Summary
Analytical Data Review:
The Analysis of Fish Tissues and Sediment Samples by LSU

The state trustees and Marathon Pipe Line Company entered into a verbal agreement to subject all data collected in response to the Marathon Pipe Line gasoline release of May 24, 1996 to data review/validation as per *USEPA National Functional Guidelines for Organic Data Review* (February 1994). The intent of this agreement was to ensure that all site-related decisions would be based upon technically valid and legally defensible environmental data of known and documented quality. Data validation activities determine the level of data quality provided by the laboratory. Validation procedures involve auditing the raw laboratory data *i.e.*, data obtained directly from the instrument printouts rather than the laboratory's report, in order to verify the final laboratory-reported analytical results.

The analytical procedure employed by LSU is not an EPA-approved reference method and the gas chromatographic conditions used were considered proprietary and were not made available for review. Therefore, data provided by LSU could not be fully validated in accordance with USEPA functional guidelines. However, to the extent possible, data were reviewed and qualified per USEPA guidelines and other publications referenced by the LSU investigators.

Significant issues regarding the LSU methodology and the findings of the data review process include the following:

- **Selective Ion Monitoring**: The selective ion monitoring (SIM) mass spectrometric method employed by LSU, while very sensitive, is unable to provide full scan mass spectra for confirmation of the presence of compounds of interest. In addition, interference contributions from other nontarget compounds cannot be demonstrated. Consequently, unequivocal compound of interest identification is not possible *i.e.*, positive chemical identification via SIM cannot withstand a data challenge.

- **Blank Contamination**: The methodology utilized by LSU was apparently intended for trace level, part per trillion (ng/kg), residue analyses. However, project samples were demonstrated to contain compound concentrations above the parts per billion

(µg/kg) range. This discrepancy between sample concentrations and method sensitivity, seems to have resulted in contamination of the laboratory's sample processing equipment and/or other aspects of the analytical system, as is evidenced in widespread blank contamination. Consequently, positive results may be false (*e.g.*, 74.4% false positives in the fish tissue) or inaccurately biased on the "high" side.

- **Matrix Spike Recovery**: Matrix spike recovery results demonstrated that the laboratory reported 3 times the actual amount of naphthalene, 42 times the actual amount of 2-methylnaphthalene, and 21 times the actual amount of 1-methylnaphthalene which was spiked into the sample. The magnitude of these spike recovery results suggests that the laboratory may have a severe problem in some aspect of sample handling.

- **Quantitation Practices**: In some instances, the laboratory used averages of external standard and internal standard results, as well as, averages of the averages. Likewise, fish tissue results provided by LSU on answer sheets represented averages of multiple analyses. However, review of the data revealed that not all analytical results available for a given fish tissue sample were used in the calculation of that sample's average value. Rather, the laboratory selected particular replicate results for use in the determination of each sample mean. No selection criteria were presented by the laboratory and the number of replicates used to calculate the reported average concentrations varied. This arbitrary selection of analytical results, as well as, the inconsistent number of replicate analyses used, introduces a bias into the determination of these sample means. Therefore, sample averages thus calculated may be biased high or low, depending on the analytical results selected.

The LSU trace level method for semivolatile analyses was developed for research use rather than as a method to be employed for regulatory purposes. As such, it is suitable for academic situations in which the limits of analyte amounts and interference amounts are known before the method is utilized.

The LSU data, although not directly comparable due to lack of full validation and differences in methodology, were supportive of validated edible fish tissue and mussel tissue results described in an earlier report, *Interim Report, Natural Resource and Habitat Injury and Recovery Assessment* (C-K Associates and Terra Consulting Group, Inc., December 1996). The LSU qualified results revealed the detection of 2-methylnaphthalene in one choupique (0.272 mg/kg) and one catfish (0.091 mg/kg) collected at the two sampling stations closest to the release site *i.e.*, Stations 3 and 4,

respectively. In the earlier report, 3 bluegill and 2 largemouth bass also collected at Stations 3 and 4, had detectable concentrations of 2-methylnaphthalene ranging from 0.014J to 2.60 mg/kg. As in the earlier reported data, qualified LSU data revealed no detections of naphthalene or 2-methylnaphthalene in any mussel tissue.

FISH TISSUE SAMPLES

TERRA
CONSULTING GROUP

Analytical Data Review: The Analysis of Fish Tissue by LSU for Semivolatile Compounds in Response to the Marathon Pipe Line Company Gasoline Release

Introduction

In response to the Marathon Pipe Line Company gasoline release from a pipe line right-of-way adjacent to U.S. Highway 61 (Airline Highway) in St. James Parish, fish tissue and organs were collected and subsequently analyzed by Dr. Jay Means of Louisiana State University, School of Veterinary Medicine, Department of Physiology, Pharmacology, and Toxicology (LSU). The extraction procedure employed by LSU utilized a tissue sample preparation technique known as Matrix Solid Phase Dispersion (MSPD) which was modified by his laboratory for the analysis of semivolatile compounds. As with the sediment sample analyses performed by LSU and discussed in an earlier report, *Interim Report, Natural Resource and Habitat Injury and Recovery Assessment* (Appendix H; C-K Associates and Terra Consulting Group, Inc., December 1996), the analytical finish (instrumental analysis) employed was not an United States Environmental Protection Agency (USEPA) approved reference method. As such, validation of the LSU data in accordance with the USEPA guidance document, *USEPA National Functional Guidelines for Organic Data Review* (USEPA February 1994)was precluded. However, to the extent possible and where appropriate, data were evaluated and qualified.

LSU Method of Analysis

The tissue extraction method utilized by LSU for semivolatile analyses was modified to be very sensitive and to possess a higher degree of specificity than routine gas chromatographic methods. Modifications applied to the sample preparation involved "scaling up" the amount of tissue that was extracted so that the detection limit of the method would be enhanced. Although this technique is a common and acceptable practice, it requires that the laboratory employ additional precautions addressing the

method contamination which may occur during the extraction process when these type of modifications are used.

The detection limits were further enhanced by programming the mass spectrometer to monitor only for specific ions (selective ion monitoring or SIM), rather than all ions, in a chemical's mass spectrum, in order to quantify the analytes of interest. Although this technique increases sensitivity, SIM has a lower degree of specificity than the routine repetitive scanning gas chromatographic / mass spectrometric (GC/MS) methods specified by the USEPA. Specifically, the LSU method does not have the ability to provide full scan mass spectra for the compounds of interest. Consequently, confirmation of the presence or identity of the analytes detected by the LSU method by comparing the mass spectra of the sample to spectra of known reference materials was not possible.

Data Review

The data review performed by Terra involved receiving, categorizing and reviewing a 4,012 page data package provided by LSU. The results of this data review are discussed below and an index to this data package, particularly to the instrument data system printouts, can be found in Appendix I, LSU Fish Tissue Data Package Index.

Averaged Results

The fish tissue analytical results entered into the Terra database were obtained from cross-tabulation style answer sheets produced and provided by LSU. The result for each fish tissue sample provided in these LSU data sheets was found to represent an average value derived from multiple analyses. Reporting the average of multiple analyses for organic fractions (e.g., semivolatile analyses) is not a common practice. However, these average values, as calculated and reported by LSU, were recorded and entered into the Terra database as if they were each a single analysis for the respective samples.

Review of the LSU data revealed that not all analytical results available for a given sample were used in the calculation of that sample's average. Rather, LSU selected particular replicate analyses for use in the determination of each sample mean. However, no selection criteria were presented by LSU and the number of replicates used to calculate reported average concentrations was inconsistent. Whereas average results for fish tissue samples were derived from two or three determinations, method blank average results were calculated from up to five analyses. The purposive selection of analytical results, as well as, the inconsistent number of replicate analyses used for the calculation of sample averages, introduces a bias into the determination of these sample means. Therefore, sample averages thus calculated may be biased high or low, depending on the analytical results selected for the calculations.

Sample Identification and Chain of Custody

Analytical samples were obtained from the dissection of four different species of fish. Each species was furthered sub-sampled for three different types of tissues, *i.e.*, edible muscle tissue, gill tissue, and liver tissue. Mussels were also collected for LSU's study. The soft tissue of the mussels were homogenized so that representative sub-samples could be taken and subsequently analyzed for semivolatiles. Table 1 below lists the fish species sampled, the respective tissue types used for semivolatile analysis and the identifications assigned by LSU. The sample identifications assigned by LSU to the mussel samples are listed in Table 2.

The naming convention utilized by LSU incorporated the type of fish, the tissue analyzed, and the location from which the sample was taken (*e.g.*, Site 1, Site 2 , Site 3, Site 4 or Site 5). The individual result values were contained in each column. In order to accommodate the field and data structure of the Terra database, a shorter, unique sample name was derived for each reporting entity. Unique sample names were assigned to each of the sample entities listed above and to the edible catfish tissue and mussels.

Table 1. Fish and Fish Tissues Identified by LSU for Analysis

Species	Species (LSU designation)	Site Designation	Terra Identification		
			Edible Tissue (T)	Liver (LV)	Gill (G)
Lepomis macrochirus	Blue Gill (BG)	Site 1	TBGS1	LVBGS1	GBGS1
Lepomis macrochirus	Blue Gill (BG)	Site 2	TBGS2	LVBGS2	GBGS2
Lepomis macrochirus	Blue Gill (BG)	Site 3	TBGS3	LVBGS3	GBGS3
Lepomis macrochirus	Blue Gill (BG)	Site 4	TBGS4	LVBGS4	GBGS4
Lepomis macrochirus	Blue Gill (BG)	Site 5	TBGS5	LVBGS5	GBGS5
Micropterus salmoides	Large Mouth Bass (L)	Site 1	TLS1	LVLS1	GLS1
Micropterus salmoides	Large Mouth Bass (L)	Site 2	TLS2	LVLS2	GLS2
Micropterus salmoides	Large Mouth Bass (L)	Site 3	TLS3	LVLS3	GLS3
Micropterus salmoides	Large Mouth Bass (L)	Site 4	TLS4	LVLS4	GLS4
Amia calva	Shoepick / Spick (P)*	Site1	TPS1	LVPS1	GPS1
Amia calva	Shoepick / Spick (P)	Site2	TPS2	LVPS2	GPS2
Amia calva	Shoepick / Spick (P)	Site3	TPS3	LVPS3	GPS3
Amia calva	Shoepick / Spick (P)	NA	NA	NA	NA
Amia calva	Shoepick / Spick (P)	Site5	TPS5	LVPS5	GPS5
Ictalurus punctatus	Catfish (CAT)	Site 1	CATS1		
Ictalurus punctatus	Catfish (CAT)	Site 2	CATS2		
Ictalurus punctatus	Catfish (CAT)	Site 3	CATS3		
Ictalurus punctatus	Catfish (CAT)	Site 4	CATS4		
Ictalurus punctatus	Catfish (CAT)	Site 5	CATS5		

*Choupique

Table 2. Mussel Tissues Identified by LSU for Analysis

Species	Species (LSU designation)	Site Designation	Edible Tissue (T)
Not available	Mussel (MU)	Site 1	MUS1
	Mussel (MU)	Site 2	MUS2
	Mussel (MU)	Site 3	MUS3
	Mussel (MU)	Site 4	MUS4
	Mussel (MU)	Site 5	MUS5

The chain of custody documents supplied by the laboratory contain sample identifications for each individual fish taken and the species of fish. The instrumental reports provided by LSU contain abbreviations such as "S1G11" or "S1F5". Presumably, S1G11 is a shortened form of Site 1 Gill 11, meaning fish 11 from Site1 is a "gill" or Bluegill. However, the "G" was also used for "Shoepick" (Choupique) so that the "G" may have actually referred to the gill tissues. Both S1F5 and S1G11 were taken from a table in the LSU data package that consisted entirely of gill organ results. Consequently, a consistent meaning was not readily apparent as no explanations were given in the raw data provided by the laboratory.

