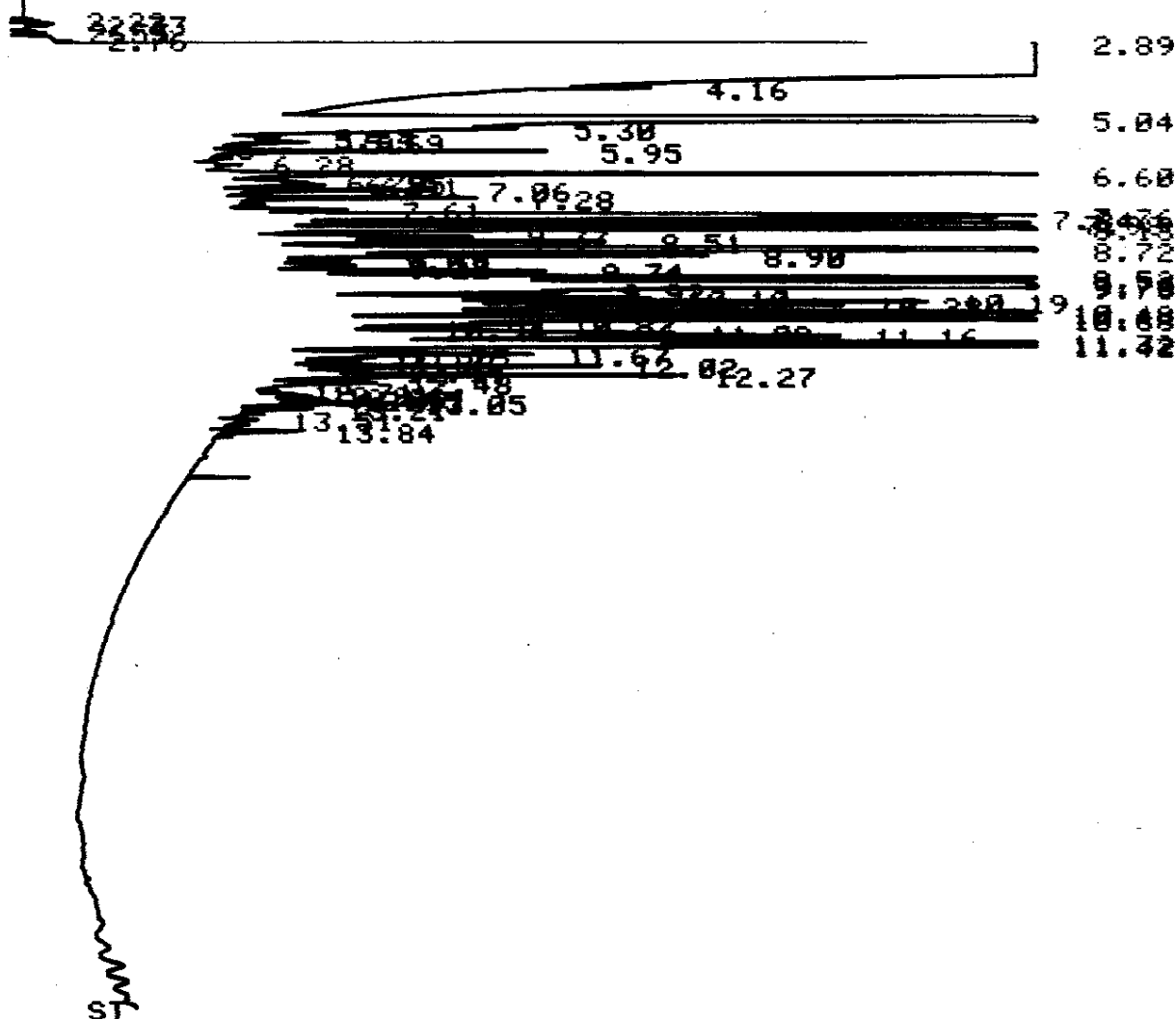
AREA %

RT	AREA	AREA %
2.89	157900000	99.865
5.04	203000	0.128
10.20	9958	0.006

DIL FACTOR: 1.0000 E+ 0

ATTN 21 4
CHANGE RUN 3 @
START VL

2) Naphtha 264 ppm #1661-1



HP RUN # 4
ID:2
AREA %

AUG/16/85
BOTTLE 4

TIME 10:55:39

RT	AREA	AREA %
2.89	135600000	99.678
5.04	167500	0.123
5.30	3970	0.003
5.95	2196	0.002
6.60	7656	0.006
7.06	1429	0.001
7.28	1674	0.001
7.76	8220	0.006
7.84	5459	0.004
7.98	6354	0.005

8.37	2532	0.002
8.51	3173	0.002
8.72	33560	0.025
8.90	3448	0.003
9.01	1242	0.001
9.17	1027	0.001
9.34	2633	0.002
9.52	10260	0.008
9.70	17480	0.013
9.78	13460	0.010
9.92	2592	0.002
10.10	4430	0.003
10.19	4973	0.004
10.28	4545	0.003
10.36	2950	0.002
10.48	17320	0.013
10.68	31310	0.023
10.82	3270	0.002
10.94	1248	0.001
11.09	4644	0.003
11.16	6484	0.005
11.32	6940	0.005
11.42	23640	0.017
11.67	3522	0.003
11.75	1049	0.001
11.88	1398	0.001
12.02	3254	0.002
12.14	1277	0.001
12.27	4251	0.003
12.48	1792	0.001
13.05	1354	0.001

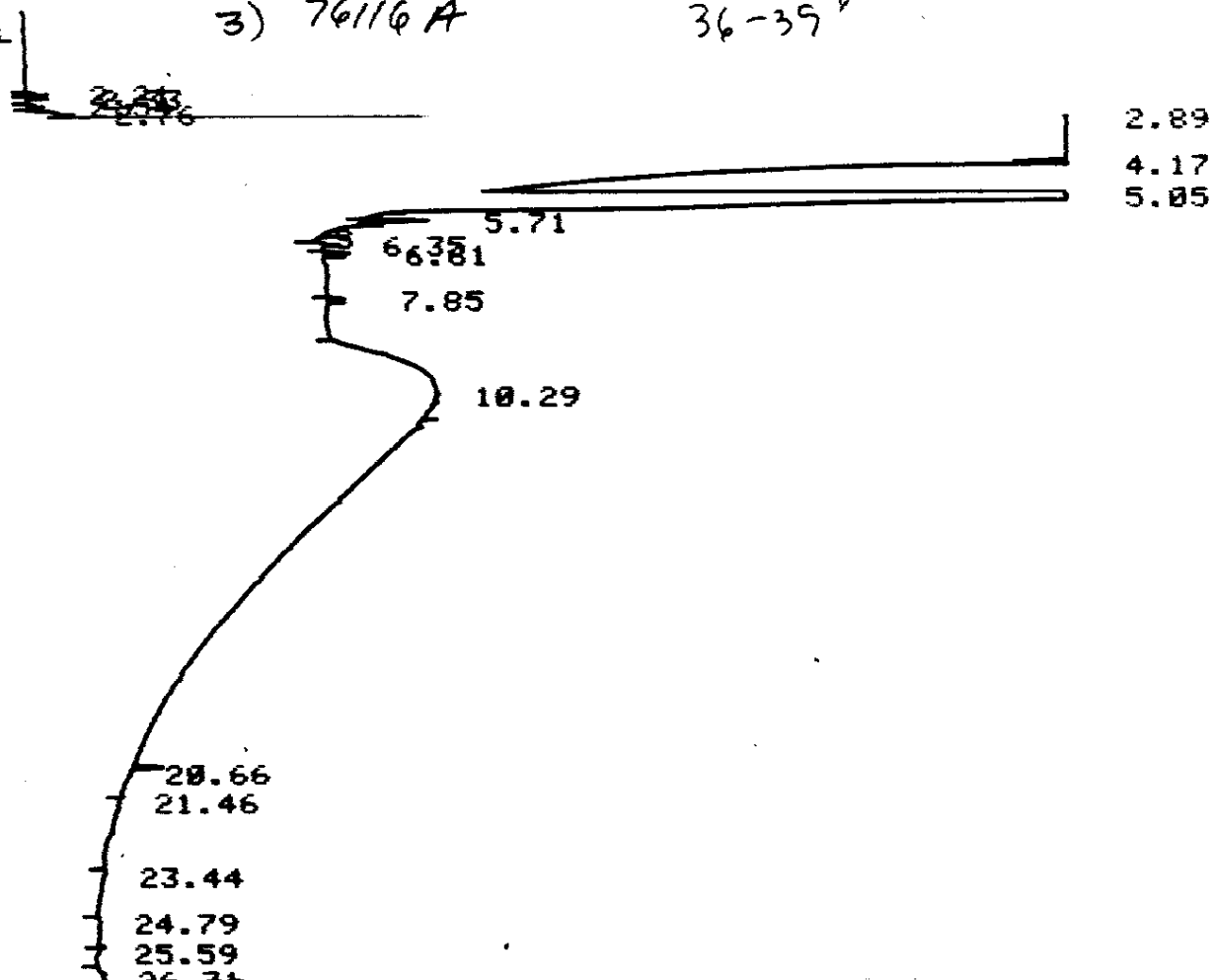
40

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑
START VL

3) 76116 A

36-39



26.55
27.56
28.26
28.98
29.13
ST

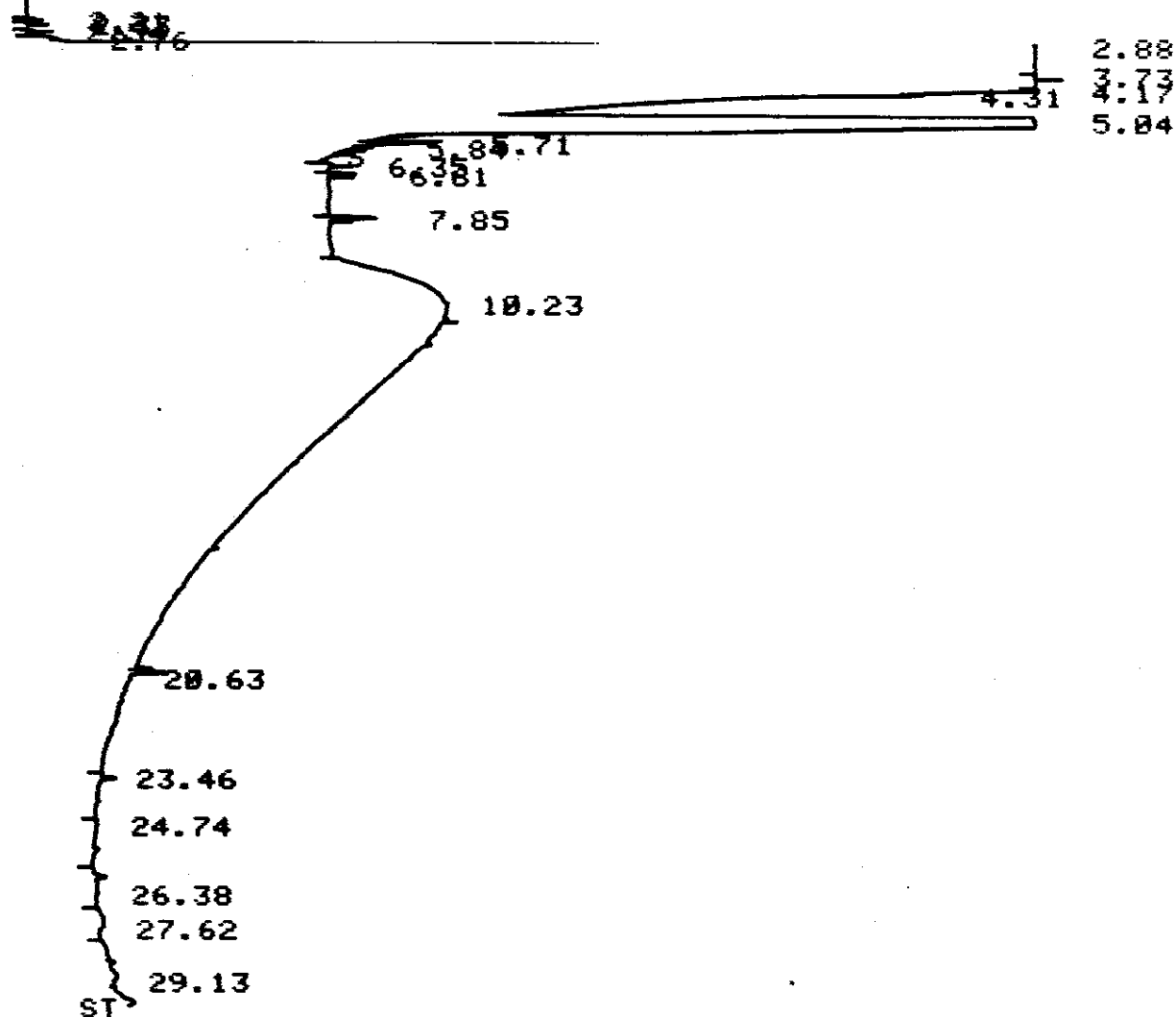
HP RUN # 5 AUG/16/85 TIME 11:42:39
ID:2 BOTTLE 5
AREA %

RT	AREA	AREA %
2.89	158900000	99.731
4.17	2085	0.001
5.05	417100	0.262
10.29	8736	0.005

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VL

4) 76116B 36-39"



HP RUN # 6 AUG/16/85 TIME 12:29:46
ID:2 BOTTLE 6
AREA %

RT	AREA	AREA %
2.88	159400000	99.687
3.73	5243	0.003

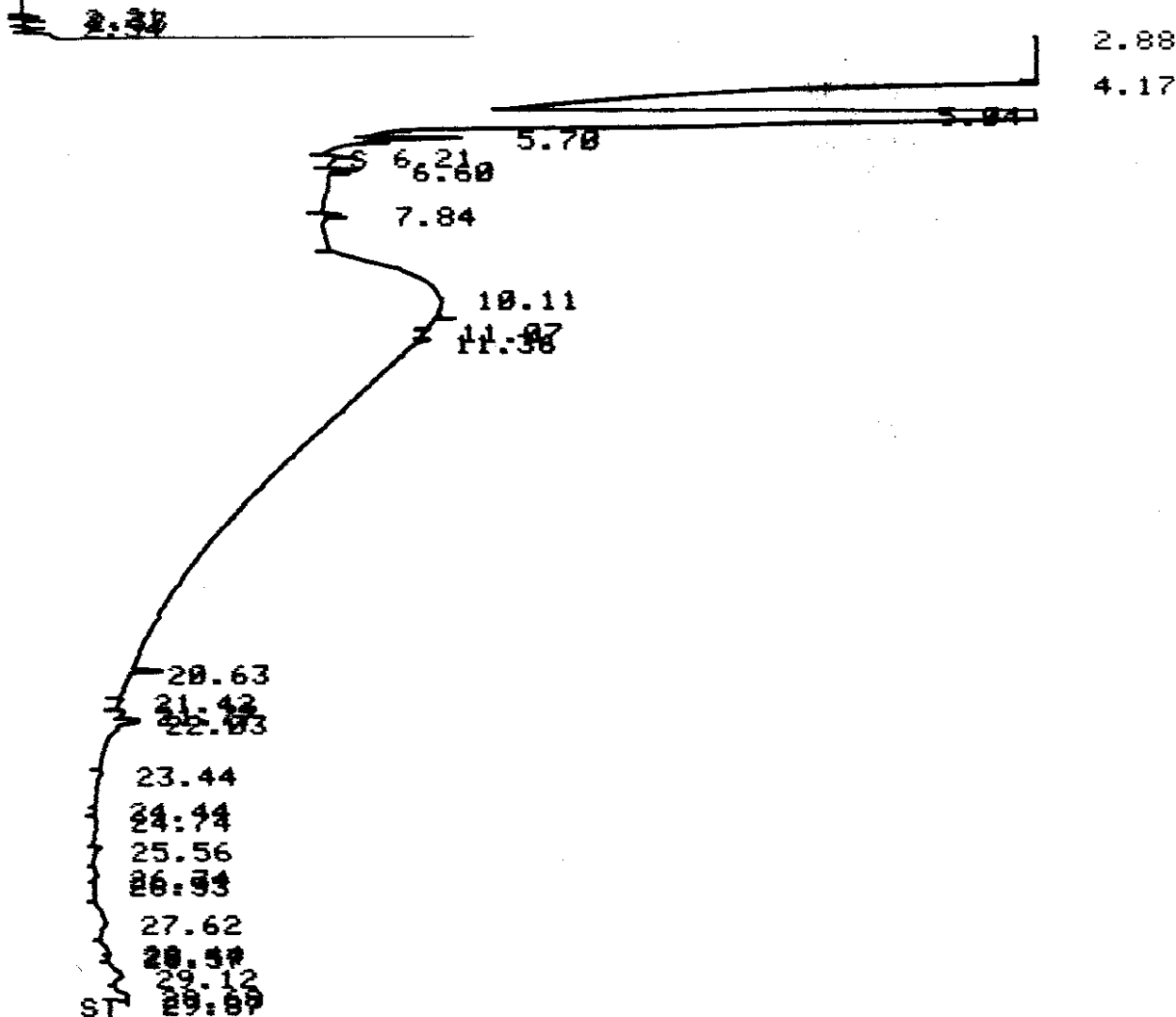
5.04 487500 0.005
10.23 6000 0.004

DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
START VL

5) 76117A

39-43"



HP RUN # 7
ID:2
AREA %

AUG/16/85
BOTTLE 7

TIME 13:17:10

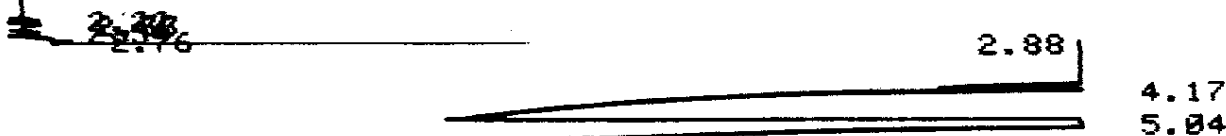
RT	AREA	AREA %
2.88	162700000	99.704
4.17	2388	0.001
5.04	474200	0.291
10.11	6664	0.004

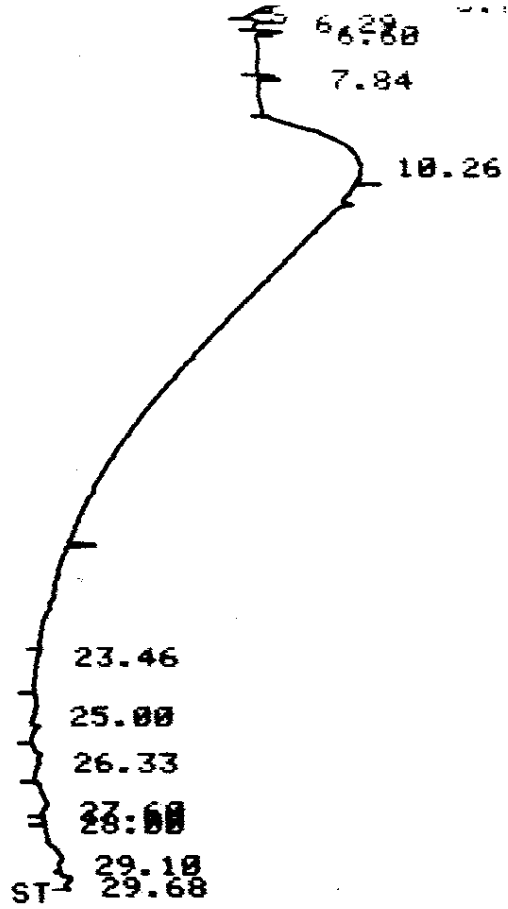
DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
START VL

6) 76117B

39-43"





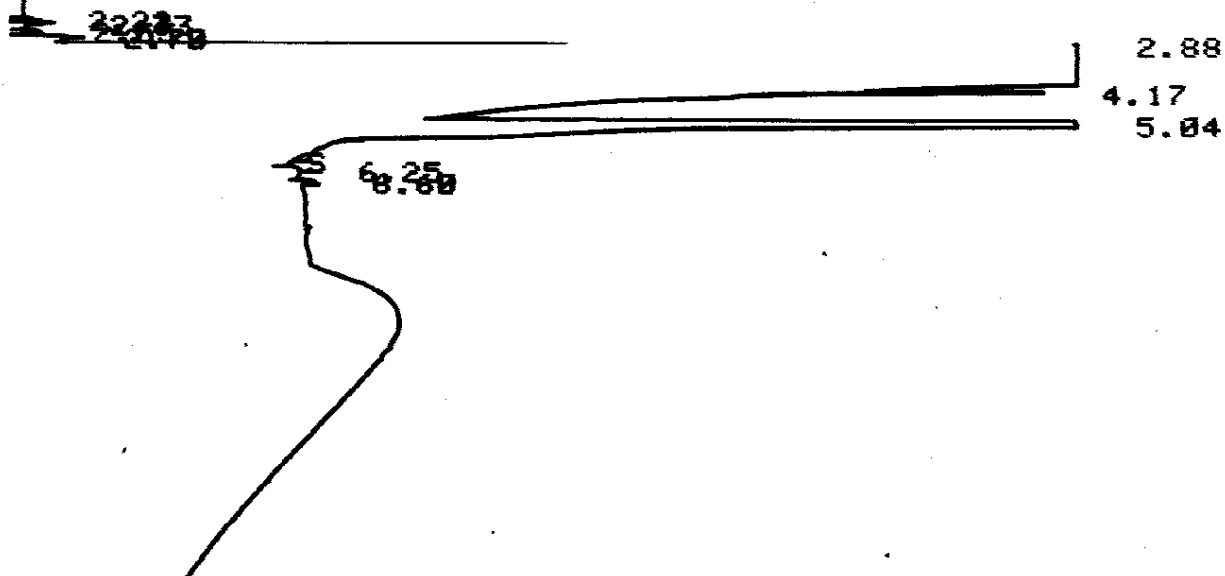
HP RUN # 8 AUG/16/85 TIME 14:04:40
 ID:2 BOTTLE 8
 AREA %

