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REPORT OF ENVIRONMENTAL SAMPLING
UNIMAR YARD 1 DRY DOCK FACILITY
LAKE UNION
SEATTLE, WASHINGTON

FOR

UNITED MARINE INTERNATIONAL, INC.

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FOR

UNITED MARINE INTERNATIONAL, INC.

GeoEngineers

September 19, 1991

Geotechnical,
Geoenvironmental and
Geologic Services

United Marine International, Inc.
1441 Northlake Way
Seattle, Washington 98103

Attention: Ms. Ruth Nelson

We are submitting eight copies of our Report of Environmental Sampling for the UNIMAR (Unimar International, Inc. and United Marine Shipbuilding, Inc.) Yard 1 Dry Dock Facility, Lake Union, Seattle, Washington. This study was performed in compliance with the Consent Decree (No. C85-382R) filed against UNIMAR (formerly owned and operated by WFI Industries, Inc. and Marine Power and Equipment Company Inc.) to evaluate the toxicity and extent of the sandblasting material beneath and near the Yard 1 facility. This data evaluation report is based on the information obtained during the January 1991 site study conducted jointly by UNIMAR, GeoEngineers and FishPro.

Information and conclusions contained in our report are based on our field observations, chemical analytical results and an evaluation of the data with regard to current regulatory guidelines.

Please call if you have any questions regarding this report.

Yours very truly,
GeoEngineers, Inc.

James A Miller

James A. Miller, P.E.
Principal

PGW:JAM

File No. 1299-003-B04

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REPORT OF ENVIRONMENTAL SAMPLING
UNIMAR YARD 1 DRY DOCK FACILITY
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SEATTLE, WASHINGTON
FOR
UNITED MARINE INTERNATIONAL, INC.

INTRODUCTION

The results of our environmental sampling investigation of the bottom sediments and water at the Lake Union Yard 1 Dry Dock facility are presented in this report. The Yard 1 facility is located along the north shore of Lake Union in Seattle, Washington. The UNIMAR (Unimar International, Inc. and United Marine Shipbuilding, Inc.) facility was formerly owned and operated by WFI Industries, Inc. and MPE (Marine Power and Equipment Company, Inc). The facility has been operated by UNIMAR and it's predecessors since 1967. Four to six dry dock facilities of various capacities have been in operation at the site since the mid-1950s. The two largest dry docks at the site reportedly began operations between 1979 and 1982. Four dry docks were operated at the Yard 1 facility (Figure 1) during the field sampling program. Three dry docks (#2, #9 and #6) are presently operated at the Yard 1 Facility (dry dock #8 has been removed). The dry docks are used for ship construction and repair, which usually include sandblasting and painting operations. Sandblasting grit and paint residue have accumulated on the bottom of Lake Union in and near the dry docks as a result of past operations and practices. Various types of sandblasting materials (river sand, silica sand and sandblasting grit) have been used at the facility during past operations.

PREVIOUS ON-SITE INVESTIGATIONS

The EPA (Environmental Protection Agency) collected 137 bottom sediment cores at the Yard 1 facility to estimate the extent of the sandblasting materials on the lake bottom. Sediment core logs, analytical results and bioassay results are presented in EPA's draft report "Marine Power and Equipment, Technical Status Report," dated March 3, 1987.

Additional sampling (32 sediment cores) and analysis of the bottom sediment were undertaken by MPE to further characterize the bottom sediments. The sediment core logs and analytical results for the MPE sampling are presented in GeoEngineers' "Report of Environmental Consultation, Bottom Sediment Conditions, Marine Power and Equipment, Yard 1 Dry Dock Facility, Seattle, Washington," dated June 1, 1988.

A previous GeoEngineers analysis of sandblasting material estimated approximately 5,300 cubic yards of potentially contaminated sediment on the lake bottom at the Yard 1 facility. The estimate of sandblasting material volume has been updated to approximately 6,500 cubic yards in-place. The approximate distribution of the sandblasting material is presented in Figure 2. This estimate has been compiled from data collected during past studies conducted by the EPA and GeoEngineers and has been updated to include the January 1991 sediment core data.

INDUSTRIAL IMPACTS IN LAKE UNION

The 1916 completion of the Montlake Channel and Hiram M. Chittenden Locks provided navigable passage from Lake Washington through Lake Union to Puget Sound and accelerated the rise of commercial and maritime industries along the shoreline. A report completed for the Washington State Pollution Commission in 1943 listed 45 industries along the shoreline of the Lake Union and the Lake Washington Ship Canal. Twenty of the 45 industries were listed as sources of pollution, not including the marinas and boat yards. The industries included 10 marine shops and metal foundries; 10 lumber and plywood mills; 12 fuel and oil storage and service facilities shops; eight sand, gravel, concrete and asphalt companies; the Seattle City Light Power Plant; and Gas Works Park (formerly Seattle Gas Plant), which was listed as the worst source of lake water pollution. Numerous studies have been completed for characterizing the extent of contamination at Gas Works Park, including the bottom sediments of Lake Union. A general summary of studies performed at Gas Works Park is presented at the end of this report.

A 1977 report, "A Baseline Study of the Water Quality, Sediments, and Biota of Lake Union," by Richard Tomlinson and others completed for METRO (Municipality of Metropolitan Seattle) describes the environmental impacts to Lake Union. Prior to 1960, raw sewage and stormwater outfalls discharged polluted water directly to Lake Union through numerous combined stormwater and sewage outfalls that often became overloaded with flow during rainfall events. Most of these outfalls were upgraded with the creation of Metro in the early 1960s, but 19 were still reported in use as of 1986. These combined outfalls discharged a total of more than 450 million gallons of raw sewage and stormwater into Lake Union annually. The poor water quality emanating from these outfalls is a result of pollution associated with urban runoff (houses, city streets, highways and paved areas), accidental spills, improper disposal practices and previously unregulated material storage and handling practices. The pollutants detected in samples collected from these combined outfalls include heavy metals, PCBs (polychlorinated biphenyls), oil and grease, nitrate, phosphate, coliform bacteria and pesticides.

It has been suggested that ground water discharging from upland contaminant sources such as Gas Works Park may be contributing pollutant loadings to the sediments on the lake bottom. It was reported in the METRO report that 68 percent of the total lead in Lake Union sediments enters the lake via atmospheric fallout and rainfall.

SCOPE OF WORK

The purpose of this study is to further characterize the chemical characteristics and toxicity of bottom sediments at the UNIMAR Yard 1 Dry Dock facility. Samples were collected from the bottom sediments and subsurface waters of Lake Washington, Lake Union and the Yard 1 Dry Dock facility. These samples were tested for the chemical constituents and biological test methods as described in the QA/QC plan (FishPro, QA/QC Plan for Sediment and Water Sampling at UNIMAR Yard 1 Dry Dock Facility, Seattle Washington, August 1990) and evaluated to determine the environmental risk associated with the sandblasting materials. The scope of services completed during this investigation includes the following:

1. Collect sediment core samples from nine sampling stations located in Lake Union, Lake Washington and the Yard 1 Dry Dock facility. Collect one duplicate sediment sample at a random location within the Yard 1 Dry Dock facility.
2. Collect two horizontal discrete depth water samples from locations in Lake Union (one background sample and one site-specific sample) and collect one duplicate sample at the sample location within the Yard 1 Dry Dock facility.
3. Collect rinseate water blanks from the sampling equipment prior to start of sampling and between sampling locations to document sample equipment decontamination procedures.

4. Submit the water and sediment core samples, along with all appropriate chain-of-custody documentation, to an analytical laboratory for testing.
5. Test sediment and water samples for the analyses specified in the QA/QC plan.
6. Evaluate and characterize the toxicity of the sediments based on the data compiled from the analytical testing results.
7. Combine the results of the chemical analyses with the bioassay and benthic survey to determine the environmental significance of the sediment characteristics.
8. Address the conceptual remedial plans with regard to corrective action, no action, and environmental concerns.

The chemical analytical results were evaluated based on background data compiled from the Lake Union and Lake Washington sediment samples, PSDDA (Puget Sound Dredge Disposal Analysis) guidelines, and evolving fresh water sediment regulations. The sediment core sampling, equipment decontamination, and field blank sample procedures are presented in Appendix A. The sediment core logs are presented in Appendix B. The analytical testing results are presented in Appendix C. A copy of the analytical testing method used for the analysis of tributyltin is presented in Appendix D.

FIELD STUDIES

SEDIMENT SAMPLING

A total of 42 samples from nine sediment core stations were sampled with a Shelby Tube (5-foot-long stainless steel tube that was hand-driven into the sediment by a SCUBA diver) and a Van Veen sampler to evaluate the toxicity and chemical characteristics associated with the lake bottom sediments. Two to 12 discrete sediment samples were collected from each core station according to the sample intervals specified in the QA/QC plan. The sediment samples were collected in intervals ranging between 0.3 feet and 4.7 feet. Sediment stations 1 through 7 are located at the Yard 1 Dry Dock facility (Figures 1 and 2) and stations 8 and 9 are located in Lake Union (Figure 3) and Lake Washington (Figure 4), respectively. Duplicate sediment sample 11 was collected at station 6. A description summary of the sediment sampling procedures are presented in Appendix A.

WATER SAMPLING

Two discrete water samples were collected with a horizontal Beta sampler 1 foot above the lake bottom mudline at stations 2 (sample 2A) and 8 (sample 8A). In addition, duplicate water sample 12 was collected at station 2. A total of six rinseate samples were collected from the Shelby Tube, Van Veen and Beta samplers prior to the start of sampling and at one sampling station. Presampling rinseate blanks were collected for the Shelby Tube (10A) and the Van Veen (10C) at station 9 and for the Beta sampler (10E) at station 8. Sampling rinseate blanks were collected for the Shelby Tube (10B), Van Veen (10D) and Beta samplers (10F) during sampling at stations 6 and 7.

Two of the sediment samples (2C and 8C) were partitioned into separate interstitial water and sediment samples by the analytical laboratory.

DEVIATIONS FROM PROPOSED SAMPLING PLAN

SEDIMENT SAMPLING

The deviations from the proposed QA/QC plan for the sediment sampling include relocating sediment sampling stations 1, 2, 4, 6, 7, and 9, and accepting less than a full sample core at station 9.

Sediment sampling station 1 was relocated approximately 30 feet to the south to provide acceptable recovery for the Van Veen and Shelby Tube samplers. The proposed station 1 had numerous pieces of debris that made obtaining a representative sample in the Van Veen impossible. The Shelby Tube repeatedly encountered refusal at a depth of approximately 1.5 feet and could not be driven to the required depth.

Sediment sampling station 2 was relocated approximately 20 feet to the west to allow better access for sampling with the Van Veen from the sampling platform (floating crane barge). The proposed station was located in an area with limited working area and difficult access because of the close proximity of dry dock 3 and a nearby vessel.

Sediment sampling station 4 was relocated approximately 25 feet to the west to allow the sampling platform to be docked alongside dry dock 6. The relocation allowed for better accessibility for the diver and the Van Veen sampler.

Sediment sampling stations 6 and 7 were relocated 25 feet and 15 feet, respectively, closer to the eastern shoreline bulkhead because of the positioning of the sampling platform.

Sediment sampling station 9 was relocated to the north end of Lake Washington to provide for a more representative background sample location. The maximum length of the sediment core recovered at station 9 was 2.6 feet after three cores recoveries of 2.5 feet were considered unacceptable. This length was less than the accepted criterion of 3.5 feet for the core sample recovery. The core was repeatedly driven to the 5-foot depth, but the full length of the core could not be recovered.

WATER SAMPLING

Duplicate water sample 12 was not collected immediately after water sample 2A was collected. Water sample 2A was collected prior to sampling the sediments with the Van Veen and Shelby Tube samplers. The duplicate water sample was collected at station 2 four days after the sediment sampling was performed.

ANALYTICAL TESTING METHODS

SUMMARY OF ANALYTICAL METHODS

Forty-two sediment samples were analyzed by ATI (Analytical Technologies, Inc.) of Renton, Washington, for various analytes including metals, semivolatile organic compounds (BNAs [base/neutral acid extractables] and PAHs [polynuclear aromatic hydrocarbons]), TPH (total petroleum hydrocarbons), PCBs (polychlorinated biphenyls) and moisture. Eleven of the 42 sediment samples were tested by TCLP (toxicity characteristic leaching procedure) for metals to evaluate the leaching potential of metals from the sediments. The laboratory analytical methods are presented on pages 2 and 3 at the front of each section of the certified analytical reports in Appendix C.

Three discrete water samples were tested by ATI for total metals and hardness and were submitted to their subcontracted laboratory (Tox Scan Inc., Watsonville, California) for analysis of butyltin species, in particular, TBT (tributyltin). Two sediment samples were centrifuged to separate the interstitial water from the sediment. The water samples were later analyzed for total metals and TBT. The laboratory methods for the water analyses are presented on pages 2 and 3 at the front of each section of the certified analytical reports in Appendix C. A complete description of the laboratory method used for analyzing TBT, "Measurements of Butyltin Species in Sediments by n-Pentyl Derivation with (GC/MS) Gas Chromatography/ Mass Spectrometry," is presented in Appendix D.

DEVIATIONS FROM THE PROPOSED WORK PLAN FOR SAMPLE ANALYSES

Two sets of sediment samples, 1B and 1C, were collected from station 1 on separate days and recorded on separate chain-of-custody sheets (lab IDs 9101-191-11 and -12, and 9101-203-1 and -2). The first set of samples was not analyzed because of insufficient core recovery. The precise sediment core sample interval for the first set of samples was questionable because a rock had blocked the core barrel. The second set of samples collected from station 1 was submitted for analyses. Sample 1C of this set was mistakenly recorded on the second chain-of-custody sheet; therefore, this sample was analyzed for PCBs and not PAHs. This error was not discovered until after the holding times expired.

Sediment sample 8D was not analyzed for the proposed list of analyses specified in the QA/QC plan because the sample quantity was insufficient. Sample 8D was analyzed only for the metals on the metals screen list in the QA/QC plan. Sediment sample 8C was analyzed for the full suite of testing as a replacement for 8D.

Sample 8C was centrifuged in the laboratory to separate the interstitial water from the sediment and recorded on page 2 of the chain-of-custody sheet as samples 8C water (lab ID 9101-167-15) and 8C sediment (lab ID 9101-167-16). The sediment portion of sample 8C was analyzed for the analyses specified for 8D, including TPH, PAHs and BNAs, PCBs, total metals and TCLP metals. The interstitial water portion of sample 8C was analyzed for total metals and TBT.

Sample 8F was not analyzed for PAHs because the sample quantity was insufficient. Sample 8F was analyzed for TPH and metals.

The detection limits specified in the final QA/QC plan for copper and silver (0.002 mg/l) were not met by the laboratory because of a communications error in reporting the required detection limits to the laboratory. It was not possible to reanalyze the water samples because the samples were disposed of before this error was discovered.

DEVIATIONS FROM THE PROPOSED WORK PLAN FOR ANALYTICAL TESTING METHODS

Three of the proposed analytical laboratory test methods (hardness, EP Toxicity and TBT) were replaced with alternative or updated methods during the investigation. The proposed analytical test for hardness (EPA Method 130.1), which is performed by automatic titration, was replaced by EPA Method 130.2, which is performed by manual titration. The proposed leaching procedure for metals analysis, EP toxicity was replaced by the new TCLP (toxicity characteristic leaching procedure) for metals. Theses two replacement procedures were approved by the EPA before beginning the study. The proposed analytical method for TBT analysis by Atomic Absorption/Hydride was replaced with an updated method for TBT analysis, "Measurements of Butyltin Species in Sediments by n-Pentyl Derivation with GC/MS." The request for changing this method was documented in the January 17, 1991 letter to the EPA from GeoEngineers. Verbal approval for the updated TBT analytical method was granted in the field during a site visit by the EPA.

The detection limits specified in the final draft QA/QC plan for silver and copper (0.002 mg/l) in water samples were not met by the analytical laboratory because of a communications error in reporting the final required detection limits to the laboratory.

SAMPLE INTEGRITY

All samples were immediately placed on ice upon collection, placed in insulated shipping containers, and delivered to the laboratory with chain-of-custody seals intact and containing all completed chain-of-custody paperwork.

Samples 1B through 1H, 3A and 3B, sampled on Friday, January 25, 1991, arrived at the laboratory on Monday, January 28, 1991 with unfrozen ice packs (lab ID 9101-203). This shipping container was sealed with fresh ice packs on Friday evening, but did not arrive at the laboratory until Monday morning with the COC seals intact. All other samples arrived at the laboratory cold and intact.

QA/QC SAMPLE VALIDATION

Overall, the QA/QC data indicate that the accuracy and reproducibility of the samples were acceptable. All soil and water samples were extracted and analyzed within the recommended holding times with the exception of mercury, which was tested after the 28-day holding time during the TCLP metals analyses. The TCLP testing procedures specified in the QA/QC plan required the samples to be air-dried 60 days before extraction.

Low concentrations of copper and zinc were noted in the sediment sample reagent blanks for two of the batch analyses. These low levels do not represent a significant deviation from the recommended QA/QC procedures. No contaminants were reported in the water sample reagent blanks.

There were some general matrix interference problems noted in a few of the analytical batches, which were indicated by percent recoveries that were out of the control limits for mercury and zinc.

The QA/QC data for the PCB analyses were well within all acceptable limits for surrogate recoveries and no contamination was detected in the reagent blanks. The detection limits for sediment samples for stations 6 and 7 were elevated as a result of the significant levels of contamination present in the matrix.

The QA/QC data for the TPH analyses were within acceptable limits and no contamination was detected in the reagent blank samples.

The QA/QC data for the PAH analyses were within acceptable limits for the surrogate recoveries and no contamination was reported in the reagent blank samples. The QA/QC data for the BNA analyses were generally within acceptable limits. The accuracy of EPA Methods 8270 and 8310 was demonstrated in sample 8C, which was analyzed by both methods and attained similar results. Surrogate recoveries were generally within acceptable limits. Tentatively identified compounds were reported at very low concentrations in some of the reagent blanks.

The QA/QC data for the TCLP metals analyses were within acceptable limits and no contamination was reported in the reagent blank.

A total of six field rinseate samples were collected prior to the first sampling attempts (10A, 10C and 10E) and during the field study (10B, 10D and 10F) to confirm decontamination procedures. The results of the analytical testing for the equipment blanks are presented in each analytical summary section.

SUMMARY OF ANALYTICAL TESTING RESULTS--SEDIMENT SAMPLES

METALS

Quantification of metals in the sediment samples was performed by analyzing the samples for either total metals (arsenic, barium, cadmium, chromium, copper, lead, mercury, nickel, selenium, silver and zinc) or the metals on the metals screen list (cadmium, copper, lead, and zinc). A summary of the metals analyses for sediment samples is presented in Table 1.

The following sections present the general trends in the sediment concentrations of the background samples compared to the on-site samples. Duplicate sample 6A, designated sample 11, is included in the evaluation but will not be referenced unless a significant difference is noted between the two samples.

Arsenic:

Background Sampling Stations 9 and 8 - Arsenic was detected in background Lake Washington sediment samples 9A and 9B at concentrations of 8.9 mg/kg (milligrams per kilogram) and 2.3 mg/kg, respectively. The arsenic concentrations detected in background Lake Union sediment samples 8B (71 mg/kg) and 8C (24 mg/kg) were approximately nine and ten times greater than the concentrations found in Lake Washington for similar sampling intervals.

Sampling Stations 1 Through 7 - Arsenic was detected in the sediment samples from stations 1 through 7 at concentrations between 150 mg/kg and 3,100 mg/kg in the shallow interval (0 to 0.3 feet) (Figure 5). These concentrations are significantly greater than background concentrations. Arsenic detected in the sediment samples in the deep interval (0.3 to 5.0 feet) at stations 3 through 7 ranged from 26 mg/kg to 180 mg/kg (Figure 6), which are generally greater than the background concentrations. The highest arsenic concentrations (greater than 1,800 mg/kg) were detected at stations 1, 2, and 4.

Barium:

Background Sampling Stations 9 and 8 - Barium was detected in background sediment samples 9A and 9B at concentrations of 110 mg/kg and 51 mg/kg, respectively. Barium was detected in the background Lake Union sediment samples at generally similar concentrations of 92 mg/kg in both 8B and 8C.

Sampling Stations 1 Through 7 - Barium was detected in the sediment samples from stations 1 through 7 at concentrations between 32 mg/kg and 380 mg/kg. The highest barium concentrations (greater than 210 mg/kg) were generally detected at stations 1, 2 and 4. The concentrations detected from the other stations were generally less than two times the background results.

Cadmium:

Background Sampling Stations 9 and 8 - Cadmium was not detected in the background sediment samples at station 9. Cadmium was detected in the background sediment samples from station 8 at concentrations generally between 2 mg/kg and 3.6 mg/kg. However, 12 mg/kg cadmium was detected in 8M, which is from the 4.5 to 4.8 feet interval. The high concentration detected in 8M shows the possible background range of cadmium concentrations present in Lake Union.

Sampling Stations 1 Through 7 - Cadmium was generally detected in the sediment samples from the stations 1 through 7 at concentrations ranging from 4.4 mg/kg to 27 mg/kg for the shallow interval (Figure 7). Most of the samples resulted in cadmium concentrations greater than the background concentrations. The cadmium concentrations in the shallow interval were generally about two times greater than the concentrations in the deeper interval (Figure 8).

Chromium:

Background Sampling Stations 9 and 8 - Chromium was detected in background sediment samples 9A and 9B at concentrations of 39 mg/kg and 32 mg/kg, respectively. Chromium was detected in background sediment samples 8B and 8C at similar concentrations of 47 mg/kg and 32 mg/kg, respectively.

Sampling Stations 1 Through 7 - Chromium was detected in the sediment samples from stations 1 through 7 at concentrations generally ranging from 18 mg/kg to 89 mg/kg. However, a chromium concentration of 230 mg/kg was detected in sample 2C. Most of the sample results were 1.5 to 2 times greater than the background results. The deeper interval concentrations are generally similar to or slightly greater than the background concentrations.

Copper:

Background Sampling Stations 9 and 8 - Copper was detected in background sediment samples 9A and 9B at concentrations of 38 mg/kg and 15 mg/kg, respectively. The copper concentrations detected in background sediment samples 8B (170 mg/kg) and 8C (94 mg/kg) were approximately four and six times the concentrations detected in samples from background station 9 for similar depth intervals. Copper was detected in the discrete background sediment samples from station 8 at concentrations generally between of 12 mg/kg and 180 mg/kg, with a significant decrease (less than 39 mg/kg) below the 3.0- to 3.5-foot interval.

Sampling Stations 1 Through 7 - Copper was detected in the sediment samples from stations 1 through 7 at concentrations between 230 mg/kg and 5,900 mg/kg in the shallow interval (Figure 9). These concentrations are significantly greater than background concentrations (9A and 8B). Copper was generally detected at concentrations exceeding background concentrations in sediments at stations 3B through 7B, with values between 89 mg/kg and 252 mg/kg in the deep interval (Figure 10). The highest copper concentrations (greater than 1,200 mg/kg) were detected in samples from stations 1, 2, 4 and 6. The copper concentrations detected in the sediment samples from stations 1 and 2 decreased significantly (to less than 100 mg/kg) below the 3.0-foot (1G) and 4.0-foot (2I) discrete sample depths.

Lead:

Background Sampling Stations 9 and 8 - Lead was detected in background sediment sample 9A at a concentrations of 68 mg/kg and was not detected in sample 9B. The lead concentrations detected in background sediment samples 8B (250 mg/kg) and 8C (130 mg/kg) were approximately four to greater than ten times the concentrations present at background station 9 for similar depth intervals. Lead was detected in the discrete background sediment samples from station 8 at concentrations generally between less than 40 mg/kg and 350 mg/kg, with an apparent decrease (to less than 50 mg/kg) below the 3.0- to 3.5-foot interval.

Sampling Stations 1 Through 7: Lead was detected in the sediment samples from stations 1 through 7 at concentrations between 210 mg/kg and 2,900 mg/kg in the shallow interval (Figure 11). These concentrations are significantly greater than background concentrations (9A and 8B). Lead was generally detected at concentrations greater than background concentrations in sediment samples 3B through 7B, with values between 97 mg/kg to 230 mg/kg from the deep interval (Figure 12). The highest lead concentrations (greater than 1,700 mg/kg) were detected at stations 1, 2, and 4. The lead concentrations detected in the sediment samples from stations 1 and 2 decreased significantly (to less than 100 mg/kg) below the 3.0-foot (1G) and 4.0-foot (2I) discrete sample depths.

Mercury:

Background Sampling Stations 9 and 8 - Mercury was not detected in the background sediment samples at station 9. Mercury was detected in background sediment samples 8B and 8C at concentrations of 1.53 mg/kg and 1.18 mg/kg, respectively. The station 8 sample results will be used as background concentrations for Lake Union.

Sampling Stations 1 Through 7 - Mercury either was not detected or was detected in sediment samples from stations 1 through 7 at concentrations ranging from 0.80 mg/kg to 1.52 mg/kg. The sample results show no significant difference compared to the Lake Union background concentrations for mercury.

Nickel:

Background Sampling Stations 9 and 8 - Nickel was detected in background sediment samples 9A and 9B at concentrations of 41 mg/kg and 31 mg/kg, respectively. Nickel was detected in the background sediments samples from 8B and 8C at slightly greater concentrations of 48 mg/kg and 49 mg/kg, respectively.

Sampling Stations 1 Through 7 - Nickel was generally detected in sediment samples from stations 1 through 7 at concentrations ranging from 13 mg/kg to 78 mg/kg; the greatest concentration (130 mg/kg) was detected in sample 2C. Most of the sample results generally were less than 1.5 times the background results. The deeper interval sample concentrations were generally slightly greater than the background concentrations with the exception of the samples from station 3, which were less than 0.5 times the background concentrations.

Selenium:

Sampling Stations 1 Through 9 - Selenium was not detected in the background sediment samples or in the Yard 1 Dry Dock samples.

Silver:

Sampling Stations 1 Through 9 - Silver was not detected in the background sediment samples but was detected at concentrations of 2 mg/kg to 4.7 mg/kg in sediment samples from stations 1 through 4.

Zinc:

Background Sampling Stations 9 and 8 - Zinc was detected in background sediment samples 9A and 9B at concentrations of 120 mg/kg and 44 mg/kg, respectively. The zinc concentrations detected in background sediment samples 8B (340 mg/kg) and 8C (260 mg/kg) were approximately three to six times greater than the concentrations present in samples from background station 9 at similar depth intervals. Zinc was detected in the discrete background sediment samples from station 8 at concentrations between 39 mg/kg and 430 mg/kg, with an apparent decrease (to less than 100 mg/kg) below the 3.0- to 3.5-foot interval.

Sampling Stations 1 Through 7 - Zinc was generally detected in the sediment samples from stations 1 through 7 at concentrations significantly greater than background concentrations (9A and 8B) with values between 660 mg/kg and 13,000 mg/kg in the shallow interval (Figure 13). Zinc was generally detected at concentrations between 210 mg/kg and 620 mg/kg from the deep interval (Figure 14); these concentrations are greater than background concentrations in samples 3B through 7B. The highest zinc concentrations (greater than 4,000 mg/kg) were detected in samples from stations 1, 2 and 4. The zinc concentrations detected in the sediment samples from stations 1 and 2 decreased significantly (to less than 190 mg/kg) below the 3.0-foot (1G) and 4.0-foot (2I) discrete sample depths.

SEMIVOLATILE ORGANIC COMPOUNDS

The quantification of semivolatile organic compounds in the sediment samples was performed by analyzing the samples by EPA Methods 8310 (PAHs) and 8270 (BNAs). The data summarized in Table 2 were compiled from the entire list of semivolatile analyses. Table 2 includes only the constituents detected and the method detection limits. Estimated values are included in Table 2 but tentatively identified compounds are not included, because these compounds have no intrinsic value in our analysis. Tentatively identified compounds (detected by the mass spectrometer) have molecular spectral patterns that are similar in composition to one or more compounds, thus individual compounds cannot be distinguished and are placed into molecular categories (compound classes) during the computer search.

The majority of the semivolatile organic compounds results fall into two main categories: LPAHs (low molecular weight polynuclear aromatic hydrocarbons) including acenaphthene, acenaplthylene, fluorene, naphthalene, phenanthrene, and 2-methylnaphthalene; and HPAHS (high molecular weight polynuclear aromatic hydrocarbons) including anthracene, benzo(a)anthracene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(g,h,i)perylene, benzo(a)-pyrene, chrysene, dibenz(a,h)anthracene, fluoranthene, indeno(1,2,3-cd)-

pyrene, and pyrene. Two additional compounds presented in Table 2 under the miscellaneous category include bis(2-ethylhexyl)phthalate and dibenzofuran. The following sections briefly summarize and compare the general trends of the background and on-site data results for the LPAHs, HPAHs and miscellaneous compounds.

General Summary LPAHs, HPAHs and Miscellaneous Compounds:

Background Sampling Stations 9 and 8 - Semivolatile organic compounds were not detected in background sediment sample 9A greater than the method detection limit (0.68 mg/kg). The total concentrations (sum) of semivolatile organic compounds in the sediment samples from background station 8 are approximately 45 mg/kg for LPAHs and 211 mg/kg for HPAHs for the shallow interval and approximately 5 mg/kg for LPAHs and 41 mg/kg for HPAHs for the 0.3-foot to 2.0-foot interval. The Lake Union background results represent a significant difference in concentrations compared to the Lake Washington background results. Miscellaneous compounds were not detected in sediments at station 8. No comparisons will be made with the miscellaneous compounds.

Sampling Stations 1 Through 7 - The total LPAHs detected in sediment samples from stations 2, and 4 through 7 were generally at concentrations greater than 55 mg/kg, which is significantly greater than the total background concentrations from 8B (Figures 15 and 16). The total LPAH concentrations in these sediment samples range from approximately 56 mg/kg (5A) to 349 mg/kg (6A) for the shallow interval, while the results from the deeper interval range from 13 mg/kg (5B) to 384 mg/kg (6B). The sediment sample results for stations 1 and 3 indicate that the detected total LPAH concentrations are much less than the total background concentration for 8B (46 mg/kg) in the shallow interval, with approximate concentrations between 16 mg/kg to 20 mg/kg. However, the total LPAH concentrations for 3B (20 mg/kg) was greater (21 mg/kg) than background sample 8C (5 mg/kg) in the deeper interval. Stations 6 and 7 have the highest overall LPAH sediment contamination while stations 1 and 3 have the lowest. Stations 2, 4 and 5 generally fall within the low to middle range of overall LPAH concentrations.

The total HPAH concentrations detected in the samples at stations 1 through 7 generally are either less than 76 mg/kg or greater than 145 mg/kg (Figures 17 and 18). Total HPAHs were detected in the greatest concentrations at stations 4 through 7, with ranges of approximately 145 mg/kg to

173 mg/kg at stations 4, 6 and 7 for both sample intervals, and approximately 302 mg/kg and 88 mg/kg for the shallow and deeper intervals at station 5. Total HPAH concentrations were the lowest in the sediment samples from stations 1 through 3 (between 26 mg/kg and 76 mg/kg for both intervals). Overall, however, significant HPAH concentrations were detected in the background and on-site samples.

A general evaluation of the semivolatile organic compounds data shows that sediments at stations 1 through 3 have the lowest LPAH and HPAH concentrations, while the highest concentrations are present at stations 4 through 7 and background station 8.

No general trends are evident in the sediment samples for the two miscellaneous semivolatile compounds. Bis(2-ethylhexyl)phthalate was detected in samples from stations 1 through 4 at concentrations between 1.8 mg/kg (estimated value) and 10 mg/kg. Dibenzofuran was detected in samples from stations 1, 2, 4 and 6 at concentrations between 1.0 mg/kg (estimated value) and 14 mg/kg.

<u>Rinseate Samples 10A Through 10C</u> - PAHs were not detected in the rinseate samples greater than the method detection limits (0.05 μg/l to 1.0 μg/l) from the Shelby Tube (10A and 10B) and the Van Veen (10C and 10C) samplers. Rinseate samples for PAHs were not collected for the Beta sampler.

TOTAL PETROLEUM HYDROCARBONS

A summary of the TPH (total petroleum hydrocarbon) results is presented in Table 3. The sediment samples from stations 1 and 3 through 9 were analyzed for TPH. Samples from station 2 were not analyzed for TPH.

<u>Background Sampling Stations 9 and 8</u> - Total petroleum hydrocarbons were detected in the background Lake Washington sediment samples 9A and 9B at concentrations of 65 mg/kg and 13 mg/kg, respectively. The TPH concentrations detected in background Lake Union sediment samples 8B (120 mg/kg) and 8F (350 mg/kg) were approximately five to ten times the concentrations found in Lake Washington for generally similar sample intervals.

<u>Sampling Stations 1, and 3 Through 7</u> - Total petroleum hydrocarbons were detected at concentrations between 39 mg/kg (5B) and 1,600 mg/kg (1A)

in the sediment samples from the Yard 1 Dry Dock facility. No general trends appear in the spatial distribution of TPH concentrations in the sediment samples.

Rinseate Samples 10A Through 10D - TPH was not detected in the rinseate samples greater than the method detection limits (1 mg/l) from the Shelby Tube (10A and 10B) and the Van Veen (10C and 10D) samplers. Rinseate samples for TPH were not collected from the Beta sampler.

POLYCHLORINATED BIPHENYLS

PCBs (polychlorinated biphenyls) were not detected above the detection limits in the background and the on-site sediments samples analyzed. A tentative identification was made for a PCB-like pattern in sample 1B (0.43 mg/kg), but a positive match could not be verified. PCBs were not detected in the Van Veen and Shelby Tube rinseate samples (10A through 10D) at concentrations greater than the detection limits (1.0 μg/l). Table 4 lists the method detection limits for PCB analyses in the sediment and rinseate water samples.

SUMMARY OF ANALYTICAL TESTING RESULTS--WATER SAMPLES

METALS

Beta Water Samples: Three water samples were collected with the horizontal Beta sampler within 1 foot of the bottom of Lake Union at sampling stations 8 (38.5-foot depth), and 2 (40.0 foot depth). Duplicate sample 12 (40.0-foot depth) was also collected at sampling station 2. Samples were submitted to the analytical laboratory for analysis of total metals, butyltin species, and hardness. Field water quality parameters (temperature, specific conductance, pH and dissolved oxygen) were measured in triplicate with field meters and the average of each set of field data was calculated. The analytical testing results and the field water quality results are summarized in Table 5.

Background Sample 8A - Zinc was detected in background water sample 8A at a concentration equal to the detection limit (0.01 mg/l [milligrams per liter]). Tributyltin was detected at a concentration of 9.2 ng/l (nanograms per liter) and hardness was measured at concentration of 42 mg/l as $CaCO_3$ (calcium carbonate). No other metals were detected in water sample 8A.

Sample 2A - Metals were not detected in water sample 2A at concentrations greater than the detection limits listed in Table 5. The concentrations of the butyltin species detected in sample 2A were 4 ng/l DBT (dibutyltin), 4 ng/l TRBT (tetrabutyltin), 5 ng/l TBT (tributyltin) and 6 ng/l MBT (monobutyltin). Hardness was measured at a concentration of 40 mg/l as $CaCO_3$.

Sample 12 (Duplicate 2A) - Lead and mercury were detected in water sample 12 at concentrations equal to the detection limits of 0.005 mg/l and 0.0005 mg/l, respectively. Zinc was detected at a concentration of 0.04 mg/l. The concentrations of the butyltin species detected in the water sample ranged from 21 ng/l MBT to 185 ng/l TBT. The higher metals concentration detected in sample 12, compared to 2A, may have been caused by disturbing the sediments at station 2 four days before collecting water sample 12. Hardness was measured at a concentration of 40 mg/l as $CaCO_3$.

Beta Rinseate Samples 10E and 10F - Two Beta rinseate samples were collected prior to initial sampling attempts (10E) and during the field investigation (10F) to confirm decontamination procedures. The rinseate samples were analyzed for the constituents previously mentioned, including total metals and butyltin species. The testing results are summarized in Table 5.

Zinc was the only metal detected as a low level contaminant (0.02 mg/l) in Beta rinseate sample 10E. This may explain the low concentration of zinc detected in sample 8A. Butyltin species were not detected in sample 10E above the method detection limit (1.0 ng/l).

Metals were not detected in Beta rinseate sample 10F at concentrations greater than the detection limits listed in Table 5. MBT and DBT were detected at low concentrations of 15 ng/l and 4 ng/l, respectively. Sample 10F was collected after duplicate sample 12 was collected; therefore, the butyltin concentrations detected in 10F may reflect minor residual contamination from sample 12. The butyltin species concentrations measured in water sample 12 were much greater than the rinseate sample 10F results.

INTERSTITIAL WATER SAMPLES

Two sediment samples (2C and 8C) were centrifuged to separate the interstitial water from the sediment, and the water samples were analyzed for total metals and butyltin species. The sediment sample from 2C was also analyzed for butyltin species. The results are presented in Appendix C. The test results for the two water samples are summarized in Table 5.

Samples 2C and 8C - Arsenic, barium and the butyltin species were detected in the interstitial water samples removed from sediment sample 2C and background sediment sample 8C. Other metals were not detected in the interstitial water samples. Arsenic was detected in water sample 2C at a concentration of 0.898 mg/l, which is 20 times greater than the concentration detected in background sample 8C (0.042 mg/l). Barium was detected in samples 2C and 8C at generally similar concentrations of 0.19 mg/l (2C) and 0.16 mg/l (8C). The butyltin concentrations detected in 2C (27 ng/l MBT, 40 ng/l DBT, 22 ng/l TBT) are significantly less than the concentrations detected in background sample 8C (82 ng/l MBT, 90 ng/l DBT, 310 ng/l TBT). TRBT was not detected in sample 2C, while a concentration of 160 ng/l was detected in sample 8C.

SHELBY TUBE AND VAN VEEN EQUIPMENT BLANKS

Two field rinseate samples were collected prior to the initial sampling attempts (10A and 10C) and two were collected during the field study (10B and 10D) to confirm decontamination procedures. The rinseate samples were analyzed for the constituents previously mentioned, including total metals. The testing results are summarized in Table 5.

Shelby Tube Rinseate Samples 10A and 10B - Metals were not detected at concentrations greater than the detection limits (Table 5) in rinseate sample 10A, collected at station 9 prior to the start of sampling. Copper and lead were detected at low concentrations of 0.10 mg/l and 0.009 mg/l, respectively, in rinseate sample 10B, which was collected at station 7 prior to the start of sampling. These low level residual metal contaminants would not have noticeably affected the final sediment sample results.

Van Veen Rinseate Samples 10C and 10D - Barium, copper, lead and zinc were detected at low concentrations in Van Veen rinseate sample 10C, which was collected at station 9 prior to the start of sampling. Barium, copper and zinc were detected at concentrations slightly above the detection limits for each metal. The lead concentration detected in rinseate sample 10C (0.45 mg/l) suggests minor contamination resulting from spraying the rinseate water over the lead weights attached to the Van Veen. Lead contamination would not likely affect the samples because the weights do not come in direct contact with the sediment during collection. Lead was not detected in rinseate sample 10D at concentration greater than the detection limits. Sample 10D was collected from the Van Veen sampler at station 7 prior to the start of sampling.

TCLP METAL RESULTS

Eleven sediment samples were tested by TCLP (toxicity characteristic leaching procedure) for metals to assist in evaluating the potential for upland disposal of these materials. This procedure is intended to mimic the potential generation of metals in leachate following disposal in a solid waste landfill. The TCLP testing results are summarized in Table 6.

GENERAL TCLP RESULTS

Selenium and silver were not detected in any of the TCLP extracts at concentrations greater than the detection limits. Mercury was detected only in sample 4A at a concentration equal to the detection limit (0.0005 mg/l). These results are representative of the relatively low concentrations of these metals in the sediment samples.

Arsenic: Arsenic was detected only in extracts 1A, 2A and 3A at concentrations between 0.05 mg/l to 0.17 mg/l. These arsenic concentrations are significantly less than the dangerous waste characterization criteria (5 mg/l) for TCLP extracts.

Barium: Barium was detected in the sample extracts (excluding background extracts) at concentrations between 0.060 mg/l and 0.33 mg/l. These barium concentrations are significantly less than the dangerous waste characterization criteria (100 mg/l) for TCLP extracts.

Cadmium: Cadmium was detected in the sample extracts (excluding background extracts) at concentrations between 0.011 mg/l to 0.050 mg/l. These cadmium concentrations are significantly less than the dangerous waste characterization criteria (1 mg/l) for TCLP extracts.

Chromium: Chromium was detected in four of the sample extracts (excluding background extracts) at concentrations between 0.005 mg/l to 0.012 mg/l. These chromium concentrations are significantly less than the dangerous waste characterization criteria (5 mg/l) for TCLP extracts.

Copper: Copper was detected in the sample extracts (excluding background extracts) at concentrations between 0.051 mg/l to 3.7 mg/l. There are no dangerous waste criteria for copper.

Lead: Lead was detected in the sample extracts (excluding background extracts) at concentrations between 0.28 mg/l to 3.0 mg/l. These lead concentrations are less than the dangerous waste characterization criteria (5 mg/l) for TCLP extracts.

Nickel: Nickel was detected in the sample extracts (excluding background extracts) at concentrations between 0.10 mg/l to 0.41 mg/l. There are no dangerous waste criteria for nickel.

Zinc: Zinc was detected in the sample extracts (excluding background results) at concentrations between 8.2 mg/l to 83 mg/l. There are no dangerous waste criteria for zinc.

REGULATORY SIGNIFICANCE OF WATER SAMPLES

The water quality data summarized in Table 5 were evaluated for environmental toxicity as regulated by the Water Quality Standards For Surface Waters of the State of Washington, WAC 173-201-010 through 173-201-120, 1988, and the U.S. EPA Quality Criteria For Water, 1986. Table 7 summarizes the fresh water quality regulatory standards for metals at 40 mg/l hardness; these standards are applicable to Beta water samples collected in Lake Union. Barium was not listed on the table because it is not regulated by the EPA and is not considered toxic in fresh water.

The metals concentrations detected in water samples 2A, 12 and 8A did not exceed the acute and chronic fresh water criteria in surface waters for arsenic, barium, cadmium, chromium, mercury, nickel, selenium and zinc. The lead concentration detected in water sample 12 (0.005 mg/l) did not exceed the acute fresh water criteria (0.025 mg/l), although it exceeded the chronic fresh water criteria (0.001 mg/l).

The detection limits specified in the final QA/QC plan for copper and silver (0.002 mg/l) were not performed by the laboratory because of a communications error in reporting the required detection limits to the laboratory. It was not possible to reanalyze the water samples because the samples were disposed of before this error was discovered.

The detection limit for silver (0.02 mg/l) is not less than the acute water quality standard (0.0008 mg/l). The silver concentrations detected in the sediments (less than 2.0 mg/kg to 4.2 mg/kg) suggest very low silver concentrations in the water samples.

The detection limit for copper (0.02 mg/l) is not less than the acute fresh water quality standard (0.0075 mg/l). We cannot estimate the water quality data for copper below the detection limits, although the survival rate of the bioassay analyses suggests very low copper concentrations.

The EPA developed provisional water quality criteria for TBT in 1987 at concentrations of 531 ng/l acute and 47 ng/l chronic to protect aquatic life; however, the EPA set an advisory criterion at 10 ng/l chronic. The concentrations of TBT detected in the three water samples (5 ng/l [2A] to 185 ng/l [12]) did not exceed the acute criteria; however, the results from sample 12 exceeded the chronic criteria.

A comparison of the results for Beta water samples (2A, 12, 8A) and interstitial water samples (2C and 8C) shows significantly greater arsenic and barium concentrations in the interstitial water samples and also a copper concentration (0.03 mg/l) greater than the detection limit in 8C. The copper concentration detected exceeds the acute criteria and the TBT concentration exceeds the chronic criteria in interstitial background water sample 8C. The arsenic concentration detected in 2C exceeds the acute criteria and the TBT concentration detected exceeds the advisory chronic criteria. The comparison also shows the TBT concentration is significantly greater in the background interstitial water sample than in the on-site interstitial water sample.

REGULATORY SIGNIFICANCE OF THE SEDIMENT SAMPLES

The sediment quality data presented in Tables 1 through 4 were evaluated for environmental toxicity associated with various remedial alternatives, including the no-action alternative, dredging the contaminated sediment, and capping the contaminated sediment with clean material. The sediment quality results were not evaluated in this report for environmental toxicity under the Final Sediment Management Standards, April 1991, Chapter 173-204 WAC developed by Ecology (Washington State Department of Ecology). These recently adopted sediment standards were developed for <u>marine</u> sediments, although Ecology has reserved the option to apply these regulations to fresh water sediments on a case-by-case basis. It would be not be appropriate to evaluate the <u>fresh water</u> sediments in Lake Union by these regulations for this phase of the investigation. The environmental toxicity of the sediments was evaluated for dredging and disposal as specified in the PSDDA (Puget Sound Dredge Disposal Analysis) Reports

Management Plan (June 1988) and the Evaluation Procedures (September 1989) developed by the U.S. Army Corps of Engineers, Washington State Department of Natural Resources, the U.S. EPA Region X and Ecology.

NO-ACTION ALTERNATIVE

A no-action alternative would allow the contaminated sediment to remain in place and would limit the disturbance of in-place sediments in Lake Union. An overall evaluation of the sediment quality shows that the types and concentrations of chemical contaminants present in the on-site bottom sediments and in background Lake Union samples have had possible adverse effects on biological activity in the Lake Union bottom sediments. Based on the bioassay results, there appears to be a significant difference between the mortality rate of the benthic organisms in the Yard 1 sediments compared to the Lake Union and Lake Washington background samples. There appears to be a relatively direct correlation between the overall concentrations of contaminants present in the Yard 1 sediments and the mortality rates of the benthic organisms, although the water quality data at the bottom of Lake Union did not show any short term acute toxic effects to the bioassay organisms as demonstrated in the water bioassay tests.

The exact nature of the chemical or chemicals that have the greatest adverse effects on the benthic organisms have not been determined as part of this study.

Environmental Significance of No-Action Alternative: The no-action alternative was evaluated to demonstrate the environmental significance associated with either performing no action or by performing alternative remedial options (dredging or capping). The acceptance of the no-action alternative as a remedial option may produce the following negative impacts and positive consequences to the Lake Union and the environment.

The negative impacts to Lake Union resulting from accepting a no-action alternative program generally would be caused by the physical presence of the contaminated bottom sediments. The negative impacts include the following: (1) the contaminated bottom sediments were demonstrated to be toxic to benthic organisms; (2) the chemical constituents in the sediments potentially may be bioaccumulated and possibly biomagnified (increasingly greater concentrations of the chemicals) up through the food chain to ultimate human consumption; and (3) the potential exists for the slow release of the constituents into the Lake Union water as dissolved compounds at concentrations near or below practical analytical quantification limits.

The positive consequences to accepting the no-action alternative include the following: 1) the potential environmental consequences associated with disturbing the sediments during dredging or during capping operations would not occur; 2) the environmental consequences associated with physically removing (treating contaminated water), containing and transporting contaminated sediment and processing the contaminated sediment for disposal would not occur; 3) the sediments appear to pose negligible environmental risk to the lake water quality because no short-term acute toxic effects were demonstrated in the water bioassay; and 4) the depth of water for operations and navigation purposes would be maintained.

As demonstrated by the water sampling at station 12 (where the water column was slightly affected [elevated butyltin species concentrations] by sediment disturbances 4 days prior to collecting the water sample), disturbances of the sediment may introduce a temporary pulse of suspended or dissolved chemical constituents into the lake bottom water. Dredging or capping activities may potentially disturb the sediments even if appropriate measures are taken to limit dispersion of the sediments (using a silt curtain) during remedial activities. A rapid pulse of suspended and dissolved chemical constituents may introduce a potentially lethal dose of chemical constituents into Lake Union, which may cause a short-term to possibly longer-term degradation of the water quality during remedial actions. A release of a contaminant plume may affect the Lake Union ecosystem to an unknown extent for an unspecified (suspected short-term) period of time. Capping the bottom sediment would reduce the available free board space for continuing operations at the Yard 1 facility. Natural sedimentation, in comparison, is expected to slowly cover the contaminated sediment in time, which would accomplish the same goal as capping and may be worth the environmental risk in the short term. There is no guarantee that a sediment cap would not be damaged, removed or recontaminated through contact with the contaminated material over time, thus negating the remedial alternative.

DREDGE DISPOSAL ANALYSIS

The sediment quality results (chemical and biological) were evaluated according to the guidelines specified in the PSDDA dredge material evaluation procedures. The chemical data were directly compared to the screening levels and the maximum levels presented in PSDDA Table A-7 "Screening Level (SL) and Maximum Level (ML) Guideline Chemistry Values," updated February 20, 1990 (Appendix E).

According to PSDDA, open-water disposal of dredge spoils with contaminant concentrations greater than the SLs and MLs are acceptable only in the presence of favorable bioassay results. The SLs and MLs are used as guidance for determining when bioassay testing is required.

A general ranking system was developed by PSDDA for the various navigated waterways in Puget Sound as a guide for planning proposed dredge activities. Lake Union was given a high ranking for the potential presence of chemicals of concern in the PSDDA guidelines. Selection of the required number of chemical and biological samples for PSDDA evaluation was based on the ranking system, historical data and estimated dredge quantities.

Unconfined Open-Water Disposal: Unconfined open-water disposal involves placing dredge spoils in an unconfined setting where the spoils are not isolated or separated from the environment. Unconfined disposal provides numerous potential pathways for chemicals entrained in the sediments to be released into the environment during the disposal activities.

The primary goal of the dredge disposal analysis is to determine if dredge spoils are suitable for unconfined open-water disposal. The suitability of dredge disposal is based on demonstrated ecological or human health effects, as developed in the PSDDA guidelines. Dredge spoils with contaminant concentrations greater than maximum level standards are potentially unacceptable for unconfined disposal. A brief comparison of the chemical results, presented below, demonstrates that unconfined open-water disposal may be unacceptable according to PSDDA guidelines. The results are evaluated for the Yard 1 facility, including sampling stations 1 through 7, shallow and deep intervals.

Metals - Arsenic concentrations detected generally exceeded the 57 mg/kg SL (screening level) in the sediment samples on-site and exceeded the 700 mg/kg ML (maximum level) at sampling stations 1, 2, and 4.

Cadmium concentrations detected exceeded the 0.96 mg/kg SL in sediment samples from all sampling stations and exceeded the 9.6 mg/kg ML in samples from stations 2, 4, 5, 6 and 7.

Copper concentrations detected exceeded the 81 mg/kg SL in sediment samples from all sampling stations and exceeded the 810 mg/kg ML in sampling stations 1, 2, 4 and 6.

Lead concentrations detected exceeded the 66 mg/kg SL in sediment samples from all sampling stations and exceeded the 660 mg/kg ML in sampling stations 1, 2 and 4.

Mercury concentrations detected exceeded the 0.21 mg/kg SL in the sediment samples from stations 1, 2, 4, 5 and 6. The ML for mercury was not exceeded in any of the samples.

The SL for nickel was not exceeded in any of the samples collected.

The silver concentrations detected exceeded the 1.2 mg/kg SL in sediment samples from stations 1, 2, 3 and 4. The ML for silver was not exceeded in any of the samples.

Zinc concentrations detected exceeded the 160 mg/kg SL in sediment samples from all sampling stations and equaled or exceeded the 1,600 mg/kg ML in sediment samples from stations 1, 2, 4, 5 and 6.

The tributyltin concentration detected in sediment sample 2C (0.19 mg/kg) exceeded the (0.03 mg/kg) SL. TBT was analyzed in only one sediment sample.

PAHs - The calculated total LPAH concentrations detected exceeded the 0.6 mg/kg SL and the 6.1 mg/kg ML in all the sediment samples. Generally, the greatest LPAH concentrations were observed at stations 4, 6 and 7. The lowest LPAH concentrations were observed at stations 1, 2, 3 and 5.

The calculated total HPAH concentrations detected exceeded the 1.8 mg/kg SL in all the sediment samples and the 51 mg/kg ML in the sediment samples from all sampling stations except station 3A. Generally, the greatest HPAH concentrations were observed at sampling stations 4 through 7 and background station 8. The lowest LPAH concentrations were observed at sampling stations 1 through 3.

Miscellaneous Compounds - The concentrations detected for dibenzofuran and bis(2-ethylhexyl)phthalate generally exceeded the SL and ML for those samples where these compound were detected at concentrations greater than the method detection limits.

PCBs - PCBs were not detected in the sediments at concentrations greater than the detection limits and generally not exceeding the SL and ML criteria, although the detection limits at sampling station 6 were greater than the regulatory guidelines because of matrix interferences.

Confined Disposal: Confined aquatic disposal is the placement of contaminated dredge spoils in a suitable (and approved) deep water disposal site after which the disposal material is capped with clean materials. The primary goal of capping is to isolate the contaminated spoils from the environment, thereby effectively eliminating the potential for subsequent adverse environmental effects. No approved confined disposal sites presently exist.

Confined aquatic disposal is an option that would require further, detailed evaluation to determine the suitability of this option. This option may be more costly than alternative disposal methods.

Upland Disposal: Upland disposal involves placing contaminated dredge spoils within an environmentally acceptable disposal site. Upland disposal options include nearshore disposal within a diked structure, disposal in a permitted solid waste landfill and disposal in a hazardous waste landfill.

Nearshore disposal involves placing the dredge material within an engineered sediment retention structure that allows the interstitial water to drain from the sediments before the contaminated material is capped with a clean soil cover. The contaminated materials placed in the disposal site would potentially release contaminated interstitial water into the environment at levels that may exceed regulatory guidelines. Collection and treatment of the interstitial water would be required to minimize environmental impacts and comply with regulatory standards. Ongoing environmental monitoring may also be required at the disposal location to evaluate the environmental consequences of nearshore disposal.

Upland disposal in a solid waste landfill would require transporting the contaminated dredge material to a permitted solid waste disposal facility. No processing would be done on the material except for reducing the moisture content to acceptable standards prior to disposal. Landfill disposal may be possible when the material is not characterized as a dangerous waste and when the landfill is willing to accept the waste.

The TCLP test results for metals show that the contaminated sediment (as dredge spoils) would not be classified as a dangerous waste. However, the HPAH data for the Yard 1 sediments result in PAH concentrations that are close to the dangerous waste designation levels.

The main environmental risk associated with landfill disposal is the possibility of future environmental liability. These materials may be determined to be toxic by future regulatory standards and may require responsible parties to assist in potential remedial activities at the landfill. To reduce the risk of this future liability, the material could be transported to a permitted hazardous waste landfill at a substantially greater cost.