Blank Contamination

Review of the data package provided by LSU showed the presence of significant amounts of target analytes in the method blanks. Possible causes of this blank contamination include the use of contaminated sample processing equipment and / or contaminated chemical solvents by the laboratory. The presence of target analytes in the method blanks indicates that contamination of samples occurred within the laboratory setting. Therefore, the source of the contaminants found in the field samples is questionable. Accordingly, the sample results were reviewed and qualified in the manner described for addressing blank contamination in the USEPA Functional Guidelines. The guidelines for qualifying GC/MS blanks are normally used with single determinations per sample and blank, but the same principles may be applied to averaged results.

All values reported in the Terra database were taken from answer sheets containing final results presented by the LSU laboratory. Intermediate calculation spreadsheets provided by LSU were not used for reporting or qualifying the data. Four blank results were provided on the final answer sheets and were identified as being associated with particular sample results *i.e.*, liver, gill, catfish, or mussel. However, no blank results were provided for other edible tissue preparations (Bluegill, Largemouth Bass and

Choupique). Therefore, these edible tissue preparation results were qualified using the average of all four of the provided blank results. Typically, however, sample results with no corresponding blank are considered unusable.

The importance of qualifying the fish sample results for blank contamination is shown by the following statistics based on qualifying the results using the five times (5 x) rule from USEPA Functional Guidelines. The tables from which these were derived are available for review.

- Total Number of Possible Results = 3,773
- Total Positives = 1,876
- Percentage of positives = 49.7%
- Percentage of false positives (flagged UB) = 74.4%

Based on USEPA guidance, seventy-four percent (74%) of all the positive analytical results reported by LSU in fish and mussel tissue are considered to be false positives. The false positives that were reported by LSU appear to be a function of the sample preparation techniques employed by the laboratory and/or the methods use of specific ions rather than full mass spectra.

EDIBLE FISH TISSUE

LSU Fish Data Crosstab

	TBGS1 5/31/96 TRG		TBGS2 5/31/96 TRG		TBGS3 5/31/96 TRG		TBGS4 5/31/96 TRG	
	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	26	26U	12	12U	13	13U	23	23U
2-Methylnaphthalene	40	40U	27	27U	26	26U	43	43U
Naphthalene	67	67U	48	48U	49	49U	91	91U

	TBGS5 5/31/96 TRG		TLS1 5/31/96 TRG		TLS2 5/31/96 TRG		TLS3 5/31/96 TRG	
	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	8	8U	16	16U	17	17U	23	23U
2-Methylnaphthalene	12	12U	30	30U	35	35U	42	42U
Naphthalene	28	28U	49	49U	88	88U	83	83U

	TLS4 5/31/96 TRG		TPS1 5/31/96 TRG		TPS2 5/31/96 TRG		TPS3 5/31/96 TRG	
	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	12	12U	24	24U	43	43U	172	172
2-Methylnaphthalene	19	19U	37	37U	76	76U	272	272
Naphthalene	44	44U	72	72U	194	194U	301	301U

	TPS5 5/31/96 TRG		CATS1 5/31/96 TRG		CATS2 5/31/96 TRG		CATS3 5/31/96 TRG	
	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	57	57U	8	8U	13	13U	18	18U
2-Methylnaphthalene	97	97U	20	20U	30	30U	42	42U
Naphthalene	155	155U	78	78U	94	94U	125	125U

Lab Result = data as received from LSU.
Qualified data= data after validation; positive detects are shaded.
U = Indicates undetected result at the numerical level reported. Values are indistinguishable from method blank levels and therefore results are considered non-detects.
All results are reported in µg/kg = parts per billion (ppb).

EDIBLE FISH TISSUE (Continued)

LSU Fish Data Crosstab

	CATBLANK N/A BLK	CATS4 5/31/96 TRG		CATS5 5/31/96 TRG	
		Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	6	57	57	8	8U
2-Methylnaphthalene	16	91	91	21	21U
Naphthalene	63	82	82U	62	62U

Lab Result = data as received from LSU.
Qualified data= data after validation; positive detects are shaded.
U = Indicates undetected result at the numerical level reported. Values are indistinguishable from method blank levels and therefore results are considered non-detects.
All results are reported in µg/kg = parts per billion (ppb).

MUSSEL TISSUE

LSU Fish Data Crosstab

	MUBLANK N/A BLK	MUS1 5/31/96 TRG		MUS2 5/31/96 TRG		MUS3 5/31/96 TRG		MUS4 5/31/96 TRG	
		Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	20	54	54U	42	42U	41	41U	33	33U
2-Methylnaphthalene	43	117	117U	88	88U	97	97U	75	75U
Naphthalene	102	207	207U	157	157U	126	126U	109	109U

	MUS5 5/31/96 TRG	
	Lab Result	Qualified Data
1-Methylnaphthalene	36	36U
2-Methylnaphthalene	84	84U
Naphthalene	137	137U

Lab Result = data as received from LSU.
Qualified data= data after validation; positive detects are shaded.
U = Indicates undetected result at the numerical level reported. Values are indistinguishable from method blank levels and therefore results are considered non-detects.
All results are reported in µg/kg = parts per billion (ppb).

GILL AND LIVER TISSUE

LSU Fish Data Crosstab

	GBGS1 5/31/96 TRG		GBGS2 5/31/96 TRG		GBGS3 5/31/96 TRG		GBGS4 5/31/96 TRG	
	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	32	32U	123	123U	32	32U	52	52U
2-Methylnaphthalene	58	58U	245	245U	65	65U	77	77U
Naphthalene	97	97U	382	382U	108	108U	66	66U

	GBGS5 5/31/96 TRG		GILLBLANK N/A BLK	GLS1 5/31/96 TRG		GLS2 5/31/96 TRG		GLS3 5/31/96 TRG	
	Lab Result	Qualified Data		Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	25	25U	42	53	53U	67	67U	23	23U
2-Methylnaphthalene	52	52U	85	111	111U	150	150U	55	55U
Naphthalene	86	86U	164	159	159U	242	242U	95	95U

	GLS4 5/31/96 TRG		GPS1 5/31/96 TRG		GPS2 5/31/96 TRG		GPS3 5/31/96 TRG	
	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	31	31U	61	61U	93	93U	66	66U
2-Methylnaphthalene	69	69U	114	114U	179	179U	117	117U
Naphthalene	111	111U	204	204U	287	287U	106	106U

	GPS5 5/31/96 TRG		LVBGS1 5/31/96 TRG		LVBGS2 5/31/96 TRG		LVBGS3 5/31/96 TRG	
	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	197	197U	33	33	7	7U	36	36
2-Methylnaphthalene	343	343U	57	57U	19	19U	79	79
Naphthalene	528	528U	100	100U	61	61U	165	165U

Lab Result = data as received from LSU.
Qualified data= data after validation; positive detects are shaded.
U = Indicates undetected result at the numerical level reported. Values are indistinguishable from method blank levels and therefore results are considered non-detects.
All results are reported in µg/kg = parts per billion (ppb).

GILL AND LIVER TISSUE (Continued)

LSU Fish Data Crosstab

	LVBGS4 5/31/96 TRG		LVBGS5 5/31/96 TRG		LVBLANK N/A BLK	LVGBS2 5/31/96 TRG		LVLS1 5/31/96 TRG	
	Lab Result	Qualified Data	Lab Result	Qualified Data		Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	89	89	24	24U	5	7	7U	11	11U
2-Methylnaphthalene	127	127	55	55U	15	19	19U	27	27U
Naphthalene	131	131U	128	128U	41	61	61U	75	75U

	LVLS2 5/31/96 TRG		LVLS3 5/31/96 TRG		LVLS4 5/31/96 TRG		LVPS1 5/31/96 TRG	
	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	9	9U	22	22U	25	25U	23	23U
2-Methylnaphthalene	24	24U	51	51U	45	45U	33	33U
Naphthalene	67	67U	103	103U	79	79U	61	61U

	LVPS2 5/31/96 TRG		LVPS3 5/31/96 TRG		LVPS5 5/31/96 TRG	
	Lab Result	Qualified Data	Lab Result	Qualified Data	Lab Result	Qualified Data
1-Methylnaphthalene	21	21U	726	726	235	235
2-Methylnaphthalene	26	26U	936	936	251	251
Naphthalene	74	74U	307	307	180	180U

Lab Result = data as received from LSU.
Qualified data= data after validation; positive detects are shaded.
U = Indicates undetected result at the numerical level reported. Values are indistinguishable from method blank levels and therefore results are considered non-detects.
All results are reported in µg/kg = parts per billion (ppb).

APPENDIX I. Inventory of LSU Data						
Sequence Log Page 30						
P31-32	Sample	1	6219A01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	8/7/96	11:20 AM
P33-34	Sample	2	6219A02	CAT LIVER S2 F20	8/7/96	12:45 PM
P35-36	Sample	3	6219A03	CAT LIVER S2 F17	8/7/96	4:20 PM
Sequence Log Page 61						
p63-68	Sample	1	6218B01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	8/6/96	10:14 AM
p69-70	Sample	2	6218B05	CAT GILL S1 F8	8/6/96	11:27 AM
p71-72	Sample	3	6218B18	CAT LIVER S2 F17	8/6/96	12:40 PM
p73-74	Sample	4	6218B19	CAT LIVER S4 F29	8/6/96	4:20 PM
p75-76	Sample	5	6218B20	CAT LIVER S2 F19	8/6/96	5:37 PM
Not Present	Sample	6	6218B21	CAT LIVER S2 F20 (NP=Not Present)	NP	NP
Not Present	Sample	7	6218C22	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	NP	NP
p77-116	Mass Chromatograms					
Sequence Log Page 117						
p118-123	Sample	1	6218A01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	8/5/96	1:04 PM
p124-125	Sample	2	6218A02	CAT GILL S2 F17	8/5/96	2:17 PM
p126-127	Sample	3	6218A03	CAT GILL S4 F26	8/5/96	3:27 PM
p128-129	Sample	4	6218A04	CAT GILL S4 F33	8/5/96	4:40 PM
Not Present	Sample	5	6218A05	CAT GILL S1 F8	NP	NP
p130-131	Sample	6	6218A06	CAT GILL S3 F21	8/5/96	7:00 PM
p132-133	Sample	7	6218A07	CAT GILL S2 F14	8/5/96	8:14 PM
p134-135	Sample	8	6218A08	CAT GILL S4 F32	8/5/96	9:28 PM
p136-137	Sample	9	6218A09	CAT GILL S 5 F 6	8/5/96	10:41 PM
Not Present	Sample	10	6218B10	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	NP	NP
p138-139	Sample	11	6218B11	CAT LIVER BLANK 8/5/96	8/6/96	1:08 AM
p140-141	Sample	12	6218B12	CAT LIVER S2 F12 SPK - 6218B12.D	8/6/96	2:21 AM
p142-143	Sample	13	6218B13	CAT LIVER S2 F12 DUP - 6218B13.D	8/6/96	3:35 AM
p144-145	Sample	14	6218B14	CAT LIVER S2 F12 - 6218B14.D	8/6/96	4:48 AM
p146-147	Sample	15	6218B15	CAT LIVER S3 F21 - 6218B15.D	8/6/96	6:02 AM
p148-149	Sample	16	6218B16	CAT LIVER S1 F8 - 6218B16.D	8/6/96	7:16 AM
p150-151	Sample	17	6218B17	CAT LIVER S4 F26 - 6218B17.D	8/6/96	8:29 AM
Not Present	Sample	18	6218B05	CAT LIVER S2 F17	NP	NP
Not Present	Sample	19	6218B19	CAT GILL S1 F8	NP	NP
Not Present	Sample	20	6218B20	CAT LIVER S4 F29	NP	NP
Not Present	Sample	21	6218B21	CAT LIVER S2 F19	NP	NP
Not Present	Sample	22	6218C22	CAT LIVER S2 F20	NP	NP
Not Present	Sample	23	6218C22	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	NP	NP
p152-289	Mass Chromatograms					
Sequence Log Page 290						
p291-296	Sample	1	6216A01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	8/3/96	4:44 PM
p297-298	Sample	2	6216A02	CAT GILL BLANK 7/31	8/3/96	5:57 PM
p299-300	Sample	3	6216A03	CAT GILL S2 F12 SPK	8/3/96	7:11 PM
p301-302	Sample	4	6216A04	CAT GILL S2 F12 DUP	8/3/96	8:25 PM
p303-304	Sample	5	6216A05	CAT GILL S2 F12	8/3/96	9:39 PM
p305-306	Sample	6	6216A06	CAT GILL S1 F2	8/3/96	10:52 PM
p307-308	Sample	7	6216A07	CAT GILL S1 F4	8/4/96	12:05 AM
p309-310	Sample	8	6216A08	CAT GILL S4 F25	8/4/96	1:19 AM
Not Present	Sample	9	6216A09	CAT GILL S2 F17	NP	NP
Not Present	Sample	10	6216A10	CAT GILL S4 F26	NP	NP
Not Present	Sample	11	6216A11	CAT GILL S4 F33	NP	NP
Not Present	Sample	12	6216A12	CAT GILL S1 F8	NP	NP
Not Present	Sample	13	6216A13	CAT GILL S3 F21	NP	NP
Not Present	Sample	14	6216A14	CAT GILL S2 F14	NP	NP
Not Present	Sample	15	6216A15	CAT GILL S4 F32	NP	NP
Not Present	Sample	16	6216A16	CAT GILL S5 F6	NP	NP
Not Present	Sample	17	6216B01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	NP	NP
p311-374	Mass Chromatograms					
Sequence Log Page 375						
p376-381	Sample	1	6214B01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	8/2/96	10:55 AM
p382-383	Sample	2	6214B07	CAT GILL S3 F23 - p382 with header is illegible due to diagonal copy		
p384-385	Sample	3	6214B08	CAT GILL S5 F36	8/2/96	2:55 PM
p386-387	Sample	4	6214B09	CAT GILL S5 F35	8/2/96	4:08 PM
p388-389	Sample	5	6214B10	CAT GILL S1 F1	8/2/96	5:21 PM
p390-391	Sample	6	6214B11	CAT GILL S5 F37	8/2/96	6:31 AM
p392-393	Sample	7	6214B12	CAT GILL S4 F29	8/2/96	7:45 PM
p394-395	Sample	8	6214B13	CAT GILL S5 F34	8/2/96	8:58 PM
p396-397	Sample	9	6214B14	CAT GILL S2 F20	8/2/96	10:12 PM
Not Present	Sample	10	6214B15	CAT GILL S5 F6	NP	NP
Not Present	Sample	11	6214C01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	NP	NP