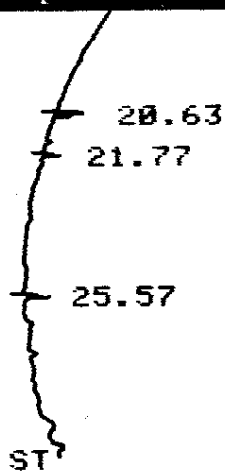
RT	AREA	AREA %
2.88	153500000	99.735
4.17	2080	0.001
5.04	400900	0.260
10.26	5508	0.004

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
 START VL

7) M. Blank E.S.#3912 ext'd 6-10-85





HP RUN # 9
ID: 2
AREA %

AUG/16/85
BOTTLE 9

TIME 14:52:23

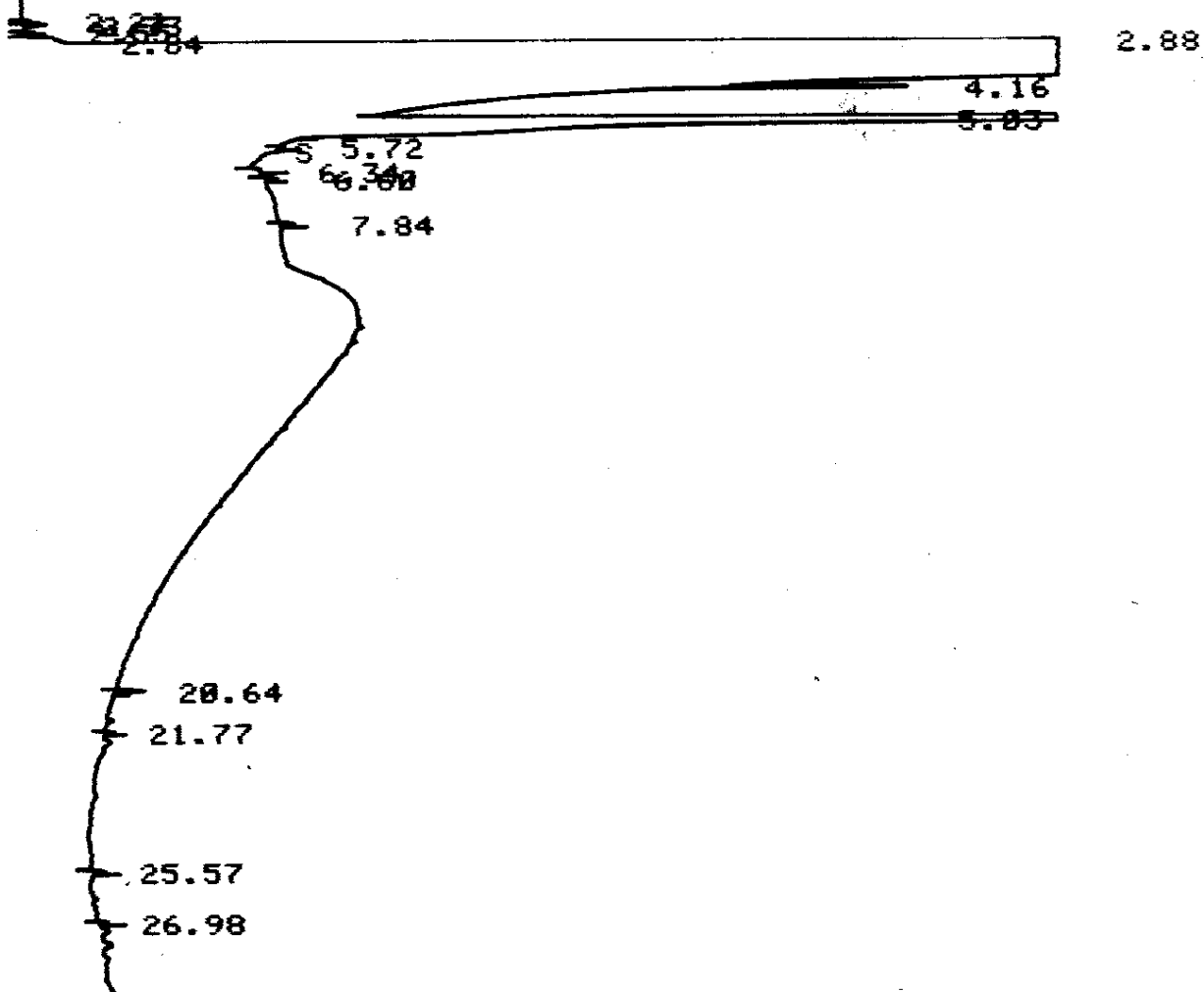
RT	AREA	AREA %
2.88	158000000	99.838
4.17	1047	0.001
5.04	255900	0.162

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑
START VL

8) 76110

18-21

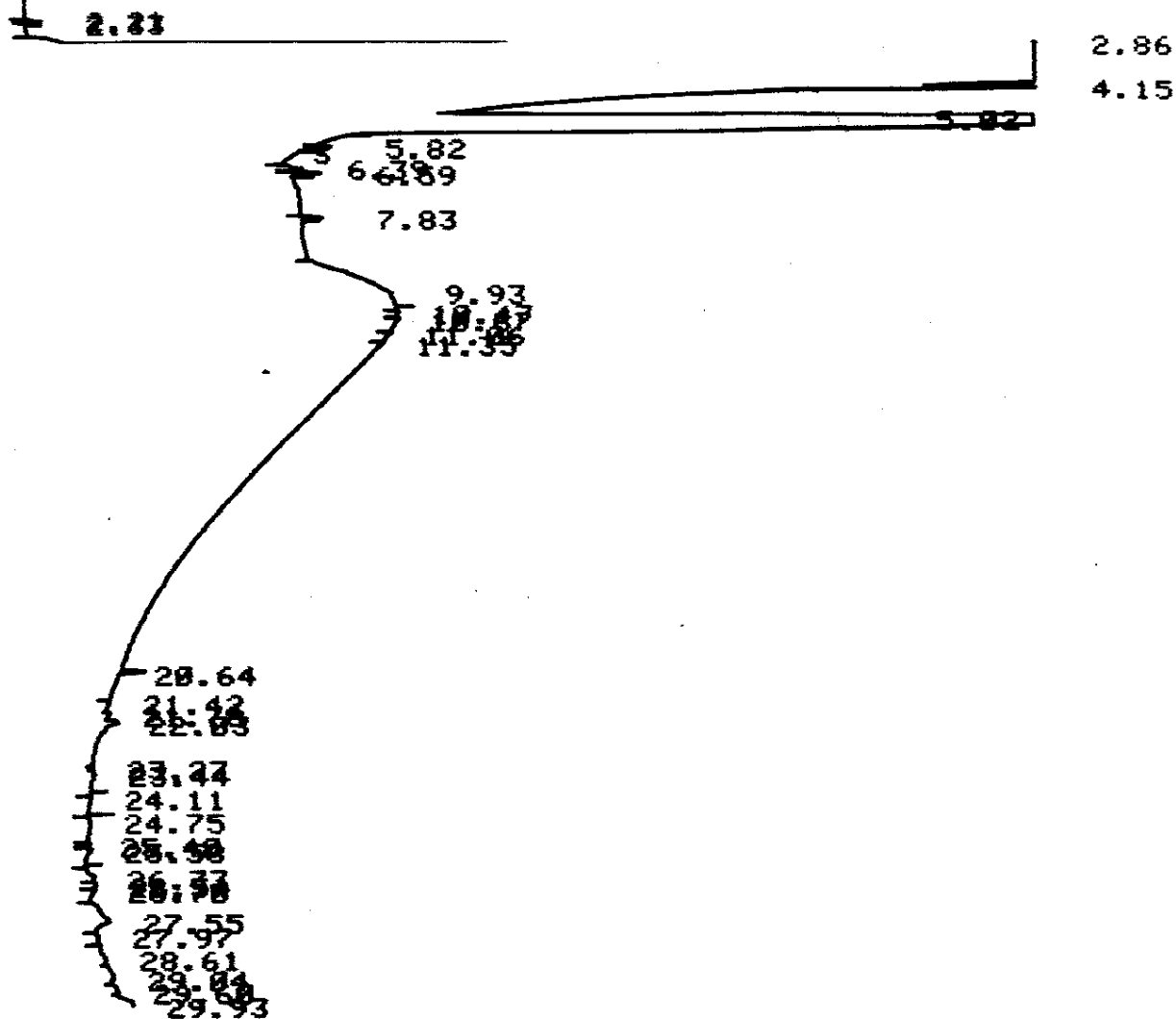


2.86 157700000 99.849
5.02 238500 0.151

DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
START VL

21) Blank E.S. # 3933 Ext'd 6-20-85



HP RUN # 23
ID: 2
AREA %

AUG/17/85
BOTTLE 23

TIME 02:07:44

RT	AREA	AREA %
2.86	159200000	99.685
4.15	2521	0.002
5.02	500000	0.313
27.55	1033	0.001

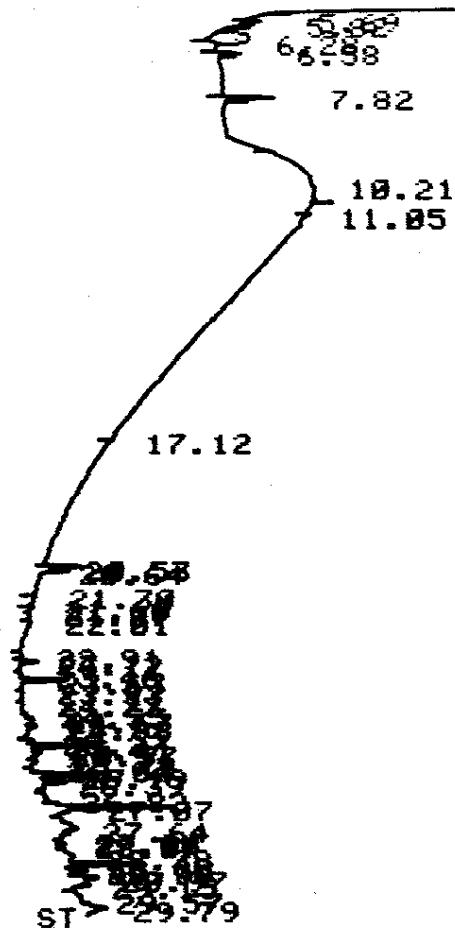
DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
START VL

22) 76949/76959

CBC-5 (0-3")





HP RUN # 24
ID: 2
AREA %

AUG/17/85
BOTTLE 24

TIME 02:55:51

RT	AREA	AREA %
2.86	158100000	99.752
3.72	4479	0.003
4.15	1897	0.001
5.02	373800	0.236
10.21	2071	0.001
26.39	1114	0.001
27.07	2488	0.002
27.64	3079	0.002
28.38	1946	0.001
28.62	1595	0.001

DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
START VL

23) 76950/76960

LC5 (3.6")

2.86

2.86

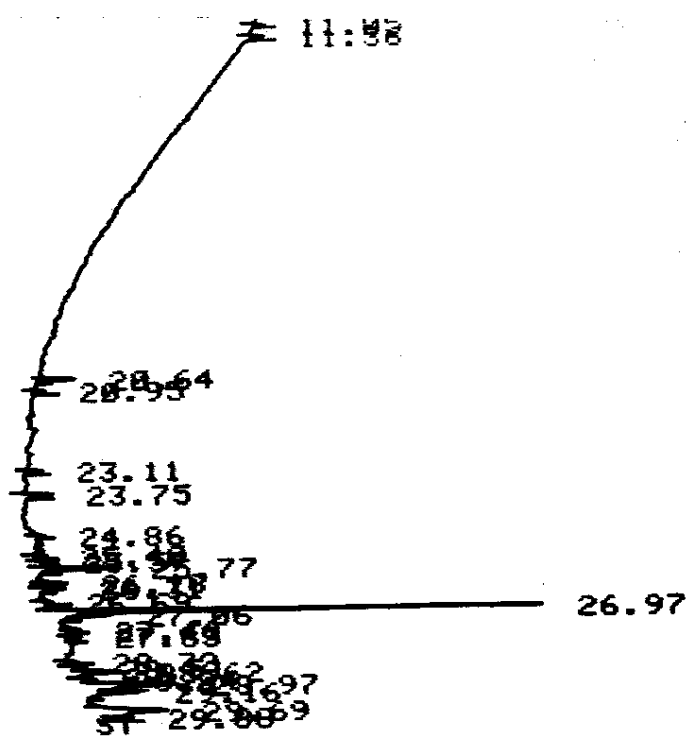
4.15

5.02

5.82
6.88

7.81

11.05



HP RUN # 25
ID: 2
AREA %

AUG/17/85
BOTTLE 25

TIME 03:43:58

RT	AREA	AREA %
2.86	150400000	99.746
4.15	1823	0.001
5.02	376800	0.250
26.97	3211	0.002
28.97	1155	0.001

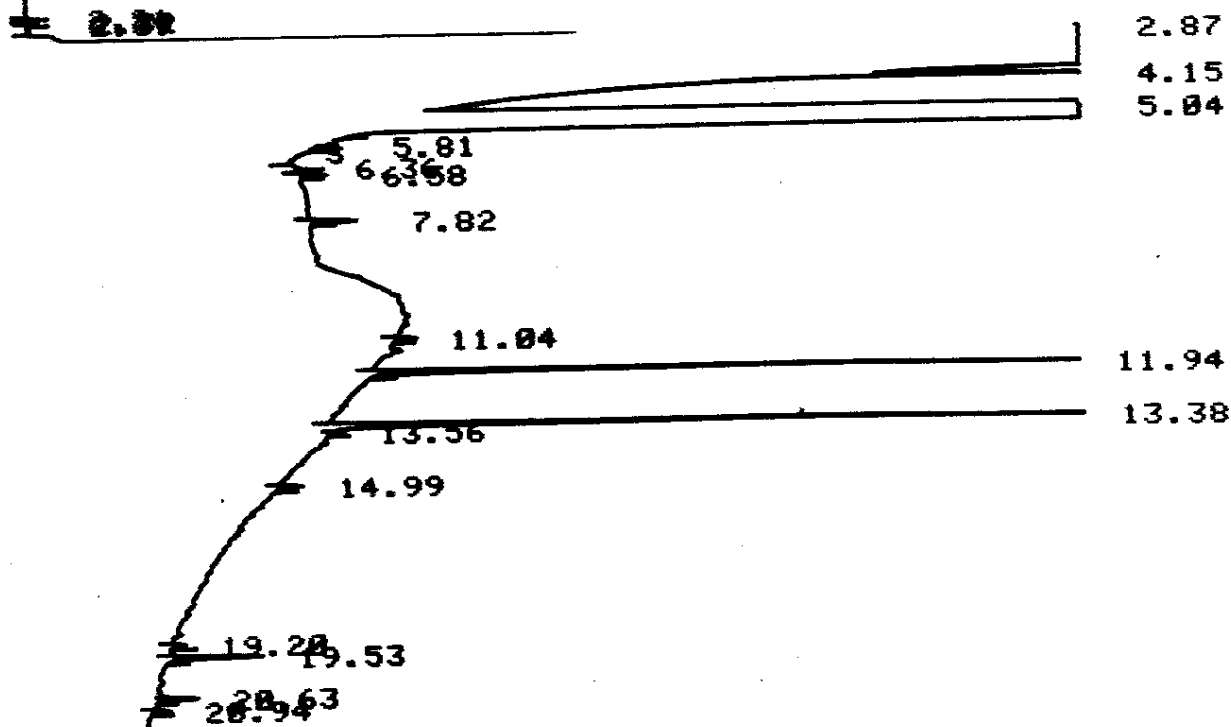
DIL FACTOR: 1.0000 E+ 0

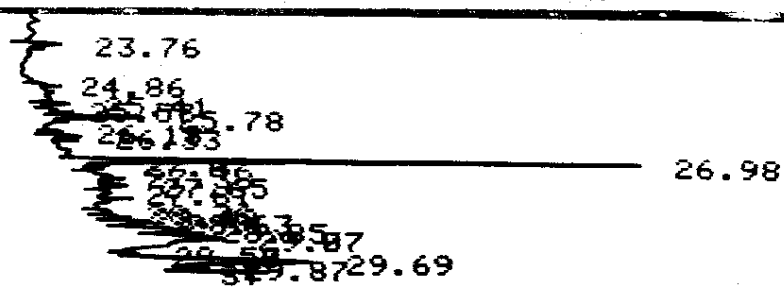
ATTN 2↑
START VL

4

24) 76955/76965

5(6-9')



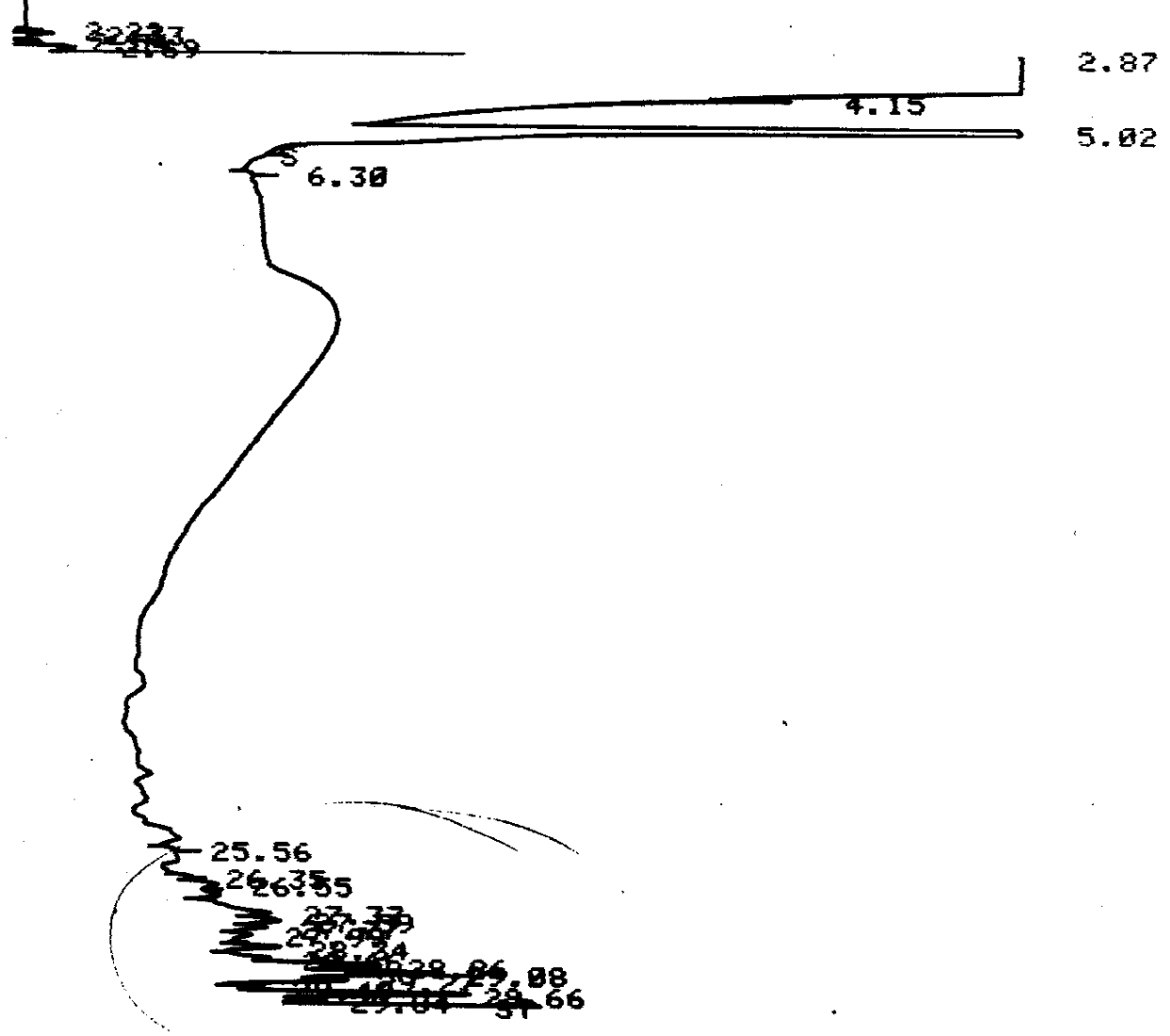


HP RUN # 26 AUG/17/85 TIME 04:32:17
 ID:2 BOTTLE 26
 AREA %

RT	AREA	AREA %
2.87	154800000	99.337
4.15	2300	0.001
5.04	1012000	0.649
11.94	4076	0.003
13.38	6580	0.004
26.98	3648	0.002
29.07	2756	0.002
29.69	2075	0.001

