

FINAL

**INTERIM ASSESSMENT OF THE PRESENCE AND
CAUSES OF AMBIENT WATER AND SEDIMENT
TOXICITY IN VINCE BAYOU, SEGMENT 1007A**

Prepared For

TOTAL MAXIMUM DAILY LOAD PROGRAM

**TEXAS NATURAL RESOURCE CONSERVATION COMMISSION
P.O. BOX 13087, MC - 150
AUSTIN, TEXAS 78711-3087**

Prepared By

PARSONS

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FEBRUARY 2003

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PREPARED IN COOPERATION WITH THE TEXAS COMMISSION ON
ENVIRONMENTAL QUALITY AND THE U.S. ENVIRONMENTAL PROTECTION AGENCY

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EXECUTIVE SUMMARY Vince Bayou Segment 1007A (Toxicity in Sediment)

The Texas Commission on Environmental Quality (TCEQ) is responsible for administering provisions of the constitution and laws of the State of Texas to promote judicious use and the protection of the quality of waters in the State. A major aspect of this responsibility is the continuous monitoring and assessment of water quality to evaluate compliance with state water quality standards which are established within Texas Water Code, '26.023 and Title 30 Texas Administrative Code, '307.1-307.10. Texas Surface Water Quality Standards 30 TAC 370.4(d) specify that surface waters will not be toxic to aquatic life. Pursuant to the federal Clean Water Act '303(d), states must establish Total Maximum Daily Loads (TMDLs) for pollutants contributing to violations of water quality standards. The purpose of this TMDL Study was to assess the presence and causes of ambient toxicity in seven Texas waterbodies listed on the Draft 2000 Federal Clean Water Act (CWA) '303(d) List in an effort to comply with Texas law.

In order to assess the waterbodies, this study provided goals as follows:

- Confirmation that toxicity is present more than 10% of the time, through the collection of up to date toxicity testing.
- The identification of the substance(s) or factors causing the toxicity where present.
- The identification of the sources of the toxicant(s).
- Confirmation, via chemical analysis, that water quality standards are being maintained.

This study was limited to the following seven waterbodies of concern:

1. Alligator Bayou (Segment 0702A) in Jefferson County (toxicity in water and sediment),
2. Bryan Municipal Lake (Segment 1209A) in Brazos County (toxicity in sediment),
3. Finfeather Lake (Segment 1209B) in Brazos County (toxicity in sediment),
4. Vince Bayou (Segment 1007A) in Harris County (toxicity in sediment),
5. Arroyo Colorado Tidal (Segment 2201) in Cameron County (toxicity in sediment),
6. Rio Grande (Segment 2304) in Kinney, Maverick, and Webb Counties (toxicity in water), and
7. Rio Grande (Segment 2306) in Presidio County (toxicity in water).

The TCEQ selected Parsons to conduct a more thorough and intensive assessment of the existence of toxicity and identification of likely toxicants in the waterbodies. The Texas Surface Water Quality Standards specify that surface waters will not be toxic to aquatic life. Pursuant to the federal Clean Water Act §303(d), States must establish total maximum daily loads (TMDLs) for pollutants contributing to violations of surface water quality standards. Ambient toxicity testing complements routine chemical monitoring to identify waterbodies with aquatic life impairment. The waterbody assessments are each described in six different reports. Finfeather Lake and Bryan Municipal Lake are described in the same report due to their close proximity and likely cause.

Vince Bayou has been included by TCEQ in the state's 303(d) list based on sediment toxicity tests results for the assessment period from 1991 to 1996. No water toxicity was documented in any of 18 samples collected from the bayou. Vince Bayou is a tributary to the Houston Ship Channel (Segment 1007) and designated 1007A. Potential chemicals of concern in sediments identified for Segment 1007 and tributaries include copper, lead, mercury, and zinc, and dioxin for fish/shellfish consumption, based on the draft 2000 TCEQ 303(d) list.

Three of the seven stations established by TCEQ (Stations 11299, 14368, 14371), were selected for monitoring in this assessment of Vince Bayou. Monitoring included field measurements of water quality, and collection of sediments for chemical analyses and toxicity testing. Sediment samples collected April 18 and May 24, 2001 from Station 14368 were significantly toxic to both the *Neanthes* and *Leptocheirus* surrogate species using Whole Sediment Test methods. Following the May 24 sampling event, a Toxicity Identification Evaluation (TIE) was initiated at Station 14368.

Sediment collected from Station 11299 no longer produced toxicity after August 10, 1993. The three sediment samples collected at Station 11299 on and before August 10, 1993 exhibited toxicity using EPA's elutriate test method to *Cyprinodon variegatus*. The six sediment samples collected after August 10, 1993, including those collected by Parsons, did not exhibit toxicity using either the elutriate or whole sediment test methods.

Vince Bayou Sediment Toxicity Test Results

Vince Bayou 1007A		% Survival	
		Neanthes	Leptocheirus
April 18-19, 2001	Control	100	99
	11299	100	96
	14368	96	7
	14368-Dup1*	28	2
	14371	92	85
May 24, 2001	Control	96	96
	11299	96	96
	14368	32	13
	14371	92	92
June 14, 2001	Control	92	95
	11299		
	14368	92	1
	14371		
July 19 & 26, 2001	Control	100	98
	11299	100	96
	14368	92	5
October 30, 2001	Control	100	98
	14368	100	5

January 9, 2002	Control		99
	14368		32
	11301		44
	11171		94
	E. Jackson		94
April 3, 2002	Control	100	100
	14368	100	86**
April 23, 2002	Control	100	100
	14368	100	92
	11301	100	92
	11171	88	90
	E. Jackson	100	98
May 29, 2002	Control	100	100
	14368	100	99

Shaded cell - denotes exceedance of recommended criteria; * - collected in approximately the same location (for quality control purposes)

** - significantly different, but not toxic according to recommended criteria

NA – Not Analyzed

Summary of Sediment Toxicity Test Results

Station	Lethal Neanthes	Lethal Leptocheirus
12999	0/3	0/3
14368	1/8	6/10
14371	0/2	0/2
11301	0/1	1/2
11171	0/1	0/2

Toxicant identification for Station 14368 sediments showed a SPE-extraction as marginally effective in reducing toxicity. The results were inconclusive as the treatment was effective in only one out of five tests conducted with two tests species. An additional procedure was subsequently employed, passing the pore water through the polymeric adsorbent resin Amberlite XAD-4. In two separate test procedures this treatment effectively removed toxicity suggesting that organics (possibly petroleum hydrocarbons) are possible contaminants. This conclusion is supported by the detection of several PAHs in the sediment at concentrations well above toxicity screening criteria.

Phase 1 TIE tests were performed to determine some physical characteristics of the toxicant in samples from Station 14368. During these tests, it was determined that toxicity was removed from the porewaters by adjusting the pH to 3.0 and sparging the porewater samples with air. The next approach to the TIE was to try to capture the sparged gas fraction

from pH 3.0 adjusted porewater onto a charcoal cartridge and in methanol. In addition, an attempt to move the toxic fraction, via sparged gas, to control water and recover the toxic parameter(s) were made. Neither the charcoal or methanol fractions revealed any significant results. Several volatile traps were used, none of which detected any compounds of interest.

All of the TIE work was performed on sediment samples that were collected prior to April 2002. It was discovered that sometime after the January 2002 sampling event, Stations 14368 and 11301, which had previously and consistently shown toxicity, were now not toxic for either *Leptocheirus* or *Neanthes*. For the April 3, 2002 sampling event, Station 14368 was only slightly toxic for *Leptocheirus* and not toxic at all for *Neanthes*. Since this sampling event, no significant differences from the control in percent survival were observed. In May and June of 2002, several attempts were made by Parsons sampling crew to identify areas of similar looking sediments and collect samples both upstream and downstream from the previously toxic areas, without success.

The TIE procedure identified caprolactam, caprolactam-related products, and an unknown pore-water toxicant that combined with caprolactam to produce the toxicity. Our interpretation of the results of the TIE procedures which identified caprolactam is that there is evidence that caprolactam-related substances are contributing to the toxicity observed in the pore water. We also have evidence that the increased toxicity of caprolactam seen in association with the cleaned-up pore water is not dependent upon the direct action of the pore water on the caprolactam (i.e. inducing ring opening or polymerization) since the increased toxicity of caprolactam can be induced by independent exposures to cleaned-up pore water (non-toxic by itself) and caprolactam in clean seawater (non-toxic by itself; see the results of the dual exposure experiment).

Caprolactam is primarily used in the manufacture of Nylon 6 and other synthetic fibers. Caprolactam is also used in brush bristles, textile stiffeners, film coatings, synthetic leather, plastics, plasticizers, paint vehicles, cross-linking for polyurethanes, and in the synthesis of lysine (USEPA 1988, USDHHS 1993).

Parsons' recommendation is continued periodic monitoring of sediment toxicity. In addition, effluent and sludge sampling should be performed on potential sources followed by the development of a TMDL for caprolactam.

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LIST OF ACRONYMS

Cfs	Cubic feet per second
CRP	Clean Rivers Program
CWA	Clean Water Act
DQO	Data quality objectives
FM	Farm to market
Km	Kilometer
LCS	Laboratory control standards
m	Meter
mg/L	Milligrams per liter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
QAO	Quality assurance officer
QAPP	Quality assurance projected plan
QC	Quality control
SSI	Screening site inspection
SWQM	Surface water quality manual
TAC	Texas Administrative Code
TIE	Toxicity identification evaluation
TMDL	Total maximum daily load
TCEQ	Texas Commission on Environmental Quality
TNRCC	Texas Natural Resources Conservation Commission
USDHHS	U.S. Department of Health and Human Services
USEPA	United States Environmental Protection Agency
USGS	United States Geologic Survey
VB	Vince Bayou
WWTP	Wastewater treatment plant

SECTION 1 INTRODUCTION

The federal Clean Water Act (CWA), §305(b), requires states to produce a periodic inventory comparing water quality conditions to established water quality standards for surface waters. Standards for the State of Texas are specified in Texas Water Code, §26.023 and Title 30 Texas Administrative Code (TAC) §§307.1-307.10. Texas Surface Water Quality Standards 30 TAC 307.4(d) specify that surface waters will not be toxic to aquatic life. Pursuant to the federal CWA §303(d), states must establish total maximum daily loads (TMDL) for pollutants contributing to violations of water quality standards.

1.1 BACKGROUND INFORMATION

Segment 1007A Vince Bayou is identified on the State of Texas 1999 and draft 2000, 303(d) lists as partially supporting aquatic life due to ambient sediment toxicity. Vince Bayou is a tidal tributary to the Houston Ship Channel/Buffalo Bayou (Segment 1007), located in Harris County, Texas. The bayou receives discharges from municipal and industrial facilities plus non-point source runoff.

As shown in the maps in Figure 1.1 and Figure 1.2, Segment 1007A of the San Jacinto River Basin is located in Harris County, Texas in the City of Pasadena. The bayou is located in southeast Harris County and runs northwest, through Pasadena, for approximately 9 miles to its mouth on Buffalo Bayou.

The purpose of this assessment is to verify the presence of toxicity in sediments of Vince Bayou and its tributaries and, if toxicity is found, determine its cause(s) and source(s) in the bayou.

1.2 DESCRIPTION OF THE SAMPLING STATIONS

The TCEQ has established seven historic sampling stations on Vince Bayou. The sampling station descriptions are as follows:

- 11299: Vince Bayou 300 yards upstream of the Houston Ship Channel Confluence
- 11300: Vince Bayou at North Richey Street in Pasadena, TX
- 14368: Vince Bayou downstream of the City of Pasadena WWTP Outfall, 33 feet downstream of West Richey Street
- 14369: Vince Bayou at West Harris Avenue in Pasadena
- 14370: Vince Bayou at South Shaver Street in Pasadena, TX
- 14371: Little Vince Bayou at West Richey Street in Pasadena, TX

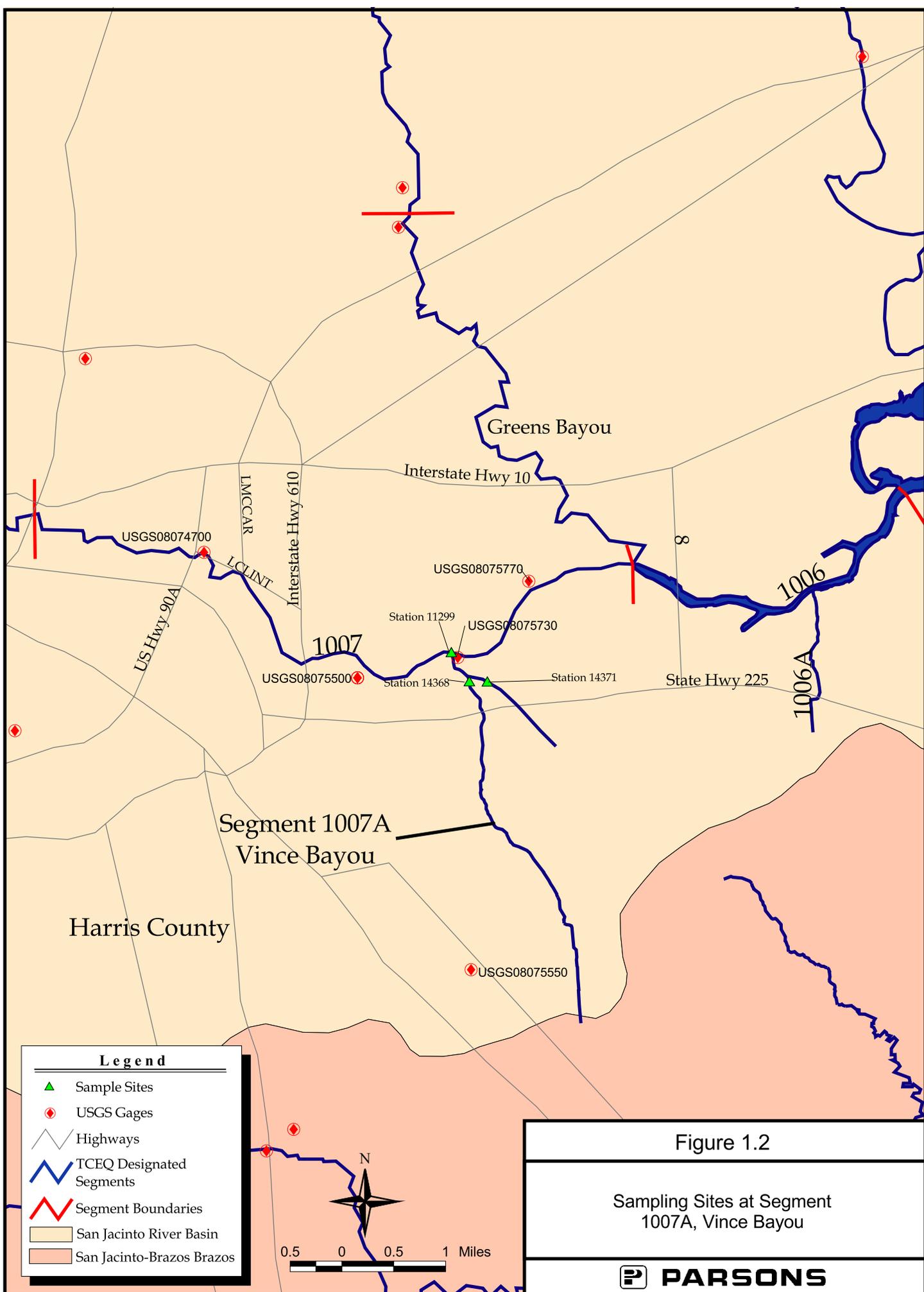


2000 0 2000 Feet

Figure 1.1

Dischargers and Sampling Stations
In Vince Bayou





Three of the seven stations established by TCEQ on Vince Bayou, Stations 11299, 14368 and 14371, were selected for monitoring in this assessment. Criteria used to select stations for this investigation were: 1) The station must be a TCEQ station for which past monitoring data are available; 2) Past monitoring by TCEQ has indicated water quality impairment at the station; and 3) Pollutant loading is known or suspected near the station.

SECTION 2 PROBLEM DEFINITION

2.1 VINCE BAYOU 303(D) LISTING

Vince Bayou was included by TCEQ in the state's 303(d) list based on three sediment toxicity tests results for the assessment period from 1991 to 1996. Elutriate from sediment samples collected from station 11299 in October 1992, April 1993, and August 1993 were toxic to the test species *Cyprinodon variegatus* (*C. variegatus*). No toxicity was observed in five sediment samples subsequently taken from the same station, nor in single samples from six other locations (Stations SS18, 11299, 11300, 14368, 14369, 14370, 14371). No water toxicity was documented in any of 18 samples collected from the bayou. Table 2.1 summarizes the basis for inclusion of Vince Bayou in the §303(d) list. Table 2.2 provides the historical toxicity tests results. Appendix A presents the historical chemical analysis data.

Guidance developed by TCEQ for Texas Surface and Drinking Water Quality Data, requires that data used to evaluate waterbodies for 303(d) listing and TMDL development not be more than 5 years old. Therefore, tasks within this assessment include collection of additional water and sediment samples to confirm the toxicity; if toxic, at what location(s). Then determine the cause and the source of the toxicity. Results of the analysis will determine whether to proceed with TMDL development or establish the basis for removing the bayou from the 303(d) list.

The historical sediment toxicity tests were performed by the U.S. Environmental Protection Agency (USEPA) laboratory in Houston using the sediment elutriate test. This test requires mixing the sediment in lab water for a specified period of time, then letting the sediment settle. The toxicity test is performed on the supernatant. It is believed that this test maximizes the amount of potentially toxic dissolved compounds in the supernatant and may overstate the actual whole sediment toxicity to endemic benthic organisms. In addition, measured water column concentrations may also be overstated due to the elutriate procedures.

2.2 CHEMICALS OF CONCERN

Table 2.3 lists historical data for sediment chemistry at station 11299 from 1995 to 2000. The data indicate that average concentrations of copper, lead, mercury, and zinc were higher than the screening criteria. Three other metals (arsenic, cadmium, and nickel) also exceeded the criteria at maximum concentrations. Sediments were reported as predominantly sandy (68 percent), with an elevated content of total solids (56 percent) and total organic carbon (20g per kg dry weight).

Elevated nutrients and dissolved oxygen have been eliminated as possible concerns because additional data do not indicate adverse water quality impacts associated with nutrients. The Texas Department of Health's (TDH) fish/shellfish consumption advisory related to dioxins applies to this segment. This section of Vince Bayou is saline in nature due to the influence of segment 1007.

**Table 2.1
Historical Toxicity Tests Results Justifying 303(d) Listing for Vince Bayou**

Species	Number of Tests*	Exhibits Primary Toxicity	Exhibits Secondary Toxicity	Total Exhibiting Toxicity	Total % Toxic
<u>Cyprinodon variegatus</u>					
Water Toxicity	16	0	NP	0	0
Sediment Toxicity	14	3	NP	3	21
Total	30	3	NP	3	

NP = Not Performed

* Samples were collected from 18 sampling events that occurred between November 1992 and November 1996

**Table 2.2
Historical Sediment Toxicity Results**

Vince Bayou 1007		% Survival
		Cyprinodon Variegatus
June 19, 1997	Control	100
	ss18	100
October 24, 1995	Control	93
	11299	87
April 19, 1995	Control	100
	11299	93
October 11, 1994	Control	90
	11299	87
April 22, 1994	Control	93
	11299	83
October 15, 1993	Control	97
	11299	87
August 10, 1993	Control	90
	14370	97
	11300	90
	14369	100
	14371	93
	11299	73
	14368	97
April 7, 1993	Control	93
	11299	0
October 14, 1992	Control	93
	11299	0

Bold - denotes significant difference from the control

Table 2.3
Vince Bayou
Historical Sediment Chemistry Detections

PARAMETER	Historical Average*	Historical Minimum*	Historical Maximum*	Lowest Screening Criteria**	UNITS
METALS IN BOTTOM DEPOSITS					
(mg/kg dry wt.)					
Aluminum	15285	6370	24200		mg/kg
Arsenic	4.9	1.6	8.2	7.24	mg/kg
Barium	162	66	257		mg/kg
Cadmium, Total	1	0.2	1.87	0.68	mg/kg
Chromium, Total	31.2	17.0	45.3	52.3	mg/kg
Copper	41	20	61	18.7	mg/kg
Lead	143	54	231	30.2	mg/kg
Manganese	150	103	197		mg/kg
Mercury, Total	0.5	0.2	0.86	0.13	mg/kg
Nickel	13.8	6.4	21.2	15.9	mg/kg
Silver	2	4.0	ND		mg/kg
Zinc	203	73	333	124	mg/kg
SEDIMENT COMPOSITION					
(percent dry weight)					
Clay, particle size < 0.0039 mm	23	14	32		%
Silt, particle size 0.0039 to 0.0625	9	7	10		%
Sand, particle size 0.0625 to 2 mm	68	60	75		%
SOLIDS IN SEDIMENT					
Total Solids (percent by dry weight)	56	49	63		%
Total Organic Carbon (mg/kg dry wt.)	20400	17800	23000		mg/Kg

Notes:

* TNRCC database information for station 11299 for the period April 19, 1995 to January 1, 1999.

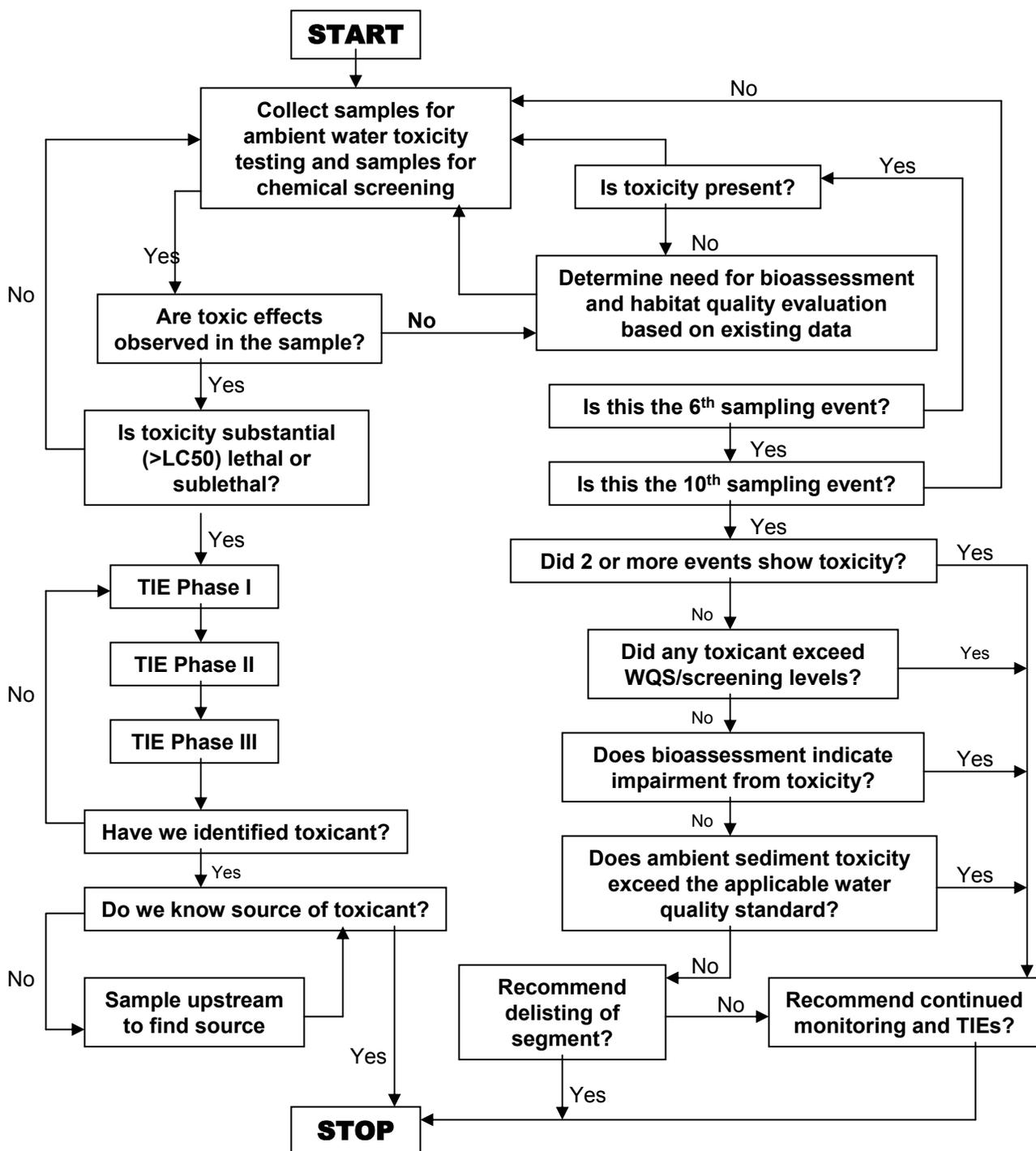
** Criteria is from *Equilibrium and Non-Equilibrium Partitioning-Based Sediment Quality Screening Indices* tables. The basis for criteria selection is presented in Appendix G

Shading represents results which are above the Lowest Screening Criteria.

SECTION 3 ASSESSMENT OBJECTIVE, SCOPE AND STRATEGY

The objective of this assessment is one part of the larger objective of establishing fully supported designated uses for the bayou. The assessment seeks to determine the presence and causes of ambient water and sediment toxicity. Figure 3.1 provides a conceptual toxicity strategy flow diagram for this assessment study.

Figure 3.1 Conceptual Toxicity Strategy Flow Diagram



SECTION 4 ASSESSMENT METHODS

4.1 STUDY DESIGN

The general approach used in this assessment is a two-step investigative process. The first step involves determining if impairment of the designated uses continues. Delisting of the waterbody from the 303(d) list would be pursued if monitoring results demonstrate the waterbody is no longer impaired. Second, if toxicity is found to be present, a Toxicity Identification Evaluation (TIE) will be performed to identify the toxicant or toxicants causing the impairment. Based on results of the TIE, attempts will be made to identify the source(s) of the toxicity.

4.2 SAMPLING METHOD

Field measurements and sediment samples were collected from Stations 14368, 11299 and 14371 on Vince Bayou and Little Vince Bayou (Segment 1007A) during 12 sampling events starting in April 2001 and ending in June, 2002. Table 4.1 identifies the stations sampled, sampling frequencies, toxicity tests conducted, and chemical parameters analyzed.

Field staff of Parsons followed the field sampling procedures for field, biological, and conventional chemical parameters documented in the TCEQ *Surface Water Quality Monitoring Procedures Manual* (TCEQ, 1999a) and the TCEQ *Receiving Water Assessment Procedures Manual* (TCEQ, 1999b). Additional procedures for field sampling outlined in this section reflect specific requirements for sampling under this TMDL Project and/or provide additional clarification.

Four general water chemistry parameters were routinely analyzed during sample collections. Temperature, pH, dissolved oxygen, and specific conductivity were measured with a YSI 600 XL Multi-Parameter Probe. These parameters were measured when samples were collected from a sample location.

4.3 SAMPLING EVENTS

The following subsections provide a summary of samples gathered for each specific trip.

Table 4.1
Summary of Water and Sediment Sampling Events in Vince Bayou, Segment 1007

ANALYSES	April 18, 2001			May 24, 2001			June 14, 2001			July 18, 2001			July 26, 2001			August 9, 2001			Total
	Stations	Stations	Stations	Stations	Stations	Stations	Stations	Stations	Stations	Stations	Stations	Stations	Stations	Stations	Stations	Stations			
	11299	14368	14371	11299	14368	14371	11299	14368	14371	11299	14368	14371	11299	14368	14371	11299	14368	14371	
Field-measured parameters																			
Temperature, DO, pH, conductivity	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	13
SEDIMENT TOXICITY EVALUATION																			
Chronic toxicity bioassays																			
<i>Neanthes</i>	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	10
<i>Leptochirus</i>	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	10
Total metals																			
As, Cd, Cr, Cu, Pb, Hg, Ni, Se, Ag, Zn				1						1			1		1				4
VOCs																			
Includes priority pollutant list				1						1			1		1				4
SVOCs																			
Includes priority pollutant list				1						1			1		1				4
PCBs				1						1			1		1				4
Pesticides/Herbicides including modern compounds				1						1			1		1				4
Polycyclic aromatic hydrocarbons				1						1			1		1				4
Total PAHs analysis (includes priority pollutant list)				1						1			1		1				4
Bioavailability evaluation																			
TOC, AVS, SEM				1						1			1		1				4
Grain-size evaluation																			
Percent sand, silt, clay				1						1			1		1				4

Table 4.1

Summary of Water and Sediment Sampling Events in Vince Bayou, Segment 1007

ANALYSES	October 10, 2001		January 9, 2002		April 3, 2002		April 23, 2002		May 29, 2002		June 27, 2002		Total
	Stations	14368	Stations	14368	Stations	14368	Stations	14368	Stations	14368	Stations	14368	
Field-measured parameters													
Temperature, DO, pH, conductivity	1		1		1		1		1		1		19
SEDIMENT TOXICITY EVALUATION													
Chronic toxicity bioassays													
<i>Neanthes</i>	1			1			1			1			12
<i>Leptoichirus</i>	1			1			1			1			13
Total metals													
As, Cd, Cr, Cu, Pb, Hg, Ni, Se, Ag, Zn													5
VOCs													
Includes priority pollutant list													5
SVOCs													
Includes priority pollutant list													5
PCBs													5
Pesticides/Herbicides including modern compounds													5
Polycyclic aromatic hydrocarbons													5
Total PAHs analysis (includes priority pollutant list)													5
Bioavailability evaluation													
TOC, AVS, SEM													5
Grain-size evaluation													
Percent sand, silt, clay													5

4.3.1 First Sampling Event (April 18, 2001)

Sonde readings and sediment samples were collected at Stations 14368 and 11299 of Vince Bayou. The sonde readings consist of temperature, conductivity, dissolved oxygen, and pH measurements. Duplicate sediment samples were collected at Station 14368. Due to the discovery of a dead body, sampling was postponed. The following day, sonde readings and sediment samples were collected at Station 14371 of Little Vince Bayou. At this location, there was a lot of concrete and rip rap. Even approximately 50 feet downstream, the bayou bottom was predominately concrete and rip rap. Therefore, in order to collect sediment, the crew moved approximately 45 feet downstream of the bridge on Richey Road.

4.3.2 Second Sampling Event (May 24, 2001)

Water parameter readings were recorded and sediment samples collected from all three stations. The water parameters monitored included chlorine, pH, conductivity, and temperature. The first site visited was Station 14368, followed by Station 14371 at Little Vince Bayou. As in the case of the first sampling event, the sediment collected at Station 14371 contained many rocks. The sampling location was then moved 150 feet north of Richey Road, where the water parameters were tested and sediment collected. It was noted that Little Vince Bayou had a large amount of trash, tires, and debris present in the water. The water was also nearly stagnant with practically no flow to the Houston Ship Channel. The third site visited was Station 11299.

4.3.3 Third Sampling Event (June 14, 2001)

Water parameter measurements and sediment samples were collected at Station 14368. Since toxicity had been identified in earlier sampling events, only this station was sampled to begin work on the TIE. The water parameters were collected using the YSI sonde device included temperature, conductivity, dissolved oxygen, specific conductivity, percent dissolved oxygen, and pH. In addition to the YSI data, chlorine measurements were taken. For sediment, a split sample was collected. Sediment was collected in three buckets and combined to form a composite sample for USEPA elutriate test.

4.3.4 Fourth Sampling Event (July 18, 2001)

Data and sediment samples were collected at Station 14368 for sediment toxicity and chemistry. This station was included for additional chemistry analyses because toxicity had been detected at this location. This sampling event was scheduled for earlier in the month, but Tropical Storm Allison delayed the sampling.

4.3.5 Fifth Sampling Event (July 26, 2001)

Sediment samples were collected at Station 14368 for sediment organic chemistry since the FedEx shipment got lost. In addition, sediment was collected at Station 11299 for toxicity and chemistry analyses, and YSI data were recorded.

4.3.6 Sixth Sampling Event (August 9, 2001)

YSI and GPS data were recorded at the three stations. Readings were first taken at Station 11299 (GPS coordinates 10880366 N, -973610 E.), then at Station 14368 (GPS coordinates 10879146.6 N, -972441.9 E), and finally at Station 14371 (GPS coordinates 10878641.4 N, -970084.1 E).

4.3.7 Seventh Sampling Event (October 10, 2001)

The field crew arrived at Vince Bayou station 14368 at 1400. Water parameter measurements and sediment samples were collected at Station 14368. The water parameters were collected using the YSI sonde device included temperature, conductivity, dissolved oxygen, specific conductivity, percent dissolved oxygen, and pH. Sediment samples were collected and sent to TRAC Laboratories via FedEx.

4.3.8 Eighth Sampling Event (January 9, 2002)

The field crew arrived at Vince Bayou station 14368 at 08:20. The weather was sunny, clear, with low humidity and a temperature of 65 degrees Fahrenheit (°F). YSI was calibrated and water quality measurements were taken. The sediment sample was dark brown in color with no appreciable odor. Samples were also collected at Stations 11301, 11171 and “E. Jackson.”

4.3.9 Ninth Sampling Event (April 3, 2002)

The field crew arrived at Vince Bayou to collect sediment samples from Station 14368. Arrived at station at 1315. YSI was calibrated and water quality measurements were taken. Sediment samples were collected, and they appeared to be blank in color and fine to medium grained.

4.3.10 Tenth Sampling Event (April 23, 2002)

The field crew arrived at Vince Bayou at Vince Bayou to collect sediment sample from Station 14368. Arrived at station at 0840. YSI was calibrated and water quality measurements were taken. Two 3.5 gallon buckets of sediment were collected. The sediment was blank in color and had a strong organic odor, similar to rotting vegetation.

4.3.11 Eleventh Sampling Event (May 29, 2002)

The field crew arrived at Vince Bayou Station 14368 at 0845. Water parameter measurements and sediment samples were collected. The water parameters were collected using the YSI sonde device. The weather was partially cloudy, humid and a temperature of 80°F. Sediment sample was a mixture of blank sediment with a clay sediment colored grayish brown. The odor was of organic rotting material. Sediment samples were collected and sent to TRAC Laboratories via FedEx.

4.3.12 Twelfth Sampling Event (June 27, 2002)

The field crew arrived at Vince Bayou Station 14368 at 0840. Water parameter measurements and sediment samples were collected at Station 14368. The water parameters were collected using the YSI sonde device included temperature, conductivity, dissolved oxygen, specific conductivity, percent dissolved oxygen, and pH. Sediment samples were collected and sent to TRAC Laboratories via FedEx.