p398-469	Mass Chromatograms					
Sequence Log Page 470						
p471-476	Sample	1	6214A01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	8/1/96	6:18 PM
p477-478	Sample	2	6214A02	CAT GILL BLK 7/29/96	8/1/96	7:31 PM
p479-480	Sample	3	6214A03	CAT GILL S2 F19 SPK	8/1/96	8:44 PM
p481-482	Sample	4	6214A04	CAT GILL S2 F19 DUP	8/1/96	9:58 PM
p483-484	Sample	5	6214A05	CAT GILL S2 F19	8/1/96	11:11 PM
p485-486	Sample	6	6214A06	CAT GILL S5 F42	8/2/96	12:25 PM
p487-534	Mass Chromatograms					
Sequence Log Page 535						
p540-541	Sample	1	6212A01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/30/96	6:43 PM
p544-545	Sample	2	6212A02	CAT LIVER BLK 7/12/96	7/30/96	7:56 PM
p546-547	Sample	3	6212A03	CAT LIVER S4 F25 SPK	7/30/96	9:10 PM
p548-549	Sample	4	6212A04	CAT LIVER S4 F25 DUP	7/30/96	10:23 PM
p550-551	Sample	5	6212A05	CAT LIVER S4 F25	7/30/09	11:37 PM
p552-553	Sample	6	6212A06	CAT LIVER S4 F33	7/31/96	12:50 AM
p554-555	Sample	7	6212A07	CAT LIVER S4 F33 DUP	7/31/96	2:04 AM
p556-557	Sample	8	6212A08	CAT LIVER S1 F1	7/31/96	3:18 AM
p536-539	Sample	9	6212B01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/31/96	4:31 AM
p542-543				CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/31/96	4:31 AM
p564-565	Sample	10	6212B09	CAT LIVER S5 F6	7/31/96	5:45 AM
p566-567	Sample	11	6212B10	CAT LIVER S5 F35	7/31/96	6:58 AM
p568-569	Sample	12	6212B11	CAT LIVER S5 F36	7/31/96	8:11 AM
p570-571	Sample	13	6212B12	CAT LIVER S5 F34	7/31/96	9:25 AM
p572-573	Sample	14	6212B13	CAT LIVER S1 F2	7/31/96	10:38 AM
p576-577	Sample	15	6212B14	CAT LIVER S4 F32	7/31/96	11:50 AM
p578-579	Sample	16	6212B15	CAT LIVER S5 F37	7/31/96	1:03 PM
p558-563	Sample	17	6212B16	CAT LIVER S5 F42	7/31/96	2:15 PM
Not Present	Sample	18	6212C17	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/31/96	3:28 PM
Not Present	Sample	19	6212C18	2-FBP 5 UL 100ppm in 200ul HEX(5 o -	NP	NP
Not Present	Sample	20	6212C19	EPA IS 40 ul 40 ppm in 200 ul HEX(18 -	NP	NP
p580-723	Mass Chromatograms					
Sequence Log Page 724						
p729-730	Sample	1	6211A01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/29/96	2:30 PM
p735-736	Sample	2	6211A02	CAT LIVER BLK 7/9/96	7/29/96	3:43 PM
Not Present	Sample	3	6211A03	CAT LIVER S2 F14 SPK	NP	NP
p737-738	Sample	4	6211A04	CAT LIVER S2 F14 DUP	7/29/96	6:08 PM
Not Present	Sample	5	6211A05	CAT LIVER S2 F14	NP	NP
p739-740	Sample	6	6211A06	CAT LIVER S1 F4	7/29/96	8:35 PM
Not Present	Sample	7	6211A07	CAT LIVER S2 F19	NP	NP
Not Present	Sample	8	6211A08	CAT LIVER S2 F20	NP	NP
p731-732	Sample	9	6211B01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/30/96	12:16 AM
Not Present	Sample	10	6211B09	CAT LIVER S2 F12	NP	NP
p741-742	Sample	11	6211B10	CAT LIVER S3 F23	7/30/96	2:43 AM
Not Present	Sample	12	6211B11	CAT LIVER S4 F26	NP	NP
Not Present	Sample	13	6211B12	CAT LIVER S3 F21	NP	NP
Not Present	Sample	14	6211B13	CAT LIVER S4 F29	NP	NP
Not Present	Sample	15	6211B14	CAT LIVER S2 F17	NP	NP
Not Present	Sample	16	6211B15	CAT LIVER S1 F8	NP	NP
p725-728	Sample	17	6211C16	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/30/96	10:09 AM
p733-734				CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/30/96	10:09 AM
p743-877	Mass Chromatograms					
Sequence Log Page 878						
p882-883	Sample	1	6205A01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/23/96	3:43 PM
p886-887	Sample	2	6205A02	GILL BLK 7/8/96	7/23/96	4:56 PM
p888-889	Sample	3	6205A03	GILL F3 S13 SPK	7/23/96	6:09 PM
p890-891	Sample	4	6205A04	GILL F3 S13 DUP	7/23/96	7:23 PM
p892-893	Sample	5	6205A05	GILL F3 S13 DUP	7/23/96	8:36 PM
p894-895	Sample	6	6205A06	GILL F5 S6	7/23/96	9:50 PM
p896-897	Sample	7	6205A07	GILL F3 S1	7/23/96	11:04 PM
p898-899	Sample	8	6205A08	GILL F4 S1	7/24/96	12:17 AM
p900-901	Sample	9	6205A09	GILL F3 S10	7/24/96	1:31 AM
p902-903	Sample	10	6205A10	GILL F5 S8	7/24/96	2:45 AM
p904-905	Sample	11	6205A11	GILL F4 S8	7/24/96	3:58 AM
p906-907	Sample	12	6205A12	GILL F3 S12	7/24/96	5:12 AM
p908-909	Sample	13	6205A13	GILL F5 S7	7/24/96	6:25 AM
p910-911	Sample	14	6205A14	GILL F3 S3	7/24/96	7:39 AM
p912-913	Sample	15	6205A15	GILL F4 S7	7/24/96	8:51 AM
p914-915	Sample	16	6205A16	GILL F2 S14	7/24/96	10:04 AM
p879-881	Sample	17	6205B01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/24/96	11:17 AM
p884-885				CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/24/96	11:17 AM
p916-1051	Mass Chromatograms					