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
 START VL 35) Blank CH₂Cl₂



HP RUN # 27
ID: 2
AREA %

AUG/17/85
BOTTLE 27

TIME 05:28:32

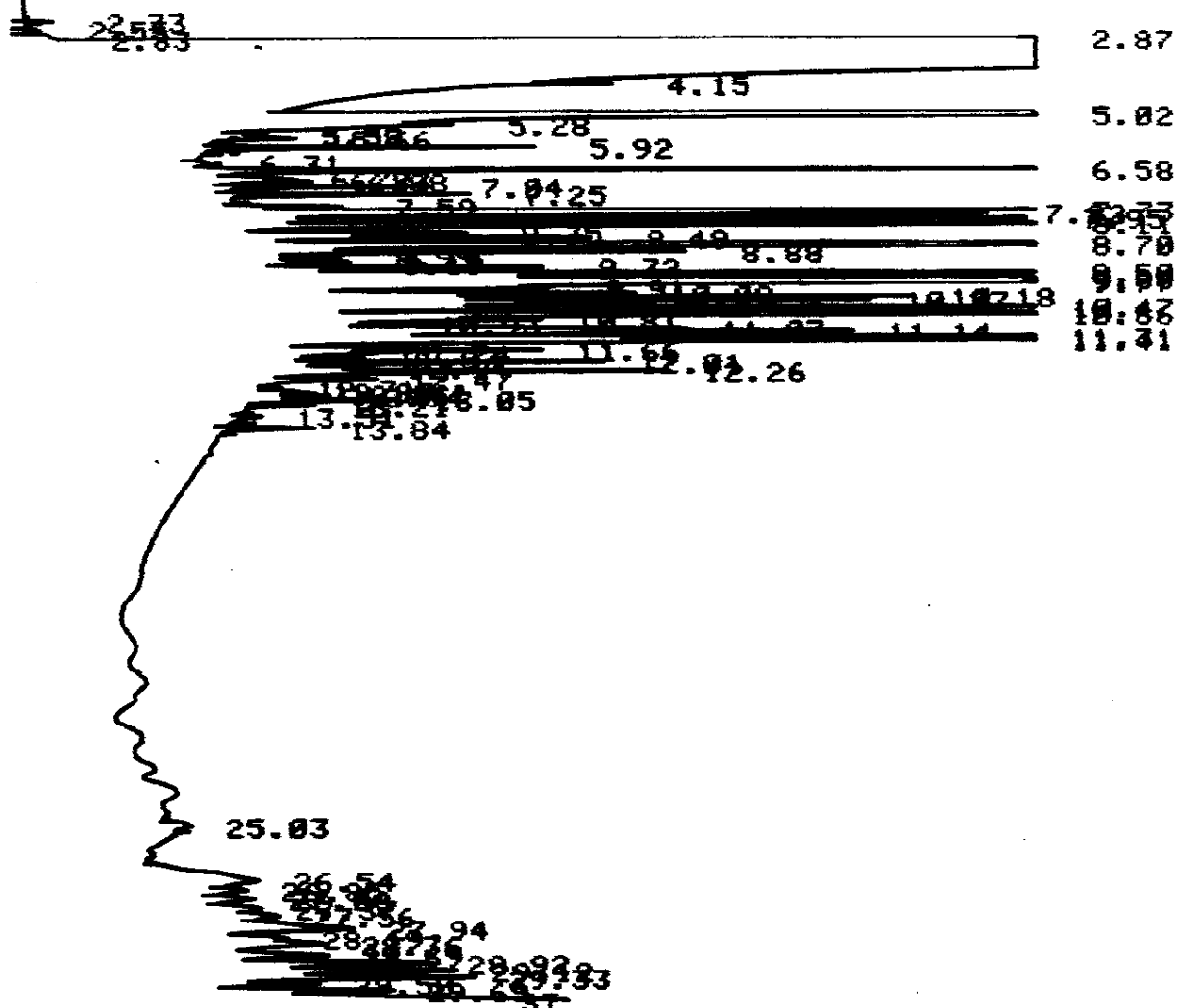
RT	AREA	AREA %
2.87	1478000000	99.864
5.02	186300	0.126
27.37	1629	0.001
27.59	1454	0.001
28.86	2693	0.002
29.08	4228	0.003
29.27	2044	0.001
29.66	2882	0.002

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑
START VL

4

26) Naphtha 264ppm #1661-1



HP RUN # 28
ID: 2
AREA %

AUG/17/85
BOTTLE 28

TIME 06:08:30

RT	AREA	AREA %
2.87	1308000000	99.631
5.02	162200	0.124
5.28	3087	0.002
5.92	2169	0.002

7.04	1423	0.000
7.25	1701	0.001
7.73	8260	0.006
7.82	5545	0.004
7.95	6326	0.005
8.11	12400	0.009
8.35	2664	0.002
8.49	3220	0.002
8.70	33750	0.026
8.88	3485	0.003
8.99	1223	0.001
9.32	2672	0.002
9.50	10380	0.008
9.68	17730	0.014
9.77	13310	0.010
9.91	2634	0.002
10.08	4478	0.003
10.18	5084	0.004
10.27	4640	0.004
10.36	2961	0.002
10.47	17650	0.013
10.66	32010	0.024
10.81	3414	0.003
10.93	1309	0.001
11.07	4893	0.004
11.14	6594	0.005
11.31	7290	0.006
11.41	24190	0.018
11.66	3727	0.003
11.74	1145	0.001
11.87	1539	0.001
12.01	3473	0.003
12.14	1388	0.001
12.26	4559	0.003
12.47	2145	0.002
13.05. 34	1550	0.001
26.54	4886	0.004
26.85	2385	0.002
27.08	2385	0.002
27.37	2450	0.002
27.56	3216	0.002
27.94	6363	0.005
28.19	2365	0.002
28.36	4336	0.003
28.69	3939	0.003
28.92	4817	0.004
29.12	4739	0.004
29.33	3900	0.003
29.50	1000	0.001
29.69	1385	0.001

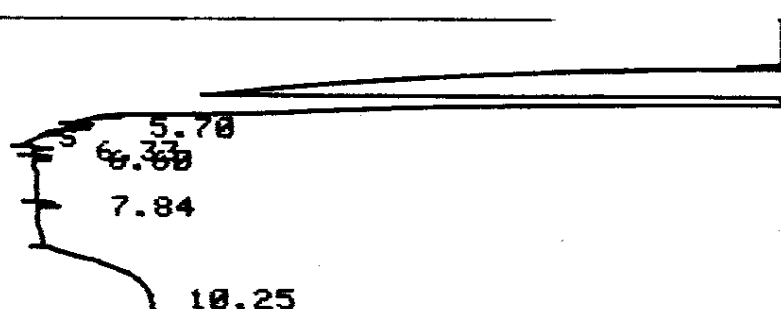
DIL FACTOR: 1.0000 E+ 0

ATTN 21 4
START VL

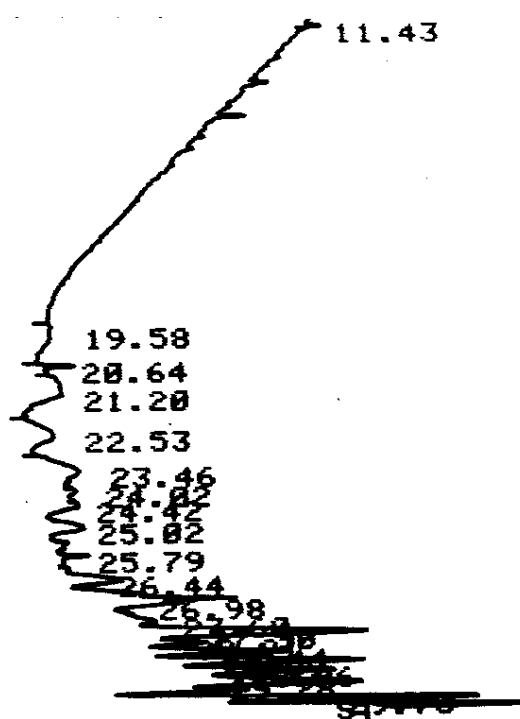
27) 76956/76966

6(9-12")

2.33



2.88
4.16
5.03



HP RUN # 29
ID:2
AREA %

AUG/17/85
BOTTLE 29

TIME 06:56:45

RT	AREA	AREA %
2.88	164100000	99.763
4.16	1211	0.001
5.03	306000	0.186
10.25	6344	0.004
21.20	2793	0.002
22.53	1980	0.001
23.46	3820	0.002
24.42	2468	0.002
25.02	2586	0.002
25.79	2950	0.002
26.44	4792	0.003
26.98	8464	0.005
27.60	5912	0.004
27.90	9046	0.005
28.21	4004	0.002
28.44	5386	0.003
28.69	5999	0.004
28.96	5677	0.003
29.28	8444	0.005
29.73	2794	0.002

DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
START VL

2.83

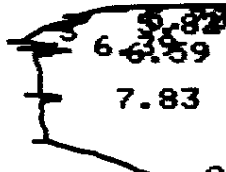
28) 76957/76967

6(12-15")

2.87

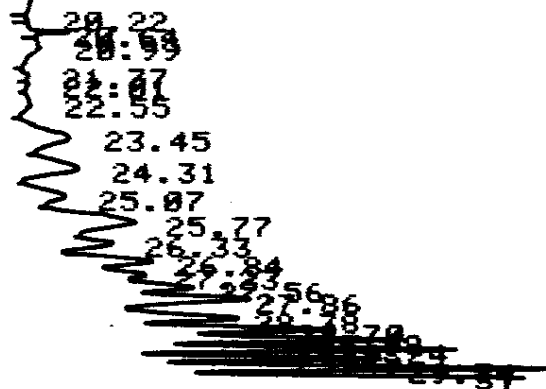
4.16

5.03



Q QQ

18:57
11.06



HP RUN # 30
ID:2
AREA %

AUG/17/85
BOTTLE 30

TIME 07:44:45

RT	AREA	AREA %
2.87	167400000	99.619
4.16	3009	0.002
5.03	562200	0.335
23.45	2924	0.002
24.31	3057	0.002
25.07	2130	0.001
25.77	7100	0.004
26.33	3725	0.002
26.84	4656	0.003
27.23	3790	0.002
27.56	6052	0.004
27.86	7726	0.005
28.38	7538	0.004
28.70	6598	0.004
28.98	6508	0.004
29.24	5339	0.003
29.49	3137	0.002
29.74	5314	0.003

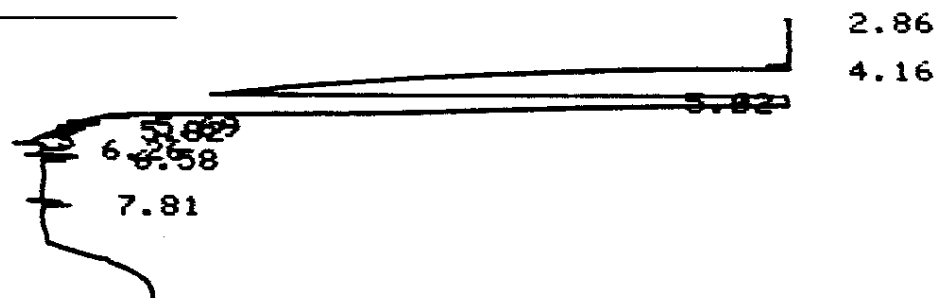
DIL FACTOR: 1.0000 E+ 0

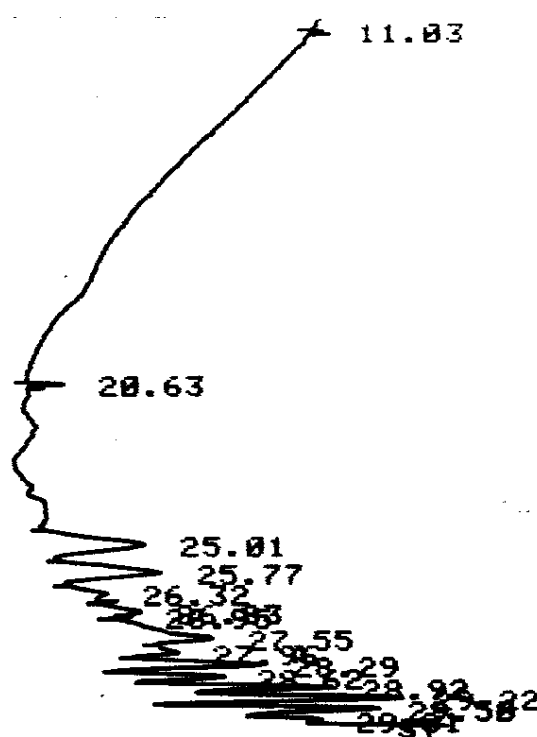
ATTN 21 4
START VL

2.23

29) 76958/76968

6 (15-18")





HP RUN # 31
ID:2
AREA %

AUG/17/85
BOTTLE 31

TIME 08:32:43

RT	AREA	AREA %
2.86	172200000	99.654
4.16	3025	0.002
5.02	561900	0.325
25.01	3814	0.002
25.77	3498	0.002
27.55	4815	0.003
27.96	2414	0.001
28.29	3697	0.002
28.62	2144	0.001
28.92	3675	0.002
29.22	5212	0.003
29.58	2994	0.002

DIL FACTOR: 1.0000 E+ 0

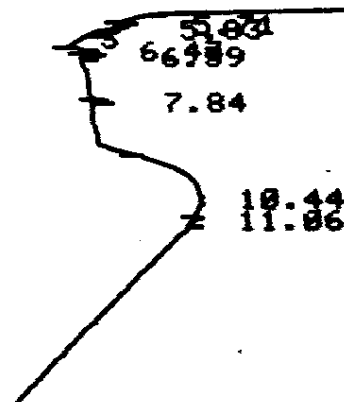
ATTN 2+ 4
START VL

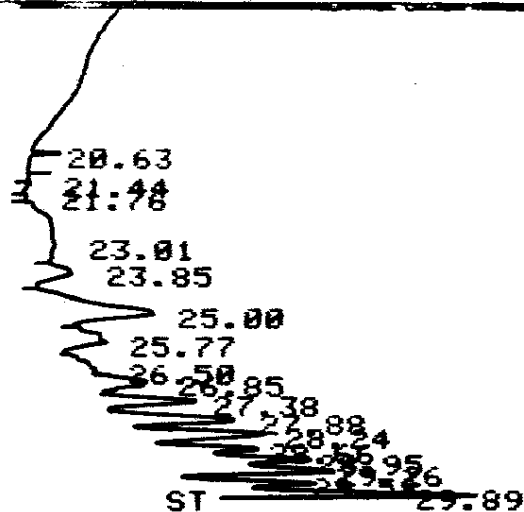
30) 76957/76967 Dup.

6(125')



4.16
5.03





HP RUN # 32
ID:2
AREA %

AUG/17/85
BOTTLE 32

TIME 09:20:49

RT	AREA	AREA %
2.88	158000000	99.721
4.16	1695	0.001
5.03	358500	0.226
10.44	5833	0.004
23.01	4705	0.003
23.85	2675	0.002
25.00	8586	0.005
25.77	5362	0.003
26.50	3930	0.002
26.85	6242	0.004
27.38	6220	0.004
27.88	8250	0.005
28.24	7984	0.005
28.66	4392	0.003
28.95	4998	0.003
29.26	5089	0.003
29.58	2602	0.002
29.89	4894	0.003

DIL FACTOR: 1.0000 E+ 0

ATTN 21 4
START VL

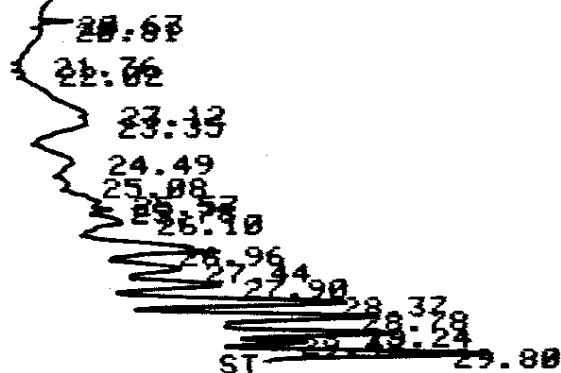
31) 76958/76968 Dup.

6 (15-18")

2.33
2.75

2.89
4.17
5.04

5.73
6.361
7.85
10.16
11.08
11.42
12.28
12.96
13.87



HP RUN # 33
ID:2
AREA %

AUG/17/85
BOTTLE 33

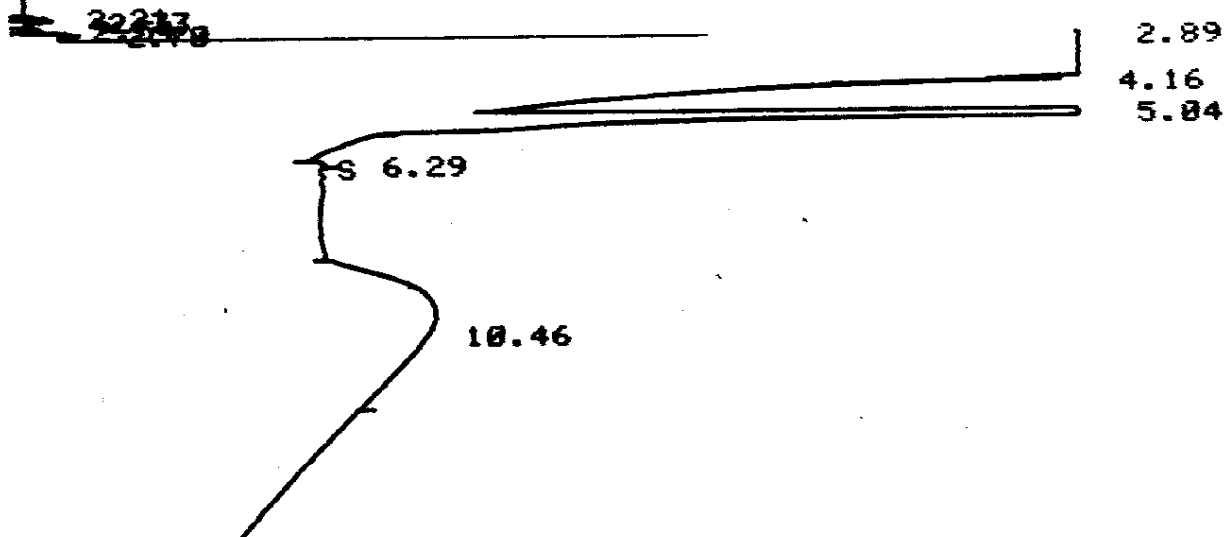
TIME 10:08:46

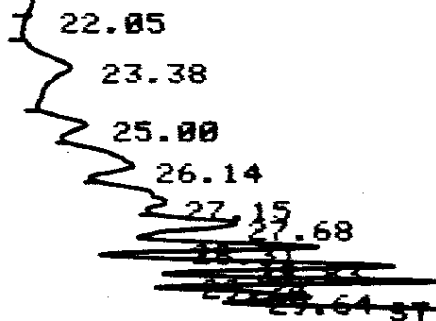
RT	AREA	AREA %
2.89	159300000	99.683
4.17	1970	0.001
5.04	426200	0.267
10.16	6526	0.004
23.12	4085	0.003
23.35	2892	0.002
24.49	3870	0.002
25.08	1770	0.001
25.57	4124	0.003
25.78	1271	0.001
26.10	5751	0.004
26.96	9516	0.006
27.44	7740	0.005
27.90	6239	0.004
28.37	7104	0.004
28.78	6510	0.004
29.24	4568	0.003
29.48	1354	0.001
29.80	4826	0.003

DIL FACTOR: 1.0000 E+ 0

ATTN 2+ 4
START VL

32) Blank CH₂Cl₂





HP RUN # 34
ID:2
AREA %

AUG/17/85
BOTTLE 34

TIME 10:56:02

RT	AREA	AREA %
2.89	154800000	99.813
5.04	198700	0.128
10.46	26290	0.017
23.38	5217	0.003
25.00	4457	0.003
26.14	10110	0.007
27.15	11630	0.007
27.68	11440	0.007
28.31	6550	0.004
28.83	7450	0.005
29.24	5861	0.004
29.64	2992	0.002

DIL FACTOR: 1.0000 E+ 0

ATTN 2↑ 4
START VL STOP

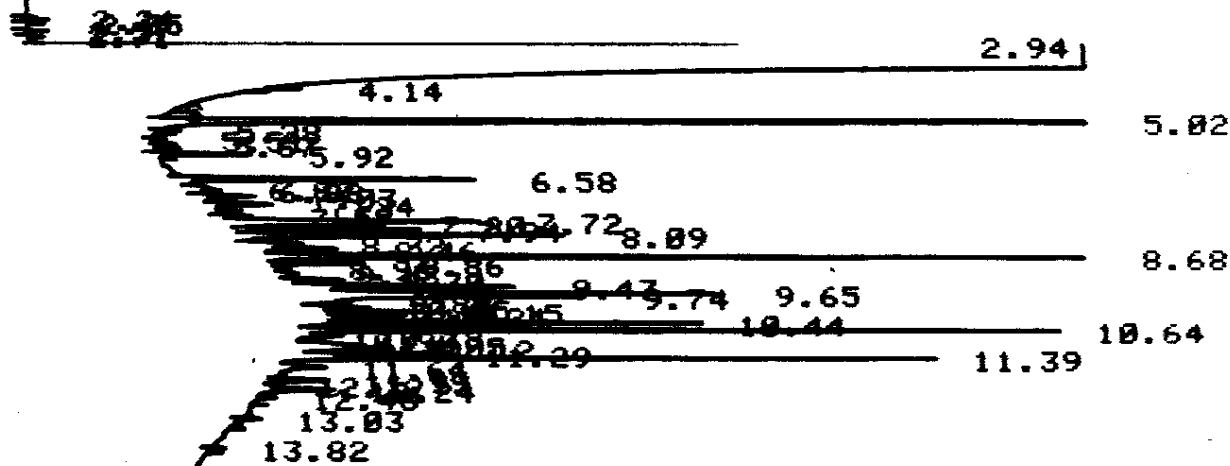
air run out

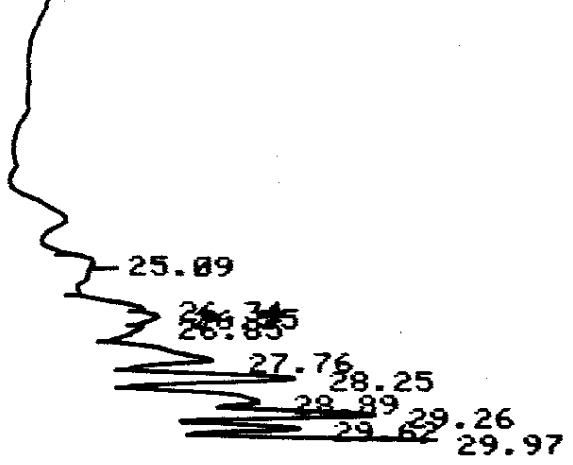
HP RUN # 35
ESCAPE
START VL

AUG/17/85

TIME 12:40:43

33) Naptha 264 ppm # 1661-1





HP RUN # 36
ID:2
AREA %

AUG/17/85
BOTTLE 35

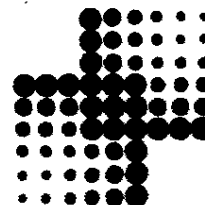
TIME 12:56:20

RT	AREA	AREA %
2.94	47498888	99.688
5.82	49240	0.183
6.58	1822	0.004
7.72	1932	0.004
7.88	1129	0.002
7.94	1568	0.003
8.89	3864	0.006
8.32	1867	0.002
8.46	1886	0.002
8.68	7812	0.016
8.86	1273	0.003
9.13	1347	0.003
9.38	1334	0.003
9.47	3134	0.007
9.65	4391	0.009
9.74	3434	0.007
9.88	1188	0.002
10.85	1859	0.004
10.15	1635	0.003
10.24	1498	0.003
10.32	1129	0.002
10.44	4316	0.009
10.64	6578	0.014
10.78	1549	0.003
11.85	1748	0.004
11.12	2882	0.004
11.29	1837	0.004
11.39	5134	0.011
11.64	2734	0.006
12.24	1812	0.002
26.34	1578	0.003
26.55	3171	0.007
26.85	2453	0.005
27.76	6893	0.013
28.25	4939	0.010
28.89	4748	0.010
29.26	5488	0.012
29.62	2384	0.005

DIL FACTOR: 1.0000 E+ 0

ATTN 21 4

APPENDIX X
STANDARD OPERATING PROCEDURE, LAB NOTES AND
COMMENTS FOR GREASE AND OIL EXTRACTION;
AND GENERAL ANALYSIS COMMENTS





ENVIRODYNE ENGINEERS

12161 Lockwood Road
St. Louis, Missouri 63146
(314) 434-0960

August 20, 1985
2086-00110

Dr. Jacqueline Michel
Research Planning Institute, Inc.
925 Gervais St.
Columbia, SC 29201

Dear Dr. Michel:

Enclosed are the latest revisions/reanalyses results for several of the Dupont L-Area basin samples. The standard operating procedures for petroleum hydrocarbons and bulk chemistry are also enclosed. A brief discussion of the enclosed information follows.