4.4 ANALYTICAL METHODS

Appendix E lists a combination of the analytical methods used and potential methods for potential toxicant identification. The analyses listed in Appendix E are USEPA-approved methods as cited in TCEQ TMDL guidance document, Clean Rivers Program, or Surface Water Quality Monitoring program guidelines and in 40 Code of Federal Regulations, Section 136, Part B. Exception to this includes analyses and sample matrices for which no regulated methods exist, or where USEPA has not approved any method with adequate sensitivity for TMDL data requirements.

4.5 TOXICITY TESTING METHODS

The toxicity of sediments was assessed by the following methods using the marine amphipod *Leptocheirus plumulosus* and the marine polychaete worm *Neanthes arenaceodentata*:

- For *L. plumulosus*: Methods for Measuring the Toxicity of Sediment-Associated Contaminants with Estuarine and Marine Amphipods (USEPA/600/R-94/025).
- For *N. arenaceodentata* ASTM. 2000. Standard Guide for Designing Biological Tests with Sediments. E1525-94a. In *Annual Book of ASTM Standards*, Vol. 11.05, Philadelphia, PA.

For toxicity testing, marine amphipods and polychaetes were exposed for 10 days to sediment collected from three stations positioned along Segment 1007. Mortality at the end of the 10-day exposure period was statistically compared to mortality found in control exposures where the organisms were exposed to clean sediments supplied by the testing laboratory.

Whereas USEPA approved methods have been developed to identify causes of toxicity in effluents and ambient water, approved methods are not yet available for performing TIEs on sediments. In recent years, considerable progress has been made by USEPA and other research entities to develop TIE methods for sediments. The sediment TIE methods used in this investigation were developed through the coordinated efforts of scientists at USEPA's laboratory in Duluth, Minnesota, scientists at North Texas State University (UNT), TRAC Laboratories and Parsons using the most recent scientific advances in the subject area.

4.6 QUALITY CONTROL REQUIREMENTS

Refer to the Assessment of the Presence and Causes of Ambient Toxicity Quality Assurance Project Plan (QAPP), Revision 4, FY 2002-03.

4.6.1 Sampling Quality Control Requirements and Acceptability Criteria

The minimum field quality control (QC) requirements followed by Parsons are outlined in the TCEQ *Surface Water Quality Monitoring Procedures Manual* and in Section B5 of the project QAPP. Sampling QC involved field duplicates, matrix spikes and matrix spike duplicates.

4.6.2 Laboratory Measurement Quality Control Requirements and Acceptability Criteria

These requirements and criteria were applicable to all laboratories used for analysis of various required parameters. Detailed laboratory QC requirements were contained within each individual method and laboratory quality assurance manuals. As described in Section B5 of the project QAPP, the minimum requirements followed by analytical laboratories included: 1) laboratory duplicates; 2) laboratory control standards (LCS); 3) matrix spikes (MS) and matrix spike duplicates; 4) method blanks; and 5) additional QC samples such as surrogates, internal standards, continuing calibration samples, and interference check samples. Laboratory QC sample results were reported with the data report (see Section C2 of the project QAPP).

4.6.3 Failures in Quality Control Requirements

As described in Section B5 of the project QAPP, sampling QC excursions were evaluated by the Parsons Project Manager, in consultation with the Parsons Quality Assurance Officer (QAO). Differences in field duplicate sample results were used to assess the entire sampling process, including environmental variability. The arbitrary rejection of results based on pre-determined limits was not practical, therefore, the professional judgment of the Parsons Project Manager and QAO was relied upon in evaluating results. Rejecting sample results based on wide variability was a possibility. Corrective action included identification of the cause of the failure where possible. Response actions typically included re-analysis of questionable samples. In some cases, a site was re-sampled to achieve project goals. The disposition of such failures and conveyance to the TCEQ are discussed in Section B4 of the project QAPP under Failures or Deviations in Analytical Methods Requirements and Corrective Actions.

Refer to Appendix E for the summarization of QA/QC findings, data acceptability and qualifiers to deviations.

4.7 DATA MANAGEMENT

Data Management Protocols are addressed in the Data Management Plan in Appendix E of the project QAPP.

4.8 STREAM HABITAT CHARACTERIZATION

Stream habitat characterization utilizing TCEQ procedures was performed during the August sampling event by completing copies of the TCEQ's receiving water assessment forms (Stream Physical Characteristics Worksheets) for each location. The detailed habitat forms are located in Appendix H.

SECTION 5 RESULTS OF AMBIENT SEDIMENT ANALYSIS

5.1 FIELD MEASUREMENTS

All field measurements were within expected ranges during these sampling results. Table 5.1 presents the results from these events. Although the reported dissolved oxygen concentrations appear low, the dissolved oxygen standard for Segment 1007 is 1.0 mg/l.

5.2 AMBIENT SEDIMENT TOXICITY RESULTS

Sediment toxicity was evaluated by a 10-day sediment exposure test with the marine amphipod, *L. plumulosus* and the marine polychaete worm, *Neanthes arenaceodentata* using methods specified in Section 4.4 of the report. Criteria for determining whether significant sediment toxicity has occurred to *Neanthes* and *Leptocheirus* are specified in the Technical Memorandum in Appendix F to this report. The following conditions must each be met for sediment to be considered toxic:

1. There is a statistically significant reduction in survival, at alpha equal to 0.05;
2. Mortality in the sample exceeds that of the control by 20 percent; and
3. Mortality in the sample must also be less than the minimum control mortality allowed according to the USEPA methods.

If one or more of the three criteria were not met, the sediment sample was not considered significantly toxic. Similar conditions to these have been used previously by TCEQ in TPDES permits as conditions that trigger a TIE/TRE. These conditions assume that a sample is ecologically significant and that some quantifiable increase in survival of the test organisms maybe observed in conducting a TIE.

Table 5.2 presents toxicity analysis results for Vince Bayou sediments conducted at Stations 11299, 14368 and 14371. Test methods followed USEPA's chronic estuarine and marine sediment testing protocols that evaluate organism survival over a 10-day test period. Test species were *Leptocheirus plumulosus* and *Neanthes arenaceodentata*. Sampling was conducted at all locations on April 18-19, May 24, June 14, July 19 and 26, October 30, 2001 and January 9, April 3, April 23, and May 29, 2002. Toxicity was present in the first six out of nine samples at Station 14368. One sample collected from Station 11301 on January 9, 2002 was found to be toxic. The toxicity disappeared after January 2002.

Toxicity was documented at Station 14368 during the first six sampling events conducted. Reduced survival was observed for both test species, but *L. plumulosus* was consistently the most sensitive organism. A sediment TIE procedure was initiated at this location based on these results, as discussed in Section 6.

**Table 5.1
Field Measurement
Vince Bayou
Station 11299**

Date M/D/Y	Temperature °C	DO Conc mg/L	pH	Cond mS/cm	TRC mg/l
4/18/2001	24.27	3.96	7.28	EM	NR
5/24/2001	14.5	NR	7.03	EM	NR
7/26/2001	30.82	3.83	7.09	17567	NR
8/9/2001	31.54	1.78	6.91	18061	NR
Station 14368					
Date M/D/Y	Temp °C	DO Conc mg/L	pH	Cond mS/cm	TRC mg/l
4/18/2001	24.53	5.37	7.04	EM	NR
5/24/2001	14.5	NR	6.94	EM	NR
6/14/2001	28.65	3.39	7.45	490	NR
7/18/2001	30.75	1.83	7.26	12370	NR
8/9/2001	30.2	3.78	7.11	6039	NR
10/30/2001	22.46	EM	EM	5860	<0.1
1/9/2002	14	3.43	7.24	8468	NR
4/3/2002	Field measurements not taken				
4/23/2002	25.37	1.38	6.98	972	NR
5/29/2002	26.52	7.52	6.34	NR	NR
6/27/2002	29.05	1.24	7.29	6858	NR
Station 14371					
Date M/D/Y	Temp °C	DO Conc mg/L	pH	Cond mS/cm	TRC mg/l
4/18/2001	23.7	5.37	6.86	EM	NR
4/19/2001	20.79	4.37	7.67	EM	NR
5/24/2001	14.5	NR	7.37	EM	0
8/9/2001	30.05	2.2	7.30	5097	NR

°C - degrees Celcius

mg/L - milligrams per liter

mS/cm - milli Siemens per centimeter

ft - feet

pH is in standard units

Cond - Conductivity

DO Conc - Dissolved oxygen concentration

NR - Not Recorded

Missing results will be completed upon review of field notes.

EM - Equipment Malfunction

Table 5.2
Vince Bayou 1007A
10 Day Marine Sediment Exposure Results

Vince Bayou 1007A		% Survival	
		Neanthes	Leptocheirus
April 18-19, 2001	Control	100	99
	11299	100	96
	14368	96	7
	14368-Dup1*	28	2
	14371	92	85
May 24, 2001	Control	96	96
	11299	96	96
	14368	32	13
	14371	92	92
June 14, 2001	Control	92	95
	11299		
	14368	92	1
	14371		
July 19 & 26, 2001	Control	100	98
	11299	100	96
	14368	92	5
October 30, 2001	Control	100	98
	14368	100	5
January 9, 2002	Control		99
	14368		32
	11301		44
	11171		94
	E. Jackson		94
April 3, 2002	Control	100	100
	14368	100	86**
April 23, 2002	Control	100	100
	14368	100	92
	11301	100	92
	11171	88	90
	E. Jackson	100	98
May 29, 2002	Control	100	100
	14368	100	99

Bold - denotes significant difference from the control

* sample collected in approximately the same location; for quality control purposes only

** significantly different, but not toxic according to recommended criteria

E. Jackson is located approximately mid way between Stations 11301 and 11171; sediment collected and tested in effort to isolate toxicity.

5.3 CHEMICAL ANALYSIS RESULTS

Sediment samples were collected on May 24, 2001 for chemical analysis at Station 11299. This station was selected because it is TCEQ's historic sampling site, and had previously documented toxicity. The sediment sample was collected 150 feet north of Richey Road, at the same location where sediment was collected for toxicity analysis.

Table 5.3 presents sediment analysis results for chemicals found above detectable concentrations. A complete listing of analytes is presented in Appendix D. The collected sediment was primarily composed of sand and silt.

As indicated by the chemical screening of sediment from Station 11299, copper, lead, zinc and several PAHs could be potential toxicants to organisms. Exceedances of the screening criteria were moderate for the metals, but more significant for PAHs. However, the sample had elevated concentrations of total organic carbon (24.7 g per kg) and acid volatile sulfides (1.3 mmol per g dry wt.) that are likely to significantly reduce the bioavailability of organic chemicals and metals, respectively. This is consistent with the fact that no toxicity has been detected at Station 11299, during the testing.

On July 26, 2001, additional sampling was conducted for chemical analysis of the sediment at Station 11299, as well as Station 14368 where toxicity was observed. Sediment collected from Station 14368 contained more heavy metals than sediment from Station 11299.

Sediment from Station 11301, which is upstream of Station 14368 at the West Shaw Avenue Bridge, was collected to determine the extent of contamination. The lead concentration in the sediment at Station 11301 was high. Copper was detected but was not quantifiable. PAHs were also elevated.

**Table 5.3
Chemical Analysis Detections**

		Station ID 11299	Station ID 11299	Station ID 14368	Station ID 14368	Station ID 11301		
PARAMETER		5/24/01 RESULT	7/26/01 RESULT	7/26/01 RESULT	4/23/02 RESULT	4/23/02 RESULT	Lowest Screening Value*	UNITS
Ions	Chloride	1160	1660	3990	134	96.4		mg/Kg-dry wt
	Sulfate	76.7	166	189	106	119		mg/Kg-dry wt
Metals	Aluminum	11100	6410	17000	10400 J	7890 J	7.24	mg/Kg-dry wt
	Arsenic	3.95	3.26	5.41	7.6	2.29		mg/Kg-dry wt
	Barium	86.8	53.8	256	115	74.9		mg/Kg-dry wt
	Cadmium	0.514	0.347	1.31	0.312	0.18	0.676	mg/Kg-dry wt
	Calcium	23100	21300	32800	115000 J	28700 J		mg/Kg-dry wt
	Chromium	22.3	12.9	35.3	68.8	23	52.3	mg/Kg-dry wt
	Copper	23.1	25.2	53.2	40.3 J	20.1 J	18.7	mg/Kg-dry wt
	Iron	12000	6600	16200	1990 J	9770 J		mg/Kg-dry wt
	Lead	35.2	32.1	173	60.5	86.3	30.24	mg/Kg-dry wt
	Magnesium	3110	2710	4620	3700 J	2890 J		mg/Kg-dry wt
	Nickel	10.1	7.13	16.4	16.9	9.24	15.9	mg/Kg-dry wt
	Potassium	1670	936	2280	1280 J	1260 J		mg/Kg-dry wt
	Selenium	ND	ND	ND	1.98	ND		mg/Kg-dry wt
	Silver	ND	ND	6.51	ND	ND	0.73	mg/Kg-dry wt
	Sodium	1330	1510	3230	910	252		mg/Kg-dry wt
	Zinc	133	83.2	317	81.9	88.4	124	mg/Kg-dry wt
Mercury	0.106	0.459	0.128	0.109	ND	0.13	mg/Kg-dry wt	
Volatiles	Chlorobenzene	ND	ND	2 J	ND	ND	413	µg/Kg-dry wt
	o-Xylene	ND	ND	5.4 J	ND	ND		µg/Kg-dry wt
	Toluene	ND	ND	ND	15.1	ND		µg/Kg-dry wt
Semi-Volatiles	Anthracene	55	58 J	140 J	110 J	130 J	46.85	µg/Kg-dry wt
	Benzo(a)anthracene	385	453	547	506	1030	74.8	µg/Kg-dry wt
	Benzo(a)pyrene	501	747	754	506	1250	88.8	µg/Kg-dry wt
	Benzo(b)fluoranthene	777	830	1110	612	13400	27372	µg/Kg-dry wt
	Benzo(g,h,i)perylene	389	281	318	ND	ND	720	µg/Kg-dry wt
	Benzo(k)fluoranthene	489	708	722	461	1200	3600	µg/Kg-dry wt
	Bis(2-ethylhexyl)phthalate	1050	582	22400	940	474	182	µg/Kg-dry wt
	Butyl benzyl phthalate	ND	ND	ND	ND	ND	900	µg/Kg-dry wt
	Chrysene	617	714	961	736	1490	108	µg/Kg-dry wt
	Di-n-octylphthalate	ND	170 J	1100	ND	ND	885363	µg/Kg-dry wt
	Fluoranthene	944	978	1580	1590	2640	113	µg/Kg-dry wt
	Fluorene	ND	ND	120 J	ND	ND	19	µg/Kg-dry wt
	Indeno[1,2,3-cd]pyrene	325	299	250 J	328	860		µg/Kg-dry wt
	Phenanthrene	319	331	857	328	1170	86.7	µg/Kg-dry wt
Pyrene	780	812	1260	1060	2030	153	µg/Kg-dry wt	

**Table 5.3
Chemical Analysis Detections**

PARAMETER		5/24/01 RESULT	7/26/01 RESULT	7/18/01 RESULT	4/23/02 RESULT	4/23/02 RESULT	Lowest Screening Value*	UNITS
Pest/ PCBs	Chlordane	ND	6.6 J	31 J	ND	ND		µg/Kg-dry wt
	d-BHC	ND	ND	ND	ND	ND		µg/Kg-dry wt
	4,4'-DDD	ND	11 J	ND	ND	ND	1.22	µg/Kg-dry wt
	4,4'-DDE	ND	12 J	ND	ND	ND	2.07	µg/Kg-dry wt
	4,4'-DDT	ND	5.5 J	27 J	ND	ND	1	µg/Kg-dry wt
	PCB-1248	ND	ND	4000 J	ND	ND		µg/Kg-dry wt
	PCB-1254	11000	ND	ND	ND	ND		µg/Kg-dry wt
Organo-phosphorus Compounds	Chloropyrifos	14.0 J	ND	ND	ND	ND		µg/Kg-dry wt
SEM	Cadmium	0.5	0.19	0.83	ND	0.0037		µmol/dry g
	Copper	1.01	ND	ND	2.2 J	1.2 J		µmol/dry g
	Lead	48.6	13	140	0.31 J	0.49 J		µmol/dry g
	Mercury	0.0006 J	ND	ND	0.00024	0.0007		µmol/dry g
	Nickel	3.16	0.98	3.5	0.12	0.19		µmol/dry g
	Silver	1.066	ND	ND	NA	NA		µmol/dry g
	Zinc	161.28	49	180	2 J	2.7 J		µmol/dry g
Total Organic Carbon (TOC)		24700	16580	23940	8100	8200		mg/Kg C
Acid Volatile Sulfide (AVS)		1323	420	2200	26.2	24.4		µmol/dry g
Grain Size	Gravel	NA	NA	NA	8.9	0		
	Sand	42	68	39	72	55		%
	Silt	33	21	44	12	27		%
	Clay	25	11	18	8	18		%

Notes:

* Criteria is from *Equilibrium and Non-Equilibrium Partitioning-Based Sediment Quality Screening Indices* tables.

The value is the lowest value from the Indices as stated in the Appendix.

J- result is estimated

ND- result was Not Detected

mg/kg-dry = milligrams per kilogram dry weight

ug/kg-dry = microgram per kilogram dry weight

umol/dry g = microgram per mole per dry gram

% = percent

SECTION 6 TOXICITY IDENTIFICATION EVALUATION

6.1 IDENTIFICATION OF CLASSES OF COMPOUNDS

Station 14368 was determined to have significant toxicity on the first two events. Therefore, after the second event, the sampling focused on obtaining fresh samples for the TIE at Station 14368. It should be noted that the April 18-19, 2001 14368-duplicate was found to be toxic to *Neanthes* while the 14368 test was not. Typically a conflict in the duplicate and test results indicate possible contamination. In this case the duplicate and test samples were both be toxic to *Leptocheirus*. Therefore, the April 2001 sample is reported as toxic to *Neanthes*.

Station 11299 previously showed toxicity using the elutriate test by USEPA, but has not shown toxicity to date in samples collected by TCEQ or this study. Station 14368 has shown toxicity to both *Leptocheirus* and *Neanthes*, but *Leptocheirus* appears more sensitive.

Toxicant identification for Station 14368 sediments, based on standard phase procedures, showed a SPE-extraction as effectively reducing toxicity. See Tables 6.1 and 6.2. These results, however, were inconclusive as the treatment was effective in only one out of 5 tests conducted with two tests species.

Additional phase 1 TIE tests were performed to determine some physical characteristics of the toxicant in samples from station 14368. During these tests, it was determined that toxicity was removed from the porewaters by adjusting the pH to 3.0 and sparging the porewater samples with air. Only the combination of adjusting the pH and sparging removed the toxicity. The next approach to the TIE was to try to capture the sparged gas fraction from pH 3.0 adjusted porewater onto a charcoal cartridge and in methanol. In addition, an attempt to move the toxic fraction, via sparged gas, to control water and recover toxicity was made. Neither the charcoal nor methanol fractions revealed any significant results. Several volatile traps were used, none of which detected any compounds of interest.

In subsequent TIEs, it has been discovered that the toxic fraction is not volatilizing, but sorbing at pH 3.0 to the suspended material in the porewater. This was determined by using a 0.2-micron filtration at pH 3.0 whereby toxicity was completely removed. Currently, tests are being performed to recover the toxic fraction from the filters. The filters were analyzed by GC/MS and many compounds were recovered, although none of which are believed to be contributing to the toxicity of interest. An attempt to clean the sample of uninvolved background components is underway by pH adjustments, filtration techniques and the use of C-18 cartridges. This should make isolating compounds of interest more attainable. Apparently, by lowering the pH to 3 the toxicant absorbs onto particles that can be filtered. If pH is raised back up to initial pH without filtering, the toxicant moves back into solution. Figure 6.1 summarizes the TIE evolution as previously described.

Table 6.1
Phase 1 TIE of Station 14368 Sediments using *Leptocheirus plumulosus*

20 - 24 June 2001			10 - 14 July 01			16 - 20 July 01		
TIE fraction	LC50 (% Pore-water)	Toxic Units (TU)	TIE fraction	LC50 (% Pore-water)	Toxic Units (TU)	TIE fraction	LC50 (% Pore-water)	Toxic Units (TU)
Initial test	28.0	3.57	Initial test			Initial test		
Baseline	35.1	2.85	Baseline	40.1	2.49	Baseline	35.4	2.82
Aeration	44.1	2.27	Aeration	40.1	2.49	Aeration	31.2	3.20
Filtration	44.1	2.27	Filtration	35.4	2.82	Filtration	35.4	2.82
SPE	>100	NA	SPE	35.4	2.82	SPE	35.2	2.84
SPE Elution	0.45X	2.22	SPE Elution			SPE Elution		
EDTA	44.1	2.27	EDTA	45.7	2.20	EDTA	31.2	3.20
Na ₂ S ₂ O ₃	45.5	2.2	Na ₂ S ₂ O ₃	41.7	2.40	Na ₂ S ₂ O ₃	27.3	3.66

Table 6.2
Phase 1 TIE of Station 14368 Sediments using *Mysidopsis bahia*

20 - 24 June 2001			10 - 14 July 01			16 - 20 July 01		
TIE fraction	LC50 (% Pore-water)	Toxic Units (TU)	TIE fraction	LC50 (% Pore-water)	Toxic Units (TU)	TIE fraction	LC50 (% Pore-water)	Toxic Units (TU)
Initial test			Initial test			Initial test		
Baseline			Baseline	45.7	2.18	Baseline	35.4	2.82
Aeration			Aeration	31.2	3.20	Aeration	40.1	2.49
Filtration			Filtration	35.4	2.82	Filtration	40.1	2.49
SPE			SPE	35.4	2.82	SPE	35.4	2.82
SPE Elution			SPE Elution			SPE Elution		
EDTA			EDTA	31.2	3.20	EDTA	33.3	3.00
Na ₂ S ₂ O ₃			Na ₂ S ₂ O ₃	35.4	2.82	Na ₂ S ₂ O ₃	31.2	3.20

All of the TIE work is being performed on sediment samples that were collected prior to April 2002. It was discovered that sometime after the January 2002 sampling event, stations 14368 and 11301, which had previously and consistently shown toxicity, were now not toxic for either *Leptocheirus* or *Neanthes*. For the April 3, 2002 sampling event, station 14368 was only slightly toxic for *Leptocheirus* and not toxic at all for *Neanthes*. Since this sampling event, there have not been any significant differences from the control in percent survival or sub-lethal effects. In May and June of 2002, several attempts were made by Parsons sampling crew to identify areas of similar looking sediments and collect samples both upstream and downstream from the usually toxic areas, without success. This fresh toxic sample cannot be found anywhere in Vince Bayou at present. For this reason, all TIE manipulations used sediments collected in early 2002.

The following discusses results of procedures employed after more conventional TIE procedures failed to produce a method by which sediment pore water toxicity could be both removed from the pore water and subsequently recovered by a method amenable to toxicant identification via analytical analysis. Experiments revealed (1) removal of the toxic component(s) was possible only under the condition of reduced pH (2) removal of suspended particulate matter prior to pH adjustment was necessary as toxic components appeared to sorb to suspended particulates at reduced pH (3) under conditions of reduced pH, pore water toxicant(s) consistently sorbed to an HLB SPE extraction cartridge but not to conventional C18 SPE cartridges (4) the most toxic fraction was effectively recovered from the cartridge with an 80% methanol in water elution (5) toxicity recovery was observed only in cleaned-up pore water and not in clean seawater. After caprolactam was detected in GC/MS analyses of methanol eluates, an investigation was initiated to determine the toxicity of known concentrations of caprolactam and gain understanding of the caprolactam-related compounds observed in the pore water through LC/MS analysis.

The sediment pore water was tested unaltered (baseline) and yielded an LC50 of 48.9% pore water (Table 6.3). Additional pore water was tested concurrently after being passed through an HLB SPE cartridge at reduced pH as described above. Survival data from this test are summarized in Table 6.4 and indicate a significant reduction of toxicity. Subsequent cartridge elution with varying dilutions of methanol in water yielded significant toxicant recovery in the 80% methanol fraction (Table 6.5). Two additional tests were conducted to examine the toxicity of known concentrations of caprolactam and to examine the differences, if any, of caprolactam toxicity in pore water compared to seawater. The caprolactam spiked, HLB SPE cleaned-up pore water test yielded an LC50 of 31.0 mg/L caprolactam (Table 6.6) whereas the caprolactam spiked seawater test yielded an LC50 of 453.5 mg/L caprolactam (Table 6.7).

Early GC/MS analyses of toxic fractions revealed the presence of caprolactam in some preparations, but not all. More recently, side-by-side analyses of duplicate preparations of 80% methanol elutions of HLB solid-phase cartridge extractions of toxic pore water exhibited a large caprolactam peak in one preparation and no caprolactam peak in the duplicate. This led us to speculate that caprolactam instability in the pore water matrix might be related to the inconsistency between the two preparations. However, since the HLB SPE procedure

consistently removed toxicity and caprolactam was the only major peak observed in the cartridge elution, we tested caprolactam for toxicity in both the clean seawater and in pore water which had been previously rendered non-toxic by low pH HLB SPE clean-up. Caprolactam was much more toxic (approximately one order of magnitude) in the cleaned-up pore water than in clean sea water. This pattern was also observed for the toxic materials eluted from the HLB clean-up cartridge. Since the unknown toxicant from the cartridge elution and caprolactam shared this common characteristic, we decided to examine the original toxic pore water for caprolactam and related compounds. Caprolactam can be analyzed by GC/MS but polar products resulting from the opening of the ring structure as well as related polymeric compounds are not amenable to GC/MS analysis and this may explain why our analyses of the toxic fractions sometimes contained caprolactam and sometimes did not. Consequently, we sub-contracted with Dr. Robert Voyksner, LCMS Limited, to examine selected samples by LC/MS. To date, Dr. Voyksner's work has demonstrated (1) the 80% methanol toxic fraction eluted from the HLB SPE cartridge shows the presence of LC/MS peaks with mass spectral characteristics consistent with the open ring structure of caprolactam and related polymeric materials (2) these caprolactam-related peaks are also present in the original toxic pore water and (3) these caprolactam-related peaks are absent in the non-toxic pore water resulting from HLB SPE clean-up. Analysis of HLB-cleaned-up pore water spiked with caprolactam revealed only caprolactam; spiking did not result in the formation of the caprolactam-related peaks found in the original toxic pore water and the toxic fraction eluted from the HLB cartridge.

Our interpretation of the results above is that there is evidence that caprolactam-related substances are contributing to the toxicity observed in the pore water. This toxic effect is observed only in combination with other contributing factors which remain in the pore water after HLB SPE treatment at low pH. These factors apparently interact with either spiked caprolactam or the HLB 80% toxic fraction to produce a toxic effect which is absent when either of these materials is tested for toxicity in clean sea water. We also have evidence that the increased toxicity of caprolactam seen in association with the cleaned-up pore water is not dependent upon the direct action of the pore water on the caprolactam (i.e. inducing ring opening or polymerization) since the increased toxicity of caprolactam can be induced by independent exposures to cleaned-up pore water (non-toxic by itself) and caprolactam in clean seawater (non-toxic by itself; see the results of the dual exposure experiment). This leads us to believe that the toxic interaction of caprolactam is likely manifested regardless of the integrity of the ring structure and state of polymerization in the environmental exposure (within the limits observed in the toxic pore water) and probably results from a metabolized form of the material that may be common to any form of the material that is initially taken up by the test organism.

Caprolactam is primarily used in the manufacture of Nylon 6 and other synthetic fibers. Caprolactam is also used in brush bristles, textile stiffeners, film coatings, synthetic leather, plastics, plasticizers, paint vehicles, cross-linking for polyurethanes, and in the synthesis of lysine (USEPA 1988, USDHHS 1993).

Table 6.3
Survival of *Mysidopsis Bahía*, Exposed to Unaltered Sediment Pore Water

<u>Test Treatment</u> <u>(% Pore water)</u>	<u>Mean Survival</u> <u>(%)</u>
<u>Exposure Period</u>	<u>96 Hours</u>
Seawater Control	100
25	90
50	60
100	0

LC50 = 48.9% pore water

Table 6.4
Survival of *Mysidopsis Bahía*, Exposed to Post-HLB SPE Sediment Pore Water

<u>Test Treatment</u> <u>(% Pore water)</u>	<u>Mean Survival</u> <u>(%)</u>
<u>Exposure Period</u>	<u>96 Hours</u>
Seawater Control	100
25	100
50	100
100	90

Cartridge loading period = 2.0 ml/min

Table 6.5
Survival of *Mysidopsis bahia*, Eposed to HLB Cleaned Pore Water Containing Associated Methanol Eluates

<u>Test Treatment</u> <u>(% Methanol in</u> <u>Water)</u>	<u>Mean Survival</u> <u>(%)</u>
<u>Exposure Period</u>	<u>96 Hours</u>
Methanol Blank	80
50	100
75	40
80	0
85	80
90	80
95	80
100	80

Elution period = 0.2 ml/min

Table 6.6
Survival of *Mysidopsis Bahía*, Exposed To Post-HLB SPE Sediment Pore Water Spiked with Caprolactam

<u>Test Treatment</u> <u>(mg/L</u> <u>Caprolactam in</u> <u>Pore water)</u>	<u>Mean Survival</u> <u>(%)</u>
<u>Exposure Period</u>	<u>96 Hours</u>
Pore Water Blank	100
12.5	100
25	60
50	20
100	0
200	0

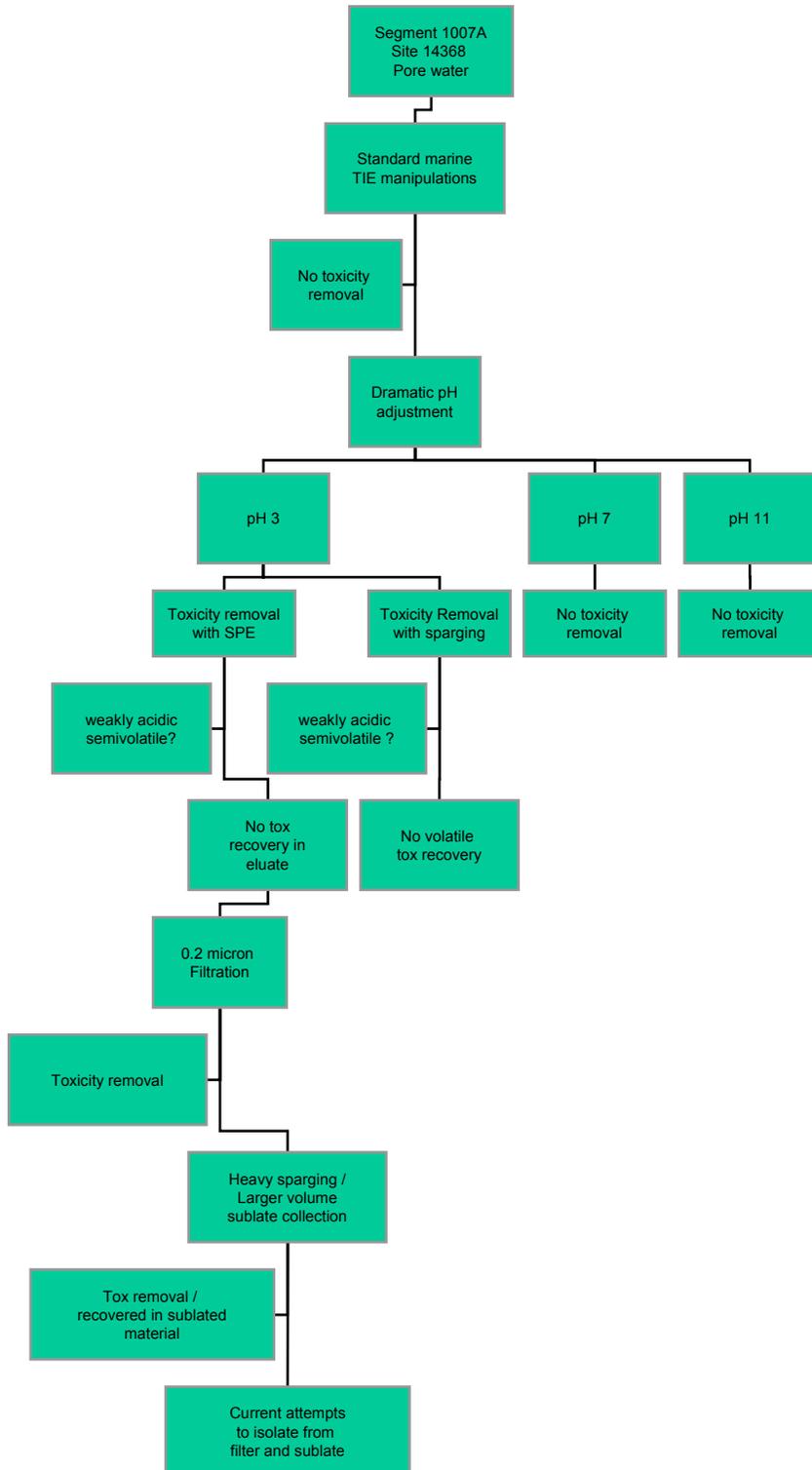
LC50 = 31.0 mg/L caprolactam

Table 6.7
Survival of *Mysidopsis Bahía*, Exposed to Laboratory Seawater Spiked with Caprolactam

<u>Test Treatment</u> <u>(mg/L</u> <u>Caprolactam in</u> <u>Seawater)</u>	<u>Mean Survival</u> <u>(%)</u>
<u>Exposure Period</u>	<u>96 Hours</u>
Seawater Blank	100
250	80
500	60
1000	0
2000	0
4000	0

LC50 = 453.5 mg/L caprolactam

Figure 6.1
TIE Evolution
Vince Bayou - Segment 1007A
Station 14368



SECTION 7 SOURCE ANALYSIS AND IDENTIFICATION

Source Analysis has not been initiated as the TIE was just completed and confirmation of the toxicant was finalized.

SECTION 8 SUMMARY AND CONCLUSIONS

Vince Bayou was included by TCEQ in the state's 303(d) list based on sediment toxicity tests results for the assessment period from 1991 to 1996. No water toxicity was documented in any of 18 samples collected from the bayou. Potential chemicals of concern in sediments identified for Segment 1007 and tributaries include copper, lead, mercury, zinc, and dioxin for fish/shellfish consumption.

The purpose of this assessment is to verify the presence of toxicity in sediments of Vince Bayou and its tributaries and, if toxicity is found, determine its cause(s) and source(s) in the bayou. The results of the analyses will determine whether to proceed with TMDL development or establish the basis for removing the bayou from the 303(d) list.