Sequence Log Page 1052						
p1057-1058	Sample	1	6193A01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/11/96	6:23 PM
p1061-1062	Sample	2	6193A02	GILL BLK 7/5/96	7/11/96	7:37 PM
p1063-1064	Sample	3	6193A03	GILL S2 F5	7/11/96	8:50 PM
p1065-1066	Sample	4	6193A04	GILL S2 F8	7/11/96	10:04 PM
p1067-1068	Sample	5	6193A05	GILL S4 F2	7/11/96	11:18 PM
p1069-1070	Sample	6	6193A06	GILL S2 F9	7/12/96	12:31 AM
p1071-1072	Sample	7	6193A07	GILL S4 F10	7/12/96	1:45 AM
p1073-1074	Sample	8	6193A08	GILL S2 F3	7/12/96	2:59 AM
p1075-1076	Sample	9	6193A09	GILL S2 F15	7/12/96	4:13 AM
p1077-1078	Sample	10	6193A10	GILL S4 F3	7/12/96	5:26 AM
p1079-1080	Sample	11	6193A11	GILL S1 F11 DUP	7/12/96	6:40 AM
p1081-1082	Sample	12	6193A12	GILL S2 F4 - REP on printout	7/12/96	7:53 AM
p1053-1056	Sample	13	6193B01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/12/96	9:06 AM
p1083-1186	Mass Chromatograms					
Sequence Log Page 1187						
p1192-1193	Sample	1	6191A01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/9/96	5:33 PM
p1196-1197	Sample	2	6191A02	GILL BLK 7/2/96	7/9/96	6:47 PM
p1198-1199	Sample	3	6191A03	GILL S5 F14 SPK	7/9/96	8:00 PM
p1200-1201	Sample	4	6191A04	GILL S5 F14 DUP	7/9/96	9:14 PM
p1202-1203	Sample	5	6191A05	GILL S5 F14	7/9/96	10:28 PM
Not Present	Sample	6	6191A06	GILL S2 F14	NP	NP
p1204-1205	Sample	7	6191A07	GILL S2 F7	7/10/96	12:55 AM
p1206-1207	Sample	8	6191A08	GILL S3 F2	7/10/96	2:09 AM
p1208-1209	Sample	9	6191A09	GILL S1 F5	7/10/96	3:23 AM
p1210-1211	Sample	10	6191A10	GILL S5 F13	7/10/96	4:37 AM
p1212-1213	Sample	11	6191A11	GILL S3 F15	7/10/96	5:50 AM
p1214-1215	Sample	12	6191A12	GILL S3 F11	7/10/96	7:03 AM
p1216-1217	Sample	13	6191A13	GILL S1 F2	7/10/96	8:16 AM
p1218-1219	Sample	14	6191A14	GILL S1 F3	7/10/96	9:29 AM
p1188-1191	Sample	15	6191B01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/10/96	10:42 AM
p1194-1195				CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/10/96	10:42 AM
p1220-1336	Mass Chromatograms					
Sequence Log Page 1337						
p1342-1343	Sample	1	6187A01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/5/96	7:15 PM
p1346-1347	Sample	2	6187A02	GILL BLK 6/28/96	7/5/96	8:28 PM
p1348-1349	Sample	3	6187A03	GILL S1 G11	7/5/96	9:42 PM
p1350-1351	Sample	4	6187A04	GILL S1 G15	7/5/96	10:55 PM
p1352-1353	Sample	5	6187A05	GILL S1 G9	7/6/96	12:09 AM
p1354-1355	Sample	6	6187A06	GILL S1 G17	7/6/96	1:23 AM
p1356-1357	Sample	7	6187A07	GILL S1 G7	7/6/96	2:36 AM
p1358-1359	Sample	8	6187A08	GILL S1 G13	7/6/96	3:50 AM
p1360-1361	Sample	9	6187A09	MUSSEL S2 M52 DUP RE	7/6/96	5:04 AM
p1362-1363	Sample	10	6187A10	MUSSEL S2 M56	7/6/96	6:17 AM
p1364-1365	Sample	11	6187A11	MUSSEL S1 M50	7/6/96	7:31 AM
p1366-1367	Sample	12	6187A12	MUSSEL S1 M48	7/6/96	8:45 AM
p1338-1341	Sample	13	6187B01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/6/96	9:58 AM
p1342-1471	Mass Chromatograms					
Sequence Log Page 1472						
p1473-1478	Sample	1	6185A01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/3/96	5:10 PM
p1481-1482	Sample	2	6185A02	MUSSEL BLK SPK	7/3/96	6:23 PM
p1483-1484	Sample	3	6185A03	MUSSEL S2 M52 SPK	7/3/96	7:37 PM
p1485-1486	Sample	4	6185A04	MUSSEL S2 M52	7/3/96	8:50 PM
p1487-1488	Sample	5	6185A05	MUSSEL S2 M52 DUP	7/3/96	10:04 PM
p1489-1490	Sample	6	6185A06	MUSSEL S1 M46	7/3/96	11:18 PM
p1491-1492	Sample	7	6185A07	MUSSEL S2 M54	7/4/96	12:31 AM
p1493-1494	Sample	8	6185A08	MUSSEL S2 M56	7/4/96	1:45 AM
p1495-1496	Sample	9	6185A09	MUSSEL S1 M44	7/4/96	2:59 AM
p1497-1498	Sample	10	6185A10	MUSSEL S1 M50	7/4/96	4:13 AM
p1499-1500	Sample	11	6185A11	MUSSEL S1 M48	7/4/96	5:26 AM
p1479-1480	Sample	12	6185B01	CBL STD@ 0.5 PPM W/ 0.5 PPM 2FBP	7/4/96	6:39 AM
p1481-1596	Mass Chromatograms					
Three unbatched sediment sample runs.						
p1597-1602				p1597-1602 6183D01 CBLStd.	7/2/96	7:52 AM
Sequence Log Page 1633						
p1640-1641	Sample	1	6183E13	SED: 6 LDB	7/2/96	2:18 PM
p1642-1643	Sample	2	6183E14	SED: 6 MS	7/2/96	3:30 PM
p1644-1645	Sample	3	6183E15	SED: 5 RDB	7/2/96	4:43 PM
p1634-1639	Sample	4	6183E01	CBL STOCK @ 5 PPM	7/2/96	5:56 PM
p1646-1677	Mass Chromatograms					
Sequence Log Page 1678						
p1683-1684	handwritten in		6183C01	CBLSTD	7/1/96	4:21 PM

p1685-1686	Sample	1	6183C02	MUSSEL S4 M9	7/1/96	6:22 PM
p1687-1688	Sample	2	6183C03	MUSSEL S5 M16	7/1/96	7:36 PM
p1689-1690	Sample	3	6183C04	MUSSEL S3 M36	7/1/96	8:50 PM
p1691-1692	Sample	4	6183C05	MUSSEL S5 M18	7/1/96	10:03 PM
p1693-1694	Sample	5	6183C06	MUSSEL S5 M26	7/1/96	11:17 PM
p1695-1696	Sample	6	6183C07	MUSSEL S4 M5	7/2/96	12:31 PM
p1697-1698	Sample	7	6183C08	MUSSEL S3 M32	7/2/96	1:44 AM
p1699-1700	Sample	8	6183C09	MUSSEL S4 M11	7/2/96	2:58 AM
p1701-1702	Sample	9	6183C10	MUSSEL S4 M14	7/2/96	4:12 AM
p1703-1704	Sample	10	6183C11	MUSSEL S5 M24	7/2/96	5:26 AM
p1705-1706	Sample	11	6183C12	MUSSEL S3 M42	7/2/96	6:39 AM
p1679-1682	Sample	12	6183D01	CBL STD @ 0.5 PPM W/ 0.5PPM 2FBP	7/2/96	7:52 AM
p1707-1810	Mass Chromatograms					
Sequence Log Page 1811						
p1816-1817	Sample	1	6180A01	CBL STD @ 0.5 PPM W/ 0.5PPM 2FBP	6/27/96	7:58 PM
p1820-1821	Sample	2	6180A02	MUSSEL BLK	6/27/96	9:12 PM
p1822-1823	Sample	3	6180A03	MUSSEL S4 M2	6/27/96	10:25 PM
p1824-1825	Sample	4	6180A04	MUSSEL S4 M2 DUP	6/27/96	11:39 PM
p1826-1827	Sample	5	6180A05	MUSSEL S4 M2 SPK	6/28/96	12:52 AM
p1828-1829	Sample	6	6180A06	MUSSEL S3 M10	6/28/96	2:06 AM
p1830-1831	Sample	7	6180A07	MUSSEL S5 M28	6/28/96	3:20 AM
p1832-1833	Sample	8	6180A08	MUSSEL S4 M7	6/28/96	4:33 AM
p1834-1835	Sample	9	6180A09	MUSSEL S4 M1	6/28/96	5:47 AM
p1836-1837	Sample	10	6180A10	MUSSEL S3 M34	6/28/96	7:00 AM
p1838-1839	Sample	11	6180A11	MUSSEL S3 M38	6/28/96	8:13 AM
p1840-1841	Sample	12	6180A12	MUSSEL S5 M20	6/28/96	9:26 AM
p1842-1843	Sample	13	6180A13	MUSSEL S5 M22	6/28/96	10:39 AM
p1812-1815	Sample	14	6180B01	CBL STD @ 0.5 PPM W/ 0.5PPM 2FBP		
p1844-1899	Mass Chromatograms					
Sequence Log Page 1938 (see notes pg.1940)				6/25/96 logbook p 212-213, p 1939 shows dilution schemes		NP
Not Present	Sample	1	6177A01	CBL STOCK III @ 5 PPM	NP	NP
Not Present	Sample	2	6177A02	FSHKL 96-43 BLANK	NP	NP
Not Present	Sample	3	6177A03	FSHKL 96-43	NP	NP
Not Present	Sample	4	6177A04	FSHKL 96-43 B	NP	NP
Not Present	Sample	5	6177A05	FSHKL 96-43 C	NP	NP
Not Present	Sample	6	6177A06	FSHKL 96-43 D	NP	NP
Not Present	Sample	7	6177A07	FSHKL 96-43 A	NP	NP
Not Present	Sample	8	6177B01	CBL STOCK III @ 5 PPM	NP	NP
p1960-1961	Sample	9	6177B08	CBL STD @ 0.5 PPM W/ 2FBP	6/25/96	11:55 PM
p1964-1965	Sample	10	6177B09	SED BLANK 2	6/26/96	1:09 AM
p1966-1967	Sample	11	6177B10	1RDB	6/26/96	2:23 AM
p1968-1969	Sample	12	6177B11	7LDB	6/26/96	3:37 AM
p1970-1971	Sample	13	6177B12	5MS	6/26/96	4:50 AM
p1972-1973	Sample	14	6177B13	7MS	6/26/96	6:04 AM
p1974-1975	Sample	15	6177B14	1LDB	6/26/96	7:17 AM
p1976-1977	Sample	16	6177B15	7RDB	6/26/96	8:30 AM
p1944-1945	Sample	17	6177C16	CBL STD @ 0.5 PPM W/ 2FBP	6/26/96	9:43 AM
p1948-1949	Sample	18	6177C17	2RDB	6/26/96	10:56 PM
p1950-1951	Sample	19	6177C18	2LDB	6/26/96	12:08 PM
p1952-1953	Sample	20	6177C19	4LDB	6/26/96	1:20 PM
Not Present	Sample	21	Pause	Pause	NP	NP
Not Present	rerun -	22	6177C20	6MS	NP	NP
p1954-1955	Sample	23	6177C21	3LDB	6/26/96	3:46 PM
p1978-2098	Mass Chromatograms					
Sequence Log Page 2099						
p2134-2135	Sample	1	6173A	INSTR BLANK = DCM Blank mass chrom p 2134-2135		
p2104-2105	Sample	2	6173A01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/21/96	6:02 PM
p2108-2109	Sample	3	6173A02	BLANK 6/17	6/21/96	7:16 PM
p2110-2111	Sample	4	6173A03	CATED: S2F17	6/21/96	8:30 PM
p2112-2113	Sample	5	6173A04	CATED: S1F8	6/21/96	9:43 PM
p2114-2115	Sample	6	6173A05	CATED: S1F4	6/21/96	10:57 PM
p2116-2117	Sample	7	6173A06	CATED: S2F12	6/22/96	12:11 AM
p2118-2119	Sample	8	6173A07	CATED: S5F42	6/22/96	1:24 AM
p2120-2121	Sample	9	6173A08	CATED: S3F21	6/22/96	2:38 AM
p2122-2123	Sample	10	6173A09	CATED: S4F33	6/22/96	3:52 AM
p2124-2125	Sample	11	6173A10	CATED: S5F35	6/22/96	5:06 AM
p2126-2127	Sample	12	6173A11	CATED: S1F1	6/22/96	6:19 AM
p2128-2129	Sample	13	6173A12	CATED: S5F37	6/22/96	7:33 AM
p2130-2131	Sample	14	6173A13	CATED: S4F25	6/22/96	8:47 AM
p2132-2133	Sample	15	6173A14	CATED: S2F19	6/22/96	10:00 AM
p2100-2103	Sample	16	6173B01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/22/96	11:13 AM
p2106-2107				CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/22/96	11:13 AM