Environmental Significance of Dredging: The environmental significance of dredging the contaminated sediments from the Yard 1 facility was evaluated to provide a basis for demonstrating the negative and positive effects to the water quality and ecosystem of Lake Union.

The negative effects or impacts of dredging are as follows: (1) dredging activities would cause disturbances of the contaminated sediments, which may potentially affect the lake water quality to an unknown extent; (2) dredging would require that the large quantities of contaminated water be collected, contained and treated prior to disposal; (3) the dredge sediments may require chemical treatment or stabilization before confined disposal is permitted, (these processes may generate additional waste volumes and waste streams); (4) dredging would require that an environmentally suitable confined disposal site (subaqueous or upland location) be determine or designed (at substantially greater costs); (5) dewatering of the dredge sediments during disposal may produce chemical alterations (chemical oxidation or dissolution) of the dredge materials, which may result in further environmental risks at the disposal location; (6) confined disposal of the dredge material will not reduce the overall toxicity of the material; (7) dredging would require temporary storage, loading, transporting and unloading of the contaminated sediments, which may result in additional risks to the environment; (8) long-term environmental monitoring may be required at the disposal site to define potential environmental impacts associated with the confined disposal alternative.

The positive effects of dredging include the following: (1) removal of the contaminated sediments will reduce the potential for adversely affecting the water quality of Lake Union and should promote the natural reestablishment of the bottom-dwelling aquatic organisms to Lake Union; (2) dredging will reduce the need for further studies once the ecosystem has returned to normal; (3) dredging will increase the navigable depths of water at the Yard 1 facility.

IN-PLACE CAPPING

In-place capping is a process by which relatively clean sediment is placed over the contaminated sediment, effectively isolating the contaminated material from the environment. The clean sediment cap also provides a suitable environment for benthic organisms to reestablish viable populations. In-place capping is considered a remedial technology, although the toxicity of the material is not reduced by this process.

Capping as an option at the Yard 1 Dry Dock facility may involve placing approximately 4 to 10 feet or more of a suitable capping material over the contaminated sediment. A 10-foot thick cap would reduce the average water depth at the Yard 1 facility to 30 feet. Figure 19 shows depth of water contours compiled from information supplied by UNIMAR and field measurements of water depth measured in January 1991 by GeoEngineers. A specific design of the cap would require a detailed evaluation of construction procedures, capping materials and probable burrowing depths of benthic organisms.

Environmental Significance of In-Place Capping: In-place capping of the contaminated sediments at the Yard 1 facility was evaluated for environmental significance to demonstrate the negative and positive effects on the water quality and ecosystem of Lake Union. In-place capping may be an environmentally acceptable option if the actual process does not adversely affect the environment and if the cap provides a permanent containment system (will not be degraded, damaged, removed or eroded over time by natural processes or human activity) for the contaminated material.

The negative environmental impacts of capping include releasing interstitial pore water into the surrounding lake water during consolidation of the sediments and dispersion of the fine-grained contaminated sediments into surrounding areas during placement of the cap material. Capping will reduce the navigable water depth and may impede operations and activities at the Yard 1 facility. Capping may not provide a completely permanent remedial option because ongoing natural processes (such as burrowing organisms) or human activities may degrade or breach the cap material. Capping will require ongoing environmental monitoring (additional cost expenditures) to evaluate the effectiveness of the cap material and to document reestablishment of the benthic community in the lake bottom ecosystem.

The positive aspect of capping is the benefit of isolating the contaminated sediments from the lake ecosystem without the potential adverse environmental risks and impacts that may result from dredge removal. Capping may be performed relatively easily with reduced capital expenditures compared to dredging and disposal. Capping should effectively isolate the contaminated sediments from the lake water and should provide a suitable habitat for the reestablishment of the benthic organisms. Capping, if proven to be effective, may provide the advantages of isolating the contaminated sediments, providing a new habitat for benthic organisms and satisfying environmental risk concerns.

CONCLUSIONS

As presented in FishPro's report, the water quality at the bottom of Lake Union did not show any short-term acute toxic effects to bioassay organisms as demonstrated in the water bioassay tests with nearly 100 percent survival. The concentrations of chemical constituents present in sediments at the Yard 1 Dry Dock Facility (stations 1 through 7) had a significant impact on the bioassay organisms as demonstrated by the high mortality rates observed in the bioassay analyses. The sediments in the bottom of Lake Union (station 8), were demonstrated to be more toxic to bioassay organisms than the sediments from Lake Washington (station 9). The concentrations of chemical constituents in the Yard 1 Facility sediment generally exceed the PSDDA SL and ML guideline chemistry values and were demonstrated to be toxic to the bioassay organisms, which demonstrates that unconfined open water disposal may be unacceptable according to PSDDA guidelines. The data presented in the bioassay report may suggest that a no action alternative is unacceptable, but the environmental significance of no-action may be appropriate upon further considerations of the environmental impacts associated with remedial actions such as capping or dredging.

Generally, the concentrations of chemicals present in the sediments exceed the PSDDA guidelines for unconfined open-water disposal. Dredging of the contaminated sediments is a possible viable remedial option, provided that suitable dredging procedures are used and approved confined disposal site becomes available.

Confined disposal of dredged sediments would have to be evaluated in greater detail to determine the suitability of this option. This option may be more technically demanding than other disposal options.

Nearshore disposal of bottom sediments may be an environmentally acceptable option if a suitable disposal site can win the approval of appropriate regulatory agencies. Considerable design effort would be needed to develop an appropriate plan for confined nearshore disposal of dredged sediments.

Upland disposal appears to be a viable disposal option, provided that the sediments can be permitted for disposal in a solid waste landfill. Significant efforts would be needed to dewater the dredge spoils prior to transporting the material to a solid waste landfill.

Capping may be an appropriate alternative provided that the environmental effects associated with constructing the cap are minimized or reduced to acceptable levels.

We do not feel it is appropriate at this time to recommend one remedial option over another. A thorough evaluation of the one or two best remedial options may be completed when the legal issues regarding the nature and extent of the contamination at the Yard 1 facility are resolved.

LIMITATIONS

This report has been prepared for use by United Marine International, Inc. in an investigation of the bottom sediment and water at the Yard 1 Dry Dock Facility in Seattle, Washington. The report is not intended for use by others and the information contained herein may not be applicable to other sites.

Our services have been executed in accordance with generally accepted practices in this area at the time the report was prepared. No other conditions, expressed or implied, should be understood.

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If you have any questions concerning this report, please call.

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TABLE 1
SUMMARY OF METALS ANALYSES IN SEDIMENTS (Page 1 of 2)

Sample Number	Sample Depth(ft)	EPA Method	7060	7080	7130/7031	7190	7210	7420/7421	7470/7471	7520	7740	7760	7950
		units	Arsenic	Barium	Cadmium	Chromium	Copper	Lead	Mercury	Nickel	Selenium	Silver	Zinc
1A	0.0-0.3	mg/kg	3,100	380	7.4	52	5,900	2,900	0.84	46	<3	4.2	10,000
1B	0.3-1.0	mg/kg	--	--	2.9	--	2,300	1,300	--	--	--	--	2,600
1C	1.0-1.5	mg/kg	--	--	<1	--	69	78	--	--	--	--	160
1D	1.5-2.0	mg/kg	--	--	1.3	--	34	36	--	--	--	--	94
1E	2.0-2.5	mg/kg	--	--	4.4	--	2,600	1,600	--	--	--	--	4,700
1F	2.5-3.0	mg/kg	--	--	2.1	--	1,400	1,500	--	--	--	--	2,100
1G	3.0-3.5	mg/kg	--	--	<1	--	38	45	--	--	--	--	150
1H	3.5-4.0	mg/kg	--	--	<1	--	96	31	--	--	--	--	94
2B	0.0-0.3	mg/kg	3,000	380	12	110	3,800	2,100	1.43	42	<3	<2	13,000
2C	0.3-1.5	mg/kg	2,000	196	4.4	230	2,300	1,600	1.50	130	<3	3.0	4,200
2D	1.5-2.0	mg/kg	--	--	7.5	--	3,400	2,600	--	--	--	--	6,200
2E	2.0-.25	mg/kg	--	--	8.2	--	4,600	2,700	--	--	--	--	5,700
2F	2.5-3.0	mg/kg	--	--	9.8	--	5,300	2,800	--	--	--	--	8,700
2G	3.0-3.5	mg/kg	--	--	3.9	--	2,200	2,300	--	--	--	--	6,500
2H	3.5-4.0	mg/kg	--	--	3.4	--	560	570	--	--	--	--	1,700
2I	4.0-4.5	mg/kg	--	--	1.7	--	46	95	--	--	--	--	130
2J	4.5-4.9	mg/kg	--	--	2.9	--	40	<25	--	--	--	--	190

TABLE 1
SUMMARY OF METALS ANALYSES IN SEDIMENTS (Page 2 of 2)

Sample Number	Sample Depth(ft)	EPA Method	7060	7080	7130/7031	7190	7210	7420/7421	7470/7471	7520	7740	7760	7950
		units	Arsenic	Barium	Cadmium	Chromium	Copper	Lead	Mercury	Nickel	Selenium	Silver	Zinc
3A	0.0-0.3	mg/kg	240	32	4.4	18	230	210	<0.80	13	<0.5	2	660
3B	0.3-5.0	mg/kg	64	45	3.7	18	90	97	<0.80	20	<0.5	<2	225
4A	0.0-0.3	mg/kg	1,800	210	27	89	1,500	1,700	0.80	72	<1	4.7	4,800
4B	0.3-4.3	mg/kg	180	120	10	59	240	290	0.91	59	<1	<2	620
5A	0.0-0.3	mg/kg	190	130	17	78	610	620	1.52	75	<2.0	<2	1,600
5B	0.0-5.0	mg/kg	26	110	8.2	45	89	130	1.05	55	<1	<2	210
6A	0.0-0.3	mg/kg	190	180	19	76	1,200	500	<0.80	69	<2.0	<2	1,700
11 (6A)	0.0-0.3	mg/kg	150	170	16	77	850	480	0.83	78	<2.0	<2	1,400
6B	0.3-5.0	mg/kg	67	110	10	57	252	230	<0.80	57	<2.0	<2	430
7A	0.0-0.3	mg/kg	190	130	13	59	540	470	<0.80	74	<2.0	<2	1,000
7B	0.3-5.0	mg/kg	27	90	8.0	44	130	170	<0.80	51	<2.0	<2	270
8B	0-0.3	mg/kg	71	92	2.4	47	170	250	1.53	48	<1	<2	340
8C	0.3-2.0	mg/kg	24	92	3.0	32	94	130	1.18	49	<1	<2	260
8D	0.3-0.5	mg/kg	--	--	3.6	--	62	61	--	--	--	--	160
8E	0.5-1.0	mg/kg	--	--	<2	--	56	53	--	--	--	--	150
8F	1.0-1.5	mg/kg	--	--	<4	--	120	160	--	--	--	--	294
8G	1.5-2.0	mg/kg	--	--	<3	--	150	180	--	--	--	--	301
8H	2.0-2.5	mg/kg	--	--	<3	--	180	350	--	--	--	--	430
8I	2.5-3.0	mg/kg	--	--	3	--	68	83	--	--	--	--	190
8J	3.0-3.5	mg/kg	--	--	2	--	39	45	--	--	--	--	93
8K	3.5-4.0	mg/kg	--	--	<5	--	13	<50	--	--	--	--	46
8L	4.0-4.5	mg/kg	--	--	<5	--	16	<45	--	--	--	--	63
8M	4.5-4.8	mg/kg	--	--	12	--	12	<40	--	--	--	--	39
9A	0-0.3	mg/kg	8.9	110	<2	39	38	68	<0.40	41	<1.0	<2	120
9B	0.3-2.5	mg/kg	2.3	51	<1	32	15	<10	<0.15	31	<0.5	<2	44

Notes:

11(6A) - 11 is a duplicate sample of 6A

mg/kg - milligrams per kilogram

"<" - less than

"--" - not tested

TABLE 2
SUMMARY OF SEMIVOLATILE ORGANIC COMPOUNDS ANALYSES IN SEDIMENT SAMPLES

Sample Number		1A	2B	3A	3B	4A	4B	5A	5B	6A	11 (6A)	6B	7A	7B	8B	8B	8C	9A
Sample Interval (feet)		0-0.3	0-0.3	0-0.3	0.3-5.0	0-0.3	0.3-4.3	0-0.3	0.3-5.0	0-0.3	0-0.3	0.3-5.0	0-0.3	0.3-5.0	0-0.3	0-0.3	0.3-2.0	0-0.3
EPA Method		8270	8270	8270	8310	8270	8310	8270	8310	8270	8270	8310	8270	8270	8310	8270	8310	8270
LPAHs	Units																	
Napthalene	mg/kg	1.3J	2.6	<3.5	2.4	2.6J	<3.8	<11	1.2	38	33	120	2.6J	4.2	<4.0	4.1	0.87	<0.68
2-methylnaphthalene	mg/kg	<1.8	6.9	<3.5	NA	12	NA	<11	NA	95	80	NA	2.4J	NA	NA	3.7J	NA	<0.68
Acenaphthylene	mg/kg	<1.8	<2.4	<3.5	<5.0	4.5	<7.7	<11	1.0	4.9J	<8.9	50	<4.6	16	<5.5	2.6J	0.50	<0.68
Acenaphthene	mg/kg	2.1	18	<3.5	<5.0	22	<7.7	8.8J	<0.68	40	33	150	8.3	68	<5.5	6.6	<0.57	<0.68
Fluorene	mg/kg	2.1	20	<3.5	1.0	20	4.1	6.5J	1.8	38	31	53	8.9	25	<0.55	5.9	0.40	<0.68
Phenanthrene	mg/kg	10	5.3	3.5J	7.1	56	15	15	5.6	100	88	160	27	97	9.0	16	1.7	<0.68
HPAHs																		
Anthracene	mg/kg	2.8	13	3.4J	2.4	20	5.0	9.6J	2.3	33	28	44	9.6	26	28	6.8	0.47	<0.68
Fluoranthene	mg/kg	11	25	5.3	18	34	32	52	19	31	40	47	24	60	29	43	7.7	<0.68
Pyrene	mg/kg	11	17	4.1	15	32	40	54	23	34	36	47	25	34	36	40	9.7	<0.68
Benzo(a)anthracene	mg/kg	4.4	6.0	2.0J	5.9	14	11	18	6.0	12	13	13	9.1	13	11	16	2.9	<0.68
Chrysene	mg/kg	5.5	6.8	2.1J	7.4	15	15	21	7.6	14	15	22	15	16	14	17	4.0	<0.68
Benzo(b)fluoranthene	mg/kg	6.5	6.6	2.7J	5.0	14	7.7	23	5.2	11	<8.9	7.4	11	14	11	23	2.6	<0.68
Benzo(k)fluoranthene	mg/kg	1.8	1.9J	<3.5	2.7	4.9	4.5	67	2.9	30	29	4.7	33	7.4	6.2	8.3	1.5	<0.68
Benzo(a)pyrene	mg/kg	5.4	4.8	2.6J	7.6	14	14	26	9.2	12	10	13	12	24	17	25	4.3	<0.68
Indeno(1,2,3-cd)pyrene	mg/kg	2.8	1.9J	<3.5	6.5	6.9	10	17	6.4	6.2J	4.8J	6.2	7.5	16	14	15	3.1	<0.68
Dibenz(a,h)anthracene	mg/kg	<1.8	<2.4	<3.5	1.8	2.0J	4.5	<11	0.39	<8.5	<8.9	6.5	<4.6	5.8	<1.6	2.9J	1.3	<0.68
Benzo(g,h,i)perylene	mg/kg	3.4	2.1	<3.5	6.5	8.2	13	20	8.0	6.5J	4.8J	6.5	8.8	12	18	21	4.0	<0.68
MISCELLANEOUS																		
Dibenzofuran	mg/kg	1.0J	14	<3.5	NA	6.3	NA	<11	NA	12	9.9	NA	<4.6	NA	NA	<4.0	NA	<0.68
Bis(2-ethylhexyl)phthalate	mg/kg	5.9	10	1.8J	NA	1.9J	NA	<11	NA	<8.5	<8.9	NA	<4.6	NA	NA	<4.0	NA	<0.68
Total LPAHs (1)	mg/kg	**20**	**68**	**16**	**20**	**137**	**35**	**56**	**13**	**349**	**298**	**384**	**61**	**240**	**39**	**46**	**5**	**2**
Total HPAHs (1)	mg/kg	**54**	**74**	**26**	**76**	**145**	**152**	**302**	**88**	**161**	**162**	**173**	**148**	**168**	**157**	**211**	**41**	**2**

Notes:

(1) Calculated totals include 0.5 times the detection for less than values and 0.5 times the lowest detection limit or the lowest detected concentration for the constituents not analyzed under the EPA Method 8310.
11 (6A) - 11 is a duplicate of 6A
LPAHs - Light polynuclear aromatic hydrocarbons
HPAHs - Heavy polynuclear aromatic hydrocarbons
mg/kg - milligrams per kilogram
"<" - less than method detection limit
NA = not applicable
J - estimated value

TABLE 3
SUMMARY OF TOTAL PETROLEUM HYDROCARBONS IN SEDIMENT SAMPLES

Sample Number		1A	1C (1)	3A	3B	4A	4B	5A	5B	6A	11 (6A)	6B	7A	7B	8B	8F	9A	9B
Sample Interval (feet)		0-0.3	1.0-1.3	0-0.3	0.3-5.0	0-0.3	0.3-4.3	0-0.3	0.3-5.0	0-0.3	0-0.3	0.3-5.0	0-0.3	0.3-5.0	0-0.3	1.0-1.5	0-0.3	0.3-2.5
EPA Method	Units	418.1	418.1	418.1	418.1	418.1	418.1	418.1	418.1	418.1	418.1	418.1	418.1	418.1	418.1	418.1	418.1	418.1
TPH	mg/kg	1600	950	99	230	590	420	780	39	200	110	130	160	190	120	350	65	13

Notes
(1) Laboratory results were reported for two sample 1C's listed on separate COCs; only the result presented above is a representative sample.
11 (6A) - 11 is a duplicate of 6A
mg/kg - milligrams per kilogram

TABLE 4
SUMMARY OF POLYCHLORINATED BIPHENYLS IN SEDIMENT AND RINSEATE SAMPLES

Sample Number		1A (1)	1C	2B	3A	4A	5A	6A	11 (6A)	7A	8B	8C	9A	9B
Sample Interval (feet)		0-0.3	1.0-1.5	0-0.3	0-0.3	0-0.3	0-0.3	0-0.3	0-0.3	0-0.3	0-0.3	0.3-2.0	0-0.3	0.3-2.5
EPA Method 8080	units	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
PCBs IN SEDIMENT														
PCB 1016		<0.033	<0.063	<0.033	<0.14	<0.14	<0.22	<4.1	<4.3	<2.2	<0.033	<0.033	<0.13	<0.058
PCB 1221		<0.033	<0.063	<0.033	<0.14	<0.14	<0.22	<4.1	<4.3	<2.2	<0.033	<0.033	<0.13	<0.058
PCB 1232		<0.033	<0.063	<0.033	<0.14	<0.14	<0.22	<4.1	<4.3	<2.2	<0.033	<0.033	<0.13	<0.058
PCB 1242		<0.033	<0.063	<0.033	<0.14	<0.14	<0.22	<4.1	<4.3	<2.2	<0.033	<0.033	<0.13	<0.058
PCB 1248		<0.033	<0.063	<0.033	<0.14	<0.14	<0.22	<4.1	<4.3	<2.2	<0.033	<0.033	<0.13	<0.058
PCB 1254		<0.033	<0.063	<0.033	<0.14	<0.14	<0.22	<4.1	<4.3	<2.2	<0.033	<0.033	<0.13	<0.058
PCB 1260		0.43	<0.063	<0.033	<0.14	<0.14	<0.22	<4.1	<4.3	<2.2	<0.033	<0.033	<0.13	<0.058

Sample Number		10B	10C	10D	10E
Sample Interval (feet)		Shelby Rinseates		Van Veen Rinseates	
EPA Method 8080	units	ug/l	ug/l	ug/l	ug/l
PCBs IN WATER					
PCB 1016		<1.0	<1.0	<1.0	<1.0
PCB 1221		<1.0	<1.0	<1.0	<1.0
PCB 1232		<1.0	<1.0	<1.0	<1.0
PCB 1242		<1.0	<1.0	<1.0	<1.0
PCB 1248		<1.0	<1.0	<1.0	<1.0
PCB 1254		<1.0	<1.0	<1.0	<1.0
PCB 1260		<1.0	<1.0	<1.0	<1.0

Notes

(1) A tenative identification for a PCB like pattern was made, but a positive match could not be verified.

11(6A) - sample 11 is a duplicate of 6A

"<" = less than not detected

mg/kg = milligrams per kilogram

ug/l = micrograms per liter

TABLE 5
SUMMARY OF WATER ANALYSES

Sample Number		2A	12 (2A)	8A	2C	8C	10A	10B	10C	10D	10E	10F
Sample Depth (feet)		40 (1)	40	40	0.3-1.5	0.3-2.0	Shelby	Shelby	Van Veen	Van Veen	Beta	Beta
Sample Type		Beta Samples			Interstitial Water		Rinseate Samples					
METALS	units											
Arsenic	mg/l	<0.005	<0.005	<0.005	0.898	0.042	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Barium	mg/l	<0.06	<0.02	<0.06	0.19	0.16	<0.06	<0.02	0.08	<0.02	<0.06	<0.02
Cadmium	mg/l	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003
Chromium	mg/l	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Copper	mg/l	<0.02	<0.02	<0.02	<0.02	0.03	<0.02	0.10	0.03	<0.02	<0.02	<0.02
Lead	mg/l	<0.005	0.005	<0.005	<0.005	<0.005	<0.005	0.009	0.045	<0.005	<0.005	<0.005
Mercury	mg/l	<0.0005	0.0005	<0.0005	<0.002	<0.002	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
Nickel	mg/l	<0.03	<0.1	<0.03	<0.03	<0.03	<0.03	<0.01	<0.03	<0.01	<0.03	<0.01
Selenium	mg/l	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Silver	mg/l	<0.02	<0.03	<0.02	<0.02	<0.02	<0.02	<0.03	<0.02	<0.03	<0.02	<0.03
Zinc	mg/l	<0.01	0.04	0.01	<0.01	<0.01	<0.01	<0.01	0.02	<0.01	0.02	<0.01
BUTYLTIN SPECIES												
Monobutyltin	ng/l	6	21	<1	27	82	--	--	--	--	<1	15
Dibutyltin	ng/l	4	33	<1	40	90	--	--	--	--	<1	4
Tributyltin	ng/l	5	185	9.2	22	310	--	--	--	--	<1	ND
Tetrabutyltin	ng/l	4	140	<1	<1	160	--	--	--	--	<1	ND
Hardness (2)	mg/l	40	40	42								
FIELD WATER QUALITY PARAMETERS												
D.O.	mg/l	11.4	11.7	12.0								
Conductivity	umhos/cm	132.6	122.9	150.9								
Temperature	degress F	42.7	45.8	42.7								
pH		7.20	7.8	6.20								

Notes:
(1) Corrected depth 40 feet - Sample depth listed as 39 feet on chain-of-custody
(2) hardness express as CaCO3
12 (2A) - 12 is a duplicate of 2A
mg/l = milligrams per liter
ng/l = nanograms per liter
umhos/cm = micromhos per centimeter
"--" = not analyzed
ND = not detected

TABLE 6
SUMMARY OF TCLP METALS RESULTS

Sample	Sample	EPA Method	6010	6010	6010	6010	6010	6010	7470	6010	6010	7760	7950
Number	Depth (ft)	units	Arsenic	Barium	Cadmium	Chromium	Copper	Lead	Mercury	Nickel	Selenium	Silver	Zinc
1A	0-0.3	mg/l	0.17	0.30	0.017	0.006	0.051	0.58	<0.0005	0.32	<0.05	<0.02	59
2B	0-0.3	mg/l	0.05	0.13	0.046	<0.005	2.8	0.52	<0.0005	0.30	<0.05	<0.02	83
3A	0-0.3	mg/l	0.08	0.19	0.011	<0.005	0.61	0.63	<0.0005	0.14	<0.05	<0.02	19
4A	0-0.3	mg/l	<0.05	0.060	0.042	<0.005	3.7	3.0	0.0005	0.38	<0.05	<0.02	48
5A	0-0.3	mg/l	<0.05	0.067	0.050	0.012	1.8	1.6	<0.0005	0.41	<0.05	<0.02	18
6A	0-0.3	mg/l	<0.05	0.33	0.013	0.005	0.064	0.28	<0.0005	0.10	<0.05	<0.02	8.2
11 (6A)	0-0.3	mg/l	<0.05	0.12	0.012	0.006	0.26	0.54	<0.0005	0.16	<0.05	<0.02	10
7A	0-0.3	mg/l	<0.05	0.075	0.028	0.008	1.6	1.4	<0.0005	0.41	<0.05	<0.02	15
8B	0-0.3	mg/l	<0.05	0.20	0.008	<0.005	0.051	0.23	<0.0005	0.06	<0.05	<0.02	2.0
8C	0.3-2.0	mg/l	<0.05	0.081	0.018	0.006	0.16	0.21	<0.0005	0.26	<0.05	<0.02	3.2
9A	0-0.3	mg/l	<0.05	0.31	0.004	0.006	0.019	<0.02	<0.0005	0.03	<0.05	<0.02	0.38

Note:
11 (6A) Sample 11 is a duplicate of sample 6A

TABLE 7
SUMMARY OF WATER QUALITY STANDARDS FOR SURFACE WATER[1]

	Acute[2] (mg/l)	Chronic[2] (mg/l)
Arsenic	0.36	0.19
Cadmium	0.0014	0.0006
Chromium	0.819	0.097
Copper	0.0075	0.0054
Lead	0.025	0.001
Mercury	0.0024	0.0012
Nickel	0.653	0.073
Selenium	0.26	0.035
Silver	0.008	--
Zinc	0.053	0.048

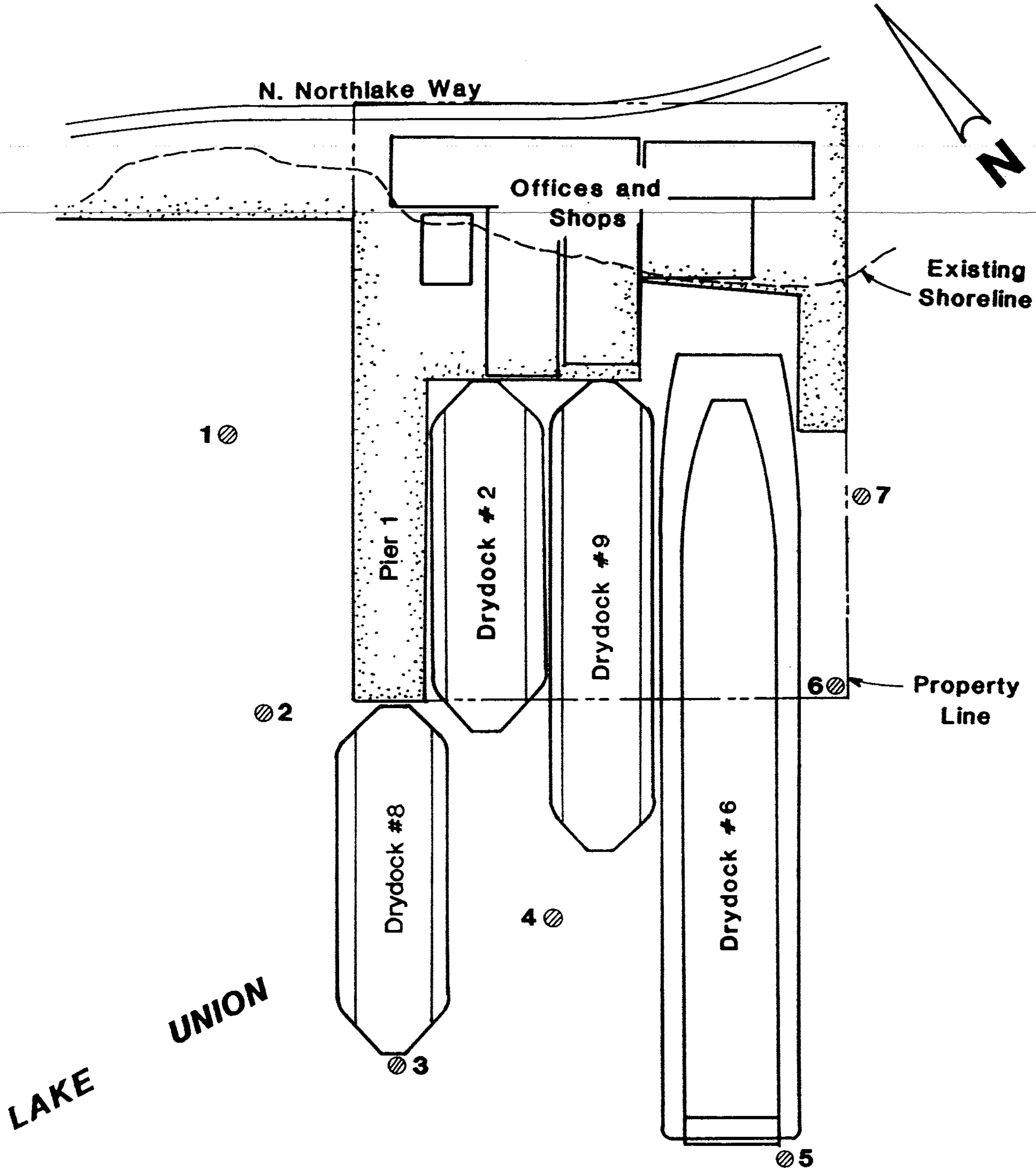
Notes:

[1] Water Quality Standards of the State of Washington WAC 173-201-010 through WAC 173-201-120 effective January 6, 1988.

[2] The acute and chronic criteria were calculated as defined in WAC 173-201-047 with an average hardness of 40 mg/l as C_aCO_3

mg/l = milligrams per liter

"--" = no level established



EXPLANATION:

1 ▨ SEDIMENT SAMPLING STATION

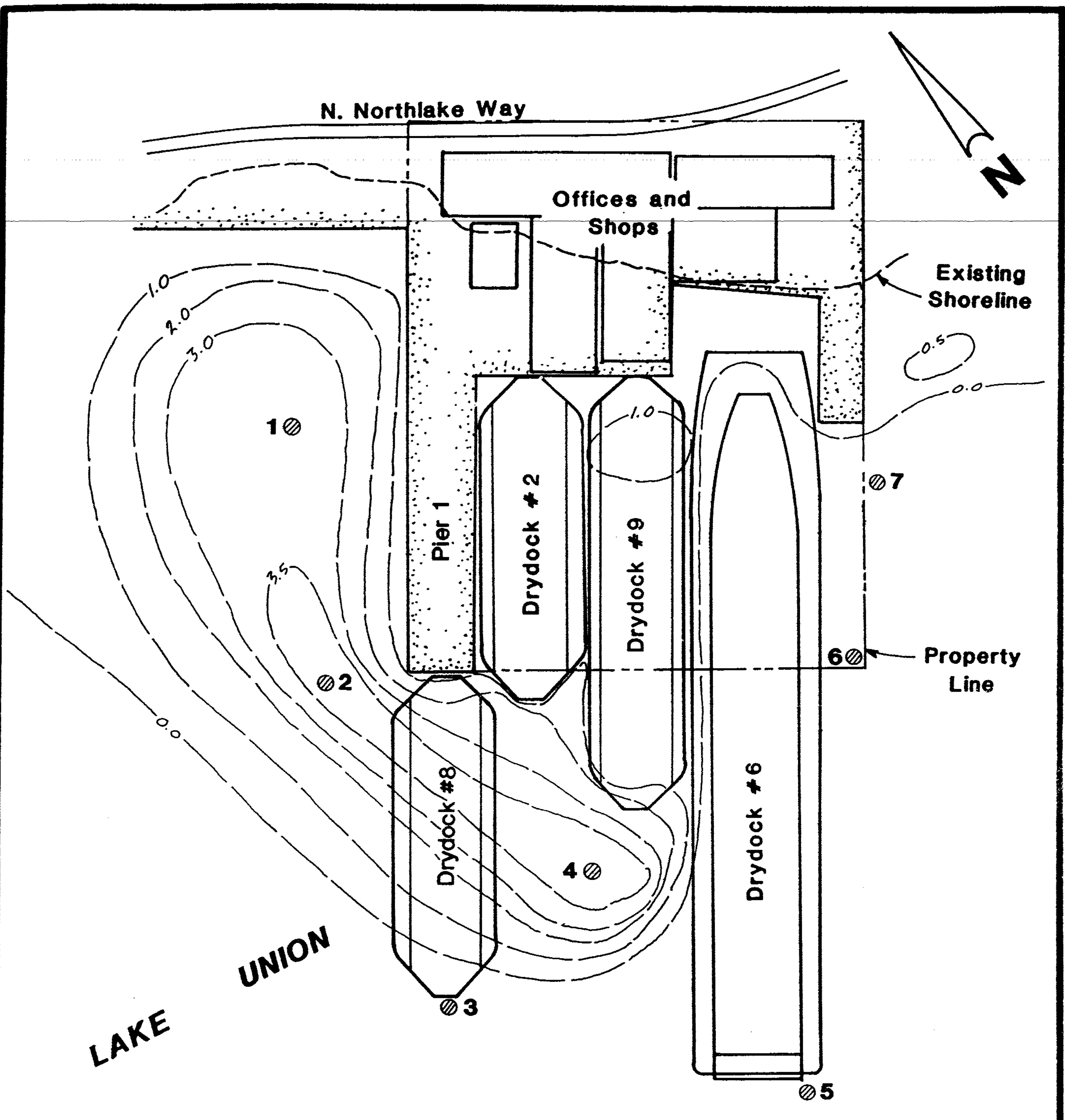
REFERENCE:
UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

GeoEngineers

SITE PLAN

FIGURE 1

6-26-91 LJD
7-23-91 RRR



EXPLANATION:

1▨ SEDIMENT SAMPLING STATION

—1.0—— SAND BLASTING MATERIAL THICKNESS CONTOUR (FEET)

0 100 200
SCALE IN FEET

REFERENCE:
UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

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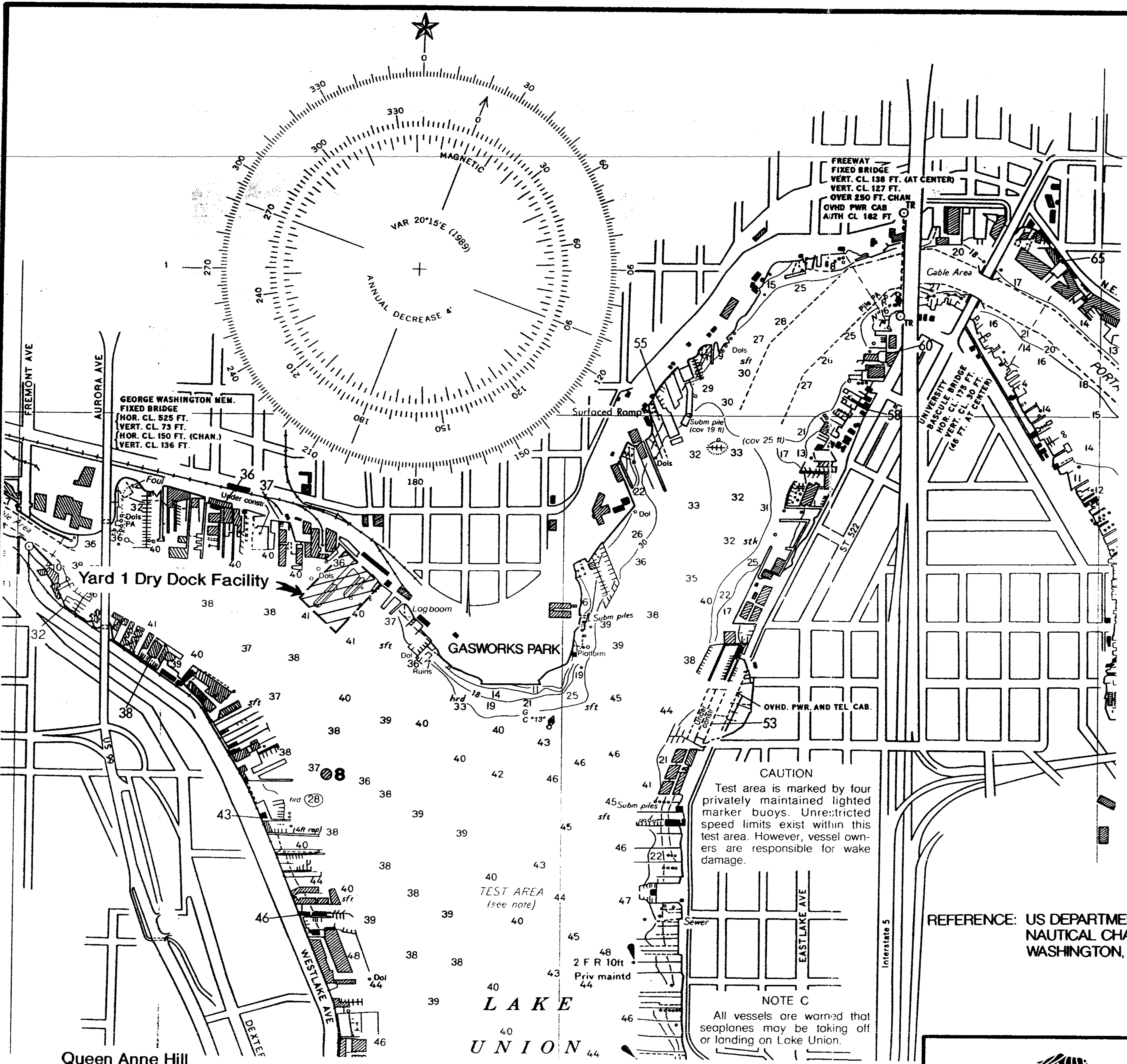
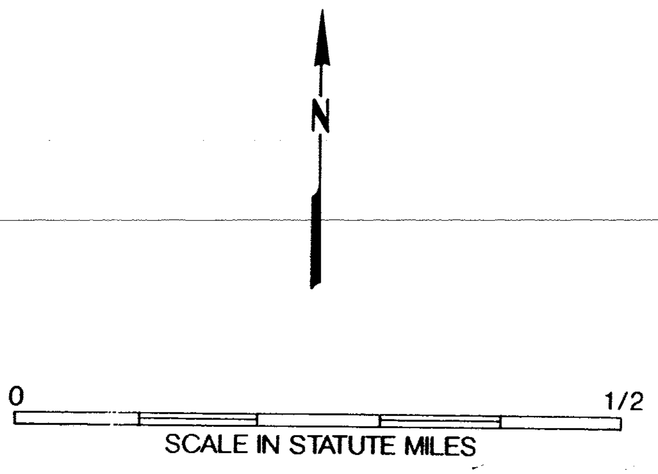
SAND BLASTING MATERIAL THICKNESS MAP

FIGURE 2

6-26-91 LJD
7-23-91 RRR





EXPLANATION:

⊘8	BACKGROUND SAMPLING STATION LOCATION AND NUMBER

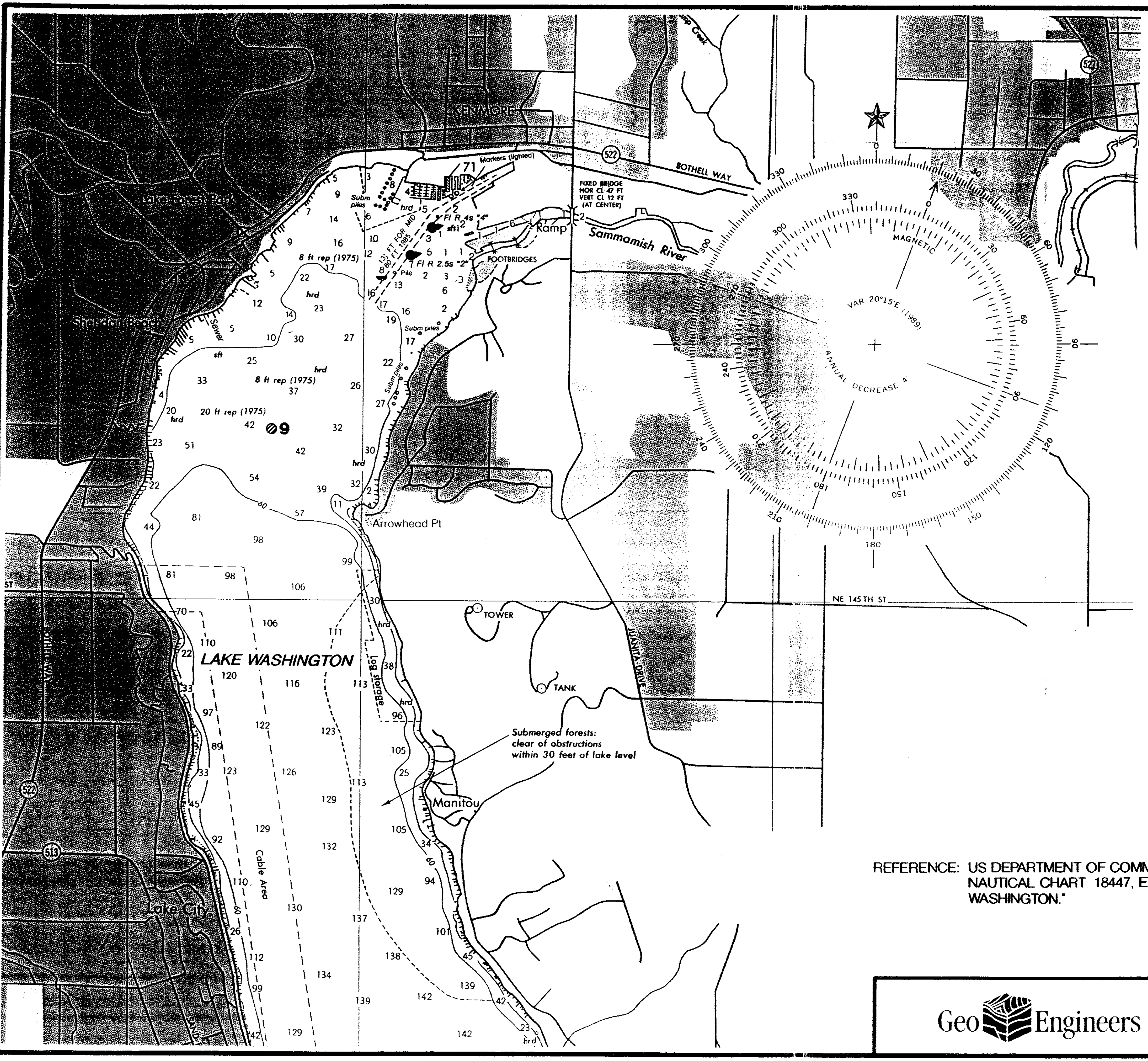
REFERENCE: US DEPARTMENT OF COMMERCE, NATIONAL OCEANIC AND ATMOSPHERIC ADMINISTRATION NAUTICAL CHART 18447, ENTITLED "LAKE WASHINGTON SHIP CANAL AND LAKE WASHINGTON, WASHINGTON."

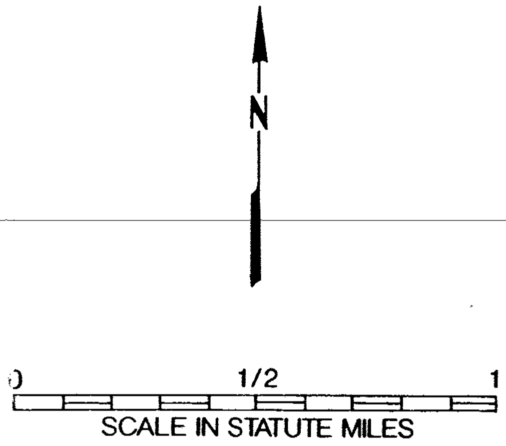
GeoEngineers

BACKGROUND SAMPLING STATION 8

FIGURE 3

1299-003-804 REV:LJD 7/19/91 (B)





SCALE IN STATUTE MILES

EXPLANATION:

⊘9	BACKGROUND SAMPLING STATION LOCATION AND NUMBER

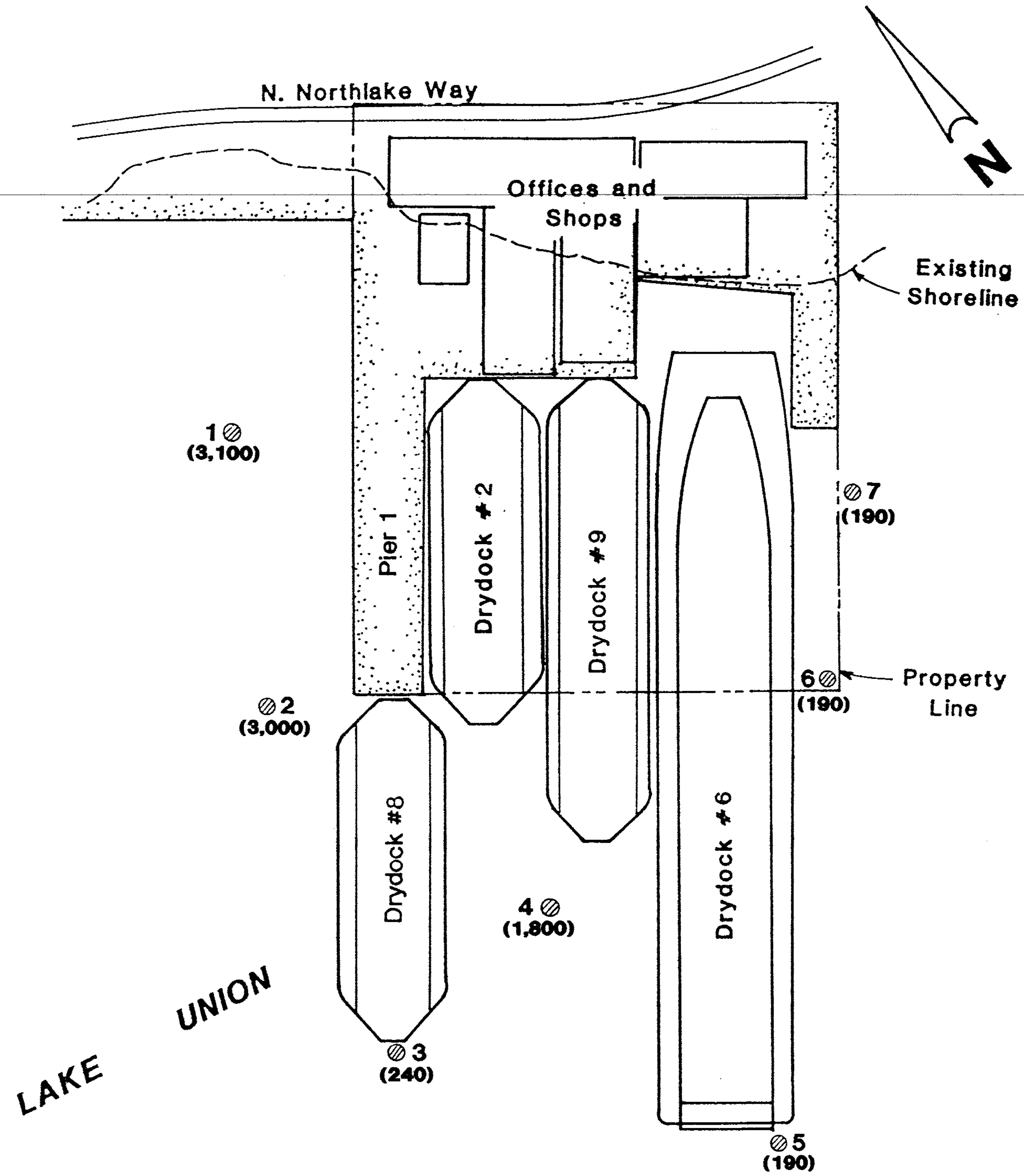
REFERENCE: US DEPARTMENT OF COMMERCE, NATIONAL OCEANIC AND ATMOSPHERIC ADMINISTRATION NAUTICAL CHART 18447, ENTITLED " LAKE WASHINGTON SHIP CANAL AND LAKE WASHINGTON, WASHINGTON."