A two-step approach was used in this assessment. The first step involves determining if impairment of the designated use continues and pursuing delisting of the waterbody from the 303(d) list, if monitoring results demonstrate the waterbody is no longer impaired. The second step, implemented if toxicity is present, is a TIE to identify the toxicant(s) the impairment, and subsequently identify the source(s) of the toxicity.

Three of the seven stations established by TCEQ (Stations 11299, 14368, and 14371), were selected for monitoring in this assessment of Vince Bayou. Three of six scheduled monitoring events have been conducted to date on April 18, May 24 and June 14, 2001. Monitoring included field measurements of water quality, and collection of sediments for chemical analyses and toxicity testing. After the second event, the sampling focused on obtaining fresh samples for the TIE and its results at Station 14368.

Station 11299 previously showed toxicity using the elutriate by USEPA, but has not shown toxicity to date in samples collected by TCEQ or this study. Station 14368 has shown toxicity to both *Leptocheirus* and *Neanthes*, but *Leptocheirus* appears more sensitive. A TIE was initiated on this station.

Toxicant identification for Station 14368 sediments, based on standard phase procedures, showed a SPE-extraction as effectively reducing toxicity. These results, however, were inconclusive as the treatment was effective in only one out of five tests conducted with two tests species. An additional procedure was subsequently employed, passing the pore water through the polymeric adsorbent resin Amberlite XAD-4. In two separate test procedures, this treatment effectively removed toxicity suggesting that organics (petroleum hydrocarbons) are possible contaminants. This conclusion is supported by the detection of several PAHs in the sediment at concentrations well above toxicity screening criteria.

Additional phase 1 TIE tests were performed to determine some physical characteristics of the toxicant in samples from station 14368. During these tests, it was determined that toxicity was removed from the porewaters by adjusting the pH to 3.0 and sparging the porewater samples with air. Only the combination of adjusting the pH and sparging removed the toxicity. The next approach to the TIE was to try to capture the sparged gas fraction from

pH 3.0 adjusted porewater onto a charcoal cartridge and in methanol. In addition, an attempt to move the toxic fraction, via sparged gas, to control water and recover toxicity was made. Neither the charcoal nor methanol fractions revealed any significant results. Several volatile traps were used, none of which detected any compounds of interest.

In subsequent TIEs, it has been discovered that the toxic fraction is not volatilizing, but sorbing at pH 3.0 to the suspended material in the porewater. This was determined by using a 0.2-micron filtration at pH 3.0 whereby toxicity was completely removed. Currently, tests are being performed to recover the toxic fraction from the filters. The filters were analyzed by GC/MS and many compounds were recovered, although none of which are believed to be contributing to the toxicity of interest. An attempt to clean the sample of uninvolved background components is underway by pH adjustments, filtration techniques and the use of C-18 cartridges. This should make isolating compounds of interest more attainable. Apparently, by lowering the pH to 3 the toxicant absorbs onto particles that can be filtered. If pH is raised back up to initial pH without filtering, the toxicant moves back into solution.

All of the TIE work was performed on sediment samples that were collected prior to April 2002. It was discovered that sometime after the January 2002 sampling event, stations 114368 and 11301, which had previously and consistently shown toxicity, were now not toxic for either *Leptocheirus* or *Neanthes*. For the April 3, 2002 sampling event, station 14368 was only slightly toxic for *Leptocheirus* and not toxic at all for *Neanthes*. Since this sampling event, there have not been any significant differences from the control in percent survival or sub-lethal effects. In May and June of 2002, several attempts were made by Parsons sampling crew to identify areas of similar looking sediments and collect samples both upstream and downstream from the usually toxic areas, without success. This fresh toxic sample cannot be found anywhere in Vince Bayou at present. For this reason, all TIE manipulations used sediments collected in early 2002.

The TIE procedure identified caprolactam, caprolactam-related products, and an unknown pore-water toxicant that combined with caprolactam to produce the toxicity. Through subsequent TIEs, it has been discovered that the toxic fraction is not volatilizing, but sorbing at pH 3.0 to the suspended material in the porewater. This was determined by using a 0.2 micron filtration at pH 3.0 whereby toxicity was completely removed. Tests were performed to recover the toxic fraction from the filters. The filters were analyzed by GC/MS and many compounds were recovered, although none of which are believed to be contributing to the toxicity of interest. An attempt to clean the sample of uninvolved background components was conducted by pH adjustments, filtration techniques and the use of C-18 cartridges. Apparently, by lowering the pH to 3 the toxicant absorbs onto particles that can be filtered. When pH was raised back up to the initial pH without filtering, the toxicant moved back into solution.

Early GC/MS analyses of toxic fractions revealed the presence of caprolactam in some preparations, but not all. More recently, side-by-side analyses of duplicate preparations of 80% methanol elutions of Hydrophilic Lipophilic Balance (HLB) solid-phase cartridge extractions of toxic pore water exhibited a large caprolactam peak in one preparation and no caprolactam peak in the duplicate. This led us to speculate that caprolactam instability in the

pore water matrix might be related to the inconsistency between the two preparations. However, since the HLB SPE procedure consistently removed toxicity and caprolactam was the only major peak observed in the cartridge elution, we tested caprolactam for toxicity in both the clean seawater and in pore water which had been previously rendered non-toxic by low pH HLB SPE clean-up. Caprolactam was much more toxic (approximately one order of magnitude) in the cleaned-up pore water than in clean sea water. This pattern was also observed for the toxic materials eluted from the HLB clean-up cartridge. Since the unknown toxicant from the cartridge elution and caprolactam shared this common characteristic, we decided to examine the original toxic pore water for caprolactam and related compounds. Caprolactam can be analyzed by GC/MS but polar products resulting from the opening of the ring structure as well as related polymeric compounds are not amenable to GC/MS analysis and this may explain why our analyses of the toxic fractions sometimes contained caprolactam and sometimes did not. Consequently, TRAC Laboratories sub-contracted with Dr. Robert Voyksner, LCMS Limited, to examine selected samples by LC/MS. To date, Dr. Voyksner's work has demonstrated (1) the 80% methanol toxic fraction eluted from the HLB SPE cartridge shows the presence of LC/MS peaks with mass spectral characteristics consistent with the open ring structure of caprolactam and related polymeric materials (2) these caprolactam-related peaks are also present in the original toxic pore water and (3) these caprolactam-related peaks are absent in the non-toxic pore water resulting from HLB SPE clean-up. Analysis of HLB-cleaned-up pore water spiked with caprolactam revealed only caprolactam; spiking did not result in the formation of the caprolactam-related peaks found in the original toxic pore water and the toxic fraction eluted from the HLB cartridge.

Our interpretation of the results above is that there is evidence that caprolactam-related substances are contributing to the toxicity observed in the pore water. This toxic effect is observed only in combination with other contributing factors which remain in the pore water after HLB SPE treatment at low pH. These factors apparently interact with either spiked caprolactam or the HLB 80% toxic fraction to produce a toxic effect which is absent when either of these materials is tested for toxicity in clean sea water. We also have evidence that the increased toxicity of caprolactam seen in association with the cleaned-up pore water is not dependent upon the direct action of the pore water on the caprolactam (i.e. inducing ring opening or polymerization) since the increased toxicity of caprolactam can be induced by independent exposures to cleaned-up pore water (non-toxic by itself) and caprolactam in clean seawater (non-toxic by itself; see the results of the dual exposure experiment). This leads us to believe that the toxic interaction of caprolactam is likely manifested regardless of the integrity of the ring structure and state of polymerization in the environmental exposure (within the limits observed in the toxic pore water) and probably results from a metabolized form of the material that may be common to any form of the material that is initially taken up by the test organism.

Caprolactam is primarily used in the manufacture of Nylon 6 and other synthetic fibers. Caprolactam is also used in brush bristles, textile stiffeners, film coatings, synthetic leather, plastics, plasticizers, paint vehicles, cross-linking for polyurethanes, and in the synthesis of lysine (USEPA 1988, USDHHS 1993).

Parsons' recommendation is continued periodic monitoring of sediment toxicity. In addition, effluent and sludge sampling should be performed on potential sources followed by the development of a TMDL for caprolactam.

SECTION 9 REFERENCES

- TRAC Laboratories, 2001. Aquatic Toxicity Identification Evaluation, Phase I, of Sediment Pore Water from Segment 1007A Using *Leptocheirus plumulosus* and *Mysidopsis bahia* TIE Test Report, August 2001, Pensacola, Florida.
- TRAC Laboratories, 2001. 10 Day Sediment Toxicity Screens Exposing *Leptocheirus plumulosus* and *Neanthes arenaceodentata* to Sediments from Segments 1007A and 2201, August 2001, Pensacola, Florida.
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- U.S. Environmental Protection Agency. Health and Environmental Effects Profile for Caprolactam. ECAO-CIN-G018. Environmental Criteria and Assessment Office, Office of Health and Environmental Assessment, Office of Research and Development, Cincinnati, OH. 1988.
- U.S. Department of Health and Human Services. Hazardous Substances Data Bank (HSDB, online database). National Toxicology Information Program, National Library of Medicine, Bethesda, MD. 1993.

**APPENDIX A
HISTORICAL DATA**

Station	Long Description	Data	Total
11299	ACID VOLATILE SULFIDE (AVS), (MMOL/KG)	Min of Value	1.49
		Max of Value	12.6
		Average of Value	7.0
		Count of Value	2
	ALKALINITY, TOTAL (MG/L AS CaCO3)	Min of Value	114
		Max of Value	182
		Average of Value	140.4
		Count of Value	17
	ALUMINUM, DISSOLVED (UG/L AS AL)	Min of Value	0
		Max of Value	97.8
		Average of Value	16.3
		Count of Value	6
	ARSENIC, DISSOLVED (UG/L AS AS)	Min of Value	0
		Max of Value	5.1
		Average of Value	2.9
		Count of Value	6
	CADMIUM, DISSOLVED (UG/L AS CD)	Min of Value	0
		Max of Value	0
		Average of Value	0.0
		Count of Value	6
CALCIUM, DISSOLVED (MG/L AS CA)	Min of Value	34.9	
	Max of Value	120	
	Average of Value	64.6	
	Count of Value	6	
CARBON, TOTAL ORGANIC (MG/L AS C)	Min of Value	0	
	Max of Value	17	
	Average of Value	9.1	
	Count of Value	17	
CHLORIDE (MG/L AS CL)	Min of Value	30.3	
	Max of Value	4790	
	Average of Value	1778.3	
	Count of Value	17	
CHLOROPHYLL-A UG/L SPECTROPHOTOMETRIC ACID. METH	Min of Value	0	
	Max of Value	20	
	Average of Value	4.3	
	Count of Value	17	
CHROMIUM, DISSOLVED (UG/L AS CR)	Min of Value	0	
	Max of Value	0	
	Average of Value	0.0	
	Count of Value	6	
COPPER, DISSOLVED (UG/L AS CU)	Min of Value	0	
	Max of Value	3	
	Average of Value	1.0	
	Count of Value	6	
FECAL COLIFORM, MF AGAR (COLONIES/100 ML)	Min of Value	10	
	Max of Value	58000	
	Average of Value	6614.7	
	Count of Value	17	
FECAL COLIFORM, MEMBR FILTER, M-FC BROTH, #/100ML	Min of Value	4800	
	Max of Value	5300	
	Average of Value	5050.0	
	Count of Value	2	
HARDNESS, DISSOLVED, CALCULATED (MG/L AS CaCO3)	Min of Value	149	
	Max of Value	1190	
	Average of Value	483.2	
	Count of Value	6	
LEAD, DISSOLVED (UG/L AS PB)	Min of Value	0	
	Max of Value	0	
	Average of Value	0.0	
	Count of Value	6	
MAGNESIUM, DISSOLVED (MG/L AS MG)	Min of Value	9.17	
	Max of Value	216	
	Average of Value	78.2	
	Count of Value	6	
NICKEL, DISSOLVED (UG/L AS NI)	Min of Value	0	
	Max of Value	0	
	Average of Value	0.0	
	Count of Value	6	
NITRITE PLUS NITRATE, TOTAL 1 DET. (MG/L AS N)	Min of Value	0	
	Max of Value	2.14	
	Average of Value	1.1	
	Count of Value	12	
NITROGEN, AMMONIA, TOTAL (MG/L AS N)	Min of Value	0.46	
	Max of Value	12	
	Average of Value	5.3	
	Count of Value	17	
NITROGEN, KJELDAHL, TOTAL, (MG/L AS N)	Min of Value	1.39	
	Max of Value	18.2	
	Average of Value	7.4	
	Count of Value	17	
NO2 PLUS NO3-N, TOTAL, WHATMAN GF/F FILT (MG/L)	Min of Value	0.245	
	Max of Value	1.3	
	Average of Value	0.6	
	Count of Value	5	

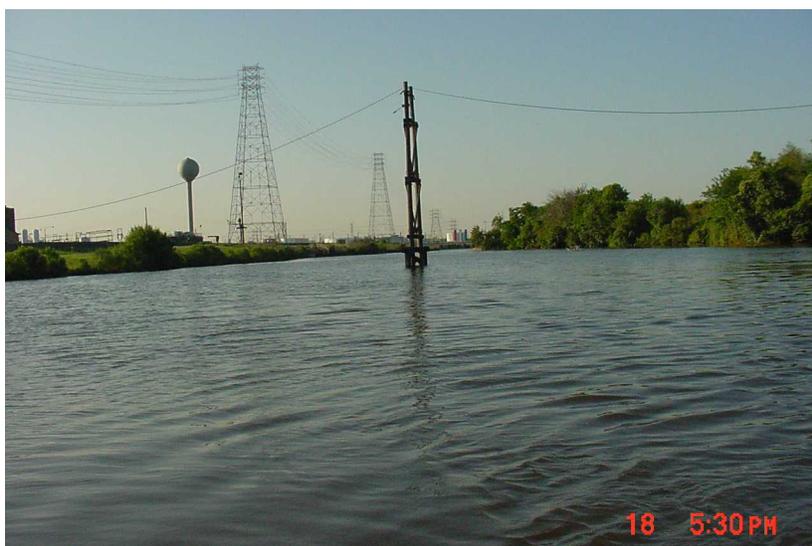
11299	OXYGEN, DISSOLVED (MG/L)	Min of Value	0
		Max of Value	9.3
		Average of Value	4.2
		Count of Value	46
	PH (STANDARD UNITS)	Min of Value	6.1
		Max of Value	7.9
		Average of Value	7.1
		Count of Value	43
	PHEOPHYTIN-A UG/L SPECTROPHOTOMETRIC ACID. METH.	Min of Value	0
		Max of Value	13.4
		Average of Value	4.6
		Count of Value	17
	PHOSPHORUS, DISSOLVED ORTHOPHOSPHORUS(MG/L AS P)	Min of Value	0.44
		Max of Value	1.03
		Average of Value	0.7
		Count of Value	11
	PHOSPHORUS, TOTAL, WET METHOD (MG/L AS P)	Min of Value	0.52
		Max of Value	1.57
		Average of Value	0.9
		Count of Value	17
PHOSPHORUS,IN TOTAL ORTHOPHOSPHATE (MG/L AS P)	Min of Value	0.43	
	Max of Value	1.16	
	Average of Value	0.7	
	Count of Value	4	
RESIDUE, TOTAL NONFILTRABLE (MG/L)	Min of Value	13	
	Max of Value	48	
	Average of Value	23.5	
	Count of Value	17	
RESIDUE, VOLATILE NONFILTRABLE (MG/L)	Min of Value	3	
	Max of Value	16	
	Average of Value	6.5	
	Count of Value	17	
RESIDUE,TOTAL FILTRABLE (DRIED AT 180C) (MG/L)	Min of Value	250	
	Max of Value	10200	
	Average of Value	3482.4	
	Count of Value	16	
SALINITY - PARTS PER THOUSAND	Min of Value	0	
	Max of Value	14.1	
	Average of Value	4.0	
	Count of Value	46	
SELENIUM, DISSOLVED (UG/L AS SE)	Min of Value	0	
	Max of Value	3.02	
	Average of Value	0.5	
	Count of Value	6	
SELENIUM, TOTAL (UG/L AS SE)	Min of Value	0	
	Max of Value	3.23	
	Average of Value	0.5	
	Count of Value	6	
SILVER, DISSOLVED (UG/L AS AG)	Min of Value	0	
	Max of Value	0	
	Average of Value	0.0	
	Count of Value	6	
SIMULTANEOUSLY EXTRACTED METALS,SUM(SEM) (MMOL/K	Min of Value	1.31	
	Max of Value	3.34	
	Average of Value	2.3	
	Count of Value	2	
SPECIFIC CONDUCTANCE,FIELD (UMHOS/CM @ 25C)	Min of Value	339	
	Max of Value	23300	
	Average of Value	7786.5	
	Count of Value	46	
SULFATE (MG/L AS SO4)	Min of Value	31	
	Max of Value	775	
	Average of Value	316.3	
	Count of Value	17	
TEMPERATURE, WATER (DEGREES CENTIGRADE)	Min of Value	13.1	
	Max of Value	34.8	
	Average of Value	22.4	
	Count of Value	46	
TRANSPARENCY, SECCHI DISC (METERS)	Min of Value	0.26	
	Max of Value	0.87	
	Average of Value	0.5	
	Count of Value	17	
ZINC, DISSOLVED (UG/L AS ZN)	Min of Value	7	
	Max of Value	21	
	Average of Value	13.2	
	Count of Value	6	
11299 Min of Value		0	
11299 Max of Value		58000	
11299 Average of Value		975.2	
11299 Count of Value		596	
Total Min of Value		0	
Total Max of Value		58000	
Total Average of Value		975.2	
Total Count of Value		596	

**APPENDIX B
PHOTO LOG**

VINCE BAYOU



Vince Bayou, Station 11299, 300 yards upstream of Houston Ship Channel confluence (2001)



Vince Bayou, Station 11299 (2001).

VINCE BAYOU



Vince Bayou, Station 14368, downstream of the City of Pasadena WWTP Outfall, 32 Feet Downstream of West Richey Street (2001).



Segment 1007A, Station 14371, Little Vince Bayou at West Richey Street in Pasadena, Texas looking downstream from sampling location (2001)

VINCE BAYOU



Segment 1007A, Station 14371, Little Vince Bayou at West Richey Street in Pasadena, Texas looking upstream at the sampling location (2001).

**APPENDIX C
TOXICITY TESTS LAB REPORTS AND DATA SUMMARY**

**Aquatic Toxicity Identification Evaluation, Phase I,
of Sediment Pore Water from Segment 1007A
Using *Leptocheirus plumulosus* and *Mysidopsis bahia***

TIE Test Report

Submitted to:

**Randy Palachek
Parsons ES
8000 Centre Park Drive, Suite 200
Austin, Texas 78754-5140**

Submitted by:

**TRAC Laboratories
14 South 2nd Street
Pensacola, Florida 32507
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Florida Department of Health and Rehabilitative Services
Certification Number E81181

**Project: TNRCC TMDL
Subcontract Number: 739598.3000-00**

August 2001

INTRODUCTION

Based on significant toxicity in whole sediment toxicity screenings of sample 14368, from segment 1007A (Vince Bayou), phase 1 toxicity identification evaluation (TIE) procedures were conducted with sediment pore water at TRAC Laboratories, Pensacola, Florida. The pore water was tested unaltered, and after various manipulations, an attempt was made to define the toxicant, or group of toxicants responsible for observed toxicity to *Leptocheirus plumulosus* and *Mysidopsis bahia*. Three separate sets of phase 1 procedures were conducted on 20 June, 14 July and 16 July, with two polymeric adsorbent treatments 15 and 20 August 2001. Test duration was 96 hours. Only *L. plumulosus* was tested in the first series dated 20 June. Both *L. plumulosus* and *M. bahia* were tested in each series thereafter. All data related to this project are stored at TRAC and presented as tables 1-3.

MATERIALS AND METHODS

Test Material

Pore water from sediment sample 14368 was obtained by centrifuging the whole sediment at approximately 2000 rpm for 45 minutes. Centrifugation was performed in a walk in refrigerator at 4°C. The overlying water was then decanted into pre-cleaned glass bottles and sealed until use.

Test Animals

L. plumulosus were supplied by Chesapeake Cultures, Inc., Hayes, Virginia and were 2-4 mm in length.

Mysidopsis bahia were obtained from TRAC's marine culture facilities and were 3 days old at test initiation.

Test Water

The dilution and control water was artificial seawater at a salinity of 20 parts per thousand (1) made of Forty Fathoms⁷ marine salt mix and deionized water. Artificial salts were used because of the relatively low salinity of the sediment pore water (~7 ppt).

Test Conditions - General

Methods for the procedures were based on the Marine Toxicity Identification Evaluation: Phase 1 Guidance, (EPA/600/R-96/054) and "Methods for Aquatic Toxicity Identification Evaluation: Phase I, Toxicity Characterization Procedures", second edition (EPA-600/6-91/003). Test chambers for all tests were 50 milliliter (ml) glass beakers. Test volume was 20 mL for all tests. Test temperatures for all treatments were 20 ± 2 °C.

RESULTS AND DISCUSSION

Statistical Analysis

Based on results of initial and baseline tests, the LC50 values (the test material concentration producing 50% mortality after a specified period of exposure) and their associated 95% confidence limits were calculated. The computer program estimated LC50 values using the following statistical methods: probit analysis or the Trimmed Spearman-Kärber Method. The method selected for reporting the results of the test data was determined by the characteristics of the data, that is, the presence or absence of 0% and 100% mortality and the number of concentrations in which mortality between 0 and 100% occurred.

Initial and Baseline Toxicity Tests

Before TIE procedures were initiated, an initial toxicity test of the sediment pore water was conducted to determine whether or not the toxicity observed in the whole sediment could be retrieved in the pore water. The initial test with *L. plumulosus* yielded an LC50 of 28.0% pore water. This effect was sufficient evidence to warrant initiation of TIE procedures. With each series of procedures, a baseline test was conducted concurrently to assess the consistency of toxicity.

Aeration Test

Two hundred ml of ASW and 200 ml of salinity adjusted pore water were aerated for one hour and then tested in 25, 50 and 100% dilutions.

Filtration Test

Glass fiber filters (1.0 micrometer nominal pore size) were prepared for the test by filtering twice with 50 ml of deionized water. Then 300 ml of ASW followed by 400 ml of salinity adjusted pore water were passed through the prepared filter. The filtered ASW and pore water were then used in the toxicity test at dilutions of 25, 50 and 100% effluent. A 200 ml portion of filtered pore water was set aside and used in the SPE-C₁₈ procedure.

Oxidant Reduction Test

Sodium thiosulfate (15 g/l Na₂S₂O₃ stock solution) was added to three dilutions of 25, 50 and 100% salinity adjusted pore water and an associated blank at a rate of 340 Φ L per 100 ml test volume, to produce a final concentration of 50.0 milligrams of Na₂S₂O₃ per liter (mg/l).

Sodium Ethylenediaminetetraacetic acid (EDTA) Chelation Test

Sodium EDTA (25 g/l stock solution) was added to three dilutions of 25, 50 and 100% salinity adjusted pore water and an associated blank at a rate of 240 Φ l per 100 ml test volume to produce a final concentration of 60 mg/l.

SPE-C₁₈ Extraction Test

The SPE column was conditioned with 25 ml HPLC grade methanol followed by 25 ml deionized water. Then 300 ml of filtered ASW were passed through the column. The first 25 ml were discarded and the next 275 ml were used as dilution water and blank for the toxicity test. The filtered salinity adjusted pore water (200 ml) was then passed through the same SPE column. The first 25 ml were discarded, the next 175 ml were used in the toxicity test.

SPE Elution

The SPE column used above was allowed to dry after the 200 ml of pore water had passed through. The column was then eluted with 1.0 ml methanol resulting in a methanol fraction with 200x the toxicity concentration of the untreated pore water. The methanol fraction was then added to dilution water at a rate of 0.3, 0.5, 1.0 and 2.0x the original toxicity.

Amberlite XAD Resin

The exchange resin (Amberlite XAD-4, polymeric adsorbent) was placed in a column and rinsed with 5 bed volumes of deionized water and then charged with 5 bed volumes of methanol. Next, 200 ml deionized water were passed through the column at a rate of ~5 bed volumes per hour. The unaltered pore water was then passed through at the above rate. The post-column deionized water and pore water were then salinity adjusted.

CONCLUSION

Comparison of treated fractions with initial and baseline tests indicated that SPE column removed more than 50% of the toxicity observed in the baseline test during the first set of procedures with *L. plumulosus*. The subsequent methanol elution recovered approximately 70% of the original toxicity. However, this was not the case in subsequent testing with either *L. plumulosus* or *M. bahia*. No toxicity removal was observed in the additional two sets of phase 1 procedures. Based on above results, an additional procedure was employed by passing the pore water through Amberlite XAD-4 resin. In two separate procedures, the resin column effectively removed toxicity. Possible contaminants suspected at this point in the study are petroleum hydrocarbons which is based in part on the physical and aromatic nature of the whole sediment. Work involving additional passes through the polymeric adsorbent and subsequent methanol elutions is currently in progress.

REFERENCES

U.S. Environmental Protection Agency, 1993. Marine Toxicity Identification Evaluation (TIE) Phase 1 Guidance Document, EPA/600/R-96/054, September 1996, Narragansett, Rhode Island.

U.S. Environmental Protection Agency, 1993. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms, EPA/600/4-90/027F, August 1993, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1991. Methods for Aquatic Toxicity Identification Evaluations. Phase I Toxicity Characterization Procedures, Second edition, EPA-600/6-91/003, February 1991, Duluth, Minnesota.

**10 Day Sediment Toxicity Screens Exposing
Leptocheirus plumulosus and *Neanthes arenaceodentata*
to Sediments from Segments 1007A and 2201**

Submitted to:

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Florida Department of Health and Rehabilitative Services
Certification Number E81181

**Project: TNRCC TMDL
Subcontract Number: 739598.3000-00
Sampling Event Numbers: 1-6**

August 2001

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1. Reference Toxicant (SDS) vs *Leptocheirus plumulosus*.
2. Reference Toxicant (SDS) vs *Neanthes arenaceodentata*.

Data Files

1. Total Ammonia Measurements from Interstitial Water.
2. Summary of Sampling Event 1: Sample Collection Dates, Test Dates and Survival Data.
3. Summary of Sampling Event 2: Sample Collection Dates, Test Dates and Survival Data.
4. Summary of Sampling Event 3: Sample Collection Dates, Test Dates and Survival Data.
5. Summary of Sampling Event 4: Sample Collection Dates, Test Dates and Survival Data.
6. Summary of Sampling Event 5: Sample Collection Dates, Test Dates and Survival Data.
7. Summary of Sampling Event 6: Sample Collection Dates, Test Dates and Survival Data.

Toxicity Test Summary Sheet

Client: Parsons ES

Subcontract Num: 739598.3000-00

Study Director: Dan Johnson

Test Material: Whole sediment samples from Segments 1007A(Vince Bayou) and 2201 (Arroyo Colorado Tidal).

Date Materials Collected: 20 April through 10 August 2001

Date of Tests: 4 May through 27 August 2001

Test Conditions: Static, 10 day duration.

Test Procedures: 1994. U.S. EPA. (EPA/600/R-94/025). Methods for Assessing the Toxicity of Sediment-associated Contaminants With Estuarine and Marine Amphipods.

1998. U.S. EPA. (EPA 823-B-98-004). Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. – Testing Manual.

Test Organisms: *Neanthes arenaceodentata* and *Leptocheirus plumulosus*

Source: *N. arenaceodentata* were supplied by Dr. Don Reish, California State, Long Beach. *L. plumulosus* were supplied by Chesapeake Cultures.

Control and Dilution Water: Natural sea water at a salinity of 30 parts per thousand (ppt) for *N. arenaceodentata* tests and 20 ppt for *L. plumulosus* tests.

Exposure Concentration: 100% sediment.

Effect Criteria: Survival.

INTRODUCTION

Sediments samples tested in this study are part of the TNRCC TMDL study. This study represents testing of 6 sampling events. Sediment samples from segment 1007A and 2201 were received from Parsons personnel and tested at TRAC Laboratories Inc., Pensacola, Florida, to determine acute effects to *Neanthes arenaceodentata* and *Leptocheirus plumulosus*. The criterion for effect was survival. Tests were conducted from 4 May through 27 August 2001. All raw data related to this study are stored at TRAC. Data are presented as hard copy data files in Excel worksheet format.

MATERIALS AND METHODS

Test Material

Sediment samples were obtained from Parsons by TRAC personnel via Federal Express. The samples were contained in 3.5 gallon plastic buckets or 1 gallon high density polyethylene jars.

A chain of custody form accompanied each sediment shipment. Sample label information was recorded in the sediment receiving log as was arrival temperature and the date received at TRAC Laboratories in Pensacola, Florida.

Sample identification, approximate volume, sieve size used for press-sieving, date of receipt and processing data were recorded in the sample log prior to test initiation.

Four samples were tested from each segment. Samples from segment 1007A (Vince Bayou) were labeled as: 14368, 11299, 14371 and a duplicate. Samples from segment 2201 (Arroyo Colorado Tidal) were labeled as: 13071, 13782, 13072 and 2201-Duplicate. Sampling and testing dates are included in Data files 2-7.

Control Water

Natural sea water collected from the Gulf of Mexico was cleaned and conditioned by running it through a sand filter continuously times. The conditioned water was then adjusted to salinities of 30ppt for *N. arenaceodentata* exposures and 20ppt for *L. plumulosus* exposures using deionized water. The salinity adjusted and conditioned water was then acclimated to the test temperature of 20°C. This treated water was then used for overlying water in the sediment exposures and positive control reference toxicant tests.

Test Animals

Neanthes arenaceodentata were obtained from Dr. Don Reish, California State University, Long Beach. The *N. arenaceodentata* were juveniles, 2-3 weeks in age.

Leptocheirus plumulosus were obtained from Chesapeake Cultures, Inc., Hayes, Virginia and were 2-4 mm in length.

Animals were shipped (via overnight courier) in their native sediment with overlying natural sea water. Upon arrival, temperature and salinity were noted, water was exchanged and renewed with fresh control water for acclimation to test conditions.

Test Conditions

Tests were conducted in a temperature-controlled (20±2°C) environmental chamber under a 24-hour light photo period. Daily animal observations were conducted and any dead organisms or molts were removed. Live *L. plumulosus* and *N. arenaceodentata* found floating during the test period were gently submerged with a pipet and allowed a 15 minute period for burrowing before replacing airlines. Each replicate was gently aerated (~100 bubbles/minute) throughout the 10-day test, and frequent daily checks insured airlines were aerating the water column.

Sediment Preparation

Sediment samples were press sieved through a 1.0 mm stainless steel sieve to remove particles and predators which might interfere with the testing process. The complete contents of each sample, including the sediment porewater, were captured and used to aid the sample in passing through the sieve.

Following the press sieving step and prior to test initiation, sediments were homogenized by blending the sediment 3 - 5 minutes with a stainless steel spoon or mechanical paddle.

Once homogenized, the sediments were measured out in 200 ml aliquots and transferred to randomly assigned one liter glass jars. Six replicates were measured out for each sediment sample. Five replicates were set up for the 10 day exposures and the sixth replicate was used to measure porewater ammonia.

Test Initiation

The randomly assigned jars containing exposure sediments were placed in the environmental chamber in numerical order. Seven hundred fifty ml of natural seawater diluted to 30ppt or 20ppt were carefully poured over a turbidity reducer to fill the test vessel. The exposure vessels were then allowed to settle 14-16 hours before test organisms were introduced.

After the settling period, physical parameters (pH, DO, temperature and salinity) were monitored and recorded on the physical data sheets prior to introduction of test organisms.

Once acclimated to laboratory conditions (Salinity, temperature and lighting), test organisms were removed from the native sediment and prepared for test sorting. *L. plumulosus* 2 - 4 mm in length were selected individually with a medium bore pipette and transferred to a 30 ml beaker containing prepared 20ppt seawater. Ten *L. plumulosus* were collected in each beaker and observed for good color, full gut, and size.

Two beakers of 10 animals were combined and added in random sequence to each exposure vessel, releasing 20 *L. plumulosus* into the sediment exposure. Two extra beakers with ten animals each were randomly selected for size measurements at test initiation and recorded on the day 0 setup sheet.

N. arenaceodentata were gently agitated with a pipet to remove them from tubes. Five worms were placed in a 30 ml beaker containing 10 ml of 30ppt seawater and then added in random sequence to each sediment replicate.

One hour after addition of test organisms, each sediment replicate was examined to ensure all animals were established in the sediment and air lines replaced.

Ammonia Analysis

The sixth replicate was brought into the environmental chamber with the 10-day sediment exposures and treated the same (aerated) as the other five replicates. A fritted glass sampler was placed approximately 2.0 cm into the sediment prior to addition of overlying water.

Hydrostatic pressure forced interstitial water into the sampler after passing through a 1.0 Φ pore glass fiber filter (Gelman Sciences, type A/E) which was wrapped around the fritted portion of the sampler to prevent clogging.

Ten to twenty ml of interstitial water were removed from the neck of the fritted sampler

16-20 hours into the test (day 0). Temperature, salinity and pH measurements were recorded prior to the total ammonia analysis. The Orion 250A pH/ISE meter and 95-12 gas-sensing ammonia electrode measured the ammonia ion after conversion to ammonia gas. Sample color and turbidity do not affect measurements by this method. Other ionic species do not interfere with this probe. The ammonia-selective electrode method (4500-NH₃, ASTM 13th Edition, 1992) was followed by raising each sample's pH to above 11 with 10 N NaOH, and measuring ammonia across the probe's membrane as it is converted from aqueous NH₃ and NH₄⁺. Potentiometric measurements were recorded for each sample in millivolts (mV) and extrapolated to mg/L of total ammonia from a standard curve constructed with each test series.

A standard ammonia curve was constructed for each test series using four standards (0.1, 1.0, 10 and 100 mg/L) diluted from a 1000 mg/L stock of ammonia. The log transformed standard concentrations were entered into a linear regression with their potentiometric responses (mV) yielding correlations of 98 to 100%. All sample measurements were then entered into this same formula to retrieve a total ammonia measurement in mg/L.

In each test series, DI water blanks were measured to calibrate a zero-ammonia point for the probe. When enough sample was available, a sample was duplicated to measure variation. Total ammonia concentrations for each sample ID are presented as Data File 1.

Test Termination

Sediment tests were terminated after 10 days. Sediment vessels were removed in numerical order from the environmental chamber animal recovery. Sediments and overlying water were passed through a 250 micron mesh sieve which was designed to capture the test organisms while allowing some sediments to pass through. Because of time constraints due to the number of exposure replicates, all material retained in the sieve was preserved in a 70% ethanol solution with rose Bengal stain. Organisms were later recovered and counted from the preserved exposures and recorded on the breakdown sheet. Once all exposure replicates were broken down and picked, the data was grouped according to the sediment ID. The randomization sheet was used to unscramble the exposure vessel numbers which in turn accounted for the five replicates. The descrambling sheet provides sample ID matched to randomized vessel numbers.