1. The uranium reanalysis to achieve a lower detection limit of 2 ug/g was done on July 31, 1985. Triplicate analysis on the samples was not possible due to the short sample quantity (5 gms of sample was required for each analysis).
2. The nickel and zinc data were recalculated using the detection limits recommended by the instrument manufacturer. We can routinely measure these metals at these lower limits, but generally use the higher detection limits because we are in the process of developing our own in-house detection limits (the start point of which is to double the instrument detection limits and then carry out tests to see how much lower you can go).
3. A reanalysis for mercury at a detection limit of 0.01 ug/g was not done because the additional sample required to meet this limit would also increase the amount of interferences present.
4. Reanalysis for beryllium, cadmium, and selenium were not possible since two separate digestions would be required and there was not sufficient sample available.
5. Analysis for hexachrome was not possible since the "wet" unprocessed sample is required for that analysis. The "grease and oil" analyses exhausted that sample supply.
6. The grease and oil extractions (with freon) for the LBC 6 6-9, 9-12, LBC 5 0-3, and 3-6 suggest the presence of high levels of hydrocarbons. However the GC analyses for these samples (based upon a naptha standard) suggest the presence of high molecular weight hydrocarbons - not low weight, petroleum hydrocarbons. The chromatographs enclosed are from a reanalysis of the sample extracts using a lower standard value. This reanalysis, along with the original analysis, support the absence of low molecular weight, petroleum hydrocarbons in the sample.

ENVIRODYNE ENGINEERS

Dr. Jacqueline Michel


August 20, 1985

Page 2

7. The NBS standards data are provided. In the case of those metals where a suitable NBS standard is not available, an aqueous standard was run to verify the calibration curve. At this time there are still a few metals which fall into that category. The spiking of a blank soil for a central standard is not normally done because of the inability to match the matrix of the unknown soil. For those metals where the recovery was poor, a special situation exists. We use the data from these metals to match against the historical recovery data. There will be differences between our data and NBS certified values because of instrumentation differences. One cannot compare Mass Spectrometry, Neutron activation, and polarity to the Inductively Coupled Argon Plasma or Atomic Absorption. In all cases an external check standard is used to verify instrument operations.

If you have any questions, please feel free to call.

Sincerely,


John J. Coniglio
Program Manager

JJC/bsm
Enclosure

SOP # EEI 17

Date: May 26, 1985

Prepared by: Charles Johnston

STANDARD OPERATING PROCEDURE
GREASE AND OIL EXTRACTION METHOD FOR SLUDGE SAMPLES

APPLICATION

Drying acidified sludge by heating leads to low results. Magnesium sulfate monohydrate is capable of combining with 75% of its own weight in water in forming $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ and is used to dry sludge. After drying, the oil and grease can be extracted with trichlorotrifluoroethane.

Ref: Standard Methods

PROCEDURE

Extraction apparatus, Soxhlet; vacuum pump or other source of vacuum; extraction thimble, paper; IPS filter paper.

In a 150 ml beaker, weigh a sample of wet sludge, 20 ± 0.5 g, of which the dry solids content is known. Acidify to pH 2.0 (generally, 0.3 ml conc HCl is sufficient). Add 25 g $\text{MgSO}_4 \cdot \text{H}_2\text{O}$. Stir to a smooth paste and spread on sides of beaker to facilitate subsequent removal. Let stand until solidified (15 to 30 minutes). Remove solids and grind in a porcelain mortar. Add the powder to a paper extraction thimble. Wipe beaker and mortar with small pieces of filter paper moistened with solvent and add to thimble. Fill thimble with glass wool or small glass beads. Extract in a Soxhlet apparatus, using trichlorotrifluoroethane, at a rate of 20 cycles/hr for 4 hours. If any turbidity or suspended matter is present in the extraction flask, remove by filtering through IPS filter paper into another weighed flask. Rinse flask and filter with solvent. Distill solvent from extraction flask in water at 70°C for 15 seconds. Draw air through it using an applied vacuum. Cool in a desiccator for 60 minutes and weigh to constant weight.

INSTRUMENT OPERATION

Soxhlet apparatus should be thoroughly cleaned with detergent and hot water. Then rinsed with acetone and freon to remove any grease and oil.

Measure all digits of display on balance. Round off insignificant figures when reporting final results of grease and oil.

REAGENTS

1. Hydrochloric acid, HCl, conc.
2. Magnesium sulfate monohydrate: Prepare $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ by overnight drying of thin layer in an oven at 150°C . (Sodium sulfate anhydrous may be substituted for magnesium sulfate.)
3. Trichlorotrifluoroethane (1,1,2-trichloro-1,2,2-trifluoroethane)

INTERFERENCE: Heavier residuals of petroleum may contain a significant portion of materials insoluble in trichlorotrifluoroethane

STANDARD PREPARATION

Measure an amount of oil (Crisco vegetable oil) for extraction. Record percent recovery.

SAMPLE PREPARATION

Preservation: Store at 4°C.

QA/QC

One blank, one standard (% recovery), and one duplicate for every ten samples. It is the responsibility of the chemist/technician to check the control charts after the analysis is performed, and notify the section manager for out of control situations.

CALCULATION

Oil and grease (as % of dry solids) = $\frac{\text{gain in weight of flask, g} \times 100}{\text{weight of wet solids, g} \times \text{dry solids fraction}}$

ug/g grease and oil = % grease and oil $\times 10^4$

DETECTION LIMIT

5 ug/g

SAFETY

Beware of heating mantles and of hot solvent when Soxhlet is running. Follow all EEI safety regulations.

DATA SHEET

Sample copy of data sheet attached; modify as needed. An updated version is being prepared.

STANDARD OPERATING PROCEDURE FLAME ATOMIC ABSORPTION

APPLICATION

Metals in solution may be readily determined by atomic absorption spectroscopy. The method is simple, rapid and applicable to a large number of metals in drinking, surface and saline waters, and domestic and industrial wastes. While drinking waters free of particulate matter may be analyzed directly, domestic and industrial wastes require processing to solubilize suspended material. Sludges, sediments and other solid type samples may also be analyzed after proper pretreatment.

PROCEDURE

Setting Instrument Conditions

1. Make sure the drain tube is filled with DI water.
2. Press power button ON.
3. Turn restart/std-by switch to OPERATE. The instrument automatically sets to the double beam mode. The AUTO button lights.
4. Place the proper burner head, air-acetylene or nitrous oxide-acetylene, in position. Wet the bottom of the burner head with DI water so that it will slide over the O-ring easier.
5. Turn the voltage knob down before inserting the hollow cathode lamp. Place the hollow cathode lamp in turret. Turn the voltage knob to proper operating current.
6. Turn the bandwidth switch to proper setting..
7. Using the wavelength selector, set the wavelength to the value recommended for the element to be analyzed.
8. Adjust the voltage as necessary when peaking out the energy meter. The optimum indication on the energy meter is within the green zone on the meter. Using the adjustable zoom lens and the wavelength selector, make sure that all settings combine to give the lowest reading on the meter.

Igniting the Flame

A. Air/Acetylene

1. Align the burner head with the hollow cathode beam.
2. Turn on the gas tanks that are needed. Make sure that the two-stage regulator is at the proper settings.

- a. Above 100 psi on the tank contents
 - b. 14-15 psi is the working range for acetylene
 - c. 50 psi is the working range for nitrous oxide
3. Set the oxidant and fuel flow rates at their proper settings.
 - a. Air (oxidant) is set at 14 SCFH
 - b. Air/fuel (fuel) is set at 6 SCFH
 4. Press the pilot button to light the flame.
 5. Aspirate with DI water.
 6. Check the nebulizer flow.
 - a. 2.5 mls/30 seconds for air/acetylene flame
 - b. 3.0 mls/30 seconds for nitrous oxide/acetylene flame
 7. Warm up the burner head for about 10 minutes with an oxidizing (fuel-lean) flame for air/acetylene and with a fuel-rich flame for nitrous oxide.

B. N₂O/Acetylene

1. Light the air/acetylene flame as above using the N₂O burner head. DO NOT ASPIRATE WATER.
2. Increase your fuel while decreasing your oxidant. The flame should become very white.
3. Warm up the burner head for 5 to 7 minutes.
4. Turn knob from air/fuel to N₂O/fuel.
5. Place aspirator tubing in DI after N₂O takeover.
6. DO NOT RUN IN A LEAN CONDITION! A "red feather" is wanted in the flame. If a red feather does not appear:
 - a. Increase the fuel
 - b. Decrease the oxidant
 - c. If necessary, increase sample flow by turning the knob on the nebulizer clockwise.
7. Once a red feather has been established, let the burner head warm up for 2 to 3 minutes.

Zeroing the Instrument

1. Zero the instrument by pressing zero while aspirating matrix blank solution.
2. After zeroing, aspirate the highest standard and press read. Obtain the greatest absorbance readings while adjusting the burner head (vertically and horizontally) and optimizing flame conditions. To stop, press read.

3. Re-zero the instrument.
4. Re-check the absorbance value again for the highest standard. (The re-zeroing and absorbance checking might have to be repeated 2 to 4 times to get the best reading.)

Calibrating the Instrument

1. SET the integration time. It is normally 4.0 seconds. It changes sometimes with sample availability. The integration time for hydride analysis is 8.0 seconds.
2. Press the mean button twice for standard deviation and relative standard deviation.
3. Aspirate the desired standard.
 - a. Press standard number
 - b. Press enter
 - c. Enter concentration value
 - d. Press enter
 - e. Press read. Take three readings.
 - f. Press enter. (The instrument will give you 4 readings since it is in auto mode.)
4. Enter the 4 to 5 standards to be used for the calibration curve by repeating step 3 for each. If the instrument gives a flex curve, check the standard make up.

Sample Analysis

1. Four readings per sample is normal procedure.
2. Periodically check the zero with matrix blank and the calibration with calibration standards. Re-zero and re-calibrate as necessary. Only use the standard used as standard #1 in calibration for re-calibration.

Instrument Shutdown

A. Air/Acetylene

1. Remove aspirator tubing from DI water.
2. Turn the knob from air/fuel to air, let the flame go out, then from air to off.
3. Turn off the acetylene gas tank depressurizing the line.

B. N₂O/Acetylene

1. Remove aspirator tubing from DI water.

2. Turn from N_2O /fuel to air/fuel. Decrease fuel and increase oxidant until slightly fuel-rich air/acetylene flame. Turn from air/fuel to air. After the flame goes out, turn off.
3. Turn off the acetylene and N_2O gas tanks depressurizing the lines.

Hydride Analysis of As and Se with the Use of the ILAVA

1. Place the hydride cell assembly, with the Teflon spacer, into the burner/premix chamber.
2. Adjust the burner mount so that the hollow cathode beam is centered in the absorption cell.
3. Ignite the air/acetylene flame as described above. Heat the hydride cell for at least 20 minutes.
4. Purge nitrogen or argon gas through the cell.
5. Program the instrument with the following information.
 - a. Single beam mode
 - b. Integration time equals 8 seconds
 - c. Peak height mode
6. Take readings one at a time with the use of the Instrumentation Laboratories Atomic Vapor Accessory (ILAVA).
7. Establish a zero baseline with the minimum amount of drift by analyzing and autozeroing on a number of matrix blanks. (All samples and standards have a matrix of 5N HCl.)
8. Run the standards in duplicate at minimum. Average the values and manually plug the absorbance readings into the atomic absorption unit after all the standards have been run.
9. Analyze the samples.
10. Periodically check for drift. Re-zero and re-calibrate as necessary.

REAGENTS

1. Concentrated HCl; concentrated HNO_3 , HF
2. Hydride Analysis: As 1% $NaBH_4$ (1% NaOH)
Se 50% KI (1% Ascorbic Acid)
3. Deionized water
4. Stock standard metal solution

STANDARD PREPARATION

All glassware must be cleaned with 1:1 HNO₃. Use 25, 50, 100, 250 and 500 ul micro-pipets in making the standard solutions. A small quantity of a 1000 mg/l stock solution is poured into a snap cap vial and micro-pipetted into a 100 ml volumetric flask. Routinely 4 to 5 standards are used for calibration curves.

As and Se standards are made up as follows:

1 ml of a 1000 mg/l solution diluted to 100 ml = 10 mg/l
1 ml of a 10 mg/l solution diluted to 100 ml = 0.1 mg/l (or 100 ug/l)
250 ul of a 100 ug/l solution diluted to 25 ml (5N HCl matrix) = 1 ug/l
750 ul of a 100 ug/l solution diluted to 25 ml (5N HCl matrix) = 3 ug/l
1250 ul of a 100 ug/l solution diluted to 25 ml (5N HCl matrix) = 5 ug/l
2000 ul of a 100 ug/l solution diluted to 25 ml (5N HCl matrix) = 8 ug/l

Standard concentrations and detection limits (in mg/l) for the designated elements are listed below. If a fifth standard is run, it is one-half the lowest concentration listed.

<u>Concentrations</u>					<u>Detection Limits</u>
Ag	0.5	1.0	1.5	2.0	0.02
Al	2.5	5.0	7.0	10.0	0.300
Ba	0.5	1.0	2.0	5.0	0.10
Be	0.5	1.0	1.5	2.0	0.01
Ca	0.5	1.0	2.0	4.0	0.01
Cd	0.5	1.0	1.5	2.0	0.04
Cr	0.5	1.0	2.0	4.0	0.04
Cu	0.5	1.0	2.0	4.0	0.04
Fe	0.5	1.0	2.0	4.0	0.05
K	0.5	1.0	2.0	5.0	0.01
Mg	0.5	1.0	1.5	2.0	0.02
Mn	0.5	1.0	2.0	5.0	0.04
Na	0.5	1.0	1.5	2.0	0.04
Ni	0.5	1.0	2.0	4.0	0.10
Ps	1.0	2.0	5.0	10.0	0.01
Zn	0.5	1.0	1.5	2.0	0.01

INTERFERENCES

1. Suspended and dissolved solids
2. Viscosity of samples
3. Chemical and spectral interference

QUALITY ASSURANCE/QUALITY CONTROL

1. Four to five standards used in calibration curve
2. Matrix blank used to zero the instrument

3. Daily control standards (used to calibrate instrument) routinely read between samples to check for drift
4. Digested sample blanks read twice for QA/QC
5. Digested standards and duplicates analyzed
6. EP Toxicity samples spiked

CALCULATION

Strip chart read outs in concentrations of mg/l or ug/l, depending on how the curve was entered. If a dilution was required, multiply by the dilution factor.

Solid:Wet Sample

$$\text{mg metal/kg sample} = \frac{(Z/1000)V}{W \times P}$$

where: Z = ug/l of metal in processed sample from calibration curve
 V = final volume of processed sample in ml
 W = weight of wet sample in grams
 P = percent solids

SAFETY

1. No smoking in laboratory or around gas tanks
2. Chains should be around gas tanks.
3. Make sure gas tanks at proper pressure settings when in use
4. Check drain tube; make sure filled with water
5. Gloves, protective glasses, laboratory coats must be worn
6. Keep aspirating tubing clear of clogs
7. Keep burner head slots clean
8. O-rings should be changed periodically to avoid wear and tear, and the possibility of flashback
9. Burner heads should not be changed when hot unless asbestos gloves are worn
10. Use proper gases with proper burner heads
11. All gas cylinders must be closed and regulator depressurized at when not in use

12. When changing tanks, check new tank for leaks with SNOOP

13. When using a 5% HF solution to clean hydride cells, caution must be used to avoid possible severe burn

STANDARD OPERATING PROCEDURE
MERCURY

APPLICATION

This method is applicable to drinking, surface, and saline waters, domestic an industrial waste, and will measure total mercury (organic, inorganic) in soils sediments, bottom deposits and sludge type materials. Reference material EPA 245.1 and 245.5

PROCEDURE

Soils

1. Rinse approximately 0.200 g of dried sample (dried at 60°C) with 5 ml DI into BOD bottles. Soil wieight must be known to 0.000g.
2. Add 5 ml aqua regia to sample.
3. Heat 2 minutes in a 95°C H₂O bath.
4. Cool samples and add 50 ml DI.
5. Add 15 ml of 5% KMnO₄ (w/v).
6. Heat 30 minutes in a 95°C H₂O bath.
7. Cool samples.
8. Add 6 ml hydroxylamine hydrochloride 12% w/v x NaCl 12% w/v.
9. Add 55 ml DI.

Water

1. Pipette 100 ml of agitated sample into BOD bottles.
2. Add 5 ml concentrated H₂SO₄.
3. Add 2.5 ml concentrated HNO₃.
4. Add 15 ml 15% w/v KMnO₄.
5. Allow to stand 15 minutes. If brown MnO₂ precipitate is prominent, add more KMnO₄, up to 30 ml and note addition in data.
6. Add 8 ml 5% potassium persulfate.
7. Digest in 95°C H₂O bath for 2 hours.
8. Add 6 ml hydroxylamine hydrochloride 12% w/v x NaCl 12% w/v.

INSTRUMENT OPERATION

The samples and standards are analyzed on an Atomic Absorption Unit (AAU) utilizing an Atomic Vapor Accessory (AVA). The AAU is turned on and put into the operator mode. A mercury hollow cathode lamp is inserted and set at 3 ma. The bandpass is set at 1, wavelength at 253.7 nm, and the strip chart is turned on. The absorbance is checked to insure that it reads 1.000 and the instrument is set into the pk ht mode with an 8 second integration time. The mercury cell is cleaned with 1:1 HNO₃ (ultra pure) and dried. It is set in place and adjusted to maximize the light beam passing through it which is shown by a minimum energy reading (minimum interference).

The AVA is integrated to the AAU and a flow gas of nitrogen is introduced at 20 P.S.I. The flow rate on the AVA is set at 6-7 liters per minute. A 5% SnCl₂ in 3 M HCl solution is introduced and the AVA is thoroughly flushed with this solution (A preliminary flushing with DI is suggested to prevent cross reactions with any reagents left in the tubing). The AVA is calibrated to deliver 2.5 ml \pm 0.25 ml SnCl₂.

DI blanks (reagent blank) are run to insure a response of approximately 0.000 is being achieved and then samples, standards, and method blanks are analyzed.

A correlation >0.9960 between the calibration points is desired and the concentration of samples is read off the plot of these points.

REAGENTS

1. Aqua regia: 3 parts concentrated HCl to one part concentrated HNO₃.
2. 5% KMnO₄ (w/v): 50 g KMnO₄/1 liter.
3. 5% Potassium persulfate: 12.5 g/250 ml.
4. Hydroxylamine hydrochloride: 60.0 NaCl and 60.0 g hydroxylamine hydrochloride/500 ml.
5. SnCl₂: 62.5 ml concentrated HCl and 12.5 g SnCl₂/250 ml.

ANALYSIS

See Procedure.

INTERFERENCE

Possible interference from sulfide is eliminated by the addition of KMnO₄. Interfering volatile materials are removed when the dead air space in BOD bottle is purged with the flow gas in AVA before addition of SnCl₂.

STANDARD PREPARATION

Dilution flasks are acidified with 0.2 ml concentrated HNO_3 . A 5-point calibration standard is prepared from a stock standard of 1,000 ppm Hg:

1 ml of 1,000 ppm diluted to 100 ml yields a 10 ppm standard solution
1 ml of 10 ppm diluted to 100 ml yields a 0.1 ppm standard solution

Calibration standard concentrations:

Method blank (100 ml DI)
0.05 ug/100 ml (0.5 ml of 0.1 ppm)
0.10 ug/100 ml (1.0 ml of 0.1 ppm)
0.30 ug/100 ml (3.0 ml of 0.1 ppm)
0.50 ug/100 ml (5.0 ml of 0.1 ppm)

The preparation of standards is completed as per the type of sample being analyzed.