GeoEngineers

BACKGROUND SAMPLING STATION 9

FIGURE 4

1299-003-804 REJ: JJD 7/19/91 (A)



EXPLANATION:

1 ▨ SEDIMENT SAMPLING STATION
(3,100) ARSENIC CONCENTRATION (MG/KG)

BACKGROUND STATION 8 = 71 MG/KG

0 100 200
SCALE IN FEET

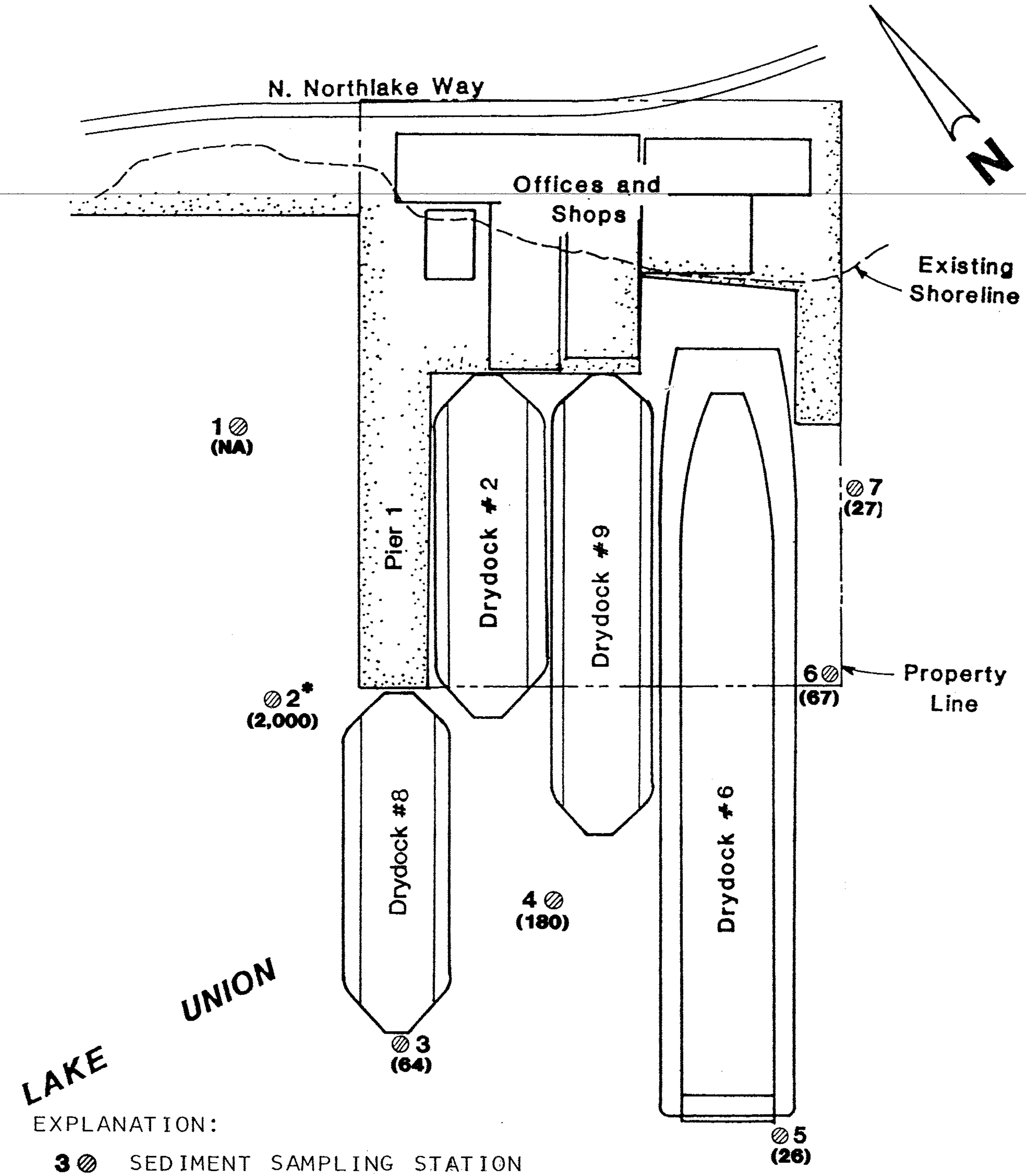
REFERENCE:
UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

GeoEngineers

ARSENIC, 0–0.3 FEET INTERVAL

FIGURE 5

6·26·91 LJD
7·23·91 RRR



EXPLANATION:

3 ▨ SEDIMENT SAMPLING STATION
(64) ARSENIC CONCENTRATION (MG/KG)

* SAMPLE INTERVAL 0.3-1.5 FEET

NA NOT ANALYZED

BACKGROUND STATION 8 = 24 MG/KG
(0.3-2.0 FEET)*

0 100 200
SCALE IN FEET

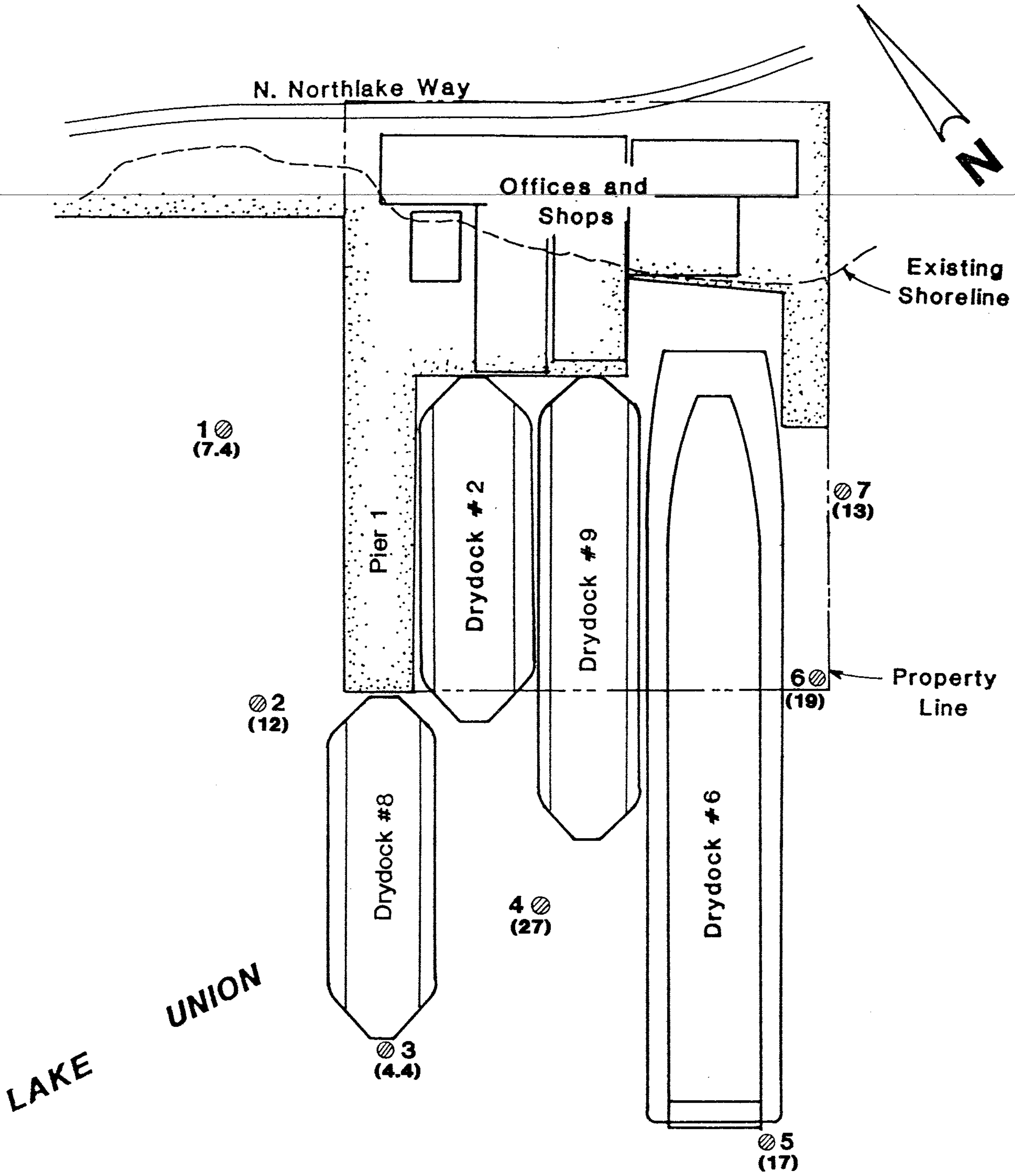
REFERENCE:
UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

GeoEngineers

ARSENIC, 0.3-5.0 FEET INTERVAL

FIGURE 6

6-26-91 LJD
7-23-91 RRR



EXPLANATION:

1▨ SEDIMENT SAMPLING STATION
(7.4) CADMIUM CONCENTRATION (MG/KG)
BACKGROUND STATION 8 - 2.4 MG/KG

REFERENCE:
UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

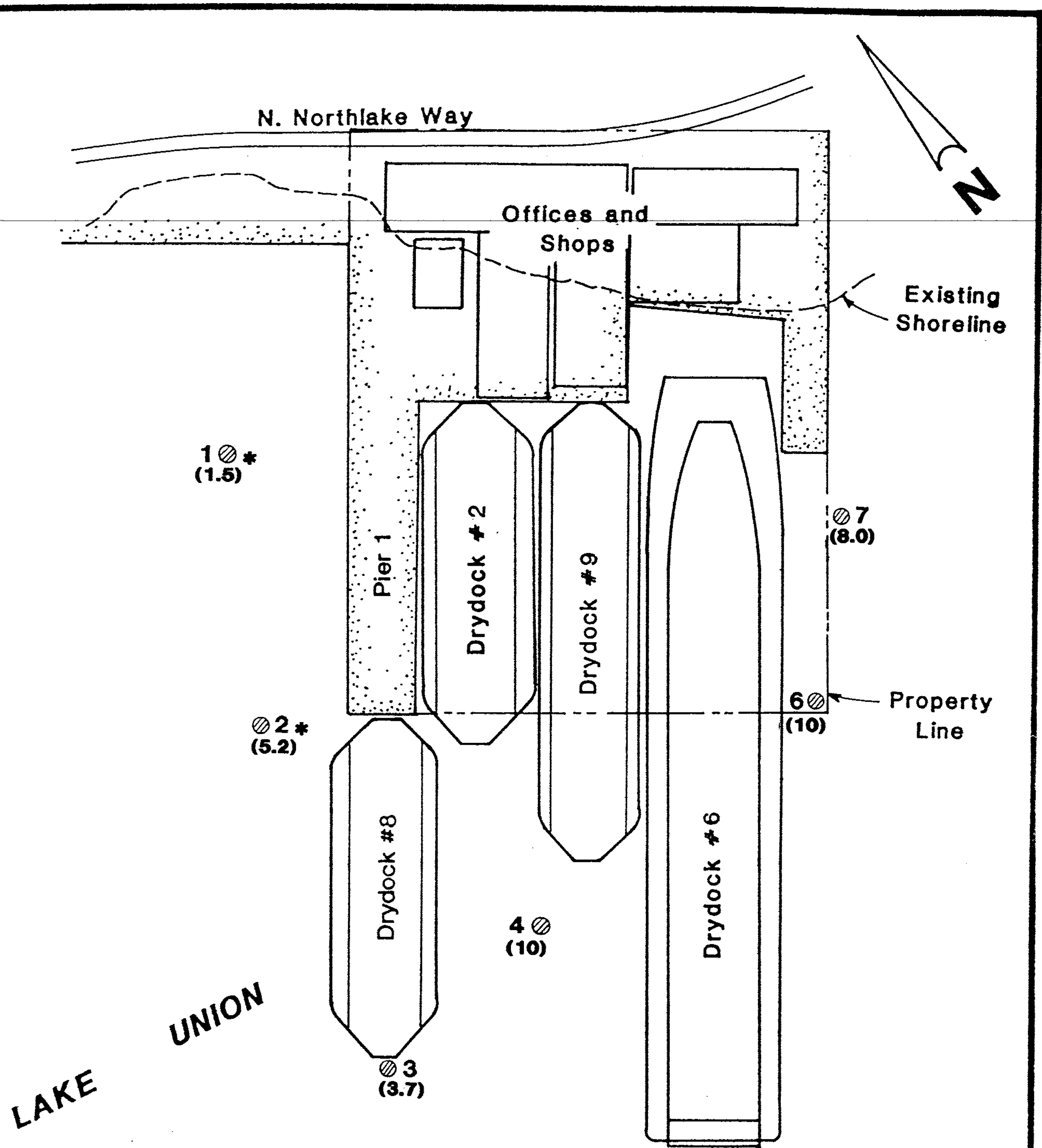
GeoEngineers

CADMIUM, 0–0.3 FEET INTERVAL

FIGURE 7

Re 6-8 KJ
6-26-91 LJD
7-23-91 RRR

[illegible]-03 [illegible]

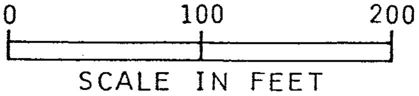


EXPLANATION:

1 ▨ SEDIMENT SAMPLING STATION
(1.5)* CADMIUM CONCENTRATION (MG/KG)

* AVERAGE CONCENTRATION FOR 0.3-5.0 FEET INTERVAL

BACKGROUND STATION 8 = APPROXIMATELY
3 MG/KG*



REFERENCE:
UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

GeoEngineers

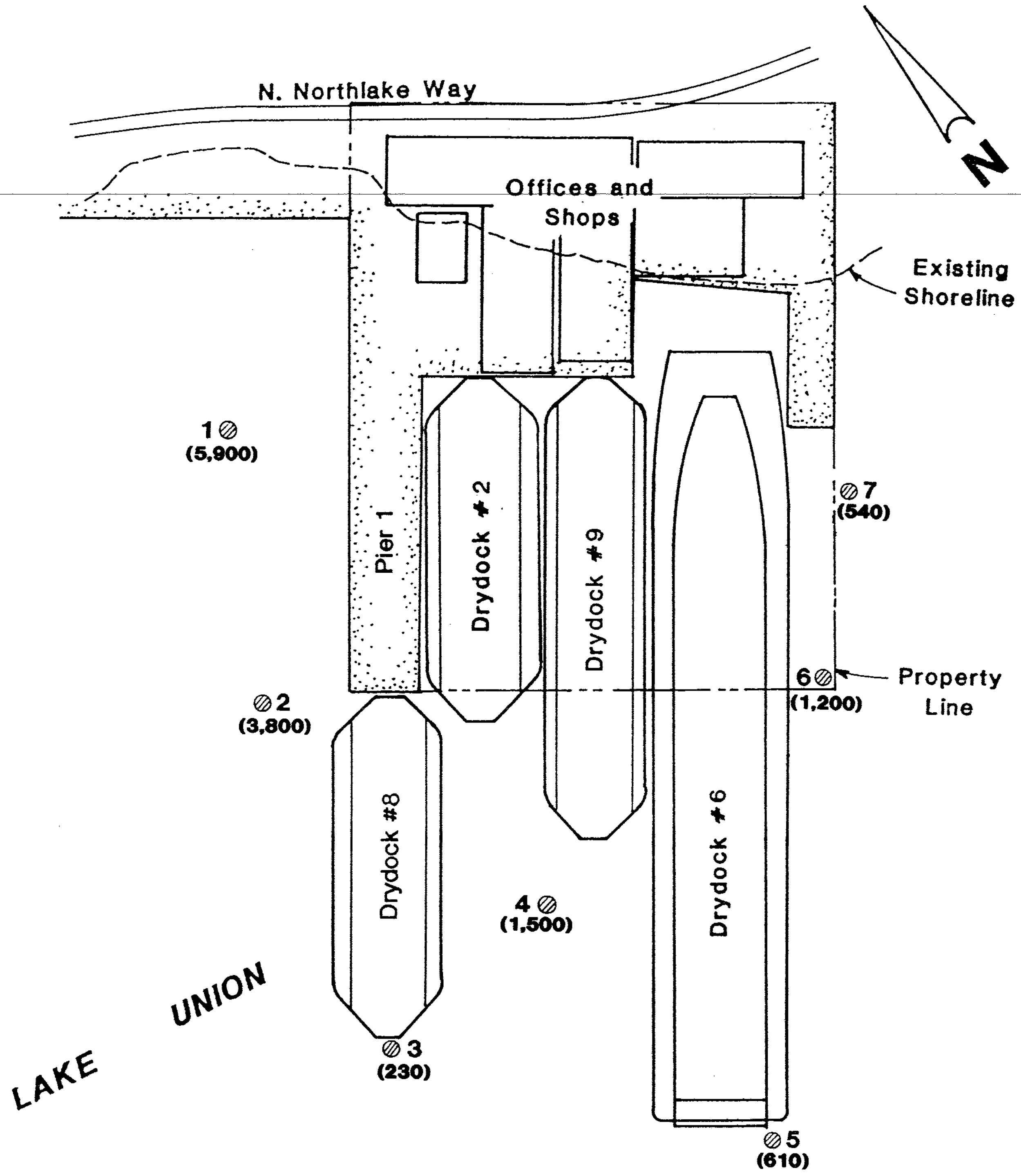
CADMIUM, 0.3-5.0 FEET INTERVAL

FIGURE 8

-03

Re 6·8·

6·26·91 LJD
7·23·91 RRR



EXPLANATION:

1 ▨ (5,900) SEDIMENT SAMPLING STATION
COPPER CONCENTRATIONS (MG/KG)

BACKGROUND STATION 8 = 170 MG/KG

0 100 200
SCALE IN FEET

REFERENCE:
UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

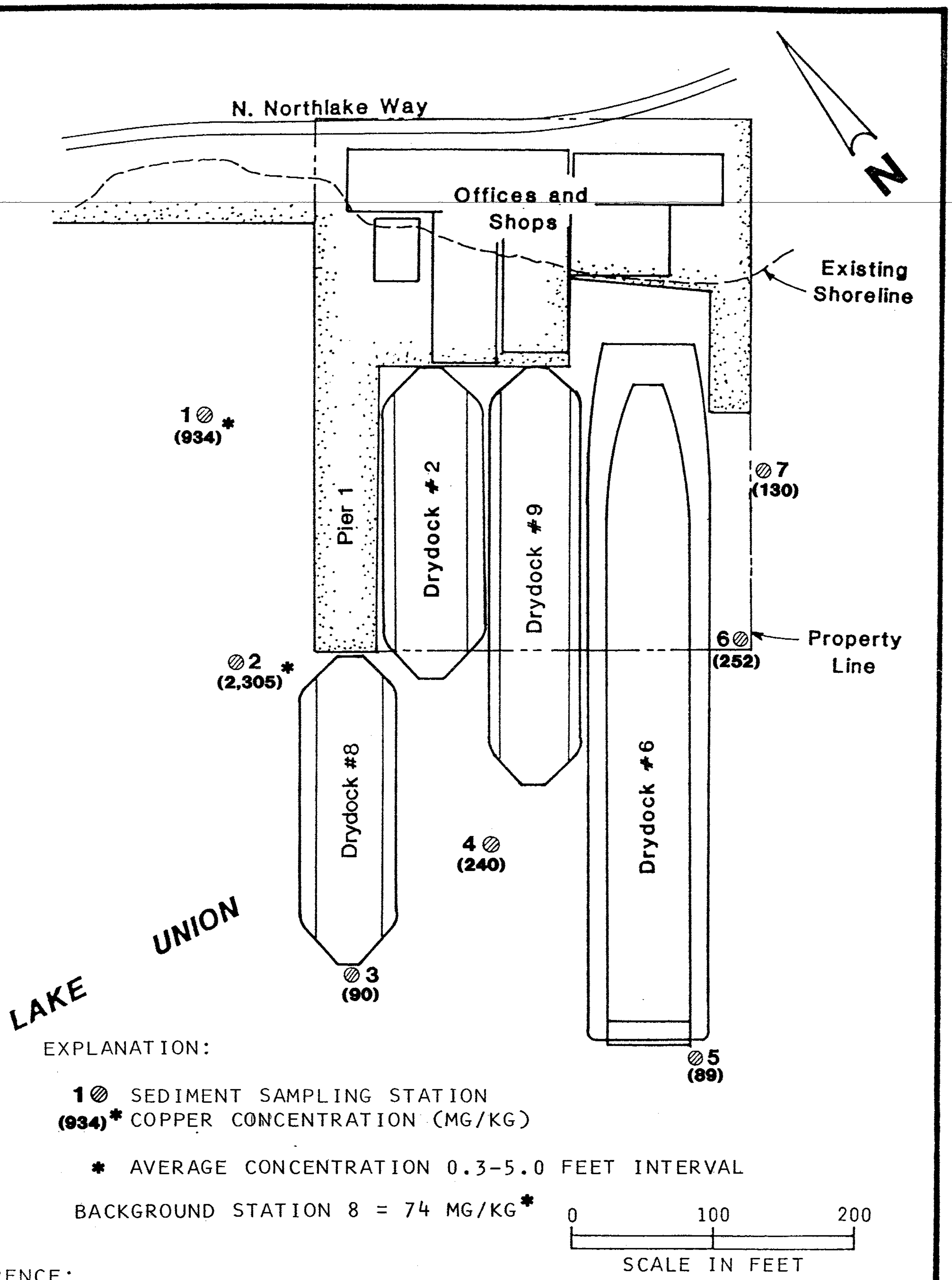
GeoEngineers

COPPER, 0-0.3 FEET INTERVAL

FIGURE 9

1799-03.4 SEW:KKI 11-17-88

Rev 1-6-89 VKJ
6-26-91 LJD
7-23-91 RRR



REFERENCE:

UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

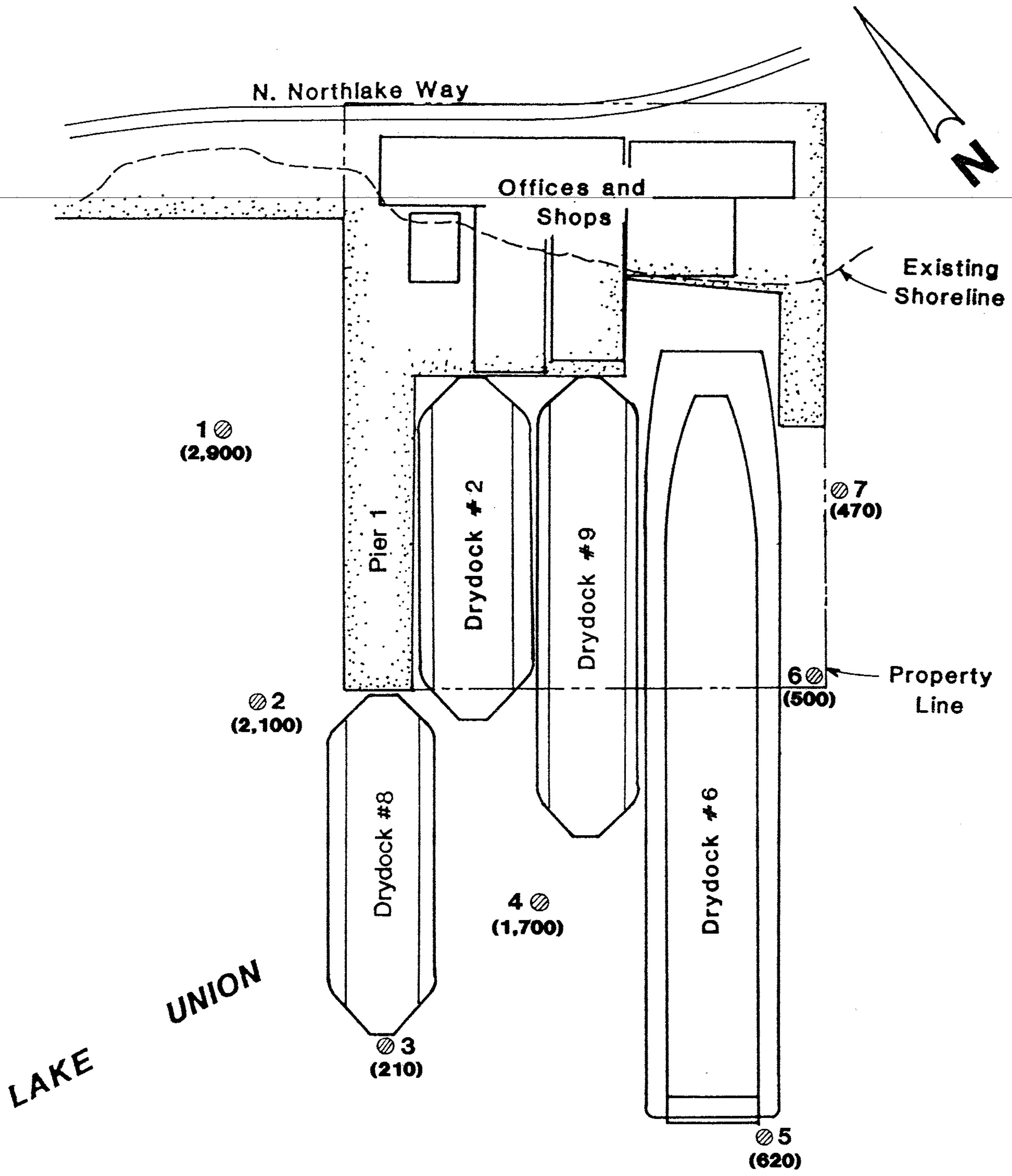
GeoEngineers

COPPER, 0.3-5.0 FEET INTERVAL

FIGURE 10

1790-03-A SEW:VKT 11-17-89

Rev. 1-6-89 VKT
6-26-91 LJD
7-23-91 RRR



EXPLANATION:

1 ▨ SEDIMENT SAMPLING STATION
(2,900) LEAD CONCENTRATION (MG/KG)

BACKGROUND STATION 8 = 250 MG/KG

0 100 200
SCALE IN FEET

REFERENCE:
UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

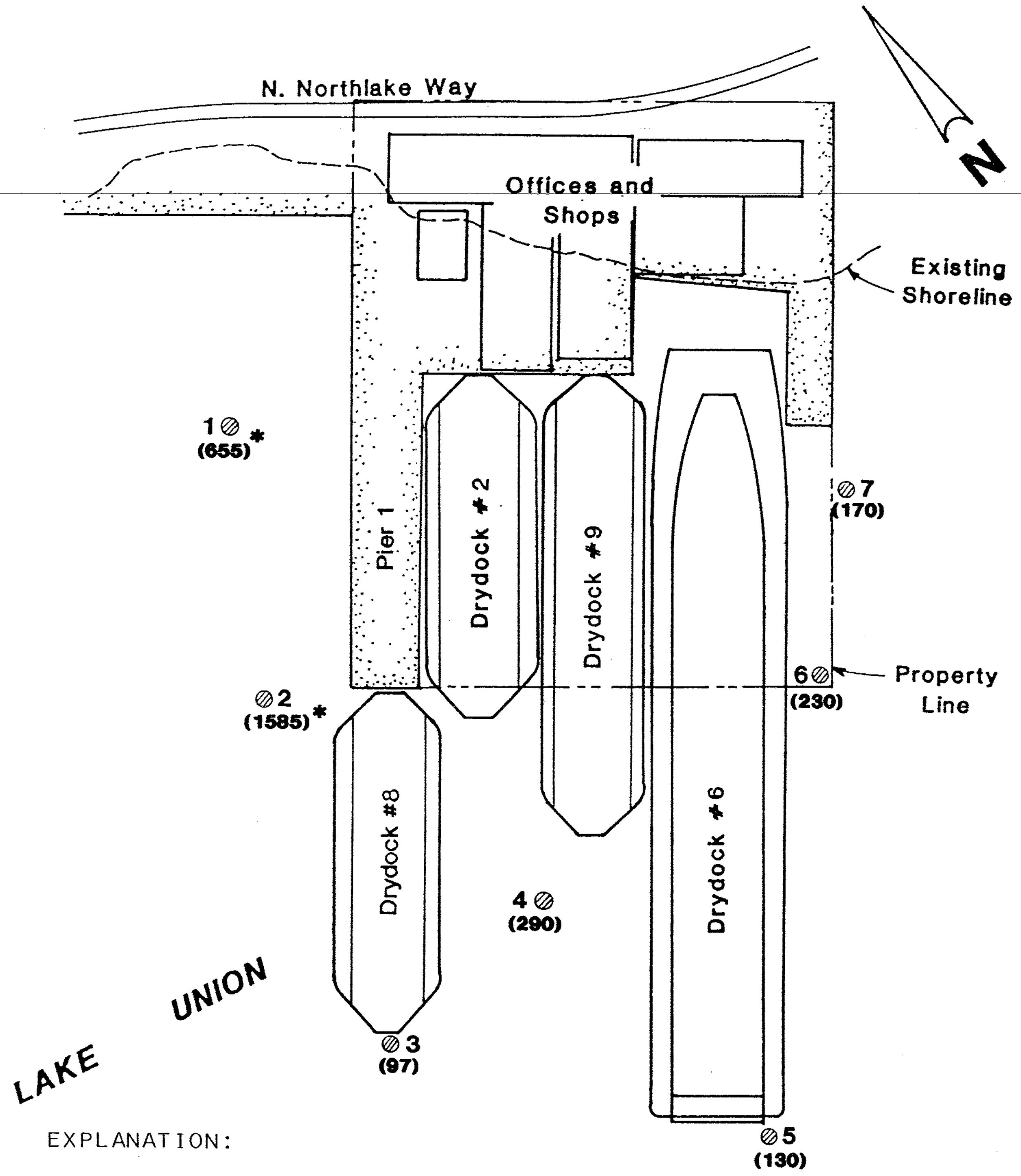
GeoEngineers

LEAD, 0–0.3 FEET INTERVAL

FIGURE 11

1?00-03 ... SEW:... 11-17-93

Re ..6-8.. PKT
6-26-91 LJD
7-23-91 RRR



EXPLANATION:

1 ▨ SEDIMENT SAMPLING STATION
(655)* LEAD CONCENTRATION (MG/KG)

* AVERAGE CONCENTRATION 0.3-5.0 FEET INTERVAL

BACKGROUND STATION 8 = 103 MG/KG*

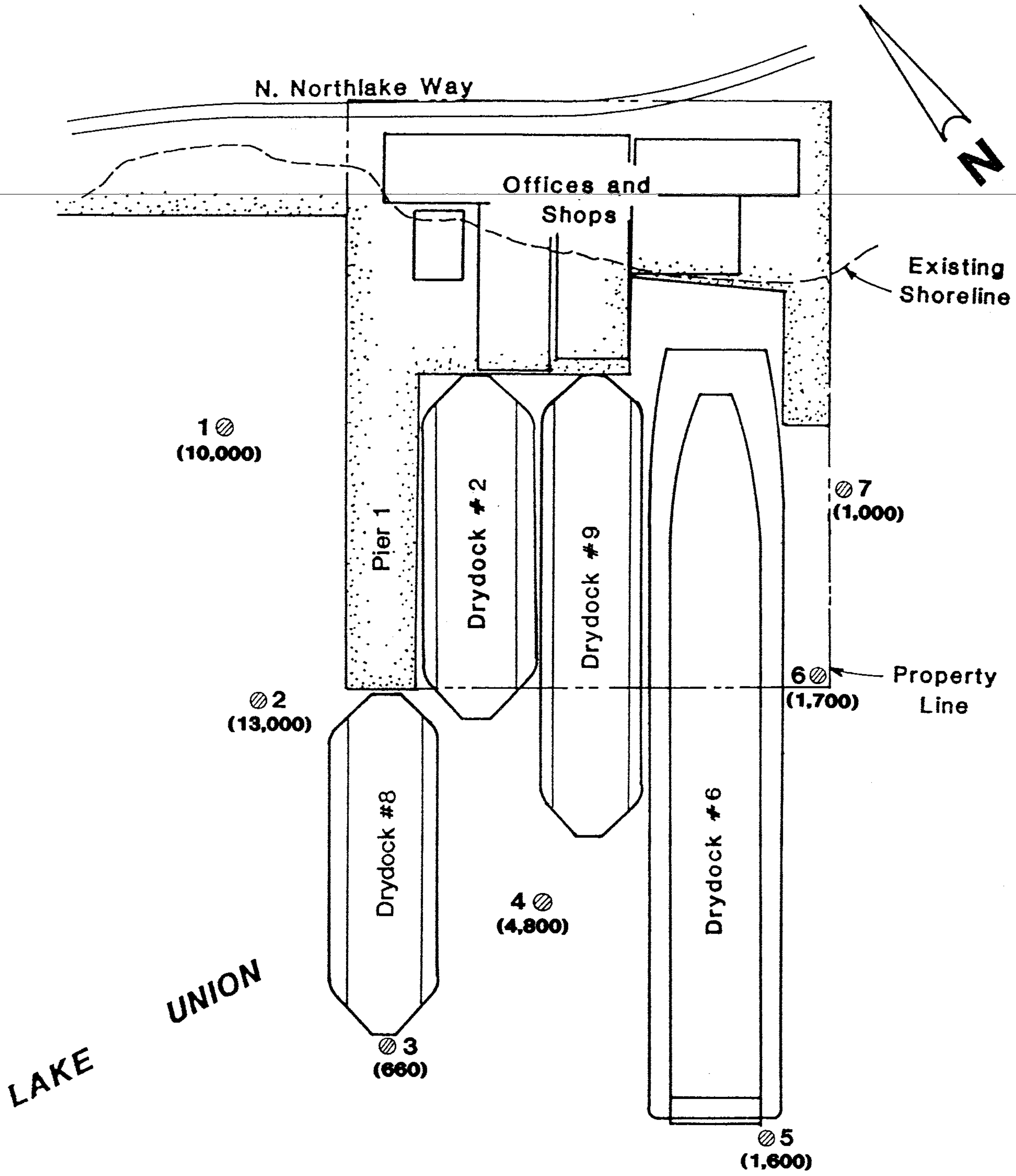
REFERENCE:
UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

GeoEngineers

LEAD, 0.3–5.0 FEET INTERVAL

FIGURE 12

-03 · EW 11- 3 Re 6-8 KT
6-26-91 LJD
7-23-91 RRR



EXPLANATION:

1 ▨ SEDIMENT SAMPLING STATION

(10,000) ZINC CONCENTRATION (MG/KG)

BACKGROUND STATION 8 = 340 MG/KG

0 100 200

SCALE IN FEET

REFERENCE:

UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

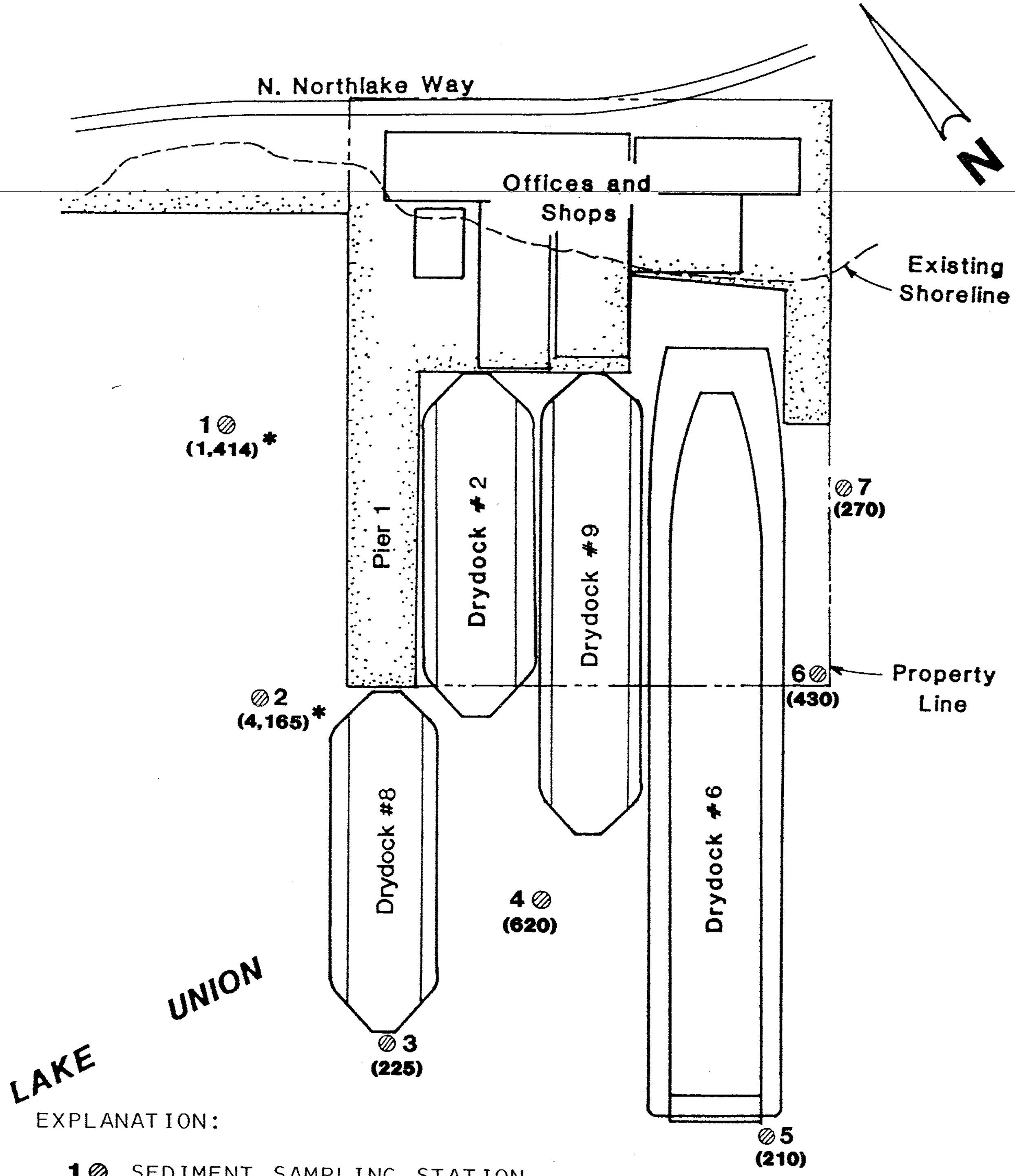
GeoEngineers

ZINC, 0–0.3 FEET INTERVAL

FIGURE 13

...-03 ... SEW:... 11-...

Rev ...6-8... KT
6-26-91 LJD
7-25-91 RRR



EXPLANATION:

1 ▨ SEDIMENT SAMPLING STATION
(1,414)* ZINC CONCENTRATION

* AVERAGE CONCENTRATION 0.3-5.0 FEET INTERVAL

BACKGROUND STATION 8 = 184 MG/KG*

0 100 200
SCALE IN FEET

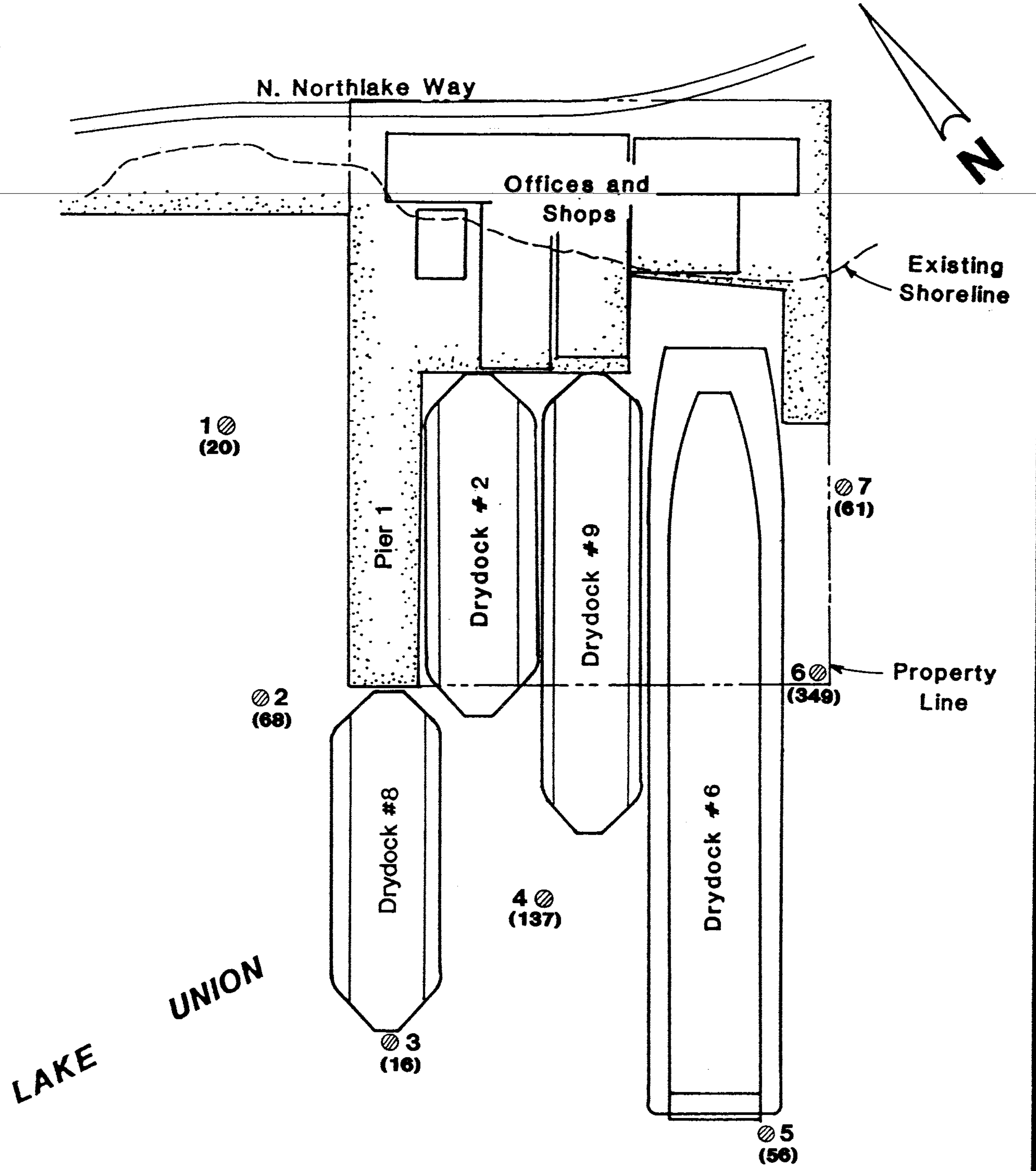
REFERENCE:
UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

GeoEngineers

ZINC, 0.3-5.0 FEET INTERVAL

FIGURE 14

6-26-91 LJD
9-23-91 RRR



EXPLANATION:

1 ▨ SEDIMENT SAMPLING STATION
(20) TOTAL LPAHS (MG/KG)

BACKGROUND STATION 8 = 46 MG/KG

0 100 200
SCALE IN FEET

REFERENCE:
UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

GeoEngineers

TOTAL LPAHS, 0-0.3 FEET INTERVAL

FIGURE 15

-03 EW 11- 3 Re 6-8 KT
6-26-91 LJD
7-23-91 RRR

ATI I.D. # 9101-238

GENERAL CHEMISTRY RESULTS

CLIENT	: GEOENGINEERS, INC.	MATRIX	: SEDIMENT
PROJECT #	: 1299-003-B04		
PROJECT NAME	: UNIMAR, INC.	UNITS	: %

ATI I.D.#	CLIENT I.D.	MOISTURE
9101-238-1	5A 0-0.3'	85
9101-238-2	6A 0-0.3'	80
9101-238-3	11 DUPLICATE	81
9101-238-4	6B 0.3-5.0'	66
9101-238-8	7A 0-0.3'	63
9101-238-9	7B 0.3-5.0'	62

GENERAL CHEMISTRY QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

SAMPLE MATRIX : SEDIMENT/SOIL

PARAMETER	UNITS	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED RESULT	SPIKE ADDED	% REC
PETROLEUM HYDROCARBONS	mg/Kg	9101-238-1	780	776	0	**	**	**
PETROLEUM HYDROCARBONS	mg/Kg	9101-238-9	189	204	8	455	255	104
PETROLEUM HYDROCARBONS	mg/Kg	BLANK SPIKE	N/A	N/A	N/A	293	268	109
MOISTURE	%	9101-242-9	29	27	7	N/A	N/A	N/A

** Due to the necessary dilution of the sample, result was not attainable.

$$\text{\% Recovery} = \frac{\text{(Spike Sample Result - Sample Result)}}{\text{Spike Concentration}} \text{ X 100}$$

$$\text{RPD (Relative \% Difference)} = \frac{\text{(Sample Result - Duplicate Result)}}{\text{Average Result}} \text{ X 100}$$

AnalyticalTechnologies,Inc.
560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

1/30/91
DATE 1/29/91 PAGE 1 OF 1

LABORATORY NUMBER: 9101-238

PROJECT MANAGER: Paul Werner
COMPANY: GeoEngineers
ADDRESS: Bellevue

PHONE: 746-5200 SAMPLED BY: P.G. Werner

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	~~TOX 9020~~ Total Metals	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)	TBT		NUMBER OF CONTAINERS
5A 0-0.3' Bottom Sediment	1/21/91	16:04	Soil	-1					X			X				X				X								X			1
6A 0-0.3' "	1/30/91	11:20		-2					X			X				X				X								X			1
" Duplicate		11:25		-3					X			X				X				X								X			1
6B 0.3-5.0'		14:30		-4						X						X				X											1
VAN VEEN RINSEATE		1215	WATER																												
10D				-5						X		X				X				X											8
Shelby Rinseate		1300																													
10B				-6						X		X				X				X											8
BETA RINSEATE 10F		1445		-7																X									X		2
Bottom SEDIMENT 6			Soil																												
~~6A~~ 7A PGW 1/30/91		1415		-8					X			X				X				X								X			1
~~6B~~ 7B "		1535		-9						X						X				X											1

PROJECT INFORMATION

PROJECT NUMBER: 1299-003-1304
PROJECT NAME: Unimar, Inc
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	24
COC SEALS/INTACT? Y/N/NA	y
RECEIVED GOOD COND./COLD	y
RECEIVED VIA: Courier	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:
Seal # 01299-009 PGW
01299-010 PGW
01299-011 PGW
7A = 7A 0-0.3' 7B = ~~7~~B 0.3-5.0 Dm 7/8/91

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 18:00	Signature: Time:	Signature: Time:
Printed Name: Paul Werner Date: 1/30/91	Printed Name: Date:	Printed Name: Date:
Company: GEI	Company:	Company:
RECEIVED BY: 1.	**RECEIVED BY: 2.**	**RECEIVED BY: (LAB) 3.**
Signature: DL Thomas Time: 11:05	Signature: Time:	Signature: Time:
Printed Name: DL Thomas Date: 1-31-91	Printed Name: Date:	Printed Name: Date:
Company:	Company:	Analytical Technologies, Inc.

I Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - O IATOR

SD

AnalyticalTechnologies,Inc. 102 026
560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 2/1/91 PAGE 1 OF 1

LABORATORY NUMBER: 102026

PROJECT MANAGER: ~~Kar~~ Donna McKinney
COMPANY: ATI - Renton
ADDRESS:
PHONE: (206)228-8335 SAMPLED BY:

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA *	8310 HPLC PNA silica gel	8080 Pesticides & PCB's	PCB's ONLY *	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)			NUMBER OF CONTAINERS
9101-238-1	1/29/91		Sed	05					✓			✓																			1
2	1/30/91			06					✓			✓																			
3				07					✓			✓																			
4			↓	03						✓																					↓
5			water	01						✓		✓																			6
6			↓	02						✓		✓																			8
8			Sed	08					✓			✓																			1
↓ 9	↓		↓	04						✓																					1

PROJECT INFORMATION

PROJECT NUMBER: 9101-238
PROJECT NAME: Geoengineers
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☐ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	18
COC SEALS/INTACT? Y/N/NA	Y
RECEIVED GOOD COND./COLD	Y
RECEIVED VIA: Fed Ex	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)
SPECIAL INSTRUCTIONS: 8310 due 2/13/91 ($250.00)

* extract & Return ASAP will fax % moi

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Time:	Signature: Time:	Signature: Time:
Printed Name: V Pennick Date: 2/1/91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Company:
RECEIVED BY: 1.	**RECEIVED BY: 2.**	**RECEIVED BY: (LAB) 3.**
Signature: Time:	Signature: Time:	Signature: Time: 1000
Printed Name: Date:	Printed Name: Date:	Printed Name: K Rado Date: 2-2-91
Company:	Company:	Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINATOR

APPENDIX

ToxScan Inc.

42 Hangar Way
Watsonville, CA 95076

(408) 724-4522

FAX (408) 724-3188

Analytical Technologies, Inc.
560 Naches Avenue SW, Suite 101
Renton, WA 98055

February 26, 1991
REVISED REPORT: August 25, 1991,
to include QA/QC

Attn: Donna McKinney

MATERIAL: Water sample received February 2, 1991
ANALYSIS COMPLETED: February 25, 1991
IDENTIFICATION: Geoengineers
TOXSCAN NUMBER: T-7221
REPORT: Quantitative chemical analysis is as follows, expressed as nanograms per liter (parts per trillion) as received:

Sample ID	Monobutyltin	Dibutyltin	Tributyltin	Tetrabutyltin	Tripropyl Tin Surrogate
9101-238-7	15	4	ND	ND	80
Spike Recovery -see T-7251 56648-5	72%	83%	84%	77%	91

ND = None detected

Detection limit = 1 part per trillion

RECEIVED AUG 29 1991

Philip D. Carpenter
Laboratory Director

Toxscan

T 2-22/

AnalyticalTechnologies,Inc.
560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 2/1/91 PAGE 1 OF 1

LABORATORY NUMBER: T-222

PROJECT MANAGER: Donna McKinney
COMPANY: ATI - Renton
ADDRESS:
PHONE: (206) 228-8335 SAMPLED BY:

SAMPLE DISPOSAL INSTRUCTIONS

☐ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)	TRIBUTYL TIN		NUMBER OF CONTAINERS
9101-238-7	1/30/91		water	-01																									✓		1

1 L amber glass bottle
[illegible]

PROJECT INFORMATION

PROJECT NUMBER: 9101-238
PROJECT NAME: Geoengineers
PURCHASE ORDER NUMBER: 02236
ONGOING PROJECT? YES ☐ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	1
COC SEALS/INTACT? Y/N/NA	
RECEIVED GOOD COND./COLD	
RECEIVED VIA:	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☐ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)
SPECIAL INSTRUCTIONS: please include proj. # on invoice
data due ASAP

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: DMcKinney Time: 14:00	Signature: Time:	Signature: Time:
Printed Name: D McKinney Date: 2/1	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: Time: 10 a.m.	Signature: Time:	Signature: Time:
Printed Name: [illegible] Date: 2/1	Printed Name: Date:	Printed Name: Date:
Company: TCI	Company:	Analytical Technologies, Inc.

Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-83 • Pensacola (904)474-1001 **DISTRIBUTION:** White, Canary - ATI • Pink - ORIG ATOR

NO 1067

RINSEATE SAMPLE 10E

WATER SAMPLE 8A

INTERSTITIAL WATER SAMPLE 8C

SEDIMENT SAMPLES 8B-8M

AnalyticalTechnologies,Inc. 560 Naches Avenue, S.W., Suite 101, Renton, WA 98055, (206) 228-8335

ATI I.D. # 9101-167

GeoEngineers

MAR 04 1991

Routing ☐ ☐
☐ ☐ ☐
File

March 4, 1991

GeoEngineers, Inc.
8410 154th Avenue N.E.
Redmond, WA 98052

Attention : Paul Werner

Project Number : 1299-003-B04

Project Name : Unimar, Inc.

On January 23, 1991, Analytical Technologies, Inc., received three water and 13 sediment samples for analysis. The samples were analyzed with EPA methodology or equivalent methods as specified in the attached analytical schedule. The results, sample cross reference, and quality control data are enclosed.

This is a partial report as it does not contain the TCLP Metals results. These results will follow in approximately 60 days.

Donna M. McKinney
Donna M. McKinney
Project Manager

Frederick W. Grothkopp
Frederick W. Grothkopp
Technical Manager

FWG/elf

ATI I.D. # 9101-167

SAMPLE CROSS REFERENCE SHEET

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ATI #	CLIENT DESCRIPTION	DATE SAMPLED	MATRIX
9101-167-1	10E BETA RINSATE	01/22/91	WATER
9101-167-2	8A WATER SAMPLE	01/22/91	WATER
9101-167-3	8B 0-0.3′	01/22/91	SEDIMENT
9101-167-4	8C 0.3-2.0′	01/22/91	SEDIMENT
9101-167-5	8D 0.3-0.5′	01/22/91	SEDIMENT
9101-167-6	8E 0.5-1.0′	01/22/91	SEDIMENT
9101-167-7	8F 1.0-1.5′	01/22/91	SEDIMENT
9101-167-8	8G 1.5-2.0′	01/22/91	SEDIMENT
9101-167-9	8H 2.0-2.5′	01/22/91	SEDIMENT
9101-167-10	8I 2.5-3.0′	01/22/91	SEDIMENT
9101-167-11	8J 3.0-3.5′	01/22/91	SEDIMENT
9101-167-12	8K 3.5-4.0′	01/22/91	SEDIMENT
9101-167-13	8L 4.0-4.5′	01/22/91	SEDIMENT
9101-167-14	8M 4.5-4.8′	01/22/91	SEDIMENT
9101-167-15	8C (INTERSTITIAL WATER)	01/22/91	WATER
9101-167-16	8C 0.3-2.0′	01/22/91	SEDIMENT

----- TOTALS -----

MATRIX	# SAMPLES
WATER	3
SEDIMENT	13

ATI STANDARD DISPOSAL PRACTICE

The samples from this project will be disposed of in thirty (30) days from the date of this report. If an extended storage period is required, please contact our sample control department before the scheduled disposal date.

ANALYTICAL SCHEDULE

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ANALYSIS	TECHNIQUE	REFERENCE	LAB
SEMI-VOLATILE COMPOUNDS	GCMS	EPA 8270	R
POLYCHLORINATED BIPHENYLS (PCBs)	GC/ECD	EPA 8080	R
POLYNUCLEAR AROMATIC HYDROCARBONS	HPLC/UV	EPA 8310	SD
TRIBUTYLTIN	GC/FPD	BATTELLE	SUB
ARSENIC	AA/GF	EPA 7060	R
BARIUM	AA/F	EPA 7080	R
CADMIUM	AA/F	EPA 7130	R
CADMIUM	AA/GF	EPA 7131	R
CHROMIUM	AA/F	EPA 7190	R
COPPER	AA/F	EPA 7210	R
LEAD	AA/F	EPA 7420	R
LEAD	AA/GF	EPA 7421	R
MERCURY	AA/COLD VAPOR	EPA 7470	R
MERCURY	AA/COLD VAPOR	EPA 7471	R
NICKEL	AA/F	EPA 7520	R
SELENIUM	AA/GF	EPA 7740	R
SILVER	AA/F	EPA 7760	R
ZINC	AA/F	EPA 7950	R
HARDNESS	TITRATION	EPA 130.2	R

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ANALYTICAL SCHEDULE
CONTINUED

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ANALYSIS	TECHNIQUE	REFERENCE	LAB
PETROLEUM HYDROCARBONS	IR	EPA 418.1	R
MOISTURE	GRAVIMETRIC	METHOD 7-2.2	R

R = ATI - Renton
SD = ATI - San Diego
T = ATI - Tempe
PNR = ATI - Pensacola
FC = ATI - Fort Collins
SUB = Subcontract

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<0.17
PHENOL	<0.17
ANILINE	<0.17
BIS(2-CHLOROETHYL)ETHER	<0.17
2-CHLOROPHENOL	<0.17
1,3-DICHLOROBENZENE	<0.17
1,4-DICHLOROBENZENE	<0.17
BENZYL ALCOHOL	<0.17
1,2-DICHLOROBENZENE	<0.17
2-METHYLPHENOL	<0.17
BIS(2-CHLOROISOPROPYL)ETHER	<0.17
4-METHYLPHENOL	<0.17
N-NITROSO-DI-N-PROPYLAMINE	<0.17
HEXACHLOROETHANE	<0.17
NITROBENZENE	<0.17
ISOPHORONE	<0.17
2-NITROPHENOL	<0.17
2,4-DIMETHYLPHENOL	<0.17
BENZOIC ACID	<0.85
BIS(2-CHLOROETHOXY)METHANE	<0.17
2,4-DICHLOROPHENOL	<0.17
1,2,4-TRICHLOROBENZENE	<0.17
NAPHTHALENE	<0.17
4-CHLOROANILINE	<0.17
HEXACHLOROBUTADIENE	<0.17
4-CHLORO-3-METHYLPHENOL	<0.17
2-METHYLNAPHTHALENE	<0.17
HEXACHLOROCYCLOPENTADIENE	<0.17
2,4,6-TRICHLOROPHENOL	<0.17
2,4,5-TRICHLOROPHENOL	<0.85
2-CHLORONAPHTHALENE	<0.17
2-NITROANILINE	<0.85
DIMETHYLPHTHALATE	<0.17
ACENAPHTHYLENE	<0.17
3-NITROANILINE	<0.85
ACENAPHTHENE	<0.17
2,4-DINITROPHENOL	<0.85
4-NITROPHENOL	<0.85

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SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
DIBENZOFURAN	<0.17
2,4-DINITROTOLUENE	<0.17
2,6-DINITROTOLUENE	<0.17
DIETHYLPHTHALATE	<0.17
4-CHLOROPHENYL-PHENYLETHER	<0.17
FLUORENE	<0.17
4-NITROANILINE	<0.85
4,6-DINITRO-2-METHYLPHENOL	<0.85
N-NITROSODIPHENYLAMINE	<0.17
4-BROMOPHENYL-PHENYLETHER	<0.17
HEXACHLOROBENZENE	<0.17
PENTACHLOROPHENOL	<0.85
PHENANTHRENE	<0.17
ANTHRACENE	<0.17
DI-N-BUTYLPHTHALATE	<0.17
FLUORANTHENE	<0.17
BENZIDINE	<1.7
PYRENE	<0.17
BUTYLBENZYLPHTHALATE	<0.17
3,3-DICHLOROBENZIDINE	<0.34
BENZO(a)ANTHRACENE	<0.17
BIS(2-ETHYLHEXYL)PHTHALATE	<0.17
CHRYSENE	<0.17
DI-N-OCTYLPHTHALATE	<0.17
BENZO(b)FLUORANTHENE	<0.17
BENZO(k)FLUORANTHENE	<0.17
BENZO(a)PYRENE	<0.17
INDENO(1,2,3-cd)PYRENE	<0.17
DIBENZ(a,h,)ANTHRACENE	<0.17
BENZO(g,h,i)PERYLENE	<0.17

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	73
2-FLUOROBIPHENYL	82
TERPHENYL-d14	76
PHENOL-d6	86
2-FLUOROPHENOL	66
2,4,6-TRIBROMOPHENOL	87

ATI I.D. # 9101-167

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SOIL
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
HYDROCARBON	1883	0.63
HYDROCARBON	1944	1.0
HYDROCARBON	2013	1.3
HYDROCARBON	2094	1.4
HYDROCARBON	2306	1.0

ATI I.D. # 9101-167-3

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 8B 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/22/91
DATE RECEIVED : 01/23/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 5

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<4.0
PHENOL	<4.0
ANILINE	<4.0
BIS(2-CHLOROETHYL)ETHER	<4.0
2-CHLOROPHENOL	<4.0
1,3-DICHLOROBENZENE	<4.0
1,4-DICHLOROBENZENE	<4.0
BENZYL ALCOHOL	<4.0
1,2-DICHLOROBENZENE	<4.0
2-METHYLPHENOL	<4.0
BIS(2-CHLOROISOPROPYL)ETHER	<4.0
4-METHYLPHENOL	<4.0
N-NITROSO-DI-N-PROPYLAMINE	<4.0
HEXACHLOROETHANE	<4.0
NITROBENZENE	<4.0
ISOPHORONE	<4.0
2-NITROPHENOL	<4.0
2,4-DIMETHYLPHENOL	<4.0
BENZOIC ACID	<20
BIS(2-CHLOROETHOXY)METHANE	<4.0
2,4-DICHLOROPHENOL	<4.0
1,2,4-TRICHLOROBENZENE	<4.0
NAPHTHALENE	4.1
4-CHLOROANILINE	<4.0
HEXACHLOROBUTADIENE	<4.0
4-CHLORO-3-METHYLPHENOL	<4.0
2-METHYLNAPHTHALENE	3.7 J
HEXACHLOROCYCLOPENTADIENE	<4.0
2,4,6-TRICHLOROPHENOL	<4.0
2,4,5-TRICHLOROPHENOL	<20
2-CHLORONAPHTHALENE	<4.0
2-NITROANILINE	<20
DIMETHYLPHTHALATE	<4.0
ACENAPHTHYLENE	2.6 J
3-NITROANILINE	<20
ACENAPHTHENE	6.6
2,4-DINITROPHENOL	<20
4-NITROPHENOL	<20

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J = Estimated value.