Reference Toxicant (Positive Control)

A positive control reference toxicant test was conducted with each shipment of test organisms. The reference toxicant used was sodium dodecyl sulfate (SDS) and the test was conducted in accordance with EPA/600/4-90/027F and EPA/620/R-95/008. Values were plotted to determine if the results were within prescribed limits. In this technique, a running plot is maintained for the toxicity values from successive tests with a given reference toxicant. For regression analysis results (i.e. LC50s), the mean (\bar{x}) and upper and lower control limits ($\sqrt{2}SD$) are recalculated with each successive point until the statistics stabilize. Control charts are presented as figures 1 and 2.

Reference Sediment (Negative Control)

All sediment tests were accompanied by a negative control reference sediment test. Replication of these control samples were the same as for the study site samples (five exposure replicates; one replicate for ammonia analysis). Negative control reference sediment (C-17) was obtained by TRAC personnel from Perdido Bay at position 30° 19.753' N, 087° 27.869' W. The principal reason for selecting C-17 as a suitable reference sediment is in the toxicological data base developed for *A. abdita* by USEPA=s EMAP Louisianian Province in previous years (1990-1994).

Statistical Analysis

The sediment samples were tested in groups of six and seven with a common negative control. ANOVA and Dunnett=s multiple range tests were used to identify samples in which survival was statistically lower from the negative controls. The survival proportions were transformed using Arcsin ($\sqrt{p_i}$) where p_i = proportion surviving in replicate I. The data was then examined for homogeneity of variance and departure from normality using Bartlett=s and Shapiro-Wilks tests, respectively. If the data were normally distributed and the variances homogenous, the transformed data was analyzed with a one-way ANOVA. If the F test of the ANOVA was significant ($p < 0.05$), differences between the mean of each sample were compared with the control using Dunnett=s test. Dunnett=s test is specifically intended to compare treatment means with a control. If the F test in the ANOVA is not significant, no further analysis is performed, and the sample means are then statistically similar to the control. When the assumptions of normality and variance homogeneity cannot be verified, Steel=s Many One Rank Test is used to examine differences between the control and each mean. Steel=s Test is specifically intended to examine differences between treatments and a control when assumptions of normality and variance homogeneity cannot be verified.

RESULTS AND DISCUSSION

Survival Information

Survival data was calculated for each replicate as percent survival; mean and standard deviation were calculated for each sample.

Statistical analysis was performed as defined above. Based on data analysis, significant reductions in survival of both species were measured in sample 14368 (segment 1007A, Vince Bayou) only. Whole sediment tests of samples from segment 1007A were conducted in the first two sampling events only. Once consistent toxicity was observed in sample 14368 from segment 1007A, testing efforts for that site shifted to TIE procedures involving porewater. However, whole sediment testing of samples from site 2201 continued through 6 events with no observed toxicity. Complete survival data are displayed in Data Files 2-7.

Physical Parameters

Salinity, dissolved oxygen and pH were measured in each test replicate on days 0, 4, 7 and 10. Temperature was measured in each exposure replicate daily and were consistently 20°C \pm 2°C. Dissolved oxygen levels were maintained with gentle aeration throughout the ten day exposure and levels stayed above 60% of saturation.

DATA FILE 1

**Total Ammonia
Measurements from Interstitial Water**

DATA FILE 2

**Summary of Sampling Event 1:
Sample Collection Dates, Test Dates and Survival Data**

DATA FILE 3

**Summary of Sampling Event 2:
Sample Collection Dates, Test Dates and Survival Data**

DATA FILE 4

**Summary of Sampling Event 3:
Sample Collection Dates, Test Dates and Survival Data**

DATA FILE 5

**Summary of Sampling Event 4:
Sample Collection Dates, Test Dates and Survival Data**

DATA FILE 6

**Summary of Sampling Event 5:
Sample Collection Dates, Test Dates and Survival Data**

DATA FILE 7

**Summary of Sampling Event 6:
Sample Collection Dates, Test Dates and Survival Data**

**Appendix C
Toxicity Tests Lab Reports
1007A N. arenaceodontata**

Segment 1007A, Vince Bayou									
Survival of <i>Neanthes arenaceodontata</i> in Ten-day Sediment Exposures Conducted 4-14 May 2001.									
Samples collected April 18-19, 2001									
All statistical analyses were performed using TOXSTAT and followed USEPA guidelines for whole effluent toxicity tests									
Sample ID	Number Surviving	Percent Survival	Mean % Survival	Standard Deviation	p Value	Statistical Difference	CV (%)		
C17 (Control)	5	100	100	0.00	0.05	N/A	0.0		
11299-1	5	100	100						
14368-1	4	80	96	8.94	0.05	NO	9.3		
14368DUP-1	0	0	28	38.99	0.05	YES	139.2		
14371-1	5	100	92	10.95	0.05	NO	11.9		
Three stations total.									
11299: Vince Bayou 300 yards upstream of the Houston Ship Channel Confluence.									
14368: Vince Bayou downstream of the City of Pasadena WWTP Outfall, 0.1 km downstream of West Ritchey Street.									
14371: Little Vince Bayou at West Ritchey Street in Pasadena, TX									

**Appendix C
Toxicity Tests Lab Reports
1007A N. arenaceodontata**

Segment 1007A, Vince Bayou									
Survival of <i>Neanthes arenaceodontata</i> in Ten-day Sediment Exposures Conducted 5-15 June 2001.									
Samples collected May 24, 2001									
All statistical analyses were performed using TOXSTAT and followed USEPA guidelines for whole effluent toxicity tests									
Sample ID	Number Surviving	Percent Survival	Mean % Survival	Standard Deviation	p Value	Statistical Difference	CV (%)		
C17 (Control)	5	100	96	8.94	0.05	N/A	9.3		
	5	100							
	4	80							
	5	100							
	5	100							
11299-2	4	80	96	8.94	0.05	NO	9.3		
	5	100							
	5	100							
	5	100							
	5	100							
14368-2	2	40	32	22.80	0.05	YES	71.3		
	2	40							
	3	60							
	0	0							
	1	20							
14371-2	4	80	92	10.95	0.05	NO	11.9		
	5	100							
	5	100							
	4	80							
	5	100							
Three stations total.									
11299: Vince Bayou 300 yards upstream of the Houston Ship Channel Confluence.									
14368: Vince Bayou downstream of the City of Pasadena WWTP Outfall, 0.1 km downstream of West Ritchey Street.									
14371: Little Vince Bayou at West Ritchey Street in Pasadena, TX									

**Appendix C
Toxicity Tests Lab Reports
1007A N. arenaceodontata**

Segment 1007A, Vince Bayou									
Survival of <i>Neanthes arenaceodontata</i> in Ten-day Sediment Exposures Conducted 29 June - 9 July 2001.									
Samples collected June 14, 2001									
All statistical analyses were performed using TOXSTAT and followed USEPA guidelines for whole effluent toxicity tests									
Sample ID	Number Surviving	Percent Survival	Mean % Survival	Standard Deviation	p Value	Statistical Difference	CV (%)		
C17 (Control)	5	100	92	10.95	0.05	N/A	11.9		
	5	100							
	5	100							
	4	80							
	4	80							
14368-3	5	100	92	10.95	0.05	NO	11.9		
	4	80							
	5	100							
	4	80							
	5	100							
Three stations total.									
11299: Vince Bayou 300 yards upstream of the Houston Ship Channel Confluence.									
14368: Vince Bayou downstream of the City of Pasadena WWTP Outfall, 0.1 km downstream of West Ritchey Street.									
14371: Little Vince Bayou at West Ritchey Street in Pasadena, TX									

**Appendix C
Toxicity Tests Lab Reports
1007A N. arenaceodontata**

Segment 1007A, Vince Bayou									
Survival of <i>Neanthes arenaceodontata</i> in Ten-day Sediment Exposures Conducted 9 - 19 October 2001.									
Samples collected July 19 and 26, 2001									
All statistical analyses were performed using TOXSTAT and followed USEPA guidelines for whole effluent toxicity tests									
Sample ID	Number Surviving	Percent Survival	Mean % Survival	Standard Deviation	p Value	Statistical Difference	CV (%)		
C17 (Control)	5	100	100	0.00	0.05	N/A	0.0		
	5	100							
	5	100							
	5	100							
	5	100							
14368-5	4	80	92	10.95	0.05	NO	11.9		
	5	100							
	5	100							
	4	80							
	5	100							
11299-5	5	100	100	0.00	0.05	NO	0.0		
	5	100							
	5	100							
	5	100							
	5	100							
Three stations total.									
11299: Vince Bayou 300 yards upstream of the Houston Ship Channel Confluence.									
14368: Vince Bayou downstream of the City of Pasadena WWTP Outfall, 0.1 km downstream of West Ritchey Street.									
14371: Little Vince Bayou at West Ritchey Street in Pasadena, TX									

Appendix C
 Toxicity Tests Lab Reports
 1007A N. arenaceodentata

Segment 1007A, Vince Bayou									
Survival of <i>Neanthes arenaceodentata</i> in Ten-day Sediment Exposures Conducted 9 - 19 November 2001.									
Samples collected October 30, 2001									
All statistical analyses were performed using TOXSTAT and followed USEPA guidelines for whole effluent toxicity tests									
Sample ID	Number Surviving	Percent Survival	Mean % Survival	Standard Deviation	p Value	Statistical Difference	CV (%)		
C17 (Control)	5	100	100	0.00	0.05	N/A	0.0		
	5	100							
	5	100							
	5	100							
	5	100							
14368-7	5	100	100	0.00	0.05	NO	0.0		
	5	100							
	5	100							
	5	100							
	5	100							

Appendix C
Toxicity Tests Lab Reports
1007A N. arenaceodentata

Segment 1007A, Vince Bayou									
Survival of <i>Neanthes arenaceodentata</i> in Ten-day Sediment Exposures Conducted 31 May - 10 June 2002.									
Samples collected April 3, 2002									
All statistical analyses were performed using TOXSTAT and followed USEPA guidelines for whole effluent toxicity tests									
Sample ID	Number Surviving	Percent Survival	Mean % Survival	Standard Deviation	p Value	Statistical Difference	CV (%)		
C17 (Control)	5	100	100	0.00	0.05	N/A	0.0		
	5	100							
	5	100							
	5	100							
	5	100							
14368-11	5	100	100	0.00	0.05	NO	0.0		
	5	100							
	5	100							
	5	100							
	5	100							
One station.									
14368: Vince Bayou downstream of the City of Pasadena WWTP Outfall, 0.1 km downstream of West Ritchey Street.									

**Appendix C
Toxicity Tests Lab Reports
1007A N. arenaceodontata**

Segment 1007A, Vince Bayou									
Survival of <i>Neanthes arenaceodontata</i> in Ten-day Sediment Exposures Conducted 3 - 13 May 2002.									
Samples collected April 23, 2002									
All statistical analyses were performed using TOXSTAT and followed USEPA guidelines for whole effluent toxicity tests									
Sample ID	Number Surviving	Percent Survival	Mean % Survival	Standard Deviation	p Value	Statistical Difference	CV (%)		
C17 (Control)	5	100	100	0.00	0.05	N/A	0.0		
14368-12	5	100	100	0.00	0.05	NO	0.0		
11301-12	5	100	100	0.00	0.05	NO	0.0		
11171-12	5	100	88	10.95	0.05	NO	12.4		
E. Jackson-12	5	100	100	0.00	0.05	NO	0.0		
Four stations total.									
14368: Vince Bayou downstream of the City of Pasadena WWTP Outfall, 0.1 km downstream of West Ritchey Street.									
11301, 11171 and E. Jackson locations unknown to TRAC.									

**Appendix C
Toxicity Tests Lab Reports
1007A N. arenaceodentata**

Segment 1007A, Vince Bayou									
Survival of <i>Neanthes arenaceodentata</i> in Ten-day Sediment Exposures Conducted 8 - 18 June 2002.									
Samples collected May 29, 2002									
All statistical analyses were performed using TOXSTAT and followed USEPA guidelines for whole effluent toxicity tests									
Sample ID	Number Surviving	Percent Survival	Mean % Survival	Standard Deviation	p Value	Statistical Difference	CV (%)		
C17 (Control)	5	100	100	0.00	0.05	N/A	0.0		
	5	100							
	5	100							
	5	100							
	5	100							
14368-11	5	100	100	0.00	0.05	NO	0.0		
	5	100							
	5	100							
	5	100							
	5	100							
One station.									
14368: Vince Bayou downstream of the City of Pasadena WWTP Outfall, 0.1 km downstream of West Ritchey Street.									

**APPENDIX D
CHEMICAL TESTS LAB REPORTS AND DATA SUMMARY**

**Aquatic Toxicity Identification Evaluation:
Investigation of Caprolactam-related Toxicity
in Sediment Pore Water from Site 14368, Segment 1007A
to *Mysidopsis bahia***

Investigation Report

Submitted to:

**Randy Palachek
Parsons ES
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Austin, Texas 78754-5140**

Submitted by:

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Florida Department of Health and Rehabilitative Services
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Project: TNRCC TMDL

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INTRODUCTION

The following outlines and discusses results of procedures employed after more conventional TIE procedures failed to produce a method by which sediment pore water toxicity could be both removed from the pore water and subsequently recovered by a method amenable to toxicant identification via analytical analysis. Experiments revealed (1) removal of the toxic component(s) was possible only under the condition of reduced pH (2) removal of suspended particulate matter prior to pH adjustment was necessary as toxic components appeared to sorb to suspended particulates at reduced pH (3) under conditions of reduced pH, pore water toxicant(s) consistently sorbed to an HLB SPE extraction cartridge but not to conventional C₁₈ SPE cartridges (4) the most toxic fraction was effectively recovered from the cartridge with an 80% methanol in water elution (5) toxicity recovery was observed only in cleaned-up pore water and not in clean seawater. After caprolactam was detected in GC/MS analyses of methanol eluates, an investigation was initiated to determine the toxicity of known concentrations of caprolactam and gain understanding of the caprolactam-related compounds observed in the pore water through LC/MS analysis.

MATERIALS AND METHODS

Pore Water Collection

Sediment pore water was obtained by centrifuging the whole sediment at approximately 2000 rpm for 45 minutes. Centrifugation was performed in a walk in refrigerator at 4°C. The overlying water was then decanted into pre-cleaned glass bottles and sealed until use.

Pore Water Preparation

Collected pore water was filtered through a type A/E glass fiber filter to remove suspended particulate matter prior to pH adjustment. The pore water was then adjusted to a pH of 3.0 with 1.0 N HCl. The pH adjusted pore water was then passed through a 6cc HLB SPE extraction cartridge (Waters Oasis®) charged with methanol. Post cartridge pore water was retained, and the cartridge was allowed to go to dryness. The cartridge was then eluted with various methanol/water dilutions. All pH adjusted blank and pore water treatments were returned to initial pH with 0.1N and 1.0N NaOH before dilution and addition of test organisms.

Test Animals

Mysidopsis bahia were obtained from TRAC's marine culture facilities and were 2 days old at test initiation.

Test Water

The dilution and blank water was artificial seawater at a salinity of 20 parts per thousand made of Forty Fathoms⁷ marine salt mix and deionized water. Artificial salts were used because of the relatively low salinity of the sediment pore water (~5 ppt).

Pore Water Tests

The sediment pore water was tested unaltered (baseline) and yielded an LC50 of 48.9% pore water (Table 1). Additional pore water was tested concurrently after being passed through an HLB SPE cartridge at reduced pH as described above. Survival data from this test are summarized in Table 2 and indicate a significant reduction of toxicity. Subsequent cartridge elution with varying dilutions of methanol in water yielded significant toxicant recovery in the 80% methanol fraction (Table 3). Two additional tests were conducted to examine the toxicity of known concentrations of caprolactam and to examine the differences, if any, of caprolactam toxicity in pore water compared to seawater. The caprolactam spiked, HLB SPE cleaned-up pore water test yielded an LC50 of 31.0 mg/L caprolactam (Table 4) whereas the caprolactam spiked seawater test yielded an LC50 of 453.5 mg/L caprolactam (Table 5).

RESULTS SUMMARY

Early GC/MS analyses of toxic fractions revealed the presence of caprolactam in some preparations, but not all. More recently, side-by-side analyses of duplicate preparations of 80% methanol elutions of HLB solid-phase cartridge extractions of toxic pore water exhibited a large caprolactam peak in one preparation and no caprolactam peak in the duplicate. This led us to speculate that caprolactam instability in the pore water matrix might be related to the

inconsistency between the two preparations. However, since the HLB SPE procedure consistently removed toxicity and caprolactam was the only major peak observed in the cartridge elution, we tested caprolactam for toxicity in both the clean seawater and in pore water which had been previously rendered non-toxic by low pH HLB SPE clean-up. Caprolactam was much more toxic (approximately one order of magnitude) in the cleaned-up pore water than in clean sea water. This pattern was also observed for the toxic materials eluted from the HLB clean-up cartridge. Since the unknown toxicant from the cartridge elution and caprolactam shared this common characteristic, we decided to examine the original toxic pore water for caprolactam and related compounds. Caprolactam can be analyzed by GC/MS but polar products resulting from the opening of the ring structure as well as related polymeric compounds are not amenable to GC/MS analysis and this may explain why our analyses of the toxic fractions sometimes contained caprolactam and sometimes did not. Consequently, we sub-contracted with Dr. Robert Voyksner, LCMS Limited, to examine selected samples by LC/MS. To date, Dr. Voyksner's work has demonstrated (1) the 80% methanol toxic fraction eluted from the HLB SPE cartridge shows the presence of LC/MS peaks with mass spectral characteristics consistent with the open ring structure of caprolactam and related polymeric materials (2) these caprolactam-related peaks are also present in the original toxic pore water and (3) these caprolactam-related peaks are absent in the non-toxic pore water resulting from HLB SPE clean-up. Analysis of HLB-cleaned-up pore water spiked with caprolactam revealed only caprolactam; spiking did not result in the formation of the caprolactam-related peaks found in the original toxic pore water and the toxic fraction eluted from the HLB cartridge.

Our interpretation of the results above is that there is evidence that caprolactam-related substances are contributing to the toxicity observed in the pore water. This toxic effect is observed only in combination with other contributing factors which remain in the pore water after HLB SPE treatment at low pH. These factors apparently interact with either spiked caprolactam or the HLB 80% toxic fraction to produce a toxic effect which is absent when either of these materials is tested for toxicity in clean sea water. We also have evidence that the increased toxicity of caprolactam seen in association with the cleaned-up pore water is not dependent upon the direct action of the pore water on the caprolactam (i.e. inducing ring opening or polymerization) since the increased toxicity of caprolactam can be induced by independent exposures to cleaned-up pore water (non-toxic by itself) and caprolactam in clean seawater (non-toxic by itself; see the results of the dual exposure experiment). This leads us to believe that the toxic interaction of caprolactam is likely manifested regardless of the integrity of the ring structure and state of polymerization in the environmental exposure (within the limits observed in the toxic pore water) and probably results from a metabolized form of the material that may be common to any form of the material that is initially taken up by the test organism. Areas for future investigation might include (1) identification of the unknown factors remaining in the non-toxic HLB-cleaned-up pore water which contribute to the caprolactam-related toxicity (2) identification of structures of caprolactam-related peaks by comparison with authentic standard materials (3) identification of factors important in determining the ring integrity and polymerization of the caprolactam-related peaks identified by LC/MS (4) examination of tissue samples for possible identification of toxic metabolites of the caprolactam-related compounds and interacting toxicants.

REFERENCES

U.S. Environmental Protection Agency, 1993. Marine Toxicity Identification Evaluation (TIE) Phase 1 Guidance Document, EPA/600/R-96/054, September 1996, Narragansett, Rhode Island.

U.S. Environmental Protection Agency, 1993. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms, EPA/600/4-90/027F, August 1993, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1991. Methods for Aquatic Toxicity Identification Evaluations. Phase I Toxicity Characterization Procedures, Second edition, EPA-600/6-91/003, February 1991, Duluth, Minnesota.

Table 1. Survival of *Mysidopsis bahia*, exposed to unaltered sediment pore water.

	Mean Survival (%)
<u>Exposure Period</u>	<u>96 Hours</u>
Seawater Control	100
25	90
50	60
100	0

LC50 = 48.9% pore water

Table 2. Survival of *Mysidopsis bahia*, exposed to post-HLB SPE sediment pore water.

<u>Test Treatment</u> <u>(% Pore water)</u>	Mean Survival (%)
<u>Exposure Period</u>	<u>96 Hours</u>
Seawater Control	100
25	100
50	100
100	90

Cartridge loading period = 2.0 ml/min

Table 3. Survival of *Mysidopsis bahia*, exposed to HLB cleaned pore water containing associated methanol eluates.

<u>Test Treatment</u> <u>(% Methanol in</u> <u>Water)</u>	Mean Survival (%)
<u>Exposure Period</u>	<u>96 Hours</u>
Methanol Blank	80
50	100
75	40
80	0
85	80
90	80
95	80
100	80

Elution period = 0.2 ml/min

Table 4. Survival of *Mysidopsis bahia*, exposed to post-HLB SPE sediment pore water spiked with caprolactam.

	Mean Survival (%)
<u>Exposure Period</u>	<u>96 Hours</u>
Pore Water Blank	100
12.5	100
25	60
50	20
100	0
200	0

LC50 = 31.0 mg/L caprolactam

Table 5. Survival of *Mysidopsis bahia*, exposed to laboratory seawater spiked with caprolactam.

<u>Test Treatment (mg/L Caprolactam in Seawater)</u>	<u>Mean Survival (%)</u>
<u>Exposure Period</u>	<u>96 Hours</u>
Seawater Blank	100
250	80
500	60
1000	0
2000	0
4000	0

LC50 = 453.5 mg/L caprolactam

Sediment Chemistry
Vince Bayou
Segment 1007A

		Station ID 11299	Station ID 11299	Station ID 14368	Station ID 14368	Station ID 11301	
PARAMETER		5/24/01 RESULT	7/26/01 RESULT	7/26/01 RESULT	4/23/02 RESULT	4/23/02 RESULT	Lowest Screening Value*
Ions	Chloride	1160	1660	3990	134	96.4	
	Sulfate	76.7	166	189	106	119	
Metals	Aluminum	11100	6410	17000	10400	7890	
	Arsenic	3.95	3.26	5.41	7.6	2.29	7.24
	Barium	86.8	53.8	256	115	74.9	
	Cadmium	0.514	0.347	1.31	0.312	0.18	0.676
	Calcium	23100	21300	32800	115000	28700	
	Chromium	22.3	12.9	35.3	68.8	23	52.3
	Copper	23.1	25.2	53.2	40.3	20.1	18.7
	Iron	12000	6600	16200	1990	9770	
	Lead	35.2	32.1	173	60.5	86.3	30.24
	Magnesium	3110	2710	4620	3700	2890	
	Nickel	10.1	7.13	16.4	16.9	9.24	15.9
	Potassium	1670	936	2280	1280	1260	
	Selenium	ND	ND	ND	1.98	ND	
	Silver	ND	ND	6.51	ND	ND	0.73
	Sodium	1330	1510	3230	910	252	
	Zinc	133	83.2	317	81.9	88.4	124
Mercury	0.106	0.459	0.128	0.109	ND	0.13	
Volatiles	1,1,1-Trichloroethane	ND	ND	ND	ND	ND	30
	1,1,2,2-Tetrachloroethane	ND	ND	ND	ND	ND	940
	1,1,2-Trichloroethane	ND	ND	ND	ND	ND	1257
	1,1-Dichloroethane	ND	ND	ND	ND	ND	27
	1,1-Dichloroethene	ND	ND	ND	ND	ND	31
	1,2-Dibromoethane	ND	ND	ND	ND	ND	
	1,2-Dichloroethane	ND	ND	ND	ND	ND	256
	1,2-Dichloropropane	ND	ND	ND	ND	ND	2075
	2-Chloroethylvinylether	ND	ND	ND	ND	ND	
	Benzene	ND	ND	ND	ND	ND	57
	Bromodichloromethane	ND	ND	ND	ND	ND	7426
	Bromoform	ND	ND	ND	ND	ND	650
	Bromomethane	ND	ND	ND	ND	ND	18
	Carbon disulfide	ND	ND	ND	ND	ND	
	Carbon tetrachloride	ND	ND	ND	ND	ND	225
	Chlorobenzene	ND	ND	2	J	ND	413
	Chloroethane	ND	ND	ND	ND	ND	7937
	Chloroform	ND	ND	ND	ND	ND	22
	Chloromethane	ND	ND	ND	ND	ND	432
	cis-1,2-Dichloroethene	ND	ND	ND	ND	ND	
	cis-1,3-Dichloropropene	ND	ND	ND	ND	ND	0.05
	Dibromochloromethane	ND	ND	ND	ND	ND	8701
	Ethylbenzene	ND	ND	ND	ND	ND	10
	Hexachlorobutadiene	ND	ND	ND	ND	ND	11
	m,p-Xylene	ND	ND	ND	ND	ND	
	Methyl tert-butyl ether	ND	ND	ND	ND	ND	
	Methylene chloride	ND	ND	ND	ND	ND	374
	o-Xylene	ND	ND	5.4	J	ND	
	Tetrachloroethene	ND	ND	ND	ND	ND	
	Toluene	ND	ND	ND	15.1	ND	
trans-1,2-Dichloroethene	ND	ND	ND	ND	ND		
trans-1,3-Dichloropropene	ND	ND	ND	ND	ND	230	
Trichloroethene	ND	ND	ND	ND	ND	215	
Vinyl chloride	ND	ND	ND	ND	ND	691	

Sediment Chemistry
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Segment 1007A

PARAMETER		5/24/01 RESULT	7/26/01 RESULT	7/26/01 RESULT	4/23/02 RESULT	4/23/02 RESULT	Lowest Screening Value*
Semi-Vol.	1,2,4-Trichlorobenzene	ND	ND	ND	ND	ND	
	1,2-Dichlorobenzene	ND	ND	ND	ND	ND	50
	1,3-Dichlorobenzene	ND	ND	ND	ND	ND	1664
	1,4-Dichlorobenzene	ND	ND	ND	ND	ND	110
	2,4,5-Trichlorophenol	ND	ND	ND	ND	ND	
	2,4,6-Trichlorophenol	ND	ND	ND	ND	ND	
	2,4-Dichlorophenol	ND	ND	ND	ND	ND	
	2,4-Dimethylphenol	ND	ND	ND	ND	ND	
	2,4-Dinitrophenol	ND	ND	ND	ND	ND	
	2,4-Dinitrotoluene	ND	ND	ND	ND	ND	293
	2,6-Dinitrotoluene	ND	ND	ND	ND	ND	10341
	2-Chloronaphthalene	ND	ND	ND	ND	ND	267345
	2-Chlorophenol	ND	ND	ND	ND	ND	
	2-Methylnaphthalene	ND	ND	ND	ND	ND	20.2
	2-Methylphenol	ND	ND	ND	ND	ND	
	2-Nitrophenol	ND	ND	ND	ND	ND	
	3,3'-Dichlorobenzidine	ND	ND	ND	ND	ND	20603
	4,6-Dinitro-2-methylphenol	ND	ND	ND	ND	ND	
	4-Bromophenyl phenyl ether	ND	ND	ND	ND	ND	1248
	4-Chloro-3-methylphenol	ND	ND	ND	ND	ND	
	4-Chlorophenyl phenyl ether	ND	ND	ND	ND	ND	456209
	4-Methylphenol	ND	ND	ND	ND	ND	
	4-Nitrophenol	ND	ND	ND	ND	ND	
	Acenaphthene	ND	ND	ND	ND	ND	6.71
	Acenaphthylene	ND	ND	ND	ND	ND	5.87
	Anthracene	55	58 J	140 J	110 J	130 J	46.85
	Benzo(a)anthracene	385	453	547	506	1030	74.8
	Benzo(a)pyrene	501	747	754	506	1250	88.8
	Benzo(b)fluoranthene	777	830	1110	612	13400	27372
	Benzo(g,h,i)perylene	389	281	318	ND	ND	720
	Benzo(k)fluoranthene	489	708	722	461	1200	3600
	Bis(2-chloroethoxy)methane	ND	ND	ND	ND	ND	
	Bis(2-chloroethyl)ether	ND	ND	ND	ND	ND	368
	Bis(2-chloroisopropyl)ether	ND	ND	ND	ND	ND	
	Bis(2-ethylhexyl)phthalate	1050	582	22400	940	474	182
	Butyl benzyl phthalate	ND	ND	ND	ND	ND	900
	Chrysene	617	714	961	736	1490	108
	Di-n-butyl phthalate	ND	ND	ND	ND	ND	11000
	Di-n-octylphthalate	ND	170 J	1100	ND	ND	885363
	Dibenzo(a,h)anthracene	ND	ND	ND	ND	ND	6.22
	Diethyl phthalate	ND	ND	ND	ND	ND	200
	Dimethyl phthalate	ND	ND	ND	ND	ND	
	Fluoranthene	944	978	1580	1590	2640	113
	Fluorene	ND	ND	120 J	ND	ND	19
	Hexachlorobenzene	ND	ND	ND	ND	ND	22
	Hexachlorocyclopentadiene	ND	ND	ND	ND	ND	
	Hexachloroethane	ND	ND	ND	ND	ND	1000
	Indeno[1,2,3-cd]pyrene	325	299	250 J	328	860	
	Isophorone	ND	ND	ND	ND	ND	
	N-Nitrosodi-n-propylamine	ND	ND	ND	ND	ND	
	N-Nitrosodiphenylamine	ND	ND	ND	ND	ND	
	Naphthalene	ND	ND	ND	ND	ND	34.6
	Nitrobenzene	ND	ND	ND	ND	ND	
	Pentachlorophenol	ND	ND	ND	ND	ND	
	Phenanthrene	319	331	857	328	1170	86.7
	Phenol	ND	ND	ND	ND	ND	
	Pyrene	780	812	1260	1060	2030	153

Sediment Chemistry
Vince Bayou
Segment 1007A

PARAMETER		5/24/01 RESULT	7/26/01 RESULT	7/18/01 RESULT	4/23/02 RESULT	4/23/02 RESULT	Lowest Screening Value*
Triazines	Atrazine	ND	ND	ND	ND	ND	
	Cyanazine	ND	ND	ND	ND	ND	
	Metolachlor	ND	ND	ND	ND	ND	
	Simazine	ND	ND	ND	ND	ND	
Pest/PCBs	a-BHC	ND	ND	ND	ND	ND	
	Alachlor	ND	ND	ND	ND	ND	
	Aldrin	ND	ND	ND	ND	ND	
	b-BHC	ND	ND	ND	ND	ND	
	Chlordane	ND	6.6 J	31 J	ND	ND	
	d-BHC	ND	ND	ND	ND	ND	
	4,4'-DDD	ND	11 J	ND	ND	ND	1.22
	4,4'-DDE	ND	12 J	ND	ND	ND	2.07
	4,4'-DDT	ND	5.5 J	27 J	ND	ND	1
	Dicofol	ND	ND	ND	ND	ND	
	Dieldrin	ND	ND	ND	ND	ND	
	Endosulfan	ND	ND	ND	ND	ND	
	Endosulfan sulfate	ND	ND	ND	ND	ND	
	Endrin	ND	ND	ND	ND	ND	
	g-BHC (Lindane)	ND	ND	ND	ND	ND	
	Heptachlor	ND	ND	ND	ND	ND	
	Heptachlor epoxide	ND	ND	ND	ND	ND	0.6
	Methoxychlor	ND	ND	ND	ND	ND	
	Mirex	ND	ND	ND	ND	ND	
	PCB-1016	ND	ND	ND	ND	ND	
	PCB-1221	ND	ND	ND	ND	ND	
	PCB-1232	ND	ND	ND	ND	ND	
	PCB-1242	ND	ND	ND	ND	ND	
	PCB-1248	ND	ND	4000 J	ND	ND	
PCB-1254	11000	ND	ND	ND	ND		
PCB-1260	ND	ND	ND	ND	ND		
Toxaphene	ND	ND	ND	ND	ND		
Organo-phosphorus Compounds	Chloropyrifos	14.0 J	ND	ND	ND	ND	
	Demeton (Total)	ND	ND	ND	ND	ND	
	Diazinon	ND	ND	ND	ND	ND	
	Guthion	ND	ND	ND	ND	ND	
	Malathion	ND	ND	ND	ND	ND	
	Parathion	ND	ND	ND	ND	ND	
Chlorinated Herbicides	2,4,5-T	ND	ND	ND	ND	ND	
	2,4,5-TP (Silvex)	ND	ND	ND	ND	ND	
	2,4-D	ND	ND	ND	ND	ND	

Sediment Chemistry
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Segment 1007A

PARAMETER		5/24/01 RESULT	7/26/01 RESULT	7/18/01 RESULT	4/23/02 RESULT	4/23/02 RESULT	Lowest Screening Value*
Carbamates	Carbaryl	ND	ND	ND	ND	ND	
	Diuron	ND	ND	ND	ND	ND	
SEM	Cadmium	0.5	0.19	0.83	ND	0.0037	
	Copper	1.01	ND	ND	2.2 J	1.2 J	
	Lead	48.6	13	140	0.31 J	0.49 J	
	Mercury	0.0006 J	ND	ND	0.00024	0.0007	
	Nickel	3.16	0.98	3.5	0.12	0.19	
	Silver	1.066	ND	ND	NA	NA	
	Zinc	161.28	49	180	2 J	2.7 J	
Total Organic Carbon		24700	16580	23940	8100	8200	
Acid Volatile Sulfide (AVS)		1323	420	2200	26.2	24.4	
Grain Size	Gravel	NA	NA	NA	8.9	0	
	Sand	41.5	68.08	38.85	71.6	54.7	
	Silt	33.15	20.89	43.52	11.5	26.9	
	Clay	25.35	11.03	17.63	8.00	18.40	

Notes:

* Criteria is from *Equilibrium and Non-Equilibrium Partitioning-Based Sediment Quality Screening Indices* tables.

The value is the lowest value from the Indices as stated in the Appendix.