Soils

Standards are brought up to a volume of 10 ml, and then treated as soil samples except 50 ml instead of 55 ml DI is added in 9.

Water

Standards are brought up to a volume of 100 ml and then treated as H_2O samples.

SAMPLE PREPARATION

See Procedure.

QA/QC

Check standards and duplicates are prepared for every 10 samples in the run. Calibration standards includes a method blank and four standards.

Soil

NBS river sediment (1.1 ± 0.5 ug/g) are prepared as samples, weight recorded to four decimal places.

Water

1.5 ug/l check standards are prepared from separate stock and dilutions that are used for preparing calibration plot. (1.5 ml of 0.1 ppm Hg treated as H_2O sample).

CALCULATION

Soils

1. Peak height is corrected to ug/100 ml from calibration plot.
2. $(\#ug/100ml) \div (g \text{ sample}) = ug/g.$

Water

1. Peak height is converted to ug/100 ml from calibration plot
2. $(\#ug/100 \text{ ml})(100ml/.1 \text{ liter}) = ug/l.$

DETECTION LIMIT

Report data $\geq 0.20 \text{ ug/l}$ or 0.20 ug/g ; anything less is reported as <0.20 .

SAFETY

All sample and standard preparation is done under hood. Dispose of concentrated Hg standards and samples in Hg waste containers. Make sure all hose connections are tight to prevent Hg vapor from leaking out of closed system.

DATA SHEET

No data sheet made up, write results directly in laboratory book.

STANDARD OPERATING PROCEDURE
HYDRIDE DIGESTION FOR WATERS

APPLICATION

This method is used to prepare all waters for determination of arsenic and selenium in waters, using the aa/ae spectrophotometer 457 and IL atomic vapor accessory 440.

PROCEDURE

1. 50 ml of well-shaken, acid preserved sample is measured in a volumetric pipet and transferred to a 300 ml tall form beaker.
2. 5.0 ml of concentrated HCl and 5.0 ml 70% HNO₃ are added to the sample.
3. The sample is covered with a watchglass and digested on the hotplate at about 90°C until near dryness (1-2 ml).
4. After cooling, 25 ml DI water is added to the sample.
5. The sample is covered again and returned to the hotplate, where it is digested to near dryness.
6. After cooling, 42 ml HCl is added to the sample.
7. The sample is transferred quantitatively to a specimen cup and diluted to the 100 ml mark with DI water.

INTERFERENCE

1. Heavy metals in the sample cause problems.
2. HNO₃ in digested sample interferes
3. Losses of selenium seem to be a surface effect of glass.
4. Losses of both arsenic and selenium occur if volume gets too low during digestion, but volume must get low enough to drive off HNO₃.

STANDARD PREPARATION

The stock standards are made up into an intermediate standard from which the standards for each run are taken. This intermediate standard is made fresh daily.

QA/QC

Normally one blank and a pair of standards are run with each set (<20) of samples. A duplicate is also done for every 10 samples.

Cleaning Glassware

Beakers and watchglasses are washed with soap and water, using a wooden-handled brush (wire handles scratch glass). They are then rinsed in tap water followed by alternate DI and 1:1 HNO_3 rinses, a minimum of 2. This followed with four rinses with DI. Beakers are drained and stored upside down on paper towels.

SAFETY

Samples are handled with gloves at all times. Beakers are placed in the hood before any reagents are added, and the digestions are done in the hood as well. The beakers are not removed from the hood until the sample has cooled. Lab coats and safety glasses are worn in the lab at all times. Also, heavier gloves and plastic aprons are available for use when needed.

STANDARD OPERATING PROCEDURE
PLASMA DIGESTION FOR WATERS

APPLICATION

This method is used to prepare all waters for determination of metals on the Plasma 200 (ICAP).

PROCEDURE

1. 200 ml of well-shaken, acid-preserved sample (pH <2) is measured in a graduate cylinder and poured into a 300 ml tall-form beaker.
2. 1 ml of 70% HNO₃ and 4 ml of 30% H₂O₂ are added to the sample.
3. The beaker is covered with a watchglass and the sample is digested on the hotplate at about 90°C until the volume is less than 20 ml.
4. After cooling, the sample is transferred quantitatively to a 50 ml conical tube and diluted to 20ml with DI.

INTERFERENCE

1. Certain samples, particularly E.P. Toxicity extracts, are frequently basis. These must have the pH adjusted to <2 with 70% HNO₃ before adding the reagents.
2. Samples high in organic compounds require additional HNO₃ and H₂O₂ in order to break these compounds down. Normally, enough H₂O₂ is added before digesting to dissolve crystals and lighten sample color (these samples are often dark colored) if possible; but some samples require digestion with repeated small aliquots (1-5 ml) of HNO₃ and H₂O₂ added as volume decreases. The ideal final product will be clear to straw colored.

STANDARD PREPARATION

For convenience, most stock standards are made up into intermediate standards from which the standards from each run are taken. These are as follows.

Standard Ia: May be HNO₃ preserved for one week; add 1 ml HNO₃/ml. 1 ml, 1000 ppm stock, diluted to 1 liter for Be, Zn, Ca, Sr, Mg, Ti, Sn, Co.

Standard Ib: May be HNO₃ preserved for one week; add 0.1 ml HNO₃/100 ml. 1 ml, 1000 ppm stock, diluted to 100 ml for Na, Mn, Fe, Ba, K, Al, Mo.

Standard Ic: Not able to preserve. 1 ml, 1000 ppm stock, diluted to 1 liter for V.

Standard IIa: May be HNO₃ preserved for one week; add 1 ml HNO₃/liter. 0.1 ml, 1000 ppm stock, diluted to 1 liter for Cd; 1 ml, 1000 ppm stock, diluted to 1 liter for Cr, Cu, Pb, Ni.

Standard IIb: Not able to preserve. 1 ml, 1000 ppm stock, diluted to 1 liter for Ag.

Standards are made from intermediates as follows:

Standard I = 10 ml Ia + 10 ml Ib + 10 ml Ic in 300 ml tall-form beaker.

Standard II = 6 ml IIa + 2 ml IIb in 300 ml tall-form beaker.

Standards are then diluted to about 200 ml with DI. A blank of 200 ml of DI is also made. The standards and the blank are digested in the same manner as the samples.

QA/QC

Normally, one blank and one set of standards are run with each batch of samples. (A batch is usually 20 samples or less). Also, one duplicate is run for every 10 samples or fraction of 10. Thus, a batch of 17 samples would include one blank, two standards and two duplicates.

Cleaning Glassware

Beakers and watchglasses are washed with soap and water, using a wooden-handled brush (wire handles scratch glass). They are then rinsed in tap water followed by alternate DI and 1:1 HNO₃ rinses, a minimum of 2. The final rinses are four with DI. Beakers are drained and stored upside down on paper towels.

SAFETY

Samples are handled with gloves at all times. Beakers are placed in the hood before any reagents are added, and the digestions are done in the hood as well. The beakers are not removed from the hood until the sample has cooled. Lab coats and safety glasses are worn in the lab at all times. Heavier gloves and plastic aprons are available when needed.

STANDARD OPERATING PROCEDURE PLASMA METALS

APPLICATION

This method may be used for the determination of dissolved suspended or total elements in drinking and surface waters, domestic and industrial wastewaters, and soil and sludge extractions. Because of the differences between various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided. Instead, the analyst is referred to the instructions provided by the manufacturer of the particular instrument.

SUMMARY OF METHOD

The method describes a technique for the sequential multi-element determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the analyte concentrations.

INSTRUMENT OPERATION

To prepare the Plasma-200 instrument for operation:

1. Turn on the cooling water at the coolant circulator on the floor.
2. Then turn on the argon supply; the operating pressure should be 60 psi at the instrument and 65 to 70 psi at the tank.
3. Turn on the circuit breaker located on the upper right portion of the lower panel.
4. Next, verify that the external venting system is open and operating at between 150 and 190 cubic feet per minute with a vaneometer.
5. Verify that there is water in the drain line.
6. Verify that the anode breaker, located to the left of the main circuit breaker, is in the UP position.
7. Press the eject button opening the cassette drive door, and insert the IL Systems Tape Cassette (green label). All the user program tapes have the systems operation recorded.

8. After inserting the IL Systems Tape Cassette, type <ENABLE> <TAPE>. The screen then displays the word TAPE for 4-1/2 minutes, followed by a count down from 190 seconds (which allows the power supply to warm up).

9. If a "TORCH ERROR" message appears, see section 5.2 of the instrument manual, correct, and clear. Verify a Hg line wavelength calibration. See 120.

10. When the wavelength calibration is complete, the CRT displays the Program Selection Menu.

11. Finally, remove the cassette from the cassette drive.

At this point, the torch may be ignited if desired. BEFORE IGNITING THE TORCH, connect the pump winding to the peristaltic pump. Make sure the capillary tubing is connected properly to the capillary and the pump winding. The pump winding should be disconnected when the IL Plasma-200 is shut down to extend its lifetime. The torch should be ignited and the system allowed to warm up for at least 20 minutes before use.

The following procedure is used for torch ignition:

1. To ignite the torch, press <ENABLE> <TORCH>. The information in Figure 3-3 then appears on the screen.

2. The ENABLE TORCH command ignites the torch and maintains it at power level 5. IL recommends that the system be warmed up at the power level which is to be used during analysis; if the power level for analysis is other than 5, change the power setting at this time by typing n POWER <RETURN> (where n is the desired power level). Most analysis is done at power level 3 or 4. Power level 6 is used for organics only. Refer to Section 3.10 of the instrument manual for operating instructions before using power level 6.

3. If the torch fails to ignite, and the CRT displays only the message "TORCH DIDN'T LIGHT," and does not display an error condition, key <ENABLE> <TORCH>. If the torch continually refuses to ignite, contact your IL service engineer. If there is an error condition displayed, see Section 5.2 of the instrument manual to troubleshoot and type in <CLEAR> <ENABLE> <TORCH>.

4. To end the torch ignition cycle (if necessary), key <ESC>.

5. While the torch is stabilizing, set the I/O Format and the required parameters; the torch should stabilize for at least 20 minutes.

Program Selection Menu

110 is the I/O Format Option. The I/O Format is used to set up the printer, the sampler, and other peripheral devices. The I/O Format must be set up for each program, because it cannot be stored on the mini-cassette. The I/O Format Menu appears displaying the status of each peripheral device. A "#" appears on the screen, prompting a numeric keyboard entry. To activate a particular device, press the appropriate number followed by <RETURN>. If the device is accidentally accessed, keying a <RETURN> will return the # prompt. To turn the device on, type 1 <RETURN>. Keying anything except 1 turns the device off. Turn on the printer (leave paging off). Set operator ID, date, and time. The other devices are optional. #9 exits the I/O Format.

120 is the Wavelength Calibration Procedure Option. This command causes the system to plot the Hg wavelength curve. If the hash marks are more than 2.5 times their own length away from the curve, the optics are out of calibration. (IN THIS CASE, CALL YOUR SERVICE ENGINEER.)

130 is the EDIT Mode Option. The EDIT Mode is used to generate and/or modify methods programs. The full ASCII keyboard must be used when in the EDIT Mode. When in this mode, an asterisk (*) appears on the CRT to prompt keyboard entries.

140 is the Shutdown Procedure Option. The Shutdown Procedure Option describes how to prepare the instrument to shut down the power supply and eventually shut down the Plasma-200 instrument. To select and enter the Shutdown Procedure option, type 140 <RETURN>. The system displays the message "PRESS ENABLE THEN 0" on the screen. Press <ENABLE> and 0. "WAIT 3 MINUTES FOR SHUTDOWN" appears on the screen. (Note: Pressing <ENABLE> and 0 at anytime begins the Shutdown Procedure.) After three minutes the message "TURN OFF CIRCUIT BREAKER" appears on the CRT, and the circuit breaker can be tripped.

PROCEDURE

See Instrument Operation to turn on the instrument. Put instrument in the EDIT Mode. To enter a new program, select the Program Number and Name Option by typing the number (from 1 through 99, inclusive) you wish to assign to the program; then, enter a space by pressing the space bar. Then, type the word ENTER followed by a space and program name (up to 18 characters); then press the RETURN key. To call up the program, type n SEE <RETURN>.

Power Level Option

The Power Level of the plasma discharge is under computer control and can be changed as needed by the operator by using the ASCII keyboard. There are six power settings (1-6) that are available, with 1 being the lowest power, and 6 being the highest power. The default power level is 3.

To select a Power Level other than 3, press the desired setting (from 1 through 6, inclusive) and type PWR. For example, if the Power Level 4 is to be entered, type 4 PWR <RETURN>. To override the existing PWR power setting, use the POWER command, which instantaneously changes the plasma power to the desired level. Then, when any new command causes the monochromator to move from rest, the plasma power returns to the PWR program power setting.

Repeat Analyses and Repeat Readings Option

The two commands *ANAL and *RDG are used to set the number of repeat analyses and repeat readings, respectively. Each analysis is one complete program cycle. If 3 *ANAL is chosen as the number of repeat analyses, the entire program is cycled through three times. If two readings of each element are required, then 2 *RDG would be entered into the program. With those selections, each element in the program would be read twice and then the cycle would be repeated three times.

1. To enter the number of Repeat Analyses for the program, type the appropriate number followed by *ANAL <RETURN>. For example, if two analyses are desired, type 2 *ANAL <RETURN>. Consequently, the complete analysis for this program is repeated three times.

2. Now, to select the number of readings for each element, type the desired number and *RDG <RETURN>. For example, if three readings are required for each element, type 3 *RDG <RETURN>.

Element Selection Option

1. Library of Elements. A library of useful analytical lines for 78 elements is available in memory. To access the possible line choices for these elements, type ELEMENT and the element symbol, then press <RETURN>. For example, if Copper is the element of interest, type ELEMENT CU <RETURN>.

2. Wavelength Selection. To select one of the wavelengths from the table, press the number of the desired line and type USE. For example, if the first Cu line is the one of choice, type 1 USE <RETURN>.

3. Alternate Wavelength Selection. To select a wavelength other than one of those displayed, type 1 USE <RETURN>. Then, type desired wavelength followed by NM. For instance, if 273.53 nm is desired, type 273.53 nm <RETURN>. As a result, the wavelength selected in step 2 is replaced by 273.53 nm.

Integration Time Option

To enter the desired Integration Time, type the desired integration time (from 0.1 seconds to 25.5 seconds, inclusive) followed by SEC. For example, if 2.0 sec is the Integration Time to be entered, type 2.0 SEC <RETURN>. The integration time can be increased to aid in achieving low detection limits if the increase in analysis time is justified.

Decimal Placement Option

To select the number of digits to the right of the decimal, use the #D command. To select the number of digits, type the desired number followed by #D. For example, for a format displaying two digits to the right of the decimal, type 2 #D <RETURN>. A high concentration curve (i.e., 1,000 ppm - 100 ppm) will not accept anything higher than 1 #D accuracy.

Generating a Calibration Curve Option

For generation of a Calibration Curve, the Plasma-200 accepts a reagent blank plus up to five standards. The "#" symbol denotes standard number; bottle number and concentration are defined by the operator. To enter the Calibration Standards, type the number of the element in the program followed by TH <RETURN>. For instance, for the first element of the program (assuming the current program has been accessed), type 1 TH <RETURN>. To enter the standard numbers, bottle numbers, and concentrations, perform the following steps. Type standard number, bottle number desired (1-99), and the concentration. For example, type 1 2 50 CONC <RETURN>.

Window Size Option

The peak search window can be varied for each element in the program. The three window sizes available are Narrow (N), Medium (M), and Wide (W). The sizes represent 0.033, 0.066, and 0.1 nm, respectively. The standard window size is Medium. If the Narrow window is to be used, type WS N <RETURN>. The smaller the window, the less chance for interfering peaks.

Trim Routine Option

Each wavelength chosen has a theoretical position stored in memory. The actual position is slightly different due to the mechanics of the monochromator.

To optimize the wavelength position, call up the desired program and line number, aspirate a test solution containing the element of interest and type TRIM <RETURN>. (It is assumed that the desired program and line report have been called up.) Move the cursor to the center of the analyte peak by pressing L for left, R for right, U for up, and D for down. Only the horizontal location must be adjusted; the vertical displacement (U and D) is for operator convenience only. Then press <RETURN>. To perform the TRIM routine for all lines of the program, call up the program, aspirate a solution containing all the elements in the program and type #TRIM <RETURN>.

Torch Height Selection Option

The Torch Height observation is variable and can be optimized for each element in the program; the MM command changes the observation height for the element in the program. The observation heights must be even numbers between 0 and 48 mm. To select a Torch Height of 18 mm, type 18 MM <RETURN>.. To determine the best viewing height, call up the line (n TH), aspirate a standard and type in TPROFILE <RETURN>. Then aspirate the blank and type ASPIRATE TPLLOT <RETURN>. The best observation height is usually the height at which a maximum intensity difference exists between sample and blank. Each "-" on the screen represents a change of 2 mm in observation height starting with 0 mm.

Restpeak Routine

The RESTPEAK routine can be used for any trimmed element in the program. If the spectral region around a peak needs to be investigated, use this command to direct the microcomputer to the analytical peak, scan a 0.25 nm (second order) region around a peak, and produce an image of the scan on the CRT.

To carry out the RESTPEAK command, first select the desired element of the program by typing the element number followed by TH and pressing <RETURN>. Next aspirate a standard containing the element of interest, then type RESTPEAK <RETURN>. After completing the RESTPEAK routine, a second emission scan at a given spectral line can be superimposed by the following steps.

1. Aspirate the sample.

2. Next, if the operator is changing bottles, it is recommended that the operator type ASPIRATE before PLOT. This causes the pump to operate at the fastpump rate for the time interval set by PDLV, flushing the sample introduction system of the original solution. Type ASPIRATE PLOT <RETURN>.

3. This process may be repeated as often as required to plot as many solutions as desired. Differences in baseline intensity between the standard and the sample indicate that background correction is needed at that wavelength.

Background Correction

Background correction is primarily used to compensate for baseline shift between standards and samples. To correct for background shift, background correction must be performed in a region of the spectrum which is free from matrix emission lines and which accurately represents the background under the analyte peak. For a more thorough discussion of background correction, consult the "Methods Manual for Inductively Coupled Plasma Emission Spectrometry."

To set the background correction position:

1. Select the line of interest by typing the line number and TH and pressing <RETURN>. For example, if the first line in the program is to be corrected, type 1 TH <RETURN>.

2. Aspirate the sample and type BKG-TRIM <RETURN>.

3. Next, select and enter one of the following:

For no background correction, press N.

For correction to the left of the peak, press L.

For correction to the right of the peak, press R.

For correction to the right and left of the peak, press B.

When the cursor appears at its default location (-0.05 nm in second order), position it as desired by pressing the L key for left, R key for right, U key for up, and D key for down. The movement of the cursor in the left and right directions can be varied by using either the fine or coarse step sizes. The system defaults to the coarse step size, but if a finer movement is needed, the cursor can be made to move in much smaller steps by typing F. After positioning the cursor, key <RETURN>.

Interfering Element Correction Routine

The Interfering Element Correction routine should be used only as a last resort. The operator should first attempt to use a line without interferences. If this cannot be done, the Interfering Element Correction routine may be used. For a more complete discussion of the Interfering Element Correction, see the "Methods Manual for Inductively Coupled Plasma Emission Spectrometry."

An interfering element is any element other than the analyte of interest that emits at the chosen wavelength. Perform a restpeak routine of the element of interest followed by a plot of suspected interferences. If any part of a peak shows in the narrowest window, an erroneously large peak will result. It can be corrected in this way.

1. Select a known concentration of Fe (the interfering element). In this example, a standard concentration of 1,000 ppm Fe is used. The 1,000 ppm Fe standard is analyzed at the V wavelength (the analyte). The apparent V concentration due to the presence of Fe is 10 ppm.
2. Then, compute a scale factor. The scale factor can be from 0.0001 to 3.0000.

$$\text{Scale Factor} = \frac{\text{Apparent Analyte Concentration}}{\text{Actual Interferent Concentration}} = \frac{10}{1000} = 0.0100$$

In this example, 0.0100 is used as the scale factor.

3. Add Fe to the program before V is added to the program. Follow standard procedures and select an Fe line. Enter the Fe line as any other line is entered in the program. The interfering element line must be in the program before the line being corrected; i.e., it must have a low line number. If the order of the interferent and analyte are reversed, the IMPROPERLY NESTED error message will appear on the screen.
4. Now select the analyte line requiring correction by typing the desired line number followed by TH. For example, if line 2 is desired, type 2 TH <RETURN>.
5. Enter the line number of the interfering element under one of the two I/E headings. For instance, if line 1 is the interfering element, type 1 I/E <RETURN>. Line 1 is now entered as the interfering element line for line 2.
6. To cancel a selected line, type 0 I/E <RETURN>.
7. Next, enter the scale factor under the corresponding S/F heading. As an example, type 0.01 S/F <RETURN>. A scale factor of 0.01 is now entered and the program will now correct for that interfering element.

The problem with using the interfering element correction is that the interference must either be fairly well resolved or be a near direct overlap. Direct overlaps are difficult to detect.

Side Line Indexing Method

A standard feature of Revision 0 software is a program called SLIM (Side Line Indexing Method). This routine allows the operator to select a reference emission line as the "searched" peak near a difficult to detect analyte peak. The reference peak is first located by the conventional monochromator search routine and then the refractor plate moves directly to the analyte peak location. The reference peak can be an integral part of the matrix, or spiked into the sample, high standard, and blank by the operator. The SLIM routine is very useful for determinations of samples where the analyte peak is obscured by a partial spectral overlap, wing broadening, or when the

analyte peak is too small to determine under normal conditions. The reference peak has to be within 0.2 nm of the analyte peak. To use the SLIM routine, aspirate a solution containing analyte only and trim. Aspirate a solution containing analyte and spiked with suspected interfering reference peak and type SLIM <RETURN>. Trim the reference peak. Enter side for analyte peak (L or R). Trim the analyte peak. Enter background correction, if necessary.