p2134-2271	Mass Chromatograms plus 2 scanned spectra					
Sequence Log Page 2274						
Not Present	Sample	1	6172C01	CAN / DCM BOTTLE BLK	NP	NP
p2281-2282	Sample	2	6172C02	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/20/96	8:58 PM
p2283-2284	Sample	3	6172C03	MSPD BLK 6/20/96	6/20/96	10:11 PM
p2285-2286	Sample	4	6172C04	CATED S1F2 SPK	6/20/96	11:25 PM
p2287-2288	Sample	5	6172C05	CATED S1F2	6/21/96	12:38 AM
p2289-2290	Sample	6	6172C06	CATED S1F2 DUP	6/21/96	1:52 AM
p2291-2292	Sample	7	6172C07	CATED S5F36	6/21/96	3:06 AM
p2293-2294	Sample	8	6172C08	CATED S4F26	6/21/96	4:19 AM
p2295-2296	Sample	9	6172C09	CATED S3F23	6/21/96	5:33 AM
p2297-2298	Sample	10	6172C10	CATED S5F34	6/21/96	6:47 AM
p2299-2300	Sample	11	6172C11	CATED S4 F32	6/21/96	8:00 AM
p2301-2302	Sample	12	6172C12	CATED S2 F20	6/21/96	9:13 AM
p2303-2304	Sample	13	6172C13	CATED S4 F29	6/21/96	10:25 AM
p2305-2306	Sample	14	6172C14	CATED S5 F6	6/21/96	11:38 AM
p2307-2308	Sample	15	6172C15	CATED S2 F14	6/21/96	12:50 PM
p2275-2280	Sample	16	6172D01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/21/96	2:03 PM
p2309-2436	Mass Chromatograms					
Sequence Log Page 2437						
p2442-2443	Sample	1	6171A01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/19/96	6:54 PM
Not Present	Sample	2	6171A02	BLK Plastic	NP	NP
Not Present	Sample	3	6171A03	BLK Glass	NP	NP
p2446-2447	Sample	4	6171A04	S1 F3 SPK	6/19/96	10:34 PM
p2448-2449	Sample	5	6171A05	S1 F3 DUP	6/19/96	11:48 PM
p2450-2451	Sample	6	6171A06	S1 F3	6/20/96	1:01 AM
p2452-2453	Sample	7	6171A07	S1 F5	6/20/96	2:12 AM
p2454-2455	Sample	8	6171A08	S1 F15	6/20/96	3:28 AM
p2456-2457	Sample	9	6171A09	S1 F2	6/20/96	4:42 AM
p2458-2459	Sample	10	6171A10	S1 F9 Glass	6/20/96	5:56 AM
p2460-1461	Sample	11	6171A11	S1 F7	6/20/96	7:09 AM
p2462-2463	Sample	12	6171A12	S1 F17	6/20/96	8:22 AM
p2438-2441	Sample	13	6171B01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/20/96	9:35 AM
p2444-2445				CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/20/96	9:35 AM
p2464-2582	Mass Chromatograms					
Sequence Log Page 2583						
Not Present	Sample	1	6169A01	FSHKL STD. @ 10ppm	NP	NP
Not Present	Sample	2	6169A02	FSHKL BLK 5/29/96	NP	NP
Not Present	Sample	3	6169A03	FSHKL BLK 6/6/96	NP	NP
Not Present	Sample	4	6169A04	FSHKL 96-21	NP	NP
Not Present	Sample	5	6169A05	FSHKL 96-29	NP	NP
Not Present	Sample	6	6169B01	FSHKL STD. @ 10ppm	NP	NP
p2588-2590	Sample	7	6169C01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/17/96	10:24 PM
p2664-2665				CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/17/96	10:24 PM
p2592-2593	Sample	8	6169C02	BLANK 6/12	6/17/96	11:38 PM
p2594-2595	Sample	9	6169C03	LIVER S4 F1	6/18/96	12:51 AM
p2596-2597	Sample	10	6169C04	LIVER S4 F3	6/18/96	2:05 AM
p2598-2569A	Sample	11	6169C05	LIVER S1 F13	6/18/96	3:18 AM
p2570A-2571A	Sample	12	6169C06	LIVER S3 F3	6/18/96	4:32 AM
p2572A-2573A	Sample	13	6169C07	LIVER S1 F11	6/18/96	5:46 AM
p2584-2587	Sample	14	6169D01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/18/96	6:59 AM
p2574-2656	Mass Chromatograms					
Sequence Log Page 2657				6/14/96 run log p 204-205		
Not Present	Sample	1	6166A01	STANDARD DWM 550	NP	NP
p2662-2663	Sample	2	6166B01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/14/96	7:49 PM
p2666-2667	Sample	3	6166B02	SED: BLANK 6/10	6/14/96	9:02 PM
p2668-2669	Sample	4	6166B03	SED: 3 MS SPK	6/14/96	10:16 PM
p2670-2671	Sample	5	6166B04	SED: 3 MS	6/14/96	11:29 PM
p2672-2673	Sample	6	6166B05	SED: 3 MS DUP	6/15/96	12:43 AM
p2674-2675	Sample	7	6166B06	SED:3 RDB	6/15/96	1:56 AM
p2676-2677	Sample	8	6166B07	SED:4 RDB	6/15/96	3:10 AM
p2678-2679	Sample	9	6166B08	SED: 4 MS	6/15/96	4:23 AM
p2680-2681	Sample	10	6166B09	SED:2 MS	6/15/96	5:37 AM
p2682-2683	Sample	11	6166B10	SED:1 MS	6/15/96	6:51 AM
p2684-2685	Sample	12	6166B11	SED:5 LDB	6/15/96	8:04 AM
p2658-2661	Sample	13	6165C01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/15/96	9:18 AM
p2783	Cholesterol			Chromatogram peak labeled Cholesterol		
Sequence Log Page 2784						
p2789-2790	Sample	1	6165A01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP		6:29 PM
p2793-2794	Sample	2	6165A02	LIV: BLANK 6/12	6/13/96	7:42 PM
p2795-2796	Sample	3	6165A03	NIST REF MUSSEL - 1974	6/13/96	8:56 PM
p2797-2798	Sample	4	6165A04	LIV: S3F12	6/13/96	10:09 PM

p2799-2800	Sample	5	6165A05	LIV:S4F2 (2X-IS)	6/13/96	11:23 PM
p2801-2802	Sample	6	6165A06	LIV: S3F10	6/14/96	12:36 AM
p2803-2804	Sample	7	6165A07	LIV:S3F2	6/14/96	1:50 AM
p2805-2806	Sample	8	6165A08	LIV: S4F10	6/14/96	3:04 AM
p2807-2808	Sample	9	6165A09	LIV: S5F8	6/14/96	4:17 AM
p2785-2786	Sample	10	6165B01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/14/96	5:31 AM
p2791-2792				CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/14/96	5:31 AM
p2813-2814				CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/14/96	5:31 AM
p2817-2818	Sample	11	6165B10	LIV: S3F11	6/14/96	6:45 AM
p2819-2820	Sample	12	6165B11	LIV: S5F6	6/14/96	7:58 AM
p2821-2822	Sample	13	6165B12	LIV: S2F15 (RED)	6/14/96	10:16 AM
p2823-2824	Sample	14	6165B13	LIV: S5F13 (RED)	6/14/96	11:29 AM
p2825-2826	Sample	15	6165B14	LIV: S5F14 (RED)	6/14/96	12:41 PM
p2827-2828	Sample	16	6165B15	LIV: S3F13 (RED)	6/14/96	1:54 PM
p2829-2830	Sample	17	6165B16	LIV: S3F15 (RED)	6/14/96	3:06 PM
p2809-2812	Sample	18	6165B17	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/14/96	4:17 PM
p2815-2816				CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/14/96	4:17 PM
p2831-2980	Mass Chromatograms					
Sequence Log Page 2981						
p2986-2987	Sample	1	6164A01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/12/96	1:12 PM
p2990-2991	Sample	2	6164A02	S3F15 rerun	6/12/96	2:25 PM
p2992-2993	Sample	3	6164A03	S5F13 rerun	6/12/96	3:37 PM
p2994-2995	Sample	4	6164A04	S5F8 rerun	6/12/96	4:50 PM
p2996-2997	Sample	5	6164A05	LIV: S2F7	6/12/96	6:50 PM
p2998-2999	Sample	6	6164A06	LIV: S2F9	6/12/96	8:03 PM
p3000-3001	Sample	7	6164A07	LIV: S2F3	6/12/96	9:17 PM
p3002-3003	Sample	8	6164A08	LIV: S5F7	6/12/96	10:31 PM
p3004-3005	Sample	9	6164A09	LIV: S4F7	6/12/96	11:44 PM
p3006-3007	Sample	10	6164A10	LIV: S2F4	6/13/96	12:58 AM
p3008-3009	Sample	11	6164A11	LIV: S2F5	6/13/96	2:12 AM
p3010-3011	Sample	12	6164A12	LIV: S2F8	6/13/96	3:26 AM
p3012-3013	Sample	13	6164A13	LIV: S2F14 (red)	6/13/96	4:39 AM
p2982-2985	Sample	14	6164B01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/13/96	5:53 AM
p3014-3125	Mass Chromatograms					
Sequence Log Page 3126						
Not Present		1	6160A01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	NP	NP
Not Present		2	6160A02	MSPD BLK 6/10/96	NP	NP
Not Present		3	6163A03	S5F6	NP	NP
Not Present		4	6163A04	S4F8	NP	NP
Not Present		5	6163A05	S5F8	NP	NP
Not Present		6	6163A06	S4F9	NP	NP
Not Present		7	6163A07	S2F5	NP	NP
Not Present		8	6163A08	S2F15	NP	NP
Not Present		9	6163A09	S4F10	NP	NP
Not Present		10	6163A10	S2F9	NP	NP
p3131-3132		11	6163B01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/11/96	9:55 PM
p3135-3136		12	6163B11	BLK 6/11/96	6/11/96	11:08 PM
p3137-3138		13	6163B12	S3F1SPK	6/12/96	12:22 AM
p3139-3140		14	6163B13	S3F1	6/12/96	1:35 AM
p3141-3142		15	6163B14	S3F1DUP	6/12/96	2:49 AM
p3143-3144		16	6163B15	S4F8	6/12/96	4:02 AM
p3127-3130		17	6163B16	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/12/96	5:16 AM
p3145-3200	Mass Chromatograms					
Sequence Log Page 3205						
p3210-3211		1	6160A01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/11/96	9:46 AM
p3214-3215		2	6160A02	S5F6	6/11/96	10:59 AM
p3216-3217		3	6163A03	S4F8	6/11/96	12:11 PM
p3218-3219		4	6163A04	S5F8	6/11/96	1:23 PM
p3220-3221		5	6163A05	S5F8	6/11/96	2:36 PM
p3222-3223		6	6163A06	S4F9	6/11/96	3:48 PM
p3224-3225		7	6163A07	S2F5	6/11/96	5:01 AM
p3226-3227		8	6163A08	S2F15	6/11/96	6:14 PM
p3228-3229		9	6163A09	S4F10	6/11/96	7:28 PM
p3230-3231		10	6163A10	S2F9	6/11/96	8:41 PM
p3206-3209		11	6163B01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/11/96	9:55 PM
p3212-3213				CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/11/96	9:55 PM
Not Present		12	6163B11	BLK 6/11/96	NP	NP
Not Present		13	6163B12	S3F1SPK	NP	NP
Not Present		14	6163B13	S3F1	NP	NP
Not Present		15	6163B14	S3F1DUP	NP	NP
Not Present		16	6163B15	S4F8	NP	NP
Not Present		17	6163B16	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	NP	NP