J- result is estimated

ND- result was Not Detected

mg/kg-dry = milligrams per kilogram dry weight

ug/kg-dry = microgram per kilogram dry weight

umol/dry g = microgram per mole per dry gram

% = percent

**APPENDIX E
DATA QUALITY OBJECTIVES AND VALIDATION REPORTS**

Appendix E Data Quality Objectives for Measurement Data

Parameter	Units	Method Type	Method	Method Description	Storet	MAL	Precision of Laboratory Duplicates (RPD)	Accuracy of Matrix Spikes % Recovery	Accuracy crm	Percent Complete
Field Parameters										
pH	pH units	YSI Multi-Parameter Probe	EPA 150.1 or TNRCC SOP	probe	00400	1.0	10	NA	+/- 0.1	90
Dissolved Oxygen (DO)	mg/L	YSI Multi-Parameter Probe	EPA 360.1 or TNRCC SOP	probe	00300	1.0	10	+/- 0.5	NA	90
Conductivity	uS/cm	YSI Multi-Parameter Probe	EPA 120.1 or TNRCC SOP	probe	00094	1	10	+/- 5	+/- 5	90
Temperature	° Celcius	YSI Multi-Parameter Probe	EPA 170.1 or TNRCC SOP	probe	00010	NA	10	NA	NA	90
Salinity	ppt	YSI Multi-Parameter Probe	TNRCC SOP	probe	00480	NA	NA	NA	NA	90
Instantaneous Stream Flow	cfs	Flowmeter	TNRCC SOP	sensor	00061	NA	NA	NA	NA	90
Flow Severity	1-no flow, 2-low, 3-normal, 4-flood, 5-high, 6-dry	Observation	TNRCC SOP	Field observation	01351	NA	NA	NA	NA	90
Conventional Parameters										
Total Residual Chlorine	mg/L	DPD	EPA 330.5	colorimetric	50060	0.1	20%	NA	NA	90
Sediment Grain-size	% particle size	Frac. Separation & gravimetric determination	EPA 3.4, 3.5 (600/2-78-054)	Separation and gravimetric	89991, 82009, 82008, 80256	NA	NA	NA	NA	90
Total Suspended Solids	mg/L	gravimetric	EPA 160.2	gravimetric	00530	4.0	20	NA	+/- 10%	90
Total Organic Carbon (TOC)	mg/L	oxidation	EPA 415.1	oxidation	00680	1.0	20	78-120	+/- 10%	90
Total Organic Carbon (TOC) in sediment	mg/kg	Combustion	B&B Laboratories SOP 1005 See Appendix I	Combustion	81951	0.3	15	80-120	+/- 5%	90
Oil & Grease	mg/L	Extraction Gravimetry	EPA 413.1	Freon Extractable Material	00556	1.0	20	80-120	+/-10%	90
Dissolved Organic Carbon (DOC)	mg/L	oxidation	EPA 415.2	oxidation	00681	0.1	20	78-120	+/- 10%	90
Total Alkalinity, as CaCO ₃	mg/L	potentiometric	EPA 310.1-2	potentiometri ^c	00410	3.0	20	78-120	NA	90
Total Dissolved Solids (TDS)	mg/L	residue gravimetric	EPA 160.1	residue gravimetric	70300	10.0	20	NA	NA	90
Sulfate in water	mg/L	ion chromatoph ^{gry}	EPA 300.0/9056	IC	00945	3	20	70-113	+/- 10%	90
Sulfate in sediment	mg/kg	ion chromatoph ^{gry}	EPA 300.0/9056	IC	85818	10	30	80-120	80-120	90
Sulfide in water	mg/L	colorimetric	EPA 371.2	colorimetric	00745	1.0	20	80-120	+/-10%	90
Fluoride in water	mg/L	colorimetric	EPA 340.3/9056	Colorimetric/ ^{IC}	00950	0.5	20	80-120	+/-10%	90

Appendix E Data Quality Objectives for Measurement Data

Parameter	Units	Method Type	Method	Method Description	Storet	MAL	Precision of Laboratory Duplicates (RPD)	Accuracy of Matrix Spikes % Recovery	Accuracy crm	Percent Complete
Chloride in water	mg/L	colorimetric	EPA 325.2/9256	Colorimetric automated ferricyanide/IC	00940	1.0	20	80-120		90
Chloride in sediment	mg/kg	IC	EPA 300.0	IC	00943	10	30	80-120	80-120	90
Ammonia-N	mg/L	colorimetric	EPA 350.1	colorimetric	00610	0.02	20	68-135	NA	90
o-Phosphorus	mg/L	colorimetric, absorbic acid	EPA 365.3	IC	00671	0.01	20	80-120	NA	90
Potassium, total recoverable in water	mg/L	ICP/AES	EPA 200.7	ICP/AES	00937	0.05	20	80-149	90-110	90
Potassium in sediment	mg/kg	ICP/MS	EPA 6020	ICP/MS	00938	25	25	NA	80-120	90
Sodium, total recoverable in water	mg/L	ICP/AES	EPA 200.7	ICP/AES	00929	0.2	20	79-137	90-110	90
Sodium in sediment	mg/kg	ICP/MS	EPA 6020	ICP/MS	00934	25	25	NA	80-120	90
Nitrate/nitrite-N	mg/L	ion chromatography	EPA 353.2	Colorimetric automated cadmium reduction	00630	0.01	20	83-125	+/- 10%	90
Total Kjeldahl Nitrogen	mg/L	colorimetric, automated phenate	EPA 351.2	colorimetric	00625	0.1	20	72-133	+/- 10%	90
Total Phosphorus (TPO)	mg/L	colorimetric, automated, block digester	365.1-4	colorimetric	00665	0.02	20	74-118	+/- 10%	90
Cyanide	mg/L	spectrophotometric	EPA 335.2	spectrophotometric	00720	5	20	80-120	+/- 10%	90
Turbidity	NTU	nephelometric	EPA 180.1	nephelometric	82079	0.05	20	NA	+/- 10%	90
Carbonaceous Biochemical Oxygen Demand (BOD)	mg/L	potentiometric	EPA 405.1	potentiometric	00307	1.0	25	NA	+/- 5%	90
Chemical Oxygen Demand (COD)	mg/L	colorimetric	EPA 410.1-3	colorimetric	00335 or 00340	10	25	NA	+/- 5%	90
Acid volatile sulfide in sediment	umol/g	colorimetry	EPA Draft 1991	Purge and trap, colorimetry	50088	0.5	40	60-130	NA	90
SEM Simultaneous extraction, sum of concentrations: Cd, Cu, Pb, Hg, Ni, Ag, and Zn	umol/g	CVAAS Hg, ICP Other elements	EPA 200.7/245.5	Purge and Trap, Atomic spectroscopy	50087	0.05-0.5 w/ metal	40	NA	NA	90
Metals, trace metals, and related parameters										
Aluminum, dissolved in water	µg/L	ICP-MS	EPA 200.8	ICP-MS	01106	10	25	80-120	80-120	90
Aluminum, total in water	µg/L	ICP-MS	EPA 200.8	ICP-MS	01105	10	25	80-120	80-120	90
Aluminum in sediment	mg/kg	Primary Direct	EPA 200.8 or 6010B/6020	ICP-MS	01108	12.5	25	NA	80-120	90
Arsenic, dissolved in water	µg/L	HGA/FS	EPA 200.8	HGA/FS	01000	10	25	55-146	55-146	90
Arsenic, total in water	µg/L	HGA/FS	EPA 1632	HGA/FS	01002	0.5	25	55-146	55-146	90
Arsenic in sediment	mg/kg	Primary Direct	EPA 6020/200.8	ICP-MS	01003	2.5	25	80-120	80-120	90
Barium, dissolved in water	µg/L	Primary Direct	EPA 200.8	ICP-MS	01005	10	25	80-120	80-120	90

Appendix E Data Quality Objectives for Measurement Data

Parameter	Units	Method Type	Method	Method Description	Storet	MAL	Precision of Laboratory Duplicates (RPD)	Accuracy of Matrix Spikes % Recovery	Accuracy crm	Percent Complete
Barium in sediment	mg/kg	Primary Direct	EPA 6020/200.8	ICP-MS	01008	2.5	25	80-120	80-120	90
		ICP-MS	EPA 200.8	ICP-MS	01025	0.1	25	80-120	80-120	90
Cadmium, dissolved in water	µg/L	Alternate Direct	EPA 200.9	GFAAS	01025	0.05	25	64-145	64-145	90
		Primary Direct	EPA 200.8	ICP-MS	01027	0.1	25	84-113	84-113	90
Cadmium in sediment	mg/kg	Alternate Direct	EPA 200.9	GFAAS	01027	0.05	25	64-145	64-145	90
		Primary Direct	EPA 200.8 or 6010B/6020	ICP-MS	01028	0.2	25	80-120	80-120	90
Calcium, dissolved in water	mg/L	ICP/AES	EPA 200.7	ICP-AES	00915	0.05	20	84-113	84-113	90
		Alternate Direct	EPA 215.1	Flame AAS	00915	0.03	20	80-120	80-120	90
Calcium, total recoverable in water	mg/L	ICP/AES	EPA 200.7	ICP-AES	00916	0.05	20	84-113	84-113	90
		Primary Direct	EPA 200.8 or 6010B/6020	ICP-MS	00917	12.5	25	80-120	80-120	90
Chromium, dissolved in water	µg/L	ICP-MS	EPA 200.8	ICP-MS	01030	2.0	25	80-120	80-120	90
		Primary Direct	EPA 200.8	ICP-MS	01034	2.0	25	80-120	80-120	90
Chromium (hexavalent), total in water	µg/L	Ion Chromatography	EPA 1636	IC	01032	5.0	20	79-122	79-122	90
		Primary Direct	EPA 6020/200.8	ICP-MS	01029	2	25	80-120	80-120	90
Copper, dissolved in water	µg/L	ICP-MS	EPA 200.8	ICP-MS	01040	0.2	25	51-145	51-145	90
		Primary Direct	EPA 200.8	ICP-MS	01042	0.2	25	51-145	51-145	90
Copper in sediment	mg/kg	Primary Direct	EPA 6020/200.8	ICP-MS	01043	2.5	25	80-120	80-120	90
		Primary Direct	EPA 130.1-2	Titrametric EDTA	00900	1.0, as CaCO ₃	20	80-120	80-120	90
Iron, total recoverable in water	µg/L	ICP-AES	EPA 200.7	ICP-AES	01045	0.05				90
		ICP/MS	EPA 6020A	ICP/MS	01170	12.5				90
Lead, dissolved in water	µg/L	ICP-MS	EPA 200.8	ICP-MS	01049	0.05	25	72-143	72-143	90
		Primary Direct	EPA 200.8	ICP-MS	01051	0.05	25	72-143	72-143	90
Lead, in sediment	mg/kg	Primary Direct	EPA 200.8 or 6010B/6020	ICP-MS	01052	2	25	80-120	80-120	90
		Alternate Direct	EPA 200.7	ICP-AES	00925	0.05	20	80-120	80-120	90
Magnesium, dissolved in water	mg/L	ICP/AES	EPA 200.7	ICP-AES	00925	0.003	20	80-120	80-120	90
		Alternate Direct	EPA 242.1	Flame AAS	00925					90
Magnesium, total recoverable in water	mg/L	ICP/AES	EPA 200.7	ICP-AES	00927	0.05	20	80-120	80-120	90
		Primary Direct	EPA 6020	ICP/MS	00924	2.5	25	NA	80-120	90
Mercury, dissolved in water	µg/L	Primary Direct	EPA 1631	P/T CVA/F	71890	0.0005	25	71-125	71-125	90
		Primary Direct	EPA 1631	P/T CVA/F	71900	0.0005	25	71-125	71-125	90
Mercury, total recoverable in water	µg/L	Primary Direct	EPA 245.5	P/T CVA/F	71921	0.05	25	80-120	80-120	90
		Primary Direct	EPA 245.5	P/T CVA/F	71921	0.05	25	80-120	80-120	90

Appendix E Data Quality Objectives for Measurement Data

Nickel, dissolved in water	µg/L	ICP-MS	EPA 200.8	ICP-MS	01065	1.0	20	68-134	68-134	90
		Alternate Direct	EPA 200.9	GFAAS	01065	2.0	25	65-145	65-145	90
		Primary Direct	EPA 200.8	ICP-MS	01067	1.0	20	68-134	68-134	90
		Alternate Direct	EPA 200.9	GFAAS	01067	2.0	25	65-145	65-145	90
Nickel in sediment	mg/kg	Primary Direct	EPA 6020/200.8	ICP-MS	01068	2.5	20	80-120	80-120	90
	µg/L	Primary Direct	EPA 200.8	ICP-MS	01145	1 or 2	25	59-149	59-149	90
Selenium, dissolved in water	µg/L	Alternate Direct	EPA 200.9	GFAAS	01145	2	25	56-131	56-131	90
		ICP-MS	EPA 200.8	ICP-MS	01147	2	25	59-149	59-149	90
Selenium, total recoverable in water	µg/L	Alternate Direct	EPA 200.9	GFAAS	01147	2	25	56-131	56-131	90
		Primary Direct	EPA 6010B/6020/200.8	ICP-MS	01148	5	25	80-120	80-120	90
Silver, dissolved in water	µg/L	ICP-MS	EPA 200.8	ICP-MS	01075	0.1	25	74-119	74-119	90
	µg/L	Primary Direct	EPA 200.8	ICP-MS	01077	0.1	25	74-119	74-119	90
Silver in sediment	mg/kg	Primary Direct	EPA 6020/200.8	ICP-MS	01078	1	25	75-125	75-125	90
	µg/L	ICP-MS	EPA 200.8	ICP-MS	01090	0.5	25	46-146	46-146	90
Zinc, dissolved in water	µg/L	Alternate Direct	EPA 200.7	ICP-AES	01090	5.0	25	67-142	67-142	90
		Alternate Direct	EPA 200.9	GFAAS	01090	0.5	25	67-142	67-142	90
Zinc, total in water	µg/L	Primary Direct	EPA 200.8	ICP-MS	01092	0.5	25	46-146	46-146	90
		Alternate Direct	EPA 200.7	ICP-MS	01092	5.0	25	80-120	80-120	90
Zinc, in sediment	mg/kg	Alternate Direct	EPA 200.9	GFAAS	01092	0.5	25	67-142	67-142	90
		Primary Direct	EPA 6020/200.8	ICP-MS	01093	2.5	25	80-120	80-120	90
Organic and Organometal Compounds										
Acenaphthene in water	µg/L	Primary	EPA 8270C	GC/MS	34205	4	30	49-125	49-125	90
Acenaphthene in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34208	133	30	47-145	47-145	90
Anthracene in water	µg/L	Primary	EPA 8270C	GC/MS	34220	4	30	45-165	45-165	90
Anthracene in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34223	660	30	27-133	27-133	90
Acenaphthylene in water	µg/L	Primary	EPA 8270C	GC/MS	34200	4	30	47-125	47-125	90
Acenaphthylene in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34203	660	30	33-145	33-145	90
Acrolein in sediment (Propenal)	µg/kg	Primary	EPA8260B	GC/MS	34213	51	40	25-175	25-175	90
Acrylonitrile in water	µg/L	Primary	EPA8260B	GC/MS	34215	50	20	50-150	50-150	90
Acrylonitrile in sediment	µg/kg	Primary	EPA8260B	GC/MS	34218	3.71	40	25-175	25-175	90

Appendix E Data Quality Objectives for Measurement Data

Alachlor in water	µg/L	Primary	EPA 8081	GC/ECD	77825	0.10	25	50-150	50-150	90
		Alternate	EPA 525.1	L/S Extraction + Capillary GC/MS	77825	0.3	25			90
Alachlor in sediment		Alternate	EPA 645	GC		0.6	25			90
		Alternate	EPA 1656	GC/ECD		0.06	25	23-101		90
Aldrin in water	µg/kg	Primary	EPA 8081	GC/ECD	75050	100	30	50-150	50-150	90
	µg/L	Primary	EPA 8081	GC/ECD	39330	0.05	25	20-100	20-100	90
Aldrin in sediment	µg/kg	Primary	EPA 8081	GC/NPD	39333	50	30	50-150	50-150	90
	µg/L	Primary	EPA 619	GC	39630	0.15	25	62-191	62-191	90
Atrazine in water		Alternate	EPA 525.1	L/S Extraction + Capillary GC/MS		0.42	25			90
		Alternate	EPA 1656	GC/ECD		1.5	25	31-132		90
Atrazine in sediment	µg/kg	Primary	EPA 8141	GC/NPD	39631	50	30			90
	µg/L	Primary	EPA 8260B	GC/MS	34030	1	20	75-125	75-125	90
Benzene in water	µg/kg	Primary	EPA 8260B	GC/MS	34237	10	40	25-165	25-165	90
	µg/L	Primary	EPA 8260B	GC/MS	32104	1	20	75-125	75-125	90
Bromoform in water	µg/kg	Primary	EPA 8260B	GC/MS	34290	10	40	30-180	30-180	90
	µg/L	Primary	EPA 8260B	GC/MS	30202	1	20	62-147	62-147	90
Bromomethane in water	µg/kg	Primary	EPA 8260B	GC/MS	88802	5	30	70-130	70-130	90
	µg/L	Primary	EPA 8270C	GC/MS	34526	4	30	51-133	51-133	90
Benz (a) Anthracene in water	µg/kg	Primary	EPA 8270C	GC/MS	34529	660	30	33-143	33-143	90
	µg/L	Primary	EPA 8270C	GC/MS	34247	4	30	41-125	41-125	90
Benzo (a) Pyrene in water	µg/kg	Primary	EPA 8270C	GC/MS	34250	660	30	17-163	17-163	90
	µg/L	Primary	EPA 8270C	GC/MS	34230	4	30	37-152	37-152	90
Benzo (b) fluoranthene in water	µg/kg	Primary	EPA 8270C	GC/MS	34233	133	30	24-159	24-159	90
	µg/L	Primary	EPA 8270C	GC/MS	34521	4	30	34-149	34-149	90
Benzo (ghi) Perylene in water	µg/kg	Primary	EPA 8270C	GC/MS	34524	660	30	15-219	15-219	90
	µg/L	Primary	EPA 8270C	GC/MS	34242	4	30	34-149	34-149	90
Benzo (k) Fluoranthene in water	µg/kg	Primary	EPA 8270C	GC/MS	34245	660	30	11-162	11-162	90
	µg/L	Primary	EPA 8081	GC/ECD	39337	0.05	25	35-117	35-117	90
BHC, alpha in water	µg/kg	Primary	EPA 8081	GC/ECD	39076	50	30	38-137	38-137	90
	µg/L	Primary	EPA 8081	GC/ECD	39338	0.05	25	51-121	51-121	90
BHC, beta in sediment	µg/kg	Primary	EPA 8081	GC/ECD	34257	50	30	51-133	51-133	90
	µg/L	Primary	EPA 8081	GC/ECD	34259	0.05	25	32-121	32-121	90
BHC, delta in water	µg/kg	Primary	EPA 8081	GC/ECD	34262	50	30	43-131	43-131	90
	µg/L	Primary	EPA 8081	GC/ECD	39782	0.05	25	41-114	41-114	90
BHC, gamma (Lindane) in water	µg/kg	Primary	EPA 8081	GC/ECD	39783	50	30	47-132	47-132	90
	µg/L	Primary	EPA 8270C	GC/MS	34278	4	30	49-125	49-125	90
Bis (2-Chloroethoxy) Methane in water	µg/kg	Primary	EPA 8270C	GC/MS	34281	660	30	33-184	33-184	90
	µg/L	Primary	EPA 8270C	GC/MS	34273	4	30	44-125	44-125	90
Bis (2-Chloroethyl) Ether in water	µg/kg	Primary	EPA 8270C	GC/MS	34276	133	30	12-158	12-158	90
	µg/L	Primary	EPA 8270C	GC/MS	34283	4	30	36-166	36-166	90

Appendix E Data Quality Objectives for Measurement Data

Bis (2-Chloroisopropyl) Ether in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34286	133	30	36-166	36-166	90
Bis (2-Ethylhexyl) Phthalate in water	µg/L	Primary	EPA 8270C	GC/MS	39100	4	30	33-129	33-129	90
Bis (2-Ethylhexyl) Phthalate in sediment	µg/kg	Primary	EPA 8270C	GC/MS	39102	660	30	8-158	8-158	90
4-Bromophenyl Phenyl Ether in water	µg/L	Primary	EPA 8270C	GC/MS	34636	4	30	53-127	53-127	90
4-Bromophenyl Phenyl Ether in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34639	660	30	53-130	53-130	90
N-Butylbenzyl Phthalate in water	µg/L	Primary	EPA 8270C	GC/MS	34292	10	30	26-125	26-125	90
N-Butylbenzyl Phthalate in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34295	660	30	15-152	15-152	90
Carbaryl (Sevin) in water	µg/L	Primary	EPA 8321	HPLC/MS	39750	1	25	40-131	40-131	90
Carbaryl (Sevin) in sediment	µg/kg	Primary	EPA 8321	HPLC/MS	81818	20	25	34-129	34-129	90
Carbon disulfide in water	µg/L	Primary	EPA 8260B	GC/MS	77041	25	20	50-150	50-150	90
		Alternate	EPA 1624	Isotope Dilution GC/MS	77041	25				90
Carbon disulfide in sediment	µg/kg	Primary	EPA 8260B	GC/MS	78544	50	30	50-150	50-150	90
		Alternate	EPA 1624	Isotope Dilution GC/MS	78544		25			90
Carbon Tetrachloride in water	µg/L	Primary	EPA 8260B	GC/MS	32102	1	20	62-125	62-125	90
Carbon Tetrachloride in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34299	10	40	60-150	60-150	90
Chlorobenzene in water	µg/L	Primary	EPA 8260B	GC/MS	34301	1	20	75-125	75-125	90
Chlorobenzene in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34304	10	40	20-175	20-175	90
Chlorodibromomethane in water	µg/L	Primary	EPA 8260B	GC/MS	32105	1	20	73-125	73-125	90
Chlorodibromomethane in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34309	5	40	40-160	40-160	90
Chloroethane in water	µg/L	Primary	EPA 8260B	GC/MS	34311	1	50	53-145	53-145	90
Chloroethane in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34314	5	40	15-255	15-255	90
2-Chloroethylvinyl ether in water	µg/L	Primary	EPA 8260B	GC/MS	34576	50	20	50-150	50-150	90
2-Chloroethylvinyl ether in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34579	60	40	15-300	15-300	90
Chloroform in water	µg/L	Primary	EPA 8260B	GC/MS	32106	1	20	74-125	74-125	90
Chloroform in sediment	µg/L	Primary	EPA 8260B	GC/MS	34318	10	40	40-150	40-150	90
Chlordane in water	µg/L	Primary	EPA 8081	GC/ECD	39350	0.05	25	45-122	45-122	90
		Alternate	EPA 1656	GC/ECD	39350	1-2	25	69-133	69-133	90
		Alternate	EPA 525.1	L/S Extraction + Capillary GC/MS	39350	1-2	25			90
Chlordane in sediment	µg/kg	Primary	EPA 8081	GC/ECD	39351	50	30	56-142	56-142	90
		Alternate	EPA 1656	GC/ECD			25	69-133	69-133	90
Chloromethane in water	µg/L	Primary	EPA 8260B	GC/MS	30201	1	20	60-140	60-140	90
Chloromethane in sediment	µg/kg	Primary	EPA 8260B	GC/MS	88835	10	30	70-130	70-130	90
2-Chloronaphthalene in water	µg/L	Primary	EPA 8270C	GC/MS	34581	4	30	60-125	60-125	90
2-Chloronaphthalene in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34584	660	30	60-130	60-130	90
2-Chlorophenol in water	µg/L	Primary	EPA 8270C	GC/MS	34586	4	30	41-125	41-125	90
2-Chlorophenol in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34589	133	30	31-135	31-135	90
4-Chlorophenyl Phenyl Ether in water	µg/L	Primary	EPA 8270C	GC/MS	34641	4	30	51-132	51-132	90
4-Chlorophenyl Phenyl Ether in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34644	133	30	25-158	25-158	90
Chloropyrifos (Dursban) in water	µg/L	Primary	EPA 8141	GC/NPD	81403	0.5	25	45-118	45-118	90
Chloropyrifos (Dursban) in sediment	µg/kg	Primary	EPA 8141	GC/NPD	81404	50	30	40-129	40-129	90
Chrysene in water	µg/L	Primary	EPA 8270C	GC/MS	34320	4	30	55-133	55-133	90
Chrysene in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34323	133	30	17-168	17-168	90
Cyanazine in water	µg/L	Primary	EPA 619	GC/NPD	81757	0.5	25	30-232	30-232	90
Cyanazine in sediment	µg/kg	Primary	EPA 619-m	GC/NPD	03999	50	30			90

Appendix E Data Quality Objectives for Measurement Data

2,4-D in water	µg/L	Primary	EPA 8151	GC/ECD	39730	0.5	25	72-146	72-146	90
2,4-D in sediment	µg/kg	Primary	EPA 8151	GC/ECD	39731	200	30	89-175	89-175	90
Demeton in water	µg/L	Primary	EPA 8141	GC/NPD	39560	1	25	14-107	14-107	90
Demeton in sediment	µg/kg	Primary	EPA 8141	GC/NPD	82400	100	30	5-108	5-108	90
Diazinon in water	µg/L	Primary	EPA 8141	GC/NPD	39570	0.1	25	34-126	34-126	90
Diazinon in sediment	µg/kg	Primary	EPA 8141	GC/NPD	39571	50	30	39-124	39-124	90
1,2-Dibromoethane in water	µg/L	Primary	EPA 8260B	GC/MS	77651	1	20	75-125	75-125	90
1,2-Dibromoethane in sediment	µg/kg	Primary	EPA 8260B	GC/MS	88805	10	30	70-130	70-130	90
Dicofof (Kelthane) in water	µg/L	Primary	EPA 8081	GC/ECD	39780	0.10	25			90
Dicofof (Kelthane) in sediment	µg/kg	Primary	EPA 8081	GC/ECD	79799	100	30			90
Dieldrin in water	µg/L	Primary	EPA 8081	GC/ECD	39380	0.02	25	52-120	52-120	90
		Alternate	EPA 1656	GC/ECD	39380	0.02	25	48-158	48-158	90
Dieldrin in sediment	µg/kg	Primary	EPA 8081	GC/ECD	39383	50	30	56-125	56-125	90
		Alternate	EPA 1656	GC/ECD	38383		25	48-158	48-158	90
BromoDichloromethane in water	µg/L	Primary	EPA 8260B	GC/MS	32101	1	20	75-125	75-125	90
BromoDichloromethane in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34330	10	40	40-160	40-160	90
1,1-Dichloroethane in water	µg/L	Primary	EPA 8260B	GC/MS	34496	1	20	72-125	72-125	90
1,1-Dichloroethane in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34499	5	40	45-165	45-165	90
1,2-Dichloroethane in water	µg/L	Primary	EPA 8260B	GC/MS	34531	1	20	68-127	68-127	90
1,2-Dichloroethane in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34534	5	40	40-165	40-165	90
1,1-Dichloroethylene in water	µg/L	Primary	EPA 8260B	GC/MS	34501	1	20	75-125	75-125	90
1,1-Dichloroethylene in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34504	5	40	15-260	15-260	90
1,2-Dichloropropane in water	µg/L	Primary	EPA 8260B	GC/MS	34541	1	20	70-125	70-125	90
1,2-Dichloropropane in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34544	5	40	15-255	15-255	90
cis 1,3-Dichloropropene in water	µg/L	Primary	EPA 8260B	GC/MS	34704	1	20	74-125	74-125	90
cis 1,3-Dichloropropene in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34702	10	30	70-130	70-130	90
1,3-Dichloropropylene in water	µg/L	Primary	EPA 8260B	GC/MS	34565	10	40	15-280	15-280	90
1,3-Dichloropropylene in sediment	µg/kg	Primary	EPA 8321	HPLC/MS	39650	1	25	57-133	57-133	90
Diuron (Karmex) in water	µg/L	Primary	EPA 8321	HPLC/MS	73030	20	25	25-133	25-133	90
Diuron (Karmex) in sediment	µg/kg	Primary	EPA 8081	GC/ECD	39373	50	30	36-129	36-129	90
DDT in sediment	µg/kg	Alternate	EPA 1656	GC/ECD	39373	12	25	79-119	79-119	90
DDT in water	µg/L	Primary	EPA 8081	GC/ECD	39370	0.05	25	27-142	27-142	90
		Alternate	EPA 1656	GC/ECD	39370	0.036	25	79-119	79-119	90
DDE in sediment	µg/kg	Primary	EPA 8081	GC/ECD	39368	50	30	58-127	58-127	90
		Alternate	EPA 1656	GC/ECD	39368	4	25	54-126	54-126	90
DDE in water	µg/L	Primary	EPA 8081	GC/ECD	39365	0.05	25	29-120	29-120	90
		Alternate	EPA 1656	GC/ECD	39365	0.030	25	54-126	54-126	90
DDD in sediment	µg/kg	Primary	EPA 8081	GC/ECD	39363	50	30	51-129	51-129	90
		Alternate	EPA 1656	GC/ECD	39363	11	25	57-129	57-129	90
DDD in water	µg/L	Primary	EPA 8081	GC/ECD	39360	0.05	25	44-119	44-119	90
		Alternate	EPA 1656	GC/ECD	39360	0.015	25	57-129	57-129	90
Dibenzo (a,h) Anthracene in water	µg/L	Primary	EPA 8270C	GC/MS	34556	4	30	50-125	50-125	90
Dibenzo (a,h) Anthracene in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34559	660	30	15-227	15-227	90
1,2-Dichlorobenzene in water	µg/L	Primary	EPA 8260B	GC/MS	34536	4	30	42-155	42-155	90
1,2-Dichlorobenzene in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34539	660	30	32-130	32-130	90
1,3-Dichlorobenzene in water	µg/L	Primary	EPA 8260B	GC/MS	34566	4	30	36-125	36-125	90
1,3-Dichlorobenzene in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34569	660	30	15-172	15-172	90
1,4-Dichlorobenzene in water	µg/L	Primary	EPA 8260B	GC/MS	34571	4	30	30-125	30-125	90
1,4-Dichlorobenzene in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34574	660	30	20-130	20-130	90
3,3-Dichlorobenzidine in water	µg/L	Primary	EPA 8270C	GC/MS	34631	4	30	29-175	29-175	90

Appendix E Data Quality Objectives for Measurement Data

3,3-Dichlorobenzidine in sediment	Primary	EPA 8270C	GC/MS	34634	133	30	15-262	15-262	90
trans-1,2-Dichloroethene in water	Primary	EPA 8260B	GC/MS	34546	1	20	75-125	75-125	90
trans-1,2-Dichloroethene in sediment	Primary	EPA 8260B	GC/MS	34549	10	30	75-125	75-125	90
2,4-Dichlorophenol in water	Primary	EPA 8270C	GC/MS	34601	4	30	46-125	46-125	90
2,4-Dichlorophenol in sediment	Primary	EPA 8270C	GC/MS	34604	133	30	36-135	36-135	90
trans-1,3-Dichloropropene in water	Primary	EPA 8260B	GC/MS	34699	1	20	66-125	66-125	90
trans-1,3-Dichloropropene in sediment	Primary	EPA 8260B	GC/MS	34697	10	30	70-130	70-130	90
Diethyl Phthalate in water	Primary	EPA 8270C	GC/MS	34336	10	30	37-125	37-125	90
Diethyl Phthalate in sediment	Primary	EPA 8270C	GC/MS	34339	660	30	15-130	15-130	90
2,4-Dimethylphenol in water	Primary	EPA 8270C	GC/MS	34606	4	30	10-139	10-139	90
2,4-Dimethylphenol in sediment	Primary	EPA 8270C	GC/MS	34609	133	30	30-149	30-149	90
Dimethyl Phthalate in water	Primary	EPA 8270C	GC/MS	34341	4	30	25-175	25-175	90
Dimethyl Phthalate in sediment	Primary	EPA 8270C	GC/MS	34344	660	30	15-130	15-130	90
Di-n-Butyl Phthalate in water	Primary	EPA 8270C	GC/MS	39110	10	30	34-136	34-136	90
Di-n-Butyl Phthalate in sediment	Primary	EPA 8270C	GC/MS	39112	330	30	1-130	1-130	90
4,6-Dinitro-ortho-cresol in water	Primary	EPA 8270C	GC/MS	34657	10	30	26-134	26-134	90
4,6-Dinitro-ortho-cresol in sediment	Primary	EPA 8270C	GC/MS	34660	330	30	25-144	25-144	90
2,4-Dinitrophenol in water	Primary	EPA 8270C	GC/MS	34616	20	30	30-151	30-151	90
2,4-Dinitrophenol in sediment	Primary	EPA 8270C	GC/MS	34619	660	30	25-161	25-161	90
2,4-Dinitrotoluene in water	Primary	EPA 8270C	GC/MS	34611	4	30	39-139	39-139	90
2,4-Dinitrotoluene in sediment	Primary	EPA 8270C	GC/MS	34614	133	30	39-139	39-139	90
2,6-Dinitrotoluene in water	Primary	EPA 8270C	GC/MS	34626	4	30	51-125	51-125	90
2,6-Dinitrotoluene in sediment	Primary	EPA 8270C	GC/MS	34629	133	30	50-158	50-158	90
Di-n-Octyl Phthalate in water	Primary	EPA 8270C	GC/MS	34596	10	30	38-127	38-127	90
Di-n-Octyl Phthalate in sediment	Primary	EPA 8270C	GC/MS	34599	660	30	4-146	4-146	90
Endosulfan in water	Primary	EPA 8081	GC/ECD	39388	0.05	25	55-123	55-123	90
Endosulfan in sediment	Primary	EPA 8081	GC/ECD	39389	50	30	56-142	56-142	90
Endosulfan Sulfate in water	Primary	EPA 8081	GC/ECD	34351	0.05	25	51-126	51-126	90
Endosulfan Sulfate in sediment	Primary	EPA 8081	GC/ECD	34354	50	30	25-153	25-153	90
Endrin in water	Primary	EPA 8081	GC/ECD	39390	0.05	25	40-138	40-138	90
Endrin in sediment	Primary	EPA 8081	GC/ECD	39393	50	30	44-129	44-129	90
Ethylbenzene in water	Primary	EPA 8260B	GC/MS	34371	1	20	75-125	75-125	90
Ethylbenzene in sediment	Primary	EPA 8260B	GC/MS	34374	5	40	25-175	25-175	90
Fluorene in water	Primary	EPA 8270C	GC/MS	34381	4	30	48-139	48-139	90
Fluorene in sediment	Primary	EPA 8270C	GC/MS	34384	660	30	59-130	59-130	90
Fluoranthene in water	Primary	EPA 8270C	GC/MS	34376	4	30	26-137	26-137	90
Fluoranthene in sediment	Primary	EPA 8270C	GC/MS	34379	133	30	26-137	26-137	90
Guthion (Azinphos methyl) in water	Primary	EPA 8141	GC/NPD	39580	5.0	25	13-155	13-155	90
Guthion (Azinphos methyl) in sediment	Primary	EPA 8141	GC/NPD	39581	500	30	36-153	36-153	90
Heptachlor in water	Primary	EPA 8081	GC/ECD	39410	0.05	25	12-122	12-122	90
Heptachlor in sediment	Primary	EPA 8081	GC/ECD	39413	50	30	37-149	37-149	90