The high standard, the blank, and all samples must contain some Al. The Al concentration does not have to be constant. Calibration is carried out in the standard manner.

If the use of the SLIM routine is no longer required for a particular line in a program, typing -SLIM <RETURN> will remove the routine for that particular line. If the position of the reference peak has to be changed, typing TRIM <RETURN> will allow the readjustment of the reference peak position. If the analyte peak position needs to be reset, typing SLIM <RETURN> will allow the operator to change the position of the analyte peak only.

Calibration

After all options are optimized, calibrate the elements. Emission line intensities obtained during calibration and the corresponding concentration values are fit to a quadratic equation by a least squares method. During analysis, concentration values are calculated from intensities using this equation. This method is used to correct for any nonlinearity of the calibration curve.

To perform calibration, type CALIBRATE <RETURN>. The question "NO. OF READINGS TO STANDARDIZE?" appears on the CRT screen. Enter the number of readings (the number of readings must be between 1 and 10) by pressing the desired number and <RETURN>. For example, if three readings are to be used to standardize, type 3 <RETURN>. In response, the system displays the standard to be aspirated. Aspirate the standards as requested by the Plasma-200, then press <READ>.

Program Storage Routine

After a program is written, it can be stored on tape or disk. The information stored consists of each user program in memory with element information, the trimmed line positions for each element, and the latest calibration data. The user programs can either be stored by themselves or with the system program. To store system and user program, enter <ENABLE> <TAPE>, followed by 4 <RETURN>. To modify programs, refer to instrument manual.

Recalibration

After a calibration curve is entered, a slope recalibration may be performed using the reagent blank and the highest standard. This operation may be performed with either keyboard and is recommended each time a program is re-entered from the cassette reader.

To perform recalibration on the full keyboard, type in the EDIT mode RECALIBRATE <RETURN>. The question "NO. OF READINGS TO STANDARDIZE?" then appears on the CRT. Enter the desired number of readings (between 1 and 10)

by pressing the desired number of readings and <RETURN>. For example, if two readings are to be taken, press 2 <RETURN>. Next, aspirate the requested bottle number (the highest standard) and press <READ>. Finally, aspirate the blank as requested by the Plasma-200 and press the <READ> key.

REAGENTS

The only reagents used are pure grade HNO_3 to digest (see digestion procedure) and make up the standard curves. Triton X-100 can be used if there is a problem matching sample and standard viscosity. It can also be used to aspirate through the spray chamber to keep the surface smooth and clean for top aspiration efficiency.

ANALYSIS

Recalibrate analyte and interfering elements of analyte following procedure. Return to operator mode <PROG>, aspirate sample, and type in <SAM#>, nnnnn (number has to be less than 67000), and <READ>.

Analysis Errors

OV FLOW Signal intensity exceeds dynamic range of the instrument
 (i.e., 25000 cts/25 ms)

OVRANGE Sample is greater than 125 percent of high standard

If either of these messages occurs, analyte can either be run diluted or on a less sensitive wavelength. Refer to manual or any table of atomic emissions.

INTERFERENCE

The main interference in plasma spectroscopy is spectral. An analyte may or may not be influenced by any interfering element. Using a narrow window helps to eliminate interference, but if the concentration of the interfering element is high, it will inevitably interfere with the analyte. The best procedure is to choose an alternate wavelength. If no alternate wavelength is suitable, then interfering element correction must be used (see procedure).

Baseline shift or a continuous spectrum interference must be background corrected (see procedure). Some elements may be corrected using the SLIM routine (see procedure). It is a good idea to inspect the spectral region of each analyte on a representative matrix. The best way of handling interferences must then be decided.

STANDARD PREPARATION

Depending upon the sensitivity of the wavelength chosen for the analyte, a blank and four standards are made up usually spreading the range 0, X, 2.5X, 5X, 10X. If several analytes do not interfere with each other, they may be mixed together in a cocktail. The matrix must match closely the samples; usually one to two percent HNO_3 .

SAMPLE PREPARATION

See digestion procedure.

QA/QC

A blank, standard, and duplicate are digested with every set of samples. The instrument, when warmed up properly, is very stable. A check standard (usually close in value to sample concentration) is read in the middle and end of the run. If you suspect drift or carryover, run the check standard more often. If the check standard is not satisfactory (within ± 80 percent of true and/or mean value), a recalibration must be performed. If there is a lot of carryover, it is a good practice to aspirate blank water first so as to get a stable and accurate recalibration. The digested blank, standard, and duplicate are recorded and calculated into mean and standard deviation.

CALCULATION

Waters: $\text{Response (ug/ml)} \times \text{concentration (ml/ml)} \times \text{dilution (ml/ml)}$

Soils or solids: $\text{Response (ug/ml)} \times \frac{\text{final volume (ml)}}{\text{weight of sample (g)}} \times \text{dilution (ml/ml)}$

DETECTION LIMIT

The detection limit for most metals is listed in the manual. Experimentally, they can be determined by calculating a value that is ± 2 times the mean of a series of blank responses. Obviously, interferences (even if corrected), the use of a less sensitive line, or dilution will all raise the detection limit accordingly.

SAFETY

The drain line must be attached and full of water. The pump must be hooked up and aspirating solution while the torch is on. Do not attempt to work on instrument while voltages are on.

STANDARD OPERATING PROCEDURE CHEMICAL OXYGEN DEMAND

APPLICATION

This method applied to both wastewater and soils/sludges. The chemical oxygen demand (COD) is used as a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. For samples from a specific source, COD can be related empirically to biochemical oxygen demand (BOD), organic carbon, or organic matter. The test is useful for monitoring and control after correlation has been established. The dichromate reflux method is preferred over procedures using other oxidants because of superior oxidizing ability, applicability to a wide variety of samples, and ease of manipulation. Oxidation of most organic compounds is 95 to 100% of the theoretical value. Pyridine and related compounds resist oxidation and volatile organic compounds are oxidized only to the extent that they remain in contact with the oxidant. Ammonia, present either in the waste or liberated from nitrogen-containing organic matter, is not oxidized in the absence of significant concentration of free chloride ions.

Most types of organic matter are oxidized by a boiling mixture of chromic and sulfuric acids. A sample is refluxed in strongly acid solution with a known excess of potassium dichromate ($K_2Cr_2O_7$). After digestion, the remaining unreduced $K_2Cr_2O_7$ is titrated with ferrous ammonium sulfate to determine the amount of $K_2Cr_2O_7$ consumed and the oxidizable organic matter is calculated in terms of oxygen equivalent. Keep ratios of reagent weights, volumes and strengths constant when sample volumes other than 50 ml are used. The standard 2-hour reflux time may be reduced if it has been shown that a shorter period yields the same results.

PROCEDURE

1. Treatment of Water Samples with COD of >50 mg Oxygen Per Liter

Place 50 ml sample (for samples with COD >900 mg O_2 /liter, use smaller sample portion diluted to 50 ml) in a 500 ml refluxing flask. Add 1 g $HgSO_4$, several glass beads, and very slowly add 5.0 ml sulfuric acid reagent, with mixing to dissolve $HgSO_4$. Cool while mixing to avoid possible loss of volatile materials. Add 10 ml of 0.2500 $K_2Cr_2O_7$ solution and mix. Attach flask to condenser and turn on cooling water. Add remaining sulfuric acid reagent (25 ml) through open end of condenser. Continue swirling and mixing while adding the sulfuric acid reagent. CAUTION: Mix reflux mixture thoroughly before applying heat to prevent local heating of flask bottom and a possible blowout of flask contents. Reflux for 2 hours.

Cool and wash down condenser with distilled water. Disconnect reflux condenser and dilute mixture to about twice its volume with distilled water. Cool to room temperature and titrate excess $K_2Cr_2O_7$ with FAS, using 0.10 to 0.15 ml (2 to 3 drops) ferroin indicator. Although the quantity of ferroin indicator is not critical, use the same volume for all titrations. Take as the end point of the titration the first sharp color change from blue-green to reddish brown. The blue-green may reappear. In the same manner, reflux and titrate a blank containing the reagents and a volume of distilled water equal to that of sample.

2. Alternate Procedure for Low-COD Samples

Follow Procedure 1 with two exceptions: 1) use 10 ml of 0.025N $K_2Cr_2O_7$, and 2) titrate with 0.025M FAS. (CAUTION: Exercise extreme care with this procedure because even a trace of organic matter on the glassware or from the atmosphere may cause gross errors.) If a further increase in sensitivity is required, concentrate a larger volume of sample before digesting under reflux as follows: add all reagents to a sample larger than 50 ml and reduce total volume to 150 ml by boiling in the refluxing flask open to the atmosphere without the condenser attached. Compute amount of $HgSO_4$ to be added (before concentration) on the basis of a weight ratio of 10:1, $HgSO_4:Cl^-$, using the amount of Cl^- present in the original volume of sample. Carry a blank reagent through the same procedure. This technique has the advantage of concentrating the same without significant losses of easily digested volatile materials. Hard-to-digest volatile materials such as volatile acids are lost, but an improvement is gained over ordinary evaporative concentration methods.

3. Determination of Standard Solution

Evaluate the technique and quality of reagents by conducting the test on a standard potassium hydrogen phthalate solution.

4. Alternate Procedure for Soil Samples

Follow Procedure 1 with the following exceptions: 1) accurately weigh 0.2 to 0.5 g that will consume one-half of the $K_2Cr_2O_7$, and 2) add 20 ml of distilled water to each flask. Add $HgSO_4$ and H_2SO_4 according to Procedure 1.

All glassware can be cleaned with 1:1 HNO_3 with plenty of distilled water rinse to remove traces of HNO_3 .

A blank and standard must be run with each set.

INSTRUMENT OPERATION

The reflex apparatus consist of 500 or 250 ml Erlenmeyer flasks with ground-glass 24/40 neck and 300 mm jacket Liebig, West or equivalent condenser with 24/40 ground-glass joint, and a hot plate having sufficient power to produce at least 1.4 W/cm² of heating surface, or equivalent. Turn hot plate on ahead of time. Turn on condenser cooling water.

REAGENTS

1. Standard Potassium Dichromate Solution, 0.0417M/0.2500N

Dissolve 12.259 g $K_2Cr_2O_7$, primary standard grade, previously dried at 103°C for 2 hours in distilled water and dilute to 1000 ml.

2. Sulfuric Acid Reagent

Add Ag_2SO_4 , reagent or technical grade, crystals or powder, to concentrated H_2SO_4 at the rate of 5.5 g Ag_2SO_4 /kg H_2SO_4 . Let stand 1 to 2 days to dissolve Ag_2SO_4 .

3. Ferroun Indicator Solution

Dissolve 1.485 g 1,10-phenanthroline monohydrate and 695 mg $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in distilled water and dilute to 100 ml. This indicator solution may be purchased already prepared.

4. Standard FAS Titrant, Approximately 0.25M

Dissolve 98 g $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ in distilled water. Add 20 ml concentrated H_2SO_4 , cool, and dilute to 100 ml. Standardize this solution daily against standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution as follows. Dilute 10 ml standard $\text{K}_2\text{Cr}_2\text{O}_7$ to about 100 ml; add 30 ml concentrated H_2SO_4 and cool while samples are on hot plate. Titrate with FAS titrant using 0.10 to 0.15 ml (2 to 3 drops) ferroin indicator before titrating samples.

$$\text{Normality of FAS Solution} = \frac{\text{Volume } \text{K}_2\text{Cr}_2\text{O}_7 \text{ solution titrated, ml}}{\text{Volume FAS used in titration, ml}} \times 0.25N \text{ } \text{K}_2\text{Cr}_2\text{O}_7$$

5. Mercuric Sulfate

HgSO_4 , crystals or powder.

6. Sulfamic Acid

Required only if the interference of nitritesis to be eliminated

7. Potassium Hydrogen Phthalate (KHP) Standard

Lightly crush and then dry potassium hydrogen phthalate ($\text{HOOCCH}_2\text{CH}_2\text{COOK}$) to constant weight at 120°C . Dissolve 425 mg in distilled water and dilute to 1000 ml. KHP has a theoretical COD of 1.176 mg O_2/mg and this solution has a theoretical COD of 500 $\mu\text{g } \text{O}_2/\text{ml}$. This solution is stable when refrigerated for up to 3 months in the absence of visible biological growth.

INTERFERENCE

Volatile straight-chain aliphatic compounds are not oxidized to any appreciable extent. This failure occurs partly because volatile organics are present in the vapor space and do not come in contact with the oxidizing liquid. Straight-chain aliphatic compounds are oxidized more effectively when Ag_2SO_4 is added as a catalyst. However, Ag_2SO_4 reacts with chloride, bromide and iodide to produce precipitates that are oxidized only partially. The difficulties caused by the presence of the halides can be overcome largely, though not completely, by complexing with HgSO_4 before the refluxing procedure. Although 1 g HgSO_4 is specified by 50 ml sample, a lesser amount may be used where sample chloride concentration is known to be less than 2000 mg/l, as long as a 10:1 ratio of $\text{HgSO}_4:\text{Cl}^-$ is maintained. Do not use the test for samples containing more than 2000 mg Cl^-/l . Techniques designed to measure COD in saline water are available.

NO_2^- exerts a COD of 1.1 mg $\text{O}_2/\text{mg } \text{NO}_2^-/\text{N}$. Because concentrations of NO_2^- in waters rarely exceed 1 or 2 mg $\text{NO}_2^-/\text{N/l}$, the interference is considered insignificant and usually is ignored. To eliminate a significant interference due to NO_2^- , add 10 mg sulfamic acid for each mg NO_2^-/N present in the sample volume used; add the same amount of sulfamic acid to the reflux vessel containing the distilled water blank.

Reduced inorganic species such as ferrous iron, sulfide, manganous manganese, etc., are oxidized quantitatively under the test conditions. For samples containing significant levels of these species, stoichiometric oxidation can be assumed from known initial concentration of the the interfering species and corrections can be made to the COD value obtained.

STANDARD PREPARATION

A 500 mg/l KHP standard is analyzed on the high method COD (see Procedures 1 and 3, and Reagent 7.

A 50 mg/l KHP standard is analyzed on the low method COD (Procedure 2). Dilute 5 ml of Reagent 7 to 50 ml in flask and analyze.

SAMPLE PREPARATION

Samples must be preserved by acidification to pH ≤ 2 with concentrated H_2SO_4 .

QUALITY ASSURANCE/QUALITY CONTROL

A blank and standard are analyzed with every ten samples. The standard must be within 2 standard deviations of the mean value. If the standard fails to be within the limits, the run must be reanalyzed.

CALCULATION

$$COD \text{ as mg } O_2/l = \frac{(A - B) \times N \times 8000}{ml \text{ sample}}$$

where A = ml FAS used for blank
B = ml FAS used for sample

DETECTION LIMIT

High Level Water: 50 mg/l anything; <50 mg/l should be rerun on low level
High Level Soil: 2500 ug/g
Low Level Water: 5 mg/l

SAFETY

1. Slowly add concentrated H_2SO_4 reagent with plenty of swirling.
2. Make sure that H_2SO_4 -water mixture is well mixed and cooling water is sufficient to condense organic vapors.
3. Used titration mixture is classified as hazardous waste and must be diluted and stored in labeled plastic containers to be treated later.

DATA SHEET

Sample copy of data sheet attached.



COD DETERMINATIONS

SECRET NO. _____ OF _____
PAGE NO. _____

$$\text{COD mg/l} = \frac{(\text{Diff})(n)(8,000)}{\text{ml Sample}}$$

Sample		Normality of Titrant	
1	2	3	4
5	6	7	8
9	10	11	12
13	14	15	16
17	18	19	20
21	22	23	24
25	26	27	28
29	30	31	32
33	34	35	36
37	38	39	40
41	42	43	44
45	46	47	48
49	50	51	52
53	54	55	56
57	58	59	60
61	62	63	64
65	66	67	68
69	70	71	72
73	74	75	76
77	78	79	80
81	82	83	84
85	86	87	88
89	90	91	92
93	94	95	96
97	98	99	100

STANDARD OPERATING PROCEDURES CYANIDE

APPLICATION

This method is applicable to the determination of cyanide in drinking, surface and saline waters, domestic and industrial wastes.

The colorimetric procedure is used for concentrations below 1 mg/l of cyanide and is sensitive to about 0.02 mg/l. The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined.

In the colorimetric measurement the cyanide is converted to cyanogen chloride, CNCl , by reaction with chloramine-T at a pH less than eight without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read at 578 nm for pyridine-barbituric acid. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

Cyanide is defined as cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

PROCEDURE

Samples Without Sulfide

1. Place 500 ml of sample, or an aliquot diluted to 500 ml in the 1 liter boiling flask. (Add a few boiling beads to flask prior to adding sample. Make sure water is running through cold finger at a reasonable rate.) Add 50 ml of sodium hydroxide to the absorbing tube and dilute if necessary with distilled water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber and trap in the train.
2. Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube. (CAUTION: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to readjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.)
3. Slowly add 25 ml concentrated sulfuric acid through the air inlet tube. Rinse the tube with distilled water and allow the airflow to mix the flask contents for 3 minutes. Pour 20 ml of magnesium chloride into the air inlet and wash down with a stream of water.
4. Heat the solution to boiling, taking care to prevent the solution from backing up into and overflowing from the air inlet tube. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

5. Drain the solution from the absorber into a 250 ml volumetric flask and bring up to volume with distilled water washings from the absorber tube.

Samples With Sulfide

1. Place 500 ml of sample, or an aliquot diluted to 500 ml in the 1 liter boiling flask. Pipet 50 ml of sodium hydroxide to the absorbing tube. Add 25 ml of lead acetate to the sulfide scrubber. Connect the boiling flask, condenser, scrubber and absorber in the train. The flow meter is connected to the outlet tube of the cyanide absorber.

2. Start a stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately 1.5 liters per minute enters the boiling flask through the air inlet tube. The bubble rate may not remain constant while heat is being applied to the flask. It may be necessary to readjust the air rate occasionally.

3. If samples contain NO_3 and/or NO_2 add 2 g of sulfamic acid solution after the air rate is set through the air inlet tube. Mix for three minutes prior to addition of H_2SO_4 .

4. Slowly add 50 ml 18N sulfuric acid through the air inlet tube. Rinse the tube with distilled water and allow the airflow to mix the flask contents for three minutes. Pour the tube with distilled water and allow the airflow to mix the flask contents for three minutes. Pour 20 ml of magnesium chloride into the air inlet and wash down with a stream of water.

5. Heat the solution to boiling. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

Analysis can be stopped at this point, coloring can be done next day (time not factor, solution is very stable at this point).

Coloring Procedure

Withdraw 25 ml or less of the solution from the flask and transfer to 50 ml volumetric flask. If less than 25 ml is taken, dilute to 25 ml with 0.25 N sodium hydroxide solution. Add 15.0 ml of sodium phosphate solution and mix.

1. Pyridine - Barbituric Acid Method: Add 2 ml of chloramine T and mix. After one to two minutes, add 5 ml of pyridine-barbituric acid solution and mix. Dilute to mark with distilled water and mix again. Allow eight minutes for color development then read absorbance at 578 nm in a 1 cm cell within 15 minutes.

QA/QC

Prepare a series of standards by pipeting suitable volumes of standard solution into 250 ml volumetric flasks. To each standard add 50 ml of 1.25N sodium hydroxide and dilute to 250 ml with distilled water. Prepare as follows:

ml of Standard Solution
(1.0 = 5 ug CN)

Concentrate mgCN
per 250 ml

0
1.0
2.0
5.0
10.0
15.0
20.0

Blank
5
10
25
50
75
100

INSTRUMENT OPERATION

Apparatus

Reflux distillation apparatus such as shown in Figure 2. The boiling flask should be of 1 liter size with inlet tube and provision for condenser. The gas absorber may be a Fisher-Milligan scrubber. Spectrophotometer suitable for measurements at 578 nm or 620 nm with a 1.0 cm cell or larger.

Reflux distillation apparatus for sulfide removal. The sulfide scrubber may be a Wheaton Bubber #709682 with 29 42 joints, size 100 ml. The air inlet tube should not be fritted. The cyanide absorption vessel should be the same as the sulfide scrubber. The air inlet tube should be fritted. Flow meter, such as Lab Crest with stainless steel float (Fisher 11-164-50).

REAGENTS

1. Sodium hydroxide solution, 1.25N: Dissolve 50 g of NaOH in distilled water, and dilute to 1 liter with distilled water.
2. Cadmium carbonate: powdered.
3. Ascorbic acid: crystals.
4. Dilute sodium hydroxide solution, 0.25N: Dilute 200 ml of sodium hydroxide solution to 1,000 ml with distilled water.
5. Sulfuric acid: concentrated.
6. Sodium dihydrogenphosphate, 1 M: Dissolve 138 g of $\text{NaH}_2\text{PO}_4\cdot\text{H}_2\text{O}$ in 1 liter of distilled water. Refrigerate this solution.
7. Stock cyanide solution: Dissolve 2.51 g of KCN and 2 g KOH in 1 liter of distilled water. Standardize with 0.0192 n AgNO_3 . Dilute to appropriate concentration so that 1 ml = 1 mg CN.
8. Standard cyanide solution, intermediate: Dilute 50.0 ml of stock (1 ml = 1 mg CN) to 1000 ml with distilled water (1 ml = 50.0 ug).
9. Standard cyanide solution: Prepare fresh daily by diluting 100.0 ml of intermediate cyanide solution to 1,000 ml with distilled water and store in a glass stoppered bottle [1 ml = 5.0 ug CN(5.0 mg/l)].