p3232-3311	Mass Chromatograms					
Sequence Log Page 3314						
p3321-3322		1	6159A01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/10/96	6:32 PM
p3325-3326		2	6159A02	MSPD BLK 6/10/96	6/10/96	7:45 PM
p3327-3328		3	6159A03	S4F7	6/10/96	8:58 PM
p3329-3330		4	6159A04	S2F3	6/10/96	10:12 PM
p3331-3332		5	6159A05	S2F7	6/10/96	11:25 PM
p3333-3334		6	6159A06	S2F14	6/11/96	12:38 AM
p3335-3336		7	6159A07	S3F1	6/11/96	1:52 AM
p3337-3338		8	6159A08	S4F2	6/11/96	3:05 AM
p3339-3340		9	6159A09	S3F13	6/11/96	4:19 AM
p3341-3342		10	6159A10	S2F8	6/11/96	5:32 AM
p3343-3344		11	6159A11	S5F7	6/11/96	6:46 AM
p3323-3324		12	6159B01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/11/96	7:58 AM
p3345-3440	Mass Chromatograms					
Sequence Log Page 3446						
p3451-3452		1	6158A01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/7/96	8:10 PM
p3455-3456		2	6158A02	MSPD BLK 6/7/96	6/7/96	9:24 PM
p3457-3458		3	6158A03	S5F14 SPIKE	6/7/96	10:37 PM
p3459-3460		4	6158A04	S5F14	6/7/96	11:51 PM
p3461-3462		5	6158A05	S5F14 DUP	6/8/96	1:04 AM
p3463-3464		6	6158A06	S3F2	6/8/96	2:18 AM
p3465-3466		7	6158A07	S3F12	6/8/96	3:31 AM
p3467-3468		8	6158A08	S3F3	6/8/96	4:45 AM
p3469-3470		9	6158A09	S3F11	6/8/96	5:59 AM
p3471-3472		10	6158A10	S3F10	6/8/96	7:12 AM
p3473-3474		11	6158A11	S4F3	6/8/96	8:26 AM
p3447-3450		12	6158B01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/8/96	9:39 AM
p3475-3590	Mass Chromatograms					
Sequence Log Page 3597					6/6/96	8:30 PM
p3606-3607		1	6157B02	BLK 6/6/96 DCM / ACN	6/6/96	9:44 PM
p3608-3609		2	6157B03	Site 5 Fish 13 DCM/ACN	6/6/96	10:57 PM
p3610-3611		3	6157B04	Site 3 Fish 15 DCM/ACN	6/7/96	12:11 AM
p3612-3613		4	6157B05	Site 3 Fish 15 ACN	6/7/96	1:24 AM
p3614-3615		5	6157B06	Site 5 Fish 13 ACN	6/7/96	2:38 AM
p3616-3617		6	6157B07	Site 5 Fish 13 SPK ACN	6/7/96	3:52 AM
p3618-3619		7	6157B08	Site 3 Fish 15 ACN/DCM	6/7/96	5:05 AM
p3598-3601		8	6157C01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/7/96	6:19 AM
p3620-3673	Mass Chromatograms					
Sequence Log Page 3682						
Not Present		1	6156A01	1 BLK 6\4\96	NP	NP
Not Present		2	6156A02	1 BLK CAN	NP	NP
Not Present		3	6156A03	2 FISH	NP	NP
Not Present		4	6156B01	2 FISH Repeat	NP	NP
p3687-3688		5	6156C01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/4/96	9:07 PM
p3691-3692		6	6156C02	1 BLK 6/4/96	6/4/96	10:21 PM
p3693-3694		7	6156C03	2 FISH #17	6/4/96	11:34 PM
p3695-3696		8	6156C04	3 FISH #17 DUP	6/5/96	12:48 AM
p3697-3698		9	6156C05	4 FISH #17 DUP2	6/5/96	2:02 AM
p3699-3700		10	6156C06	5 FISH #9	6/5/96	3:15 AM
p3701-3702		11	6156C07	6 FISH #13	6/5/96	4:29 AM
p3703-3704		12	6156C08	7 FISH #2	6/5/96	5:43 AM
p3705-3706		13	6156C09	8 FISH #15	6/5/96	6:56 AM
p3707-3708		14	6156C10	9 FISH #7	6/5/96	8:09 AM
p3683-3686		15	6156E01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/5/96	9:22 AM
p3709-3710		16	6156C11	10 FISH # 5	6/5/96	10:35 AM
p3711-3712		17	6156C12	11 FISH # 11	6/5/96	11:47 AM
p3713-3714		18	6156C13	12 FISH # 3	6/5/96	1:00 PM
p3715-3716		19	6156C14	13 FISH # 3 SPK	6/5/96	2:13 PM
p3717-3718		20	6156D01	CBL STD @ 0.5 PPM W/ 0.5 PPM 2FBP	6/5/96	3:25 PM
Not Present		21	6156D02	PNA @ 2 PPM (US-106)	NP	NP
Not Present		22	6156E16	12 FISH # 3 (SCAN MODE)	NP	NP
Not Present		23	6156E20	Gasoline: TK500-4 rec'd 6/4/96dj	NP	NP
p3719-3857	Mass Chromatograms					
Sequence Log Page 3858						
p3863-3864		1	6155C01	CBL STD @ 0.5 PPM W/ 2-FBP 0.5 PPM	6/3/96	7:18 PM
p3865-3866		2	6155C02	1 BLANK 10ul EPA@40	6/3/96	8:31 PM
p3867-3868		3	6155C03	2 FISH #17 10 ul EPA@40	6/3/96	9:45 PM
p3869-3870		4	6155C04	3 FISH #17DUP. 10ul EPA@40	6/3/96	10:59 PM
p3871-3872		5	6155C06	5 FISH #3 10 ul EPA@40	6/4/96	12:12 AM
p3873-3874		6	6155C07	6 FISH #7 10ul EPA@40	6/4/96	1:26 AM
p3875-3876		7	6155C08	7 FISH #15 10ul EPA@40	6/4/96	2:39 AM

p3877-3878		8	6155C09	8 FISH #2 10ul EPA@40	6/4/96	3:53 AM
p3879-3880		9	6155C10	9 FISH #9 10ul EPA@40	6/4/96	5:06 AM
p3881-3882		10	6155C11	10 FISH #13 10ul EPA@40	6/4/96	6:20 AM
p3883-3884		11	6155C12	11 FISH #5 10ul EPA@40	6/4/96	7:33 AM
p3885-3886		12	6155C13	12 FISH #11 10ul EPA@40	6/4/96	8:46 AM
Not Present		13	6155C05	4 FISH #17DUP2 10 ul EPA@40	NP	NP
p3887-3888		14	6155C14	13 FISH #17SPIKE: 10ul CBL@ 0.5	6/4/96	11:11 AM
Not Present		15	6155D01	CBL STD @ 0.5 PPM W/ 2-FBP 0.5 PPM	NP	NP
p3889-4012	Mass Chromatograms					
Sequence Log Page 3859						
Not Present		1	6155A01	CBL STD @ 0.5 PPM W/ 2-FBP 0.5 PPM	NP	NP
Not Present		2	6155A02	2 TISSUE 150ul EPA	NP	NP
Not Present		3	6155A03	7 TISSUE 10ul CBL STOCK	NP	NP
Not Present		4	6155B01	CBL STD @ 0.5 PPM W/ 2-FBP 0.5 PPM	NP	NP

TERRA

CONSULTING GROUP

SEDIMENT SAMPLES

Executive Summary

A total of 20 sediment samples were collected from seven locations on Blind River on June 5, 1996 in response to the Marathon Pipe Line Company gasoline release from a pipe line right-of-way adjacent to U. S. Highway 61 (Airline Highway) in St. James Parish. The state-collected sediment samples were analyzed for semivolatile compounds by Dr. Jay Means of the Louisiana State University, Department of Physiology, Pharmacology, and Toxicology via a special trace level method developed by his laboratory (1). The analytical procedure employed is not an United States Environmental Protection Agency (USEPA) approved reference method and, therefore, data provided by LSU could not be fully validated in accordance with the U. S. EPA guidance document, *National Functional Guidelines for Organic Data Review* (2). To the extent possible, data were evaluated and qualified. Volatile analyses were also performed, however, no quality assurance/quality control information was available for review.

The method utilized by LSU for semivolatile analyses is very sensitive and has a higher degree of specificity than routine gas chromatographic methods. However, it has a lower degree of specificity than the routine repetitive scanning gas chromatographic / mass spectrometric (GC/MS) methods specified by the EPA. Specifically, the LSU method does not have the ability to provide full scan mass spectra for compounds of interest. Consequently, confirmation of the presence or identity of the analytes detected by the LSU method by comparing the mass sample spectra to spectra of known reference

materials is not possible. The method employed by LSU is not a suitable substitute for SW-846 GC/MS methods for proving the presence of contaminants.

The data reported by LSU are very prone to contain values which are higher than what may actually be in the sample. The positive results should only be used if an undefined and positive bias can be tolerated or in conjunction with other data generated from methods generally accepted or recommended by the EPA. Mass spectra are not available and were not subsequently produced by LSU for the positive results that were reported. Consequently, a positive result may be totally false and cannot withstand a data challenge.

Introduction

A total of 20 sediment samples were collected from seven locations on Blind River on June 5, 1996 in response to the Marathon Pipe Line Company gasoline release from a pipe line right-of-way adjacent to U. S. Highway 61 (Airline Highway) in St. James Parish. Sediment samples were collected from the left descending bank (LDB), the right descending bank (RDB), and at midstream (MS). The midstream sample identification, MS, is not to be confused with the typical designation for matrix spike used by most laboratories.

Dr. Jay Means of the Louisiana State University, Department of Physiology, Pharmacology, and Toxicology analyzed the state-collected sediment samples for volatile and semivolatile compounds. Semivolatile analyses were conducted via a special trace level method developed by his laboratory (1). The analytical procedure employed is not an United States Environmental Protection Agency (USEPA) approved reference method. The gas chromatographic conditions used by Dr. Means were not made available because they are considered proprietary. As such, data provided by LSU could not be fully validated in accordance with the U. S. EPA guidance document, *National Functional Guidelines for Organic Data Review* (2). To the extent possible, data were evaluated and qualified. Volatile analyses were also performed, however, no quality assurance/quality control information was available for review.

The semivolatile results shown in Table 1 are displayed on a wet weight basis and contain qualifiers from the validator (person(s) in Terra reviewing the data supplied by LSU) based on the relative quality of the flagged data element when compared to the rest of the data set. This data set was considered as an isolated entity (from the other analytical data sets generated by independent parties for this project) since it was not comparable to data derived from any agency-approved methods from which acceptable performance statistics from multiple laboratories are available. The analytical results reviewed by Terra Consulting Group, Inc. (Terra) were derived from a spreadsheet printout of final answers which was provided by Dr. Means' laboratory. In many instances, multiple values for the same sample were generated by LSU, however, the values reported here were based on those results chosen by the laboratory.

This report describes some of the issues associated with reviewing the data generated by LSU and is organized in terms of the analytical elements typically examined during data validation. These elements are:

- holding times
- mass spectrometer performance check
- initial calibration
- continuing calibration
- system monitoring compounds (surrogates)
- matrix spike and matrix spike duplicates

- compound quantitation
- blank contamination
- system performance
- overall assessment of the data
- positive results
- negative results

Table 2 presents the results of volatile analyses conducted by Dr. Means' laboratory. These results have not been evaluated by Terra due to lack of the necessary quality assurance/quality control information. The data presented in this table reflect only a unit change from ug/kg to mg/kg and rounding to two significant figures. No conversion to a wet weight basis was necessary.

LSU Methodology

The sediment samples were analyzed with a special trace level method developed and used by Dr. Jay Means at the School of Veterinary Medicine at Louisiana State University (Baton Rouge) in the Department of Physiology, Pharmacology, and Toxicology (1,3). The method utilizes an internal standard extraction procedure followed by instrumental analysis with combined capillary gas chromatography and low resolution electron impact ionization selected ion monitoring mass spectrometry (EI-SIM) to detect and quantify up to 60 aromatic hydrocarbons at contaminant concentrations below one part per billion (ug/kg). The mass spectrometer was programmed to perform as a mass selective gas

chromatographic detector. No mass spectra were generated by Dr. Means' method. Further, the gas chromatographic program utilized by Dr. Means was not provided as it was considered proprietary. This was unfortunate since one of the criteria used to accurately identify the polynuclear aromatic hydrocarbons is based on the determination of relative retention time. In the absence of the parameters utilized by Dr. Means and without full mass spectra, qualitative confirmation of the compounds reported by LSU could not be performed.

In the extraction step, deuterated standards of the polycyclic compounds are added prior to the extraction process and act as whole method internal standards. A three day, three stage, sequential tumbling extraction was performed and the volume of extract was concentrated using rotary evaporation and subsequently reduced under a stream of nitrogen to a final volume of 0.2 ml (in some instances it was 0.4 ml). The method utilized an internal standard-to-analyte response ratio to compensate for inaccuracy introduced by the sample preparation process. A single instrumental internal standard was also used to monitor instrument variation independently of the preparation procedure. An external standard calibration method was used to calculate the weight detected and apparently the analyst had the option of applying a recovery ratio correction based on the internal standards carried through the method. This procedure complicated Terra's attempt to validate LSU's data. The criteria apparently used by LSU for applying the external standard calculation option was based on the recovery values being less than 10 percent. However, LSU also used external standards to calculate results when the recoveries where greater than 10 percent. Additional complications involved in verifying

the quantitation methodology were incurred as a result of the LSU quantitation practices. In some instances, it appears that the LSU laboratory used averages of external standard and internal standard results as well as averages of these averages.