ATI I.D. # 9101-167-3

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/22/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/23/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: 8B 0-0.3'	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 5

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
DIBENZOFURAN	<4.0
2,4-DINITROTOLUENE	<4.0
2,6-DINITROTOLUENE	<4.0
DIETHYLPHTHALATE	<4.0
4-CHLOROPHENYL-PHENYLETHER	<4.0
FLUORENE	5.9
4-NITROANILINE	<20
4,6-DINITRO-2-METHYLPHENOL	<20
N-NITROSODIPHENYLAMINE	<4.0
4-BROMOPHENYL-PHENYLETHER	<4.0
HEXACHLOROBENZENE	<4.0
PENTACHLOROPHENOL	<20
PHENANTHRENE	16
ANTHRACENE	6.8
DI-N-BUTYLPHTHALATE	<4.0
FLUORANTHENE	43
BENZIDINE	<40
PYRENE	40
BUTYLBENZYLPHTHALATE	<4.0
3,3-DICHLOROBENZIDINE	<8.1
BENZO(a)ANTHRACENE	16
BIS(2-ETHYLHEXYL)PHTHALATE	<4.0
CHRYSENE	17
DI-N-OCTYLPHTHALATE	<4.0
BENZO(b)FLUORANTHENE	23
BENZO(k)FLUORANTHENE	8.3
BENZO(a)PYRENE	25
INDENO(1,2,3-cd)PYRENE	15
DIBENZ(a,h,)ANTHRACENE	2.9 J
BENZO(g,h,i)PERYLENE	21

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	62
2-FLUOROBIPHENYL	107
TERPHENYL-d14	97
PHENOL-d6	87
2-FLUOROPHENOL	58
2,4,6-TRIBROMOPHENOL	113

J = Estimated value.

ATI I.D. # 9101-167-3

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 8B 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/22/91
DATE RECEIVED : 01/23/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 5

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
NAPHTHALENE, 1,8-DIMETHYL-	890	7.1
HYDROCARBON	1398	26
BENZO(E)PYRENE	1885	12
HEPTACOSANE	2010	13
DIBENZO(DEF,MNO)CHRYSENE	2216	10

SEMI-VOLATILE ORGANIC
QUALITY CONTROL DATA

CLIENT	: GEOENGINEERS, INC.		SAMPLE I.D.	: BLANK SPIKE
PROJECT #	: 1299-003-B04		DATE EXTRACTED	: 01/30/91
PROJECT NAME	: UNIMAR, INC.		DATE ANALYZED	: 02/08/91
EPA METHOD	: 8270		MATRIX	: SOIL
			UNITS	: mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
1,2,4-TRICHLOROBENZENE	<0.17	3.33	2.39	72	2.86	86	18
ACENAPHTHENE	<0.17	3.33	2.19	66	2.42	73	10
2,4-DINITROTOLUENE	<0.17	3.33	2.46	74	2.56	77	4
PYRENE	<0.17	3.33	2.34	70	2.62	78	11
N-NITROSO-DI-N-PROPYLAMINE	<0.17	3.33	2.26	68	2.68	81	17
1,4-DICHLOROBENZENE	<0.17	3.33	2.23	67	2.65	79	17
PENTACHLOROPHENOL	<0.85	13.3	11.6	87	11.3	85	3
PHENOL	<0.17	6.67	3.98	60	4.79	72	18
2-CHLOROPHENOL	<0.17	6.67	4.03	60	4.90	73	19
4-CHLORO-3-METHYLPHENOL	<0.17	6.67	4.44	67	4.99	75	12
4-NITROPHENOL	<0.85	13.3	11.5	86	11.6	87	1

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-167

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SOIL
EPA METHOD : 8080 (PCB)
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/04/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
PCB 1016	<0.033
PCB 1221	<0.033
PCB 1232	<0.033
PCB 1242	<0.033
PCB 1248	<0.033
PCB 1254	<0.033
PCB 1260	<0.033

ATI I.D. # 9101-167-3

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/22/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/23/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: 8B 0-0.3'	DATE ANALYZED	: 02/04/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
PCB 1016	<0.033
PCB 1221	<0.033
PCB 1232	<0.033
PCB 1242	<0.033
PCB 1248	<0.033
PCB 1254	<0.033
PCB 1260	<0.033

ATI I.D. # 9101-167-16

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/22/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/23/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: 8C 0.3-2.0'	DATE ANALYZED	: 02/04/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
PCB 1016	<0.033
PCB 1221	<0.033
PCB 1232	<0.033
PCB 1242	<0.033
PCB 1248	<0.033
PCB 1254	<0.033
PCB 1260	<0.033

POLYCHLORINATED BIPHENYLS (PCB)
QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
EPA METHOD : 8080 (PCB)

SAMPLE ID : BLANK SPIKE
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/04/91
MATRIX : SOIL
UNITS : mg/Kg

COMPOUND	SAMPLE RESULT	CONC SPIKED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % RECOVERY	RPD
PCB 1260	<0.033	0.33	0.345	104	0.351	106	2

$$\% \text{ Recovery} = \frac{(\text{Spike Sample result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-167

POLYNUCLEAR AROMATICS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8310	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
NAPHTHALENE	<0.083
ACENAPHTHYLENE	<0.17
ACENAPHTHENE	<0.17
FLUORENE	<0.017
PHENANTHRENE	<0.0083
ANTHRACENE	<0.0083
FLUORANTHENE	<0.017
PYRENE	<0.017
BENZO(a)ANTHRACENE	<0.017
CHRYSENE	<0.017
BENZO(b)FLUORANTHENE	<0.017
BENZO(k)FLUORANTHENE	<0.017
BENZO(a)PYRENE	<0.017
DIBENZ(a,h)ANTHRACENE	<0.034
BENZO(g,h,i)PERYLENE	<0.017
INDENO(1,2,3-cd)PYRENE	<0.017

ATI I.D. # 9101-167-3

POLYNUCLEAR AROMATICS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 8B
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8310
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/22/91
DATE RECEIVED : 01/23/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/09/91
UNITS : mg/Kg
DILUTION FACTOR : 10

COMPOUND	RESULT
NAPHTHALENE	<4.0
ACENAPHTHYLENE	<5.5
ACENAPHTHENE	<5.5
FLUORENE	<0.55
PHENANTHRENE	9.0
ANTHRACENE	28
FLUORANTHENE	29
PYRENE	36
BENZO(a)ANTHRACENE	11
CHRYSENE	14
BENZO(b)FLUORANTHENE	11
BENZO(k)FLUORANTHENE	6.2
BENZO(a)PYRENE	17
DIBENZ(a,h)ANTHRACENE	<1.6
BENZO(g,h,i)PERYLENE	18
INDENO(1,2,3-cd)PYRENE	14

ATI I.D. # 9101-167-16

POLYNUCLEAR AROMATICS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 8C 0.3-2.0'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8310
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/22/91
DATE RECEIVED : 01/23/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/09/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
NAPHTHALENE	0.87
ACENAPHTHYLENE	0.50
ACENAPHTHENE	<0.57
FLUORENE	0.40
PHENANTHRENE	1.7
ANTHRACENE	0.47
FLUORANTHENE	7.7
PYRENE	9.7
BENZO(a)ANTHRACENE	2.9
CHRYSENE	4.0
BENZO(b)FLUORANTHENE	2.6
BENZO(k)FLUORANTHENE	1.5
BENZO(a)PYRENE	4.3
DIBENZ(a,h)ANTHRACENE	1.3
BENZO(g,h,i)PERYLENE	4.0
INDENO(1,2,3-cd)PYRENE	3.1

ATI I.D. # 9101-167

POLYNUCLEAR AROMATICS
QUALITY CONTROL DATA

CLIENT	: GEOENGINEERS, INC.		SAMPLE I.D.	: 101289-32
PROJECT #	: 1299-003-B04		DATE EXTRACTED	: 01/30/91
PROJECT NAME	: UNIMAR, INC.		DATE ANALYZED	: 02/08/91
EPA METHOD	: 8310		MATRIX	: SOIL
			UNITS	: mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
ACENAPHTHYLENE	<0.17	16.6	11	66	11	66	0
PHENANTHRENE	<0.034	1.77	1.4	79	1.5	93	7
PYRENE	<0.017	1.76	1.8	102	1.9	108	5
BENZO(k)FLUORANTHENE	0.0083	1.50	1.2	76	1.4	89	15

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-167

POLYNUCLEAR AROMATICS
QUALITY CONTROL DATA

CLIENT	: GEOENGINEERS, INC.		SAMPLE I.D.	: 101289-32
PROJECT #	: 1299-003-B04		DATE EXTRACTED	: 01/30/91
PROJECT NAME	: UNIMAR, INC.		DATE ANALYZED	: 02/08/91
EPA METHOD	: 8310		MATRIX	: SOIL
			UNITS	: mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
ACENAPHTHYLENE	<0.17	16.6	11	66	N/A	N/A	N/A
PHENANTHRENE	<0.034	1.77	1.3	73	N/A	N/A	N/A
PYRENE	<0.017	1.76	1.7	97	N/A	N/A	N/A
BENZO(k)FLUORANTHENE	<0.0083	1.58	1.3	82	N/A	N/A	N/A

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

GAS CHROMATOGRAPHY RESULTS *

CLIENT	: GEOENGINEERS, INC.	MATRIX	: WATER
PROJECT #	: 1299-003-B04		
PROJECT NAME	: UNIMAR, INC.	UNITS	: ng/L

PARAMETER	10E BETA RINSATE -1	8A WATER SAMPLE -2	8C INTERSTITIAL WATER -15
MONOBUTYLTIN	<1	<1	82
DIBUTYLTIN	<1	<1	90
TRIBUTYLTIN	<1	9.2	310
TETRABUTYLTIN	<1	<1	160

* Analyzed by GC/FPD, Method Battelle N05196300.

ATI I.D. # 9101-167

METALS RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

PARAMETER	10E BETA RINSATE -1	8A WATER SAMPLE -2	8C INTERSTITIAL WATER -15	REAGENT BLANK
ARSENIC	<0.005	<0.005	0.042	<0.005
BARIUM	<0.06	<0.06	0.16	<0.06
CADMIUM	<0.0003	<0.0003	<0.0003	<0.0003
CHROMIUM	<0.02	<0.02	<0.02	<0.02
COPPER	<0.02	<0.02	0.03	<0.02
LEAD	<0.005	<0.005	<0.005	<0.005
MERCURY	<0.0005	<0.0005	<0.002*	<0.0005
NICKEL	<0.03	<0.03	<0.03	<0.03
SELENIUM	<0.005	<0.005	<0.005	<0.005
SILVER	<0.02	<0.02	<0.02	<0.02
ZINC	0.02	0.01	<0.01	<0.01

* Increased detection limit due to limited sample.

ATI I.D. # 9101-167

METALS QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

COMPOUND	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED SAMPLE	SPIKE CONC	% REC
ARSENIC	9101-167-15	0.042	0.036	15	0.052	0.025	40*
ARSENIC	BLANK SPIKE	N/A	N/A	N/A	0.024	0.025	96
BARIUM	9101-167-15	0.16	0.18	12	16.8	20.0	83
CADMIUM	9101-174-2	0.0012	0.0009	29	0.0019	0.0010	70
CHROMIUM	9101-191-13	<0.02	<0.02	0	1.98	2.50	79
COPPER	9101-174-2	0.65	0.59	10	3.04	2.50	96
LEAD	9101-184-3	0.113	0.112	1	**	**	**
LEAD	BLANK SPIKE	N/A	N/A	N/A	0.033	0.025	132
MERCURY	9101-202-2	<0.0005	<0.0005	0	0.0030	0.0020	150*
MERCURY	BLANK SPIKE	N/A	N/A	N/A	0.0029	0.0020	145
NICKEL	9101-167-15	<0.03	<0.03	0	2.48	2.50	99
SELENIUM	9101-167-15	<0.005	<0.005	0	0.018	0.025	72
SILVER	9101-191-13	<0.02	<0.02	0	0.94	1.00	94
ZINC	9101-167-15	<0.01	<0.01	0	0.49	0.50	98

* Out of limits.

** Due to the necessary dilution of the sample, result was not attainable.

$$\text{\% Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : SEDIMENT

ELEMENT	DATE PREPARED	DATE ANALYZED
ARSENIC	01/30/91	02/08/91
BARIUM	01/30/91	02/12/91
CADMIUM	01/30/91	02/06/91
CADMIUM	01/30/91	02/07/91
CHROMIUM	01/30/91	02/07/91
COPPER	01/30/91	02/07/91
LEAD	01/30/91	02/06/91
LEAD	01/30/91	02/07/91
MERCURY	01/30/91	02/22/91
NICKEL	01/30/91	02/07/91
SELENIUM	01/30/91	02/08/91
SILVER	01/30/91	02/08/91
ZINC	01/30/91	02/07/91

METALS RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

ATI I.D. #	CLIENT I.D.	ARSENIC	BARIUM	CADMIUM	CHROMIUM
9101-167-3	8B 0-0.3′	71	92	2.4	47
9101-167-5	8D 0.3-0.5′	-	-	3.6	-
9101-167-6	8E 0.5-1.0′	-	-	<2	-
9101-167-7	8F 1.0-1.5′	-	-	<4	-
9101-167-8	8G 1.5-2.0′	-	-	<3	-
9101-167-9	8H 2.0-2.5′	-	-	<3	-
9101-167-10	8I 2.5-3.0′	-	-	3	-
9101-167-11	8J 3.0-3.5′	-	-	2	-
9101-167-12	8K 3.5-4.0′	-	-	<5	-
9101-167-13	8L 4.0-4.5′	-	-	<5	-
9101-167-14	8M 4.5-4.8′	-	-	12	-
9101-167-16	8C 0.3-2.0′	24	92	3.0	32
REAGENT BLANK	-	<0.5	<6	<1	<2

METALS RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

ATI I.D. #	CLIENT I.D.	COPPER	LEAD	MERCURY	NICKEL
9101-167-3	8B 0-0.3'	170	250	1.53	48
9101-167-5	8D 0.3-0.5'	62	61	-	-
9101-167-6	8E 0.5-1.0'	56	53	-	-
9101-167-7	8F 1.0-1.5'	120	160	-	-
9101-167-8	8G 1.5-2.0'	150	180	-	-
9101-167-9	8H 2.0-2.5'	180	350	-	-
9101-167-10	8I 2.5-3.0'	68	83	-	-
9101-167-11	8J 3.0-3.5'	39	45	-	-
9101-167-12	8K 3.5-4.0'	13	<50	-	-
9101-167-13	8L 4.0-4.5'	16	<45	-	-
9101-167-14	8M 4.5-4.8'	12	<40	-	-
9101-167-16	8C 0.3-2.0'	94	130	1.18	49
REAGENT BLANK	-	<2	<10	<0.15	<3

METALS RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

ATI I.D. #	CLIENT I.D.	SELENIUM	SILVER	ZINC
9101-167-3	8B 0-0.3'	<1	<2	340
9101-167-5	8D 0.3-0.5'	-	-	160
9101-167-6	8E 0.5-1.0'	-	-	150
9101-167-7	8F 1.0-1.5'	-	-	294
9101-167-8	8G 1.5-2.0'	-	-	301
9101-167-9	8H 2.0-2.5'	-	-	430
9101-167-10	8I 2.5-3.0'	-	-	190
9101-167-11	8J 3.0-3.5'	-	-	93
9101-167-12	8K 3.5-4.0'	-	-	46
9101-167-13	8L 4.0-4.5'	-	-	63
9101-167-14	8M 4.5-4.8'	-	-	39
9101-167-16	8C 0.3-2.0'	<1	<2	260
REAGENT BLANK	-	<0.5	<2	<1

METALS QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

PARAMETER	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED SAMPLE	SPIKE CONC	% REC
ARSENIC	9101-167-16	24	30	22	**	**	**
ARSENIC	BLANK SPIKE	N/A	N/A	N/A	1.5	1.3	115
BARIUM	9101-167-16	92	92	0	1,400	1,600	82
CADMIUM	9101-167-16	3.0	2.3	26	74.8	82.0	88
CHROMIUM	9101-167-16	32	32	0	334	410	74
COPPER	9101-167-16	94	95	1	411	410	77
LEAD	9101-167-16	130	130	0	927	820	97
MERCURY	9101-216-4	1.05	<0.80	0	1.78	1.67	44*
MERCURY	BLANK SPIKE	N/A	N/A	N/A	0.44	0.50	88
NICKEL	9101-167-16	49	46	6	418	410	90
SELENIUM	9101-167-16	<1	<1	0	1.9	4.1	46*
SELENIUM	BLANK SPIKE	N/A	N/A	N/A	1.1	1.2	92
SILVER	9101-167-16	<2	<2	0	74	82	90
ZINC	9101-167-16	260	270	4	506	410	60*
ZINC	BLANK SPIKE	N/A	N/A	N/A	230	250	92

* Out of limits due to matrix interference.

** Due to the necessary dilution of the sample, result was not attainable.

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

GENERAL CHEMISTRY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : WATER

ANALYSIS	DATE PREPARED	DATE ANALYZED
HARDNESS	02/08/91	02/08/91

ATI I.D. # 9101-167

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

ATI I.D.#	CLIENT I.D.	HARDNESS
9101-167-2	8A WATER SAMPLE	42
REAGENT BLANK	-	<5

GENERAL CHEMISTRY QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

PARAMETER	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED RESULT	SPIKE ADDED	% REC
HARDNESS	9102-011-2	27	24	12	552	500	105

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

ATI I.D. # 9101-167

GENERAL CHEMISTRY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : SEDIMENT

ANALYSIS	DATE PREPARED	DATE ANALYZED
PETROLEUM HYDROCARBONS	01/28/91	01/28/91
MOISTURE	-	01/29/91

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

ATI I.D. #	CLIENT I.D.	PETROLEUM HYDROCARBONS
9101-167-3	8B 0-0.3'	120
9101-167-7	8F 1.0-1.5'	350
9101-167-16	8C 0.3-2.0'	70
REAGENT BLANK	-	<5

GENERAL CHEMISTRY RESULTS

CLIENT	: GEOENGINEERS, INC.	MATRIX	: SEDIMENT
PROJECT #	: 1299-003-B04		
PROJECT NAME	: UNIMAR, INC.	UNITS	: %

ATI I.D. #	CLIENT I.D.	MOISTURE
9101-167-3	8B 0-0.3′	79
9101-167-5	8D 0.3-0.5′	67
9101-167-6	8E 0.5-1.0′	69
9101-167-7	8F 1.0-1.5′	80
9101-167-8	8G 1.5-2.0′	82
9101-167-9	8H 2.0-2.5′	83
9101-167-10	8I 2.5-3.0′	71
9101-167-11	8J 3.0-3.5′	79
9101-167-12	8K 3.5-4.0′	90
9101-167-13	8L 4.0-4.5′	89
9101-167-14	8M 4.5-4.8′	88
9101-167-16	8C 0.3-2.0′	70

GENERAL CHEMISTRY QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

PARAMETER	UNITS	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED RESULT	SPIKE ADDED	% REC
PETROLEUM HYDROCARBONS	mg/Kg	9101-167-16	70	75	7	348	248	112
MOISTURE	%	9101-167-13	89	89	0	N/A	N/A	N/A
MOISTURE	%	9101-220-2	71	72	1	N/A	N/A	N/A

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

AnalyticalTechnologies,Inc.
560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 1/22/91 PAGE 1 OF 2

LABORATORY NUMBER: 9101-167

PROJECT MANAGER: PAUL WERNER
COMPANY: GEO ENGINEERS
ADDRESS: 140th Ave NE Bellevue WA 98005
PHONE: 746-5200 SAMPLED BY: P. Werner

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	~~TOX 9020~~ * METALS SCREEN	% Moisture	EP TOX Metals (8) EP EXT	~~Priority Pollutant Metals (13)~~ **	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)	Tri butyl Tin TBT	Hardness	NUMBER OF CONTAINERS
10E BETA RINSEATE	1/22/91	1030	WATER	-1																			X						+		2
8A WATER SAMPLE	1/22/91	1035	WATER	-2																			X						X	X	3
8B Bottom SEDIMENT 0-0.3'		1320	Soil	-3					X	X		X				X							X					X			1
8C Bottom SEDIMENT 0.3-2.0'		13:59	Soil ~~[illegible]~~	-4																			~~X~~	Soil				Soil ~~X~~			1
8D 0.3-0.5'		14:00		-5						~~X~~ NO		~~X~~ NO				~~X~~ NO				X YES			~~X~~ NO				NO	~~X~~	[illegible] 1/23/91		1
8E 0.5-1.0'		13:27		-6																X											1
8F 1.0-1.5'		1329		-7					NO ~~X~~	[illegible]						X				X											1
8G 1.5-2.0'		1332		-8																X											1
8H 2.0-2.5'		1334		-9																X											1
8I 2.5-3.0'		1340		-10																X											1
8J 3.0-3.5		1350		-11																X											1
8K 3.5-4.0	↓	1353	↓	-12																X											1

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PROJECT INFORMATION

PROJECT NUMBER: 1299-003-R01
PROJECT NAME: UNIMAR
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	
COC SEALS/INTACT? Y/N/NA	Y
RECEIVED GOOD COND./COLD	Y
RECEIVED VIA: Courier	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:
SEALS 1299-03 PGW
1299-04 PGW
* As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Se, Ag, Zn
** Cd, Cu, Pb, Zn

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 15:30	Signature: Time:	Signature: Time:
Printed Name: PAUL WERNER Date: 1/22/91	Printed Name: Date:	Printed Name: Date:
Company: GEI	Company: -	Company:
RECEIVED BY: 1.	**RECEIVED BY: 2.**	**RECEIVED BY: (LAB) 3.**
Signature: D.L. Thomas Time: 4:40	Signature: Time:	Signature: Time:
Printed Name: D.L. Thomas Date: 1-23-91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Analytical Technologies, Inc.

AnalyticalTechnologies,Inc.
560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 1/22/91 PAGE 2 OF 3

LABORATORY NUMBER: 9101-167

PROJECT MANAGER: PAUL WERNER
COMPANY: GEO ENGINEERS
ADDRESS: SEE PAGE 1
PHONE: 746-5200 SAMPLED BY: P. WERNER

SAMPLE DISPOSAL INSTRUCTIONS

☐ ATI Disposal @ $5.00 each ☐ Return

					ANALYSIS REQUEST																										
																								TCLP ONLY							
SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	~~Priority Pollutant Metals (13)~~	8080 Pesticide (4)	8240 ZH-EXT	8270	8150 Herbicides (2)	Metals (8)	Metals Screen	Tributyl Tin	NUMBER OF CONTAINERS
Bottom SEDIMENT	/																														
8L ; 4.0 - 4.5'	1/22/91	1355	Soil	-13																									X		1
8M 4.5 - 4.8'	↓	1405	↓	-14																									X		1
8C	1/22/91	13:59	H2O	-15																			X							X	
8C ↓	↓	↓	sed	-16						X		X				X							X					X			

PROJECT INFORMATION

PROJECT NUMBER: 1299-003-B04
PROJECT NAME: Unimar, Inc
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS
COC SEALS/INTACT? Y/N/NA 4
RECEIVED GOOD COND./COLD 4
RECEIVED VIA: Courier

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)
SPECIAL INSTRUCTIONS:

RELINQUISHED BY: 1.
Signature: Paul Werner Time: 15:30
Printed Name: PAUL WERNER Date: 1/22/91
Company:

RELINQUISHED BY: 2.
Signature: Time:
Printed Name: Date:
Company:

RELINQUISHED BY: 3.
Signature: Time:
Printed Name: Date:
Company:

RECEIVED BY: 1.
Signature: DL Thomas Time: 4:40
Printed Name: DL Thomas Date: 1-23-91
Company:

RECEIVED BY: 2.
Signature: Time:
Printed Name: Date:
Company:

RECEIVED BY: (LAB) 3.
Signature: Time:
Printed Name: Date:
Company: Analytical Technologies, Inc.

A s: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 nsacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGIN

10 SD

AnalyticalTechnologies,Inc.

560 Naches Avenue SW, Suite 101 Renton, WA 98055 (206)228-8335

Chain of Custody

DATE 1/28/91 PAGE 1 OF 1

LABORATORY NUMBER: 101314

PROJECT MANAGER: Donna McKinney
COMPANY: ATI - Renton
ADDRESS:
PHONE: (206)228-8335 SAMPLED BY:

SAMPLE DISPOSAL INSTRUCTIONS

☒ ~~ATI Disposal @ $5.00 each~~ [✓ ☒ Return]

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA *	8310 HPLC PNA, Silica (*)	8080 Pesticides & PCB's	PCB's ONLY *	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)			NUMBER OF CONTAINERS
9101-167-3	1/22/91		Sed.	01					X	X		X																			1
-16	1/22/91		↓	02						X		X																			1

PROJECT INFORMATION

PROJECT NUMBER: 9101-167
PROJECT NAME: Geoengineers
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☐ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	2
COC SEALS/INTACT? Y/N/NA	NA
RECEIVED GOOD COND./COLD	Y
RECEIVED VIA:	FX

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS: will fax % Moi - 8310 data due 2/5
Normal $ ($250)

* extract + Return ASAP

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: D McKinney Time: 2:00	Signature: Time:	Signature: Time:
Printed Name: D. McKinney Date: 1/21/91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: Time:	Signature: Time:	Signature: Time: 0935
Printed Name: Date:	Printed Name: Date:	Printed Name: Kaido Date: 1-29-91
Company:	Company:	Analytical Technologies, Inc.

AT' s: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 nsacola (904)474-1001 **DISTRIBUTION:** White, Canary - ATI • Pink - ORIGIN/

APPENDIX

ToxScan Inc.

42 Hangar Way
Watsonville, CA 95076

(408) 724-4522

FAX (408) 724-3188

Analytical Technologies, Inc.
560 Naches Avenue SW, Suite 101
Renton, WA 98055

February 22, 1991
REVISED REPORT: August 25, 1919,
to include QA/QC

Attn: Donna McKinney

MATERIAL: Water samples received January 29, 1991
ANALYSIS COMPLETED: February 21, 1991
IDENTIFICATION: Geo engineers
TOXSCAN NUMBER: T-7197
REPORT: Quantitative chemical analysis is as follows, expressed as nanograms per liter (parts per trillion) as received:

Sample ID	Monobutyltin	Dibutyltin	Tributyltin	Tetrabutyltin	Tripropyl Tin Surrogate
9101-167-1	ND	ND	ND	ND	53
9101-167-2	ND	ND	9.2	ND	45
9101-167-15	82	90	310	160	100
9101-167-1 -Spike recovery	66%	78%	86%	75%	59

ND = None detected

Detection limit = 1 part per trillion

Philip D. Carpenter
Laboratory Director

.10XSCAN

T 71.7

AnalyticalTechnologies,Inc.
560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 1/28/91 PAGE 1 OF 1

LABORATORY NUMBER: T-7197

PROJECT MANAGER: Donna McKinney
COMPANY: ATI - Renton
ADDRESS:
PHONE: (206) 228-8335 SAMPLED BY:

SAMPLE DISPOSAL INSTRUCTIONS

☐ ATI Disposal @ $5.00 each [✓ ☒ Return]

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)	Tributyl Tin		NUMBER OF CONTAINERS
9101-167-1	1/22/91		water	-01																									✓		1
-2				-02																									✓		1
↓ -15 *	↓		↓	-03																									✓		1

-01 1L amb glass bottle
-02 ↓
-03 ~50-70 ml clear glass bottle

C - 210

PROJECT INFORMATION

PROJECT NUMBER: 9101-167
PROJECT NAME: Geoengineers
PURCHASE ORDER NUMBER: 02208
ONGOING PROJECT? YES ☐ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS 3
COC SEALS/INTACT? Y/N/NA
RECEIVED GOOD COND./COLD
RECEIVED VIA:

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☐ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)
SPECIAL INSTRUCTIONS: data due ASAP!

* interstitial H₂O Please include project # on invoice.

RELINQUISHED BY: 1.
Signature: D McKinney Time: 12:00
Printed Name: D. MCKINNEY Date: 1/28/91
Company: ATI

RELINQUISHED BY: 2.
Signature: Time:
Printed Name: Date:
Company:

RELINQUISHED BY: 3.
Signature: Time:
Printed Name: Date:
Company:

RECEIVED BY: 1.
Signature: K. Wanna Time: 10 a.m.
Printed Name: K. Wanna Date: 1/29/91
Company: TCI

RECEIVED BY: 2.
Signature: Time:
Printed Name: Date:
Company:

RECEIVED BY: (LAB) 3.
Signature: Time:
Printed Name: Date:
Company: Analytical Technologies, Inc.

RINSEATE SAMPLS 10A
10C

SEDIMENT SAMPLES 9A-9B

AnalyticalTechnologies,Inc. 560 Naches Avenue, S.W., Suite 101, Renton, WA 98055, (206) 228-8335

ATI I.D. # 9101-148

GeoEngineers

FEB 19 1991

Routing PGW ☒ ☐
☐ ☐ ☐
File 1299-003-B04

February 18, 1991

GeoEngineers, Inc.
8410 154th Avenue N.E.
Redmond, WA 98052

Attention : Paul Werner

Project Number : 1299-003-B04

Project Name : Unimar, Inc.

On January 22, 1991, Analytical Technologies, Inc., received two water and two sediment samples for analysis. The samples were analyzed with EPA methodology or equivalent methods as specified in the attached analytical schedule. The results, sample cross reference, and quality control data are enclosed.

This is a partial report as it does not contain the TCLP Metals results. These results will follow in approximately 60 days.

Donna M. McKinney
Donna M. McKinney
Project Manager

Frederick W. Grothkopp
Frederick W. Grothkopp
Technical Manager

FWG/elf

SAMPLE CROSS REFERENCE SHEET

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ATI #	CLIENT DESCRIPTION	DATE SAMPLED	MATRIX
9101-148-1	VAN VEEN-RINSATE 10C	01/21/91	WATER
9101-148-2	SHELBY RINSATE 10A	01/21/91	WATER
9101-148-3	9A 0-0.3'	01/21/91	SEDIMENT
9101-148-4	9B 0.3-2.5'	01/21/91	SEDIMENT

----- TOTALS -----

MATRIX	# SAMPLES
WATER	2
SEDIMENT	2

ATI STANDARD DISPOSAL PRACTICE

The samples from this project will be disposed of in thirty (30) days from the date of this report. If an extended storage period is required, please contact our sample control department before the scheduled disposal date.

ANALYTICAL SCHEDULE

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ANALYSIS	TECHNIQUE	REFERENCE	LAB
SEMI-VOLATILE COMPOUNDS	GCMS	EPA 8270	R
POLYCHLORINATED BIPHENYLS (PCBs)	GC/ECD	EPA 8080	R
POLYNUCLEAR AROMATIC HYDROCARBONS	HPLC/UV	EPA 8310	SD
ARSENIC	AA/GF	EPA 7060	R
BARIUM	AA/F	EPA 7080	R
CADMIUM	AA/GF	EPA 7131	R
CADMIUM	AA/F	EPA 7130	R
CHROMIUM	AA/F	EPA 7190	R
COPPER	AA/F	EPA 7210	R
LEAD	AA/GF	EPA 7421	R
LEAD	AA/F	EPA 7420	R
MERCURY	AA/COLD VAPOR	EPA 7470	R
MERCURY	AA/COLD VAPOR	EPA 7471	R
NICKEL	AA/F	EPA 7520	R
SELENIUM	AA/GF	EPA 7740	R
SILVER	AA/F	EPA 7760	R
ZINC	AA/F	EPA 7950	R
PETROLEUM HYDROCARBONS	IR	EPA 418.1	R
MOISTURE	GRAVIMETRIC	METHOD 7-2.2	R

R = ATI - Renton
SD = ATI - San Diego
T = ATI - Tempe

PNR = ATI - Pensacola
FC = ATI - Fort Collins
SUB = Subcontract

ATI I.D. # 9101-148

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SOIL
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 01/23/91
DATE ANALYZED : 01/31/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<0.17
PHENOL	<0.17
ANILINE	<0.17
BIS(2-CHLOROETHYL)ETHER	<0.17
2-CHLOROPHENOL	<0.17
1,3-DICHLOROBENZENE	<0.17
1,4-DICHLOROBENZENE	<0.17
BENZYL ALCOHOL	<0.17
1,2-DICHLOROBENZENE	<0.17
2-METHYLPHENOL	<0.17
BIS(2-CHLOROISOPROPYL)ETHER	<0.17
4-METHYLPHENOL	<0.17
N-NITROSO-DI-N-PROPYLAMINE	<0.17
HEXACHLOROETHANE	<0.17
NITROBENZENE	<0.17
ISOPHORONE	<0.17
2-NITROPHENOL	<0.17
2,4-DIMETHYLPHENOL	<0.17
BENZOIC ACID	<0.85
BIS(2-CHLOROETHOXY)METHANE	<0.17
2,4-DICHLOROPHENOL	<0.17
1,2,4-TRICHLOROBENZENE	<0.17
NAPHTHALENE	<0.17
4-CHLOROANILINE	<0.17
HEXACHLOROBUTADIENE	<0.17
4-CHLORO-3-METHYLPHENOL	<0.17
2-METHYLNAPHTHALENE	<0.17
HEXACHLOROCYCLOPENTADIENE	<0.17
2,4,6-TRICHLOROPHENOL	<0.17
2,4,5-TRICHLOROPHENOL	<0.85
2-CHLORONAPHTHALENE	<0.17
2-NITROANILINE	<0.85
DIMETHYLPHTHALATE	<0.17
ACENAPHTHYLENE	<0.17
3-NITROANILINE	<0.85
ACENAPHTHENE	<0.17
2,4-DINITROPHENOL	<0.85
4-NITROPHENOL	<0.85

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ATI I.D. # 9101-148

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/23/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 01/31/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
DIBENZOFURAN	<0.17
2,4-DINITROTOLUENE	<0.17
2,6-DINITROTOLUENE	<0.17
DIETHYLPHTHALATE	<0.17
4-CHLOROPHENYL-PHENYLETHER	<0.17
FLUORENE	<0.17
4-NITROANILINE	<0.85
4,6-DINITRO-2-METHYLPHENOL	<0.85
N-NITROSODIPHENYLAMINE	<0.17
4-BROMOPHENYL-PHENYLETHER	<0.17
HEXACHLOROBENZENE	<0.17
PENTACHLOROPHENOL	<0.85
PHENANTHRENE	<0.17
ANTHRACENE	<0.17
DI-N-BUTYLPHTHALATE	<0.17
FLUORANTHENE	<0.17
BENZIDINE	<1.7
PYRENE	<0.17
BUTYLBENZYLPHTHALATE	<0.17
3,3-DICHLOROBENZIDINE	<0.34
BENZO(a)ANTHRACENE	<0.17
BIS(2-ETHYLHEXYL)PHTHALATE	<0.17
CHRYSENE	<0.17
DI-N-OCTYLPHTHALATE	<0.17
BENZO(b)FLUORANTHENE	<0.17
BENZO(k)FLUORANTHENE	<0.17
BENZO(a)PYRENE	<0.17
INDENO(1,2,3-cd)PYRENE	<0.17
DIBENZ(a,h,)ANTHRACENE	<0.17
BENZO(g,h,i)PERYLENE	<0.17

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	63
2-FLUOROBIPHENYL	66
TERPHENYL-d14	62
PHENOL-d6	77
2-FLUOROPHENOL	61
2,4,6-TRIBROMOPHENOL	60

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/23/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 01/31/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
UNKNOWN	238	0.20
UNKNOWN	302	1.5

ATI I.D. # 9101-148-3

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 9A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/21/91
DATE RECEIVED : 01/22/91
DATE EXTRACTED : 01/23/91
DATE ANALYZED : 01/31/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<0.68
PHENOL	<0.68
ANILINE	<0.68
BIS(2-CHLOROETHYL)ETHER	<0.68
2-CHLOROPHENOL	<0.68
<0.68	
HEXACHLOROETHANE	<0.68
NITROBENZENE	<0.68
ISOPHORONE	<0.68
2-NITROPHENOL	<0.68
2,4-DIMETHYLPHENOL	<0.68
BENZOIC ACID	<3.4
BIS(2-CHLOROETHOXY)METHANE	<0.68
2,4-DICHLOROPHENOL	<0.68
1,2,4-TRICHLOROBENZENE	<0.68
NAPHTHALENE	<0.68
4-CHLOROANILINE	<0.68
HEXACHLOROBUTADIENE	<0.68
4-CHLORO-3-METHYLPHENOL	<0.68
2-METHYLNAPHTHALENE	<0.68
HEXACHLOROCYCLOPENTADIENE	<0.68
2,4,6-TRICHLOROPHENOL	<0.68
2,4,5-TRICHLOROPHENOL	<3.4
2-CHLORONAPHTHALENE	<0.68
2-NITROANILINE	<3.4
DIMETHYLPHTHALATE	<0.68
ACENAPHTHYLENE	<0.68
3-NITROANILINE	<3.4
ACENAPHTHENE	<0.68
2,4-DINITROPHENOL	<3.4
4-NITROPHENOL	<3.4

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ATI I.D. # 9101-148-3

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/21/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/22/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/23/91
CLIENT I.D.	: 9A 0-0.3′	DATE ANALYZED	: 01/31/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
DIBENZOFURAN	<0.68
2,4-DINITROTOLUENE	<0.68
2,6-DINITROTOLUENE	<0.68
DIETHYLPHTHALATE	<0.68
4-CHLOROPHENYL-PHENYLETHER	<0.68
FLUORENE	<0.68
4-NITROANILINE	<3.4
4,6-DINITRO-2-METHYLPHENOL	<3.4
N-NITROSODIPHENYLAMINE	<0.68
4-BROMOPHENYL-PHENYLETHER	<0.68
HEXACHLOROBENZENE	<0.68
PENTACHLOROPHENOL	<3.4
PHENANTHRENE	<0.68
ANTHRACENE	<0.68
DI-N-BUTYLPHTHALATE	<0.68
FLUORANTHENE	<0.68
BENZIDINE	<6.8
PYRENE	<0.68
BUTYLBENZYLPHTHALATE	<0.68
3,3-DICHLOROBENZIDINE	<1.4
BENZO(a)ANTHRACENE	<0.68
BIS(2-ETHYLHEXYL)PHTHALATE	<0.68
CHRYSENE	<0.68
DI-N-OCTYLPHTHALATE	<0.68
BENZO(b)FLUORANTHENE	<0.68
BENZO(k)FLUORANTHENE	<0.68
BENZO(a)PYRENE	<0.68
INDENO(1,2,3-cd)PYRENE	<0.68
DIBENZ(a,h,)ANTHRACENE	<0.68
BENZO(g,h,i)PERYLENE	<0.68

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	64
2-FLUOROBIPHENYL	67
TERPHENYL-d14	62
PHENOL-d6	80
2-FLUOROPHENOL	64
2,4,6-TRIBROMOPHENOL	65

ATI I.D. # 9101-148-3

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/21/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/22/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/23/91
CLIENT I.D.	: 9A 0-0.3'	DATE ANALYZED	: 01/31/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION	
UNKNOWN NITROGENATED	161	1.2	
UNKNOWN	305	15	B
OXYGENATED HYDROCARBON	1409	1.3	
UNKNOWN	1826	0.67	

B = Also found in blank.

SEMI-VOLATILE ORGANIC QUALITY CONTROL DATA

CLIENT	: GEOENGINEERS, INC.		SAMPLE I.D.	:	BLANK SPIKE
PROJECT #	: 1299-003-B04		DATE EXTRACTED	:	01/23/91
PROJECT NAME	: UNIMAR, INC.		DATE ANALYZED	:	01/31/91
EPA METHOD	: 8270		MATRIX	:	SOIL
			UNITS	:	mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
1,2,4-TRICHLOROBENZENE	<0.17	3.33	2.15	65	2.21	66	2
ACENAPHTHENE	<0.17	3.33	1.95	58	2.02	61	4
2,4-DINITROTOLUENE	<0.17	3.33	2.29	69	2.39	72	5
PYRENE	<0.17	3.33	2.06	62	2.37	71	14
N-NITROSO-DI-N-PROPYLAMINE	<0.17	3.33	2.38	71	2.63	79	10
1,4-DICHLOROBENZENE	<0.17	3.33	2.20	66	2.24	67	2
PENTACHLOROPHENOL	<0.85	13.34	8.04	61	7.87	59	2
PHENOL	<0.17	6.67	3.79	57	3.83	57	1
2-CHLOROPHENOL	<0.17	6.67	3.98	60	3.97	60	0
4-CHLORO-3-METHYLPHENOL	<0.17	6.67	3.46	52	3.27	49	6
4-NITROPHENOL	<0.85	13.34	11.9	90	12.6	94	5

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/23/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/03/91
SAMPLE MATRIX	: WATER	UNITS	: ug/L
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 1

COMPOUND	RESULT
PCB 1016	<1.0
PCB 1221	<1.0
PCB 1232	<1.0
PCB 1242	<1.0
PCB 1248	<1.0
PCB 1254	<1.0
PCB 1260	<1.0

ATI I.D. # 9101-148-1

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/21/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/22/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/23/91
CLIENT I.D.	: VAN VEEN-RINSATE 10C	DATE ANALYZED	: 02/03/91
SAMPLE MATRIX	: WATER	UNITS	: ug/L
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 1

COMPOUND	RESULT
PCB 1016	<1.0
PCB 1221	<1.0
PCB 1232	<1.0
PCB 1242	<1.0
PCB 1248	<1.0
PCB 1254	<1.0
PCB 1260	<1.0

ATI I.D. # 9101-148-2

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/21/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/22/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/23/91
CLIENT I.D.	: SHELBY RINSATE 10A	DATE ANALYZED	: 02/03/91
SAMPLE MATRIX	: WATER	UNITS	: ug/L
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 1

COMPOUND	RESULT
PCB 1016	<1.0
PCB 1221	<1.0
PCB 1232	<1.0
PCB 1242	<1.0
PCB 1248	<1.0
PCB 1254	<1.0
PCB 1260	<1.0

POLYCHLORINATED BIPHENYLS (PCB)
QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
EPA METHOD : 8080 (PCB)

SAMPLE ID : 9101-148-2
DATE EXTRACTED : 01/23/91
DATE ANALYZED : 02/03/91
MATRIX : WATER
UNITS : ug/L

COMPOUND	SAMPLE RESULT	CONC SPIKED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % RECOVERY	RPD
PCB 1260	<1.0	9.3	9.03	97	9.83	106	8

$$\% \text{ Recovery} = \frac{(\text{Spike Sample result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

POLYCHLORINATED BIPHENYLS (PCB)
QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
EPA METHOD : 8080 (PCB)

SAMPLE ID : BLANK SPIKE
DATE EXTRACTED : 01/23/91
DATE ANALYZED : 02/03/91
MATRIX : WATER
UNITS : ug/L

COMPOUND	SAMPLE RESULT	CONC SPIKED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % RECOVERY	RPD
PCB 1260	<1.0	10	9.77	98	N/A	N/A	N/A

$$\% \text{ Recovery} = \frac{(\text{Spike Sample result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-148

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/23/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/03/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
PCB 1016	<0.033
PCB 1221	<0.033
PCB 1232	<0.033
PCB 1242	<0.033
PCB 1248	<0.033
PCB 1254	<0.033
PCB 1260	<0.033

ATI I.D. # 9101-148-3

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 9A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8080 (PCB)
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/21/91
DATE RECEIVED : 01/22/91
DATE EXTRACTED : 01/23/91
DATE ANALYZED : 02/03/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
PCB 1016	<0.13
PCB 1221	<0.13
PCB 1232	<0.13
PCB 1242	<0.13
PCB 1248	<0.13
PCB 1254	<0.13
PCB 1260	<0.13

ATI I.D. # 9101-148-4

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/21/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/22/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/23/91
CLIENT I.D.	: 9B 0.3-2.5'	DATE ANALYZED	: 02/03/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
PCB 1016	<0.058
PCB 1221	<0.058
PCB 1232	<0.058
PCB 1242	<0.058
PCB 1248	<0.058
PCB 1254	<0.058
PCB 1260	<0.058

ATI I.D. # 9101-148

POLYCHLORINATED BIPHENYLS (PCB)
QUALITY CONTROL

CLIENT	: GEOENGINEERS, INC.		SAMPLE ID	:	9101-148-4
PROJECT #	: 1299-003-B04		DATE EXTRACTED	:	01/23/91
PROJECT NAME	: UNIMAR, INC.		DATE ANALYZED	:	02/03/91
EPA METHOD	: 8080 (PCB)		MATRIX	:	SEDIMENT
			UNITS	:	mg/Kg

COMPOUND	SAMPLE RESULT	CONC SPIKED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % RECOVERY	RPD
PCB 1260	<0.058	0.58	0.586	101	0.553	95	6

$$\% \text{ Recovery} = \frac{(\text{Spike Sample result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-148

POLYCHLORINATED BIPHENYLS (PCB)
QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
EPA METHOD : 8080 (PCB)

SAMPLE ID : BLANK SPIKE
DATE EXTRACTED : 01/23/91
DATE ANALYZED : 02/03/91
MATRIX : SOIL
UNITS : mg/Kg

COMPOUND	SAMPLE RESULT	CONC SPIKED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % RECOVERY	RPD
PCB 1260	<0.033	0.33	0.376	114	0.346	105	8

% Recovery = (Spike Sample result - Sample Result) / Spike Concentration X 100

RPD (Relative % Difference) = (Spiked Sample Result - Duplicate Spike Sample Result) / Average of Spiked Sample X 100

POLYNUCLEAR AROMATICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : WATER
EPA METHOD : 8310

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 01/23/91
DATE ANALYZED : 02/07/91
UNITS : ug/L
DILUTION FACTOR : 1

COMPOUND	RESULT
NAPHTHALENE	<0.50
ACENAPHTHYLENE	<1.0
ACENAPHTHENE	<1.0
FLUORENE	<0.10
PHENANTHRENE	<0.05
ANTHRACENE	<0.05
FLUORANTHENE	<0.10
PYRENE	<0.10
BENZO(a)ANTHRACENE	<0.10
CHRYSENE	<0.10
BENZO(b)FLUORANTHENE	<0.10
BENZO(k)FLUORANTHENE	<0.10
BENZO(a)PYRENE	<0.10
DIBENZ(a,h)ANTHRACENE	<0.20
BENZO(g,h,i)PERYLENE	<0.10
INDENO(1,2,3-cd)PYRENE	<0.10

ATI I.D. # 9101-148-1

POLYNUCLEAR AROMATICS ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/21/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/22/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/23/91
CLIENT I.D.	: VAN VEEN-RINSATE 10C	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: WATER	UNITS	: ug/L
EPA METHOD	: 8310	DILUTION FACTOR	: 1

COMPOUND	RESULT
NAPHTHALENE	<0.50
ACENAPHTHYLENE	<1.0
ACENAPHTHENE	<1.0
FLUORENE	<0.10
PHENANTHRENE	<0.05
ANTHRACENE	<0.05
FLUORANTHENE	<0.10
PYRENE	<0.10
BENZO(a)ANTHRACENE	<0.10
CHRYSENE	<0.10
BENZO(b)FLUORANTHENE	<0.10
BENZO(k)FLUORANTHENE	<0.10
BENZO(a)PYRENE	<0.10
DIBENZ(a,h)ANTHRACENE	<0.20
BENZO(g,h,i)PERYLENE	<0.10
INDENO(1,2,3-cd)PYRENE	<0.10

ATI I.D. # 9101-148-2

POLYNUCLEAR AROMATICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : SHELBY RINSATE 10A
SAMPLE MATRIX : WATER
EPA METHOD : 8310

DATE SAMPLED : 01/21/91
DATE RECEIVED : 01/22/91
DATE EXTRACTED : 01/23/91
DATE ANALYZED : 02/08/91
UNITS : ug/L
DILUTION FACTOR : 1

COMPOUND	RESULT
NAPHTHALENE	<0.50
ACENAPHTHYLENE	<1.0
ACENAPHTHENE	<1.0
FLUORENE	<0.10
PHENANTHRENE	<0.05
ANTHRACENE	<0.05
FLUORANTHENE	<0.10
PYRENE	<0.10
BENZO(a)ANTHRACENE	<0.10
CHRYSENE	<0.10
BENZO(b)FLUORANTHENE	<0.10
BENZO(k)FLUORANTHENE	<0.10
BENZO(a)PYRENE	<0.10
DIBENZ(a,h)ANTHRACENE	<0.20
BENZO(g,h,i)PERYLENE	<0.10
INDENO(1,2,3-cd)PYRENE	<0.10

POLYNUCLEAR AROMATICS
QUALITY CONTROL DATA

CLIENT	: GEOENGINEERS, INC.		SAMPLE I.D.	:	9101-148-1
PROJECT #	: 1299-003-B04		DATE EXTRACTED	:	01/23/91
PROJECT NAME	: UNIMAR, INC.		DATE ANALYZED	:	02/07/91
EPA METHOD	: 8310		MATRIX	:	WATER
			UNITS	:	ug/L

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
ACENAPHTHYLENE	<1.0	87.4	64	73	46	53	33*
PHENANTHRENE	<0.05	8.84	6.3	71	6.5	74	3
PYRENE	<0.10	8.46	6.6	78	5.9	70	11
BENZO(k)FLUORANTHENE	<0.10	6.84	6.0	88	5.9	86	2
DIBENZ(a,h)ANTHRACENE	<0.20	9.46	7.5	79	7.1	75	6

* Out of limits.

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

POLYNUCLEAR AROMATICS
QUALITY CONTROL DATA

CLIENT	: GEOENGINEERS, INC.		SAMPLE I.D.	: BLANK SPIKE
PROJECT #	: 1299-003-B04		DATE EXTRACTED	: 01/23/91
PROJECT NAME	: UNIMAR, INC.		DATE ANALYZED	: 02/07/91
EPA METHOD	: 8310		MATRIX	: WATER
			UNITS	: ug/L

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
ACENAPHTHYLENE	<1.0	87.4	47	54	N/A	N/A	N/A
PHENANTHRENE	<0.05	8.84	7.1	80	N/A	N/A	N/A
PYRENE	<0.10	8.46	6.9	82	N/A	N/A	N/A
BENZO(k)FLUORANTHENE	<0.10	6.84	6.6	96	N/A	N/A	N/A
DIBENZ(a,h)ANTHRACENE	<0.20	9.46	7.4	78	N/A	N/A	N/A

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : WATER

ELEMENT	DATE PREPARED	DATE ANALYZED
ARSENIC	01/23/91	02/08/91
BARIUM	01/23/91	01/28/91
CADMIUM	01/23/91	02/12/91
CHROMIUM	01/23/91	02/08/91
COPPER	01/23/91	01/23/91
LEAD	01/23/91	02/12/91
MERCURY	01/23/91	01/29/91
NICKEL	01/23/91	02/07/91
SELENIUM	01/23/91	02/08/91
SILVER	01/23/91	01/24/91
ZINC	01/23/91	02/05/91

METALS RESULTS

CLIENT	: GEOENGINEERS, INC.	MATRIX	: WATER
PROJECT #	: 1299-003-B04		
PROJECT NAME	: UNIMAR, INC.	UNITS	: mg/L

PARAMETER	VEN VEEN-RINSATE 10C -1	SHELBY RINSATE 10A -2	REAGENT BLANK
ARSENIC	<0.005	<0.005	<0.005
BARIUM	0.08	<0.06	<0.06
CADMIUM	<0.0003	<0.0003	<0.0003
CHROMIUM	<0.02	<0.02	<0.02
COPPER	0.03	<0.02	<0.02
LEAD	0.045	<0.005	<0.005
MERCURY	<0.0005	<0.0005	<0.0005
NICKEL	<0.03	<0.03	<0.03
SELENIUM	<0.005	<0.005	<0.005
SILVER	<0.02	<0.02	<0.02
ZINC	0.02	<0.01	<0.01

METALS QUALITY CONTROL

CLIENT	: GEOENGINEERS, INC.	MATRIX	: WATER
PROJECT #	: 1299-003-B04		
PROJECT NAME	: UNIMAR, INC.	UNITS	: mg/L

COMPOUND	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED SAMPLE	SPIKE CONC	% REC
ARSENIC	9101-174-2	<0.005	<0.005	0	0.016	0.025	64
ARSENIC	BLANK SPIKE	N/A	N/A	N/A	0.024	0.025	96
BARIUM	9101-142-1	0.29	0.27	7	10.08	10.00	98
CADMIUM	9101-174-2	0.0012	0.0009	29	0.0019	0.0010	70
CHROMIUM	9101-191-13	<0.02	<0.02	0	1.98	2.50	79
COPPER	9101-148-2	<0.02	<0.02	0	1.0	1.0	100
LEAD	9101-184-3	0.113	0.112	1	**	**	**
LEAD	BLANK SPIKE	N/A	N/A	N/A	0.033	0.025	132
MERCURY	9101-202-2	<0.0005	<0.0005	0	0.0030	0.0020	150*
MERCURY	BLANK SPIKE	N/A	N/A	N/A	0.0029	0.0020	145
NICKEL	9101-174-2	<0.03	<0.03	0	2.33	2.50	93
SELENIUM	9101-174-2	<0.005	<0.005	0	0.021	0.025	84
SILVER	9101-142-1	<0.02	<0.02	0	1.0	1.0	100
ZINC	9101-174-2	0.60	0.55	9	2.76	2.50	86

* Out of limits.