10. Standard silver nitrate solution, 0.0192 N: Prepare by crushing approximately 5 g AgNO_3 crystals and drying to constant weight at 40°C . Weigh out 3.2647 g of dried AgNO_3 , dissolve in distilled water, and dilute to 1,000 ml (1 ml = 1 mg CN).

11. Chloramine T solution: Dissolve 1.0 g of white, water soluble Chloramine T in 100 ml of distilled water and refrigerate until ready to use. Prepare fresh weekly.

12. Color Reagent: Pyridine-Barbituric Acid Reagent: Place 15 g of barbituric acid in a 250 ml volumetric flask and add just enough distilled water to wash the sides of the flask and wet the barbituric acid. Add 75 ml of pyridine and mix. Add 15 ml of HCl , mix, and cool to room temperature. Dilute to 250 ml with distilled water and mix. This reagent is stable for approximately six months if stored in a cool, dark place.

13. Magnesium chloride solution: Weigh 510 g of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ into a 1,000 ml flask, dissolve and dilute to 1 liter with distilled water.

14. Sulfamic acid.

ANALYSIS

See Procedure

INTERFERENCE

1. Interferences are eliminated or reduced by using the distillation.
2. Sulfides adversely affect the colorimetric procedure. Samples that contain hydrogen sulfide, metal sulfides or other compounds that may produce hydrogen sulfide during the distillation should be distilled by the optional procedure.
3. High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation nitrate and nitrite will form nitrous acid which will react with some organic compounds to form oximes. These compounds formed will decompose under test conditions to generate HCN . The interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid.

STANDARD PREPARATION

Standard Curve for Samples Without Sulfide

1. Prepare a series of standards by pipeting suitable volumes of standard solution into 250 ml volumetric flasks. To each standard add 50 ml of 1.25 N sodium hydroxide and dilute to 250 ml with distilled water. Prepare as follows:

ml of Working Standard Solution
(1 ml = 10 ug CN)

Concentrate ug CN
per 250 ml

0	Blank
1.0	10
2.0	20
5.0	50
10.0	100
15.0	150
20.0	200

2. It is not imperative that all standards be distilled in the same manner as the samples. It is recommended that at least two standards (a high and low) be distilled and compared to similar values on the curve to insure that the distillation technique is reliable. If distilled standards do not agree within $\pm 10\%$ of the undistilled standards the analyst should find the cause of the apparent error before proceeding.

3. Prepare a standard curve by plotting absorbance of standard vs. cyanide concentrations.

4. To check the efficiency of the sample distillation, add an increment of cyanide from either the intermediate standard or the working standard to 500 ml of sample to insure a level of 20 ug/l. Proceed with the analysis.

Standard Curve for Samples With Sulfide

1. It is imperative that all standards be distilled in the same manner as the samples. Standards distilled by this method will give a linear curve, but as the concentration increases, the recovery decreases. It is recommended that at least three standards be distilled.

2. Prepare a standard curve by plotting absorbance of standard vs. cyanide concentrations.

SAMPLE PREPARATION

The sample should be collected in plastic or glass bottles of 1 liter or larger size. All bottles must be thoroughly cleansed and thoroughly rinsed to remove soluble material from containers.

Oxidizing agents such as chlorine decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper (KI-starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.

Samples must be preserved with 2 ml of 10 N sodium hydroxide per liter of sample (pH >12) at the time of collection. Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored in a refrigerator or in an ice chest filled with water and ice to maintain temperature at 4°C.

1. Alternatively, if the sample contains more than 1 mg of CN transfer the distillate, or a suitable aliquot diluted to 250 ml, to a 500 ml Erlenmeyer flask. Add 10-12 drops of the benzalrhodanine indicator.

2. Titrate with standard silver nitrate to the first change in color from yellow to brownish-pink. Titrate a distilled water blank using the same amount of sodium hydroxide and indicator as in the sample.

3. The analyst should familiarize himself with the end point of the titration and the amount of indicator to be used before actually titrating the samples. A 5 to 10 ml microburet may be conveniently used to obtain a more precise titration.

CALCULATION

1. If the colorimetric procedure is used, calculate the cyanide, in ug/l, in the original sample as follows:

$$\text{CN, ug/l} = \frac{A \times 1,000}{B} \times \frac{25}{C}$$

where:

A = ug CN read from standard curve

B = ml of original sample for distillation

C = ml taken for colorimetric analysis

2. Using the titrimetric procedure, calculate concentration of CN as follows:

$$\text{CN, mg/l} = \frac{(A - B)1,000}{\text{ml original sample}} \times \frac{250}{\text{ml of aliquot titrated}}$$

where:

A = volume of AgNO_3 for titration of sample

B = volume of AgNO_3 for titration of blank

DETECTION LIMIT

5 ug/l

SAFETY

Use caution while refluxing samples and adding acid. Follow all safety regulations.

EPA METHOD 335.2

PROJECT _____
ANALYST _____

1

[illegible]

NOTES AND CALCULATIONS: TITRIMETRIC METHOD: CN $\text{mg/L} = \frac{(A-B) \times 1000}{C} \times \frac{D}{E}$ COLORIMETRIC METHOD: CN $\text{mg/L} = \frac{F \times 1000}{C} \times \frac{25}{E}$

$$m/\lambda = \frac{f = 1000}{c} = \frac{25}{\lambda}$$

STANDARD OPERATING PROCEDURE
TOTAL AND DISSOLVED ORGANIC CARBON

APPLICATION

This method is applicable to drinking and surface waters, and sewage and industrial waste effluents.

PROCEDURE

For Total Organic Carbon (TOC) acidified samples are analyzed as received. Dissolved Organic Carbon (DOC) samples are analyzed using the same procedure except the samples are filtered through a 0.45 micron filter prior to analysis.

An acidified persulfate reagent is continuously pumped from an external reservoir to the injection port and then into the bottom of UV reactor. The reactor is a constant volume design; the excess liquid is pumped to waste from a drain port.

The reactor liquid is continuously sparged and this sparge/carrier gas flows out on the top of the reactor to a non-dispersive infra-red detector (NDIR). When a sample is injected, it is carried into the reactor by the reagent flow. The oxidation of organics occurs rapidly, and the resultant carbon dioxide is sparged from the liquid and carried to the NDIR. The NDIR produces an electrical output (peak) which is integrated and sealed by the number processor and then displayed and printed. The concentration of the sample injected is displayed and printed directly in the concentration units for which the instrument has been calibrated.

The instrument is designed for three levels of carbon analysis. High level samples are run with the detector module set on 40 ul, and 40 ul samples are injected into the reaction module. Low level carbon samples are run with the detector module set on 1 ml, and 1 ml samples are injected into the reaction module. We do not generally use the mid-range carbon setting of 200 ul.

INSTRUMENT OPERATION

1. Fill the reactor with persulfate reagent by temporarily lifting the lamp and cap assembly up and pouring the reagent in. Fill to about 1 inch below the recycle port.
2. Add clean water to the "U" tube trap until the level is just at the bottom of the bulb section on the right.
3. Turn on oxygen gas tank; regulator reads 30 psi.
4. Turn on switches:
 - a. Main power on EM-2 microprocessor
 - b. Power, pump and lamp on UV-persulfate reaction module

5. Let instrument warm up for at least one-half hour to ensure complete sparging of the persulfate solution and stabilization of all electronics and the UV lamp.

6. Calibrate the instrument, if necessary, following the instructions on pages 3-6 to 3-9 in the instrument manual. The instrument, once calibrated, retains its calibration (even when shut-off) until it is erased manually. Check the calibration by running a standard after the instrument is warmed up.

7. The samples and standards are purged for a few minutes with oxygen to drive off any inorganic carbon prior to analysis.

8. Standards, blanks and samples are injected into the injection port with 40 μ l or 1 ml aliquots using glass syringes. Rinse the syringe 10-12 times with water and 10-12 times with standard or sample to be analyzed before filling the syringe to inject into the instrument. Repeat for each sample.

Instrument Maintenance

1. Reaction vessel is cleaned with chromic acid cleaning solution once a week.

2. The tin scrubber which traps any chlorine generated is changed every 3-4 weeks.

3. Change pump tubing as necessary, usually every 2-3 weeks.

Instrument Troubleshooting

The instrument will print error messages when not operating properly. See Table I, page 3-10, in the instrument manual.

REAGENTS

Potassium Persulfate Solution: Dissolve 20 grams of reagent grade potassium persulfate and 1 ml of reagent grade concentrated nitric acid in 1 liter of the purest water available.

INTERFERENCE

High Chloride: See Appendix B of instrument manual.

STANDARD PREPARATION

2,000 ppm Carbon Standard: Weigh 425 mg of potassium hydrogen phthalate ($C_8H_5O_4K$), KHP, dried to a constant weight. Transfer the KHP quantitatively with the purest water available to a 100 ml flask, dissolve, add 0.1 ml of concentrated nitric acid (HNO_3) and dilute to volume with water. Store this solution in dark glass under refrigeration. Replace monthly.

10 ppm Carbon Standard: 1.00 ml of the 2000 ppm standard is diluted to 200 ml with pure water in a volumetric flask.

SAMPLE PREPARATION

Sulfuric acid is used to preserve the samples to a pH of less than 2. If the samples are unpreserved, add acid prior to analysis. (DOC samples are generally received unpreserved.) Acidify DOC samples after filtering through a 0.45 micron filter and prior to analysis.

QA/QC

A blank of the purest water available, a standard, and a duplicate are run per every ten samples analyzed.

CALIBRATION

When calibrated properly (as described previously), the instrument display and printer will read directly in concentration units.

DETECTION LIMIT

0.1 mg/l

SAFETY

Safety glasses, gloves and lab coat are worn in the laboratory. Special care is taken when working with syringes to avoid injury.

DATA SHEET

Sample copy of Data Sheet attached.

anion exchange column. Plutonium is eluted with sulfurous acid, the eluate evaporated to dryness and dissolved for electrodeposition on stainless steel discs. Quantitation and chemical recovery are determined from alpha spectroscopy counting.

1.2 Soil

1.2.1 Gamma Spectroscopy

One-hundred grams of dried and pulverized sample is placed in a Marinelli beaker and counted for eight hours, on a Ge(Li) Detector, which is coupled to a 2048 computer based, multi-channel analyzer (Northern Scientific). The resulting spectrum is fed into a computer and specific nuclides, if present, are identified and quantized in terms of energy and net count rate with the aid of the computer.

1.2.2 Gross Alpha and Gross Beta

A suitable aliquot of prepared sample is muffled, dissolved in acid, nitrated, evaporated and transferred to a tared two-inch stainless steel planchet. The planchet is counted for Gross Alpha and Gross Beta activity as in Method 3.1.1.

1.2.3 Radium (total)

Preparation of the soil for analysis requires that it first be dried, muffled to remove carbon, and pulverized. Aliquots for sample and sample spike with added radium-226 tracer are leached in dilute nitric acid, filtered, and the leachate evaporated to dryness. The residue is dissolved in nitric acid, insoluble residue filtered off and sulfuric acid added as an anion source for the precipitate. The samples are heated and barium carrier added, precipitating the barium/radium as a sulfate. Samples and corresponding spikes are alpha counted. Chemical recovery is determined from radium-226 tracer spike recovery.

1.2.4 Strontium-89/90

Strontium-90

A ten-gram aliquot is spiked with Strontium-85 tracer and dissolved in HNO_3 - HF mixture. After dissolution of the soil, the solution is evaporated to dryness several times with HCl. Oxalic acid is added and the solution adjusted to a pH of 5.5 - 6.0 with NH_4OH . The solution is allowed to stand for several hours and is then stirred and filtered. The filtrate is discarded.

The precipitate and paper are transferred to a dish and dried overnight at 110°C . The oxalate precipitate is ignited in a muffle furnace at 400 - 500°C for two hours. The temperature is raised slowly to about 700°C and heating continued for two hours. The precipitate is cooled and transferred to an appropriate size beaker and dissolved in 1:1 HNO_3 . About six drops of H_2O_2 (30%) is added to facilitate dissolution, and then the solution is gently heated to boiling. The solution is cooled to room temperature.

The solution is transferred to a suitable size beaker and evaporated to dryness.

The residue is dissolved in 25 ml of 0.08 N HCl and transferred to a 125 ml separatory funnel using two 5 ml rinses of 0.08 N HCl.

After the 14-day ingrowth period is established, the Yttrium-90 is extracted with 5% di-2-ethylhexyl phosphoric acid (D_2EHPA) in toluene, back extracted into an aqueous phase, precipitated as the oxalate and counted in a low background internal gas flow proportional counter (Beckman Low Beta II) to determine the Strontium-90 content of the sample. The Strontium-85 tracer is counted on the Gamma Spectrometer to determine the percent recovery of the method.

Strontium-89

A ten-gram aliquot of sample containing standardized stable Strontium carrier is dried at 110°C for twenty-four hours, ashed in a muffle furnace with the addition of concentrated fuming nitric acid. The Strontium is precipitated with concentrated fuming nitric acid, redissolved in water, made basic with dilute ammonium hydroxide and precipitated as the oxalate. The dried oxalate precipitate is counted in a low-background proportional counter, which has 60% Strontium, Yttrium-90 efficiency. The Strontium-89 activity is determined by subtracting the previously measured Strontium-90 activity and its corresponding Yttrium-90 ingrowth from the measured Gross Strontium activity.

1.2.5 Isotopic Uranium

CEP uses the following analytical method for analyzing Uranium-234, 235, 238 in soil: A ten-gram aliquot is spiked with Uranium-232 tracer. Total dissolution of the soil is performed using a hydrofluoric-nitric acid mixture, nitrated and evaporated to dryness. The residue is dissolved in concentrated nitric acid and again taken to dryness and redissolved in dilute acid. The sample is purified with an ion exchange resin column. The Uranium is electroplated and the discs counted on a solid state alpha spectrometer and the chemical recovery is determined from the Uranium-232 tracer peak.

1.2.6 Uranium, total

The soil sample is dried, muffled to remove carbon and pulverized. An aliquot is taken, leached in nitric acid, filtered, and brought to a known volume with deionized water. Analysis is then performed according to Method 1.1.8.

1.2.7 Cesium-137

See method 1.2.1, Gamma Spectrometry in Soil

1.2.8 Isotopic Plutonium

The soil sample is totally dissolved using a 40% solution of HF and Pu-242 tracer is added before dissolution. The sample is fumed with HF and converted to sulfate and then brought up with HNO₃. The Plutonium is separated using an ion exchange resin. The Plutonium is eluted off the column and electroplated on a stainless steel disc. The disc is counted on a solid state alpha spectrometer and chemical recovery is determined from the tracer peak.

1.3 Vegetation

1.3.1 Gamma Spectroscopy

The wet sample is placed in a Marinelli beaker and counted with a multichannel analyzer equipped with an intrinsic detector which is coupled to a 4096 channel, computer-based, multichannel analyzer (Northern Scientific TN4500). The resulting spectrum is analyzed by computer, and specific nuclides, if present, identified and quantified on a dry weight basis.

1.3.2 Gross Alpha and Gross Beta

A suitable aliquot of the sample is dried at 110°C for twenty-four hour, ashed in a muffle furnace, dissolved in dilute acid and transferred to a tared planchet. The Alpha and Beta radioactivity are determined using a low-background gas flow, proportional counter (Beckman Wide Beta II). The activity is corrected for counter efficiency and self absorption.

1.3.3 Strontium-89/90

The sample is prepared according to method 1.3.2 above and then analyzed according to method 1.2.4 Strontium-89 and 90 in Soil.

STANDARD OPERATING PROCEDURE BULK CHEMISTRY

APPLICATION

This method is applicable for soils and sediments. This method is suitable for extraction of metal complexes. The assumption is made that the oxide form is preferred by most metals in soil.

PROCEDURE

The nitric acid digestion is used for all bulk metals with the exception of silica, aluminum, calcium and iron. Calcium is extracted by DI because of its high solubility rate. Silica, aluminum and iron are extracted by the following procedure.

Transfer 10 ml of NaOH solution measured with a plastic graduated cylinder to each of two nickle crucibles, and evaporate the solution to dryness on a hot plate. (A small amount of spattering can be ignored.) Except for addition of no soil, carry one of the crucibles through the entire procedure described below to obtain the reference blank solution. To the other crucible, add a sample of about 0.05 g of 100-mesh soil that has been weighed to the nearest 0.1 mg. Cover crucible, and heat to dull redness for about 5 minutes. Grasp the crucible with nickel or platinum-tipped tongs, and rotate it so that the melt is spread over the sides of the lower half of the crucible. Allow the melt to cool, add approximately 50 ml of water, cover the crucible, and let it stand overnight. Transfer the contents of the crucible to a 600 ml beaker containing about 400 ml of water and 20 ml of 6N HCl. (Do not allow the nickel crucible to come in contact with the acid solution.) Scrub the crucible well with a rubber policeman, and wash any remaining residue or solution into the beaker. Transfer the solution to a 1-liter volumetric flask, add distilled water to make 1-liter, and mix the contents well. (This is referred to as the sample solution.)

INSTRUMENT OPERATION

See ICP Methods. Silica was determined spectrophotometrically as follows.

Color Development

Withdraw 10 ml of the sample solution with a pipette, and transfer the aliquot to a 100-ml volumetric flask. Treat a 10 ml aliquot of the reference blank solution in the same manner as the sample solution. Add 1 ml of the ammonium molybdate reagent solution, swirling the contents of the flask during addition. Mix the solutions well, and allow the flask to stand for 10 minutes. Now add 4 ml of the tartaric acid solution while swirling contents of the flask, and mix the solution well. Add 1 ml of reducing solution while swirling the contents of the flask, add distilled water to make 100 ml, mix the contents well, and allow the flask to stand at least 30 minutes. Determine the percent transmission at 650 mμ, setting the reference blank solution at 100. Calculate the concentration of silicon from the standard curve.

Standard Curve

Pipette 0, 1, 2, 3, 4, 5, 6, 7 and 8 ml of the standard silicon solution into 100 ml volumetric flasks. Add sufficient reference blank solution to each flask so that the volume of silicon standard plus reference blank totals 10 ml. Develop the color as described above, starting with "add 1 ml of the ammonium molybdate reagent solution...." Plot the percent transmission versus silicon concentration on semilogarithmic graph paper.

Results were calculated as follows:

$$\text{mg Si/liter (from standard curve)} \times \text{liter/g sample} \times 10 \text{ (d.f.)} = \text{mg Si/g sample (as per procedure)}$$

See ICP Methods for Al and Fe determinations.

REAGENTS

Reagents are specific for each extraction procedure.

ANALYSIS

See ICAP Methods.

QA/QC

Normal QA/QC procedures to be followed.

DETECTION LIMIT

See ICAP Methods.

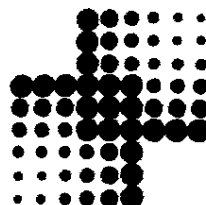
SAFETY

Use normal Laboratory safety procedures.

APPENDIX XI
ANALYTICAL RESULTS
"LABORATORY AND FIELD"

NOTE

Laboratory Radiometric Data
from CED are of very poor quality.