It is important to note that although LSU uses common, EPA analytical terms in describing their methodology, the context in which they apply these terms is different. As a result, this may cause some confusion and misinterpretation by a data validator or reader on attempting to apply standard EPA analytical terminology to LSU's method. For example, the internal standards used throughout the entire method are termed as surrogates by the laboratory. The single instrumental internal standard is called an internal standard. The corrections for surrogate recovery are actually the equivalent of an internal standard correction for the entire method. The EPA internal standard correction for semivolatile extraction based methods is only applied as an instrumental correction. (The internal standard method works best when the amount of internal standard introduced into the instrument remains constant for all the analyses, including standards, samples and blanks.) Environmental samples often require dilution because of sample matrix or target compound concentration issues. Any sample dilutions that are required proportionally reduce the amount of internal standard introduced into the instrument. If large dilutions are required, the amount of internal standard introduced into the instrument becomes undetectable and cannot be used reliably as a proportional scale for the target compounds or compounds of interest.

Although the method utilized by LSU is very sensitive and has a higher degree of specificity than routine gas chromatographic methods, it has a lower degree of specificity

than the routine repetitive scanning gas chromatographic / mass spectrometric (GC/MS) methods specified by the EPA. Specifically, the LSU method does not have the ability to provide full scan mass spectra for compounds of interest. Consequently, confirmation of the presence or identity of the analytes detected by the LSU method by comparing the mass sample spectra to spectra of known reference materials was not possible. Methods utilizing GC/MS promulgated by the EPA have the capability to produce electron impact spectra for comparison to authentic references as well as to demonstrate the presence of interference contributions from relatively large, but non-target sample components. The method employed by LSU is not a suitable substitute for SW-846 GC/MS methods when proving the presence of contaminants. Although, the LSU method is more sensitive and can measure concentrations of the compounds of interest at lower concentrations than the EPA methods, it is not clear what value this provides in terms of data use by any regulatory authority for this environmental event.

The method has not been validated in a regulatory sense by different laboratories nor has it been promulgated by the EPA for general use. Further, there are not any rules or guidelines for data validation specifically developed for this method. Therefore, the data supplied by LSU was reviewed in terms of the method applied and the publications referenced by the author. Some spike recovery statistics were available from the literature referenced by LSU (3) and involved the spiking of twelve seawater samples, extraction with C18 SPE disks, extraction of the disks and analysis of the extracts. Whether or not an internal standard method was used with these seawater spikes was not clear. It was also unclear as to how to apply these statistics to the sediment sample results

produced by LSU. However, the high recoveries reported by LSU for naphthalene (351% and 287%) indicate the potential for significantly overstating the amount of naphthalene present in the samples the LSU lab analyzed.

Holding Times

All samples except one (5RDB) were extracted within 7 days of sample collection and the extracts were analyzed within 21 days of extraction, meeting the EPA recommended holding times for water and soil samples (1). Although sample 5RDB was documented in the extraction logbook to have been extracted on 6/12/96, the extraction date listed on the laboratory quantitation reports was actually 6/22/96. This sample was noted to contain the odor of gasoline and was determined to be the second highest level sample in terms of target analyte concentration. As such, the sample required dilution and subsequent reanalysis.

It is possible that the lab used up the extract from the initial 6/12/96 extraction by diluting and screening it for approximate concentration level prior to obtaining a final analysis and then prepared a fresh extract on 6/22/96. Since the sample contained higher than trace levels of gasoline, and the recommended 14 day holding time was not grossly exceeded, the results for sample 5RDB were not flagged or considered invalid due to extraction holding time.

Mass Spectrometer Instrument Check

The LSU laboratory performed a mass spectrometer tuning or tuning check using the instrument manufacturer's procedure (Hewlett Packard). This involved the gaseous introduction of PFTBA (perfluortributylamine) vapor for each analytical work shift. This technique will correct any problems which could have developed over time (such as mass axis drift) but will not document that such problems occurred. Mass axis drift may occur before the next tuning procedure is conducted. The length of time between each mass spectrometer tune (analytical work shift) was 16 hours. This was exceeded, however, for the sample extracts reported to have been analyzed on 6/14/96 and 6/15/96. Hard copy results of the tuning procedure containing a date and time stamp from the data system were provided and used for this determination. These show accurate mass identification and acceptable mass peak resolution. Relative abundances also appear stable but do not correspond to the same conditions as the sample analysis since the introduction of the tuning compound (PFTBA) was not made through the gas chromatographic column (as required by EPA methodologies). Stability of relative abundances indirectly contributes to the stability of responses to compounds introduced via the gas chromatograph. The stability of the chromatographic response can be directly demonstrated by monitoring the response factors or relative response factors created from the calibration standard analyses. The percent difference statistic measured from the initial calibration and each daily or continuing calibration would typically be used to demonstrate this stability. Unfortunately, the data supplied by LSU did not contain any initial calibration information so that the stability comparison could not be made.

The acceptable length of time between mass spectrometer stability checks specified by EPA methods is 12 hours. Accordingly, the acceptable procedure for data acquired once this time has been exceeded would be to recheck the instrument stability. This was not done for the sequence of analyses performed between 6/14/96 and 6/15/96. The automatic run sequence apparently terminated prematurely following the completion of the run that began at 1:56 a.m. on 6/15/96. The analytical sequence for the GC/MS was restarted without performing a "fresh" tuning check to determine the stability of the instrument. The next sample analysis was begun approximately 13 hours later at 3:10 p.m. A calibration standard was analyzed which was date and time stamped for 9:18 a.m. for the next day. The 16 hour analytical work shift ended at 10:26 am. The particular mass spectrometer used by LSU is physically a small model and may be susceptible to problems associated with thermal drift. The impact of drift that may have occurred on the quantitative aspect of the analyses could have been shown by comparing the compound responses of the standard run at the beginning of the sample sequence to the standard run at the end of the sample sequence. Unfortunately, there was no standard analyzed for the 6/15/96 run sequence so this comparison could not be performed for all of the samples. The samples that may be affected are 4RDB, 4MS, 2MS, 1MS and 5LDB.

Initial Calibration

A multilevel initial calibration was not provided with the data package. Usually, a three-point calibration or a five-point calibration curve is generated by laboratories under the

same conditions as the sample analyses. This establishes a working range of concentrations and becomes the documented range of the method for the samples in the data package. For the samples analyzed by LSU, no range was demonstrated. The GC/MS instrument has an upper response limit. Near and above this limit, an increase in the amount of compound does not produce a corresponding increase in the response. This limit is often difficult to define and cannot be pinpointed accurately because the response falls off progressively over a small range of amounts rather than at a single defined value. In most environmental analytical efforts or studies, the highest concentration level in the multilevel calibration is typically used to approximate the upper calibration limit. The internal standard responses fall between extremes of the calibration curve so that the responses may vary in either direction to correctly compensate for variations in sample preparation. Though the internal standards are kept at a constant weight for the multilevel calibration, the weights of their non-deuterated analogs are increased from near the detection limit to the upper limit where the response ceases to increase as weight or concentration increases. The response to a given weight of non-deuterated analog is taken to be the same as the response to the deuterated internal standard compound so that the multilevel calibration curve demonstrates that the response levels are away from both ends of the calibration range. Though the size of the calibration range is addressed in one or more publications cited by LSU, these do not provide the range of the responses so that the use of an acceptable internal standard level can be easily verified.

Continuing Calibration

Single concentration level standards were analyzed with each 16 hour data acquisition batch. Sequence logs provided with each small batch of raw sample data indicates that there was an intent to analyze a standard before and after samples were analyzed. Typically, data validation procedure requires at least some spot check calculations by the validator reviewing the data. The data submitted by LSU to Terra for validation and review was checked by recalculating the results from the raw data area counts. The calibration standard used for calculating response and ultimately sample concentrations was time stamped and demonstrated to be analyzed in the middle of the sample run sequence and not at the end as recorded on the data system sequence log and the Instrument Logbook. In fact, there appears to be a 13 hour gap in the analysis of the sediment extracts between vial 7 and vial 8 in the 12 vial analytical sequence during which the standard actually used for calibration was analyzed. The filename of this standard differs from the name shown on the sequence log by one letter but corresponds to the instrument log filename. The filename used by LSU is comprised of a prefix and a suffix combination, where only the suffix was shown for the samples following the first injection. The Instrument Log provided by LSU does not contain the time at which the injections are made. The samples appear to be quantitated using calibration standard values obtained, more or less, with the sample analyses rather than using values from an initial calibration (of which there is no evidence that one was performed).

System Monitoring Compounds (Surrogates)

The compounds that were identified as surrogates by the LSU laboratory were not measured using the preferred method of quantitation which utilizes an internal standard for the determination because they were the internal standards. EPA methods require that the surrogates are quantitated using internal standards which are, in turn, used to quantitate target compounds. As such, the recovery behavior demonstrated by the surrogates is used to represent the recovery of the target analytes. The LSU laboratory used an external standard method to quantitate the internal standard compounds which it actually labels as surrogates. If the amount found was greater than 10 percent of the theoretical amount that should have been found (and the laboratory chose to follow its method), the laboratory used the surrogate recoveries to correct the target analyte amounts. When used to correct the target analyte amounts, the surrogates become, in function, internal standards. The compounds designated by the LSU laboratory as surrogates are the same compounds specified and used by USPEA methods as internal standards: d8-Naphthalene, d10-Acenaphthene, d10-Phenanthrene, d12-Chrysene and d12-Perylene.

The laboratory provided a table of surrogate recoveries in the spreadsheet printout of SEDWRKS.XLS. The recovery values ranged from 13% to 182%.

There are no pre-defined quality control (QC) limits which these values must meet for the data to be considered acceptable. Usually, the target compound data is derived from the

internal standard calculations. The recovery data presented by LSU were derived entirely from external standard calculations, therefore a meaningful comparison was not possible.

Matrix Spikes / Matrix Spike Duplicates

Please note that the samples labeled with the suffix "MS" in the sample data provided by LSU actually means Mid-Stream and not Matrix Spike. The laboratory spiked the samples with all of the analytes and presented the results in the same spreadsheet format as the sample results. Additional columns for percent difference and percent recovery were also conveniently provided. Sample 3 MS was chosen by LSU for spiking. A duplicate analysis of sample 3 MS was also performed. This was a second analysis of the unspiked sample and was not an analysis of a second spiked sample; therefore, a matrix spike duplicate was not analyzed. The reported spike recoveries were:

- 302% for Naphthalene,
- 4227% for 2-Methyl-naphthalene, and
- 2141% for 1-Methylnaphthalene.

The percent difference values calculated from the duplicate analyses were:

- 7% for Naphthalene,
- 32% for 2-Methylnaphthalene, and

- 30% for 1-Methylnaphthalene.

The recovery results demonstrate that the laboratory reported:

- 3 times the true amount added of Naphthalene,
- 42 times the true amount added of 2-Methylnaphthalene, and
- 21 times the true amount added of 1-Methyl-naphthalene.

The laboratory did not explicitly state the amount spiked into the samples. The same uniform spike level of each target compound was not used. Based on the review of the data supplied by LSU, the spiking levels used by LSU were determined to be on the order of 3 to 20 ug/kg (ppb). These amounts were less than the measured sample concentrations. Consequently, the levels used by LSU were inappropriate for the analyses that were performed. As a result, deficiencies in their analytical precision appear as gross inaccuracies. The magnitude of the spike results reported by LSU appears to indicate that the lab may have a severe problem in some aspect of sample handling. It may be possible that the problem was an isolated occurrence and does not affect all of the results.

Compound Quantitation

Positive values from three samples were qualified "J" as estimated values because the external standard method was used (possibly due to dilution). The internal standard method could have been used without violating the lab's "10 percent rule" but was not.

The internal standard method gives values from two to six times those reported by the laboratory. These discrepancies were considered significant enough to warrant flagging or qualifying the associated values.