** Due to the necessary dilution of the sample, result was not attainable.

$$\% \text{ Recovery} = \frac{\text{(Spike Sample Result - Sample Result)}}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{\text{(Sample Result - Duplicate Result)}}{\text{Average Result}} \times 100$$

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : SEDIMENT

ELEMENT	DATE PREPARED	DATE ANALYZED
ARSENIC	01/28/91	02/06/91
BARIUM	01/28/91	02/07/91
CADMIUM	01/28/91	01/31/91
CHROMIUM	01/28/91	02/07/91
COPPER	01/28/91	02/07/91
LEAD	01/28/91	01/31/91
MERCURY	01/28/91	02/04/91
NICKEL	01/28/91	02/07/91
SELENIUM	01/28/91	02/06/91
SILVER	01/28/91	01/31/91
ZINC	01/28/91	02/05/91

ATI I.D. # 9101-148

METALS RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

PARAMETER	9A 0-0.3′ -3	9B 0.3-2.5′ -4	BLANK
ARSENIC	8.9	2.3	<0.5
BARIUM	110	51	<6
CADMIUM	<2	<1	<1
CHROMIUM	39	32	<2
COPPER	38	15	<2
LEAD	68	<10	<10
MERCURY	<0.40	<0.15	<0.15
NICKEL	41	31	<3
SELENIUM	<1.0	<0.5	<0.5
SILVER	<2	<2	<2
ZINC	120	44	<1

ATI I.D. # 9101-148

METALS QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT
UNITS : mg/Kg

COMPOUND	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED SAMPLE	SPIKE CONC	% REC
ARSENIC	9101-143-3	1.1	1.2	9	2.0	1.3	69
BARIUM	9101-143-3	30	34	12	422	525	75
CADMIUM	9101-143-3	<1	<1	0	24	26	92
CHROMIUM	9101-143-3	19	19	0	114	131	72
COPPER	9101-143-3	11	11	0	127	131	88
LEAD	9101-143-3	<10	<10	0	230	260	88
MERCURY	9101-143-3	<0.15	<0.15	0	0.55	0.46	120
NICKEL	9101-143-3	43	44	2	164	131	92
SELENIUM	9101-143-3	<0.5	<0.5	0	0.7	1.3	54
SILVER	9101-143-3	<2	<2	0	24	26	92
ZINC	9101-143-3	26	27	4	142	131	89

$$\%\ \text{Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

ATI I.D. # 9101-148

GENERAL CHEMISTRY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : WATER

ANALYSIS	DATE PREPARED	DATE ANALYZED
PETROLEUM HYDROCARBONS	01/23/91	01/23/91

ATI I.D. # 9101-148

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER
UNITS : mg/L

ATI I.D. #	CLIENT I.D.	PETROLEUM HYDROCARBONS
9101-148-1	VAN VEEN-RINSATE 10C	<1
9101-148-2	SHELBY RINSATE 10A	<1
REAGENT BLANK	-	<1

GENERAL CHEMISTRY QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

PARAMETER	UNITS	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED RESULT	SPIKE ADDED	% REC
PETROLEUM HYDROCARBONS	mg/L	9101-148-2	<1	<1	0	4.86	10.0	48

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

GENERAL CHEMISTRY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : SEDIMENT

ANALYSIS	DATE PREPARED	DATE ANALYZED
PETROLEUM HYDROCARBONS	01/24/91	01/24/91
MOISTURE	-	01/23/91

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

ATI I.D. #	CLIENT I.D.	PETROLEUM HYDROCARBONS
9101-148-3	9A 0-0.3'	65
9101-148-4	9B 0.3-2.5'	13
REAGENT BLANK	-	<5

ATI I.D. # 9101-148

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : %

ATI I.D. #	CLIENT I.D.	MOISTURE
9101-148-3	9A 0-0.3'	75
9101-148-4	9B 0.3-2.5'	43

GENERAL CHEMISTRY QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

PARAMETER	UNITS	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED RESULT	SPIKE ADDED	% REC
PETROLEUM HYDROCARBONS	mg/Kg	9101-132-6	10,900	8,660	23	**	**	**
PETROLEUM HYDROCARBONS	mg/Kg	BLANK SPIKE	N/A	N/A	N/A	209	219	95
MOISTURE	%	9101-148-4	43	41	5	N/A	N/A	N/A

** Due to the necessary dilution of the sample, result was not attainable.

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

AnalyticalTechnologies,Inc.

560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 1/21/91 PAGE 1 OF 1

LABORATORY NUMBER: 9101-148

PROJECT MANAGER: PAUL WERNER
COMPANY: GEO ENGINEERS, INC
ADDRESS: 140 Th AVE NE Suite 105
Bellevue, WA 98005
PHONE: 746-5200 SAMPLED BY: P. WERNER

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

	SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	* Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8) **			NUMBER OF CONTAINERS	
10 C	VAN VEEN - RINSEATE	1/21/91	1245	WATER	1						X		X				X							X								8	✓
10 A	Shelby RINSEATE	1/21/91	1345	WATER	2						X		X				X							X								8	✓
9 A	Bottom SEDIMENT 9A	1/21/91	1540	Sed.	3					X			X				X							X					X			1	✓ 1L
9 B	↓ 9B	1/21/91	1610	Sed.	4								X				X							X								1	✓ L

PROJECT INFORMATION

PROJECT NUMBER: 1299-003-B04
PROJECT NAME: UNIMAK, INC
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	18
COC SEALS/INTACT? Y/N/NA	4/4
RECEIVED GOOD COND./COLD	4/4
RECEIVED VIA: Courier	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK

GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:
SEALS 01299-01 PLW * EPTOX TOTALS + Cu, Ni, Zn
01299-02 PLW ** TCLP METALS + Cu, Ni, Zn

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 2:45	Signature: Time:	Signature: Time:
Printed Name: PAUL WERNER Date: 1/21/91	Printed Name: Date:	Printed Name: Date:
Company: GEO ENG	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: Time:	Signature: Time:	Signature: Time: 1000A
Printed Name: Date:	Printed Name: Date:	Printed Name: V. PENNICK Date: 1/22/91
Company:	Company:	Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINATOR

NO. 2044

SD

Analytical Technologies, Inc.

560 Naches Avenue SW, Suite 101 Renton, WA 98055 (206)228-8335

Chain of Custody

DATE 1/22/91 PAGE 1 OF 1

LABORATORY NUMBER: 101240

PROJECT MANAGER: Donna McKinney

COMPANY: ATI

ADDRESS:

PHONE: (206)228-8335 SAMPLED BY:

SAMPLE DISPOSAL INSTRUCTIONS

☐ ATI Disposal @ $5.00 each [✓ ☒ Return]

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA *	8310 HPLC PNA + silica gel	8080 Pesticides & PCB's	PCB's ONLY *	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)			NUMBER OF CONTAINERS
9101-148-1	1/21/91		H2O	01						✓		✓																			6
2	↓		↓	02						✓		✓																			6
3	↓		Sed	03					✓			✓																			1
4	↓		↓	04								✓																			1

PROJECT INFORMATION

PROJECT NUMBER: 9101-148

PROJECT NAME: Geoengineers

PURCHASE ORDER NUMBER:

ONGOING PROJECT? YES ☐ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	14
COC SEALS/INTACT? Y/N/NA	NA
RECEIVED GOOD COND./COLD	N
RECEIVED VIA:	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☐ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK

GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:

* extract only + return extract ASAP, please.

data due 2/1/91 on 8310 = $250.00 (client's price)

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: D McKinney Time: 14	Signature: Time:	Signature: Time:
Printed Name: D McKinney Date:	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: Time:	Signature: Time:	Signature: Time: 930
Printed Name: Date:	Printed Name: Date:	Printed Name: Date: 1/23/9
Company:	Company:	Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)22 35 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - C GINATOR

Analytical**Technologies**,Inc. To Renton

5550 Morehouse Drive • San Diego, CA 92121-1709

Chain of Custody

9101-198

DATE 1-25-91 PAGE 1 OF 3

PROJECT MANAGER: Carrie Hussinger

COMPANY: ATI SD

ADDRESS:

BILL TO: same

COMPANY:

ADDRESS:

SAMPLERS: (Signature)

PHONE NUMBER ( ) -

Recommended Quantity and Preservative (Provide triple volume on QC Samples)

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	Petroleum Hydrocarbons 418.1 1L (H_2 SO_4)/100g	Oil and Grease 413.2 1L (H_2 SO_4)/100g	Gasoline (MOD 8015/DOHS) 4 oz (HCl)/50g	Diesel (MOD 8015/DOHS) 4 oz (HCl)/50g	Gasoline/BTXE (MOD 8015/8020) 4 oz (HCl)/50g	(C6-C25,MOD 8015) 4 oz (HCl)/50g	BTXE (8020) 2X40ml (HCl)/50g	Chlorinated Hydrocarbons (8010) 2X40ml (HCl)/50g	Aromatic Hydrocarbons (8020) 2X40ml (HCl)/50g	Chlorinated/Aromatic Hydrocarbons (8010/8020) 2X40ml (HCl)/50g	Organic Pb 500ml/50g	Pesticides/PCB (8080) 1L/50g	Base/NEU/Acid Cmpds GC/MS (8270) 1L/100g	Volatile Cmpds GC/MS (8240) 2X40ml (HCl)/100g	Polynuclear Aromatic (8310) 1L/100g	CCR Metals 500ml/100g	Priority Pollutant Metals 500ml/100g	Number of Containers
RB 1-23	1/23	Soil	extract	-1													X					1
101240-03				-2													X					1
SPK 1-23				-3													X					1
SOUD				-4													X					1
05.ext.w.51.01			SPK	-5													X					1
05.ext.w.49.02				-6													X					1
RB 1-23		H_2O	extract	-7												X						1
101240-01				-8												X						1
-02				-9												X						1

PROJECT INFORMATION

PROJECT NUMBER: 101240

PROJECT NAME:

PURCHASE ORDER NUMBER:

VIA: Fed EX

TAT: ☐ 24HR ☐ 48 HRS ☐ 1 WK ☐ 2 WKS

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	21
CHAIN OF CUSTODY SEALS Y/N/NA	y
INTACT? Y/N/NA	y
RECEIVED GOOD COND./COLD	y
LAB NUMBER	

SAMPLE DISPOSAL INSTRUCTIONS

☐ ATI Disposal @ $5.00 each ☐ Return ☐ Pickup (will call)

Comments: due 2/1 Fedx Seal

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Time: 1130	Signature: Time:	Signature: Time:
Printed Name: Michelle Holloway Date: 1/23/91	Printed Name: Date:	Printed Name: Date:
Company: ATI SD	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: D.L. Thomas Time: 10:35	Signature: Time:	Signature: Time:
Printed Name: D.L. Thomas Date: 1-25-91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Analytical Technologies, Inc.

ATI LABORATORIES: San Diego (619) 458-9141 • Phoenix (602) 438-1530 • Seattle (206) 22

DISTRIBUTION: White, Canary - ANALYTICAL TECHNOLOGIES, INC. • Pink - ORIGINA

AnalyticalTechnologies,Inc.
5550 Morehouse Drive • San Diego, CA 92121-1709

To Renton

Chain of Custody

4101-178

DATE 1-25-91 PAGE 2 OF 3

PROJECT MANAGER: Carrie Hassinger
COMPANY: ATI
ADDRESS:

BILL TO: same
COMPANY:
ADDRESS:

SAMPLERS: (Signature) ______ PHONE NUMBER ( ) .

Recommended Quantity and Preservative (Provide triple volume on QC Samples)

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	Petroleum Hydrocarbons 418.1 1L (H_2 SO_4)/100g	Oil and Grease 413.2 1L (H_2 SO_4)/100g	Gasoline (MOD 8015/DOHS) 4 oz (HCl)/50g	Diesel (MOD 8015/DOHS) 4 oz (HCl)/50g	Gasoline/BTXE (MOD 8015/8020) 4 oz (HCl)/50g	(C6-C25,MOD 8015) 4 oz (HCl)/50g	BTXE (8020) 2X40ml (HCl)/50g	Chlorinated Hydrocarbons (8010) 2X40ml (HCl)/50g	Aromatic Hydrocarbons (8020) 2X40ml (HCl)/50g	Chlorinated/Aromatic Hydrocarbons (8010/8020) 2X40ml (HCl)/50g	Organic Pb 500ml/50g	Pesticides/PCB (8080) 1L/50g	Base/NEU/Acid Cmpds GC/MS (8270) 1L/100g	Volatile Cmpds GC/MS (8240) 2X40ml (HCl)/100g	Polynuclear Aromatic (8310) 1L/100g	CCR Metals 500ml/100g	Priority Pollutant Metals 500ml/100g	Number of Containers
101240-02ms		H2O	extract	-10												X						1
↓ -02msd		↓	↓	-11												X						1
SPK 1-23			↓	-12												X						1
05-ext-w.35.02			SPC	-13												X						1
05-ext-w.37.02		↓	↓	-14												X						1
RR 1-23		Soil	extract	-15												X						1
101240-03		↓	↓	-16												X						1
↓ -04				-17												X						1
-04MS		↓	↓	-18												X						1

PROJECT INFORMATION

PROJECT NUMBER: 101240
PROJECT NAME:
PURCHASE ORDER NUMBER:
VIA: Fed Ex
TAT: ☐ 24HR ☐ 48 HRS ☐ 1 WK ☐ 2 WKS

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	
CHAIN OF CUSTODY SEALS Y/N/NA	y
INTACT? Y/N/NA	y
RECEIVED GOOD COND./COLD	y
LAB NUMBER	

SAMPLE DISPOSAL INSTRUCTIONS

☐ ATI Disposal @ $5.00 each ☐ Return ☐ Pickup (will call)

Comments: due 2/1

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Time: 1230	Signature: Time:	Signature: Time:
Printed Name: Michael Hallgren Date: 1/25/91	Printed Name: Date:	Printed Name: Date:
Company: ATI SD	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: DK Thomas Time: 10:30	Signature: Time:	Signature: Time:
Printed Name: DK Thomas Date: 1-26-91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Analytical Technologies, Inc.

LABORATORIES: San Diego (619) 458-9141 • Phoenix (602) 438-1530 • Seattle (206) 228-

DISTRIBUTION: White, Canary - ANALYTICAL TECHNOLOGIES, INC. • Pink - ORIGINAT

11/30/91   9104-144

Analytical Technologies, Inc.

560 Naches Avenue SW, Suite 101 Renton, WA 98055 (206)228-8335

Chain of Custody

DATE 1/29/91 PAGE 1 OF 1

LABORATORY NUMBER: 9101-238

PROJECT MANAGER: Paul Werner

COMPANY: Geo Engineers

ADDRESS: Bellevue

PHONE: 746-5200   SAMPLED BY: P. C. Werner

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each   ☐ Return

ANALYSIS REQUEST

	SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	~~TOX 9020~~ Total Metals	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	TCLP ONLY					TBT		NUMBER OF CONTAINERS
																									8080 Pesticide (4)	8240 ZH-EXT	8270	8150 Herbicides (2)	Metals (8)			
8	5A 0-0.3' Bottom Sediment	1/27/91	10:09	Soil	-1					X			X				X				X								X			1
9	6A 0-0.3' "	1/30/91	11:20	↓	-2					X			X				X				X								X			1
10	11 Duplicate	↓	11:25		-3					X			X				X				X								X			1
	6B 0.3-5.0'		14:30	↓	-4						X						X				X											1
	VAN VEEN RINSEATE		1215	WATER																												
	10D		↓	↓	-5						X		X				X				X											8
	Shelby Rinseate		1300																													
	10B		↓		-6						X		X				X				X											8
	BETA RINSEATE 10F		1445	↓	-7.																X									X		2
	Bottom SEDIMENT 8			Soil																												
11	~~6A~~ 7A PGW 1/31/91		1415	↓	-8					X			X				X				X								X			1
	~~6B~~ 7B "	↓	1535	↓	-9						X						X				X											1

C - 254

PROJECT INFORMATION

PROJECT NUMBER: 1299-003-R04

PROJECT NAME: Unimar, Inc

PURCHASE ORDER NUMBER:

ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	24
COC SEALS/INTACT? Y/N/NA	y
RECEIVED GOOD COND./COLD	y
RECEIVED VIA: Courier	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS   (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK

GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:
Sem # 01299-009 PGW
01299-010 PGW
01299-011 PGW
TCLP + Cu Ni Zn

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 18:00	Signature: Time:	Signature: Time:
Printed Name: Paul Werner Date: 1/30/91	Printed Name: Date:	Printed Name: Date:
Company: GEI	Company:	Company:
RECEIVED BY: 1.	**RECEIVED BY: 2.**	**RECEIVED BY: (LAB) 3.**
Signature: D L Thomas Time: 11:05	Signature: Time:	Signature: Time:
Printed Name: DL Thomas Date: 1-31-91	Printed Name: Date:	Printed Name: Date:
Company:	Company:	Analytical Technologies, Inc.

Analytical Technologies, Inc. To Renton
5550 Morehouse Drive • San Diego, CA 92121-1709

Chain of Custody

9101-198 1-25-91

DATE________ PAGE 3 OF 3

PROJECT MANAGER: Carrie Lassinger
COMPANY: ATI-SD
ADDRESS:

BILL TO: same
COMPANY:
ADDRESS:

SAMPLERS: (Signature) ______________ PHONE NUMBER ( ) -

Recommended Quantity and Preservative (Provide triple volume on QC Samples)

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	Petroleum Hydrocarbons 418.1 1L (H_2SO_4)/100g	Oil and Grease 413.2 1L (H_2SO_4)/100g	Gasoline (MOD 8015/DOHS) 4 oz (HCl)/50g	Diesel (MOD 8015/DOHS) 4 oz (HCl)/50g	Gasoline/BTXE (MOD 8015/8020) 4 oz (HCl)/50g	(C6-C25,MOD 8015) 4 oz (HCl)/50g	BTXE (8020) 2X40ml (HCl)/50g	Chlorinated Hydrocarbons (8010) 2X40ml (HCl)/50g	Aromatic Hydrocarbons (8020) 2X40ml (HCl)/50g	Chlorinated/Aromatic Hydrocarbons (8010/8020) 2X40ml (HCl)/50g	Organic Pb 500ml/50g	Pesticides/PCB (8080) 1L/50g	Base/NEU/Acid Cmpds GC/MS (8270) 1L/100g	Volatile Cmpds GC/MS (8240) 2X40ml (HCl)/100g	Polynuclear Aromatic (8310) 1L/100g	CCR Metals 500ml/100g	Priority Pollutant Metals 500ml/100g	Number of Containers
101240-04/MSD	1/23		Soil ex	-19												X						1
SPK 1-23				-20												X						1
SDUP 1-23				-21												X						1

PROJECT INFORMATION

PROJECT NUMBER: 101240
PROJECT NAME:
PURCHASE ORDER NUMBER:
VIA: Fed Ex
TAT: ☐ 24HR ☐ 48 HRS ☐ 1 WK ☐ 2 WKS

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	
CHAIN OF CUSTODY SEALS Y/N/NA	y
INTACT? Y/N/NA	y
RECEIVED GOOD COND./COLD	y
LAB NUMBER	

SAMPLE DISPOSAL INSTRUCTIONS

☐ ATI Disposal @ $5.00 each ☐ Return ☐ Pickup (will call)

Comments: due 2/1

RELINQUISHED BY: 1.
Signature: Time: 1330
Printed Name: Michel Hillyer Date: 1/25/91
Company: ATI-SD

RELINQUISHED BY: 2.
Signature: Time:
Printed Name: Date:
Company:

RELINQUISHED BY: 3.
Signature: Time:
Printed Name: Date:
Company:

RECEIVED BY: 1.
Signature: DK Thomas Time: 10:30
Printed Name: DW Thomas Date: 1-26-91
Company: ATI

RECEIVED BY: 2.
Signature: Time:
Printed Name: Date:
Company:

RECEIVED BY: (LAB) 3.
Signature: Time:
Printed Name: Date:
Analytical Technologies, Inc.

DISTRIBUTION: White, Canary - ANALYTICAL TECHNOLOGIES, INC. • Pink - ORIGINAT

TLCP SAMPLES 1A, 2B, 3A, 4A, 5A, 6A, 7A, 8A, 8C, 9A, 11 DUPLICATE

APPENDIX C

Analytical**Technologies,**Inc.
560 Naches Avenue, S.W., Suite 101, Renton, WA 98055, (206) 228-8335

ATI I.D. # 9101-203

GeoEngineers

MAR 06 1991

Routing ☐ ☐ ☐ ☐ ☐
File 1299-003-B04

March 4, 1991

GeoEngineers, Inc.
8410 154th Avenue N.E.
Redmond, WA 98052

Attention : Paul Werner

Project Number : 1299-003-B04

Project Name : Unimar

On January 28, 1991, Analytical Technologies, Inc., received nine sediment samples for analysis. The samples were analyzed with EPA methodology or equivalent methods as specified in the attached analytical schedule. The results, sample cross reference, and quality control data are enclosed.

This is a partial report as it does not contain the TCLP results. These results will follow in approximately 60 days.

Donna M. McKinney
Donna M. McKinney
Project Manager

Donna M. McKinney for FWG
Frederick W. Grothkopp
Technical Manager

FWG/tc

ATI I.D. # 9101-203

SAMPLE CROSS REFERENCE SHEET

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR

ATI #	CLIENT DESCRIPTION	DATE SAMPLED	MATRIX
9101-203-1	1B 0.3'-1.0'	01/25/91	SEDIMENT
9101-203-2	1C 1.0'-1.5'	01/25/91	SEDIMENT
9101-203-3	1D 1.5'-2.0'	01/25/91	SEDIMENT
9101-203-4	1E 2.0'-2.5'	01/25/91	SEDIMENT
9101-203-5	1F 2.5'-3.0'	01/25/91	SEDIMENT
9101-203-6	1G 3.0'-3.5'	01/25/91	SEDIMENT
9101-203-7	1H 3.5'-4.0'	01/25/91	SEDIMENT
9101-203-8	3A 0-0.3'	01/25/91	SEDIMENT
9101-203-9	3B 0.3-5.0'	01/25/91	SEDIMENT

----- TOTALS -----

MATRIX	# SAMPLES
SEDIMENT	9

ATI STANDARD DISPOSAL PRACTICE

The samples from this project will be disposed of in thirty (30) days from the date of this report. If an extended storage period is required, please contact our sample control department before the scheduled disposal date.

ATI I.D. # 9101-203

ANALYTICAL SCHEDULE

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR

ANALYSIS	TECHNIQUE	REFERENCE	LAB
SEMI-VOLATILE COMPOUNDS	GCMS	EPA 8270	R
POLYCHLORINATED BIPHENYLS (PCBs)	GC/ECD	EPA 8080	R
POLYNUCLEAR AROMATIC HYDROCARBONS	HPLC/UV	EPA 8310	R
ARSENIC	AA/GF	EPA 7060	R
BARIUM	AA/F	EPA 7080	R
CADMIUM	AA/F	EPA 7130	R
CHROMIUM	AA/F	EPA 7190	R
COPPER	AA/F	EPA 7210	R
LEAD	AA/F	EPA 7420	R
MERCURY	AA/COLD VAPOR	EPA 7471	R
NICKEL	AA/F	EPA 7520	R
SELENIUM	AA/GF	EPA 7740	R
SILVER	AA/F	EPA 7760	R
ZINC	AA/F	EPA 7950	R
PETROLEUM HYDROCARBONS	IR	EPA 418.1	R
MOISTURE	GRAVIMETRIC	METHOD 7-2.2	R

R = ATI - Renton
SD = ATI - San Diego
T = ATI - Tempe
PNR = ATI - Pensacola
FC = ATI - Fort Collins
SUB = Subcontract

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<0.17
PHENOL	<0.17
ANILINE	<0.17
BIS(2-CHLOROETHYL)ETHER	<0.17
2-CHLOROPHENOL	<0.17
1,3-DICHLOROBENZENE	<0.17
1,4-DICHLOROBENZENE	<0.17
BENZYL ALCOHOL	<0.17
1,2-DICHLOROBENZENE	<0.17
2-METHYLPHENOL	<0.17
BIS(2-CHLOROISOPROPYL)ETHER	<0.17
4-METHYLPHENOL	<0.17
N-NITROSO-DI-N-PROPYLAMINE	<0.17
HEXACHLOROETHANE	<0.17
NITROBENZENE	<0.17
ISOPHORONE	<0.17
2-NITROPHENOL	<0.17
2,4-DIMETHYLPHENOL	<0.17
BENZOIC ACID	<0.85
BIS(2-CHLOROETHOXY)METHANE	<0.17
2,4-DICHLOROPHENOL	<0.17
1,2,4-TRICHLOROBENZENE	<0.17
NAPHTHALENE	<0.17
4-CHLOROANILINE	<0.17
HEXACHLOROBUTADIENE	<0.17
4-CHLORO-3-METHYLPHENOL	<0.17
2-METHYLNAPHTHALENE	<0.17
HEXACHLOROCYCLOPENTADIENE	<0.17
2,4,6-TRICHLOROPHENOL	<0.17
2,4,5-TRICHLOROPHENOL	<0.85
2-CHLORONAPHTHALENE	<0.17
2-NITROANILINE	<0.85
DIMETHYLPHTHALATE	<0.17
ACENAPHTHYLENE	<0.17
3-NITROANILINE	<0.85
ACENAPHTHENE	<0.17
2,4-DINITROPHENOL	<0.85
4-NITROPHENOL	<0.85

CONTINUED NEXT PAGE

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SOIL
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
DIBENZOFURAN	<0.17
2,4-DINITROTOLUENE	<0.17
2,6-DINITROTOLUENE	<0.17
DIETHYLPHTHALATE	<0.17
4-CHLOROPHENYL-PHENYLETHER	<0.17
FLUORENE	<0.17
4-NITROANILINE	<0.85
4,6-DINITRO-2-METHYLPHENOL	<0.85
N-NITROSODIPHENYLAMINE	<0.17
4-BROMOPHENYL-PHENYLETHER	<0.17
HEXACHLOROBENZENE	<0.17
PENTACHLOROPHENOL	<0.85
PHENANTHRENE	<0.17
ANTHRACENE	<0.17
DI-N-BUTYLPHTHALATE	<0.17
FLUORANTHENE	<0.17
BENZIDINE	<1.7
PYRENE	<0.17
BUTYLBENZYLPHTHALATE	<0.17
3,3-DICHLOROBENZIDINE	<0.34
BENZO(a)ANTHRACENE	<0.17
BIS(2-ETHYLHEXYL)PHTHALATE	<0.17
CHRYSENE	<0.17
DI-N-OCTYLPHTHALATE	<0.17
BENZO(b)FLUORANTHENE	<0.17
BENZO(k)FLUORANTHENE	<0.17
BENZO(a)PYRENE	<0.17
INDENO(1,2,3-cd)PYRENE	<0.17
DIBENZ(a,h)ANTHRACENE	<0.17
BENZO(g,h,i)PERYLENE	<0.17

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	73
2-FLUOROBIPHENYL	82
TERPHENYL-d14	76
PHENOL-d6	86
2-FLUOROPHENOL	66
2,4,6-TRIBROMOPHENOL	87

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
HYDROCARBON	1883	0.63
HYDROCARBON	1944	1.0
HYDROCARBON	2013	1.3
HYDROCARBON	2094	1.4
HYDROCARBON	2306	1.0

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR
CLIENT I.D. : 3A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/25/91
DATE RECEIVED : 01/28/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 5

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<3.5
PHENOL	<3.5
ANILINE	<3.5
BIS(2-CHLOROETHYL)ETHER	<3.5
2-CHLOROPHENOL	<3.5
1,3-DICHLOROBENZENE	<3.5
1,4-DICHLOROBENZENE	<3.5
BENZYL ALCOHOL	<3.5
1,2-DICHLOROBENZENE	<3.5
2-METHYLPHENOL	<3.5
BIS(2-CHLOROISOPROPYL)ETHER	<3.5
4-METHYLPHENOL	<3.5
N-NITROSO-DI-N-PROPYLAMINE	<3.5
HEXACHLOROETHANE	<3.5
NITROBENZENE	<3.5
ISOPHORONE	<3.5
2-NITROPHENOL	<3.5
2,4-DIMETHYLPHENOL	<3.5
BENZOIC ACID	<18
BIS(2-CHLOROETHOXY)METHANE	<3.5
2,4-DICHLOROPHENOL	<3.5
1,2,4-TRICHLOROBENZENE	<3.5
NAPHTHALENE	<3.5
4-CHLOROANILINE	<3.5
HEXACHLOROBUTADIENE	<3.5
4-CHLORO-3-METHYLPHENOL	<3.5
2-METHYLNAPHTHALENE	<3.5
HEXACHLOROCYCLOPENTADIENE	<3.5
2,4,6-TRICHLOROPHENOL	<3.5
2,4,5-TRICHLOROPHENOL	<18
2-CHLORONAPHTHALENE	<3.5
2-NITROANILINE	<18
DIMETHYLPHTHALATE	<3.5
ACENAPHTHYLENE	<3.5
3-NITROANILINE	<18
ACENAPHTHENE	<3.5
2,4-DINITROPHENOL	<18
4-NITROPHENOL	<18

CONTINUED NEXT PAGE

ATI I.D. # 9101-203-8

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT	: GEOENGINEERS, INC.		DATE SAMPLED	: 01/25/91
PROJECT #	: 1299-003-B04		DATE RECEIVED	: 01/28/91
PROJECT NAME	: UNIMAR		DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: 3A 0-0.3'		DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SEDIMENT		UNITS	: mg/Kg
EPA METHOD	: 8270		DILUTION FACTOR	: 5

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT	
DIBENZOFURAN	<3.5	
2,4-DINITROTOLUENE	<3.5	
2,6-DINITROTOLUENE	<3.5	
DIETHYLPHTHALATE	<3.5	
4-CHLOROPHENYL-PHENYLETHER	<3.5	
FLUORENE	<3.5	
4-NITROANILINE	<18	
4,6-DINITRO-2-METHYLPHENOL	<18	
N-NITROSODIPHENYLAMINE	<3.5	
4-BROMOPHENYL-PHENYLETHER	<3.5	
HEXACHLOROBENZENE	<3.5	
PENTACHLOROPHENOL	<18	
PHENANTHRENE	3.5	J
ANTHRACENE	3.4	J
DI-N-BUTYLPHTHALATE	<3.5	
FLUORANTHENE	5.3	
BENZIDINE	<35	
PYRENE	4.1	
BUTYLBENZYLPHTHALATE	<3.5	
3,3-DICHLOROBENZIDINE	<7.1	
BENZO(a)ANTHRACENE	2.0	J
BIS(2-ETHYLHEXYL)PHTHALATE	1.8	J
CHRYSENE	2.1	J
DI-N-OCTYLPHTHALATE	<3.5	
BENZO(b)FLUORANTHENE	2.7	J
BENZO(k)FLUORANTHENE	<3.5	
BENZO(a)PYRENE	2.6	J
INDENO(1,2,3-cd)PYRENE	<3.5	
DIBENZ(a,h)ANTHRACENE	<3.5	
BENZO(g,h,i)PERYLENE	<3.5	

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	60
2-FLUOROBIPHENYL	107
TERPHENYL-d14	98
PHENOL-d6	90
2-FLUOROPHENOL	63
2,4,6-TRIBROMOPHENOL	113

J = Estimated value.

ATI I.D. # 9101-203-8

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR
CLIENT I.D. : 3A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/25/91
DATE RECEIVED : 01/28/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 5

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
HYDROCARBON	1779	9.0
HYDROCARBON	1830	10
HYDROCARBON	1884	17 B
HYDROCARBON	1944	12 B
HYDROCARBON	2014	16 B

B = Also found in blank.

SEMI-VOLATILE ORGANIC
QUALITY CONTROL DATA

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR
EPA METHOD : 8270

SAMPLE I.D. : BLANK SPIKE
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
MATRIX : SOIL
UNITS : mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
1,2,4-TRICHLOROBENZENE	<0.17	3.33	2.39	72	2.86	86	18
ACENAPHTHENE	<0.17	3.33	2.19	66	2.42	73	10
2,4-DINITROTOLUENE	<0.17	3.33	2.46	74	2.56	77	4
PYRENE	<0.17	3.33	2.34	70	2.62	78	11
N-NITROSO-DI-N-PROPYLAMINE	<0.17	3.33	2.26	68	2.68	81	17
1,4-DICHLOROBENZENE	<0.17	3.33	2.23	67	2.65	79	17
PENTACHLOROPHENOL	<0.85	13.3	11.6	87	11.3	85	3
PHENOL	<0.17	6.67	3.98	60	4.79	72	18
2-CHLOROPHENOL	<0.17	6.67	4.03	60	4.90	73	19
4-CHLORO-3-METHYLPHENOL	<0.17	6.67	4.44	67	4.99	75	12
4-NITROPHENOL	<0.85	13.3	11.5	86	11.6	87	1

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

SEDIMENT SAMPLES

1B-1H

3A-3B

ATI I.D. # 9101-203

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SOIL
EPA METHOD : 8080 (PCB)
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/04/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
PCB 1016	<0.033
PCB 1221	<0.033
PCB 1232	<0.033
PCB 1242	<0.033
PCB 1248	<0.033
PCB 1254	<0.033
PCB 1260	<0.033

ATI I.D. # 9101-203-2

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR
CLIENT I.D. : 1C 1.0'-1.5'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8080 (PCB)
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/25/91
DATE RECEIVED : 01/28/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/04/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
PCB 1016	<0.063
PCB 1221	<0.063
PCB 1232	<0.063
PCB 1242	<0.063
PCB 1248	<0.063
PCB 1254	<0.063
PCB 1260	<0.063

ATI I.D. # 9101-203-8

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR
CLIENT I.D. : 3A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8080 (PCB)
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/25/91
DATE RECEIVED : 01/28/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/04/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
PCB 1016	<0.14
PCB 1221	<0.14
PCB 1232	<0.14
PCB 1242	<0.14
PCB 1248	<0.14
PCB 1254	<0.14
PCB 1260	<0.14

POLYCHLORINATED BIPHENYLS (PCB)
QUALITY CONTROL

CLIENT	: GEOENGINEERS, INC.	SAMPLE ID	: BLANK SPIKE
PROJECT #	: 1299-003-B04	DATE EXTRACTED	: 01/30/91
PROJECT NAME	: UNIMAR	DATE ANALYZED	: 02/04/91
EPA METHOD	: 8080 (PCB)	MATRIX	: SOIL
		UNITS	: mg/Kg

COMPOUND	SAMPLE RESULT	CONC SPIKED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % RECOVERY	RPD
PCB 1260	<0.033	0.33	0.345	104	0.351	106	2

% Recovery = (Spike Sample result - Sample Result) / Spike Concentration X 100

RPD (Relative % Difference) = (Spiked Sample Result - Duplicate Spike Sample Result) / Average of Spiked Sample X 100

ATI I.D. # 9101-203

POLYNUCLEAR AROMATICS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SOIL
EPA METHOD : 8310
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
NAPHTHALENE	<0.083
ACENAPHTHYLENE	<0.17
ACENAPHTHENE	<0.17
FLUORENE	<0.017
PHENANTHRENE	<0.0083
ANTHRACENE	<0.0083
FLUORANTHENE	<0.017
PYRENE	<0.017
BENZO(a)ANTHRACENE	<0.017
CHRYSENE	<0.017
BENZO(b)FLUORANTHENE	<0.017
BENZO(k)FLUORANTHENE	<0.017
BENZO(a)PYRENE	<0.017
DIBENZ(a,h)ANTHRACENE	<0.034
BENZO(g,h,i)PERYLENE	<0.017
INDENO(1,2,3-cd)PYRENE	<0.017

ATI I.D. # 9101-203-9

POLYNUCLEAR AROMATICS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/25/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/28/91
PROJECT NAME	: UNIMAR	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: 3B 0.3-5.0'	DATE ANALYZED	: 02/09/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8310	DILUTION FACTOR	: 10

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
NAPHTHALENE	2.4
ACENAPHTHYLENE	<5.0
ACENAPHTHENE	<5.0
FLUORENE	1.0
PHENANTHRENE	7.1
ANTHRACENE	2.4
FLUORANTHENE	18
PYRENE	15
BENZO(a)ANTHRACENE	5.9
CHRYSENE	7.4
BENZO(b)FLUORANTHENE	5.0
BENZO(k)FLUORANTHENE	2.7
BENZO(a)PYRENE	7.6
DIBENZ(a,h)ANTHRACENE	1.8
BENZO(g,h,i)PERYLENE	6.5
INDENO(1,2,3-cd)PYRENE	6.5

ATI I.D. # 9101-203

POLYNUCLEAR AROMATICS
QUALITY CONTROL DATA

CLIENT	: GEOENGINEERS, INC.		SAMPLE I.D.	: 101289-32
PROJECT #	: 1299-003-B04		DATE EXTRACTED	: 01/30/91
PROJECT NAME	: UNIMAR		DATE ANALYZED	: 02/08/91
EPA METHOD	: 8310		MATRIX	: SOIL
			UNITS	: mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
ACENAPHTHYLENE	<0.17	16.6	11	66	11	66	0
PHENANTHRENE	<0.034	1.77	1.4	79	1.5	85	7
PYRENE	<0.017	1.76	1.8	102	1.9	108	5
BENZO(k)FLUORANTHENE	0.0083	1.50	1.2	79	1.4	93	15

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

POLYNUCLEAR AROMATICS
QUALITY CONTROL DATA

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR
EPA METHOD : 8310

SAMPLE I.D. : BLANK SPIKE
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
MATRIX : SOIL
UNITS : mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
ACENAPHTHYLENE	<0.17	16.6	11	66	N/A	N/A	N/A
PHENANTHRENE	<0.034	1.77	1.33	73	N/A	N/A	N/A
PYRENE	<0.017	1.76	1.7	97	N/A	N/A	N/A
BENZO(k)FLUORANTHENE	<0.0083	1.58	1.3	82	N/A	N/A	N/A

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR
SAMPLE MATRIX : SEDIMENT

ELEMENT	DATE PREPARED	DATE ANALYZED
ARSENIC	02/13/91	02/26/91
BARIUM	02/13/91	02/26/91
CADMIUM	02/13/91	02/14/91
CADMIUM	02/13/91	02/26/91
CHROMIUM	02/13/91	02/26/91
COPPER	02/13/91	02/14/91
COPPER	02/13/91	02/26/91
LEAD	02/13/91	02/20/91
LEAD	02/13/91	02/26/91
MERCURY	02/22/91	02/22/91
NICKEL	02/13/91	02/26/91
SELENIUM	02/13/91	02/19/91
SILVER	02/13/91	02/26/91
ZINC	02/13/91	02/20/91
ZINC	02/13/91	02/26/91

ATI I.D. # 9101-203

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR

MATRIX : SEDIMENT

UNITS : mg/Kg

PARAMETER	1B 0.3' -1.0' -1	1C 1.0' -1.5' -2	1D 1.5' -2.0' -3	1E 2.0 -2.5' -4	1F 2.5 -3.0 -5
CADMIUM	2.9	<1	1.3	4.4	2.1
COPPER	2,300	69	34	2,600	1,400
LEAD	1,300	78	36	1,600	1,500
ZINC	2,600	160	94	4,700	2,100

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR

MATRIX : SEDIMENT

UNITS : mg/Kg

PARAMETER	1G 3.0 -3.5 -6	1H 3.5 -4.0 -7	3A 0 -0.3' -8	3B 0.3 -5.0' -9	REAGENT BLANK -
ARSENIC	-	-	240	64	<0.5
BARIUM	-	-	32	45	<1
CADMIUM	<1	<1	4.4	3.7	<1
CHROMIUM	-	-	18	18	<1
COPPER	38	96	230	90	3.7
LEAD	45	31	210	97	<2.5
MERCURY	-	-	<0.80	<0.80	<0.15
NICKEL	-	-	13	20	<1
SELENIUM	-	-	<0.5	<0.5	<0.5
SILVER	-	-	2	<2	<2
ZINC	150	94	660	225	2.8

METALS QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR

MATRIX : SEDIMENT/SOIL
UNITS : mg/Kg

COMPOUND	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED SAMPLE	SPIKE CONC	% REC
ARSENIC	9101-216-4	26	23	12	35	9.4	96
BARIUM	9101-216-4	110	110	0	1,910	1,880	96
CADMIUM	9101-203-7	<1	<1	0	24	30	80
CADMIUM	9101-216-4	8.2	8.0	2	92	94	89
CHROMIUM	9101-216-4	45	45	0	525	470	102
COPPER	9101-203-7	96	79	19	220	150	83
COPPER	9101-216-4	89	85	5	522	470	92
LEAD	9101-203-7	31	38	20	340	290	106
LEAD	9101-216-4	130	129	1	979	940	90
MERCURY	9101-216-4	1.05	<0.80	0	1.78	1.67	44*
MERCURY	BLANK SPIKE	N/A	N/A	N/A	0.44	0.50	88
NICKEL	9101-216-4	55	54	2	499	470	94
SELENIUM	9101-216-4	<1.0	<1.0	0	6.4	9.4	68
SILVER	9101-216-4	<2	<2	0	113	94	120
ZINC	9101-203-7	94	120	24*	310	150	144*
ZINC	9101-216-4	210	203	3	694	470	103

* Out of limits due to matrix interference.

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

GENERAL CHEMISTRY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR
SAMPLE MATRIX : SEDIMENT

ANALYSIS	DATE PREPARED	DATE ANALYZED
PETROLEUM HYDROCARBONS	02/04/91	02/04/91
MOISTURE	-	01/30/91

ATI I.D. # 9101-203

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR

MATRIX : SEDIMENT
UNITS : mg/Kg

ATI I.D. #	CLIENT I.D.	PETROLEUM HYDROCARBONS
9101-203-2	1C 1.0'-1.5'	510
9101-203-8	3A 0-0.3'	99
9101-203-9	3B 0.3-5.0'	230
REAGENT BLANK	-	<5

ATI I.D. # 9101-203

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR

MATRIX : SEDIMENT

UNITS : %

ATI I.D. #	CLIENT I.D.	MOISTURE
9101-203-1	1B 0.3'-1.0'	41
9101-203-2	1C 1.0'-1.5'	48
9101-203-3	1D 1.5'-2.0'	55
9101-203-4	1E 2.0'-2.5'	30
9101-203-5	1F 2.5'-3.0'	34
9101-203-6	1G 3.0'-3.5'	40
9101-203-7	1H 3.5'-4.0'	15
9101-203-8	3A 0-0.3'	76
9101-203-9	3B 0.3-5.0'	66

GENERAL CHEMISTRY QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR

SAMPLE MATRIX : SEDIMENT/SOIL

PARAMETER	UNITS	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED RESULT	SPIKE ADDED	% REC
PETROLEUM HYDROCARBONS	mg/Kg	9101-238-1	780	776	0	**	**	**
PETROLEUM HYDROCARBONS	mg/Kg	BLANK SPIKE	N/A	N/A	N/A	293	268	109
MOISTURE	%	9101-203-9	66	66	0	N/A	N/A	N/A

** Due to the necessary dilution of the sample, result was not attainable.

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \text{ X } 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \text{ X } 100$$

AnalyticalTechnologies,Inc.
560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 1/25/91 PAGE 1 OF 1

LABORATORY NUMBER: 9101-203

PROJECT MANAGER: PAUL WERNER
COMPANY: GEO ENGINEERS
ADDRESS: Bellevue WA
PHONE: 746-5200 SAMPLED BY: P.G. WERNER

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 ~~Pesticides~~ & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	~~EP TOX~~ TCLP Metals (8) EP EXT *	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)	METALS SCREEN **		NUMBER OF CONTAINERS
Bottom SEDIMENT																															
1B 0.3'-1.0'	1/25/91	1400	SEDIMENT	-1																									X		1
1C 1.0'-1.5'		1405		-2							X					X													X		1
1D 1.5'-2.0'		1410		-3																									X		1
1E 2.0-2.5		1419		-4																									X		1
1F 2.5-3.0		1421		-5																									X		1
1G 3.0-3.5		1425		-6																									X		1
1H 3.5-4.0'		1430		-7																									X		1
		1555	FW																												
3A 0-0.3'		~~1600~~		-8					X	~~X~~		X				X						X						X			1
3B 0.3-5.0		1600		-9						X	~~X~~					X						X									1

PROJECT INFORMATION

PROJECT NUMBER: 1299-003-B04
PROJECT NAME: Unimar
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	9
COC SEALS/INTACT? Y/N/NA	Y
RECEIVED GOOD COND./COLD	Y/NO
RECEIVED VIA:	FedX

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK

GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:
SEAL # 01299-007 FGW *TEPMET + Cu, Ni, Zn
**Cd, Cu, Pb, Zn
Totals
Not EPTOX ICE Not Frozen Samples warm

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 700	Signature: Time:	Signature: Time:
Printed Name: PAUL WERNER Date: 1/25/91	Printed Name: Date:	Printed Name: Date:
Company: GEI	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: D.L. Thomas Time: 1011	Signature: Time:	Signature: Time:
Printed Name: D.L. Thomas Date: 1-28-91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Company: Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINATOR

NO. [illegible]

TO SD

AnalyticalTechnologies,Inc.
560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 1/28/91 PAGE 1 OF 1

LABORATORY NUMBER: 101312

PROJECT MANAGER: Donna McKinney
COMPANY: ATI - Renton
ADDRESS:
PHONE: (206) 228-8335 SAMPLED BY:

SAMPLE DISPOSAL INSTRUCTIONS

☐ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA *	8310 HPLC PNA [illegible]	8080 Pesticides & PCB's	PCB's ONLY *	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)			NUMBER OF CONTAINERS
9101-203-2	1/25/91		Soil	01								X																			1
↓ -8	↓		↓	02					X			X																			1
↓ -9	↓		↓	03						X																					1

PROJECT INFORMATION

PROJECT NUMBER: 9101-203
PROJECT NAME: Geoengineers
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☐ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	3
COC SEALS/INTACT? Y/N/NA	N/A
RECEIVED GOOD COND./COLD	Y
RECEIVED VIA:	X

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☐ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)
SPECIAL INSTRUCTIONS: will fax % moi 8310 (8250") due 2/8/91

* extract + Return ASAP!

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: D McKinney Time: 12 00	Signature: Time:	Signature: Time:
Printed Name: D. McKinney Date: 1/28/91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: Time:	Signature: Time:	Signature: Time: 1935
Printed Name: Date:	Printed Name: Date:	Printed Name: [illegible] Rado Date: 1-29-91
Company:	Company:	Analytical Technologies, Inc.

I Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINATOR

WATER SAMPLE 2A

INTERSTIAL WATER 2C

SEDIMENT SAMPLES 1A-1C
2A-2J

AnalyticalTechnologies,Inc. 560 Naches Avenue, S.W., Suite 101, Renton, WA 98055, (206) 228-8335

ATI I.D. # 9101-191

GeoEngineers

MAR 01 1991

Routing ☐ ☐ ☐ ☐ ☐

File 1299-003-B4

February 28, 1991

GeoEngineers, Inc.
8410 154th Avenue N.E.
Redmond, WA 98052

Attention : Paul Werner

Project Number : 1299-003-B04

Project Name : Unimar, Inc.

On January 25, 1991, Analytical Technologies, Inc., received two water and 12 sediment samples for analysis. The samples were analyzed with EPA methodology or equivalent methods as specified in the attached analytical schedule. The results, sample cross reference, and quality control data are enclosed.

Please note that this is a partial report as it does not contain the TCLP Metals results. These results will follow in approximately 60 days.

Donna M. McKinney
Donna M. McKinney
Project Manager

Frederick W Grothkopp
Frederick W. Grothkopp
Technical Manager

FWG/elf

ATI I.D. # 9101-191

SAMPLE CROSS REFERENCE SHEET

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ATI #	CLIENT DESCRIPTION	DATE SAMPLED	MATRIX
9101-191-1	SAMPLE 2A @ 39'	01/24/91	WATER
9101-191-2	2B 0-0.3'	01/24/91	SEDIMENT
9101-191-3	2D 1.5-2.0'	01/24/91	SEDIMENT
9101-191-4	2E 2.0-2.5'	01/24/91	SEDIMENT
9101-191-5	2F 2.5-3.0'	01/24/91	SEDIMENT
9101-191-6	2G 3.0-3.5'	01/24/91	SEDIMENT
9101-191-7	2H 3.5-4.0'	01/24/91	SEDIMENT
9101-191-8	2I 4.0-4.5'	01/24/91	SEDIMENT
9101-191-9	2J 4.5-4.8'	01/24/91	SEDIMENT
9101-191-10	1A 0-0.3'	01/24/91	SEDIMENT
9101-191-11	1B 0.3-1.0'	01/23/91	SEDIMENT
9101-191-12	1C 1.0-1.3'	01/23/91	SEDIMENT
9101-191-13	2C (INTERSTIAL WATER)	01/24/91	WATER
9101-191-14	2C 0.3-1.5'	01/24/91	SEDIMENT

----- TOTALS -----

MATRIX	# SAMPLES
WATER	2
SEDIMENT	12

ATI STANDARD DISPOSAL PRACTICE

The samples from this project will be disposed of in thirty (30) days from the date of this report. If an extended storage period is required, please contact our sample control department before the scheduled disposal date.

ANALYTICAL SCHEDULE

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ANALYSIS	TECHNIQUE	REFERENCE	LAB
SEMI-VOLATILE COMPOUNDS	GCMS	EPA 8270	R
POLYCHLORINATED BIPHENYLS (PCBs)	GC/ECD	EPA 8080	R
POLYNUCLEAR AROMATIC HYDROCARBONS	HPLC/UV	EPA 8310	SD
TRIBUTYLTIN	GC/FPD	BATTELLE	SUB
ARSENIC	AA/GF	EPA 7060	R
BARIUM	AA/F	EPA 7080	R
CADMIUM	AA/F	EPA 7130	R
CADMIUM	AA/GF	EPA 7131	R
CHROMIUM	AA/F	EPA 7190	R
COPPER	AA/F	EPA 7210	R
LEAD	AA/F	EPA 7420	R
LEAD	AA/GF	EPA 7421	R
MERCURY	AA/COLD VAPOR	EPA 7470	R
MERCURY	AA/COLD VAPOR	EPA 7471	R
NICKEL	AA/F	EPA 7520	R
SELENIUM	AA/GF	EPA 7740	R
SILVER	AA/F	EPA 7760	R
ZINC	AA/F	EPA 7950	R
HARDNESS	TITRATION	EPA 130.2	R

CONTINUED NEXT PAGE

ANALYTICAL SCHEDULE
CONTINUED

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ANALYSIS	TECHNIQUE	REFERENCE	LAB
PETROLEUM HYDROCARBONS	IR	EPA 418.1	R
MOISTURE	GRAVIMETRIC	METHOD 7-2.2	R

R = ATI - Renton
SD = ATI - San Diego
T = ATI - Tempe
PNR = ATI - Pensacola
FC = ATI - Fort Collins
SUB = Subcontract

ATI I.D. # 9101-191

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<0.17
PHENOL	<0.17
ANILINE	<0.17
BIS(2-CHLOROETHYL)ETHER	<0.17
2-CHLOROPHENOL	<0.17
1,3-DICHLOROBENZENE	<0.17
1,4-DICHLOROBENZENE	<0.17
BENZYL ALCOHOL	<0.17
1,2-DICHLOROBENZENE	<0.17
2-METHYLPHENOL	<0.17
BIS(2-CHLOROISOPROPYL)ETHER	<0.17
4-METHYLPHENOL	<0.17
N-NITROSO-DI-N-PROPYLAMINE	<0.17
HEXACHLOROETHANE	<0.17
NITROBENZENE	<0.17
ISOPHORONE	<0.17
2-NITROPHENOL	<0.17
2,4-DIMETHYLPHENOL	<0.17
BENZOIC ACID	<0.85
BIS(2-CHLOROETHOXY)METHANE	<0.17
2,4-DICHLOROPHENOL	<0.17
1,2,4-TRICHLOROBENZENE	<0.17
NAPHTHALENE	<0.17
4-CHLOROANILINE	<0.17
HEXACHLOROBUTADIENE	<0.17
4-CHLORO-3-METHYLPHENOL	<0.17
2-METHYLNAPHTHALENE	<0.17
HEXACHLOROCYCLOPENTADIENE	<0.17
2,4,6-TRICHLOROPHENOL	<0.17
2,4,5-TRICHLOROPHENOL	<0.85
2-CHLORONAPHTHALENE	<0.17
2-NITROANILINE	<0.85
DIMETHYLPHTHALATE	<0.17
ACENAPHTHYLENE	<0.17
3-NITROANILINE	<0.85
ACENAPHTHENE	<0.17
2,4-DINITROPHENOL	<0.85
4-NITROPHENOL	<0.85

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ATI I.D. # 9101-191

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
DIBENZOFURAN	<0.17
2,4-DINITROTOLUENE	<0.17
2,6-DINITROTOLUENE	<0.17
DIETHYLPHTHALATE	<0.17
4-CHLOROPHENYL-PHENYLETHER	<0.17
FLUORENE	<0.17
4-NITROANILINE	<0.85
4,6-DINITRO-2-METHYLPHENOL	<0.85
N-NITROSODIPHENYLAMINE	<0.17
4-BROMOPHENYL-PHENYLETHER	<0.17
HEXACHLOROBENZENE	<0.17
PENTACHLOROPHENOL	<0.85
PHENANTHRENE	<0.17
ANTHRACENE	<0.17
DI-N-BUTYLPHTHALATE	<0.17
FLUORANTHENE	<0.17
BENZIDINE	<1.7
PYRENE	<0.17
BUTYLBENZYLPHTHALATE	<0.17
3,3-DICHLOROBENZIDINE	<0.34
BENZO(a)ANTHRACENE	<0.17
BIS(2-ETHYLHEXYL)PHTHALATE	<0.17
CHRYSENE	<0.17
DI-N-OCTYLPHTHALATE	<0.17
BENZO(b)FLUORANTHENE	<0.17
BENZO(k)FLUORANTHENE	<0.17
BENZO(a)PYRENE	<0.17
INDENO(1,2,3-cd)PYRENE	<0.17
DIBENZ(a,h)ANTHRACENE	<0.17
BENZO(g,h,i)PERYLENE	<0.17

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	73
2-FLUOROBIPHENYL	82
TERPHENYL-d14	76
PHENOL-d6	86
2-FLUOROPHENOL	66
2,4,6-TRIBROMOPHENOL	87

ATI I.D. # 9101-191

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
HYDROCARBON	1883	0.63
HYDROCARBON	1944	1.0
HYDROCARBON	2013	1.3
HYDROCARBON	2094	1.4
HYDROCARBON	2306	1.0

ATI I.D. # 9101-191-2

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT	GEOENGINEERS, INC.	DATE SAMPLED	01/24/91
PROJECT #	1299-003-B04	DATE RECEIVED	01/25/91
PROJECT NAME	UNIMAR, INC.	DATE EXTRACTED	01/30/91
CLIENT I.D.	2B 0-0.3'	DATE ANALYZED	02/08/91
SAMPLE MATRIX	SEDIMENT	UNITS	mg/Kg
EPA METHOD	8270	DILUTION FACTOR	5

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<2.4
PHENOL	<2.4
ANILINE	<2.4
BIS(2-CHLOROETHYL)ETHER	<2.4
2-CHLOROPHENOL	<2.4
1,3-DICHLOROBENZENE	<2.4
1,4-DICHLOROBENZENE	<2.4
BENZYL ALCOHOL	<2.4
1,2-DICHLOROBENZENE	<2.4
2-METHYLPHENOL	<2.4
BIS(2-CHLOROISOPROPYL)ETHER	<2.4
4-METHYLPHENOL	<2.4
N-NITROSO-DI-N-PROPYLAMINE	<2.4
HEXACHLOROETHANE	<2.4
NITROBENZENE	<2.4
ISOPHORONE	<2.4
2-NITROPHENOL	<2.4
2,4-DIMETHYLPHENOL	<2.4
BENZOIC ACID	<12
BIS(2-CHLOROETHOXY)METHANE	<2.4
2,4-DICHLOROPHENOL	<2.4
1,2,4-TRICHLOROBENZENE	<2.4
NAPHTHALENE	2.6
4-CHLOROANILINE	<2.4
HEXACHLOROBUTADIENE	<2.4
4-CHLORO-3-METHYLPHENOL	<2.4
2-METHYLNAPHTHALENE	6.9
HEXACHLOROCYCLOPENTADIENE	<2.4
2,4,6-TRICHLOROPHENOL	<2.4
2,4,5-TRICHLOROPHENOL	<12
2-CHLORONAPHTHALENE	<2.4
2-NITROANILINE	<12
DIMETHYLPHTHALATE	<2.4
ACENAPHTHYLENE	<2.4
3-NITROANILINE	<12
ACENAPHTHENE	18
2,4-DINITROPHENOL	<12
4-NITROPHENOL	<12

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ATI I.D. # 9101-191-2

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/24/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/25/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: 2B 0-0.3'	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 5

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT	
DIBENZOFURAN	14	
2,4-DINITROTOLUENE	<2.4	
2,6-DINITROTOLUENE	<2.4	
DIETHYLPHTHALATE	<2.4	
4-CHLOROPHENYL-PHENYLETHER	<2.4	
FLUORENE	20	
4-NITROANILINE	<12	
4,6-DINITRO-2-METHYLPHENOL	<12	
N-NITROSODIPHENYLAMINE	<2.4	
4-BROMOPHENYL-PHENYLETHER	<2.4	
HEXACHLOROBENZENE	<2.4	
PENTACHLOROPHENOL	<12	
PHENANTHRENE	53	
ANTHRACENE	13	
DI-N-BUTYLPHTHALATE	<2.4	
FLUORANTHENE	25	
BENZIDINE	<24	
PYRENE	17	
BUTYLBENZYLPHTHALATE	<2.4	
3,3-DICHLOROBENZIDINE	<4.7	
BENZO(a)ANTHRACENE	6.0	
BIS(2-ETHYLHEXYL)PHTHALATE	10	
CHRYSENE	6.8	
DI-N-OCTYLPHTHALATE	<2.4	
BENZO(b)FLUORANTHENE	6.6	
BENZO(k)FLUORANTHENE	1.9	J
BENZO(a)PYRENE	4.8	
INDENO(1,2,3-cd)PYRENE	1.9	J
DIBENZ(a,h)ANTHRACENE	<2.4	
BENZO(g,h,i)PERYLENE	2.1	J

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	51
2-FLUOROBIPHENYL	81
TERPHENYL-d14	75
PHENOL-d6	64
2-FLUOROPHENOL	40
2,4,6-TRIBROMOPHENOL	71

J = Estimated value.