L-AREA OIL & GAS BASIN (RADEOLOGICAL LABORATORY RESULTS)

THIS DATA IS IN 8-BIT ASCII FORMAT. THE FORMAT OF THE DATA IS AS FOLLOWS:

BYTE	ITEM	FORTTRAN FORMAT CODE
------	------	----------------------

RECORD #1 (ONCE FOR EACH LBC #)

1-4	LBC #	4A1
5-6	NUMBER OF INTERVALS	I2

RECORD #2 (REPEATED FOR EACH INTERVAL)

1-5	INCHES	5A1
6-10	SAMPLE #	5A1
11-12	# OF ELEMENTS IN SAMPLE	I2

RECORD #3 (REPEATED N TIMES FOR EACH ELEMENT FOUND IN INTERVAL)

1-5	NUCLIDE	5A1
10-17	Pci/g	8A1
18-24	+/- ERROR	7A1

RECORD #4 (CONTAINS 2 80-BYTE RECORDS FOR EACH INTERVAL)

1-11	Pu238	11A1
12-23	Pu239/240	12A1
24-33	Sr89	10A1
34-44	Sr90	11A1

LBC6 6

0-5 76953 9

CS137 1130.0 3.0

CS134 2.72 0.3

CD60 2070.0 4.0

LA140 7330.0 1590.0

ZR95 29.7 1.7

NB95 6.05 1.46

NA22 39.6 1.0

EU152 65.4 2.0

CD109 388.0 11.0

0.09 0.04 0.08 0.03 <0.06 12.3 1.0

<0.05 <0.05 <0.06 11.3 1.5

3-6 76954 9

ZR95 47.4 3.0

CS134 4.61 1.45

NB95 7.91 2.56

CD109 419.0 13.0

NA22 62.8 1.9

EU152 162.0 29.0

LA140 8050.0 2790.0

CS137 1130.0 4.0

CD60 7160.0 8.0

<0.05 <0.05 <0.06 51.5 9.0

<0.05 <0.05 <0.06 69.1 2.5

6-9 7685510

EU152 472.0 3.0

ZN65 270.0 8.0

CS137 560.0 3.0

NA22 81.3 1.9

CD60 10200.0 68.0

CD109 412.0 12.0

CS134 5.81 0.6

LA140 17600.0 3090.0

ZR95 63.1 3.8

NB95 11.2 1.0

6.74 0.48 <0.05 <0.06 1426.0 17.0

7-1276956 9

CD109 639.0 18.0

ZR95 103.0 5.0

NA22 117.0 2.0

CS137 366.0 3.0

CS134 7.42 0.8

CD60 27300.0 12.0

NB95 14.0 1.0

LA140 16300.0 3370.0

EU152 1010.0 5.0

0.15 0.12 0.04 0.13 <0.06 12.3 4.0

12-1575557 5

CD109 174.0 10.0

EU152 124.0 5.0

CS137 146.0 3.0

CD60 165.0 3.0

LA140	7050.0	2660.0					
ZR95	15.9	2.3					
NA22	20.4	1.0					
CS134	3.48	0.4					
NB95	7.02	0.8					
1.22 0.1	7.09 0.3	<0.06	1.0	0.4			
1.07 0.1	7.05 0.27	<0.06	1.4	0.6			
15-187595810							
NA22	14.4	1.0					
CS134	3.47	0.4					
NB95	6.79	0.8					
ZR95	21.9	1.0					
CB109	82.9	6.7					
CO57	2.41	0.93					
EU152	114.0	2.0					
CS137	108.0	2.0					
CO60	3870.0	4.0					
ZN65	79.2	5.0					
3.52 0.13	29.79 3.0	12.3 0.6	2.3	1.0			
2.66 0.12	22.69 2.3	12.3 0.8	2.9	1.4			

T4421. EDIT2. DATA (LAB1)

L-AREA OIL & CHEMICAL BASIN (LAB RESULTS METALS, PETROLEUM, HYDROCARBONS AND OIL & GREASE)

THIS DATA IS IN 8-BIT ASCII FORMAT. THE FORMAT ON THE DISK IS AS FOLLOWS:

BYTE	ITEM	FORTRAN FORMAT CODE
------	------	---------------------

RECORD #1 (ONCE FOR EACH LBO #)

1-4	LBO #	4A1
5-6	NUMBER OF INTERVALS	I2

RECORD #2 (REPEATED 3X FOR EACH INTERVAL) CONTAINS 1 60-BYTE RECORDS EACH

1-5	INCHES	5A1
6-10	SAMPLE #	5A1
11-16	Sb	5A1
17-21	As	5A1
22-26	Be	4A1
28-29	Cd	4A1
30-35	Cr	6A1
36-40	Pb	5A1
41-45	Hg	5A1
46-51	Ni	5A1
52-56	Se	5A1
57-61	Ag	5A1
62-67	U	6A1
68-73	Zn	6A1
74-80	TCC	7A1
1-2	COD	5A1
3-10	CN	5A1
11-15	PETROL HYDROCARBONS	5A1
16-20	GREASE & OIL	5A1

— Envirodyne Lab ID

L8C5 4

0-3 76953

0-3 76959

0-3

3-6 76950

4.3 49800

3-6 76960

3-6

6-9 76951 45.92 8.5 2.0 9.7 1782.0 21.0 0.2 90.9 0.490.62 175.0 1105.0 100000

8500 0.25

6-9 76961

6-9

9-147695235.99 7.1 2.0 7.78039.0202.0 0.85 373.0 0.680.37 177.0 334.0 32000

10900 0.25

9-1476962

9-14

L8C619

0-3 76953

0-3 76963

0-3

3-6 76954

3-6 76964

3-6

6-9 76955

4.8 48000

6-9 76965

6-9

9-1276956

4.8 5090

9-1276966

9-12

12-1576957 5.5 4.7 2.0 4.42365.0 66.3 0.2 115.0 0.640.035 61.7 41.5 6137

10800 0.25 4.8 1630

12-1576967 4.04 5.2 2.0 4.72469.0 67.3 0.2 125.0 0.690.073 60.1 43.7 6605

10800 0.25 4.8

12-15

15-1876958 3.34 4.7 <2.0 3.92011.0 54.1<0.2 77.5 0.82011.0 1.0 1.0 1.0
10200<0.25<4.8 1650
15-1876968 4.42 4.3 <2.0 3.71966.0 58.6 0.74 98.2 0.52011.0 1.0 1.0 1.0
10000<0.25<4.8
15-18
18-2176110 1.4792.72 <2.0<2.0 474.0 36.8<0.2 9.2<0.5 0.081 1.0 1.0 1.0
3400<0.25<4.42 270
18-2176123 1.0162.56 <2.0<2.0 463.0 33.2 0.2 8.0<0.5 0.081 1.0 1.0 1.0
900<0.25<4.42 125
18-21 0.8502.76 <2.0<2.0 462.0 24.1<0.2 5.8<0.5 0.073 1.0 1.0 1.0
900 0.70<4.42
21-2476111 1.0092.30 <2.0<2.0 117.0 31.3<0.2 < 2.0<0.5 0.079 1.0 1.0 1.0
< 600<0.25<4.42 188
21-2476124 1.0562.29 <2.0<2.0 110.0 28.3<0.2 < 2.0<0.5 0.085 1.0 1.0 1.0
500<0.25<4.42
21-24 0.88 2.23 <2.0<2.0 108.0 27.4<0.2 < 2.0<0.5 0.071 1.0 1.0 1.0
< 400<0.25<4.42
24-2776112 2.7491.35 <2.0<2.0 320.0 33.2<0.2 14.5<0.5 0.133 7.29 8.8 1270
1200<0.25<4.42 200
24-2776125 2.7381.42 <2.0<2.0 315.0 29.3<0.2 16.2<0.5 0.128 4.3 1142
2900<0.25<4.42 354
24-27 16.24 1.59 <2.0<2.0 311.0 69.6<0.2 18.8<0.5 0.127 10.7 1137
2300<0.25<4.42
27-3076113 4.0220.903<2.0<2.0 259.0 35.2<0.2 13.5<0.5 0.132 5.72 5.5 1124
1500<0.25<4.42 255
27-3076126 6.82 1.04 <2.0<2.0 254.0 42.8<0.2 14.8<0.5 0.181 4.6 1114
2400<0.25<4.42
27-30 2.821 .973<2.0<2.0 255.0 27.2<0.2 13.6<0.5 0.123 4.1 1131
1600<0.25<4.42
30-3376114 0.71 2.99 <2.0<2.0 33.0 20.1<0.2 4.8<0.5 0.037 2.0 1.1 713
< 600<0.25<4.42 35
30-3376127 1.1782.92 <2.0<2.0 33.3 26.6<0.2 5.0<0.5 0.031 1.0 1.0 717
< 400<0.25<4.42
30-33 0.5513.46 <2.0<2.0 32.9 19.1<0.2 4.7<0.5 0.025 1.0 1.0 711
< 400<0.25<4.42
33-3676115 0.6021.64 <2.0<2.0 30.3 20.7<0.2 6.6<0.5 0.025 3.19 1.0 1030
< 600<0.25<4.42 84
33-3676128 0.6091.72 <2.0<2.0 28.2 20.0<0.2 6.5<0.5 0.034 1.0 1.0 815
< 400<0.25<4.42
33-36 0.4091.78 <2.0<2.0 27.3 13.7<0.2 5.8<0.5 0.035 1.0 1.0 771
500<0.25<4.42
36-3976116 0.5231.23 <2.0<2.0 13.1 15.8<0.2 < 2.0<0.5 0.031 2.0 1.0 771
< 600<0.25<4.42 154
36-3976129 0.6361.22 <2.0<2.0 14.1 19.9<0.2 6.4<0.5 0.029 2.0 1.0 566
< 400<0.25<4.8
36-39 0.7541.3 <2.0<2.0 13.9 19.7<0.2 1.0<0.5 0.021 4.3 1.0 116
< 400<0.25<4.8
39-4376117 1.0121.10 <2.0<2.0 6.9 20.7<0.2 3.4<0.5 0.041 2.0 1.0 1.0
< 600<0.25<4.42 82
39-4376130 1.6101.36 <2.0<2.0 6.9 11.8<0.2 2.0<0.5 0.029 1.0 1.0 1.0
700<0.25<4.8
39-43 1.8531.18 <2.0<2.0 6.9 20.7<0.2 2.0<0.5 0.073 1.0 1.0 1.0
< 600<0.25<4.8

48-5176118 0.6350.913<2.0<2.0 4.2 11.9<0.2 2.4<0.5 0.031<2.0 < 1.0 47-
< 600<0.25<4.42
48-5176131

48-51

54-5776119 0.5340.799<2.0<2.0 4.4 17.4<0.2 2.0<0.5 0.021<2.0 < 1.0 471
< 600<0.25<4.42 212
54-5776132

54-57

60-6376120 0.3170.654<2.0<2.0 < 4.0 12.3<0.2 < 2.0<0.5 0.031<2.0 < 1.0 445
< 600<0.25<4.42
60-6376130

60-63

66-6976121 1.7200.709<2.0<2.0 4.3 20.0<0.2 4.6<0.5 0.044<2.0 < 1.0 626
< 600<0.25<4.42 825
66-6976134

66-69

72-7576122 1.0580.985<2.0<2.0 4.9 24.1<0.2 3.9<0.5 0.024<2.0 < 1.0 727
< 500 <4.42
72-7576135

72-75

T4421, EDIT 2, DATA (FIELD)

1-AREA OIL & CHEMICAL BASIN (FIELD) L-8 RESULTS

THIS DATA IS IN 8-BIT ASCII FORMAT. THE FORMAT IN THE FILE IS AS FOLLOWS:

BYTE ITEM FORMULA FORMAT CODE

RECORD #1 (ONCE FOR EACH LEC #)

1-4	LEC #	41
5-6	NUMBER OF INTERVALS	11

RECORD #2 (REPEATED FOR EACH INTERVAL)

1-10	INCHES	10A1
11-18	Ca-60	6A1
19-20	Ca-60 +/-	2A1
21-23	Sb-125	3A1
24-26	Sb-125 +/-	3A1
27-33	Cs-137	7A1
34-36	Cs-137 +/-	3A1
37-43	Eu-152	7A1
44-47	Eu-152 +/-	4A1
48-52	Eu-154	3A1
53-58	Eu-154 +/-	3A1
56-59	Eu-155	4A1
60-62	Eu-155 +/-	3A1
63-68	GROSS ALPHA	3A1
66-69	GROSS ALPHA +/-	4A1
70-75	GROSS BETA	6A1
76-77	GROSS BETA +/-	2A1
78-80	MOISTURE	3A1

LBC-1 2
 9-15 13400.0 1 690.0 2 430.0 3 240.0 12 52 8 8500 1 80
 3-38 19.0 5 20.0 4 1 100 57 6 17

LBC-224
 0-3 2720.0 2 1180.0 3 100.0 8 7 22 980 2 92
 3-6 2510.0 2 1040.0 2 104.0 20 366 27 39 10 2020 2 84
 6-9 5140.0 1 77 22 1430.0 2 250.0 4 195.0 17 130 13 17 15 3680 1 82
 9-11 7160.0 1 1710.0 2 130.0 18 230.0 13 100.14 47 9 3830 1 80
 11-17 4500.0 1 440.0 2 180.0 11 130.0 16 92 8 73 3 7920 1 58
 17-20 120.0 2 60.0 3 1 100 170 5 17
 20-23 86.0 3 63.0 3 1 100 140 5 17
 23-26 160.0 2 66.0 3 1 100 130 5 19
 26-29 140.0 2 62.0 3 1 100 100 6 18
 29-32 47.0 4 56.0 3 1 100 39 5 17
 32-35 38.0 4 44.0 3 1 100 38 6 17
 35-38 34.0 4 42.0 4 1 100 85 6 17
 38-41 37.0 4 32.0 4 1 100 49 7 16
 41-44 29.0 5 29.0 4 1 100 63 7 16
 44-47 35.0 4 27.0 4 1 100 73 6 13
 47-50 32.0 4 24.0 4 1 100 70 6 16
 50-53 39.0 4 18.0 5 1 100 72 6 15
 53-56 47.0 3 18.0 5 1 100 100 6 19
 56-59 34.0 4 15.0 5 1 100 75 6 16
 59-62 31.0 4 14.0 6 1 100 56 7 13
 62-65 15.0 6 9.1 6 1 100 51 7 13
 65-68 14.0 7 13.0 5 1 100 70 6 16
 68-71 21.0 5 9.6 7 1 100 49 7 13
 71-74 25.0 5 12.0 7 1 100 61 5 9

LBC-314
 0-3 950.0 2 570.0 2 9 20 910 2 82
 3-6 960.0 2 700.0 2 68.0 8 8 21 1260 1 81
 6-9 3260.0 1 620.0 2 140.0 5 40 10 2060 1 87
 9-11 8180.0 1105 14 450.0 3 420.0 5 310.0 2 182 6 40 10 5280 1 59
 11-15 7360.0 1 210.0 4 250.0 8 170.0 3 140 5 11 18 4960 1 44
 15-18 360.0 2 91.0 3 9.1 13 3 31 500 3 26
 18-21 63.0 3 67.0 3 1 100 110 5 17
 21-24 160.0 2 64.0 3 1 100 160 5 18
 24-27 120.0 2 46.0 3 1 100 140 5 18
 27-30 120.0 2 33.0 4 1 100 120 5 17
 30-33 110.0 2 28.0 4 1 100 120 5 16
 33-36 100.0 2 21.0 5 1 100 100 6 16
 36-39 70.0 3 20.0 5 1 100 93 6 16
 39-44 50.0 3 18.0 5 1 100 87 6 16

LBC-412
 0-3 2200.0 2 54 70 3160.0 1 230.0 4 210.0 8 33 11 2720 2 84
 3-6 7100.0 1 99 24 1560.0 2 300.0 4 175.0 13 73 17 27 12 7290 1 30
 6-9 21300.0 1135 18 780.0 3 1110.0 2 430.0 8 57 8 16100 1 78
 S(BL) 9-15 7600.0 1 240.0 4 390.0 8 110.0 14 17 14 10400 1 80
 CS(CL) 9-15 390.0 2 77.0 3 11.0 11 3 30 880 3 26
 15-18 88.0 3 54.0 3 1 100 260 4 20
 18-21 43.0 4 45.0 3 1 45 200 5 19
 21-24 44.0 3 26.0 4 1 100 130 5 18
 24-27 59.0 3 16.0 5 1 100 140 5 17
 27-30 33.0 4 14.0 5 1 100 120 5 16
 30-33 32.0 4 6.7 9 1 100 90 6 16

		40.0	4	42.0	25		2	40	170	1	10
		22.0	5	46.0	13		1	50	140	1	10
27		8.8	8				1	50	110	5	10
27-30		4.9	9				1	100	73	6	10
30-33		2.8	10				1	100	50	7	10
33-36	5.410	5.7	30				1	100	75	6	10
36-39	7.210						1	45	52	7	10
39-42	62.0 3	4.4	12				1	100	225	2	10

LBC-813

0-3	2100.0 2 41 30	870.0	2	115.0	16	110.0	13	66	20	20	10	1120	2	84
3-8	18500.0 1160 20	700.0	3	1140.0	2	730.0	10	240	18	20	5	3400	1	79
8-11	1360.0 1	105.0	3	73.0	9	53.0	15	98	25	3	20	400	2	23
BLU 11-14	229.0 2	11.0	3	7.0	27	7.5	3					130	4	27
RED 11-14	372.0 2	68.0	3	15.0	20	16.0	30	40	30	1	40	200	4	20
14-17	20.0 3	55.0	3							1	100	90	6	20
RED 17-20	32.0 4	25.0	4							1	100	120	6	17
YEL 17-20	60.0 4	28.0	4	2.0	55					1	100	160	5	16
BLU 17-20	48.0 4	39.0	3							1	100	60	8	19
20-23	10.0 6	17.0	7							1	100	100	6	16
23-26	4.9 8	6.1	7							1	100	40	7	15
26-29	3.812	3.1	10							1	100	40	8	15
29-32	5.410	2.8	10							1	100	4010	14	

LBC-9 9

0-3	1840.0 2 50 25	790.0	3	130.0	18	100.0	10	29	10	7	15	940	2	87
3-6	5650.0 1100 20	1040.0	2	180.0	10	240.0	3	90	7	27	7	3000	1	79
6-12	1650.0 1	95.0	4	62.0	10	41.0	4	18	9	13	17	2080	1	56
12-15	92.0 2	58.0	3			1.1	30					130	5	21
15-18	73.0 3	43.0	3	1.5	33	1.1	35					160	4	17
18-21	58.0 3	21.0	4									120	5	16
21-24	52.0 3	18.0	5									50	7	18
24-27	32.0 4	13.0	6									50	7	15
27-31	38.0 4	9.6	7									50	5	13

T4421. EDIT 2 DATA (LAB2)

L-AREA DTL. CHEMICAL BASIN (LAB RESULTS, EPA TOXICITY, AND BULK CHEMISTRY)

THIS DATA IS IN 8-BIT ASCII FORMAT. THE FORMAT ON THE DISK IS AS FOLLOWS:

BYTE ITEM FORTRAN FORMAT CODE

RECORD #1 (ONCE FOR EACH LBC #)

1-4	LBC #	4A1
5-6	NUMBER OF INTERVALS	12

RECORD #2 (REPEATED 3X FOR EACH INTERVAL) CONTAINS 2 80-BYTE RECORDS EACH

1-5	INCHES	5A1	
6-10	SAMPLE #	5A1	
11-16	As	6A1	-----
17-22	Ba	6A1	
23-28	Cd	6A1	
29-34	Cr	6A1	
35-40	Pb	6A1	EPA TOXICITY
41-45	Hg	5A1	
46-51	Se	6A1	
52-58	Ag	7A1	-----
59-61	SiO2	3A1	
62-64	Al2O3	3A1	
65-68	Fe2O3	4A1	
69-73	MgO	5A1	
74-79	CaO	5A1	
1-3	Na2O	3A1	BULK CHEMISTRY
4-7	K2O	4A1	
8-12	TiO2	5A1	
13-18	P2O5	6A1	
19-25	MnO	7A1	

LBC-5

0-3 76949

0-3 76959

0-3

3-6 76950

3-6 76960

3-6

6-9 76951

5282.60 69.051.4 12460.0

6-9 76961

6-9

9-1476952

3532.15 48.039.3 4070.0

LBC619

0-3 76953

0-3 76963

0-3

3-6 76954

3-6 76964

3-6

6-9 76955<0.002 0.013<0.004<0.004<0.20<0.002<0.45

6-9 76965

6-9

9-12 0.013<0.004<0.20<0.002<0.45

9-1276

9-12

12-1576957<0.002 0.023<0.002 0.005<0.004<0.20<0.002<0.45 69526371.8167.0 78.0

1321.13 68.012.2 346.0

12-1576967

68929778.6172.0 91.0

1191.19 45.012.2 399.0

12-15

15-1876959<0.002 0.022<0.002 0.004<0.004<0.20<0.002<0.45 69727768.0129.0 84.0

1191.03 35.013.1 271.0

15-1876968

69926462.3162.0 89.0

741.03 45.012.2 276.0
15-18

18-2178110<0.002<0.004<0.002<0.004<0.004 0.40<0.002 0.010884913155.1 71.0 1.5
1531.02 8.3 3.73 45.0
18-2178123 63725755.8198.0 1.5
18-21 572.752 6.7 3.96 45.0
18-21 640.684 7.3 3.73 49.0
21-2478111<0.002<0.004<0.002<0.004<0.004 0.50<0.002<0.00851832953.2 44.0 1.5
1231.28 1.2 3.14 9.3
21-2478124 5833.737.5 37.0 1.5
1431.24 1.7 2.32 9.6
21-24 45128549.2 55.0 1.4
1261.20 2.8 2.27 9.0
24-2778112<0.002 0.010<0.002 0.004<0.004 0.21<0.002 0.000772528823.3155.0 1.5
1151.32< 1.7 2.86 65.0
24-2778125 74426729.8133.0 1.5
1151.31< 1.7 3.01 63.0
24-27 75726230.9274.0 1.5
1651.75 16.0 2.91 59.0
27-3078113<0.002<0.004<0.002 0.004<0.004 0.24<0.002 0.000870229424.8159.0 1.5
1201.61< 1.7 2.81 53.0
27-3078126 72125426.6174.0 1.8
1601.86 1.8 2.74 49.0
27-30 75723048.6101.0 1.5
1141.58< 1.7 2.53 52.0
30-3378114<0.002 0.009<0.002<0.004<0.004 0.56<0.002 0.000975522551.2 33.0 1.5
911.58 7.0 1.83 11.0
30-3378127 67124030.0 70.0 1.5
1071.58 2.8 1.73 8.8
30-33 60922047.5 35.0 1.5
841.51 2.2 1.67 8.4
33-3678115<0.002 0.011<0.002<0.004 0.014 0.32<0.002 0.001083021450.9 26.0 1.5
1191.66 2.3 0.979 7.6
33-3678128 77224449.8 28.0 1.5
871.51 6.8 0.889 7.0
33-36 74723248.9 14.0 1.5
1101.54 2.3 0.847 7.5
36-3978116<0.002 0.016<0.002<0.004<0.004<0.20<0.002<0.000576622538.3 40.0 1.5
841.79< 1.7 0.399 5.2
36-3978129 81520130.6 48.0 1.5
1251.86 4.5 0.399 4.9
36-39 78722329.2 60.0 1.5
971.93 7.0 0.353 5.6
39-4378117<0.002<0.004<0.002<0.004<0.004<0.21<0.002<0.000569522929.4 63.0 1.5
1662.46 3.0 0.422 4.3
39-4378130 83020428.5 65.0 1.4
2152.43 5.0 0.399 4.5
39-43 83422636.0 60.0 1.5
1702.81 1.7 0.399 4.4
48-5178118<0.002 0.034<0.002<0.004<0.004 0.71<0.002<0.000580817528.9 40.0 1.5
1611.99 1.7 0.274 5.4
48-5178131

48-51

54-51 0.014<0.004 0.73<0.002<0.000582315129.7 25.0 1.1

2212.

54-51

54-57

60-6376120<0.002 0.020<0.002 0.007<0.004 0.28<0.002 0.000892510618.0 3.8< 1.5

1281.56 1.8 0.188 6.5

60-6376133

60-63

66-6976121<0.002 0.023<0.002<0.004<0.004 0.30<0.002<0.000585013319.8 46.0< 1.5

1701.84 3.3 0.165 3.6

66-6976134

66-69

72-7576122<0.002 0.042<0.002<0.004<0.004 1.38<0.002<0.000580417823.7 45.04 1.5

1982.02 4.0 0.284 8.3

72-7576135

72-75