Appendix E Data Quality Objectives for Measurement Data

Heptachlor epoxide in water	Primary	EPA 8081	GC/ECD	39420	0.05	25	52-121	52-121	90
	Alternate/Confirmatory	EPA 1656 EPA 525.1	GC/ECD L/S Extraction + Capillary GC/MS	39420 39420	0.04 0.7	25 25	49-131 49-131	48-158 48-158	90 90
	Primary	EPA 8081	GC/ECD	39423	50	30	55-140	55-140	90
Hexachlorobenzene in water	Alternate	EPA 1656	GC/ECD	39423	1.0	25	49-131	49-131	90
	Primary	EPA 8270C	GC/MS	39700	4	30	46-133	46-133	90
	Primary	EPA 8270C	GC/MS	39701	133	30	15-152	15-152	90
Hexachlorobutadiene in water	Primary	EPA 8260B	GC/MS	34391	1	20	59-128	59-128	90
	Primary	EPA 8260B	GC/MS	39705	5	30	24-130	24-130	90
	Primary	EPA 8270C	GC/MS	34386	10	30	20-125	20-125	90
Hexachlorocyclopentadiene in sediment	Primary	EPA 8270C	GC/MS	34389	330	30	31-135	31-135	90
	Primary	EPA 8270C	GC/MS	34396	4	30	25-153	25-153	90
	Primary	EPA 8270C	GC/MS	34399	133	30	40-130	40-130	90
Indeno[1,2,3-cd]pyrene in water	Primary	EPA 8270C	GC/MS	34403	4	30	27-160	27-160	90
	Primary	EPA 8270C	GC/MS	34406	133	30	25-170	25-170	90
	Primary	EPA 8270C	GC/MS	34408	4	30	26-175	26-175	90
Isophorone in water	Primary	EPA 8270C	GC/MS	34411	133	30	25-175	25-175	90
	Primary	EPA 8141	GC/NPD	39530	0.5	25	40-132	40-132	90
	Primary	EPA 8141	GC/NPD	39531	50	30	45-127	45-127	90
Malathion in sediment	Primary	EPA 8081	GC/ECD	39480	0.05	25	39-160	39-160	90
	Primary	EPA 8081	GC/ECD	39481	50	30	37-144	37-144	90
	Primary	EPA 8260B	GC/MS	34416	5	40	15-305	15-305	90
Methoxychlor in water	Primary	EPA 8260B	GC/MS	34421	5	40	15-320	15-320	90
	Primary	EPA 8260B	GC/MS	34423	1	20	75-125	75-125	90
	Primary	EPA 8260B	GC/MS	34426	5	40	15-250	15-250	90
Methylene Chloride in sediment	Primary	EPA 8270C	GC/MS	34452	4	30	44-125	44-125	90
	Primary	EPA 8270C	GC/MS	34455	133	30	34-135	34-135	90
	Primary	EPA 8270C	GC/MS	45502	660	30	21-133	21-133	90
3-Methyl-4-Chlorophenol in water	Primary	EPA 8270C	GC/MS	77152	4	30	25-125	25-125	90
	Primary	EPA 8270C	GC/MS	77146	4	30	25-125	25-125	90
	Primary	EPA 8270C	GC/MS	78872	134	30	25-135	25-135	90
4-Methyl phenol (o-cresol)in water	Primary	EPA 8270C	GC/MS	78803	134	30	25-135	25-135	90
	Primary	EPA 8260B	GC/MS	46491	5	20	65-135	65-135	90
	Primary	EPA 8260B	GC/MS	50928	10	30	70-130	70-130	90
Methyl tert-butyl ether in sediment	Primary	EPA 8141	GC/NPD	82612	0.5	25			90
	Primary	EPA 8141	GC/NPD	38923	50	30			90
	Primary	EPA 8081	GC/ECD	39755	0.1	25			90
Metolachlor in water	Primary	EPA 8081	GC/ECD	79800	100	30			90
	Primary	EPA 8270C	GC/MS	34696	4	30	50-125	50-125	90
	Primary	EPA 8270C	GC/MS	34445	660	30	21-133	21-133	90
Mirex in sediment	Primary	EPA 8270C	GC/MS	34447	4	30	46-133	46-133	90
	Primary	EPA 8270C	GC/MS	34450	133	30	36-143	36-143	90
	Primary	EPA 8270C	GC/MS	34433	4	30	27-125	27-125	90
Naphthalene in water	Primary	EPA 8270C	GC/MS	34436	133	30	25-135	25-135	90
	Primary	EPA 8270C	GC/MS	34445	660	30	50-125	50-125	90
	Primary	EPA 8270C	GC/MS	34447	4	30	46-133	46-133	90
Naphthalene in sediment	Primary	EPA 8270C	GC/MS	34450	133	30	36-143	36-143	90
	Primary	EPA 8270C	GC/MS	34433	4	30	27-125	27-125	90
	Primary	EPA 8270C	GC/MS	34436	133	30	25-135	25-135	90
Nitrobenzene in water	Primary	EPA 8270C	GC/MS	34433	4	30	27-125	27-125	90
	Primary	EPA 8270C	GC/MS	34436	133	30	25-135	25-135	90
	Primary	EPA 8270C	GC/MS	34436	133	30	25-135	25-135	90
N-Nitrosodiphenylamine in water	Primary	EPA 8270C	GC/MS	34436	133	30	25-135	25-135	90
	Primary	EPA 8270C	GC/MS	34436	133	30	25-135	25-135	90
	Primary	EPA 8270C	GC/MS	34436	133	30	25-135	25-135	90

Appendix E Data Quality Objectives for Measurement Data

N-Nitrosodi-n-propylamine in water	µg/L	Primary	EPA 8270C	GC/MS	34428	4	30	37-125	37-125	90
N-Nitrosodi-n-propylamine in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34431	133	30	27-135	27-135	90
2-Nitrophenol in water	µg/L	Primary	EPA 8270C	GC/MS	34591	4	30	44-125	44-125	90
2-Nitrophenol in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34594	133	30	34-135	34-135	90
4-Nitrophenol in water	µg/L	Primary	EPA 8270C	GC/MS	34646	4	30	15-131	15-131	90
4-Nitrophenol in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34649	133	30	25-141	25-141	90
Parathion in water	µg/L	Primary	EPA 8141	GC/NPD	39540	0.5	25	39-136	39-136	90
Parathion in sediment	µg/kg	Primary	EPA 8141	GC/NPD	39541	50	30	33-139	33-139	90
Pentachlorophenol in water	µg/L	Primary	EPA 8270C	GC/MS	39032	4	30	28-136	28-136	90
Pentachlorophenol in sediment	µg/kg	Primary	EPA 8270C	GC/MS	39061	133	30	38-146	38-146	90
Pyrene in water	µg/L	Primary	EPA 8270C	GC/MS	34469	4	30	47-136	47-136	90
Pyrene in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34472	660	30	52-130	52-130	90
Phenanthrene in water	µg/L	Primary	EPA 8270C	GC/MS	34461	4	30	54-125	54-125	90
Phenanthrene in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34464	13310	30	54-130	54-130	90
Phenol in water	µg/L	Primary	EPA 8270C	GC/MS	34694	4	30	15-125	15-125	90
Phenol in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34695	133	30	25-135	25-135	90
PCBs in water	µg/L	Primary	EPA 8082	GC/ECD	39516	0.5	25	30-117	30-117	90
		Alternate	EPA 1656	GC/ECD	39516	0.35	25	75-119	75-119	90
PCB-1242 in water	µg/L	Primary	EPA 8082	GC/ECD	39496	0.35	25			90
		Alternate	EPA 1656	GC/ECD	39496	0.35	25	75-119	75-119	90
PCB-1254 in water	µg/L	Primary	EPA 8082	GC/ECD	39504	0.35	25			90
		Alternate	EPA 1656	GC/ECD	39504	0.35	25	75-119	75-119	90
PCB-1221 in water	µg/L	Primary	EPA 8082	GC/ECD	39488	0.35	25			90
		Alternate	EPA 1656	GC/ECD	39488	0.35	25	75-119	75-119	90
PCB-1232 in water	µg/L	Primary	EPA 8082	GC/ECD	39492	0.35	25			90
		Alternate	EPA 1656	GC/ECD	39492	0.35	25	75-119	75-119	90
PCB-1248 in water	µg/L	Primary	EPA 8082	GC/ECD	39500	0.35	25			90
		Alternate	EPA 1656	GC/ECD	39500	0.35	25	75-119	75-119	90
PCB-1260 in water	µg/L	Primary	EPA 8082	GC/ECD	39508	0.35	25			90
		Alternate	EPA 1656	GC/ECD	39508	0.35	25	75-119	75-119	90
PCB-1016 in water	µg/L	Primary	EPA 8082	GC/ECD	34671	0.35	25			90
		Alternate	EPA 1656	GC/ECD	34671	0.35	25	75-119	75-119	90
PCBs in sediment total	µg/kg	Primary	EPA 8082	GC/ECD	39519	200	30			90
	µg/kg	Alternate	EPA 1656	GC/ECD	39519	1.0	25	75-119	75-119	90
PCB-1242 In Sediment	µg/kg	Primary	EPA 8082	GC/ECD	39499	200	30			90
	µg/kg	Alternate	EPA 1656	GC/ECD	39499	1.0	25	75-119	75-119	90
PCB-1254 In Sediment	µg/kg	Primary	EPA 8082	GC/ECD	39507	200	30			90
	µg/kg	Alternate	EPA 1656	GC/ECD	39507	1.0	25	75-119	75-119	90

Appendix E Data Quality Objectives for Measurement Data

PCB-1221 In Sediment	µg/kg	Primary	EPA 8082	GC/ECD	39491	200	30				90
PCB-1221 In Sediment	µg/kg	Alternate	EPA 1656	GC/ECD	39491	1.0	25	75-119	75-119		90
PCB-1232 In Sediment	µg/kg	Primary	EPA 8082	GC/ECD	39495	200	30				90
	µg/kg	Alternate	EPA 1656	GC/ECD	39495	1.0	25	75-119	75-119		90
PCB-1248 In Sediment	µg/kg	Primary	EPA 8082	GC/ECD	39503	200	30				90
	µg/kg	Alternate	EPA 1656	GC/ECD	39503	1.0	25	75-119	75-119		90
PCB-1260 In Sediment	µg/kg	Primary	EPA 8082	GC/ECD	39511	200	30	61-118	61-118		90
	µg/kg	Alternate	EPA 1656	GC/ECD	39511	1.0	25	75-119	75-119		90
PCB-1016 In Sediment	µg/kg	Primary	EPA 8082	GC/ECD	39514	200	30	56-113	56-113		90
	µg/kg	Alternate	EPA 1656	GC/ECD	39514	1.0	25	75-119	75-119		90
Simazine in water	µg/L	Primary	EPA 8141	GC/NPD	39055	0.5	25	35-135	35-135		90
Simazine in sediments	µg/L	Primary	EPA 8141	GC/NPD	39046	50	30	35-135	35-135		90
2,4,5-T in water	µg/L	Primary	EPA 8151	GC/ECD	39740	0.10	25	45-134	45-134		90
2,4,5-T in sediment	µg/kg	Primary	EPA 8151	GC/ECD	39741	40	30	48-153	48-153		90
2,4,5-TP (Silvex) in water	µg/L	Primary	EPA 8151	GC/ECD	39760	0.1	25	46-125	46-125		90
2,4,5-TP (Silvex) in sediment	µg/kg	Primary	EPA 8151	GC/ECD	39761	40	30	54-145	54-145		90
1,1,2,2-Tetrachloroethane in water	µg/L	Primary	EPA 8260B	GC/MS	34516	1	20	74-125	74-125		90
1,1,2,2-Tetrachloroethane in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34519	5	40	35-170	35-170		90
Tetrachloroethene in water	µg/L	Primary	EPA 8260B	GC/MS	34475	1	20	71-125	71-125		90
Tetrachloroethene in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34478	10	30	70-130	70-130		90
1,2,4-Trichlorobenzene in water	µg/L	Primary	EPA 8270C	GC/MS	34551	4	30	44-142	44-142		90
1,2,4-Trichlorobenzene in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34554	133	30	34-152	34-152		90
Trichloroethylene in water	µg/L	Primary	EPA 8260B	GC/MS	39180	1	20	71-125	71-125		90
Trichloroethylene in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34487	10	40	60-170	60-170		90
1,1,1-trichloro-ethane in water	µg/L	Primary	EPA 8260B	GC/MS	34506	1	20	75-125	75-125		90
1,1,1-trichloro-ethane in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34509	5	25	70-130	70-130		90
1,1,2-trichloro-ethane in water	µg/L	Primary	EPA 8260B	GC/MS	34511	1	20	75-127	75-127		90
1,1,2-trichloro-ethane in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34514	5	25	70-130	70-130		90
2,4,5-Trichlorophenol in water	µg/L	Primary	EPA 8270C	GC/MS	77687	4	30	25-175	25-175		90
2,4,5-Trichlorophenol in sediment	µg/kg	Primary	EPA 8270C	GC/MS	78401	133	30	25-175	25-175		90
2,4,6-Trichlorophenol in water	µg/L	Primary	EPA 8270C	GC/MS	34621	4	30	39-128	39-128		90
2,4,6-Trichlorophenol in sediment	µg/kg	Primary	EPA 8270C	GC/MS	34624	133	30	29-138	29-138		90
Toluene in water	µg/L	Primary	EPA 8260B	GC/MS	34010	1	20	74-125	74-125		90
Toluene in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34483	10	30				90
Toxaphene in water	µg/L	Primary	EPA 8081	GC/ECD	39400	1.0	25	28-131	28-131		90
		Alternate/Confirmatory	EPA 1656	GC/ECD	39400	2.7	25	76-122	76-122		90
		Alternate/Confirmatory	EPA 525.1	L/S Extraction + Capillary GC/MS	39400	20	25				90
Toxaphene in sediment	µg/kg	Primary	EPA 8081	GC/ECD	39403	500	30	21-113	21-113		90
	µg/kg	Alternate	EPA 1656	GC/ECD	39403	5.0	25	76-122	76-122		90

Appendix E Data Quality Objectives for Measurement Data

Vinyl Chloride in water	µg/L	Primary	EPA 8260B	GC/MS	39175	1	20	46-134	46-134	90
Vinyl Chloride in sediment	µg/kg	Primary	EPA 8260B	GC/MS	34495	10	40	15-325	15-325	90
m,p-xylene in water	µg/L	Primary	EPA 8260B	GC/MS	85795	1	20	75-125	75-125	90
o-xylene in water	µg/L	Primary	EPA 8260B	GC/MS	77135	1	20	75-125	75-125	90
m,p-xylene in sediment	µg/kg	Primary	EPA 8260B	GC/MS	45516	10	30	70-130	70-130	90
o-xylene in sediment	µg/kg	Primary	EPA 8260B	GC/MS	78402	10	30	70-130	70-130	90
Tributyltin in water	µg/L	Primary	EV-024/025		30340	0.010	25			90
Toxicity in ambient marine water	% Survival Yes/No*	<i>Mysidopsis bahia</i>	EPA 600-4-91-003; 1007.0	Chronic Toxicity Screening Test	89805	NA	NA	NA	NA	90
Toxicity in ambient marine water	% Survival Yes/No*	<i>Menidia Berrylina</i>	EPA 600-4-91-003; 1006.0	Chronic Toxicity Screening Test	89806	NA	NA	NA	NA	90
Toxicity in marine sediment	% Survival Yes/No*	<i>Leptocheirus</i>	EPA 600-R-94-025; 100.4	Whole Sediment Toxicity Test	89815	NA	NA	NA	NA	90
Toxicity in marine sediment	% Survival Yes/No*	<i>Neanthes</i>	EPA 823-B-98-004	Whole Sediment Toxicity Test	89816	NA	NA	NA	NA	90
Freshwater toxicity	% Survival Yes/No*	<i>Ceriodaphnia dubia</i>	EPA 600-4-91-002; 1002.0	7-day subchronic test for survival, reproduction	89802	NA	NA	NA	NA	90
Freshwater toxicity	% Survival Yes/No*	<i>Pimephales promelas</i>	EPA 600-4-91-002; 1000.0	7-day test for larval survival, growth	89803	NA	NA	NA	NA	90
Toxicity for freshwater whole sediments	% Survival Yes/No	<i>Hyallela azteca</i>	EPA 600-R-94-024; 100.1	10-day survival test for sediments	89813	NA	NA	NA	NA	90
Toxicity for freshwater whole sediments	% Survival Yes/No	<i>Chironomus tentans</i>	EPA 600-R-94-024; 100.2	10-day survival and growth tests for sediments	89814	NA	NA	NA	NA	90
Benthic Macro invertebrate sampling	number	counts	TNRCC SOP	TNRCC SOP	Texas Species Code**	NA	NA	NA	NA	90
Nekton Sampling	number	counts	TNRCC SOP	TNRCC SOP	Texas Species Code**	NA	NA	NA	NA	90
Stream Habitat	NA	Counts	TNRCC SOP	TNRCC SOP	NA	NA	NA	NA	NA	90
Sediment Core Upper Depth	Inches	Grab	TNRCC SOP	TNRCC SOP	81900	NA	NA	NA	NA	90
Sediment Core Lower Depth	Inches	Grab	TNRCC SOP	TNRCC SOP	81901	NA	NA	NA	NA	90

* 1 = toxic; 2 = sublethal; 3 = none

** Individual species will be reported by TNRCC species code (TNRCC 1999)

DATA VERIFICATION REPORT
for sediment samples collected from Segment 1007A
VINCE BAYOU TMDL SITE

May 24, 2001

Data Verification by: Sandra de las Fuentes

The following data verification summary report covers environmental sediment samples collected from the Vince Bayou Segment 1007A, Station 11299, on May 23, 2001.

A Chemist with Parsons has reviewed the data submitted by DHL Analytical, B&B Laboratories, APPL, Inc. and TRAC Environmental Technology and Chemistry.

The samples in this event were analyzed for volatiles, semivolatiles, pesticides (including triazines, PCBs, organophosphorus compounds, herbicides and carbamates), total metals, anions, simultaneously extracted metals (SEM), acid volatile sulfide (AVS), total organic carbon (TOC) and grain size.

There were no field quality control samples collected at this site. No trip blanks were analyzed for volatiles and no field blanks or equipment blanks were collected in association with the sediment samples in this DVR. Therefore, the possibility of contamination during sampling or handling could not be evaluated for these samples.

All samples were collected by Parsons and were analyzed by the various laboratories following procedures outlined in the Assessment of the Presence and Causes of Ambient Toxicity Quality Assurance Project Plan (QAPP).

REVIEW CRITERIA

All data submitted by the various laboratories has been reviewed. Field and laboratory QC sample information was examined, including: laboratory blanks, laboratory control samples (LCS), laboratory duplicates, standard reference material (SRM) samples, matrix spikes and matrix spike duplicate (MS and MSD) samples, surrogate spikes and Chain-of-Custody (COC) forms. The findings presented in this report are based on the reviewed information and whether the requirements specified in the project QAPP were met.

VOLATILES

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001 and was analyzed for volatile organic compounds (VOCs). The VOC analyses were performed using USEPA SW846 Method 8260B.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the MS/MSD samples, LCS samples and surrogate spikes. A sample from another client was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this group. It should be noted that only a small subset of analytes was reported for the MS/MSD.

The percent recoveries for the LCS were all within acceptance criteria.

The percent recoveries for the MS/MSD were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was analyzed in association with the samples. The blank was free of target analytes above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All volatile results for the samples in this report were considered usable. The completeness for the VOC portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

SEMIVOLATILES

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001 and was analyzed for semivolatile organic compounds (SVOCs). The SVOC analyses were performed using USEPA SW846 Method 8270C.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the MS/MSD samples, LCS samples, and the surrogate spikes. A sample (10643-2) from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this group. It should be noted that only a small subset of analytes was reported for the MS/MSD.

All MS/MSD and surrogate %Rs were within acceptance criteria.

All LCS %Rs were within acceptance criteria.

All of the surrogate recoveries were within laboratory specified acceptance criteria for the LCS and MB except for the following:

Sample	Analyte	%R	QC Criteria
LCS	2,4,6-Tribromophenol	135	19-122
MB	4-terphenyl-d14	141	18-137

Since this surrogate compound was above control limits and all the percent recoveries for the LCS compounds were within acceptance criteria, no corrective action was taken. No action was taken for the non-compliant surrogate recovery in the MB since this surrogate compound was only slightly above control limits.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries.

All MS/MSD RPDs were within laboratory specified acceptance criteria with the exception of the following:

Analyte	MS %R	MSD %R	% RPD	QC Criteria
pentachlorophenol	72.5	53.2	30.7	30%

Pentachlorophenol was slightly above laboratory specified acceptance criteria. No corrective action was taken since the recoveries were within acceptance criteria for this compound in both the MS and MSD.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was analyzed in association with the samples. The blank was free of target analytes above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All semivolatile results for the samples in this report were considered usable. The completeness for the SVOC portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

TRIAZINES

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001, and was analyzed for triazine. The triazine compounds, atrazine, cyanazine, metolachlor and simazine, were analyzed using USEPA SW846 Method 8141A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the MS/MSD samples, LCS sample and surrogate spikes. Sample, 10643-2(ARF 35491) from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this data group.

The LCS percent recoveries were within acceptance criteria.

All MS/MSD percent recoveries were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the triazine analyses. The blank was free of any triazines above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All triazine results for the sample in this report were considered usable. The completeness for the triazine portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

PESTICIDES / PCBS

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001, and was analyzed for pesticides and PCBs. The pesticide/PCB analyses were performed using USEPA SW846 Method 8081A/8082.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample, MS/MSD samples and surrogate spikes. Sample, 10643-2(ARF 35491) from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this data group.

The LCS percent recoveries were within acceptance criteria except for the following:

Analyte	LCS %R	Lab Tolerance
Dicofol	240	50-150

Dicofol was recovered high in the LCS by laboratory acceptance criteria. The QAPP did not provide accuracy acceptance criteria, therefore non-detect results in the sample were not flagged.

All MS/MSD percent recoveries were within acceptance criteria except for the following:

Analyte	MS %R	MSD %R	Tolerance
Aldrin	42.5	37.4	46-155
b-BHC	(55.2)	46.0	51-133
chlordane	(56.9)	52.4	56-142
DDE	(64.3)	53.6	58-127
DDT	(41.8)	34.1	36-129
Endosulfan	(61.7)	51.2	56-142
Methoxychlor	(39.8)	33.2	37-144
PCB-1016	120	135	56-113

() indicates recovery met criteria.

The sample batched with the non-compliant MS/MSD %R was not flagged since the MS/MSD sample was taken from another TMDL site.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the pesticide/PCB analyses. The blank was free of any pesticides or PCBs of concern above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All pesticide/PCB results for the samples in this report were considered usable. The completeness for the pesticide/PCB portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

ORGANOPHOSPHORUS COMPOUNDS

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001, and was analyzed for organophosphorus compounds. The organophosphorus compounds, Chloropyrifos, Demeton, Diazinon, Guthion, Malathion and Parathion were analyzed using USEPA SW846 Method 8141A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample, MS/MSD samples, and surrogate spikes. Sample, 10643-2(ARF 35491) from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this data group.

The LCS percent recoveries were within acceptance criteria.

All MS/MSD percent recoveries were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the organophosphorus compound analyses. The blank was free of any organophosphorus compounds above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All organophosphorus compound results for the sample in this report were considered usable. The completeness for the organophosphorus compound portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

HERBICIDES

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001, and was analyzed for herbicides. Herbicides, 2,4,5-T, 2,4,5-TP (Silvex) and 2,4-D, were analyzed using USEPA SW846 Method 8151A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample, MS/MSD samples and the surrogate spike. Sample, 10643-2 (ARF 35491) from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this data group.

The LCS percent recoveries were within acceptance criteria.

All MS/MSD percent recoveries were within acceptance criteria with the exception of the following:

Analyte	MS %R	MSD %R	QC Criteria
2,4-D	69.1	69.8	89-175

The MS/MSD %R were below acceptance criteria, although no flags were applied to the non-detected results for this compound since the MS/MSD sample was taken from another TMDL site.

The surrogate spike recovery met laboratory specified tolerance in the samples, QC and method blanks.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

The method blank was run in association with the herbicide analyses. The blank was free of any herbicides above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All herbicide results for the samples in this report were considered usable. The completeness for the herbicide portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

CARBAMATES

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001, and was analyzed for carbamates. The carbamate compounds, carbaryl and diuron were analyzed using USEPA SW846 Method 8321A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample and surrogate spikes.

The LCS percent recoveries were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the carbamates analyses. The blank was free of any carbamates of concern above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All carbamate results for the samples in this report were considered usable. The completeness for the carbamates portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

TOTAL METALS AND IONS

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001 and was analyzed for total metals (aluminum, arsenic, barium, cadmium, calcium, chromium, copper, iron, lead, magnesium, mercury, nickel, potassium, selenium, silver, sodium and zinc). The mercury analyses were performed using USEPA SW846 Method 7471A. All other metals were determined using USEPA SW846 Method 6020B.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the LCS and MS/MSD samples. A sample (10643-2) from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this.

All LCS %Rs met acceptance criteria.

All MS and MSD %Rs met acceptance criteria except for the following:

	Analyte	MS %R	MS %R	QC Criteria
10643-2	Aluminum	-131	-111	80-120%
	Barium	73.2	78.8	
	Calcium	49.6	55.5	
	Iron	-77.4	-45.2	
	Lead	69.6	58.7	
	Mercury	(115)	122	
	Magnesium	58.2	60.5	
	Potassium	62.5	65.7	
	Sodium	53.2	54.3	
	Zinc	76.1	78.6	

There were no flags added since the sample used for the MS/MSD was from a different TMDL site as the sample in this group.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries and field duplicate analyte values.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the procedures outlined in the QAPP with the exceptions noted above.

All samples were prepared and analyzed within the hold time required by the method.

All laboratory blanks were free of target analytes above the MAL.

No calibration, analytical spike or dilution test information was provided for the analyses.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All metals results for the samples in this report were considered usable. The completeness for the metals portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

ANIONS (CHLORIDE AND SULFATE)

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001 and was analyzed for chloride and sulfate using USEPA SW846 Method 9056.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the LCS and LCSD samples.

All LCS and LCSD %Rs met acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the LCS/LCSD recoveries and field duplicate analyte values.

LCS/LCSD RPDs were within laboratory specified acceptance criteria for chloride and sulfate.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the procedures outlined in the QAPP.

All samples were prepared and analyzed within the hold time required by the method.

All laboratory blanks were free of target analytes above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All metals results for the samples in this report were considered usable. The completeness for the metals portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

SEM IN SEDIMENT

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001, and was analyzed for Simultaneously Extracted Metals (SEM), including cadmium, copper, lead, mercury, nickel, silver and zinc.

The metals analyses were performed using a modified EPA 1620 method, which is equivalent to EPA 200.7 and EPA 245.5.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the LCS and MS/MSD samples. Another client's sample was used for the MS/MSD for the batch QC for this group. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this group.

All LCS %Rs met QAPP acceptance criteria.

There was no accuracy data provided for silver and mercury.

No accuracy criteria for the MS/MSD samples were listed in the QAPP for the SEM analyses. The tolerances listed for metals analyses were used to evaluate the MS/MSD samples.

All MS %Rs met the QAPP metals acceptance criteria except for the following:

Analyte	MS %R	MSD %R	QC Criteria
Copper	76	79	80-120%
Lead	(109)	265	
Zinc	136	(101)	

() indicates recovery met criteria

Because no tolerances were specified in the QAPP for SEM matrix spike accuracy and since this sample is from another client, no corrective action was necessary.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries.

All MS/MSD RPDs were within laboratory specified acceptance criteria except for the following:

Analyte	MS %R	MSD %R	RPD	QC Limits
Lead	109	265	84%	20%

Since this sample is from another client, no corrective action was necessary.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the procedures outlined in the QAPP.

All samples were prepared and analyzed within the hold time specified in the QAPP.

All laboratory blanks were reviewed and found to be free of SEM above the MAL, except for the following:

Sample ID	Analyte	Conc. (ug/dry g)	MDL (ug/dry g)
MB	Zinc	3.09	0.24

No flags were applied since the result for zinc in the sample was greater than 5 times the result in the method blank.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All SEM results for the samples in this report were considered usable. The completeness for the SEM portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

AVS IN SEDIMENT

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001, and was analyzed for Acid Volatile Sulfide (AVS). The AVS analyses were performed using EPA method 376.3.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the LCS and MS/MSD samples. Another client's sample was used for the MS/MSD for the batch QC for this group. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this group.

All LCS %Rs met acceptance criteria.

All MS and MSD %Rs met acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the procedures outlined in the QAPP with the exceptions noted above.

All samples were prepared and analyzed within the hold time required by the QAPP.

All laboratory blanks were reviewed and found to be free of AVS at the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All AVS results for the samples in this report were considered usable. The completeness for the AVS portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

TOC

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001, and was analyzed for total organic carbon (TOC). The TOC analyses were performed using B&B Laboratories, Inc. Standard Operating Procedure 1005.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the standard reference material (SRM) samples.

TOC met acceptance criteria in both SRM samples analyzed.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

Two method blanks were analyzed in association with the samples. Both blanks were free of TOC at the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All TOC results for the samples in this report were considered usable. The completeness for the TOC portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

GRAIN SIZE

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on May 24, 2001, and was analyzed for grain size by GS-92-01-B&B Method. Grain size results are reported as a percent of sand, silt or clay based on the weight of the sample.

Accuracy

Accuracy could not be evaluated by this method.

Precision

Precision could not be evaluated by this method.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

There were no method blanks required by this method.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results for grain size for the sample in this report were considered usable. The completeness for the grain size compound portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

DATA VERIFICATION REPORT
for sediment samples collected from Segment 1007A
VINCE BAYOU TMDL SITE
July 18 and 26, 2001

Data Verification by: Sandra de las Fuentes

The following data verification summary report covers environmental sediment samples collected from the Vince Bayou Segment 1007A, Stations 11299 and 14368, on July 18 and 26, 2001.

A Chemist with Parsons has reviewed the data submitted by DHL Analytical, B&B Laboratories, APPL, Inc. and TRAC Environmental Technology and Chemistry.

The samples in this event were analyzed for volatiles, semivolatiles, pesticides (including triazines, PCBs, organophosphorus compounds, herbicides and carbamates), total metals, anions, simultaneously extracted metals (SEM), acid volatile sulfide (AVS), total organic carbon (TOC) and grain size.

The samples collected for pesticides were taken on two separate sampling events. The first event occurred on July 18, 2001 and samples 14368-5 and 14368-5 Dup were collected. The second event occurred on July 26, 2001 and sample 11299-5 was collected. APPL, Inc. analyzed the samples from the two sampling events in separate sample groups, ARF 35921 and ARF 35985, respectively. They are described in this report according to the sample group.

There were no field quality control samples collected at this site. No trip blanks were analyzed for volatiles and no field blanks or equipment blanks were collected in association with the sediment samples in this DVR. Therefore, the possibility of contamination during sampling or handling could not be evaluated for these samples.

All samples were collected by Parsons and were analyzed by the various laboratories following procedures outlined in the Assessment of the Presence and Causes of Ambient Toxicity Quality Assurance Project Plan (QAPP).

REVIEW CRITERIA

All data submitted by the various laboratories has been reviewed. Field and laboratory QC sample information was examined, including: laboratory blanks, laboratory control samples (LCS), laboratory duplicates, standard reference material (SRM) samples, matrix spikes and matrix spike duplicate (MS and MSD) samples, surrogate spikes and Chain-of-Custody (COC) forms. The findings presented in this report are based on the reviewed information and whether the requirements specified in the project QAPP were met.

VOLATILES

General

This sample group consisted of two (2) environmental sediment samples. The samples were collected on July 26, 2001 and were analyzed for volatile organic compounds (VOCs). The VOC analyses were performed using USEPA SW846 Method 8260B.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the MS/MSD samples, LCS samples and surrogate spikes. A sample from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this group. It should be noted that only a small subset of analytes was reported for the MS/MSD.

The percent recoveries for the LCS were all within acceptance criteria except for the following:

Sample	Analyte	%R	QC Criteria
LCS	Chloromethane	56.3	70-130
	Hexachlorobutadiene	133	24-130

The reported concentration for Chloromethane in the LCS was considered estimated (possibly biased low) and the samples were flagged "UJ" for non-detect results. Hexachlorobutadiene was recovered only slightly high therefore the non-detect results in the samples were not flagged.

The percent recoveries for the MS/MSD were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;

- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was analyzed in association with the samples. The blank was free of target analytes above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All volatile results for the samples in this report were considered usable. The completeness for the VOC portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

SEMIVOLATILES

General

This sample group consisted of two (2) environmental sediment samples. The samples were collected on July 26, 2001 and were analyzed for semivolatile organic compounds (SVOCs). The SVOC analyses were performed using USEPA SW846 Method 8270C.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the MS/MSD samples, LCS samples, and the surrogate spikes. A sample from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this group. It should be noted that only a small subset of analytes was reported for the MS/MSD.

All MS/MSD and surrogate %Rs were within acceptance criteria.

All LCS %Rs were within acceptance criteria.

All of the surrogate recoveries were within laboratory specified acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and

- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was analyzed in association with the samples. The blank was free of target analytes above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All semivolatile results for the samples in this report were considered usable. The completeness for the SVOC portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

TRIAZINES (ARF 35921)

General

This sample group consisted of two (2) samples, one (1) environmental sediment sample and a field duplicate sample. The samples were collected on July 18, 2001, and were analyzed for triazine. The triazine compounds, atrazine, cyanazine, metolachlor and simazine, were analyzed using USEPA SW846 Method 8141A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the MS/MSD samples, LCS sample and surrogate spikes. A sample from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this data group.

The LCS percent recoveries were within acceptance criteria.

All MS/MSD percent recoveries were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD and the field duplicate analyte values. Sample 14368-5 DUP was collected as analyzed as the field duplicate of sample 14368-5.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

All field duplicate RPDs were within acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;

- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the triazine analyses. The blank was free of any triazines above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All triazine results for the sample in this report were considered usable. The completeness for the triazine portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

TRIAZINES (ARF 35985)

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on July 26, 2001, and was analyzed for triazine. The triazine compounds, atrazine, cyanazine, metolachlor and simazine, were analyzed using USEPA SW846 Method 8141A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample and surrogate spikes.