METALS RESULTS

CLIENT	: GEOENGINEERS, INC.	MATRIX	: SEDIMENT
PROJECT #	: 1299-003-B04		
PROJECT NAME	: UNIMAR, INC.	UNITS	: mg/Kg

ATI I.D. #	CLIENT I.D.	SILVER	ZINC
9101-191-2	2B 0-0.3'	<2	13,000
9101-191-3	2D 1.5-2.0'	-	6,200
9101-191-4	2E 2.0-2.5'	-	5,700
9101-191-5	2F 2.5-3.0'	-	8,700
9101-191-6	2G 3.0-3.5'	-	6,500
9101-191-7	2H 3.5-4.0'	-	1,700
9101-191-8	2I 4.0-4.5'	-	130
9101-191-9	2J 4.5-4.8'	-	190
9101-191-10	1A 0-0.3'	4.2	10,000
9101-191-14	2C 0.3-1.5'	3.0	4,200
REAGENT BLANK	-	<2	<1

METALS QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT/SOIL

UNITS : mg/Kg

PARAMETER	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED SAMPLE	SPIKE CONC	% REC
ARSENIC	9101-191-12	23	22	4	**	**	**
ARSENIC	BLANK SPIKE	N/A	N/A	N/A	1.5	1.3	115
BARIUM	9101-191-12	54	51	6	820	500	150*
BARIUM	BLANK SPIKE	N/A	N/A	N/A	400	500	80
CADMIUM	9101-191-12	<1	<1	0	24	25	96
CHROMIUM	9101-197-12	20	20	0	95	101	74
COPPER	9101-191-12	44	46	4	133	126	71
LEAD	9101-191-12	180	160	12	540	252	142
MERCURY	9101-216-4	1.05	<0.80	0	1.78	1.67	44*
MERCURY	BLANK SPIKE	N/A	N/A	N/A	0.44	0.55	88
NICKEL	9101-191-12	18	18	0	119	126	80
SELENIUM	9101-191-12	<0.5	<0.5	0	0.7	1.3	54
SILVER	9101-191-12	<2	<2	0	25	25	100
ZINC	9101-191-12	150	130	14	318	126	133

* Out of limits due to matrix interference.

** Due to the necessary dilution of the sample, result was not attainable.

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

GENERAL CHEMISTRY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : WATER

ANALYSIS	DATE PREPARED	DATE ANALYZED
HARDNESS	02/08/91	02/08/91

ATI I.D. # 9101-191

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

ATI I.D.#	CLIENT I.D.	HARDNESS
9101-191-1	SAMPLE 2A @ 39′	40
REAGENT BLANK	-	<5

GENERAL CHEMISTRY QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

PARAMETER	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED RESULT	SPIKE ADDED	% REC
HARDNESS	9102-011-2	27	24	12	552	500	105

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

GENERAL CHEMISTRY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : SEDIMENT

ANALYSIS	DATE PREPARED	DATE ANALYZED
PETROLEUM HYDROCARBONS	01/28/91	01/28/91
MOISTURE	-	01/29/91

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

ATI I.D. #	CLIENT I.D.	PETROLEUM HYDROCARBONS
9101-191-10	1A 0-0.3'	1,600
9101-191-12	1C 1.0-1.3'	950
REAGENT BLANK	-	<5

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : %

ATI I.D. #	CLIENT I.D.	MOISTURE
9101-191-2	2B 0-0.3'	64
9101-191-3	2D 1.5-2.0'	68
9101-191-4	2E 2.0-2.5'	61
9101-191-5	2F 2.5-3.0'	69
9101-191-6	2G 3.0-3.5'	63
9101-191-7	2H 3.5-4.0'	68
9101-191-8	2I 4.0-4.5'	57
9101-191-9	2J 4.5-4.8'	77
9101-191-10	1A 0-0.3'	52
9101-191-14	2C 0.3-1.5'	62

GENERAL CHEMISTRY QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

PARAMETER	UNITS	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED RESULT	SPIKE ADDED	% REC
PETROLEUM HYDROCARBONS	mg/Kg	9101-167-16	70	75	7	348	248	12
MOISTURE	%	9101-191-14	62	62	0	N/A	N/A	N/A

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

AnalyticalTechnologies,Inc.

560 Naches Avenue SW, Suite 101 Renton, WA 98055 (206)228-8335

Chain of Custody

DATE 1/24/91 PAGE 1 OF 2

LABORATORY NUMBER: 9101-191

PROJECT MANAGER: PAUL WERNER

COMPANY: GEO ENGINEERS

ADDRESS:

PHONE: 746-5200

SAMPLED BY: P. G. WERNER

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

see -13,14

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	~~TOC 9060~~ METALS SCREEN*	TOX 9020	% Moisture	EP TOX Metals (8) ~~EP-EXT~~ **	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)	TBT	Hardness	NUMBER OF CONTAINERS
SAMPLE 2A @ 39'	1/24/90	1030	Water	1																		X							X	X	3
2B 0-0.3'	↓	15:09	SEDIMENT	2					X			X										X						X			1
2C 0.3-1.5' (Interstitial WATER)		1500	↓	~~X~~																	[illegible]	~~X~~						[illegible]	~~X~~		1
2D 1.5-2.0'		1505		3															X												1
2E 2.0-2.5'		1515		4															X												1
2F 2.5-3.0'		1525		5															X												1
2G 3.0-3.5'		1535		6															X												1
2H 3.5-4.0'		1536		7															X												1
2I 4.0-4.5'		1538		8															X												1
2J 4.5-4.8'		1540		9															X												1
1A 0-0.3		900		10					X			X				X						X						X			1
	↓		↓	~~4~~					~~X~~			~~X~~				~~X~~		PCW		1/24/91		~~X~~						~~X~~			[illegible]

PROJECT INFORMATION

PROJECT NUMBER: 1299-003-P04

PROJECT NAME: Unimar Inc

PURCHASE ORDER NUMBER:

ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	14
COC SEALS/INTACT? Y/N/NA	Y
RECEIVED GOOD COND./COLD	Y
RECEIVED VIA: COURIER	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK

GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:

SEAL # 01299-05 PCW * Cd, Cu, Pb, Zn

SEAL # 01299-06 PCW ** EPTOX + Cu, Ni, Zn

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 18:00 1/24/91	Signature: Time:	Signature: Time:
Printed Name: PAUL WERNER Date: 1/24/91	Printed Name: Date:	Printed Name: Date:
Company: GEI	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: D L Thomas Time: 12:00	Signature: Time:	Signature: Time:
Printed Name: D L Thomas Date: 1-25-91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINATOR

Analytical**Technologies,**Inc.
560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 1/24/91 PAGE 2 OF 2

LABORATORY NUMBER: 9101-191

PROJECT MANAGER: Paul Weriver
COMPANY: Geo Engineers
ADDRESS:
PHONE: 746-5200
SAMPLED BY: P.G. Weriver

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

					ANALYSIS REQUEST																				TCLP ONLY							
SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX ~~9020~~ Metals Scr	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	8080 Pesticide (4)	8240 ZH-EXT	8270	8150 Herbicides (2)	Metals (8)	Metals Screen	TBT	NUMBER OF CONTAINERS
*1B 0.3-1.0'	1/23/91	1652	Sediment	11																									X		
*1C 1.0-1.3'	1/23/91	1655	↓	12						X						X													X		
* { 2C			H2O	13																		X								X	
2C			Sed	14																		X								X	

PROJECT INFORMATION

PROJECT NUMBER: 1299-603-1304
PROJECT NAME: Damer
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☐ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	
COC SEALS/INTACT? Y/N/NA	y
RECEIVED GOOD COND./COLD	y
RECEIVED VIA: Courier	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:

HOLD Analysis *1B *1C Until Further Notice

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 1800	Signature: Time:	Signature: Time:
Printed Name: Paul Weriver Date: 1/24/91	Printed Name: Date:	Printed Name: Date:
Company: GEI	Company:	Company:
RECEIVED BY: 1.	**RECEIVED BY: 2.**	**RECEIVED BY: (LAB) 3.**
Signature: D L Thomas Time: 12:00	Signature: Time:	Signature: Time:
Printed Name: DW Thomas Date: 1-25-91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINATOR

APPENDIX

ToxScan Inc.

42 Hangar Way
Watsonville, CA 95076

(408) 724-4522

FAX (408) 724-3188

Analytical Technologies, Inc.
560 Naches Avenue SW, Suite 101
Renton, WA 98055

February 22, 1991
REVISED REPORT: August 25, 1991,
to include QA/QC

Attn: Donna McKinney

MATERIAL:	Water samples received January 29, 1991
ANALYSIS COMPLETED:	February 21, 1991
IDENTIFICATION:	Geo engineers
TOXSCAN NUMBER:	T-7198
REPORT:	Quantitative chemical analysis is as follows, expressed as nanograms per liter (parts per trillion) as received:

Sample ID	Monobutyltin	Dibutyltin	Tributyltin	Tetrabutyltin	Tripropyl Tin Surrogate
91018-191-1	6	4	5	4	100
9101-191-13	27	40	22	ND	100
Spike Recovery -see T-7197 9101-167-1	66%	78%	86%	75%	59

ND = None detected

Detection limit = 1 part per trillion

Philip D. Carpenter
Laboratory Director

TOXSCAN

AnalyticalTechnologies,Inc.
560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 1/28/91 PAGE 1 OF 1

LABORATORY NUMBER: T- 7198

PROJECT MANAGER: Donna McKinney
COMPANY: ATI- Renton
ADDRESS:
PHONE: (206)228-8335 SAMPLED BY:

SAMPLE DISPOSAL INSTRUCTIONS

☐ ATI Disposal @ $5.00 each [✓ ☒ Return]

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)	[illegible]		NUMBER OF CONTAINERS
9101-191-1	1/24/91		water	-01																									X		1
-13*				-02																									X		1
-14			Sed.	-03																									X		1

-01 1L amb glass bottle
-02 [illegible] glass bottle
-03 125ml glass jar

PROJECT INFORMATION

PROJECT NUMBER: 9101-191
PROJECT NAME: Geoengineers
PURCHASE ORDER NUMBER: 02208
ONGOING PROJECT? YES ☐ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS 3
COC SEALS/INTACT? Y/N/NA
RECEIVED GOOD COND./COLD
RECEIVED VIA:

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☐ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)
SPECIAL INSTRUCTIONS: data due ASAP!

* interstitial water Please include project # on invoice

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: D McKinney Time: 12:00	Signature: Time:	Signature: Time:
Printed Name: D. McKinney Date: 1/28/91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: K. Weaver Time: 10:41	Signature: Time:	Signature: Time:
Printed Name: Kay Weaver Date: [illegible]	Printed Name: Date:	Printed Name: Date:
Company: [illegible]	Company:	Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINATOR

WATER SAMPLE 12

SEDIMENT SAMPLES 4A-4B, 5B

AnalyticalTechnologies,Inc. 560 Naches Avenue, S.W., Suite 101, Renton, WA 98055, (206) 228-8335

ATI I.D. # 9101-216

March 5, 1991

GeoEngineers, Inc.
8410 154th Avenue N.E.
Redmond, WA 98052

Attention : Paul Werner

Project Number : 1299-003-B04

Project Name : Unimar, Inc.

On January 29, 1991, Analytical Technologies, Inc., received three sediment samples and one water sample for analysis. The samples were analyzed with EPA methodology or equivalent methods as specified in the attached analytical schedule. The results, sample cross reference, and quality control data are enclosed.

This is a partial report as it does not contain the TCLP results. These results will follow in approximately 60 days.

Donna M. McKinney
Donna M. McKinney
Project Manager

Donna M. McKinney for FWG
Frederick W. Grothkopp
Technical Manager

FWG/tc

SAMPLE CROSS REFERENCE SHEET

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ATI #	CLIENT DESCRIPTION	DATE SAMPLED	MATRIX
9101-216-1	LOCATION #12 WATER	01/28/91	WATER
9101-216-2	4A 0-0.3′	01/28/91	SEDIMENT
9101-216-3	4B 0.3-4.2′	01/28/91	SEDIMENT
9101-216-4	5B 0.3-5.0′	01/28/91	SEDIMENT

----- TOTALS -----

MATRIX	# SAMPLES
WATER	4

ATI STANDARD DISPOSAL PRACTICE

The samples from this project will be disposed of in thirty (30) days from the date of this report. If an extended storage period is required, please contact our sample control department before the scheduled disposal date.

ATI I.D. # 9101-216

ANALYTICAL SCHEDULE

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ANALYSIS	TECHNIQUE	REFERENCE	LAB
SEMI-VOLATILE COMPOUNDS	GCMS	EPA 8270	R
POLYCHLORINATED BIPHENYLS (PCBs)	GC/ECD	EPA 8080	R
POLYNUCLEAR AROMATIC HYDROCARBONS	HPLC/UV	EPA 8310	SD
TRIBUTYLTIN	GC/FPD	BATTELLE	SUB
ARSENIC	AA/GF	EPA 7060	R
BARIUM	ICAP	EPA 6010	R
CADMIUM	AA/GF	EPA 7131	R
CADMIUM	ICAP	EPA 6010	R
CHROMIUM	ICAP	EPA 6010	R
COPPER	AA/F	EPA 7210	R
LEAD	AA/GF	EPA 7421	R
LEAD	ICAP	EPA 6010	R
MERCURY	AA/COLD VAPOR	EPA 7470	R
MERCURY	AA/COLD VAPOR	EPA 7471	R
NICKEL	ICAP	EPA 6010	R
SELENIUM	AA/GF	EPA 7740	R
SILVER	ICAP	EPA 6010	R
ZINC	ICAP	EPA 6010	R
PETROLEUM HYDROCARBONS	IR	EPA 418.1	R
HARDNESS	TITRATION	EPA 130.2	R
MOISTURE	GRAVIMETRIC	METHOD 7-2.2	R

R = ATI - Renton
SD = ATI - San Diego
T = ATI - Tempe

PNR = ATI - Pensacola
FC = ATI - Fort Collins
SUB = Subcontract

ATI I.D. # 9101-216

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<0.17
PHENOL	<0.17
ANILINE	<0.17
BIS(2-CHLOROETHYL)ETHER	<0.17
2-CHLOROPHENOL	<0.17
1,3-DICHLOROBENZENE	<0.17
1,4-DICHLOROBENZENE	<0.17
BENZYL ALCOHOL	<0.17
1,2-DICHLOROBENZENE	<0.17
2-METHYLPHENOL	<0.17
BIS(2-CHLOROISOPROPYL)ETHER	<0.17
4-METHYLPHENOL	<0.17
N-NITROSO-DI-N-PROPYLAMINE	<0.17
HEXACHLOROETHANE	<0.17
NITROBENZENE	<0.17
ISOPHORONE	<0.17
2-NITROPHENOL	<0.17
2,4-DIMETHYLPHENOL	<0.17
BENZOIC ACID	<0.85
BIS(2-CHLOROETHOXY)METHANE	<0.17
2,4-DICHLOROPHENOL	<0.17
1,2,4-TRICHLOROBENZENE	<0.17
NAPHTHALENE	<0.17
4-CHLOROANILINE	<0.17
HEXACHLOROBUTADIENE	<0.17
4-CHLORO-3-METHYLPHENOL	<0.17
2-METHYLNAPHTHALENE	<0.17
HEXACHLOROCYCLOPENTADIENE	<0.17
2,4,6-TRICHLOROPHENOL	<0.17
2,4,5-TRICHLOROPHENOL	<0.85
2-CHLORONAPHTHALENE	<0.17
2-NITROANILINE	<0.85
DIMETHYLPHTHALATE	<0.17
ACENAPHTHYLENE	<0.17
3-NITROANILINE	<0.85
ACENAPHTHENE	<0.17
2,4-DINITROPHENOL	<0.85
4-NITROPHENOL	<0.85

CONTINUED NEXT PAGE

ATI I.D. # 9101-216

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
DIBENZOFURAN	<0.17
2,4-DINITROTOLUENE	<0.17
2,6-DINITROTOLUENE	<0.17
DIETHYLPHTHALATE	<0.17
4-CHLOROPHENYL-PHENYLETHER	<0.17
FLUORENE	<0.17
4-NITROANILINE	<0.85
4,6-DINITRO-2-METHYLPHENOL	<0.85
N-NITROSODIPHENYLAMINE	<0.17
4-BROMOPHENYL-PHENYLETHER	<0.17
HEXACHLOROBENZENE	<0.17
PENTACHLOROPHENOL	<0.85
PHENANTHRENE	<0.17
ANTHRACENE	<0.17
DI-N-BUTYLPHTHALATE	<0.17
FLUORANTHENE	<0.17
BENZIDINE	<1.7
PYRENE	<0.17
BUTYLBENZYLPHTHALATE	<0.17
3,3-DICHLOROBENZIDINE	<0.34
BENZO(a)ANTHRACENE	<0.17
BIS(2-ETHYLHEXYL)PHTHALATE	<0.17
CHRYSENE	<0.17
DI-N-OCTYLPHTHALATE	<0.17
BENZO(b)FLUORANTHENE	<0.17
BENZO(k)FLUORANTHENE	<0.17
BENZO(a)PYRENE	<0.17
INDENO(1,2,3-cd)PYRENE	<0.17
DIBENZ(a,h)ANTHRACENE	<0.17
BENZO(g,h,i)PERYLENE	<0.17

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	73
2-FLUOROBIPHENYL	82
TERPHENYL-d14	76
PHENOL-d6	86
2-FLUOROPHENOL	66
2,4,6-TRIBROMOPHENOL	87

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SOIL
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
HYDROCARBON	1883	0.63
HYDROCARBON	1944	1.0
HYDROCARBON	2013	1.3
HYDROCARBON	2094	1.4
HYDROCARBON	2306	1.0

ATI I.D. # 9101-216-2

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 4A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/28/91
DATE RECEIVED : 01/29/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 5

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<3.5
PHENOL	<3.5
ANILINE	<3.5
BIS(2-CHLOROETHYL)ETHER	<3.5
2-CHLOROPHENOL	<3.5
1,3-DICHLOROBENZENE	<3.5
1,4-DICHLOROBENZENE	<3.5
BENZYL ALCOHOL	<3.5
1,2-DICHLOROBENZENE	<3.5
2-METHYLPHENOL	<3.5
BIS(2-CHLOROISOPROPYL)ETHER	<3.5
4-METHYLPHENOL	<3.5
N-NITROSO-DI-N-PROPYLAMINE	<3.5
HEXACHLOROETHANE	<3.5
NITROBENZENE	<3.5
ISOPHORONE	<3.5
2-NITROPHENOL	<3.5
2,4-DIMETHYLPHENOL	<3.5
BENZOIC ACID	<18
BIS(2-CHLOROETHOXY)METHANE	<3.5
2,4-DICHLOROPHENOL	<3.5
1,2,4-TRICHLOROBENZENE	<3.5
NAPHTHALENE	2.6 J
4-CHLOROANILINE	<3.5
HEXACHLOROBUTADIENE	<3.5
4-CHLORO-3-METHYLPHENOL	<3.5
2-METHYLNAPHTHALENE	12
HEXACHLOROCYCLOPENTADIENE	<3.5
2,4,6-TRICHLOROPHENOL	<3.5
2,4,5-TRICHLOROPHENOL	<18
2-CHLORONAPHTHALENE	<3.5
2-NITROANILINE	<18
DIMETHYLPHTHALATE	<3.5
ACENAPHTHYLENE	4.5
3-NITROANILINE	<18
ACENAPHTHENE	22
2,4-DINITROPHENOL	<18
4-NITROPHENOL	<18

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SEMI-VOLATILE ORGANICS ANALYSIS DATA SUMMARY (CONTINUED)

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 4A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/28/91
DATE RECEIVED : 01/29/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 5

COMPOUND	RESULT	
DIBENZOFURAN	6.3	
2,4-DINITROTOLUENE	<3.5	
2,6-DINITROTOLUENE	<3.5	
DIETHYLPHTHALATE	<3.5	
4-CHLOROPHENYL-PHENYLETHER	<3.5	
FLUORENE	20	
4-NITROANILINE	<18	
4,6-DINITRO-2-METHYLPHENOL	<18	
N-NITROSODIPHENYLAMINE	<3.5	
4-BROMOPHENYL-PHENYLETHER	<3.5	
HEXACHLOROBENZENE	<3.5	
PENTACHLOROPHENOL	<18	
PHENANTHRENE	56	
ANTHRACENE	20	
DI-N-BUTYLPHTHALATE	<3.5	
FLUORANTHENE	34	
BENZIDINE	<35	
PYRENE	32	
BUTYLBENZYLPHTHALATE	<3.5	
3,3-DICHLOROBENZIDINE	<7.1	
BENZO(a)ANTHRACENE	14	
BIS(2-ETHYLHEXYL)PHTHALATE	1.9	J
CHRYSENE	15	
DI-N-OCTYLPHTHALATE	<3.5	
BENZO(b)FLUORANTHENE	14	
BENZO(k)FLUORANTHENE	4.9	
BENZO(a)PYRENE	14	
INDENO(1,2,3-cd)PYRENE	6.9	
DIBENZ(a,h)ANTHRACENE	2.0	J
BENZO(g,h,i)PERYLENE	8.2	

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	72
2-FLUOROBIPHENYL	109
TERPHENYL-d14	96
PHENOL-d6	94
2-FLUOROPHENOL	70
2,4,6-TRIBROMOPHENOL	100

J = Estimated value.

ATI I.D. # 9101-216-2

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 4A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/28/91
DATE RECEIVED : 01/29/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 5

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
NAPHTHALENE, 1,8-DIMETHYL-	878	31
NAPHTHALENE, 1,5-DIMETHYL-	890	31
NAPHTHALENE, 1,7-DIMETHYL-	894	14
NAPHTHALENE, 1,2-DIMETHYL-	909	17
PHENANTHRENE, 3-METHYL-	1294	4.9

SEMI-VOLATILE ORGANIC
QUALITY CONTROL DATA

CLIENT	: GEOENGINEERS, INC.	SAMPLE I.D.	: BLANK SPIKE
PROJECT #	: 1299-003-B04	DATE EXTRACTED	: 01/30/91
PROJECT NAME	: UNIMAR, INC.	DATE ANALYZED	: 02/08/91
EPA METHOD	: 8270	MATRIX	: SOIL
		UNITS	: mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
1,2,4-TRICHLOROBENZENE	<0.17	3.33	2.39	72	2.86	86	18
ACENAPHTHENE	<0.17	3.33	2.19	66	2.42	73	10
2,4-DINITROTOLUENE	<0.17	3.33	2.46	74	2.56	77	4
PYRENE	<0.17	3.33	2.34	70	2.62	78	11
N-NITROSO-DI-N-PROPYLAMINE	<0.17	3.33	2.26	68	2.68	81	17
1,4-DICHLOROBENZENE	<0.17	3.33	2.23	67	2.65	79	17
PENTACHLOROPHENOL	<0.85	13.3	11.6	87	11.3	85	3
PHENOL	<0.17	6.67	3.98	60	4.79	72	18
2-CHLOROPHENOL	<0.17	6.67	4.03	60	4.90	73	19
4-CHLORO-3-METHYLPHENOL	<0.17	6.67	4.44	67	4.99	75	12
4-NITROPHENOL	<0.85	13.3	11.5	86	11.6	87	1

$$\text{\% Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-216

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/04/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
PCB 1016	<0.033
PCB 1221	<0.033
PCB 1232	<0.033
PCB 1242	<0.033
PCB 1248	<0.033
PCB 1254	<0.033
PCB 1260	<0.033

ATI I.D. # 9101-216-2

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 4A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8080 (PCB)
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/28/91
DATE RECEIVED : 01/29/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/04/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
PCB 1016	<0.14
PCB 1221	<0.14
PCB 1232	<0.14
PCB 1242	<0.14
PCB 1248	<0.14
PCB 1254	<0.14
PCB 1260	<0.14

ATI I.D. # 9101-216

POLYCHLORINATED BIPHENYLS (PCB)
QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
EPA METHOD : 8080 (PCB)

SAMPLE ID : BLANK SPIKE
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/04/91
MATRIX : SOIL
UNITS : mg/Kg

COMPOUND	SAMPLE RESULT	CONC SPIKED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % RECOVERY	RPD
PCB 1260	<0.033	0.33	0.345	104	0.351	106	2

$$\% \text{ Recovery} = \frac{(\text{Spike Sample result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-216

POLYNUCLEAR AROMATICS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SOIL
EPA METHOD : 8310
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
NAPHTHALENE	<0.083
ACENAPHTHYLENE	<0.17
ACENAPHTHENE	<0.17
FLUORENE	<0.017
PHENANTHRENE	<0.0083
ANTHRACENE	<0.0083
FLUORANTHENE	<0.017
PYRENE	<0.017
BENZO(a)ANTHRACENE	<0.017
CHRYSENE	<0.017
BENZO(b)FLUORANTHENE	<0.017
BENZO(k)FLUORANTHENE	<0.017
BENZO(a)PYRENE	<0.017
DIBENZ(a,h)ANTHRACENE	<0.034
BENZO(g,h,i)PERYLENE	<0.017
INDENO(1,2,3-cd)PYRENE	<0.017

ATI I.D. # 9101-216-3

POLYNUCLEAR AROMATICS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/28/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/29/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: 4B 0.3-4.2'	DATE ANALYZED	: 02/09/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8310	DILUTION FACTOR	: 10

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
NAPHTHALENE	<3.8
ACENAPHTHYLENE	<7.7
ACENAPHTHENE	<7.7
FLUORENE	4.1
PHENANTHRENE	15
ANTHRACENE	5.0
FLUORANTHENE	32
PYRENE	40
BENZO(a)ANTHRACENE	11
CHRYSENE	15
BENZO(b)FLUORANTHENE	7.7
BENZO(k)FLUORANTHENE	4.5
BENZO(a)PYRENE	14
DIBENZ(a,h)ANTHRACENE	4.5
BENZO(g,h,i)PERYLENE	13
INDENO(1,2,3-cd)PYRENE	10

ATI I.D. # 9101-216-4

POLYNUCLEAR AROMATICS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/28/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/29/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: 5B 0.3-5.0'	DATE ANALYZED	: 02/09/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8310	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
NAPHTHALENE	1.2
ACENAPHTHYLENE	1.0
ACENAPHTHENE	<0.68
FLUORENE	1.8
PHENANTHRENE	5.6
ANTHRACENE	2.3
FLUORANTHENE	19
PYRENE	23
BENZO(a)ANTHRACENE	6.0
CHRYSENE	7.6
BENZO(b)FLUORANTHENE	5.2
BENZO(k)FLUORANTHENE	2.9
BENZO(a)PYRENE	9.2
DIBENZ(a,h)ANTHRACENE	0.39
BENZO(g,h,i)PERYLENE	8.0
INDENO(1,2,3-cd)PYRENE	6.4

POLYNUCLEAR AROMATICS
QUALITY CONTROL DATA

CLIENT	: GEOENGINEERS, INC.	SAMPLE I.D.	: 101289-32
PROJECT #	: 1299-003-B04	DATE EXTRACTED	: 01/30/91
PROJECT NAME	: UNIMAR, INC.	DATE ANALYZED	: 02/08/91
EPA METHOD	: 8310	MATRIX	: SOIL
		UNITS	: mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
ACENAPHTHYLENE	<0.17	16.6	11	66	11	66	0
PHENANTHRENE	<0.0083	1.77	1.4	79	1.5	85	7
PYRENE	<0.017	1.76	1.8	102	1.9	108	5
BENZO(k)FLUORANTHENE	<0.017	1.50	1.2	79	1.4	93	15

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

POLYNUCLEAR AROMATICS
QUALITY CONTROL DATA

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
EPA METHOD : 8310

SAMPLE I.D. : BLANK SPIKE
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/08/91
MATRIX : SOIL
UNITS : mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
ACENAPHTHYLENE	<0.17	16.6	11	66	N/A	N/A	N/A
PHENANTHRENE	<0.0083	1.77	1.3	73	N/A	N/A	N/A
PYRENE	<0.017	1.76	1.7	97	N/A	N/A	N/A
BENZO(k)FLUORANTHENE	<0.017	1.58	1.3	82	N/A	N/A	N/A

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-216

GAS CHROMATOGRAPHY RESULTS *

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : ng/L

PARAMETER	LOCATION #12 WATER -1
MONOBUTYLTIN	21
DIBUTYLTIN	33
TRIBUTYLTIN	185
TETRABUTYLTIN	140

* Analyzed by GC/FPD, Method Battelle N05196300.

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : WATER

ELEMENT	DATE PREPARED	DATE ANALYZED
ARSENIC	02/01/91	02/17/91
BARIUM	02/01/91	02/28/91
CADMIUM	02/01/91	02/26/91
CHROMIUM	02/01/91	02/28/91
COPPER	02/01/91	02/01/91
LEAD	02/01/91	02/26/91
MERCURY	02/01/91	02/20/91
NICKEL	02/01/91	02/28/91
SELENIUM	02/01/91	02/17/91
SILVER	02/01/91	02/28/91
ZINC	02/01/91	02/28/91

ATI I.D. # 9101-216

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

PARAMETER	LOCATION #12 WATER -1	REAGENT BLANK
ARSENIC	<0.005	<0.005
BARIUM	<0.02	<0.02
CADMIUM	<0.0003	<0.0003
CHROMIUM	<0.02	<0.02
COPPER	<0.02	<0.02
LEAD	0.005	<0.005
MERCURY	0.0005	<0.0005
NICKEL	<0.01	<0.01
SELENIUM	<0.005	<0.005
SILVER	<0.03	<0.03
ZINC	0.04	<0.02

METALS QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

COMPOUND	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED SAMPLE	SPIKE CONC	% REC
ARSENIC	9101-238-7	<0.005	<0.005	0	0.023	0.025	92
BARIUM	9101-238-7	<0.02	<0.02	0	10.6	10.0	106
CADMIUM	9101-238-7	<0.0003	0.0006	0	0.0010	0.0010	100
CHROMIUM	9101-238-7	<0.02	<0.02	0	2.59	2.50	104
COPPER	9101-174-2	0.65	0.59	10	3.04	2.50	96
LEAD	9101-238-7	<0.005	<0.005	0	0.027	0.025	108
MERCURY	9102-069-4	<0.0005	<0.0005	0	0.0030	0.0020	150
MERCURY	BLANK SPIKE	N/A	N/A	N/A	0.0025	0.0020	125
NICKEL	9101-238-7	<0.01	<0.01	0	2.62	2.50	105
SELENIUM	9101-238-7	<0.005	<0.005	0	0.026	0.025	104
SILVER	9101-238-7	<0.03	<0.03	0	**	**	**
SILVER	BLANK SPIKE	N/A	N/A	N/A	11.8	10.0	118
ZINC	9101-238-7	0.11	0.12	9	2.88	2.50	111

** Due to the necessary dilution of the sample, result was not attainable.

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : SEDIMENT

ELEMENT	DATE PREPARED	DATE ANALYZED
ARSENIC	02/08/91	02/28/91
BARIUM	02/08/91	02/26/91
CADMIUM	02/08/91	02/26/91
CHROMIUM	02/08/91	02/26/91
COPPER	02/08/91	02/26/91
LEAD	02/08/91	02/26/91
MERCURY	02/22/91	02/22/91
NICKEL	02/08/91	02/26/91
SELENIUM	02/08/91	02/19/91
SILVER	02/08/91	02/26/91
ZINC	02/08/91	02/26/91

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

PARAMETER	4A 0-0.3′ -2	4B 0.3-4.2′ -3	5B 0.3-5.0′ -4	REAGENT BLANK
ARSENIC	1,800	180	26	<0.5
BARIUM	210	120	110	<1
CADMIUM	27	10	8.2	<1
CHROMIUM	89	59	45	<1
COPPER	1,500	240	89	3.7
LEAD	1,700	290	130	<2.5
MERCURY	0.80	0.91	1.05	<0.15
NICKEL	72	59	55	<1
SELENIUM	<1	<1	<1	<1
SILVER	4.7	<2	<2	<2
ZINC	4,800	620	210	2.8

ATI I.D. # 9101-216

METALS QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

COMPOUND	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED SAMPLE	SPIKE CONC	% REC
ARSENIC	9101-216-4	26	23	12	35	9.4	96
BARIUM	9101-216-4	110	110	0	1,910	1,880	96
CADMIUM	9101-216-4	8.2	8.0	2	92	94	89
CHROMIUM	9101-216-4	45	45	0	525	470	102
COPPER	9101-216-4	89	85	5	522	470	92
LEAD	9101-216-4	130	129	1	979	940	90
MERCURY	9101-216-4	1.05	<0.80	0	1.78	1.67	44*
MERCURY	BLANK SPIKE	N/A	N/A	N/A	0.44	0.50	88
NICKEL	9101-216-4	55	54	2	499	470	94
SELENIUM	9101-216-4	<1	<1	0	6.4	9.4	68
SILVER	9101-216-4	<2	<2	0	113	94	120
ZINC	9101-216-4	210	203	3	694	470	103

* Out of limits due to matrix interference.

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

ATI I.D. # 9101-216

GENERAL CHEMISTRY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : WATER

ANALYSIS	DATE PREPARED	DATE ANALYZED
HARDNESS	02/08/91	02/08/91

ATI I.D. # 9101-216

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

ATI I.D. #	CLIENT I.D.	HARDNESS
9101-216-1	LOCATION #12 WATER	40
REAGENT BLANK	-	<5

GENERAL CHEMISTRY QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

SAMPLE MATRIX : WATER

PARAMETER	UNITS	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED RESULT	SPIKE ADDED	% REC
HARDNESS	mg/L	9102-011-2	27	24	12	552	500	105

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

GENERAL CHEMISTRY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : SEDIMENT

ANALYSIS	DATE PREPARED	DATE ANALYZED
PETROLEUM HYDROCARBONS	02/04/91	02/04/91
MOISTURE	-	01/31/91

GENERAL CHEMISTRY RESULTS

CLIENT	: GEOENGINEERS, INC.	MATRIX : SEDIMENT
PROJECT #	: 1299-003-B04	
PROJECT NAME	: UNIMAR, INC.	UNITS : mg/Kg

ATI I.D. #	CLIENT I.D.	PETROLEUM HYDROCARBONS
9101-216-2	4A 0-0.3'	590
9101-216-3	4B 0.3-4.2'	420
9101-216-4	5B 0.3-5.0'	39
REAGENT BLANK	-	<5

ATI I.D. # 9101-216

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : %

ATI I.D. #	CLIENT I.D.	MOISTURE
9101-216-2	4A 0-0.3′	76
9101-216-3	4B 0.3-4.2′	78
9101-216-4	5B 0.3-5.0′	75

GENERAL CHEMISTRY QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT/SOIL

PARAMETER	UNITS	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED RESULT	SPIKE ADDED	% REC
PETROLEUM HYDROCARBONS	mg/Kg	9101-238-1	780	776	0	**	**	**
PETROLEUM HYDROCARBONS	mg/Kg	BLANK SPIKE	N/A	N/A	N/A	293	268	109
MOISTURE	%	9101-228-3	14	14	0	N/A	N/A	N/A

** Due to the necessary dilution of the sample, result was not attainable.

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

AnalyticalTechnologies,Inc.
560 Naches Avenue SW, Suite 101 Renton, WA 98055 (206)228-8335

DATE 1/28/91 PAGE 1 OF 1

Chain of Custody

LABORATORY NUMBER: 9101-216

PROJECT MANAGER: Paul Werner
COMPANY: Geo Engineers
ADDRESS: Bellevue
PHONE: 746-5200 SAMPLED BY: P.G. Werner

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

					ANALYSIS REQUEST																			TCLP ONLY							
SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) ~~EP EXT~~	Priority Pollutant Metals (13)	8080 Pesticide (4)	8240 ZH-EXT	8270	8150 Herbicides (2)	Metals (8)	TBT	Hardness	NUMBER OF CONTAINERS
Location #12 Water	1/28/91	11:30	Water	-1																		X							X	X	3
Bottom Sediment			Soil																												
4A 0-0.3'		1500		-2					X			X				X						X						X			1
4B 0.3-4.2'		1322		-3						X						X						X									1
~~5A~~ 5B 0.3-5.0		1625		-4						X						X						X									1
per Paul Werner on 1/31/91																															

PROJECT INFORMATION

PROJECT NUMBER: 1299-003-B01
PROJECT NAME: Univar Inc
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	6
COC SEALS/INTACT? Y/N/NA	y
RECEIVED GOOD COND./COLD	y
RECEIVED VIA: Courier	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: [✓ (NORMAL) ☒ 2WKS] (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:
1299-008-PGW

* TEPTOX + Cu, Ni, Zn

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 15:00	Signature: Time:	Signature: Time:
Printed Name: Paul Werner Date: 1/28/91	Printed Name: Date:	Printed Name: Date:
Company: GEI	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: DL Thomas Time: 11:30	Signature: Time:	Signature: Time:
Printed Name: DL Thomas Date: 1-29-91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Analytical Technologies, Inc.

*TI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINATOR

AnalyticalTechnologies,Inc.
560 Naches Avenue SW, Suite 101 Renton, WA 98055 (206)228-8335

TO SD

Chain of Custody

DATE 1/29/91 PAGE 1 OF 1

LABORATORY NUMBER: 101385

PROJECT MANAGER: Donna McKinney
COMPANY: ATI - Renton
ADDRESS:
PHONE: (206) 228-8335 SAMPLED BY:

SAMPLE DISPOSAL INSTRUCTIONS

☐ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA *	8310 HPLC PNA Silica gel	8080 Pesticides & PCB's	PCB's ONLY *	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)			NUMBER OF CONTAINERS
9101-216-2	1/28/91		Sed	01					X			X																			1
-3				02						X																					1
-4				03						X																					1

PROJECT INFORMATION

PROJECT NUMBER: 9101-216
PROJECT NAME: Geoengineers
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☐ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	3
COC SEALS/INTACT? Y/N/NA	NA
RECEIVED GOOD COND./COLD	X
RECEIVED VIA:	X

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☐ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)
SPECIAL INSTRUCTIONS: data for 8310 due 2/11/91 (250--)
(will fax 2 moi)
* extract + return ASAP!

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: [illegible] Time: 400	Signature: Time:	Signature: Time:
Printed Name: [illegible] Date:	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Company:
RECEIVED BY: 1.	**RECEIVED BY: 2.**	**RECEIVED BY: (LAB) 3.**
Signature: Time:	Signature: Time:	Signature: Patrice Kaido Time: 0930
Printed Name: Date:	Printed Name: Date:	Printed Name: P Kaido Date: 1.30.91
Company:	Company:	Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 **DISTRIBUTION:** White, Canary - ATI • Pink - ORIGINATOR

NO 0770

APPENDIX

ToxScan Inc.

42 Hangar Way
Watsonville, CA 95076

(408) 724-4522

FAX (408) 724-3188

Analytical Technologies, Inc.
560 Naches Avenue SW, Suite 101
Renton, WA 98055

February 26, 1991
REVISED REPORT: August 25, 1991,
to include QA/QC

Attn: Donna McKinney

MATERIAL:	Water sample received January 30, 1991
ANALYSIS COMPLETED:	February 25, 1991
IDENTIFICATION:	12 Duplicate
TOXSCAN NUMBER:	T-7203
REPORT:	Quantitative chemical analysis is as follows, expressed as nanograms per liter (parts per trillion) as received:

Sample ID	Monobutyltin	Dibutyltin	Tributyltin	Tetrabutyltin	Tripropyl Tin Surrogate
9101-216-1	21	33	185	140	55
Spike Recovery -see T-7251 56648-5	72%	83%	84%	77%	91

ND = None detected

Detection limit = 1 part per trillion

Philip D. Carpenter
Laboratory Director

TOXSCAN

Analytical Technologies, Inc.

560 Naches Avenue SW, Suite 101 Renton, WA 98055 (206)228-8335

Chain of Custody

DATE 1/29/91 PAGE 1 OF 1

LABORATORY NUMBER: T-2203

PROJECT MANAGER: Donna McKinney

COMPANY: ATI - Renton

ADDRESS:

PHONE: (206)228-8335 SAMPLED BY:

SAMPLE DISPOSAL INSTRUCTIONS

☐ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)	[illegible]		NUMBER OF CONTAINERS
9101-216-1	1/25/91		water																										✓		1
source "12 Duplicate			"																												
[illegible]																															
call [illegible]																															

liter

PROJECT INFORMATION

PROJECT NUMBER: 9101-216

PROJECT NAME: Geoengineers

PURCHASE ORDER NUMBER: 02235

ONGOING PROJECT? YES ☐ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	1
COC SEALS/INTACT? Y/N/NA	
RECEIVED GOOD COND./COLD	
RECEIVED VIA:	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☐ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK

GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:

data ASAP please

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: V. Pennick Time: 4pm	Signature: Time:	Signature: Time:
Printed Name: V. Pennick Date: 1/29/91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: Time: 10 am	Signature: Time:	Signature: Time:
Printed Name: [illegible] Date: [illegible]	Printed Name: Date:	Printed Name: Date:
Company: TSI	Company:	Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINATOR

NO [illegible]700

C – 108

RINSEATE SAMPLES 10B, 10D, 10F

SEDIMENT SAMPLES 5A, 6A-6B, 7A-7B, 11 DUPLICATE

AnalyticalTechnologies,Inc. 560 Naches Avenue, S.W., Suite 101, Renton, WA 98055, (206) 228-8335

GeoEngineers

MAR 07 1991

Routing ☐ ☐
☐ ☐ ☐
File

ATI I.D. # 9101-238

March 6, 1991

GeoEngineers, Inc.
8410 154th Avenue N.E.
Redmond, WA 98052

Attention : Paul Werner

Project Number : 1299-003-B04

Project Name : Unimar, Inc.

On January 31, 1991, Analytical Technologies, Inc., received six sediment and three water samples for analysis. The samples were analyzed with EPA methodology or equivalent methods as specified in the attached analytical schedule. The results, sample cross reference, and quality control data are enclosed.

This is a partial report as it does not contain the TCLP results. These results will follow in approximately 60 days.

Donna M. McKinney
Donna M. McKinney
Project Manager

Donna M. McKinney for FWG
Frederick W. Grothkopp
Technical Manager

FWG/tc

ATI I.D. # 9101-238

SAMPLE CROSS REFERENCE SHEET

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ATI #	CLIENT DESCRIPTION	DATE SAMPLED	MATRIX
9101-238-1	5A 0-0.3'	01/29/91	SEDIMENT
9101-238-2	6A 0-0.3'	01/30/91	SEDIMENT
9101-238-3	11 DUPLICATE	01/30/91	SEDIMENT
9101-238-4	6B 0.3-5.0'	01/30/91	SEDIMENT
9101-238-5	VAN VEEN RINSEATE 10D	01/30/91	WATER
9101-238-6	SHELBY RINSEATE 10B	01/30/91	WATER
9101-238-7	BETA RINSEATE 10F	01/30/91	WATER
9101-238-8	7A 0-0.3'	01/30/91	SEDIMENT
9101-238-9	7B 0.3-5.0'	01/30/91	SEDIMENT

----- TOTALS -----

MATRIX	# SAMPLES
SEDIMENT	6
WATER	3

ATI STANDARD DISPOSAL PRACTICE

The samples from this project will be disposed of in thirty (30) days from the date of this report. If an extended storage period is required, please contact our sample control department before the scheduled disposal date.

ATI I.D. # 9101-238

ANALYTICAL SCHEDULE

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ANALYSIS	TECHNIQUE	REFERENCE	LAB
SEMI-VOLATILE COMPOUNDS	GCMS	EPA 8270	R
POLYCHLORINATED BIPHENYLS (PCBs)	GC/ECD	EPA 8080	R
POLYNUCLEAR AROMATIC HYDROCARBONS	HPLC/UV	EPA 8310	SD
TRIBUTYLTIN	GC/FPD	BATTELLE	SUB
ARSENIC	AA/GF	EPA 7060	R
BARIUM	ICAP	EPA 6010	R
CADMIUM	AA/GF	EPA 7131	R
CADMIUM	ICAP	EPA 6010	R
CHROMIUM	ICAP	EPA 6010	R
COPPER	AA/F	EPA 7210	R
COPPER	ICAP	EPA 6010	R
LEAD	AA/GF	EPA 7421	R
LEAD	ICAP	EPA 6010	R
MERCURY	AA/COLD VAPOR	EPA 7470	R
MERCURY	AA/COLD VAPOR	EPA 7471	R
NICKEL	ICAP	EPA 6010	R
SELENIUM	AA/GF	EPA 7740	R
SILVER	ICAP	EPA 6010	R
ZINC	ICAP	EPA 6010	R
PETROLEUM HYDROCARBONS	IR	EPA 418.1	R
MOISTURE	GRAVIMETRIC	METHOD 7-2.2	R

R = ATI - Renton
SD = ATI - San Diego
T = ATI - Tempe

PNR = ATI - Pensacola
FC = ATI - Fort Collins
SUB = Subcontract

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SOIL
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 02/06/91
DATE ANALYZED : 02/12/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<0.17
PHENOL	<0.17
ANILINE	<0.17
BIS(2-CHLOROETHYL)ETHER	<0.17
2-CHLOROPHENOL	<0.17
1,3-DICHLOROBENZENE	<0.17
1,4-DICHLOROBENZENE	<0.17
BENZYL ALCOHOL	<0.17
1,2-DICHLOROBENZENE	<0.17
2-METHYLPHENOL	<0.17
BIS(2-CHLOROISOPROPYL)ETHER	<0.17
4-METHYLPHENOL	<0.17
N-NITROSO-DI-N-PROPYLAMINE	<0.17
HEXACHLOROETHANE	<0.17
NITROBENZENE	<0.17
ISOPHORONE	<0.17
2-NITROPHENOL	<0.17
2,4-DIMETHYLPHENOL	<0.17
BENZOIC ACID	<0.85
BIS(2-CHLOROETHOXY)METHANE	<0.17
2,4-DICHLOROPHENOL	<0.17
1,2,4-TRICHLOROBENZENE	<0.17
NAPHTHALENE	<0.17
4-CHLOROANILINE	<0.17
HEXACHLOROBUTADIENE	<0.17
4-CHLORO-3-METHYLPHENOL	<0.17
2-METHYLNAPHTHALENE	<0.17
HEXACHLOROCYCLOPENTADIENE	<0.17
2,4,6-TRICHLOROPHENOL	<0.17
2,4,5-TRICHLOROPHENOL	<0.85
2-CHLORONAPHTHALENE	<0.17
2-NITROANILINE	<0.85
DIMETHYLPHTHALATE	<0.17
ACENAPHTHYLENE	<0.17
3-NITROANILINE	<0.85
ACENAPHTHENE	<0.17
2,4-DINITROPHENOL	<0.85
4-NITROPHENOL	<0.85

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ATI I.D. # 9101-238

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/06/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/12/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
DIBENZOFURAN	<0.17
2,4-DINITROTOLUENE	<0.17
2,6-DINITROTOLUENE	<0.17
DIETHYLPHTHALATE	<0.17
4-CHLOROPHENYL-PHENYLETHER	<0.17
FLUORENE	<0.17
4-NITROANILINE	<0.85
4,6-DINITRO-2-METHYLPHENOL	<0.85
N-NITROSODIPHENYLAMINE	<0.17
4-BROMOPHENYL-PHENYLETHER	<0.17
HEXACHLOROBENZENE	<0.17
PENTACHLOROPHENOL	<0.85
PHENANTHRENE	<0.17
ANTHRACENE	<0.17
DI-N-BUTYLPHTHALATE	0.75
FLUORANTHENE	<0.17
BENZIDINE	<1.7
PYRENE	<0.17
BUTYLBENZYLPHTHALATE	<0.17
3,3-DICHLOROBENZIDINE	<0.34
BENZO(a)ANTHRACENE	<0.17
BIS(2-ETHYLHEXYL)PHTHALATE	<0.17
CHRYSENE	<0.17
DI-N-OCTYLPHTHALATE	<0.17
BENZO(b)FLUORANTHENE	<0.17
BENZO(k)FLUORANTHENE	<0.17
BENZO(a)PYRENE	<0.17
INDENO(1,2,3-cd)PYRENE	<0.17
DIBENZ(a,h)ANTHRACENE	<0.17
BENZO(g,h,i)PERYLENE	<0.17

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	65
2-FLUOROBIPHENYL	69
TERPHENYL-d14	65
PHENOL-d6	76
2-FLUOROPHENOL	66
2,4,6-TRIBROMOPHENOL	71

ATI I.D. # 9101-238

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/06/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/12/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
PROPANOIC ACID, 2-HYDROXY-	35	0.53
HYDROCARBON	1396	0.83

ATI I.D. # 9101-238-1

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 5A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/29/91
DATE RECEIVED : 01/31/91
DATE EXTRACTED : 02/06/91
DATE ANALYZED : 02/13/91
UNITS : mg/Kg
DILUTION FACTOR : 10

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<11
PHENOL	<11
ANILINE	<11
BIS(2-CHLOROETHYL)ETHER	<11
2-CHLOROPHENOL	<11
1,3-DICHLOROBENZENE	<11
1,4-DICHLOROBENZENE	<11
BENZYL ALCOHOL	<11
1,2-DICHLOROBENZENE	<11
2-METHYLPHENOL	<11
BIS(2-CHLOROISOPROPYL)ETHER	<11
4-METHYLPHENOL	<11
N-NITROSO-DI-N-PROPYLAMINE	<11
HEXACHLOROETHANE	<11
NITROBENZENE	<11
ISOPHORONE	<11
2-NITROPHENOL	<11
2,4-DIMETHYLPHENOL	<11
BENZOIC ACID	<57
BIS(2-CHLOROETHOXY)METHANE	<11
2,4-DICHLOROPHENOL	<11
1,2,4-TRICHLOROBENZENE	<11
NAPHTHALENE	<11
4-CHLOROANILINE	<11
HEXACHLOROBUTADIENE	<11
4-CHLORO-3-METHYLPHENOL	<11
2-METHYLNAPHTHALENE	<11
HEXACHLOROCYCLOPENTADIENE	<11
2,4,6-TRICHLOROPHENOL	<11
2,4,5-TRICHLOROPHENOL	<57
2-CHLORONAPHTHALENE	<11
2-NITROANILINE	<57
DIMETHYLPHTHALATE	<11
ACENAPHTHYLENE	<11
3-NITROANILINE	<57
ACENAPHTHENE	8.8 J
2,4-DINITROPHENOL	<57
4-NITROPHENOL	<57

CONTINUED NEXT PAGE

J = Estimated value.

ATI I.D. # 9101-238-1

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 5A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/29/91
DATE RECEIVED : 01/31/91
DATE EXTRACTED : 02/06/91
DATE ANALYZED : 02/13/91
UNITS : mg/Kg
DILUTION FACTOR : 10

COMPOUND	RESULT	
DIBENZOFURAN	<11	
2,4-DINITROTOLUENE	<11	
2,6-DINITROTOLUENE	<11	
DIETHYLPHTHALATE	<11	
4-CHLOROPHENYL-PHENYLETHER	<11	
FLUORENE	6.5	J
4-NITROANILINE	<57	
4,6-DINITRO-2-METHYLPHENOL	<57	
N-NITROSODIPHENYLAMINE	<11	
4-BROMOPHENYL-PHENYLETHER	<11	
HEXACHLOROBENZENE	<11	
PENTACHLOROPHENOL	<57	
PHENANTHRENE	15	
ANTHRACENE	9.6	J
DI-N-BUTYLPHTHALATE	<11	
FLUORANTHENE	52	
BENZIDINE	<110	
PYRENE	54	
BUTYLBENZYLPHTHALATE	<11	
3,3-DICHLOROBENZIDINE	<23	
BENZO(a)ANTHRACENE	18	
BIS(2-ETHYLHEXYL)PHTHALATE	<11	
CHRYSENE	21	
DI-N-OCTYLPHTHALATE	<11	
BENZO(b)FLUORANTHENE	23	
BENZO(k)FLUORANTHENE	67	
BENZO(a)PYRENE	26	
INDENO(1,2,3-cd)PYRENE	17	
DIBENZ(a,h)ANTHRACENE	<11	
BENZO(g,h,i)PERYLENE	20	

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	66
2-FLUOROBIPHENYL	98
TERPHENYL-d14	84
PHENOL-d6	84
2-FLUOROPHENOL	72
2,4,6-TRIBROMOPHENOL	96

J = Estimated value.