The LSU laboratory originally reported all the sample results on a dry weight basis. These were subsequently converted to wet weight values by the validator using the percent moisture values provided by the laboratory. Although this appears to change the numerical values that were originally reported by LSU, it allows one to compare the sediment data from different laboratories.

Blanks

Blanks were processed through the sample preparation process and analyzed with the samples. The blank results were reported with small amounts of the compounds of interest. A value of five times the blank level was used as a delimiter of significance (according to EPA Functional Guidelines for Data Validation). Results for samples associated with contaminated blanks reported at or below these calculated levels are considered to be indistinguishable from the blank level caused by analytical system contamination. For three of the samples in this set, 1MS, 1LDB and 2LDB, the numerical results were qualified with a "U" to show that the values were not high enough above the apparent analytical system contamination to be reported in the sample. In this context, the analytical system was determined to be the entire set of sample processing equipment

and reagents including solvents, rubber stoppers, tumbling jars, and rotary evaporators and was not limited solely to the analytical instrument.

System Performance

The overall analytical system utilized by LSU appears to be in a developmental stage. The system is not suitable for establishing the presence of target analytes in the environment. Theoretically, it is suitable for academic situations in which the limits of analyte amounts and interference amounts are known before the method is employed.

The system utilized by LSU is apparently intended for trace level, part per trillion (ng/kg), residue analyses with quantitation limits at or below 1 part per billion (ug/kg). Samples from this project submitted for analysis to LSU were demonstrated to contain compound concentrations above the parts per billion range. Consequently, the method employed by Dr. Jay Means at LSU, was inappropriate as indicated by the apparent contamination of the LSU laboratory's sample processing equipment and/or other aspects of their analytical system. As a result, values that were reported are false positives or were inaccurately biased on the "high" side.

Overall Assessment of Data

The data generated should not be used to demonstrate compliance with any regulatory limit at any particular geographical location. Any utilization of the data should be

performed with caution and discretion. The sample handling problem demonstrated by the spike may have involved inadvertent problems with sample identification.

Positive Results

The data reported by LSU were very prone to contain values which were higher than what may actually be in the sample. The positive results should only be used if an undefined and positive bias can be tolerated or in conjunction with other data generated from methods generally accepted or recommended by the EPA. Mass spectra are not available and were not subsequently produced by LSU for the positive results that were reported. Consequently, a positive result may be totally false and cannot withstand a data challenge.

Negative Results

Some of the negative results reported by the LSU laboratory may be useable in a limited extent. For instance, if the negative results for samples 4RDB, 4MS, 2MS, 1MS and 5LDB are to be used, they should be interpreted to possess a detection limit 25 times the stated method detection limit. This multiplier would be based on the uncertainties associated with the compensation necessary for any possible mass axis drift (i.e., low recovery on last internal standard) and the uncertainty guidance provided by the EPA for blank contamination.

References

1) McMillin, D.J. and Means, J. C., 1995. Development and Application of (parts per trillion) GC/MS analysis method for Approximately 60 Aromatic Hydrocarbons. In: Winston, G. W. and Means, J. C., Bioavailability and Genotoxicity of Produced Water Discharges Associated with Offshore Production Operations. OCS Study MMS 95-0020.

2) EPA, 1994. USEPA Contract Laboratory Program, National Functional Guidelines for Organic Data Review. EPA540/R-94/012.

3) McMillin, D.J. and Means, J. C., 1995. Proposal to Marathon Pipe Line Company for Analysis of Volatile and Semi-volatile Hydrocarbons in Biological Tissues and Sediment Cores from the Blind Bayou Gasoline Spill Site.

Table H1

Semivolatile Analyses of Sediment Samples by LSU Trace Level Method

Client Sample	Lab Sample	Initial Wt. (g.)	% M	Date Sampled	Date Extracted	Date Analyzed	MQL (mg/kg) *	Naphthalene (mg / kg) *	2-Methyl-Naphthalene (mg / kg) *	1-Methyl-Naphthalene (mg / kg) *
1MS	6166B10	15.90	80	6/5/96	6/10/96	6/15/96	0.00012	0.0016 UB	0.0020	0.0012
1LDB	6177B14	15.10	53	6/5/96	6/12/96	6/26/96	0.00007	0.0011 UB	0.0018	0.00094
1RDB	6177B10	15.18	46	6/5/96	6/12/96	6/26/96	0.00007	0.0034	0.012	0.0078
2MS	6166B09	15.63	79	6/5/96	6/10/96	6/15/96	0.00006	0.0027	0.0023	0.0014
2LDB	6177C18	15.68	59	6/5/96	6/12/96	6/26/96	0.00007	0.0012 UB	0.00086	0.00070
2RDB	6177C17	15.45	57	6/5/96	6/12/96	6/26/96	0.00006	0.0034	0.0060	0.00420
3MS	6166B04	15.14	77	6/5/96	6/10/96	6/14/96	0.00007	0.0044	0.022	0.011
3LDB	6177C21	15.85	73	6/5/96	6/10/96	6/26/96	0.00006	0.0025	0.0043	0.0032
3RDB	6166B06	15.80	55	6/5/96	6/10/96	6/15/96	0.00006	0.0063	0.012	0.0072
4MS	6166B08	15.51	78	6/5/96	6/10/96	6/15/96	0.00007	0.011	0.096	0.051
4LDB	6177C19	15.34	62	6/5/96	6/10/96	6/26/96	0.00006	0.0049	0.016	0.013
4RDB	6166B07	16.00	56	6/5/96	6/10/96	6/15/96	0.00006	0.010	0.043	0.036
5MS	6177B12	15.53	77	6/5/96	6/12/96	6/26/96	0.00006	0.024	0.13	0.072
5LDB	6166B11	15.29	82	6/5/96	6/10/96	6/15/96	0.00013	0.035	0.25	0.16
5RDB	6183D15	15.22	87	6/5/96	6/12/96	6/27/96	0.00039	4.4	5.8 J	3.6 J
6LDB	6183D13	15.45	86	6/5/96	6/12/96	6/27/96	0.00097	9.7 J	17 J	9.7 J
6MS	6183D14	15.45	85	6/5/96	6/12/96	7/2/96	0.00060	1.8	1.4 J	0.82 J
7MS	6177B13	15.71	77	6/5/96	6/12/96	6/26/96	0.00008	0.0037	0.018	0.0097
7LDB	6177B11	15.91	52	6/5/96	6/12/96	6/26/96	0.00006	0.0053	0.0082	0.0047
7RDB	6177B15	15.64	55	6/5/96	6/12/96	6/26/96	0.00006	0.0050	0.0081	0.0050
6/10 Blank	6166B02	15.00	0		6/12/96	6/14/96	0.00007	0.00045	0.00015	0.00008

* These values are converted to wet weight from dry weight values provided by the laboratory for each sample.
J = Estimated value due to low precision of available results.
UB = The detected amount is not significantly higher than the blank and should be considered not detected at the numerical value shown. Amounts less than or equal to 5 times the blank value are not significantly higher than the blank.

Table H2

Volatile Aromatics Analyses of Sediment Samples by LSU

Client Sample	Date Sampled	MQL * (mg / kg)	Benzene (mg / kg)	Toluene (mg / kg)	Ethylbenzene (mg / kg)	m/p-Xylene (mg / kg)	o-Xylene + Styrene (mg / kg)	Isopropyl-benzene (mg / kg)	Propyl benzene (mg / kg)	1,3,5-Trimethyl-benzene (mg / kg)	tert-Butyl benzene (mg / kg)	1,2,4-Trimethyl-benzene (mg / kg)	sec-Butyl-benzene (mg / kg)	p-Isopropyl-toluene (mg / kg)	n-Butyl-benzene (mg / kg)	Naphthalene (mg / kg)
1MS	6/5/96	0 001 U	0 001 U	0 001 U	0.003 UB	0 014 UB	0 005	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 004 UB	0 001 U
1LDB	6/5/96	0 001 U	0 001 U	0 001 U	0 003 UB	0 011 UB	0 003	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 002 UB	0 003 UB
1RDB	6/5/96	0 001 U	0 001 U	0 001 U	0 003 UB	0 012 UB	0 004	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 002 UB	0 003 UB
2MS	6/5/96	0 001 U	0 001 U	0 001 U	0 003 UB	0 012 UB	0 004	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 002 UB	0 001 U
2LDB	6/5/96	0 001 U	0 001 U	0 001 U	0 001 UB	0 008 UB	0 002	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 003	0 001 U	0 002 UB	0 009 UB
2RDB	6/5/96	0 001 U	0 001 U	0 001 U	0 004 UB	0 017 UB	0 005	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 UB	0 001 U
3MS	6/5/96	0 001 U	0 001	0 001 U	0 001 UB	0 005 UB	0 002	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 003 UB	0 001 U
3LDB	6/5/96	0 001 U	0 005	0 001 U	0 003 UB	0 012 UB	0 005	0 001 U	0 003	0 026 UB	0 005	0 001 U	0 001 U	0 002	0 005 UB	0 010 UB
3RDB	6/5/96	0 001 U	0 001	0 001 U	0 003 UB	0 014 UB	0 004	0 004	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 003 UB	0 001 U
4MS	6/5/96	0 001 U	0 006	0 001 U	0 002 UB	0 011 UB	0 005	0 001 U	0 002	0 037 UB	0 014	0 036	0 001 U	0 003	0 023	0 012 UB
4LDB	6/5/96	0 001 U	0 001 U	0 001 U	0 001 UB	0 006 UB	0 002	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 001 U	0 004 UB	0 001 U
4RDB	6/5/96	0 001 U	0 001 U	0 001 U	0 002 UB	0 01 UB	0 004	0 001 U	0 003	0 013 UB	0 008	0 013	0 006	0 004	0 006 UB	0 008 UB
5MS	6/5/96	0 001 U	0 000	0 002 UB	0 008 UB	0 029 UB	0 015	0 003	0 015	0 055 UB	0 054	0 120	0 010	0 012	0 098	0 013 UB
5LDB	6/5/96	0 001 U	0 27	0 36	0 096	0 35	0 20	0 005	0 017	0 070	0 34	0 049	0 004	0 004	0 032	0 002 UB
5RDB	6/5/96	0 001 U	0 063	0 061	0 017	0 072	0 048	0 003	0 018	0 26	0 14	0 33	0 010	0 019	0 27	0 056
6LDB	6/5/96	0 001 U	1 6	7 8	7 9	30	16	3 2	10	81	41	55	4 9	5 4	2 3	12
6MS	6/5/96	0 001 U	0 029	0 079	0 046	0 20	0 13	0 035	0 10	1 1	0 41	0 87	0 44	0 60	0 52	0 21
7MS	6/5/96	0 001 U	0 002	0 001 U	0 002 UB	0 006 UB	0 003	0 001 U	0 001 U	0 002 UB	0 004	0 001 U	0 001 U	0 001 U	0 003 UB	0 012 UB
7LDB	6/5/96	0 001 U	0 003	0 003 UB	0 002 UB	0 007 UB	0 003	0 001 U	0 001 U	0 001 U	0 003	0 001 U	0 001 U	0 001 U	0 006 UB	0 005 UB
7RDB	6/5/96	0 001 U	0 002	0 001 UB	0 003 UB	0 013 UB	0 005	0 001 U	0 002	0 003 UB	0 004	0 001 U	0 003	0 002	0 004 UB	0 012 UB
Blank		0 001 U	0 001 U	0 002	0 002	0 010	0 003	0 001 U	0 001 U	0 014	0 001 U	0 001 U	0 001 U	0 001 U	0 003	0 004

All values are converted to mg / kg (ppm) from the ng / g (ppb) used by the laboratory.
The laboratory's results have not been verified and QC results such as spike recoveries are not present.
U = Not detected at or above the concentration level shown
UB = The laboratory reported a positive instrument reading but the the contaminant is not detected in the sample below the level shown due to the amount found in the blank
The MQL of 0 001 mg / kg is assumed based on the lowest reported value for any analyte The laboratory did not report MQL values for these samples