The LCS percent recoveries were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the triazine analyses. The blank was free of any triazines above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All triazine results for the sample in this report were considered usable. The completeness for the triazine portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

PESTICIDES / PCBS (ARF 35921)

General

This sample group consisted of two (2) samples, one (1) environmental sediment sample and a field duplicate sample. The samples were collected on July 18, 2001, and were analyzed for pesticides and PCBs. The pesticide/PCB analyses were performed using USEPA SW846 Method 8081A/8082.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample, MS/MSD samples and surrogate spikes. A sample from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this data group.

The LCS percent recoveries were within acceptance criteria.

All MS/MSD percent recoveries were within acceptance criteria except for the following:

Analyte	MS %R	MSD %R	Tolerance
DDT	26.5	32.6	36-129
Methoxychlor	34.4	(41.6)	37-144

() indicates recovery met criteria.

The sample batched with the non-compliant MS/MSD %R was not flagged since the MS/MSD sample was taken from another TMDL site.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD and the field duplicate analyte values. Sample 14368-5 DUP was collected as analyzed as the field duplicate of sample 14368-5.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

All field duplicate RPDs were within acceptance criteria, except for the following:

Analyte	14368-5 Conc. (ug/Kg-dry)	14368-5 Dup Conc. (ug/Kg-dry)	Tolerance
PCB-1248	4000	ND	25%

The PCB-1248 pattern in sample 14368-5 was confirmed by the laboratory and by Parsons. The field duplicate sample, 14368-5 Dup, did not contain any PCB-1248 and therefore may indicate a field and/or laboratory error. Since it was uncertain if and when the error occurred, a “J” flag was applied to both the sample and field duplicate for PCB-1248.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the pesticide/PCB analyses. The blank was free of any pesticides or PCBs of concern above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All pesticide/PCB results for the samples in this report were considered usable. The completeness for the pesticide/PCB portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

PESTICIDES / PCBS (ARF 35985)

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on July 26, 2001, and was analyzed for pesticides and PCBs. The pesticide/PCB analyses were performed using USEPA SW846 Method 8081A/8082.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample and surrogate spikes.

The LCS percent recoveries were within acceptance criteria, except for the following:

Analyte	LCS %R	Tolerance
a-BHC	147	38-137
b-BHC	140	51-133
d-BHC	138	43-131
DDD	144	51-129
DDE	143	58-127
DDT	149	36-129
Dieldrin	154	56-125
Endrin	150	44-129
g-BHC (Lindane)	141	47-132
Heptachlor Epoxide	145	55-140
Methoxychlor	147	37-144

The non-compliant compounds all recovered high in the LCS. The sample contained low concentrations of DDD, DDE and DDT. The low detections in the sample were previously flagged as estimated ("J") since they were below the RL. No additional actions were required.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks, except for the following:

Sample	Surrogate	%R	Tolerance
LCS	TCmX	121	32-117%

No actions were taken for the high surrogate recovery for this LCS since the detected compounds in the sample were previously “J” flagged as estimated. The second surrogate, DECA, was recovered within acceptance limits.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the pesticide/PCB analyses. The blank was free of any pesticides or PCBs of concern above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All pesticide/PCB results for the samples in this report were considered usable. The completeness for the pesticide/PCB portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

ORGANOPHOSPHORUS COMPOUNDS (ARF 35921)

General

This sample group consisted of two (2) samples, one (1) environmental sediment sample and a field duplicate sample. The samples were collected on July 18, 2001, and were analyzed for organophosphorus compounds. The organophosphorus compounds, Chloropyrifos, Demeton, Diazinon, Guthion, Malathion and Parathion were analyzed using USEPA SW846 Method 8141A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample, MS/MSD samples, and surrogate spikes. A sample from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this data group.

The LCS percent recoveries were within acceptance criteria.

All MS/MSD percent recoveries were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD and the field duplicate analyte values. Sample 14368-5 DUP was collected as analyzed as the field duplicate of sample 14368-5.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

All field duplicate RPDs were within acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the organophosphorus compound analyses. The blank was free of any organophosphorus compounds above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All organophosphorus compound results for the sample in this report were considered usable. The completeness for the organophosphorus compound portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

ORGANOPHOSPHORUS COMPOUNDS (ARF 35985)

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on July 26, 2001, and was analyzed for organophosphorus compounds. The organophosphorus compounds, Chlorpyrifos, Demeton, Diazinon, Guthion, Malathion and Parathion were analyzed using USEPA SW846 Method 8141A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample and surrogate spikes.

The LCS percent recoveries were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the organophosphorus compound analyses. The blank was free of any organophosphorus compounds above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All organophosphorus compound results for the sample in this report were considered usable. The completeness for the organophosphorus compound portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

HERBICIDES (ARF 35921)

General

This sample group consisted of two (2) samples, one (1) environmental sediment sample and a field duplicate sample. The samples were collected on July 18, 2001, and were analyzed for herbicides. Herbicides, 2,4,5-T, 2,4,5-TP (Silvex) and 2,4-D, were analyzed using USEPA SW846 Method 8151A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample, MS/MSD samples and the surrogate spike. A sample from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this data group.

The LCS percent recoveries were within acceptance criteria.

All MS/MSD percent recoveries were within acceptance criteria.

The surrogate spike recovery met laboratory specified tolerance in the samples, QC and method blanks.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD and the field duplicate analyte values. Sample 14368-5 DUP was collected as analyzed as the field duplicate of sample 14368-5.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

All field duplicate RPDs were within acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

The method blank was run in association with the herbicide analyses. The blank was free of any herbicides above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All herbicide results for the samples in this report were considered usable. The completeness for the herbicide portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

HERBICIDES (ARF 35985)

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on July 26, 2001, and was analyzed for herbicides. Herbicides, 2,4,5-T, 2,4,5-TP (Silvex) and 2,4-D, were analyzed using USEPA SW846 Method 8151A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample and the surrogate spike.

The LCS percent recoveries were within acceptance criteria.

The surrogate spike recovery met laboratory specified tolerance in the samples, QC and method blanks.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

The method blank was run in association with the herbicide analyses. The blank was free of any herbicides above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All herbicide results for the samples in this report were considered usable. The completeness for the herbicide portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

CARBAMATES (ARF 35921)

General

This sample group consisted of two (2) samples, one (1) environmental sediment sample and a field duplicate sample. The samples were collected on July 18, 2001, and were analyzed for carbamates. The carbamate compounds, carbaryl and diuron were analyzed using USEPA SW846 Method 8321A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample, MS/MSD samples and surrogate spikes. A sample from another TMDL site was analyzed as the MS/MSD for this data set. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this data group.

The LCS percent recoveries were within acceptance criteria.

The MS/MSD percent recoveries were outside of acceptance limits as shown in the following:

Analyte	MS %R	MSD %R	Tolerance
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Carbaryl	41.4	63.7	34-129
Diuron	(100)	163	25-133

() indicates recovery met criteria.

The sample batched with the non-compliant MS/MSD %R was not flagged since the spiked sample was taken from another TMDL site.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD and the field duplicate analyte values. Sample 14368-5 DUP was collected as analyzed as the field duplicate of sample 14368-5.

All MS/MSD RPDs were within laboratory specified acceptance criteria except for the following:

Analyte	MS %R	MSD %R	% RPD	Lab Tolerance
Carbaryl	41.4	63.7	42.3	25%
Diuron	100	163	47.9	

The sample batched with the non-compliant MS/MSD %R was not flagged since the MS/MSD sample was taken from another TMDL site.

All field duplicate RPDs were within acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the carbamates analyses. The blank was free of any carbamates of concern above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All carbamate results for the samples in this report were considered usable. The completeness for the carbamates portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

CARBAMATES (ARF 35985)

General

This sample group consisted of one (1) environmental sediment sample. The sample was collected on July 26, 2001, and was analyzed for carbamates. The carbamate compounds, carbaryl and diuron were analyzed using USEPA SW846 Method 8321A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample and surrogate spikes.

The LCS percent recoveries were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the carbamates analyses. The blank was free of any carbamates of concern above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All carbamate results for the samples in this report were considered usable. The completeness for the carbamates portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

TOTAL METALS AND IONS

General

This sample group consisted of two (2) environmental sediment samples. The samples were collected on July 26, 2001 and were analyzed for total metals (aluminum, arsenic, barium, cadmium, calcium, chromium, copper, iron, lead, magnesium, mercury, nickel, potassium, selenium, silver, sodium and zinc). The mercury analyses were performed using USEPA SW846 Method 7471A. All other metals were determined using USEPA SW846 Method 6020B.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the LCS and MS/MSD samples. A sample from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this.

All LCS %Rs met acceptance criteria.

All MS and MSD %Rs met acceptance criteria except for the following:

Sample ID	Analyte	MS %R	MS %R	QC Criteria
10643-5	Aluminum	147	156	80-120%
	Calcium	43.5	148	
	Iron	53.9	155	
	Lead	125	(107)	

() indicates recovery met criteria.

There were no flags added since the sample spiked was from a different TMDL site as the sample in this group.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries and field duplicate analyte values.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the procedures outlined in the QAPP with the exceptions noted above.

All samples were prepared and analyzed within the hold time required by the method.

All laboratory blanks were free of target analytes above the MAL.

No calibration, analytical spike or dilution test information was provided for the analyses.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All metals results for the samples in this report were considered usable. The completeness for the metals portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

ANIONS (CHLORIDE AND SULFATE)

General

This sample group consisted of two (2) environmental sediment samples. The samples were collected on July 26, 2001 and were analyzed for chloride and sulfate using USEPA SW846 Method 9056.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the LCS and LCSD samples.

All LCS and LCSD %Rs met acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the LCS/LCSD recoveries.

LCS/LCSD RPDs were within laboratory specified acceptance criteria for chloride and sulfate.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the procedures outlined in the QAPP.

All samples were prepared and analyzed within the hold time required by the method.

All laboratory blanks were free of target analytes above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All metals results for the samples in this report were considered usable. The completeness for the metals portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

SEM IN SEDIMENT

General

This sample group consisted of three (3) samples, including two environmental sediment samples and one field duplicate sample. The samples were collected on July 19 and July 26, 2001, and were analyzed for Simultaneously Extracted Metals (SEM), including cadmium, copper, lead, mercury, nickel, silver and zinc.

The metals analyses were performed using a modified EPA 1620 method, which is equivalent to EPA 200.7 and EPA 245.5.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the LCS and MS/MSD samples. A sample from another TMDL site was analyzed as the MS/MSD sample for this data set. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this group.

All LCS %Rs met QAPP acceptance criteria.

No accuracy criteria for the MS/MSD samples were listed in the QAPP for the SEM analyses. The tolerances listed for metals analyses were used to evaluate the MS/MSD samples.

All MS/MSD %Rs met the QAPP metals acceptance criteria except for the following:

Analyte	MS %R	MSD %R	QC Criteria
Silver	0	0	80-120%
Cadmium	72	(86)	
Copper	0	0	
Lead	0	52	
Zinc	65	147	

() indicates recovery met criteria

The laboratory explained the observed variances as a product of sample inhomogeneity and matrix interference. This sample was analyzed in duplicate as shown below. As a result of the high variances in both the MS/MSD spike results and the duplicate data, the concentrations for the above compounds were considered estimated although no flags were applied since the sample spiked was taken from a different TMDL site.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries and the field duplicate samples. Sample “Duplicate 11299-5” was collected in duplicate as the field duplicate for sample “11299-5”.

All MS/MSD RPDs were within laboratory specified acceptance criteria with the exception of the following:

Analyte	MS Conc. (ug/kg)	MSD Conc. (ug/kg)	RPD	QC Limits
Lead	21.6	33.1	42%	20%

There were no flags applied to the samples since the sample spiked was taken from a different TMDL site.

All field duplicate RPDs were within acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the procedures outlined in the QAPP.

All samples were prepared and analyzed within the hold time specified in the QAPP.

All laboratory blanks were reviewed and found to be free of SEM above the MAL

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All SEM results for the samples in this report were considered usable. The completeness for the SEM portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

AVS IN SEDIMENT

General

This sample group consisted of three (3) samples, including two environmental sediment samples and one field duplicate sample. The samples were collected on July 19 and July 26, 2001, and were analyzed for Acid Volatile Sulfide (AVS). The AVS analyses were performed using EPA method 376.3.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the LCS.

All LCS %Rs met acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the field duplicate samples. Sample "Duplicate 11299-5" was collected and analyzed as the field duplicate of "11299-5".

All field duplicate RPDs were within acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the procedures outlined in the QAPP with the exceptions noted above.

All samples were prepared and analyzed within the hold time required by the QAPP.

The laboratory blank was reviewed and found to be free of AVS at the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All AVS results for the samples in this report were considered usable. The completeness for the AVS portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

TOC

General

This sample group consisted of three (3) samples, including two environmental sediment samples, and one laboratory duplicate sample randomly selected by the laboratory. The samples were collected on July 19 and 26, 2001, and were analyzed for total organic carbon (TOC). The TOC analyses were performed using B&B Laboratories, Inc. Standard Operating Procedure 1005.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the standard reference material (SRM) samples.

TOC met acceptance criteria in both SRM samples analyzed.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the laboratory duplicate. Sample, 11299-5 Dup, was randomly selected by the laboratory and analyzed as a laboratory duplicate of sample, 11299-5.

The laboratory duplicate RPD was within acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

Two method blanks were analyzed in association with the samples. Both blanks were free of TOC at the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All TOC results for the samples in this report were considered usable. The completeness for the TOC portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

GRAIN SIZE

General

This sample group consisted of four (4) samples, including two environmental sediment samples, one field duplicate sample and one laboratory duplicate sample, randomly selected by the laboratory. The sample was collected on July 19 and 26, 2001, and was analyzed for grain size by GS-92-01-B&B Method. Grain size results are reported as a percent of sand, silt or clay based on the weight of the sample.

Accuracy

Accuracy could not be evaluated by this method.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the field duplicate sample and a laboratory duplicate. Sample, 11299-5 Dup, was collected in duplicate and analyzed as a field duplicate sample of 11299-5. Sample, Dup (11299-5 Dup), was randomly selected by the laboratory as a laboratory duplicate of sample, 11299-5 Dup.

The field duplicate RPD was within acceptance criteria.

The laboratory duplicate RPD was within acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

There were no method blanks required by this method.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results for grain size for the sample in this report were considered usable. The completeness for the grain size compound portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

DATA VERIFICATION REPORT
for sediment samples collected from Segment 1007A
VINCE BAYOU TMDL SITE

April 23, 2002

Data Verification by: Sandra de las Fuentes

The following data verification summary report covers environmental sediment samples collected from the Vince Bayou Segment 1007A, Stations 14368 and 11301, on April 23, 2002.

A Chemist with Parsons has reviewed the data submitted by DHL Analytical, APPL, Inc. and TRAC Environmental Technology and Chemistry.

The samples in this event were analyzed for volatiles, semivolatiles, pesticides (including triazines, PCBs, organophosphorus compounds, herbicides and carbamates), total metals, anions, simultaneously extracted metals (SEM), acid volatile sulfide (AVS), total organic carbon (TOC) and grain size.

There were no field quality control samples collected at this site. No trip blanks were analyzed for volatiles and no field blanks or equipment blanks were collected in association with the sediment samples in this DVR. Therefore, the possibility of contamination during sampling or handling could not be evaluated for these samples.

All samples were collected by Parsons and were analyzed by the various laboratories following procedures outlined in the Assessment of the Presence and Causes of Ambient Toxicity Quality Assurance Project Plan (QAPP).

REVIEW CRITERIA

All data submitted by the various laboratories has been reviewed. Field and laboratory QC sample information was examined, including: laboratory blanks, laboratory control samples (LCS), laboratory duplicates, matrix spikes and matrix spike duplicate (MS and MSD) samples, surrogate spikes and Chain-of-Custody (COC) forms. The findings presented in this report are based on the reviewed information and whether the requirements specified in the project QAPP were met.

VOLATILES

General

This sample group consisted of two (2) environmental sediment samples. The samples were collected on April 23, 2002 and were analyzed for volatile organic compounds (VOCs). The VOC analyses were performed using USEPA SW846 Method 8260B.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the MS/MSD samples, LCS samples and surrogate spikes. Sample 11301-12 was selected by the lab as the MS/MSD for this QC batch. It should be noted that only a small subset of analytes was reported for the MS/MSD.

The percent recoveries for the MS/MSD were within acceptance criteria.

The percent recoveries for the LCS were all within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was analyzed in association with the samples. The blank was free of target analytes above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All volatile results for the samples in this report were considered usable. The completeness for the VOC portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

SEMIVOLATILES

General

This sample group consisted of two (2) environmental sediment samples. The samples were collected on April 23, 2002 and were analyzed for semivolatile organic compounds (SVOCs). The SVOC analyses were performed using USEPA SW846 Method 8270C.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the MS/MSD samples, LCS samples, and the surrogate spikes. A sample from another TMDL site was selected as the MS/MSD for this QC batch. The results for the MS/MSD will be discussed although not used to qualify the data for the sample in this group. It should be noted that only a small subset of analytes was reported for the MS/MSD.

All MS/MSD %Rs were within acceptance criteria except for the following:

Analyte	MS %R	MSD %R	Tolerance
2-chlorophenol	0	0	31-135
4-chloro-3-methylphenol	0	0	34-135
4-Nitrophenol	0	0	25-141
Pentachlorophenol	24.4	29.4	38-146
Phenol	0	0	25-135

There were no flags applied since the sample used for the MS/MSD was taken from another client's sample.

All LCS %Rs were within acceptance criteria.

All of the surrogate recoveries were within laboratory specified acceptance criteria for the samples from this TMDL site. Three of the six surrogate recoveries were below acceptance criteria for the MS and MSD; no flags were applied since the sample spiked was taken from another client's sample.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;

- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was analyzed in association with the samples. The blank was free of target analytes above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All semivolatile results for the samples in this report were considered usable. The completeness for the SVOC portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

TRIAZINES

General

This sample group consisted of two (2) environmental sediment samples. The samples were collected on April 23, 2002, and were analyzed for triazines. The triazine compounds, atrazine, cyanazine, metolachlor and simazine, were analyzed using USEPA SW846 Method 8141A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample and surrogate spikes.

The LCS percent recoveries were within acceptance criteria.

Sample	Compound	%R	Tolerance
LCS	Simazine	184	35-135%

There were no flags applies to the samples since Simazine recovered high and the sample results were non-detected for this compound.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;

- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the triazine analyses. The blank was free of any triazines above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All triazine results for the sample in this report were considered usable. The completeness for the triazine portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

PESTICIDES / PCBS

General

This sample group consisted of two (2) environmental sediment samples. The samples were collected on April 23, 2002, and were analyzed for pesticides and PCBs. The pesticide/PCB analyses were performed using USEPA SW846 Method 8081A/8082.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample and surrogate spikes.

The LCS percent recoveries were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the pesticide/PCB analyses. The blank was free of any pesticides or PCBs of concern above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All pesticide/PCB results for the samples in this report were considered usable. The completeness for the pesticide/PCB portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

ORGANOPHOSPHORUS COMPOUNDS

General

This sample group consisted of two (2) environmental sediment samples. The samples were collected on April 23, 2002, and were analyzed for organophosphorus compounds. The organophosphorus compounds, Chlorpyrifos, Demeton, Diazinon, Guthion, Malathion and Parathion were analyzed using USEPA SW846 Method 8141A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample and surrogate spikes.

The LCS percent recoveries were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the organophosphorus compound analyses. The blank was free of any organophosphorus compounds above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All organophosphorus compound results for the sample in this report were considered usable. The completeness for the organophosphorus compound portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

HERBICIDES

General

This sample group consisted of two (2) environmental sediment samples. The samples were collected on April 23, 2002, and were analyzed for herbicides. Herbicides, 2,4,5-T, 2,4,5-TP (Silvex) and 2,4-D, were analyzed using USEPA SW846 Method 8151A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample and the surrogate spikes.

The LCS percent recoveries were within acceptance criteria.

The surrogate spike recovery met laboratory specified tolerance in the samples, QC and method blanks.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

The method blank was run in association with the herbicide analyses. The blank was free of any herbicides above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All herbicide results for the samples in this report were considered usable. The completeness for the herbicide portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

CARBAMATES

General

This sample group consisted of two (2) environmental sediment samples. The samples were collected on April 23, 2002, and were analyzed for carbamates. The carbamate compounds, carbaryl and diuron were analyzed using USEPA SW846 Method 8321A.

Accuracy

Accuracy was evaluated using the percent recovery (%R) results for the LCS sample and surrogate spikes.

The LCS percent recoveries were within acceptance criteria.

All surrogate spike recoveries met laboratory specified tolerance in the samples, QC and method blanks.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

One method blank was run in association with the carbamates analyses. The blank was free of any carbamates of concern above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All carbamate results for the samples in this report were considered usable. The completeness for the carbamates portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

TOTAL METALS AND IONS

General

This sample group consisted of four (4) samples, including two (2) environmental sediment samples and one pair of MS/MSD samples. The samples were collected on April 23, 2002 and were analyzed for total metals (aluminum, arsenic, barium, cadmium,

calcium, chromium, copper, iron, lead, magnesium, mercury, nickel, potassium, selenium, silver, sodium and zinc). The mercury analyses were performed using USEPA SW846 Method 7471A. All other metals were determined using USEPA SW846 Method 6020B.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the LCS and MS/MSD samples. Sample 11301-12 was selected as the MS/MSD for this QC batch.

All LCS %Rs met acceptance criteria.

All MS and MSD %Rs met acceptance criteria except for the following:

Sample ID	Analyte	MS %R	MS %R	QC Criteria
11301-12	Aluminum	178	-312	80-120%
	Barium	(118)	125	
	Calcium	-280	-638	
	Copper	75.9	-221	
	Iron	154	57.4	
	Magnesium	27	-58.4	
	Potassium	(101)	29.4	

() indicates recovery met criteria.

There were no flags applied to the barium results in the samples since the MS and MSD % recoveries for barium were only slightly above the tolerance criteria. Aluminum, calcium, copper, iron, magnesium and potassium were all flagged “J” for detected results and “UJ” for all non-detected results, for samples 11302-12 and 14368-12.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries and field duplicate analyte values.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the procedures outlined in the QAPP with the exceptions noted above.

All samples were prepared and analyzed within the hold time required by the method.

All laboratory blanks were free of target analytes above the MAL.

No calibration, analytical spike or dilution test information was provided for the analyses.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All metals results for the samples in this report were considered usable. The completeness for the metals portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

ANIONS (CHLORIDE AND SULFATE)

General

This sample group consisted of three (3) environmental sediment samples, and one laboratory duplicate sample, randomly selected by the lab. The samples were collected on April 23, 2002 and were analyzed for chloride and sulfate using USEPA SW846 Method 9056.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the LCS and LCSD samples.

All LCS and LCSD %Rs met acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the LCS/LCSD recoveries and the laboratory duplicate analyte values. Sample 11301-12 was chosen by the laboratory as the laboratory duplicate for this QC batch.

LCS/LCSD RPDs were within laboratory specified acceptance criteria for chloride and sulfate.

The laboratory duplicate analyte values were within QAPP acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the procedures outlined in the QAPP.

All samples were prepared and analyzed within the hold time required by the method.

All laboratory blanks were free of target analytes above the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All metals results for the samples in this report were considered usable. The completeness for the metals portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

SEM IN SEDIMENT

General

This sample group consisted of five (5) samples, including two (2) environmental sediment samples, one laboratory duplicate sample and one pair of MS/MSD samples, randomly selected by the laboratory. The samples were collected on April 23, 2002, and were analyzed for Simultaneously Extracted Metals (SEM), including cadmium, copper, lead, mercury, nickel, and zinc.

The metals analyses were performed using a modified EPA 821 draft method, which is equivalent to EPA 200.7 and EPA 245.5.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the LCS and MS/MSD samples. Sample 14368-12 was analyzed as the MS/MSD sample for this data set.

All LCS %Rs met QAPP acceptance criteria.

No accuracy criteria for the MS/MSD samples were listed in the QAPP for the SEM analyses. The tolerances listed for metals analyses were used to evaluate the MS/MSD samples.

All MS/MSD %Rs met the QAPP metals acceptance criteria except for the following:

Analyte	MS %R	MSD %R	QC Criteria
Cadmium	74.8	78.4	80-120%
Copper	-297	-261.9	
Lead	(116.7)	190.1	
Nickel	70.8	72.8	
Zinc	156.2	16.6	

() indicates recovery met criteria.

There were no flags applied to cadmium and nickel results in the samples since the percent recoveries were only slightly below QC criteria. The results for copper, lead and zinc in samples 14368-12 and 11301-12 were flagged “J” for detected results and “UJ” for non-detected results.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries and the laboratory duplicate samples. Sample 14368-12 was chosen by the laboratory as the laboratory duplicate for this QC batch.

All MS/MSD RPDs were within laboratory specified acceptance criteria with the exception of the following:

Analyte	MS % Rec	MSD % Rec	RPD	RPD Limit
Lead	116.7	190.1	47.8	40%
Zinc	156.2	16.6	161.6	

There were no flags applied since the results for lead and zinc were previously flagged as estimated.

All laboratory duplicate RPDs were within acceptance criteria except for the following:

Analyte	14368-12 (mg/kg)	14368-12 Dup (mg/kg)	% RPD	RPD Limits
Copper	137	24.8	138.7	40%
Nickel	6.9	10.8	44.1	

No flags were applied to the sample results for nickel since the % RPD was only slightly above acceptance criteria. No flags were applied to the copper results in the samples since the results for this compound were previously flagged as estimated.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the procedures outlined in the QAPP.

All samples were prepared and analyzed within the hold time specified in the QAPP.

All laboratory blanks were reviewed and found to be free of SEM above the MAL

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All SEM results for the samples in this report were considered usable. The completeness for the SEM portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

AVS IN SEDIMENT

General

This sample group consisted of five (5) samples, including two (2) environmental sediment samples, and one laboratory duplicate sample randomly selected by the laboratory. The samples were collected on April 23, 2002, and were analyzed for Acid Volatile Sulfide (AVS). The AVS analyses were performed using EPA method 821.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the LCS and MS/MSD samples. Sample 14368-12 was analyzed as the MS/MSD sample for this data set.

All LCS %Rs met QAPP acceptance criteria.

All MS/MSD %Rs met the QAPP metals acceptance criteria except for the following:

Analyte	MS %R	MSD %R	QC Criteria
AVS	135	-225	60-130%

The AVS concentration in the MS and MSD samples (27.4 umol/g and 24.2 umol/g, respectively) were much greater than the amount spiked (0.89 umol/g), therefore no corrective action was needed.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the MS/MSD recoveries and the laboratory duplicate samples. Sample 14368-12 was chosen by the laboratory as the laboratory duplicate for this QC batch.

All MS/MSD RPDs were within laboratory specified acceptance criteria.

All laboratory duplicate RPDs were within acceptance criteria except.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the procedures outlined in the QAPP with the exceptions noted above.

All samples were prepared and analyzed within the hold time required by the QAPP.

The laboratory blank was reviewed and found to be free of AVS at the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All AVS results for the samples in this report were considered usable. The completeness for the AVS portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

TOC

General

This sample group consisted of three (3) samples, including two environmental sediment samples, and one laboratory duplicate sample randomly selected by the laboratory. The samples were collected on April 23, 2002, and were analyzed for total organic carbon (TOC). The TOC analyses were performed using EPA 415.1.

Accuracy

Accuracy was evaluated using the percent recovery (%R) for the laboratory control sample (LCS).

TOC met acceptance criteria for % R in the LCS sample.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) obtained from the laboratory duplicate. Sample, 11301-12, was randomly selected by the laboratory and analyzed as a laboratory duplicate sample.

The laboratory duplicate RPD was within acceptance criteria.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and
- Examining laboratory blanks for contamination of samples during analysis.

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

Two method blanks were analyzed in association with the samples. Both blanks were free of TOC at the MAL.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All TOC results for the samples in this report were considered usable. The completeness for the TOC portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

GRAIN SIZE

General

This sample group consisted of two (2) samples, including two environmental sediment samples. The samples were collected on April 23, 2002, and were analyzed for grain size by EPA 3.4 and 3.5. Grain size results are reported as a percent of gravel, sand, silt or clay based on the weight of the sample.

Accuracy

Accuracy could not be evaluated by this method.

Precision

There was no precision data available for evaluation.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing actual analytical procedures to those described in the QAPP;
- Evaluating holding times; and

All samples were prepared and analyzed following the QAPP and within the hold time required by the method.

There were no method blanks required by this method.

Completeness

Completeness was evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results for grain size for the sample in this report were considered usable. The completeness for the grain size compound portion of this data set is 100%, which meets the minimum QAPP acceptance criteria of 90%.

**APPENDIX F
TECHNICAL MEMOS ON DETERMINATION OF WATER AND
SEDIMENT TOXICITY IN AMBIENT WATERS AND SEDIMENT
SCREENING LEVELS**

Technical Memorandum 2

Sediment Quality Screening Indices

This handout is provided in response to comments from the public meeting of January 10, 2001. As requested during the meeting, sediment quality indices have been compiled and presented. A brief discussion of the indices generally available and the methodology used to complete the table follows. As discussed in the Patrick Bayou QAPP, the public meeting of January 10, 2001 and the public meeting of February 20, 2001, site-specific data collected will be used in the sediment triad approach to assess the sediment quality in the bayou.

Measured concentrations of contaminants may be compared to sediment quality screening indices to indicate whether a measured concentrations of a compound may have the potential to cause toxicity. There are many ways to derive sediment quality indices. Therefore, a discussion of the ways in which indices are derived is necessary to understand the various types of indices and how they differ.

The bulk concentration of contaminants in sediment is measured. Typically most of the bulk measured contaminant is bound in organic matter (in the case of organic compounds) and acid-volatile sulfides (in the case of metals), and not biologically available to cause toxicity in sediment. In general, organic matter has a much higher capacity for binding organic contaminants than inorganic matter. The composition of the sediments governs the bioavailability and expressed toxicity of a contaminant.

Organisms differ greatly in their sensitivity to contaminants. Toxic effects may include, but are not limited to changes in growth rates, number of offspring, behavior, physiology, and mortality. Thus, a broad range of concentrations is reported to cause toxicity. For example, DDT has been observed to cause small reductions in growth of oysters at concentrations of 0.01 µg/L in water, while fireworms (*Eurythroë complanata*) will live at 1,000 µg/L of DDT. For many contaminants, toxic effects have only been measured with a few types of organisms. Water and sediment quality indices are designed to protect all organisms from any biological effects, therefore, they are typically set well below the level that has been observed to be toxic in order to include a substantial margin of safety. Thus, contaminant levels in sediments that exceed screening indices do not necessarily indicate the presence of biological effects to the indigenous species present.

Equilibrium-Partitioning Sediment Quality Indices for Organic Compounds

Sediment quality indices based on “equilibrium partitioning” are provided in this summary. This term refers to the division, at equilibrium, of organic contaminants between sediment organic matter and the pore water present between the grains of sediments. The sediment pore water fraction is assumed to be mostly bioavailable. This approach has been used in numerous studies. The USEPA (1993) recommends it as one component of the sediment quality triad. It allows consideration of site-specific bioavailability of contaminants.

Four different equilibrium partitioning-based screening indices for the organic compounds measured in this study are listed in Table 1. While equilibrium partitioning-based indices must be calculated for each location using the site-specific organic carbon concentration, these indices are illustrated using a sediment organic carbon content of 1 percent. The illustrative value of 1 percent is typically used for general publications, since it can be easily multiplied to address site-specific organic carbon. The indices would be twice as high for a sediment with 2 percent organic carbon, three times as high for a sediment with 3 percent organic carbon, and so forth. The organic carbon content of Patrick Bayou sediments sampled in this study ranged from 1.3 percent to 18.6 percent. Therefore, the equilibrium partitioning-based indices for a given location in Patrick Bayou would be 1.3 to 18.6 times higher than the concentration in Table 1.

There is a broad range in values for those contaminants for which multiple equilibrium partitioning-based indices can be calculated. This is caused by differing assumptions used in the calculations, as well as considerable uncertainties in the data sources. In Table 1, the indices are labeled as Tier 1, Tier 2, predicted, and acute. Tier 1 sediment quality indices are available for only a few contaminants. Tier 1 indices are based on an aquatic chronic toxicity data set and were verified by EPA using whole sediment toxicity tests. The toxicity is calculated as a draft EPA final chronic value, which is based on the chronic toxicity to the most sensitive species and incorporates a substantial margin of safety. Tier 2 sediment quality indices are similar to draft Tier 1 indices, but were based on draft EPA secondary chronic values, which are based on less extensive toxicity data sets. Because there is more uncertainty regarding toxicity, EPA lowered Tier 2 indices by a factor ranging from 4 to 22 to be more protective. For some measured contaminants, Tier 1 or Tier 2 indices were not available. Therefore, "Predicted" sediment quality indices were calculated in the same way that EPA developed Tier 1 and Tier 2 indices. In some cases, these "Predicted" indices were based on expected (rather than measured) partitioning behavior, and/or very limited chronic toxicity datasets. Primary data sources used for this data set was obtained from a broad range of sources, such as EPA Region 4, EPA Office of Solid Waste and Emergency Response, and others. Thus, there is substantial uncertainty in "Predicted" sediment quality indices. Finally, no chronic toxicity information was available for several compounds. Thus, "Acute" sediment quality indices were calculated based on observed acute lethal toxicity to the most sensitive aquatic organisms. Marine acute toxicity measurements were used if available. As expected, calculations based on acute toxicity are higher than those based on chronic toxicity.

Other Sediment Quality Indices for Organic Compounds

In the absence of information about the bioavailability of contaminants, several different types of other sediment quality screening indices have been developed. To determine whether there is cause for further investigation of sediment contaminants, the State of Texas Surface Water Quality Monitoring Program applies the simplest approach. They compare individual sediment contaminant measurements at a particular location (i.e., Patrick Bayou) to the 85th percentile of all concentrations of that contaminant measured in all Texas tidal streams and estuaries. This technique focuses more on sediment quality relative to other locations than the toxicity and bioavailability of a particular compound.