ATI I.D. # 9101-238-1

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 5A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/29/91
DATE RECEIVED : 01/31/91
DATE EXTRACTED : 02/06/91
DATE ANALYZED : 02/13/91
UNITS : mg/Kg
DILUTION FACTOR : 10

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
2-PHENYLNAPHTHALENE	1347	11
11H-BENZO(A)FLUORENE	1513	8.9
BENZO(E)PYRENE	1852	11
BENZO(B)PYRENE	1879	20
DIBENZO(DEF MNO)CHRYSENE	2201	8.9

ATI I.D. # 9101-238-2

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 6A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/30/91
DATE RECEIVED : 01/31/91
DATE EXTRACTED : 02/06/91
DATE ANALYZED : 02/13/91
UNITS : mg/Kg
DILUTION FACTOR : 10

COMPOUND	RESULT	
N-NITROSODIMETHYLAMINE	<8.5	
PHENOL	<8.5	
ANILINE	<8.5	
BIS(2-CHLOROETHYL)ETHER	<8.5	
2-CHLOROPHENOL	<8.5	
1,3-DICHLOROBENZENE	<8.5	
1,4-DICHLOROBENZENE	<8.5	
BENZYL ALCOHOL	<8.5	
1,2-DICHLOROBENZENE	<8.5	
2-METHYLPHENOL	<8.5	
BIS(2-CHLOROISOPROPYL)ETHER	<8.5	
4-METHYLPHENOL	<8.5	
N-NITROSO-DI-N-PROPYLAMINE	<8.5	
HEXACHLOROETHANE	<8.5	
NITROBENZENE	<8.5	
ISOPHORONE	<8.5	
2-NITROPHENOL	<8.5	
2,4-DIMETHYLPHENOL	<8.5	
BENZOIC ACID	<43	
BIS(2-CHLOROETHOXY)METHANE	<8.5	
2,4-DICHLOROPHENOL	<8.5	
1,2,4-TRICHLOROBENZENE	<8.5	
NAPHTHALENE	38	
4-CHLOROANILINE	<8.5	
HEXACHLOROBUTADIENE	<8.5	
4-CHLORO-3-METHYLPHENOL	<8.5	
2-METHYLNAPHTHALENE	95	
HEXACHLOROCYCLOPENTADIENE	<8.5	
2,4,6-TRICHLOROPHENOL	<8.5	
2,4,5-TRICHLOROPHENOL	<43	
2-CHLORONAPHTHALENE	<8.5	
2-NITROANILINE	<43	
DIMETHYLPHTHALATE	<8.5	
ACENAPHTHYLENE	4.9	J
3-NITROANILINE	<43	
ACENAPHTHENE	40	
2,4-DINITROPHENOL	<43	
4-NITROPHENOL	<43	

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J = Estimated value.

ATI I.D. # 9101-238-2

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 6A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/30/91
DATE RECEIVED : 01/31/91
DATE EXTRACTED : 02/06/91
DATE ANALYZED : 02/13/91
UNITS : mg/Kg
DILUTION FACTOR : 10

COMPOUND	RESULT	
DIBENZOFURAN	12	
2,4-DINITROTOLUENE	<8.5	
2,6-DINITROTOLUENE	<8.5	
DIETHYLPHTHALATE	<8.5	
4-CHLOROPHENYL-PHENYLETHER	<8.5	
FLUORENE	38	
4-NITROANILINE	<43	
4,6-DINITRO-2-METHYLPHENOL	<43	
N-NITROSODIPHENYLAMINE	<8.5	
4-BROMOPHENYL-PHENYLETHER	<8.5	
HEXACHLOROBENZENE	<8.5	
PENTACHLOROPHENOL	<43	
PHENANTHRENE	100	
ANTHRACENE	33	
DI-N-BUTYLPHTHALATE	<8.5	
FLUORANTHENE	31	
BENZIDINE	<85	
PYRENE	34	
BUTYLBENZYLPHTHALATE	<8.5	
3,3-DICHLOROBENZIDINE	<17	
BENZO(a)ANTHRACENE	12	
BIS(2-ETHYLHEXYL)PHTHALATE	<8.5	
CHRYSENE	14	
DI-N-OCTYLPHTHALATE	<8.5	
BENZO(b)FLUORANTHENE	11	
BENZO(k)FLUORANTHENE	30	
BENZO(a)PYRENE	12	
INDENO(1,2,3-cd)PYRENE	6.2	J
DIBENZ(a,h)ANTHRACENE	<8.5	
BENZO(g,h,i)PERYLENE	6.5	J

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	58
2-FLUOROBIPHENYL	88
TERPHENYL-d14	75
PHENOL-d6	75
2-FLUOROPHENOL	65
2,4,6-TRIBROMOPHENOL	86

J = Estimated value.

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/30/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/31/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/06/91
CLIENT I.D.	: 6A 0-0.3'	DATE ANALYZED	: 02/13/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 10

RESULTS BASED ON DRY WEIGHT

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
UNKNOWN PAH	782	85
NAPHTHALENE, 1,8-DIMETHYL-	877	87
NAPHTHALENE, 1,2-DIMETHYL-	889	80
NAPHTHALENE, 1,5-DIMETHYL-	893	40
NAPHTHALENE, 1,4-DIMETHYL-	908	40

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 11 DUPLICATE
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8270
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/30/91
DATE RECEIVED : 01/31/91
DATE EXTRACTED : 02/06/91
DATE ANALYZED : 02/13/91
UNITS : mg/Kg
DILUTION FACTOR : 10

COMPOUND	RESULT
N-NITROSODIMETHYLAMINE	<8.9
PHENOL	<8.9
ANILINE	<8.9
BIS(2-CHLOROETHYL)ETHER	<8.9
2-CHLOROPHENOL	<8.9
1,3-DICHLOROBENZENE	<8.9
1,4-DICHLOROBENZENE	<8.9
BENZYL ALCOHOL	<8.9
1,2-DICHLOROBENZENE	<8.9
2-METHYLPHENOL	<8.9
BIS(2-CHLOROISOPROPYL)ETHER	<8.9
4-METHYLPHENOL	<8.9
N-NITROSO-DI-N-PROPYLAMINE	<8.9
HEXACHLOROETHANE	<8.9
NITROBENZENE	<8.9
ISOPHORONE	<8.9
2-NITROPHENOL	<8.9
2,4-DIMETHYLPHENOL	<8.9
BENZOIC ACID	<45
BIS(2-CHLOROETHOXY)METHANE	<8.9
2,4-DICHLOROPHENOL	<8.9
1,2,4-TRICHLOROBENZENE	<8.9
NAPHTHALENE	33
4-CHLOROANILINE	<8.9
HEXACHLOROBUTADIENE	<8.9
4-CHLORO-3-METHYLPHENOL	<8.9
2-METHYLNAPHTHALENE	80
HEXACHLOROCYCLOPENTADIENE	<8.9
2,4,6-TRICHLOROPHENOL	<8.9
2,4,5-TRICHLOROPHENOL	<45
2-CHLORONAPHTHALENE	<8.9
2-NITROANILINE	<45
DIMETHYLPHTHALATE	<8.9
ACENAPHTHYLENE	<8.9
3-NITROANILINE	<45
ACENAPHTHENE	33
2,4-DINITROPHENOL	<45
4-NITROPHENOL	<45

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ATI I.D. # 9101-238-3

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/30/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/31/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/06/91
CLIENT I.D.	: 11 DUPLICATE	DATE ANALYZED	: 02/13/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 10

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT	
DIBENZOFURAN	9.9	
2,4-DINITROTOLUENE	<8.9	
2,6-DINITROTOLUENE	<8.9	
DIETHYLPHTHALATE	<8.9	
4-CHLOROPHENYL-PHENYLETHER	<8.9	
FLUORENE	31	
4-NITROANILINE	<45	
4,6-DINITRO-2-METHYLPHENOL	<45	
N-NITROSODIPHENYLAMINE	<8.9	
4-BROMOPHENYL-PHENYLETHER	<8.9	
HEXACHLOROBENZENE	<8.9	
PENTACHLOROPHENOL	<45	
PHENANTHRENE	88	
ANTHRACENE	28	
DI-N-BUTYLPHTHALATE	<8.9	
FLUORANTHENE	40	
BENZIDINE	<89	
PYRENE	36	
BUTYLBENZYLPHTHALATE	<8.9	
3,3-DICHLOROBENZIDINE	<18	
BENZO(a)ANTHRACENE	13	
BIS(2-ETHYLHEXYL)PHTHALATE	<8.9	
CHRYSENE	15	
DI-N-OCTYLPHTHALATE	<8.9	
BENZO(b)FLUORANTHENE	<8.9	
BENZO(k)FLUORANTHENE	29	
BENZO(a)PYRENE	10	
INDENO(1,2,3-cd)PYRENE	4.8	J
DIBENZ(a,h)ANTHRACENE	<8.9	
BENZO(g,h,i)PERYLENE	4.8	J

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	53
2-FLUOROBIPHENYL	80
TERPHENYL-d14	66
PHENOL-d6	65
2-FLUOROPHENOL	56
2,4,6-TRIBROMOPHENOL	76

J = Estimated value.

ATI I.D. # 9101-238-3

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/30/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/31/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/06/91
CLIENT I.D.	: 11 DUPLICATE	DATE ANALYZED	: 02/13/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 10

RESULTS BASED ON DRY WEIGHT

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
UNKNOWN PAH	784	79
NAPHTHALENE, 1,8-DIMETHYL-	879	72
NAPHTHALENE, 1,2-DIMETHYL-	891	67
NAPHTHALENE, 1,5-DIMETHYL-	895	39
NAPHTHALENE, 1,3-DIMETHYL-	910	33

ATI I.D. # 9101-238-8

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT	GEOENGINEERS, INC.	DATE SAMPLED	01/30/91
PROJECT #	1299-003-B04	DATE RECEIVED	01/31/91
PROJECT NAME	UNIMAR, INC.	DATE EXTRACTED	02/06/91
CLIENT I.D.	7A 0-0.3'	DATE ANALYZED	02/13/91
SAMPLE MATRIX	SEDIMENT	UNITS	mg/Kg
EPA METHOD	8270	DILUTION FACTOR	10

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT	
N-NITROSODIMETHYLAMINE	<4.6	
PHENOL	<4.6	
ANILINE	<4.6	
BIS(2-CHLOROETHYL)ETHER	<4.6	
2-CHLOROPHENOL	<4.6	
1,3-DICHLOROBENZENE	<4.6	
1,4-DICHLOROBENZENE	<4.6	
BENZYL ALCOHOL	<4.6	
1,2-DICHLOROBENZENE	<4.6	
2-METHYLPHENOL	<4.6	
BIS(2-CHLOROISOPROPYL)ETHER	<4.6	
4-METHYLPHENOL	<4.6	
N-NITROSO-DI-N-PROPYLAMINE	<4.6	
HEXACHLOROETHANE	<4.6	
NITROBENZENE	<4.6	
ISOPHORONE	<4.6	
2-NITROPHENOL	<4.6	
2,4-DIMETHYLPHENOL	<4.6	
BENZOIC ACID	<23	
BIS(2-CHLOROETHOXY)METHANE	<4.6	
2,4-DICHLOROPHENOL	<4.6	
1,2,4-TRICHLOROBENZENE	<4.6	
NAPHTHALENE	2.6	J
4-CHLOROANILINE	<4.6	
HEXACHLOROBUTADIENE	<4.6	
4-CHLORO-3-METHYLPHENOL	<4.6	
2-METHYLNAPHTHALENE	2.4	J
HEXACHLOROCYCLOPENTADIENE	<4.6	
2,4,6-TRICHLOROPHENOL	<4.6	
2,4,5-TRICHLOROPHENOL	<23	
2-CHLORONAPHTHALENE	<4.6	
2-NITROANILINE	<23	
DIMETHYLPHTHALATE	<4.6	
ACENAPHTHYLENE	<4.6	
3-NITROANILINE	<23	
ACENAPHTHENE	8.3	
2,4-DINITROPHENOL	<23	
4-NITROPHENOL	<23	

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J = Estimated value.

ATI I.D. # 9101-238-8

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/30/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/31/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/06/91
CLIENT I.D.	: 7A 0-0.3'	DATE ANALYZED	: 02/13/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 10

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
DIBENZOFURAN	<4.6
2,4-DINITROTOLUENE	<4.6
2,6-DINITROTOLUENE	<4.6
DIETHYLPHTHALATE	<4.6
4-CHLOROPHENYL-PHENYLETHER	<4.6
FLUORENE	8.9
4-NITROANILINE	<23
4,6-DINITRO-2-METHYLPHENOL	<23
N-NITROSODIPHENYLAMINE	<4.6
4-BROMOPHENYL-PHENYLETHER	<4.6
HEXACHLOROBENZENE	<4.6
PENTACHLOROPHENOL	<23
PHENANTHRENE	27
ANTHRACENE	9.6
DI-N-BUTYLPHTHALATE	<4.6
FLUORANTHENE	24
BENZIDINE	<46
PYRENE	25
BUTYLBENZYLPHTHALATE	<4.6
3,3-DICHLOROBENZIDINE	<9.2
BENZO(a)ANTHRACENE	9.1
BIS(2-ETHYLHEXYL)PHTHALATE	<4.6
CHRYSENE	15
DI-N-OCTYLPHTHALATE	<4.6
BENZO(b)FLUORANTHENE	11
BENZO(k)FLUORANTHENE	33
BENZO(a)PYRENE	12
INDENO(1,2,3-cd)PYRENE	7.5
DIBENZ(a,h)ANTHRACENE	<4.6
BENZO(g,h,i)PERYLENE	8.8

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	64
2-FLUOROBIPHENYL	87
TERPHENYL-d14	72
PHENOL-d6	73
2-FLUOROPHENOL	63
2,4,6-TRIBROMOPHENOL	82

ATI I.D. # 9101-238-8

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/30/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/31/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/06/91
CLIENT I.D.	: 7A 0-0.3'	DATE ANALYZED	: 02/13/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 10

RESULTS BASED ON DRY WEIGHT

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
UNKNOWN PAH	782	11
NAPHTHALENE, 1,3-DIMETHYL-	876	14
NAPHTHALENE, 1,5-DIMETHYL-	889	14
DODECANE, 2,7,10-TRIMETHYL-	1131	47
HEPTADECANE, 2,6,10,15-TETRAMETHYL-	1282	12

SEMI-VOLATILE ORGANIC
QUALITY CONTROL DATA

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
EPA METHOD : 8270

SAMPLE I.D. : BLANK SPIKE
DATE EXTRACTED : 02/06/91
DATE ANALYZED : 02/12/91
MATRIX : SOIL
UNITS : mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
1,2,4-TRICHLOROBENZENE	<0.17	3.33	2.05	61	2.26	68	10
ACENAPHTHENE	<0.17	3.33	1.52	46	1.58	47	4
2,4-DINITROTOLUENE	<0.17	3.33	1.51	45	1.60	48	6
PYRENE	<0.17	3.33	1.68	50	1.77	53	5
N-NITROSO-DI-N-PROPYLAMINE	<0.17	3.33	2.07	62	2.14	64	3
1,4-DICHLOROBENZENE	<0.17	3.33	1.85	56	1.95	59	5
PENTACHLOROPHENOL	<0.85	6.67	7.03	105	7.75	116*	10
PHENOL	<0.17	6.67	4.03	60	3.86	58	4
2-CHLOROPHENOL	<0.17	6.67	3.98	60	4.07	61	2
4-CHLORO-3-METHYLPHENOL	<0.17	6.67	3.99	60	3.96	59	1
4-NITROPHENOL	<0.85	6.67	6.40	96	6.75	101	5

* Out of limits.

$$\text{\% Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-238

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : WATER
EPA METHOD : 8080 (PCB)

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 02/05/91
DATE ANALYZED : 02/13/91
UNITS : ug/L
DILUTION FACTOR : 1

COMPOUND	RESULT
PCB 1016	<1.0
PCB 1221	<1.0
PCB 1232	<1.0
PCB 1242	<1.0
PCB 1248	<1.0
PCB 1254	<1.0
PCB 1260	<1.0

ATI I.D. # 9101-238-5

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/30/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/31/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/05/91
CLIENT I.D.	: VAN VEEN RINSEATE 10D	DATE ANALYZED	: 02/13/91
SAMPLE MATRIX	: WATER	UNITS	: ug/L
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 1

COMPOUND	RESULT
PCB 1016	<1.0
PCB 1221	<1.0
PCB 1232	<1.0
PCB 1242	<1.0
PCB 1248	<1.0
PCB 1254	<1.0
PCB 1260	<1.0

ATI I.D. # 9101-238-6

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : SHELBY RINSEATE 10B
SAMPLE MATRIX : WATER
EPA METHOD : 8080 (PCB)

DATE SAMPLED : 01/30/91
DATE RECEIVED : 01/31/91
DATE EXTRACTED : 02/05/91
DATE ANALYZED : 02/13/91
UNITS : ug/L
DILUTION FACTOR : 1

COMPOUND	RESULT
PCB 1016	<1.0
PCB 1221	<1.0
PCB 1232	<1.0
PCB 1242	<1.0
PCB 1248	<1.0
PCB 1254	<1.0
PCB 1260	<1.0

ATI I.D. # 9101-238

POLYCHLORINATED BIPHENYLS (PCB)
QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
EPA METHOD : 8080 (PCB)

SAMPLE ID : 9101-238-5
DATE EXTRACTED : 02/05/91
DATE ANALYZED : 02/13/91
MATRIX : WATER
UNITS : ug/L

COMPOUND	SAMPLE RESULT	CONC SPIKED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % RECOVERY	RPD
PCB 1260	<1.0	9.3	11.2	120	10.6	114	6

$$\% \text{ Recovery} = \frac{(\text{Spike Sample result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-238

POLYCHLORINATED BIPHENYLS (PCB)
QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
EPA METHOD : 8080 (PCB)

SAMPLE ID : BLANK SPIKE
DATE EXTRACTED : 02/05/91
DATE ANALYZED : 02/13/91
MATRIX : WATER
UNITS : ug/L

COMPOUND	SAMPLE RESULT	CONC SPIKED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % RECOVERY	RPD
PCB 1260	<1.0	10	11.2	112	N/A	N/A	N/A

$$\% \text{ Recovery} = \frac{(\text{Spike Sample result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-238

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/05/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/13/91
SAMPLE MATRIX	: SOIL	UNITS	: mg/Kg
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
PCB 1016	<0.033
PCB 1221	<0.033
PCB 1232	<0.033
PCB 1242	<0.033
PCB 1248	<0.033
PCB 1254	<0.033
PCB 1260	<0.033

ATI I.D. # 9101-238-1

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/29/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/31/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/06/91
CLIENT I.D.	: 5A 0-0.3'	DATE ANALYZED	: 02/13/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 1

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
PCB 1016	<0.22
PCB 1221	<0.22
PCB 1232	<0.22
PCB 1242	<0.22
PCB 1248	<0.22
PCB 1254	<0.22
PCB 1260	<0.22

ATI I.D. # 9101-238-2

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 6A 0-0.3′
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8080 (PCB)
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/30/91
DATE RECEIVED : 01/31/91
DATE EXTRACTED : 02/06/91
DATE ANALYZED : 02/13/91
UNITS : mg/Kg
DILUTION FACTOR : 25 *

COMPOUND	RESULT
PCB 1016	<4.1
PCB 1221	<4.1
PCB 1232	<4.1
PCB 1242	<4.1
PCB 1248	<4.1
PCB 1254	<4.1
PCB 1260	<4.1

* Dilution factor necessary due to sample matrix.

ATI I.D. # 9101-238-3

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/30/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/31/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/06/91
CLIENT I.D.	: 11 DUPLICATE	DATE ANALYZED	: 02/13/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 25 *

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
PCB 1016	<4.3
PCB 1221	<4.3
PCB 1232	<4.3
PCB 1242	<4.3
PCB 1248	<4.3
PCB 1254	<4.3
PCB 1260	<4.3

* Dilution factor necessary due to sample matrix.

ATI I.D. # 9101-238-8

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/30/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/31/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/06/91
CLIENT I.D.	: 7A 0-0.3'	DATE ANALYZED	: 02/13/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8080 (PCB)	DILUTION FACTOR	: 25 *

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
PCB 1016	<2.2
PCB 1221	<2.2
PCB 1232	<2.2
PCB 1242	<2.2
PCB 1248	<2.2
PCB 1254	<2.2
PCB 1260	<2.2

* Dilution factor necessary due to sample matrix.

POLYCHLORINATED BIPHENYLS (PCB)
QUALITY CONTROL

CLIENT	: GEOENGINEERS, INC.	SAMPLE ID	: BLANK SPIKE
PROJECT #	: 1299-003-B04	DATE EXTRACTED	: 02/06/91
PROJECT NAME	: UNIMAR, INC.	DATE ANALYZED	: 02/13/91
EPA METHOD	: 8080 (PCB)	MATRIX	: SOIL
		UNITS	: mg/Kg

COMPOUND	SAMPLE RESULT	CONC SPIKED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % RECOVERY	RPD
PCB 1260	<0.033	0.33	0.376	114	0.375	114	0

$$\% \text{ Recovery} = \frac{(\text{Spike Sample result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

POLYNUCLEAR AROMATICS ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: N/A
PROJECT #	: 1299-003-B04	DATE RECEIVED	: N/A
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/05/91
CLIENT I.D.	: REAGENT BLANK	DATE ANALYZED	: 02/12/91
SAMPLE MATRIX	: WATER	UNITS	: ug/L
EPA METHOD	: 8310	DILUTION FACTOR	: 1

COMPOUND	RESULT
NAPHTHALENE	<0.50
ACENAPHTHYLENE	<1.0
ACENAPHTHENE	<1.0
FLUORENE	<0.10
PHENANTHRENE	<0.05
ANTHRACENE	<0.05
FLUORANTHENE	<0.10
PYRENE	<0.10
BENZO(a)ANTHRACENE	<0.10
CHRYSENE	<0.10
BENZO(b)FLUORANTHENE	<0.10
BENZO(k)FLUORANTHENE	<0.10
BENZO(a)PYRENE	<0.10
DIBENZ(a,h)ANTHRACENE	<0.20
BENZO(g,h,i)PERYLENE	<0.10
INDENO(1,2,3-cd)PYRENE	<0.10

ATI I.D. # 9101-238-5

POLYNUCLEAR AROMATICS ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : VAN VEEN RINSEATE 10D
SAMPLE MATRIX : WATER
EPA METHOD : 8310

DATE SAMPLED : 01/30/91
DATE RECEIVED : 01/31/91
DATE EXTRACTED : 02/05/91
DATE ANALYZED : 02/12/91
UNITS : ug/L
DILUTION FACTOR : 1

COMPOUND	RESULT
NAPHTHALENE	<0.50
ACENAPHTHYLENE	<1.0
ACENAPHTHENE	<1.0
FLUORENE	<0.10
PHENANTHRENE	<0.05
ANTHRACENE	<0.05
FLUORANTHENE	<0.10
PYRENE	<0.10
BENZO(a)ANTHRACENE	<0.10
CHRYSENE	<0.10
BENZO(b)FLUORANTHENE	<0.10
BENZO(k)FLUORANTHENE	<0.10
BENZO(a)PYRENE	<0.10
DIBENZ(a,h)ANTHRACENE	<0.20
BENZO(g,h,i)PERYLENE	<0.10
INDENO(1,2,3-cd)PYRENE	<0.10

ATI I.D. # 9101-238-6

POLYNUCLEAR AROMATICS ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/30/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/31/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/05/91
CLIENT I.D.	: SHELBY RINSEATE 10B	DATE ANALYZED	: 02/12/91
SAMPLE MATRIX	: WATER	UNITS	: ug/L
EPA METHOD	: 8310	DILUTION FACTOR	: 1

COMPOUND	RESULT
NAPHTHALENE	<0.50
ACENAPHTHYLENE	<1.0
ACENAPHTHENE	<1.0
FLUORENE	<0.10
PHENANTHRENE	<0.05
ANTHRACENE	<0.05
FLUORANTHENE	<0.10
PYRENE	<0.10
BENZO(a)ANTHRACENE	<0.10
CHRYSENE	<0.10
BENZO(b)FLUORANTHENE	<0.10
BENZO(k)FLUORANTHENE	<0.10
BENZO(a)PYRENE	<0.10
DIBENZ(a,h)ANTHRACENE	<0.20
BENZO(g,h,i)PERYLENE	<0.10
INDENO(1,2,3-cd)PYRENE	<0.10

POLYNUCLEAR AROMATICS
QUALITY CONTROL DATA

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
EPA METHOD : 8310

SAMPLE I.D. : 9101-238-6
DATE EXTRACTED : 02/05/91
DATE ANALYZED : 02/12/91
MATRIX : WATER
UNITS : ug/L

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
ACENAPHTHYLENE	<1.0	99.9	50	50	62	62	21
PHENANTHRENE	<0.05	10.6	7.4	70	7.9	75	7
PYRENE	<0.10	10.5	9.7	92	9.6	91	1
BENZO(k)FLUORANTHENE	<0.10	9.52	7.4	78	6.7	70	10

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\underset{\text{Result}}{\text{Spiked Sample}} - \underset{\text{Sample Result}}{\text{Duplicate Spike}})}{\text{Average of Spiked Sample}} \times 100$$

POLYNUCLEAR AROMATICS
QUALITY CONTROL DATA

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
EPA METHOD : 8310

SAMPLE I.D. : BLANK SPIKE
DATE EXTRACTED : 02/05/91
DATE ANALYZED : 02/12/91
MATRIX : WATER
UNITS : ug/L

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
ACENAPHTHYLENE	<1.0	99.9	51	51	N/A	N/A	N/A
PHENANTHRENE	<0.05	10.6	7.4	70	N/A	N/A	N/A
PYRENE	<0.10	10.5	9.9	94	N/A	N/A	N/A
BENZO(k)FLUORANTHENE	<0.10	9.52	7.1	75	N/A	N/A	N/A

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-238

POLYNUCLEAR AROMATICS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SOIL
EPA METHOD : 8310
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 02/07/91
DATE ANALYZED : 02/16/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
NAPHTHALENE	<0.083
ACENAPHTHYLENE	<0.17
ACENAPHTHENE	<0.17
FLUORENE	<0.017
PHENANTHRENE	<0.0083
ANTHRACENE	<0.0083
FLUORANTHENE	<0.017
PYRENE	<0.017
BENZO(a)ANTHRACENE	<0.017
CHRYSENE	<0.017
BENZO(b)FLUORANTHENE	<0.017
BENZO(k)FLUORANTHENE	<0.017
BENZO(a)PYRENE	<0.017
DIBENZ(a,h)ANTHRACENE	<0.034
BENZO(g,h,i)PERYLENE	<0.017
INDENO(1,2,3-cd)PYRENE	<0.017

ATI I.D. # 9101-238-4

POLYNUCLEAR AROMATICS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/30/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/31/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 02/07/91
CLIENT I.D.	: 6B 0.3-5.0'	DATE ANALYZED	: 02/20/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8310	DILUTION FACTOR	: 20

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
NAPHTHALENE	120
ACENAPHTHYLENE	50
ACENAPHTHENE	150
FLUORENE	53
PHENANTHRENE	160
ANTHRACENE	44
FLUORANTHENE	47
PYRENE	47
BENZO(a)ANTHRACENE	13
CHRYSENE	22
BENZO(b)FLUORANTHENE	7.4
BENZO(k)FLUORANTHENE	4.7
BENZO(a)PYRENE	13
DIBENZ(a,h)ANTHRACENE	6.5
BENZO(g,h,i)PERYLENE	6.5
INDENO(1,2,3-cd)PYRENE	6.2

ATI I.D. # 9101-238-9

POLYNUCLEAR AROMATICS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 7B 0.3-5.0'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8310
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/30/91
DATE RECEIVED : 01/31/91
DATE EXTRACTED : 02/07/91
DATE ANALYZED : 02/20/91
UNITS : mg/Kg
DILUTION FACTOR : 10

COMPOUND	RESULT
NAPHTHALENE	4.2
ACENAPHTHYLENE	16
ACENAPHTHENE	68
FLUORENE	25
PHENANTHRENE	97
ANTHRACENE	26
FLUORANTHENE	60
PYRENE	34
BENZO(a)ANTHRACENE	13
CHRYSENE	16
BENZO(b)FLUORANTHENE	14
BENZO(k)FLUORANTHENE	7.4
BENZO(a)PYRENE	24
DIBENZ(a,h)ANTHRACENE	5.8
BENZO(g,h,i)PERYLENE	12
INDENO(1,2,3-cd)PYRENE	16

POLYNUCLEAR AROMATICS
QUALITY CONTROL DATA

CLIENT	: GEOENGINEERS, INC.		SAMPLE I.D.	: 102083-06
PROJECT #	: 1299-003-B04		DATE EXTRACTED	: 02/07/91
PROJECT NAME	: UNIMAR, INC.		DATE ANALYZED	: 02/16/91
EPA METHOD	: 8310		MATRIX	: SOIL
			UNITS	: mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
ACENAPHTHYLENE	<0.17	18.2	14	77	14	77	0
PHENANTHRENE	0.28	1.91	2.5	116	2.2	101	13
PYRENE	0.039	2.43	2.0	81	2.0	81	0
BENZO(k)FLUORANTHENE	<0.17	1.87	1.5	80	1.6	86	6

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-238

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : ng/L

PARAMETER	BETA RINSEATE 10F -7
MONOBUTYLTIN	15
DIBUTYLTIN	4
TRIBUTYLTIN	ND
TETRABUTYLTIN	ND

ND - None detected.

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : WATER

ELEMENT	DATE PREPARED	DATE ANALYZED
ARSENIC	02/01/91	02/17/91
BARIUM	02/01/91	02/28/91
CADMIUM	02/01/91	02/26/91
CHROMIUM	02/01/91	02/28/91
COPPER	02/01/91	02/01/91
COPPER	02/01/91	02/28/91
LEAD	02/01/91	02/26/91
MERCURY	02/20/91	02/20/91
NICKEL	02/01/91	02/28/91
SELENIUM	02/01/91	02/17/91
SILVER	02/01/91	02/28/91
ZINC	02/01/91	02/28/91

ATI I.D. # 9101-238

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

PARAMETER	VAN VEEN RINSEATE 10D -5	SHELBY RINSEATE 10B -6	BETA RINSEATE 10F -7	REAGENT BLANK -
ARSENIC	<0.005	<0.005	<0.005	<0.005
BARIUM	<0.02	<0.02	<0.02	<0.02
CADMIUM	<0.0003	<0.0003	<0.0003	<0.0003
CHROMIUM	<0.02	<0.02	<0.02	<0.02
COPPER	<0.02	0.10	<0.02	<0.02
LEAD	<0.005	0.009	<0.005	<0.005
MERCURY	<0.0005	<0.0005	<0.0005	<0.0005
NICKEL	<0.01	<0.01	<0.01	<0.01
SELENIUM	<0.005	<0.005	<0.005	<0.005
SILVER	<0.03	<0.03	<0.03	<0.03
ZINC	<0.01	<0.01	<0.01	<0.01

METALS QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

COMPOUND	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED SAMPLE	SPIKE CONC	% REC
ARSENIC	9101-238-7	<0.005	<0.005	0	0.023	0.025	92
BARIUM	9101-238-7	<0.02	<0.02	0	10.6	10.0	106
CADMIUM	9101-238-7	<0.0003	0.0006	0	0.0010	0.0010	100
CHROMIUM	9101-238-7	<0.02	<0.02	0	2.59	2.50	104
COPPER	9101-174-2	0.65	0.59	10	3.04	2.50	96
LEAD	9101-238-7	<0.005	<0.005	0	0.027	0.025	108
MERCURY	9102-069-4	<0.0005	<0.0005	0	0.0030	0.0020	150
MERCURY	BLANK SPIKE	N/A	N/A	N/A	0.0025	0.0020	125
NICKEL	9101-238-7	<0.01	<0.01	0	2.62	2.50	105
SELENIUM	9101-238-7	<0.005	<0.005	0	0.026	0.025	104
SILVER	9101-238-7	<0.03	<0.03	0	11.8	10.0	118
ZINC	9101-238-7	0.11	0.12	9	2.88	2.50	111

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

ATI # 9101-238

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : SEDIMENT

ELEMENT	DATE PREPARED	DATE ANALYZED
ARSENIC	02/15/91	02/28/91
BARIUM	02/15/91	02/26/91
CADMIUM	02/15/91	02/26/91
CHROMIUM	02/15/91	02/26/91
COPPER	02/15/91	02/26/91
LEAD	02/15/91	02/26/91
MERCURY	02/22/91	02/22/91
NICKEL	02/15/91	02/26/91
SELENIUM	02/15/91	02/15/91
SILVER	02/15/91	02/26/91
ZINC	02/15/91	02/26/91

ATI I.D. # 9101-238

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

PARAMETER	5A 0-0.3′ -1	6A 0-0.3′ -2	11 DUPLICATE -3	6B 0.3-5.0′ -4
ARSENIC	190	190	150	67
BARIUM	130	180	170	110
CADMIUM	17	19	16	10
CHROMIUM	78	76	77	57
COPPER	610	1,200	850	252
LEAD	620	500	480	230
MERCURY	1.52	<0.80	0.83	<0.80
NICKEL	75	69	78	57
SELENIUM	<2.0	<2.0	<2.0	<2.0
SILVER	<2	<2	<2	<2
ZINC	1,600	1,700	1,400	430

ATI I.D.# 9101-238

METALS RESULTS

CLIENT	: GEOENGINEERS, INC.	MATRIX	: SEDIMENT
PROJECT #	: 1299-003-B04		
PROJECT NAME	: UNIMAR, INC.	UNITS	: mg/Kg

PARAMETER	7A 0-0.3' -8	7B 0.3-5.0' -9	REAGENT BLANK
ARSENIC	190	27	<0.5
BARIUM	130	90	<1
CADMIUM	13	8.0	<1
CHROMIUM	59	44	<1
COPPER	540	130	3.7
LEAD	470	170	<2.5
MERCURY	<0.80	<0.80	<0.15
NICKEL	74	51	<1
SELENIUM	<2.0	<2.0	<0.5
SILVER	<2	<2	<2
ZINC	1,000	270	2.8

METALS QUALITY CONTROL

CLIENT	: GEOENGINEERS, INC.	MATRIX	: SEDIMENT/SOIL
PROJECT #	: 1299-003-B04		
PROJECT NAME	: UNIMAR, INC.	UNITS	: mg/Kg

COMPOUND	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED SAMPLE	SPIKE CONC	% REC
ARSENIC	9101-238-9	27	27	0	**	**	**
ARSENIC	BLANK SPIKE	N/A	N/A	N/A	2.0	2.5	80
ARSENIC	BLANK SPIKE	N/A	N/A	N/A	2.1	2.5	84
BARIUM	9101-238-9	90	86	4	1,440	1,380	98
CADMIUM	9101-238-9	8.0	7.6	5	70	69	90
CHROMIUM	9101-238-9	44	42	5	372	345	95
COPPER	9101-238-9	130	120	8	450	345	93
LEAD	9101-238-9	170	170	0	836	690	96
MERCURY	9102-090-5	<0.15	<0.15	0	0.33	0.49	67
NICKEL	9101-238-9	51	48	6	380	345	93
SELENIUM	9101-238-9	<0.5	<0.5	0	3.5	3.4	103
SILVER	9101-238-9	<2	<2	0	58	69	84
ZINC	9101-238-9	270	260	4	591	345	95

** Due to the necessary dilution of the sample, result was not attainable.

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

ATI I.D. # 9101-238

GENERAL CHEMISTRY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : WATER

ANALYSIS	DATE PREPARED	DATE ANALYZED
PETROLEUM HYDROCARBONS	02/04/91	02/04/91

GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

ATI I.D. #	CLIENT I.D.	PETROLEUM HYDROCARBONS
9101-238-5	VAN VEEN RINSEATE 10D	<1
9101-238-6	SHELBY RINSEATE 10B	<1
REAGENT BLANK	-	<1

ATI I.D. # 9101-238

GENERAL CHEMISTRY QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

SAMPLE MATRIX : WATER

PARAMETER	UNITS	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED RESULT	SPIKE ADDED	% REC
PETROLEUM HYDROCARBONS	mg/L	9102-037-1	<1	<1	0	4.98	9.96	50

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

ATI I.D. # 9101-238

GENERAL CHEMISTRY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : SEDIMENT

ANALYSIS	DATE PREPARED	DATE ANALYZED
PETROLEUM HYDROCARBONS	02/04/91	02/04/91
MOISTURE	-	02/02/91

ATI I.D. # 9101-238

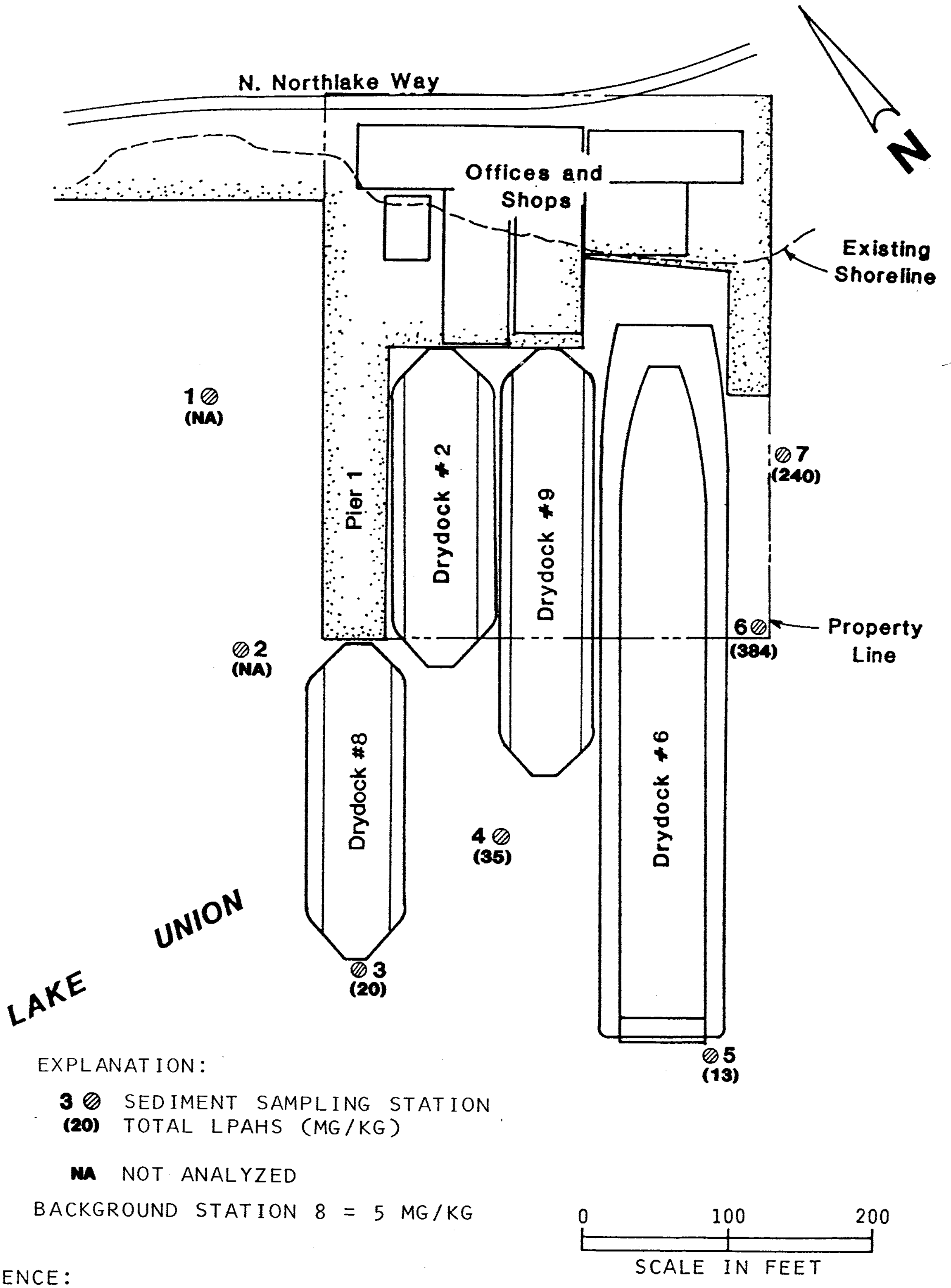
GENERAL CHEMISTRY RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

ATI I.D.#	CLIENT I.D.	PETROLEUM HYDROCARBONS
9101-238-1	5A 0-0.3'	780
9101-238-2	6A 0-0.3'	200
9101-238-3	11 DUPLICATE	110
9101-238-4	6B 0.3-5.0'	130
9101-238-8	7A 0-0.3'	160
9101-238-9	7B 0.3-5.0'	190
REAGENT BLANK	-	<5



EXPLANATION:

3 ▨ (20) SEDIMENT SAMPLING STATION TOTAL LPAHS (MG/KG)

NA NOT ANALYZED

BACKGROUND STATION 8 = 5 MG/KG

REFERENCE:

UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

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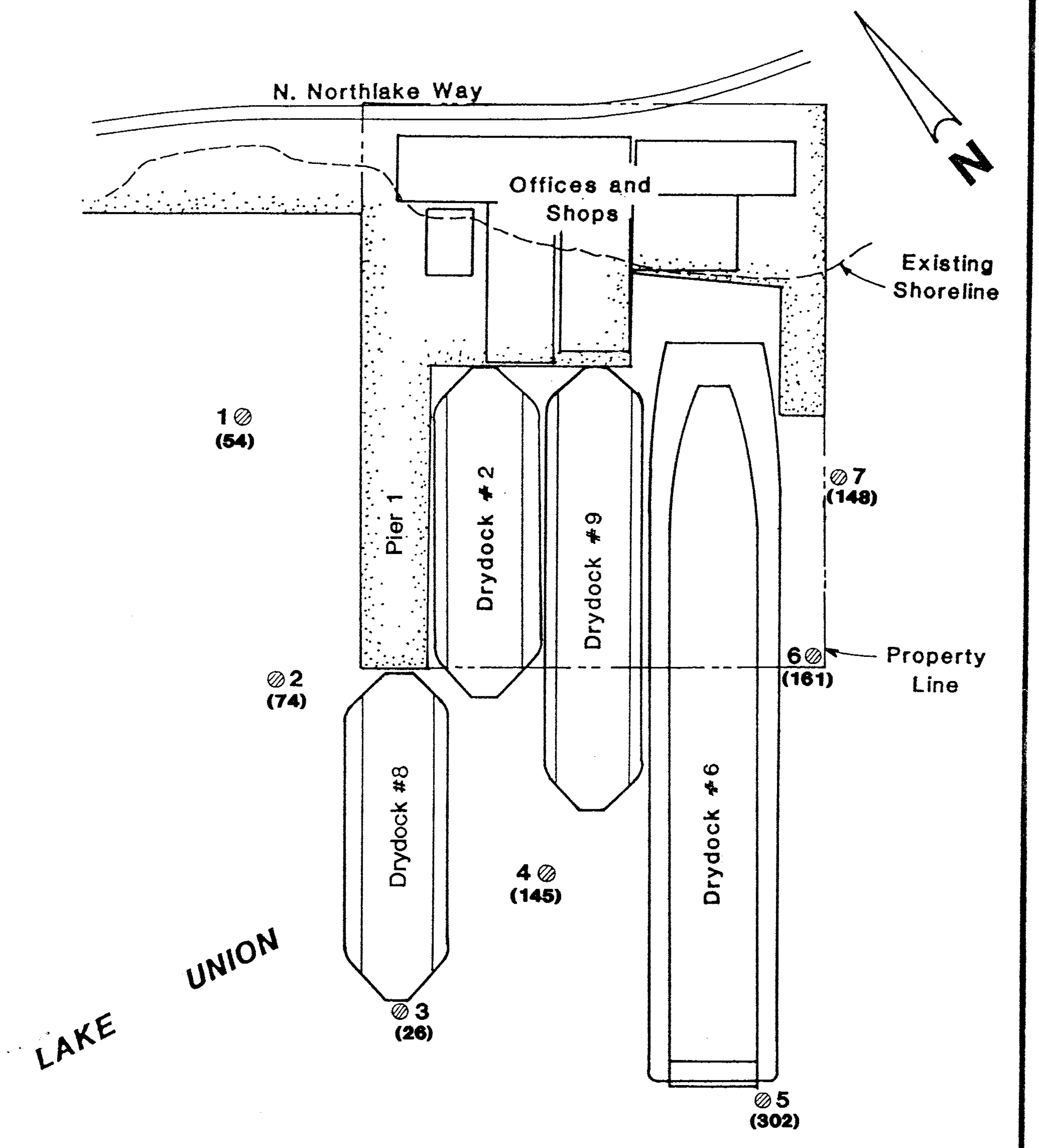
TOTAL LPAHS, 0.3–5.0 FEET INTERVAL

FIGURE 16

6-26-91 LJD
7-23-91 RRR



EXPLANATION:

1 ▨	SEDIMENT SAMPLING STATION
(54)	TOTAL HPAHS (MG/KG)

BACKGROUND STATION 8 = 211 MG/KG

REFERENCE:

UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

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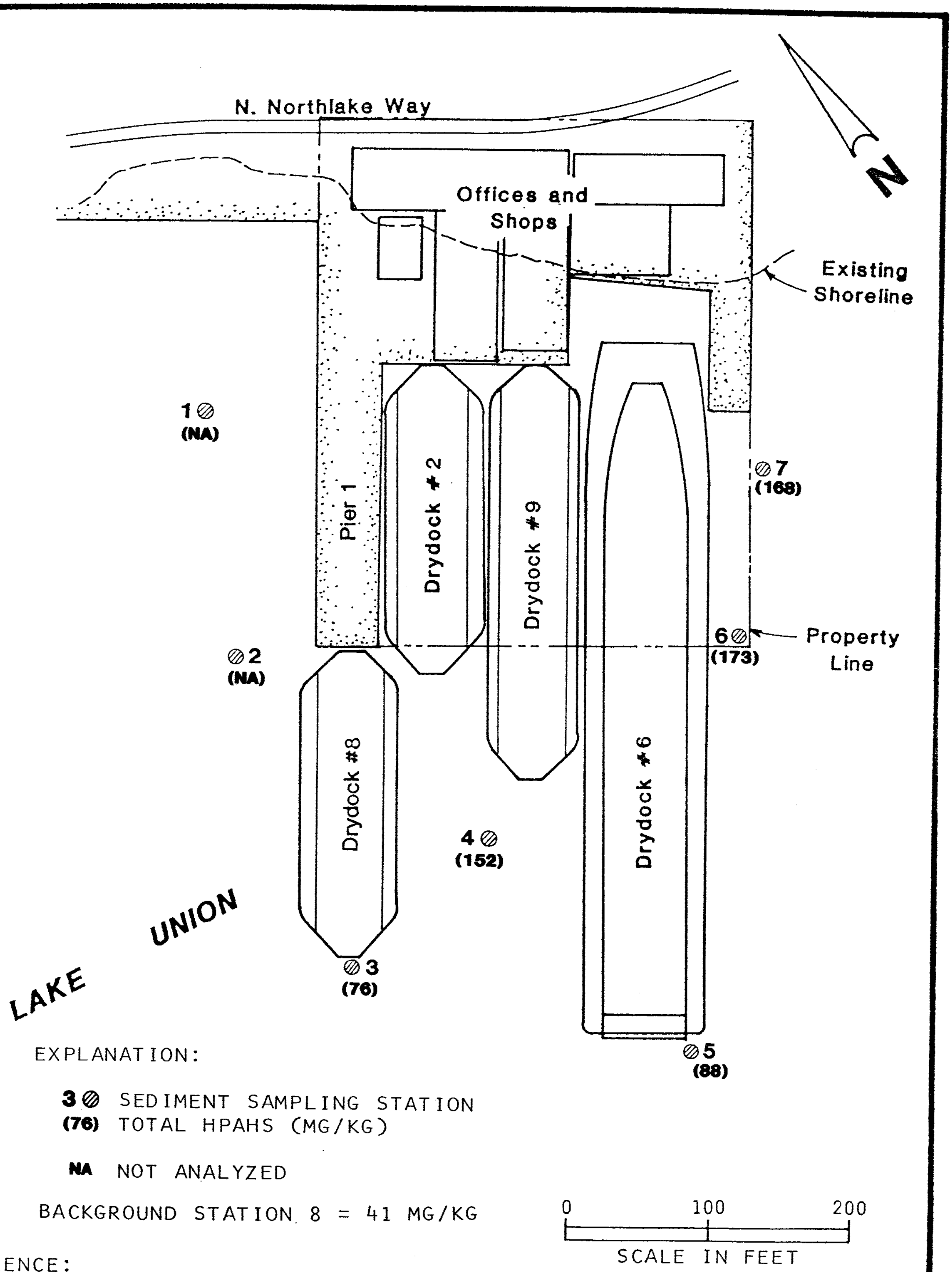
TOTAL HPAHS, 0–0.3 FEET INTERVAL

FIGURE 17

6-26-91 LJD
9-23-91 RRR



EXPLANATION:

3 ▨ SEDIMENT SAMPLING STATION
(76) TOTAL HPAHS (MG/KG)

NA NOT ANALYZED

BACKGROUND STATION 8 = 41 MG/KG

REFERENCE:
UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

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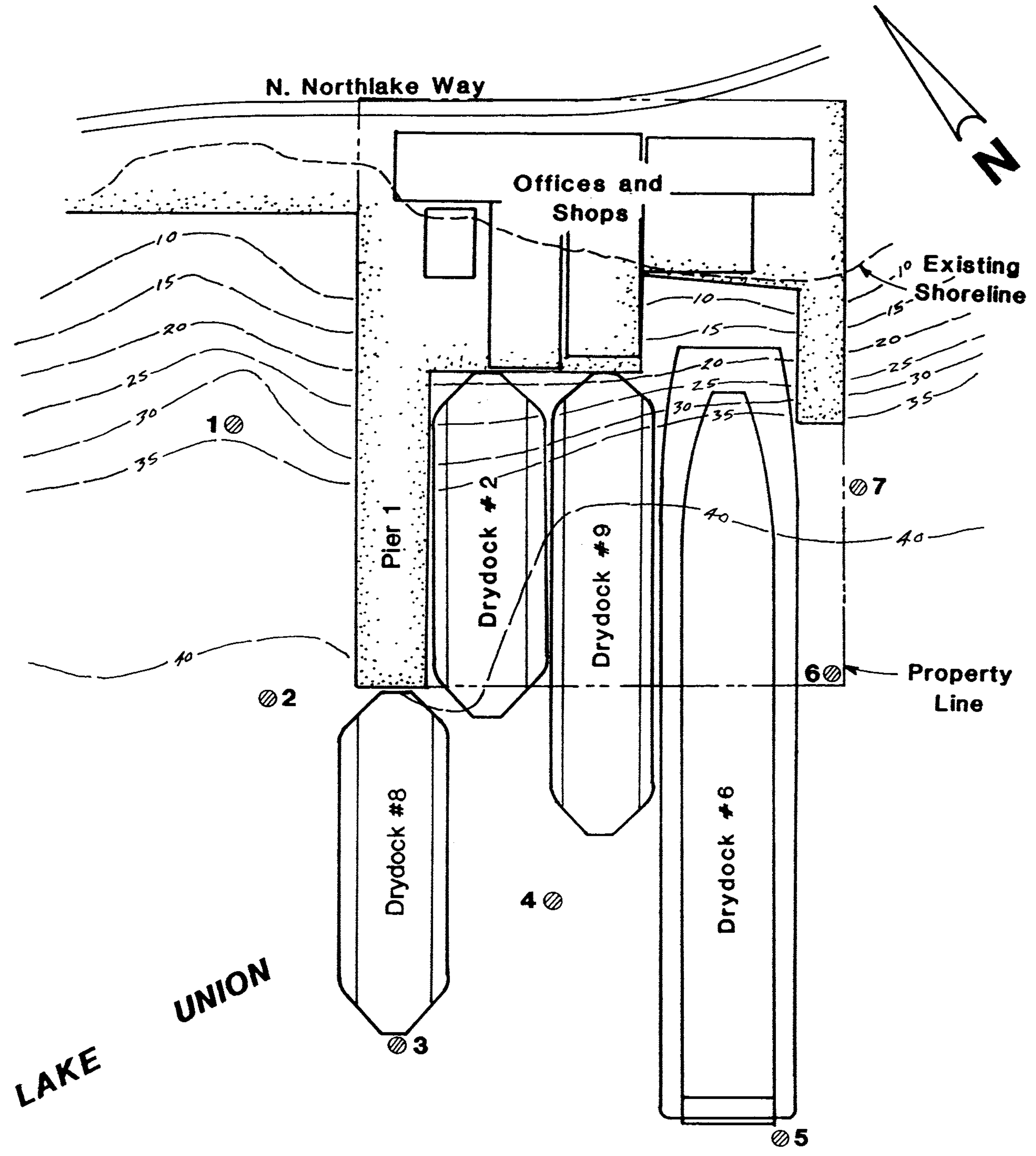
TOTAL HPAHS, 0.3-5.0 FEET INTERVAL

FIGURE 18

-03 CEW:KVT 11-
Re 6-8 KT
6-26-91 LJD
7-23-91 RRR



EXPLANATION:

1 ▨ SEDIMENT SAMPLING STATION

—10— DEPTH OF WATER CONTOUR (FEET)

REFERENCE:

UNTITLED DRAWING PROVIDED BY MARINE POWER & EQUIPMENT, INC., DATED 1-12-86.

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WATER DEPTH MAP

FIGURE 19

REV. 1-6-91 NN
6-26-91 LJD
7-23-91 RRR

APPENDIX A

FIELD PROGRAM

APPENDIX A

FIELD PROGRAM

SEDIMENT SAMPLING

Sediment core and benthic samples were collected at stations 1 through 9 for chemical analyses and descriptive logging between January 21 and January 30, 1991. The deep sediment core samples were collected by scuba divers who hand-drove 5-foot Shelby Tube (stainless steel core tube) samplers into the lake bottom sediments. The benthic and shallow sediment samples were collected from a stainless steel Van Veen grab sampler.