Another slightly more refined approach than the one described above is based on empirical relationships between bulk sediment contaminant concentrations and observed biological effects. Indices based on this approach also do not consider site-specific conditions affecting contaminant bioavailability. They are applied without knowledge of the organic carbon content of the sediment. Several government agencies have used this method to develop sediment quality indices to screen sediments for potential biological effects. No single set of such indices has been accepted by all scientific and regulatory communities. The National Oceanic and Atmospheric Administration developed the Effects Range-Median (ER-M) and Effects Range-Low (ER-L) indices (Long and Morgan, 1991; Long et al., 1995). The ER-M is the median of the range of contaminant concentrations at which adverse biological effects were observed, while the ER-L is the tenth percentile. A second set of indices, the Probable Effects Levels (PELs) and Threshold Effects Levels (TELs), were developed for the Florida Department of Environmental Protection (MacDonald, 1994). The PEL is defined as the average of: 1) the median of the range of contaminant concentrations at which biological effects were observed; and 2) the eighty-fifth percentile of the range of concentrations at which biological effects were not observed. Thus, the PEL is similar to, but slightly lower than the ER-M. The TEL is the average of: 1) the fifteenth percentile of concentrations having biological effects; and 2) the fiftieth percentile of concentrations having no effects. The Apparent Effects Threshold (AET), developed for the State of Washington, is the highest sediment chemical concentration at which statistically significant differences in observed adverse biological effects from reference conditions do not occur. This is equivalent to the concentration above which adverse biological effects typically always occur for a given site. AETs also vary with the biological indicator examined. The AET-low is the lowest AET among multiple biological indicators (e.g., growth and reproduction effects), while the AET-high is the highest AET measured, typically mortality.

Summary of Sediment Quality Indices for Organic Compounds

Various sediment quality indices are available and each of the indices was developed with a given set of assumptions. As discussed, four types of equilibrium partitioning-based indices are presented in Table 1. These types of indices are based upon USEPA protocols. This information is provided for reference. Specific data analysis methodologies that will be applied to the Patrick Bayou sediment data for organic compounds will be based upon analysis of all of the site-specific data collected, including indigenous benthic organisms.

Sediment Quality Screening for Metals

The metals lead, cadmium, nickel, silver, zinc, and copper, form strong and biologically unavailable compounds with sulfides in sediments. Numerous studies have shown that when molar concentrations of these metals in sediments do not exceed the molar concentration of acid volatile sulfide (AVS), metal toxicity is seldom observed (Pesch et al, 1995; Casas and Crecilius, 1994; DiToro et al, 1990; Hansen et al, 1996; Berry et al, 1996). AVS is the solid-phase sulfide in sediments that is soluble in cold acid (typically 1 N hydrochloric acid). Organic matter and sediment particle surfaces may provide secondary sorbent phases to reduce the bioavailability and toxicity of metals in sediments.

The equilibrium partitioning approach will be applied to predict the toxicity of divalent metals by the method recommended by the USEPA (1994). Briefly, the sum of molar concentrations of mercury, silver, copper, lead, cadmium, zinc, and nickel extracted with the AVS (simultaneously extracted metals, or SEM) is compared to the AVS concentration. If the SEM is less than AVS, it will be assumed that the metals are bound and not causing toxicity. If SEM exceeds AVS, but the available metal concentrations do not exceed their chronic toxic values, then toxicity is again considered unlikely. Finally, metal partitioning to sediment organic matter and sediment surfaces will be evaluated with partition coefficients, as with organic compounds. If the following three criteria are met, potential metal toxicity is indicated (Ankley et al, 1996).

1. $\sum_i [SEM_i] \geq [AVS]$
2. $\sum_i \frac{[SEM_i] - [AVS]}{K_{d,oc,i} * f_{oc} * [FCV_i]} \geq 1$
3. $\sum_i \frac{[SEM_i]}{K_{d,min,i} [FCV_{i,d}]} \geq 1$

where $[SEM_i]$ is the concentration of simultaneously extractable metal i , $[AVS]$ is the concentration of acid volatile sulfide, $K_{d,oc}$ is the metal distribution coefficient between sediment organic carbon and pore water, f_{oc} is the organic carbon content of the sediment, $K_{d,min}$ is a minimum metal distribution coefficient between sediment surfaces and pore water, and $[FCV]$ is the final chronic value for toxicity of each metal.

Other Sediment Quality Indices for Metals

In the absence of the site-specific data described above, several different types of other sediment quality indices have been developed. The approaches described for other sediment quality indices of organic compounds have also been applied to metals. These approaches are the same and will not be repeated here.

Summary of Sediment Quality Indices for Metals

Various sediment quality indices are available and each of the indices was developed with a given set of assumptions. As discussed, equilibrium partitioning-based indices for metals are based upon specific sets of site-specific data. In the Patrick Bayou study, total metals, AVS, SEM and organic carbon data were collected for the sediments. Specific data analysis methodologies that will be applied to the Patrick Bayou sediment data for metals will be based upon analysis of all of the site-specific data collected, including indigenous benthic organisms.

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Table 1. Equilibrium Partitioning-Based Sediment Quality Screening Indices at 1% Organic Carbon, in µg/kg Sediment

Organic Compound	Tier 1	Tier 2	Predicted	Acute
1,1,1-Trichloroethane		170	30	26,441
1,1,2,2-Tetrachloroethane		940	1,366	12,089
1,1,2-Trichloroethane			1,257	10,157
1,1-Dichloroethane			27	2,417
1,1-Dichloroethene			31	7,259
1,2-Dichlorobenzene		340	328	
1,2-Dichloroethane			256	1,184
1,2-Dichloropropane			2,075	
1,3-Dichlorobenzene		1,700	1,664	
1,4-Dichlorobenzene		350	344	
2,4-Dinitrotoluene			293	
2,6-Dinitrotoluene				10,341
2-Chloroethyl Vinyl Ether				9,727
2-Chloronaphthalene				267,345
2-Methylnaphthalene			157	
3,3'-Dichlorobenzidine				20,603
4,4'-DDD			110	
4,4'-DDE			6,187	
4,4'-DDT			26	11,047,126
4-Bromophenyl phenyl ether		1,300	1,248	
4-Chlorophenyl phenyl ether				456,209
Acenaphthene	2,320		1,718	395,891
Acenaphthylene				30,620
Acrolein			0.005	
Acrylonitrile			1.330	46
Alpha-Chlordane			65	421,670,625
Anthracene			215	7,968
Azobenzene (1,2-diphenylhydrazine)			21	
Benzene		57	160	147,632
Benzidine			1.66	24
Benzo(a)anthracene			107	10,350,786
Benzo(a)pyrene			143	30,698,790
Benzo(b)fluoranthene				27,372
Benzo(g,h,i)perylene				7,716
Benzo(k)fluoranthene				17,418
bis(2-Chloroethoxy)methane				
bis(2-Chloroethyl)ether			368	
bis(2-Chloroisopropyl)ether				
bis(2-Ethylhexyl)phthalate			885363	
Bromodichloromethane			7426	
Bromoform		650	1307	
Bromomethane			18	
Butyl benzyl phthalate		11000	10933	

Organic Compound	Tier 1	Tier 2	Predicted	Acute
Carbon tetrachloride		1200	225	45,470
Chlorobenzene		820	413	50,361
Chloroethane				7,937
Chloroform			22	745
Chloromethane			432	
Chrysene				2,809
cis-1,3-Dichloropropene			0.05	205
Dibenzo(a,h)anthracene				15,087
Dibromochloromethane			8701	
Diethyl phthalate		630	606	
Di-n-butyl phthalate		11000	11860	81,322,597
Di-n-octylphthalate			885363	
Dioxins/furans TEQ			0.26	
Ethylbenzene		4800	90	66,435
Fluoranthene	2960		6601	17,144,309
Fluorene		540	538	
Gamma-Chlordane			65	291,925,818
Heptachlor Epoxide			2.96	
Hexachlorobenzene			13570	
Hexachlorobutadiene			171	
Hexachloroethane		1000	1021	
Mean Avg. Aroclor PCB			97	80,898,414
Mean Avg. Toxaphene		100	28	
Methylene Chloride			374	1,223
Naphthalene		470	239	239,431
Phenanthrene	2380		1859	17,412,134
Pyrene				939
Trans-1,3-Dichloropropene			230	
Trichloroethene		1600	215	
Vinyl Chloride				691

Table 2. Non-Equilibrium Partitioning-Based Sediment Quality Screening Indices, in µg/kg sediment.

Contaminant	ER-L	ER-M	AET-L	AET-H	TEL	PEL
1,2-Dichlorobenzene	-	-	50	50	-	-
1,4-Dichlorobenzene	-	-	110	120	-	-
2-Methylnaphthalene	70	670	670	1900	20.2	201
4,4'-DDD	2	20	16	43	1.22	7.81
4,4'-DDE	2.2	27	9	15	2.07	374.17
4,4'-DDT	1	7	34	34	1.19	4.77
Acenaphthene	16	500	500	2000	6.71	88.9
Acenaphthylene	44	640	1300	1300	5.87	127.87
Alpha-Chlordane	0.5	6	-	-	2.26	4.79
Anthracene	85.3	1100	960	13000	46.85	245
Arsenic	8200	70000	57000	700000	7240	41600
Benzo(a)anthracene	261	1600	1600	5100	74.8	693
Benzo(a)pyrene	430	1600	1600	3600	88.8	763
Benzo(b)fluoranthene	-	-	3600	9900	-	-
Benzo(g,h,i)perylene	-	-	720	2600	-	-
Benzo(k)fluoranthene	-	-	3600	9900	-	-
Bis(2-ethylhexyl)phthalate	182	-	1300	1900	182	2650
Butyl benzyl phthalate	-	-	900	900	-	-
Cadmium	1200	9600	5100	9600	676	4210
Chromium	81000	370000	260000	270000	52300	160000
Chrysene	384	2800	2800	9200	108	846
Copper	34000	270000	390000	1300000	18700	108000
Dibenzo(a,h)anthracene	63.4	260	230	970	6.22	135
Diethyl phthalate	-	-	200	200	-	-
Ethylbenzene	-	-	10	37	-	-
Fluoranthene	600	5100	2500	30000	113	1494
Fluorene	19	540	540	3600	21.2	144
Gamma-Chlordane	0.5	6	-	-	2.26	4.79
Heptachlor Epoxide	-	-	-	-	0.6	2.67
Hexachlorobenzene	-	-	22	230	-	-
Hexachlorobutadiene	-	-	11	270	-	-
Lead	46700	218000	450000	660000	30240	112180
Mean Avg. Aroclor PCB	22.7	180	1000	3100	21.6	188.79
Mercury	150	710	590	2100	130	700
Naphthalene	160	2100	2100	2700	34.6	391
Nickel	20900	51600	110000	-	15900	42800
Phenanthrene	240	1500	1500	6900	86.7	544
Pyrene	665	2600	3300	16000	153	1398
Silver	1000	3700	3100	-	730	1770
Zinc	150000	410000	410000	1600000	124000	271000

TECHNICAL MEMORANDUM 1

February 13, 2002

Suggested Criteria For Assessing Ambient Sediment And Water Toxicity Testing Results

INTRODUCTION

This technical memorandum recommends criteria for assessing ambient sediment and water chronic toxicity testing results. It is recommended that the lethal and sublethal end-point criteria described in this memorandum be used to identify waterbodies with varying degrees of impairment of aquatic life uses. Ambient toxicity tests exceeding the recommended criteria indicate the waterbody needs additional assessment and/or should be listed on the 303(d) and 305(b) List.

The following criteria recommendations and supporting information are divided into criteria for assessing sediment and ambient water toxicity data.

SEDIMENT RECOMMENDATIONS

Sediment Criteria 1 – Use an alpha = 0.05 when the number of replicates is less than 20. Use an alpha = 0.01 when the number of replicates is 20 or more.

To maintain a high power, 20 or more replicates should be used before using an alpha = 0.01. Otherwise, use an alpha = 0.05.

Sediment Criteria 2 – The whole-sediment toxicity test is recommended for use with ambient sediment samples. Use elutriate tests only on dredge material or when testing the effects of an activity that will cause excessive resuspension of the instream sediment.

Whole sediment toxicity testing is the preferred method because of its consistency and better approximation of actual instream conditions than elutriate testing. For gathering sediment data for aquatic life use attainment determinations, comparing whole sediment test to whole sediment test are preferred. Comparing a combination of whole sediment tests to elutriate tests is like comparing apples to oranges. Both tests are good for their intended purpose; however, for consistency, whole sediment tests are recommended rather than instream sediment testing. Use elutriate tests only on dredge material or when testing the effects of an activity that will cause excessive resuspension of the sediment.

Sediment Criteria 3 – In general, sublethal effects testing is not appropriate to short-duration sediment toxicity tests. Sublethal effects sediment toxicity test methods have not been fully developed. Long-term sublethal effects testing is new and more data are needed to assess this method. Therefore, sublethal effects testing will not be used to assess attainment of aquatic life uses at this time.

More data are needed before sublethal whole sediment toxicity tests can be considered appropriate for assessing aquatic life use attainment for instream sediment. According to EPA's freshwater sediment toxicity testing manual, "*Additional studies are ongoing to more thoroughly evaluate the relative sensitivity between lethal and sublethal endpoints measured in 10-d tests and between sublethal endpoints measured in the long-term tests (28-d). Results of these studies and additional applications of the methods described in Section 14 and 15 will provide data that can be used to assist in determining where application of long-term tests will be most appropriate.*"(1)

Sediment Criteria 4 - Mortality in the sample must also be less than the minimum control mortality allowed according to the EPA method.

For ambient sediment toxicity testing, if the conditions of test acceptability are met and survival of the test organism is equal to or greater than 80 percent of the original number of test organisms, the test shall be considered to not have demonstrated significant lethality.

The first WET test "Statistical Interpretation" provision in recent TPDES permits states, "*If the conditions of test acceptability are met and the survival of the test organism is equal to or greater than 80% in the critical dilution and all dilutions below that, the test shall be considered to not have demonstrated significant lethality.*" It is recommended that similar criteria be applied to sediment toxicity testing.

Sediment Criteria 5 – The minimum significant difference (MSD) or the minimum detectable difference (MDD) should not less than 20 percent.

In general, protocols applicable to sediment toxicity are not as well established as those for water methods. However, a 1992 EPA Region 6/ Galveston Corps of Engineers Regional Implementation Agreement for the Ocean Disposal of Dredged Material Off the Texas Coast states:

"Dredged material does not meet the LPC for benthic toxicity when bioassay organism mortality (1) is statistically greater than in the reference sediment, and (2) exceeds mortality in the reference sediment by at least 10% or exceeds the reference mortality by 20% when amphipods are used."

These approaches document ample justification for the selection of a minimum significant difference in survival of the test organism relative to the control.

A.1 WATER RECOMMENDATIONS

The following criteria are recommended:

Water Criteria 1 - Use the Fisher's Exact statistical test and the t-Test for ambient water toxicity testing for survival and sublethal effects, respectively.

Use of the Fisher's Exact statistical test and the t-Test for ambient water toxicity testing for survival and sublethal effects, respectively, is recommended. The EPA Region 6

Laboratory uses the Fisher's Exact and t-Test for determining the MSD for chronic survival and sublethal effects in ambient water toxicity testing. Although EPA's chronic whole effluent toxicity (WET) test manual allows for different statistical tests and reasonable arguments can be made for using different tests, the same statistical tests should be used to allow for a more direct comparison of results from one lab to another.

Water Criteria 2 - For ambient water survival and sublethal toxicity testing, if the conditions of test acceptability are met and survival of the test organism is equal to or greater than 80 percent of the number of test organisms at the beginning of the test, the test should be considered to not have demonstrated significant lethality.

For ambient water toxicity testing, if the conditions of test acceptability are met and survival of the test organism is equal to or greater than 80 percent of the original number of test organisms, it is recommended that the test be considered to not have demonstrated significant lethality.

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Water Criteria 3 - Use an alpha = 0.05 for determining the minimum significant difference in lethal toxicity testing and an alpha = 0.01 in sublethal toxicity testing. Sublethal toxicity test failure rates of less than 30 percent, by themselves, provide inconclusive data. The waterbody should continue to be judged as fully supporting aquatic life uses if previously designated as such. Sublethal toxicity test failure rates greater than 31 percent but less than 50 percent, by themselves, provide inconclusive evidence that the stream is not supporting aquatic life uses. Nevertheless, tests failures in the above range do indicate the stream is partially supporting the use, but additional testing is warranted. Sublethal toxicity test failure rates greater than 50 percent, by themselves, provide evidence that toxicity probably exists and the stream should be designated as not supporting aquatic life uses and that additional testing and potential toxicant identification are warranted.

The current debate between U.S. Environmental Protection Agency (EPA) and the regulated community over the interlaboratory variability of WET testing and the correlation of WET test failures with instream impairment, has spurred much interest and research. In 1995 EPA amended 40 CFR Part 136 – "Guidelines Establishing Test Procedures for the Analysis of Pollutants" to include WET testing. In 1996 the City of San Bernardino, United Water Florida, and City of Washington, Georgia sued EPA over these methods. Several items identified by the plaintiffs were clarification of the WET method procedures, guidance for use of WET test in permits, and guidance addressing when and under what circumstances a TIE/TRE should be initiated. Lone Star Steel Company also sued EPA in 1996 concerning issues related to WET test failures due to pathogens. In 1997 EPA amended and added new WET method procedures. Shortly after issuing the final WET rule, EPA was sued by the Edison Electric Institute, *et al.*,

and Western Coalition of Arid States(2). These plaintiffs claimed, among other things, that the variability of the WET tests exacerbated results because of unaccounted Type I errors. A Type I error occurs when an effluent is shown to be toxic when it is, in fact, not toxic, or when an ambient toxicity test indicates impairment of aquatic life uses when, in fact, the stream is fully supportive of aquatic life uses. All these suits were settled out of court in 1998 contingent upon separate agreements(2).

EPA's Wet Variability Study

The settlement agreements required EPA to amend most of the WET test methods and issue clarifications and new guidance. Additionally, EPA was required to perform an interlaboratory WET variability study subject to independent peer review. The final Interlaboratory WET Variability Study was published in September 2001(5). Revised WET methods were proposed in October 2001 with the comment period ending January 11, 2002.

Following the 1998 settlements through proposal of the latest revisions of the WET methods, a number of reports and professional articles were published. A study published in 2000 entitled "Investigating the Incidence of Type I Errors for Chronic Whole Effluent Toxicity Testing Using *Ceriodaphnia Dubia*"(3) sought to determine the frequency of Type I errors in *C. dubia* survival and reproductive toxicity tests. Non-toxic synthetic fresh water created using EPA's recommendations(4) was sent by participating wastewater treatment plant operators to 16 laboratories. The laboratories were not aware that the samples were non-toxic. The paper's abstract contained the following conclusion:

"Of the 16 tests completed by the biomonitoring laboratories, two did not meet control performance criteria. Six of the remaining 14 valid tests (43%) indicated toxicity ($TU_c > 1$) in the sample (i.e., no-observed-effect concentration or $IC_{25} < 100\%$ (Interpreted to mean $NOEC < 100\%$ and $IC_{25} < 100\%$)). This incidence of false positives was six times higher than expected when the critical value (alpha) was set to 0.05. No plausible causes for this discrepancy were found. Various alternatives for reducing the rate of Type I errors are recommended, including greater reliance on survival endpoints and use of additional test acceptance criteria."

The survival end-points between the control and the test for the 16 labs were not significantly different. All the false-positives mentioned above were observed in the *C. dubia* reproduction tests.

Results of this study, in part, caused EPA to propose changes(6) to the method of calculating the MSD between the control and the test for both sublethal endpoints for *C. dubia* and the fathead minnow toxicity tests. EPA is proposing to allow NPDES permit holders to reduce the nominal (Type I) error rate "alpha" from 0.05 to 0.01 when results of the test are reported as a condition of the permit or when WET permit limits are

derived without allowing for receiving water dilution. EPA set an additional condition, in the revised chronic WET manual, of not exceeding the Maximum-Minimum Significant Difference (Mx-MSD) using an alpha = 0.01. The Mx-MSD for *C. dubia* reproduction and fathead growth tests is 37 percent and 35 percent, respectively. In other words, the maximum MSD for *C. dubia* reproduction test cannot exceed 37 percent of the mean young per female in the control when using an alpha = 0.01. Insufficient replicates can cause the calculated MSD to exceed the Mx-MSD.

EPA made the decision to allow permittees to change the alpha to 0.01, not because the WET test was theoretically flawed, but because, in practice, WET test results were being used to make “yes or no” regulatory decisions. The NPDES permit holders did not want to be falsely accused by EPA of harming the environment. The same can be argued when a stream segment is listed as partially or not supporting aquatic life uses in the 305(b) Report based solely on ambient-water sublethal toxicity testing results. Stream segments listed in the 305(b) report as not supporting aquatic life uses are placed on the state’s 303(d) List.

In October 2000, EPA published preliminary results of their Interlaboratory WET Variability Study required in the above mentioned out-of-court settlement. In February 2001, the Western Coalition of Arid States (West-CAS), one of the plaintiffs in the out-of-court settlement, provided EPA its comments to the preliminary variability study(7). One comment provided by West-CAS relative to this memorandum is:

“EPA underestimated the true rate of false positives by misinterpreting results from the reference toxicant tests. The Agency acknowledged that many laboratories failed to observe toxicity in the chronic Ceriodaphnia tests on reference toxicant samples. The agency asserts, incorrectly, that the failure was due to “differences in test sensitivity between laboratories.” In fact, 9 of the 11 most sensitive tests (based on percent minimum significant difference) indicated that the reference toxicant sample was not toxic. Conversely, 9 of the 11 least sensitive tests showed the sample was toxic. On average, tests that indicated toxicity(,) were 50% less sensitive than tests that indicated no toxicity. The difference in test sensitivity was statistically-significant (p=.05). If the reference toxicant sample was actually toxic, then the most sensitive tests would be the most likely to confirm the presence of toxicity. Because that did not occur in EPA’s study, and because two-thirds of the laboratories (including the referee lab) reported no statistically-significant difference in Ceriodaphnia reproduction, the only logical conclusion is that the sample was not toxic. Therefore, the laboratories observing test failures were, in fact, reporting false positives. Based on data from the nontoxic reference toxicant tests, the true rate of Type-I error exceeds 33% for the chronic Ceriodaphnia reproduction method.”

Risk Science and West-CAS provided additional comments after the final version of the variability study was published in September 2001. The following is a comment that expands on the one provided above(8).

“Two-thirds of the laboratories failed to observe a toxic response for the reference toxicant samples during the chronic Ceriodaphnia dubia tests. Given that the most sensitive c. dubia tests indicated no toxicity and the least sensitive c. dubia tests showed toxicity, how should the true nature of the original sample be classified: toxic or non-toxic?”

In March 2001, EPA published peer review comments to the variability study. The following are some of the more interesting comments from the three reviewers, X, Y and Z, on EPA’s WET Variability Study, 2001(9).

Peer Reviewer X:

Question: *Are the results scientifically acceptable within the context of the intended regulatory use?*

Answer: “Yes and No. The data are there, though they need clarifications as noted in this review. However, I am not convinced that the Study Plan allowed for direct comparisons with regulatory use. For example, test concentrations were regimented and had larger than normal gradations, and false positives were not evaluated in terms of ecological significance but rather in terms of testing only. These tests are applied, to often, as decisive when (see Section 5 of this review, below) they are far from such.”

Comment: “First, single species toxicity tests (e.g., WET tests) are valuable first tier assessments. Results should then be used as guidance for additional studies such as exposure characterizations to provide insight on causality (e.g., TIEs), or biological assessments to provide data for detecting ecological impairment. As noted by Hall and Gidding (2000) and Chapman (2000), WET tests are the beginning, not the end of evaluations.”

Peer Reviewer Z

Question: Are the results scientifically acceptable within the context of the intended regulatory use?

Answer: “YES/NO. The results are scientifically acceptable within any context since the approach was scientifically rigorous. However, there is a distinction between scientifically acceptable in terms of accepting the results versus whether or not the results are acceptable for regulatory use. This is reminiscent of the following story: “*The operation was a success, but the patient died!*” The results should be accepted, but the results seem to show that some of these tests should not be used in the regulatory context because the successful completion rate is too low and the CV values are too high.”

Additional comment by West-CAS and the peer review committee and EPA’s response to their comments may be viewed at <http://www.toxicity.com/>

Reducing Type I Errors

Many scientific articles have been published that state or infer that WET or ambient toxicity tests in and by themselves do not necessarily indicate aquatic life uses are impaired (10, 11, 12). For *C. dubia* reproductive tests, Type I errors appear to occur, in practice, in greater than 5 percent ($\alpha = 0.05$) of the tests. Reasons include sampling and laboratory contamination, improper food preparation or contamination, individually poor performing females, not discarding results following a procedural error, parasites, pH drift, poor training, inexperience, and others (6, 11, 13). Not discarding results following a procedural error is more common than expected (7, 8). As an example, in EPA's final WET variability study, the successful *C. dubia* reproductive test completion rate for labs that met the Test Acceptance Criteria was 82 percent. Nevertheless, the successful completion rate for labs that met all non-discretionary conditions in 40 CFR Part 136 was 40 percent (7). There is also much debate as to whether WET testing correlates with instream aquatic conditions. In Section 3.5.5 of the Water Environment Research Foundation report (10) it was stated that "*Ceriodaphnia chronic reproduction NOEC showed no relationship with instream biological conditions.*" This report and specifically this statement focused on comparing results of WET testing of permitted point-source discharges to instream biological (benthic macroinvertebrate) assessments. Although this report compares WET test results from discharged effluent and not ambient water, the above quote was based, in part, on results from effluent dominated streams.

The following quote summarizes the views of many scientist and toxicologist.

"Rather than relying on a discrete, yes/no decision based on hypothesis testing of ambient toxicity tests at (α) levels of 0.1, 0.05 or 0.01, statistical interpretation of toxicity data and scientific judgement should be incorporated into the decision making process of determining when a stream segment or waterbody is impaired and considered for TMDL development." (14) Nevertheless, yes or no regulatory decisions are made on scientific evidence that may not support the regulatory action taken.

CONCLUSION

The recommended Sediment Criteria mirror previously established criteria established by the U.S. Corps of Engineers or are similar to the recommended water criteria. Water Criteria 1 and 2 are minor modifications to existing TNRCC policy. The reasons for these recommendations are noted above. Water Criteria 3 is more likely to be controversial. Unfortunately, there must be a line drawn where yes or no regulatory decisions concerning toxicity testing and attainment of aquatic life uses are made. Water Criteria 3 through 6 provide this line.

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TECHNICAL MEMORANDUM 3 IDENTIFYING WATER QUALITY TARGETS

One of the key decisions to be made in the TMDL is the identification of the water quality target. An ideal water quality target would have the following properties:

- It should be easily and inexpensively measurable,
- It should be supported by an ample historical database of quality-assured ambient and source measurements,
- A numeric criterion for the target constituent should be established in Texas surface water quality standards, and
- If the constituent is found at significant concentrations in more than one environmental phase (water, suspended sediment, bottom sediment, air), the concentrations in these phases should be related by a well-understood and well-quantified physical relationship.

The ultimate goal of the TMDL is the reduction of fish tissue concentrations of dioxins to levels that would allow the Texas Department of Health to remove the seafood consumption advisory. TNRCC guidance indicates that if a numeric water quality target exists for the identified pollutant of concern, it may be presumed to be adequate and used as a target. Texas surface water quality standards for human health protection (TNRCC 2000) provide protective levels of dioxin in fish tissue and in water that would allow removal of the fish consumption advisories: 0.47 pg/g fish tissue or 0.093 pg/L water¹.

A fish tissue concentration target has the advantage that it is more closely linked to the ultimate goal of TMDL, as both are based on fish tissue concentrations. Some quality-assured historical data are available for dioxins in fish tissue, and the levels of dioxins in tissue are somewhat easily quantified. However, relating fish tissue concentrations to

¹ ***Saltwater Tissue Standard (SW_s)***

$$SW_s = \frac{(10^{-5}) \times (70\text{kg}) \times (1000\text{ug} / \text{mg})}{(100,000\text{kg} - \text{d} / \text{mg}) \times (.015\text{kg} / \text{d}) \times (5000\text{l} / \text{kg})} = 9.3 \times 10^{-8} \text{ug} / \text{l}$$

Assumptions:

- 70kg = weight of average person
- 10⁻⁵ = incremental cancer risk level for known or suspected carcinogens (1 in 100,000)
- 0.015 kg/d = consumption rate of fish and shellfish for people living near the coast (15 grams per person per day)
- 5000l/kg = bioconcentration factor
- 100,000 kg-d/mg = q₁^{*} = Cancer potency slope factor

Fish Tissue

$$9.3 \times 10^{-8} \text{ug} / \text{l} \times 5,000\text{l} / \text{kg} = 0.00047\text{ug} / \text{kg} = 0.47\text{ng} / \text{kg} = 0.47 \text{ppt}$$

Assumptions:

- 5,000 l/kg = BCF

loading requires many calculations and assumptions that introduce substantial uncertainties.

A water concentration target is easily related to loading, facilitating the TMDL calculation and load allocation. However, the measurement of water concentrations is time-consuming and requires special, expensive equipment. No quality-assured historical data are available for dioxins in ambient waters. Furthermore, the water quality standard is derived from an acceptable fish tissue concentration, as shown in ¹, using an assumed bioconcentration factor that is applied statewide to all marine waters (5,000 l/kg for the Texas WQS). Because this assumed bioconcentration factor might not be representative of the specific conditions in the HSC system, meeting the water column criterion does not guarantee that the tissue concentrations will be lowered to acceptable levels. Also, the relationship between water concentrations and fish tissue concentrations is complex and variable, introducing substantial uncertainties. This results because dioxins in water are associated with a variety of phases in addition to being dissolved in water, and the fact that tissue concentrations are a result of both bioconcentration and bioaccumulation through prey items. On average, only about 20% of the total concentration in water is predicted to be dissolved and thus bioconcentrateable (see Section 2.2).

Neither the water nor tissue-based water quality standards provide a particularly good water quality target for all aspects of the TMDL. A water-based target would be simpler to serve for development of an initial simplified TMDL and load allocation. Therefore, the proposed plan for additional data collection includes a substantial effort to quantify dioxin concentrations in water (particulate and dissolved) in the Houston Ship Channel system, as well as tributaries, runoff, and effluents. However, it will not address the issue of the food chain pathway and bioaccumulation factors needed to provide some level of assurance that the HSC system will reach acceptable levels of dioxin in tissue. Continued collection of tissue and sediment concentration data, with quantification of biota-sediment accumulation factors, will allow the use of the tissue concentration target if that approach is selected and a more detailed or dynamic model and load allocation is developed in Phase III of the project.

An alternate approach is to apply the tissue target to sediment levels using a biota-sediment accumulation factor. Because dioxin concentrations in sediment are expected to be less dynamic than in water, biota-sediment accumulation factors may be more constant and predictable than biota-water accumulation factors. In each major environmental phase, including water, air, soil, and sediments, most dioxin is associated with particles. Quantification of dioxins associated with particles is also typically simpler than measurement of the dissolved or vapor phases. Thus, a particle-based water quality target may be a useful supplement to water or biota-based targets if a strong sediment-biota accumulation factor can be established in selected species of concern.

It should be noted that the two approaches (water-based target, fish tissue target) may be used in combination to decrease the model/TMDL uncertainty. The goal from this task is to evaluate the two approaches based on the collected data and identify the appropriate approach or combination of approaches.

**APPENDIX G
STREAM HABITAT FORMS**

Part I - Stream Physical Characteristics Worksheet

Observers: JJ/WT Date: 08-09-01 Time: 0915 Weather conditions: 90's, clear
 Stream: Vince Bayou Location of site: _____ Length of stream reach: _____
 Stream Segment No.: _____ Observed Stream Uses: _____ Aesthetics (circle one): (1) wilderness (2) natural (3) **common** (4) **offensive**
 Stream Type (Circle One): perennial or intermittent w/ perennial pools Stream Bends: No. Well Defined _____; No. Moderately Defined _____; No. Poorly Defined _____
 Channel Obstructions/Modifications: _____ Piping _____ No. of Riffles: 0 Channel Flow Status (circle one): high **moderate** low no flow

Riparian Vegetation (%):
 Left Bank: Trees 25 Shrubs 25 Grasses, Forbs 80 Cult. Fields _____ Other _____
 Right Bank: Trees 28 Shrubs 25 Grasses, Forbs 50 Cult. Fields _____ Other _____

Location of Transect	Stream Width (m)	Left Bank Slope (°)	Left Bank Erosion Potential (%)	Stream Depths (m) at Points Across Transect					Right Bank Slope (°)	Right Bank Erosion Potential (%)	Tree Canopy (%)
				Thalweg Depth: ?							
11299	50	10	50						10	50	5
	Habitat Type (Circle One) Riffle Run Glide Pool		Dominant Substrate Type ?		Dominant Types Riparian Vegetation: Left Bank: tree, shrub, grass Right Bank: tree, shrub, grass					% Gravel or Larger 0	
Algae or Macrophytes (Circle One) Abundant Common Rare Absent		Width of Natural Buffer Vegetation (m) LB: 50ft RB: 50ft		Instream Cover Types: Overhanging branches					% Instream Cover 5		

Location of Transect	Stream Width (m)	Left Bank Slope (°)	Left Bank Erosion Potential (%)	Stream Depths (m) at Points Across Transect					Right Bank Slope (°)	Right Bank Erosion Potential (%)	Tree Canopy (%)
				Thalweg Depth: ?							
14368	45	10	35						10	35	25
	Habitat Type (Circle One) Riffle Run Glide Pool		Dominant Substrate Type ?		Dominant Types Riparian Vegetation: Left Bank: tree, shrub grass Right Bank: tree, shrub, grass					% Gravel or Larger 0	
Algae or Macrophytes (Circle One) Abundant Common Rare Absent		Width of Natural Buffer Vegetation (m) LB: 25ft RB: 25ft		Instream Cover Types: Macrophyte, overhanging branches					% Instream Cover <10		

Table 3
Stream Habitat Summary

Sample Location Site Number	Units	Vince Bayou 11299	Vince Bayou 14368	Vince Bayou 14371
Date		08/09/01	08/09/01	08/09/01
Aesthetics		Common/Offensive	Common/Offensive	Common/Offensive
Stream Bends		Piping 0	Piping 0	Piping 0
Obstructions		Moderate	Moderate	Moderate
Riffles				
Flow Status				
Riparian Vegetation:				
Trees	%	27	27	27
Shrubs	%	25	25	25
Grass, Forbs	%	65	65	65
Cultivated Fields	%			
Stream Width	(ft)	50	45	30
Maximum Depth	(ft)			
In-Stream Vegetation Type		Overhanging branches	Macrophyte, overhanging branches	Grass overhang
In-Stream Cover	%	5	<10	5
Dominant Substrate Type		?	?	?
Bank Erosion	%	50	35	38
Average Bank Slope	degrees	10	10	25
Tree Canopy	%	5	25	0