The divers used one of two techniques to drive the samplers 5 feet into the bottom sediments. One method was to push the tube into soft sediments by hand and the other method involved using a 10-pound drive hammer for more competent materials. After the Shelby Tubes were driven to the required sample depth, the sample tube was slowly retrieved from the lake bottom with a hand-winch or by hand with a rope. The diver maintained constant communications with workers at the surface throughout the entire process. Immediately upon retrieval from the lake bottom, the diver capped both ends of the tube with tight-fitting plastic endcaps and temporarily sealed the endcaps with self-adhering PVC (polyvinyl chloride) tape. The sample tube was raised to the surface where the core recovery was checked with a tape measure. Acceptable core recovery was predetermined to be 3.5 feet or greater.

The core samples were then extruded into aluminum foil-lined troughs for descriptive logging. The sediment core was measured, photographed and divided into discrete intervals for sampling. Sediment samples were collected from the core, place into the appropriately labeled bottles, and kept cold in insulated containers during transport to the testing laboratory. The transport containers were sealed with chain of custody seals and the appropriate chain of custody paperwork accompanied the sample bottles to the testing laboratory.

The sediment volume required for the chemical analyses in the shallow interval (0 to 0.3 feet) was greater than the volume contained in the shallow sample interval. The sample volume was supplemented with sediment obtained from the Van Veen composite sample as specified in the QA/QC plan. Samples were analyzed for the test methods specified in Table 3 of the QA/QC plan. The analytical reports are given in Appendix C.

BETA WATER SAMPLING

A total of three Beta water samples, 8A, 2A and 12, were collected for chemical analyses. Water sample 12 was collected as a duplicate of sample 2A at station 2. Prior to sampling at each location, the water depth was measured with a weighted fiberglass tape and the sample depth was recorded in the field notes. The water sample depth was marked (1 foot minus the water depth) on the messenger line of the Beta sampler, the sample was slowly lowered into position and the sampler was triggered to close with the messenger weight. The sampler was brought to the surface where the water samples were placed into the appropriate bottles, the pH of the preserved samples were verified with test paper and the samples were placed in cold, insulated transport containers. All chain of custody procedures were followed for submitting the samples to the testing laboratory. Samples were analyzed for the testing methods specified in Table 3 of the QA/QC plan. The analytical reports are given in Appendix C.

FIELD WATER QUALITY MEASUREMENTS

The temperature, pH, electrical conductivity and dissolved oxygen of the Beta water samples were measured with an Orion pH meter, a YSI temperature and conductivity meter and a YSI dissolved oxygen meter. The dissolved oxygen measurements were confirmed in the field by manual titration. The calibration of the pH and conductivity sensors was checked against standard solutions prior to measurements. The water sample measurements were recorded in triplicate and averaged for each parameter. The field water quality results are presented in Table 5.

DECONTAMINATION PROCEDURES

All sampling equipment was decontaminated between obtaining samples from sampling stations and between each sampling attempt for the core samples. The equipment was decontaminated by steam-cleaning to remove gross contamination, washing with a trisodium phosphate wash, rinsing with tap water, rinsing with distilled water, rinsing with methanol and finally rinsing thoroughly with distilled water. Shelby Tubes were then wrapped in aluminum foil for storage.

RINSEATE BLANKS

Rinseate (equipment) blanks were collected by spraying distilled water over each piece of sampling equipment and submitting the water samples to the testing laboratory. All chain of custody procedures were followed for submitting the rinseate blanks to the testing laboratory. Samples were analyzed for the testing methods specified in Table 3 of the QA/QC plan. The analytical reports are given in Appendix C.

APPENDIX B

SEDIMENT CORE LOGS

SOIL CLASSIFICATION SYSTEM

MAJOR DIVISIONS			GROUP SYMBOL	GROUP NAME
COARSE GRAINED SOILS MORE THAN 50% RETAINED ON NO. 200 SIEVE	GRAVEL MORE THAN 50% OF COARSE FRACTION RETAINED ON NO. 4 SIEVE	CLEAN GRAVEL	GW	WELL-GRADED GRAVEL, FINE TO COARSE GRAVEL
			GP	POORLY-GRADED GRAVEL
		GRAVEL WITH FINES	GM	SILTY GRAVEL
			GC	CLAYEY GRAVEL
	SAND MORE THAN 50% OF COARSE FRACTION PASSES NO. 4 SIEVE	CLEAN SAND	SW	WELL-GRADED SAND, FINE TO COARSE SAND
			SP	POORLY-GRADED SAND
		SAND WITH FINES	SM	SILTY SAND
			SC	CLAYEY SAND
FINE GRAINED SOILS MORE THAN 50% PASSES NO. 200 SIEVE	SILT AND CLAY LIQUID LIMIT LESS THAN 50	INORGANIC	ML	SILT
			CL	CLAY
		ORGANIC	OL	ORGANIC SILT, ORGANIC CLAY
	SILT AND CLAY LIQUID LIMIT 50 OR MORE	INORGANIC	MH	SILT OF HIGH PLASTICITY, ELASTIC SILT
			CH	CLAY OF HIGH PLASTICITY, FAT CLAY
		ORGANIC	OH	ORGANIC CLAY, ORGANIC SILT
HIGHLY ORGANIC SOILS			PT	PEAT

NOTES:

1. Field classification is based on visual examination of soil in general accordance with ASTM D2488-83.
2. Soil classification using laboratory tests is based on ASTM D2487-83.
3. Descriptions of soil density or consistency are based on interpretation of blowcount data, visual appearance of soils, and/or test data.

SOIL MOISTURE MODIFIERS:

Dry – Absence of moisture, dusty, dry to the touch

Moist – Damp, but no visible water

Wet – Visible free water or saturated, usually soil is obtained from below water table

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SOIL CLASSIFICATION SYSTEM

FIGURE B-1

GEI 85-88

LOG OF SEDIMENT CORE

DEPTH BELOW LAKE BOTTOM (FEET)	SOIL GROUP CLASSIFICATION SYMBOL	DESCRIPTION
		<u>SEDIMENT CORE 1</u>
0.0 - 0.4	SM	Black silty fine to medium sand (sand blast material) (loose, wet) Heavy hydrocarbon staining
0.4 - 0.8	ML	Dark grayish black sandy silt with interbedded sand and silt (sand blast material) (soft, wet) Moderate hydrocarbon staining
0.8 - 0.9	SM	Black silty sand (loose, wet) Heavy hydrocarbon staining
0.9 - 1.3	ML	Gray silt with a trace of organics (soft, wet)
1.3 - 2.0	ML	Gray silt, laminated (very soft, wet)
2.0 - 2.2	SM	Gray fine to coarse silty sand (sand blast material) (loose, wet)
2.2 - 2.5	SM	Black silty fine to coarse sand with occasional wood fragments (sand blast material) (loose, wet) Heavy hydrocarbon staining
2.5 - 2.7	ML	Gray silt with a trace of sand and organic material
2.7 - 4.0	SM	Gray silty sand with gravel (medium dense, wet) (till)
		Sediment core completed at 4.0 feet on 01/24/91
		<u>SEDIMENT CORE 2</u>
0.0 - 3.8	ML	Gray silt with fine to medium sand and a trace of silt and clay (sand blast material) (soft, wet)
3.8 - 4.2	ML	Brown and gray laminated silt with a trace of organics and clay layers
4.2 - 4.8	ML	Gray sandy silt with a trace of organics (soft, wet)
4.8 - 4.9	ML	Brown sandy silt (soft, wet)
		Sediment core completed at 4.9 feet on 01/24/91

THE DEPTHS ON THE SEDIMENT CORE LOGS, ALTHOUGH SHOWN TO 0.1 FOOT, ARE BASED ON AN AVERAGE OF MEASUREMENTS ACROSS THE SEDIMENT CORE AND SHOULD BE CONSIDERED ACCURATE TO 0.5 FOOT.

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LOG OF SEDIMENT CORE

FIGURE B-2

LOG OF SEDIMENT CORE

DEPTH BELOW LAKE BOTTOM (FEET)	SOIL GROUP CLASSIFICATION SYMBOL	DESCRIPTION
		<u>SEDIMENT CORE 3</u>
0.0 - 2.5	ML	Gray to brown silt with a trace of organics (very soft, wet) Slight to moderate petroleum staining
2.5 - 3.2	ML	Black silt with a trace of clay (very soft, wet) Heavy hydrocarbon staining
3.2 - 3.3	CL	Light gray clay (soft, wet)
3.3 - 3.7	ML	Dark gray silt (very soft, wet) Moderate petroleum staining
3.7 - 3.9	ML	Gray silt with clay layers, laminated (soft, wet)
3.9 - 4.4	ML	Gray silt (very soft, wet)
4.4 - 5.0	ML	Brown silt with clay and occasional organic material (very soft, wet)
		Sediment core completed at 5.0 feet on 01/25/91
		<u>SEDIMENT CORE 4</u>
0.0 - 2.5	ML	Gray silt with clay and a trace of fine black sand (sand blast material) (very soft, wet)
2.5 - 2.7	SM	Black silty sand (sand blast material) (soft, wet) Heavy hydrocarbon staining
2.7 - 3.8	ML	Gray silt with a trace of clay and black sand (sand blast material) (very soft, wet)
3.8 - 4.3	ML	Brown silt (very soft, wet)
		Sediment core completed at 4.3 feet on 01/28/91

THE DEPTHS ON THE SEDIMENT CORE LOGS, ALTHOUGH SHOWN TO 0.1 FOOT, ARE BASED ON AN AVERAGE OF MEASUREMENTS ACROSS THE SEDIMENT CORE AND SHOULD BE CONSIDERED ACCURATE TO 0.5 FOOT.

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LOG OF SEDIMENT CORE

FIGURE B-3

LOG OF SEDIMENT CORE

DEPTH BELOW LAKE BOTTOM (FEET)	SOIL GROUP CLASSIFICATION SYMBOL	DESCRIPTION
		SEDIMENT CORE 5
0.0 - 2.1	ML	Laminated brown and gray silt with a trace of clay and sand
2.1 - 2.5	ML	Black silt with clay and a trace of sand (very soft, wet) Heavy hydrocarbon staining
2.5 - 2.8	ML	Brown silt with a trace of organics (very soft, wet)
2.8 - 3.3	ML/CL	Dark gray laminated silt and clay (soft, wet)
3.3 - 3.7	ML	Light brown silt with a trace of organics
3.7 - 3.9	ML	Dark gray silt Moderate hydrocarbon staining
3.9 - 4.1	ML	Brown silt (soft, wet)
4.1 - 4.4	ML	Gray silt with occasional organic material and a trace of clay (soft, wet)
4.4 - 4.6	ML	Brown silt (soft, wet)
4.6 - 4.9	ML	Gray silt (soft, wet) Slight hydrocarbon staining
4.9 - 5.0	ML	Brown silt (soft, wet)
		Sediment core completed at 5.0 feet on 01/28/91

THE DEPTHS ON THE SEDIMENT CORE LOGS, ALTHOUGH SHOWN TO 0.1 FOOT, ARE BASED ON AN AVERAGE OF MEASUREMENTS ACROSS THE SEDIMENT CORE AND SHOULD BE CONSIDERED ACCURATE TO 0.5 FOOT.

LOG OF SEDIMENT CORE

DEPTH BELOW LAKE BOTTOM (FEET)	SOIL GROUP CLASSIFICATION SYMBOL	DESCRIPTION
		<u>SEDIMENT CORE 6</u>
0.0 - 0.2	ML	Black silt (very soft, wet) Heavy hydrocarbon staining and heavy sheen
0.2 - 2.1	ML	Gray silt with a trace of sand (soft, wet) Slight to moderate petroleum staining and moderate sheen
2.1 - 3.5	ML	Black silt with sand (very soft, wet) Heavy hydrocarbon staining, heavy sheen Coal tar
3.5 - 3.7	ML	Gray silt with clay (soft, wet)
3.7 - 3.9	ML	Brown silt with a trace of organic material (very soft, wet)
3.9 - 4.2	ML	Gray silt (soft, wet)
4.2 - 4.6	ML	Brown silt with a trace of organic material (soft, wet)
4.6 - 4.8	ML	Gray silt with a trace of organic material (soft, wet)
4.8 - 5.0	ML	Brown silt with a trace of organic material (soft, wet)
		Sediment core completed at 5.0 feet on 01/30/91
		<u>SEDIMENT CORE 7</u>
0.0 - 2.6	ML	Black silt (very soft, wet Heavy petroleum staining and heavy sheen Coal tar
2.6 - 3.0	ML	Brown silt (soft, wet)
3.0 - 4.1	SM	Gray silty fine sand (loose, wet)
4.1 - 4.5	ML/CL	Gray to brown laminated silt with clay layers (soft, wet) Slight hydrocarbon staining
4.5 - 4.8	SP	Gray fine sand (loose, wet)
4.8 - 5.0	ML	Brown silt with a trace of organic material (soft, wet)
		Sediment core completed at 5.0 feet on 01/30/91

THE DEPTHS ON THE SEDIMENT CORE LOGS, ALTHOUGH SHOWN TO 0.1 FOOT, ARE BASED ON AN AVERAGE OF MEASUREMENTS ACROSS THE SEDIMENT CORE AND SHOULD BE CONSIDERED ACCURATE TO 0.5 FOOT.

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LOG OF SEDIMENT CORE

FIGURE B-5

LOG OF SEDIMENT CORE

DEPTH BELOW LAKE BOTTOM (FEET)	SOIL GROUP CLASSIFICATION SYMBOL	DESCRIPTION
		<u>SEDIMENT CORE 8</u>
0.0 - 1.7	ML	Gray to brown silt with clay (liquid, wet)
1.7 - 2.2	ML	Medium brown silt
2.2 - 2.8	ML	Gray and brown layered silt with clay (very soft, wet)
2.8 - 3.1	ML	Light gray silt with small brown stringers and a trace of organic matter (very soft, wet)
3.1 - 4.8	ML	Dark brown silt with a trace of organic matter (very soft, wet)
		Sediment core completed at 4.8 feet on 01/22/91
		<u>SEDIMENT CORE 9</u>
0.0 - 0.5	ML	Brownish black silt with organic material (soft, wet)
0.5 - 1.1	SP	Grayish black silty fine to medium sand with occasional wood fragments (loose, wet)
1.1 - 1.4	ML	Gray silt with a trace of clay (soft, wet)
1.4 - 1.9	CL/OH	Brownish gray organic silt with occasional sand stringers (soft, wet)
1.9 - 2.5	ML	Gray silt with rootlets (medium stiff, wt)
		Sediment core completed at 2.5 feet on 01/21/91

THE DEPTHS ON THE SEDIMENT CORE LOGS, ALTHOUGH SHOWN TO 0.1 FOOT, ARE BASED ON AN AVERAGE OF MEASUREMENTS ACROSS THE SEDIMENT CORE AND SHOULD BE CONSIDERED ACCURATE TO 0.5 FOOT.

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LOG OF SEDIMENT CORE

FIGURE B–6

APPENDIX C

ANALYTICAL TESTING RESULTS

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : SEDIMENT

ELEMENT	DATE PREPARED	DATE ANALYZED
ARSENIC	02/04/91	02/08/91
BARIUM	02/04/91	02/12/91
CADMIUM	02/04/91	02/07/91
CHROMIUM	02/04/91	02/07/91
COPPER	02/04/91	02/07/91
LEAD	02/04/91	02/07/91
MERCURY	02/22/91	02/22/91
NICKEL	02/04/91	02/07/91
SELENIUM	02/04/91	02/08/91
SILVER	02/04/91	02/08/91
ZINC	02/04/91	02/07/91

METALS RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT
UNITS : mg/Kg

ATI I.D. #	CLIENT I.D.	ARSENIC	BARIUM	CADMIUM
9101-191-2	2B 0-0.3′	3,000	380	12
9101-191-3	2D 1.5-2.0′	-	-	7.5
9101-191-4	2E 2.0-2.5′	-	-	8.2
9101-191-5	2F 2.5-3.0′	-	-	9.8
9101-191-6	2G 3.0-3.5′	-	-	3.9
9101-191-7	2H 3.5-4.0′	-	-	3.4
9101-191-8	2I 4.0-4.5′	-	-	1.7
9101-191-9	2J 4.5-4.8′	-	-	2.9
9101-191-10	1A 0-0.3′	3,100	380	7.4
9101-191-14	2C 0.3-1.5′	2,000	196	4.4
REAGENT BLANK	-	<0.5	<6	<1

ATI I.D.# 9101-191

METALS RESULTS

CLIENT	: GEOENGINEERS, INC.	MATRIX	: SEDIMENT
PROJECT #	: 1299-003-B04		
PROJECT NAME	: UNIMAR, INC.	UNITS	: mg/Kg

ATI I.D. #	CLIENT I.D.	CHROMIUM	COPPER	LEAD
9101-191-2	2B 0-0.3'	110	3,800	2,100
9101-191-3	2D 1.5-2.0'	-	3,400	2,600
9101-191-4	2E 2.0-2.5'	-	4,600	2,700
9101-191-5	2F 2.5-3.0'	-	5,300	2,800
9101-191-6	2G 3.0-3.5'	-	2,200	2,300
9101-191-7	2H 3.5-4.0'	-	560	570
9101-191-8	2I 4.0-4.5'	-	46	95
9101-191-9	2J 4.5-4.8'	-	40	<25
9101-191-10	1A 0-0.3'	52	5,900	2,900
9101-191-14	2C 0.3-1.5'	230	2,300	1,600
REAGENT BLANK	-	<2	<2	<10

METALS RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : mg/Kg

ATI I.D. #	CLIENT I.D.	MERCURY	NICKEL	SELENIUM
9101-191-2	2B 0-0.3'	1.43	42	<3
9101-191-10	1A 0-0.3'	0.84	46	<3
9101-191-14	2C 0.3-1.5'	1.50	130	<3
REAGENT BLANK	-	<0.15	<3	<0.5

ATI I.D. # 9101-191-2

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/24/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/25/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: 2B 0-0.3'	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 5

RESULTS BASED ON DRY WEIGHT

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
UNDECANE, 3,6-DIMETHYL	1095	6.2
DODECANE, 2,7,10-TRIMETHYL	1133	18
SUBSTITUTED PHENOL	1481	12
HYDROCARBON	1595	10
CYCLIC HYDROCARBON	2011	15

ATI I.D. # 9101-191-10

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/24/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/25/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: 1A 0-0.3′	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 5

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT	
N-NITROSODIMETHYLAMINE	<1.8	
PHENOL	<1.8	
ANILINE	<1.8	
BIS(2-CHLOROETHYL)ETHER	<1.8	
2-CHLOROPHENOL	<1.8	
1,3-DICHLOROBENZENE	<1.8	
1,4-DICHLOROBENZENE	<1.8	
BENZYL ALCOHOL	<1.8	
1,2-DICHLOROBENZENE	<1.8	
2-METHYLPHENOL	<1.8	
BIS(2-CHLOROISOPROPYL)ETHER	<1.8	
4-METHYLPHENOL	<1.8	
N-NITROSO-DI-N-PROPYLAMINE	<1.8	
HEXACHLOROETHANE	<1.8	
NITROBENZENE	<1.8	
ISOPHORONE	<1.8	
2-NITROPHENOL	<1.8	
2,4-DIMETHYLPHENOL	<1.8	
BENZOIC ACID	<8.9	
BIS(2-CHLOROETHOXY)METHANE	<1.8	
2,4-DICHLOROPHENOL	<1.8	
1,2,4-TRICHLOROBENZENE	<1.8	
NAPHTHALENE	1.3	J
4-CHLOROANILINE	<1.8	
HEXACHLOROBUTADIENE	<1.8	
4-CHLORO-3-METHYLPHENOL	<1.8	
2-METHYLNAPHTHALENE	<1.8	
HEXACHLOROCYCLOPENTADIENE	<1.8	
2,4,6-TRICHLOROPHENOL	<1.8	
2,4,5-TRICHLOROPHENOL	<8.9	
2-CHLORONAPHTHALENE	<1.8	
2-NITROANILINE	<8.9	
DIMETHYLPHTHALATE	<1.8	
ACENAPHTHYLENE	<1.8	
3-NITROANILINE	<8.9	
ACENAPHTHENE	2.1	
2,4-DINITROPHENOL	<8.9	
4-NITROPHENOL	<8.9	

J = Estimated value.

CONTINUED NEXT PAGE

ATI I.D. # 9101-191-10

SEMI-VOLATILE ORGANICS ANALYSIS
DATA SUMMARY (CONTINUED)

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/24/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/25/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: 1A 0-0.3'	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 5

RESULTS BASED ON DRY WEIGHT

COMPOUND	RESULT
DIBENZOFURAN	1.0 J
2,4-DINITROTOLUENE	<1.8
2,6-DINITROTOLUENE	<1.8
DIETHYLPHTHALATE	<1.8
4-CHLOROPHENYL-PHENYLETHER	<1.8
FLUORENE	2.1
4-NITROANILINE	<8.9
4,6-DINITRO-2-METHYLPHENOL	<8.9
N-NITROSODIPHENYLAMINE	<1.8
4-BROMOPHENYL-PHENYLETHER	<1.8
HEXACHLOROBENZENE	<1.8
PENTACHLOROPHENOL	<8.9
PHENANTHRENE	10
ANTHRACENE	2.8
DI-N-BUTYLPHTHALATE	<1.8
FLUORANTHENE	11
BENZIDINE	<18
PYRENE	11
BUTYLBENZYLPHTHALATE	<1.8
3,3-DICHLOROBENZIDINE	<3.5
BENZO(a)ANTHRACENE	4.4
BIS(2-ETHYLHEXYL)PHTHALATE	5.9
CHRYSENE	5.5
DI-N-OCTYLPHTHALATE	<1.8
BENZO(b)FLUORANTHENE	6.5
BENZO(k)FLUORANTHENE	1.8
BENZO(a)PYRENE	5.4
INDENO(1,2,3-cd)PYRENE	2.8
DIBENZ(a,h)ANTHRACENE	<1.8
BENZO(g,h,i)PERYLENE	3.4

SURROGATE PERCENT RECOVERIES

NITROBENZENE-d5	79
2-FLUOROBIPHENYL	116 *
TERPHENYL-d14	113
PHENOL-d6	101
2-FLUOROPHENOL	69
2,4,6-TRIBROMOPHENOL	94

* Out of limits.

J = Estimated value.

ATI I.D. # 9101-191-10

SEMI-VOLATILE ORGANICS ANALYSIS
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT	: GEOENGINEERS, INC.	DATE SAMPLED	: 01/24/91
PROJECT #	: 1299-003-B04	DATE RECEIVED	: 01/25/91
PROJECT NAME	: UNIMAR, INC.	DATE EXTRACTED	: 01/30/91
CLIENT I.D.	: 1A 0-0.3'	DATE ANALYZED	: 02/08/91
SAMPLE MATRIX	: SEDIMENT	UNITS	: mg/Kg
EPA METHOD	: 8270	DILUTION FACTOR	: 5

RESULTS BASED ON DRY WEIGHT

COMPOUND	SCAN NUMBER	ESTIMATED CONCENTRATION
UNDECANE, 3,5-DIMETHYL-	1083	7.3
DODECANE, 2,7,10-TRIMETHYL-	1130	21
SUBSTITUTED PHENOL	1480	11
CYCLIC HYDROCARBON	2012	14
HYDROCARBON	2194	13

SEMI-VOLATILE ORGANIC QUALITY CONTROL DATA

CLIENT	: GEOENGINEERS, INC.		SAMPLE I.D.	: BLANK SPIKE
PROJECT #	: 1299-003-B04		DATE EXTRACTED	: 01/30/91
PROJECT NAME	: UNIMAR, INC.		DATE ANALYZED	: 02/08/91
EPA METHOD	: 8270		MATRIX	: SOIL
			UNITS	: mg/Kg

COMPOUND	SAMPLE RESULT	SPIKE ADDED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % REC	RPD
1,2,4-TRICHLOROBENZENE	<0.17	3.33	2.39	72	2.86	86	18
ACENAPHTHENE	<0.17	3.33	2.19	66	2.42	73	10
2,4-DINITROTOLUENE	<0.17	3.33	2.46	74	2.56	77	4
PYRENE	<0.17	3.33	2.34	70	2.62	78	11
N-NITROSO-DI-N-PROPYLAMINE	<0.17	3.33	2.26	68	2.68	81	17
1,4-DICHLOROBENZENE	<0.17	3.33	2.23	67	2.65	79	17
PENTACHLOROPHENOL	<0.85	13.3	11.6	87	11.3	85	3
PHENOL	<0.17	6.67	3.98	60	4.79	72	18
2-CHLOROPHENOL	<0.17	6.67	4.03	60	4.90	73	19
4-CHLORO-3-METHYLPHENOL	<0.17	6.67	4.44	67	4.99	75	12
4-NITROPHENOL	<0.85	13.3	11.5	86	11.6	87	1

$$\text{\% Recovery} = \frac{\text{(Spike Sample Result - Sample Result)}}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{\text{(Spiked Sample Result - Duplicate Spike Sample Result)}}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-191

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : REAGENT BLANK
SAMPLE MATRIX : SOIL
EPA METHOD : 8080 (PCB)
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : N/A
DATE RECEIVED : N/A
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/04/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
PCB 1016	<0.033
PCB 1221	<0.033
PCB 1232	<0.033
PCB 1242	<0.033
PCB 1248	<0.033
PCB 1254	<0.033
PCB 1260	<0.033

ATI I.D. # 9101-191-2

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 2B 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8080 (PCB)
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/24/91
DATE RECEIVED : 01/25/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/04/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
PCB 1016	<0.033
PCB 1221	<0.033
PCB 1232	<0.033
PCB 1242	<0.033
PCB 1248	<0.033
PCB 1254	<0.033
PCB 1260	<0.033

ATI I.D. # 9101-191-10

POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS
DATA SUMMARY

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
CLIENT I.D. : 1A 0-0.3'
SAMPLE MATRIX : SEDIMENT
EPA METHOD : 8080 (PCB)
RESULTS BASED ON DRY WEIGHT

DATE SAMPLED : 01/24/91
DATE RECEIVED : 01/25/91
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/04/91
UNITS : mg/Kg
DILUTION FACTOR : 1

COMPOUND	RESULT
PCB 1016	<0.033
PCB 1221	<0.033
PCB 1232	<0.033
PCB 1242	<0.033
PCB 1248	<0.033
PCB 1254	<0.033
PCB 1260	0.43 *

* Tentative identification. PCB-like pattern is present, but qualitative identification was difficult due to discrepancies in the profile.

POLYCHLORINATED BIPHENYLS (PCB)
QUALITY CONTROL

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
EPA METHOD : 8080 (PCB)

SAMPLE ID : BLANK SPIKE
DATE EXTRACTED : 01/30/91
DATE ANALYZED : 02/04/91
MATRIX : SOIL
UNITS : mg/Kg

COMPOUND	SAMPLE RESULT	CONC SPIKED	SPIKED SAMPLE	% REC	DUP SPIKED SAMPLE	DUP % RECOVERY	RPD
PCB 1260	<0.033	0.33	0.345	104	0.351	106	2

$$\% \text{ Recovery} = \frac{(\text{Spike Sample result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Spiked Sample Result} - \text{Duplicate Spike Sample Result})}{\text{Average of Spiked Sample}} \times 100$$

ATI I.D. # 9101-191

GAS CHROMATOGRAPHY RESULTS *

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : ng/L

PARAMETER	SAMPLE 2A @ 39' -1	2C -13
MONOBUTYLTIN	6	27
DIBUTYLTIN	4	40
TRIBUTYLTIN	5	22
TETRABUTYLTIN	4	<1

* Analyzed by GC/FPD, Method Battelle N05196300.

ATI I.D. # 9101-191

GAS CHROMATOGRAPHY RESULTS *

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : SEDIMENT

UNITS : ug/Kg

PARAMETER	2C 0.3-1.5' -14
MONOBUTYLTIN	8
DIBUTYLTIN	5
TRIBUTYLTIN	190
TETRABUTYLTIN	<1

* Analyzed by GC/FPC, Method Battelle N05196300.

ATI # 9101-191

METALS ANALYSIS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
SAMPLE MATRIX : WATER

ELEMENT	DATE PREPARED	DATE ANALYZED
ARSENIC	01/29/91	02/08/91
BARIUM	01/29/91	02/07/91
CADMIUM	01/29/91	02/12/91
CHROMIUM	01/29/91	02/08/91
COPPER	02/01/91	02/01/91
LEAD	01/29/91	02/12/91
MERCURY	01/29/91	01/29/91
NICKEL	01/29/91	02/07/91
SELENIUM	01/29/91	02/08/91
SILVER	01/29/91	02/08/91
ZINC	01/29/91	02/07/91

ATI I.D. # 9101-191

METALS RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : WATER

UNITS : mg/L

PARAMETER	SAMPLE 2A @ 39' -1	2C -13	REAGENT BLANK
ARSENIC	<0.005	0.898	<0.005
BARIUM	<0.06	0.19	<0.06
CADMIUM	<0.0003	<0.0003	<0.0003
CHROMIUM	<0.02	<0.02	<0.02
COPPER	<0.02	<0.02	<0.02
LEAD	<0.005	<0.005	<0.005
MERCURY	<0.0005	<0.002*	<0.0005
NICKEL	<0.03	<0.03	<0.03
SELENIUM	<0.005	<0.005	<0.005
SILVER	<0.02	<0.02	<0.02
ZINC	<0.01	<0.01	<0.01

* Increased detection limit due to limited sample.

METALS QUALITY CONTROL

CLIENT	: GEOENGINEERS, INC.	MATRIX	: WATER
PROJECT #	: 1299-003-B04		
PROJECT NAME	: UNIMAR, INC.	UNITS	: mg/L

COMPOUND	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED SAMPLE	SPIKE CONC	% REC
ARSENIC	9101-167-15	0.042	0.036	15	0.052	0.025	40*
ARSENIC	BLANK SPIKE	N/A	N/A	N/A	0.024	0.025	96
BARIUM	9101-167-15	0.16	0.18	12	16.8	20.0	83
CADMIUM	9101-174-2	0.0012	0.0009	29	0.0019	0.0010	70
CHROMIUM	9101-191-13	<0.02	<0.02	0	1.98	2.50	79
COPPER	9101-174-2	0.65	0.59	10	3.04	2.50	96
LEAD	9101-184-3	0.113	0.112	1	**	**	**
LEAD	BLANK SPIKE	N/A	N/A	N/A	0.033	0.025	132
MERCURY	9101-202-2	<0.0005	<0.0005	0	0.0030	0.0020	150
MERCURY	BLANK SPIKE	N/A	N/A	N/A	0.0029	0.0020	145
NICKEL	9101-167-15	<0.03	<0.03	0	2.48	2.50	99
SELENIUM	9101-167-15	<0.005	<0.005	0	0.018	0.025	75
SILVER	9101-191-13	<0.02	<0.02	0	0.94	1.00	94
ZINC	9101-167-15	<0.01	<0.01	0	0.49	0.50	98

* Out of limits due to matrix interference.

** Due to the necessary dilution of the sample, result was not attainable.

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

AnalyticalTechnologies,Inc. 560 Naches Avenue, S.W., Suite 101, Renton, WA 98055, (206) 228-8335

ATI I.D. # 9104-199

GeoEngineers
MAY 15 1991
Routing PGW ☐ ☐ ☐ ☐ ☐
File

May 14, 1991

GeoEngineers, Inc.
8410 154th Avenue N.E.
Redmond, WA 98052

Attention : Paul Werner

Project Number : 1299-003-B04

Project Name : Unimar, Inc.

On April 15, 1991, Analytical Technologies, Inc., received 11 sediment samples for analysis. The samples were analyzed with EPA methodology or equivalent methods as specified in the attached analytical schedule. The results, sample cross reference, and quality control data are enclosed.

The following ATI accession numbers 9101-148, 9101-167, 9101-191, 9101-203, 9104-216, and 9101-238 were reaccessioned for additional tests on April 15, 1991. Please note that per client request mercury was leached past the 28 day holding time.

Karen J. Meyer for DMM
Donna M. McKinney
Senior Project Manager

Frederick W. Grothkopp
Frederick W. Grothkopp
Technical Manager

FWG/tc

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COMPLIANCE STATEMENT

The work conducted through Battelle Proposal/Agreement No. N-0519-6100, "Measurement of Butyltin Species in Sediments by n-Pentyl Derivatization with Gas Chromatography/Flame Photometric Detection (GC/FPD) and Optional Confirmation by Gas Chromatography/ Mass Spectrometry (GC/MS)" was performed in compliance current with Good Laboratory Practice Standards (40 CFR Part 160), dated 1983, and proposed revisions dated 1987, with the following exceptions: The initial total organic carbon (TOC) and total extractable tin characterization analyses of the test sediments were carried out by contract laboratories not operating under Good Laboratory Practices guidelines. TOC determinations were carried out by Global Geochemistry Corporation, Canoga Park, CA. Total extractable tin determinations were carried out by Battelle Marine Research Laboratory, Sequim, WA. The data reports from the contract laboratories are on file with the raw data. Sediment sample collection was not monitored by the quality assurance unit in the field. However, procedures used were conducted as stated in the protocol, and documented in the raw data.

Allen D. Uhl

Allen D. Uhler, Ph.D.
Study Director

2-28-1989
Date

Sponsor

Date

Submitter

Date

QUALITY ASSURANCE STATEMENT

Quality Assurance Statement
for
N-0519-6100, Measurement of Butyltin Species in Sediments by n-Pentyl Derivatization with Gas Chromatography/Flame Photometric Detection (GC/FPD) and Optional Confirmation by Gas Chromatography/Mass Spectrometry (GC/MS)

In accordance with Good Laboratory Practice Standards (EPA/FIFRA 40 CFR Part 160) dated 1983 and proposed revisions dated 1987, this study has been monitored by Battelle's Quality Assurance Unit to determine whether the study was conducted in accordance with the Good Laboratory Standards and laboratory Standard Operating Procedures described above. Study audit dates, and dates the results were reported to the Study Director and management are listed in the following table.

The final report for this study has been reviewed by the Quality Assurance Unit and, to the best of my knowledge, accurately reflects the study as conducted, including all areas of compliance or noncompliance with Good Laboratory Practice Standards, the study protocol, and Battelle Ocean Sciences' Standard Operating Procedures.

Patricia D Royal

Patricia D. Royal
Manager, Quality Assurance Unit
Battelle Ocean Sciences Department

2\28\89
Date

QUALITY ASSURANCE AUDITS

CONDUCTED FOR N-0519-6100

MEASUREMENT OF BUTYLTIN SPECIES IN SEDIMENTS BY n-PENTYL DERIVATIZATION WITH GAS CHROMATOGRAPHY/FLAME PHOTOMETRIC DETECTION (GC/FPD) AND OPTIONAL CONFIRMATION BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

Date of Audit	Date of Report to Study Director	Date of Report to Management
May 12, 1988	May 12, 1988	May 12, 1988
June 8-14, 1988	June 14, 1988	September 26, 1988
July 13, 1988	July 14, 1988	September 26, 1988
July 20, 1988	July 20, 1988	September 26, 1988
August 23-24, 1988	August 25, 1988	September 26, 1988
September 29, 1988	September 30, 1988	October 13, 1988
October 12, 1988	October 13, 1988	February 28, 1989
October 20-21, 1988	October 24, 1988	February 28, 1989
February 22,24, 1989	February 27, 1989	February 28, 1989

STUDY PARTICIPANTS

IN BATTELLE PROJECT N-0519-6100

MEASUREMENT OF BUTYLTIN SPECIES IN SEDIMENTS BY n-PENTYL DERIVATIZATION WITH GAS CHROMATOGRAPHY/FLAME PHOTOMETRIC DETECTION (GC/FPD) AND OPTIONAL CONFIRMATION BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

Dr. Allen D. Uhler	Study Director
Mr. Gregory S. Durell	Analytical Chemist
Mr. William G. Steinhauer	Associate Section Manager
Mr. Theodore H. Coogan	Mass Spectrometry Facility Manager
Mr. Kenneth S. Davis	Laboratory Technician
Mr. George W. Desreuisseau	Laboratory Technician
Ms. Ann M. Spellacy	Laboratory Technician

1.0 INTRODUCTION

Tributyltin (TBT) is an organometallic compound used as a biocide in the treatment of wood products, as a disinfectant, and as an active agent in antifoulant boat paints. Since the registration of TBT-containing antifoulant paints in the 1960s, some 300 TBT antifouling formulations have been introduced in the marketplace, with an estimated usage of 300,000 lb/yr in the United States (Champ and Pugh, 1987). Currently, there are 20 different TBT compounds registered as pesticidal active ingredients. Nine of these 20 TBT-containing compounds are registered for use in antifoulant paints. Table 1 presents these nine compounds and their respective Chemical Abstract Services (CAS) registration numbers.

The release of TBT from such paints (slow diffusion from the paint or sloughing of material from the paint surface) results in introduction of TBT into the various aquatic environmental compartments. Tributyltin released into the aqueous environment undergoes degradation to dibutyltin (DBT), monobutyltin (MBT) and finally, inorganic tin via successive debutylation (Thain et al., 1987a; Thain et al., 1987b; Blunden and Champman, 1982).

In 1986, concern for the fate and environmental effect of TBT in water prompted the U.S. Environmental Protection Agency (EPA) to initiate a Special Review of TBT-containing antifoulant paints. Subsequently, in 1987, EPA issued a Notice of Preliminary Determination to cancel or modify certain registrations of TBT products used as antifoulant paints. The EPA, under authority granted by the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) also issued a Data Call In, requesting product use data, TBT release rate data, worker exposure data, ecological effects data, validated analytical methods, and environmental fate data.

1.1 SCOPE OF WORK

In response to the Data Call In, this report describes the validation of a multiresidue analytical method for the detection, measurement, and confirmation of trace levels (µg/kg) of butyltin species in estuarine and freshwater sediments. The method has been validated for the compound TBT and its degradation products DBT and MBT, as well as tetrabutyltin (TTBT: a TBT manufacturing impurity that can degrade to TBT, DBT, and MBT in the environment). The method described in this report provides detection at the sub-microgram per kilogram level (wet weight), and has been tested and validated to concentrations of 500 µg/kg.

This study was conducted to meet requirements as specified in the Pesticide Assessment Guidelines, Subdivision D, Product Chemistry (EPA, 1982) and 40 CFR Part 160, EPA FIFRA Good Laboratory Practices dated 1983, and revisions dated 1987. The protocol was reviewed by the Quality Assurance Unit on May 12, 1988. The study was initiated May 17, 1988. The laboratory work was completed February 9, 1989. The study was completed February 28, 1989.

1.2 TEST SUBSTANCE IDENTIFICATION

Butyltin chloride compounds were chosen for the preparation of TBT, DBT, and MBT fortification and chromatography standards because of their ready commercial availability, chemical stability, and water solubility. Commercially available TTBT was used in the preparation of TTBT standards.

Substance	CAS Number	Abbreviation
Tributyltin chloride	1461-22-9	TBT
Dibutyltin dichloride	683-18-1	DBT
Monobutyltin trichloride	1118-46-3	MBT
Tetrabutyltin	1461-25-2	TTBT

The manufacturer, catalog number, and chemical purity of each test substance are presented in Section 2.1. The manufacturing source, lot numbers, and manufacturer's statement of chemical purity of the test substances are on file in the study records.

1.3 PRINCIPLES

Butyltin compounds are organometallic compounds in which the butyl groups are covalently bonded to the tin atom. Tetrabutyltin is a nonpolar, nonionic compound. However in aqueous solution, tributyltin, dibutyltin, and monobutyltin are monovalent, divalent, and trivalent cations, respectively. Analysis of these compounds in sediments at environmentally significant levels by gas chromatography requires that two objectives be achieved: (1) the compounds be extracted from the sediment matrix and concentrated, and (2) the cationic compounds be derivatized in order to produce nonionic compounds that are amenable to gas chromatography. To accomplish these objectives, the method presented in this report relies on forming bromide complexes of the cationic butyltins through reaction with HBr, followed by solvent extraction of the liberated butyltin compounds. Sediment samples are mixed in a water:HBr slurry prior to organic solvent extraction in order to form the less polar bromide complexes. The butyltins are then solvent extracted from the sediment slurry with toluene and the chelating agent tropolone. Following extraction, commercially available n-pentylmagnesium bromide is used to convert the butyltins to nonpolar n-pentyl derivatives. The extract is cleaned up through a Florisil/silica liquid chromatography column, and the butyltins are quantified by gas chromatography with flame photometric detection (GC/FPD). The presence of butyltins in sediment samples can optionally be confirmed by gas chromatography with mass spectrometry (GC/MS) operated in selected ion monitoring mode (SIM).

1.3.1 Method Description

A sediment sample is centrifuged at 2500 rpm for 10 min to remove overlying water. One hundred grams of this sediment is weighed to the nearest 0.1 g, and transferred to a Teflon jar. The recovery internal standard (RIS) tripropyltin chloride (TPT) is added. Next, 25 mL of distilled water and 5 mL of 48 percent HBr are added to the sediment, and the mixture is agitated on a shaker table for 1 h. This mixture is extracted twice with 60 mL of 0.05 percent tropolone in toluene for 1 hour per cycle. Between cycles, the organic layer is separated from the sediment and water mixture by centrifugation at 2500 rpm for 5 min. The toluene extracts are combined, and dried over ca. 40 g of sodium sulfate. The toluene is concentrated to ca. 10 mL by rotary evaporation at 60-70°C, and the toluene exchanged for hexane by successive hexane additions and rotary evaporation. The labile butyltins (including the RIS TPT) are converted to the corresponding n-pentyl derivatives by adding a minimum of 5 mL 1.9 M n-pentylmagnesium bromide to the hexane solution. The reaction is allowed to proceed for 15 min at room temperature, and then is quenched by adding 25 mL distilled water, followed by 10-20 mL 10 N H_2SO_4. The hexane fraction is separated from the aqueous layer, reduced in concentration to ca. 4 mL by nitrogen gas evaporation, and subjected to combined Florisil/silica gel column cleanup. The cleanup column consists of 16 g of Florisil, topped with 7 g 1 percent deactivated silica gel, topped by 2 g of sodium sulfate. The column, prewet with hexane, is charged with the sample and eluted with 100 mL hexane. The eluate is collected in a 250-mL Kuderna-Danish apparatus, and concentrated to ca. 10 mL in a 100°C water bath. A final volume of approximately 500 µL is achieved by nitrogen gas evaporation. The previously derivatized quantitation internal standard dipropyltin (DPT) is added, and the sample transferred to a 2-mL vial and submitted for GC/FPD and/or GC/MS confirmatory analysis.

Optional procedures for gel permeation chromatography (GPC) cleanup and activated copper sulfur cleanup are presented for the treatment of especially contaminated samples not adequately processed by the standard method.

Chemical standards are prepared using commercially available tetraorganotin and organotin chloride compounds. Individual butyltin stock solutions are prepared gravimetrically in hexane. A mixed mono-, di-, tri-, and tetrabutyltin solution is then prepared volumetrically from the stock solutions in hexane. The internal standards tripropyltin chloride (TPT) and dipropyltin dichloride (DPT) are prepared gravimetrically in hexane in separate vessels. The resulting solutions are derivatized according to the above procedures, and the appropriate dilutions of the derivatized butyltins are prepared for use as quantitative gas chromatography standards. Meinema et al. (1978) have demonstrated that the techniques described in this method produce rapid and quantitative alkylation of butyltin compounds, with conversion efficiency greater than 99 percent.

Quantitative analysis is accomplished by gas chromatography, with flame photometric detection. Detection of the butyltins by the highly selective GC/FPD technique is carried out using a 610-nm bandpass optical filter. This filter allows characteristic emissions from tin to be detected, while eliminating extraneous, interfering emissions. For a further, more detailed description of GC/FPD detection for organotin compounds, the work presented by Kaplia and Vogt (1980) is recommended.

Confirmation of butyltin compounds in selected samples may be carried out by GC/MS using selective ion monitoring (SIM) techniques. Selected ion monitoring allows for the sensing of only certain mass fragments during mass spectrometry detection of gas chromatographic effluents. The benefits of this technique are twofold: increased sensitivity over full mass scan methods because the mass spectrometer is scanning only mass fragments of interest, and increased specificity relative to full mass scan techniques, because many extraneous (interfering) compounds are "transparent" to the SIM technique. Selection of the mass fragments used for butyltin confirmatory analysis are discussed in Section 2.4.2.

Quantitative determination of butyltins is carried out using GC/FPD and the method of internal standards. Response factors for each butyltin analyte are determined relative to the quantitation internal standard DPT (or, as an alternative, TPT) from three-level calibration curves. Conventional response factor calculations are described in Section 2.4.3.

When utilizing the GC/FPD determinative step, the method detection limits (MDL), defined by EPA (Federal Register, 1984) as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte is greater than zero, are MBT (0.288 µg/kg); DBT (0.570 µg/kg); TBT (0.251 µg/kg); TTBT (0.125 µg/kg). The MDLs are expressed on a wet weight basis. Levels as low as 50 ng/kg per component are detectable by GC/FPD, but cannot be reliably quantified because they are below the MDL. The MDL is discussed fully in Section 3.4.

1.3.2 Method Validation Strategy

This report describes the testing and performance of the analytical method under conditions expected to be encountered in actual environmental settings. The method was tested using a number of different sediments and at different analyte concentrations. The overall strategy was as follows:

1. Design a rapid, straightforward method capable of concomitantly extracting butyltin analytes of substantially different polarity from sediments.

2. Assess the precision and accuracy of the method through fortification and recovery experiments with different sediments at various spike levels.

3. Define the method detection limit (MDL), following EPA guidelines.

4. Evaluate possible matrix interferences and their effects on method performance. Candidate interferants include natural organic matter, sulfur, and crude oil.

5. Evaluate the effect of storage of sediments and sediment extracts on butyltin stability.

6. Using the refined method, analyze several contaminated sediments from different geographical locations in the United States.

Table 2 is a summary of the tests performed for method validation. Phase 1 experiments were conducted to evaluate the effects of naturally extracted organic matter, crude oil, and elemental sulfur on method performance, especially GC/FPD analysis. The results of these experiments are discussed in Section 3.7.

Actual method performance (in terms of recovery and reproducibility of the method) was evaluated in Phase 2 experiments. Two estuarine sediments and two freshwater sediments of significantly different composition (e.g., grain size, total organic carbon content) were utilized for spiking and recovery experiments. Initially, the ambient levels of butyltins in the test sediments were determined. Next, the test sediments were fortified at levels of approximately 5-10 times and 50-100 times greater than the measured ambient levels. Recovery and reproducibility of recovery were determined from 4 replicate measurements at each level. The results of these experiments are described in Sections 3.1 to 3.3. The method detection limit (MDL) was determined by fortifying an estuarine sediment containing very low ambient butyltin residues with TBT, DBT, MBT, and TTBT at levels near the 10-to-1 FPD signal-to-noise ratio, and performing eight replicate analyses. The MDL was then calculated based on the variance in the replicate measurements, as prescribed by EPA (Federal Register, 1984). Detailed discussions and the results of the MDL experiments are presented in Section 3.4.

Phase 3 of this study involved employing the refined analytical method for screening selected U.S. coastal sediments for butyltin contamination. Selected estuarine sediments with likely butyltin residues were collected and analyzed by the analytical method, and by the method of standard additions, in order to evaluate matrix effects bias. Sediments from Hampton Roads, Virginia, Galveston Bay, Texas, and Puget Sound, Washington were used in this phase of the study. Results are presented in Section 3.5.

The stability of the target butyltin analytes during storage was evaluated during Phase 4 of this study. An estuarine sediment with ambient levels of all four butyltin analytes was stored as bulk sediment at -20°C. Additionally, the aliquots of the same sediment were extracted and derivatized, and the extracts stored at -20°C. Both the bulk sediment and the sediment extracts were analyzed on a weekly basis for 4 weeks, and the relative stability of each butyltin was monitored as a function of time. The results of these studies are presented in Section 3.6.

2.0 MATERIALS AND METHODS

The application of this method for analysis of sediment samples requires routine laboratory glassware and apparatus. Teflon jars used in the extraction step are commercially available. The following sections describe the equipment, supplies and procedures to be used.

2.1 EQUIPMENT

The following equipment and chemicals are required to perform the analytical method.

Labware/Hardware

500-mL Teflon jars with screw caps
100-mL volumetric flask
20-mL volumetric flasks

25-mL volumetric flasks
Assorted pipets, microsyringes, and disposable micropipets
25-mL pear-shaped flasks
10-mL graduated cylinder
100-mL graduated cylinder
500-mL Erlenmeyer flask
Pyrex funnels
500-mL round-bottom flask
40-mL vial with Teflon-lined cap
250-mL separatory funnel
13 mm i.d. x 100 mm glass liquid chromatography column
22 mm i.d. x 300 mm glass liquid chromatography column
250-mL Kuderna-Danish concentrator with 3-ball Snyder column
4-mL glass vial with Teflon-lined cap
250-μL conical vial with crimp cap (for GC autosampler)
Shaker table
Centrifuge
Rotary evaporator with hot water bath and water aspirator vacuum system
Hot water bath for Kuderna-Danish concentration
Inert gas (nitrogen recommended) evaporation/concentration system

Chemicals

Reagent water (Milli-Q or equivalent)
Hexane (pesticide grade or equivalent)
Toluene (pesticide grade or equivalent)
Tropolone (Alfa Products Inc., #16526)
Azulene
Perylene
6:4:3 Cyclohexane:methanol:methylene chloride
Sand, fired
Sodium sulfate, anhydrous and fired
Sulfuric acid, 10 N (ACS reagent grade or better)
Hydrobromic acid, 48% (ACS reagent grade or better)
Florisil, PR-grade or equivalent activated at 130°C for a minimum of 5 h.
Silica Gel, 1% deactivated. 60/80 mesh.
Tetrabutyltin, 95% (Alfa Products, Inc., #71132)
Tributyltin chloride, 95% (Alfa Products, Inc., #71128)
Dibutyltin dichloride, 99.1% (Alfa Products, Inc., #71127)
Butyltin trichloride 99.8% (Alfa Products, Inc., #71125)
Tripropyltin chloride 97% (Alfa Products, Inc., #71122)
Dipropyltin dichloride, (Organometallics, Inc. no catalog number)
1.9 M n-pentylmagnesium bromide in diethylether (Alfa Products, Inc., #87296)
0.05% (w/w) tropolone in toluene
PFTBA (mass spectrometer calibration compound)

Preparation of 0.05 Percent (w/w) Tropolone in Toluene: To 3 L of toluene, add 1.3 g Tropolone. Stir with a Teflon-coated magnetic bar until dissolved.

Gas Chromatography Equipment: For both GC/FPD and GC/MS analyses, a temperature programmable capillary gas chromatograph with split/splitless injection system, fitted with a 30-m 0.25 mm i.d. 5% methyl-/95% phenylsilicone capillary column (J&W Inc. DB-5 or equivalent) is required. Helium carrier gas, air, and hydrogen are required.

GC/FPD Detection/Analysis: A hydrogen-air flame photometric detector is required. Hydrogen flow set at 75 mL/min, air flow set at 100 mL/min, and helium make-up gas flow set at 40 mL/min. The detector base is set at 250°C. For organotin analysis, the detector is equipped with a 610-nm bandpass filter (Ealing Electroptics, Inc. #35-3847 or equivalent).

GC/FPD Data Acquisition/Reduction: GC/FPD data should be acquired with an electronic integrator/plotter or GC data system capable of producing, at a minimum, a graphical representation of the GC/FPD chromatogram, GC peak retention times, peak areas, and peak heights. Data systems or integrators capable of automated quantification significantly increase sample throughput, and are highly recommended to reduce per sample costs.

Mass Spectrometry Detection/Analysis: Temperature programmable capillary gas chromatograph with split/splitless injection system, fitted with a 30 m x 0.25 mm i.d. 5% methyl-/95% phenylsilicone capillary column (J&W Inc. DB-5 or equivalent), interfaced to a quadrupole mass spectrometer capable of operation in selected ion monitoring mode. The GC/MS system should include a data system for mass spectrometry and quantitative data reduction.

2.2 PREPARATION OF STANDARDS

Stock and working standards for GC analysis are prepared according to the following procedures.

2.2.1 Individual Butyltin Stock Solutions

Individual solutions of monobutyltin trichloride, dibutyltin dichloride, tributyltin chloride, tetrabutyltin, tripropyltin chloride, and dipropyltin dichloride are prepared in hexane at the 5 mg/mL level according to the following procedure:

1. Accurately weigh out 150-200 mg of each butyltin compound, and transfer each to a separate 25-mL volumetric flask. Dilute to the mark with hexane. Label each flask appropriately.
2. Butyltins are reported in terms of their potential cation weight. Standard concentrations are not corrected for purity. To calculate concentration of appropriate compound use the following equations:

$$\text{mg/mL monobutyltin} = \frac{[(\text{mg MBT-Cl}) \times 176]}{[(25\ \text{mL}) \times 282]} = \text{mg/mL} \qquad (1)$$

$$\text{mg/mL dibutyltin} = \frac{[(\text{mg DBT-Cl}) \times 233]}{[(25\ \text{mL}) \times 304]} = \text{mg/mL} \qquad (2)$$

$$\text{mg/mL tributyltin} = \frac{[(\text{mg TBT-Cl}) \times 290]}{[(25\ \text{mL}) \times 325]} = \text{mg/mL} \qquad (3)$$

$$\text{mg/mL tetrabutyltin} = \frac{[\text{mg TTBT}]}{[25\ \text{mL}]} = \text{mg/mL} \qquad (4)$$

$$\text{mg/mL tripropyltin} = \frac{[(\text{mg TPT-Cl}) \times 248]}{[(25\ \text{mL}) \times 283]} = \text{mg/mL} \qquad (5)$$

$$\text{mg/mL dipropyltin} = \frac{[(\text{mg DPT-Cl}) \times 205]}{[25\ \text{mL} \times 276]} = \text{mg/mL} \qquad (6)$$

2.2.2 Preparation of Derivatized, Mixed Organotin Stock Solutions

A 50 µg/mL stock solution containing monobutyltin, dibutyltin, tributyltin, and tetrabutyltin is prepared in hexane. The labile compounds, MBT, DBT, TBT, are converted to the n-pentyl derivatives via a Grignard reaction prior to volumetric adjustment.

1. To a 25-mL pear-shaped flask, add ca. 10 mL hexane. To this hexane, add 1.00 mL of each 5 mg/mL stock solution of MBT, DBT, and TBT. Add 5 mL n-pentylmagnesium bromide, and allow to react a minimum of 15 min at room temperature. Every 5 min, mix by hand for approximately 15 seconds. After this 15 min period, add 25 mL distilled water and 10 mL 10 N H_2SO_4.
2. In a 250-mL separatory funnel, separate the hexane fraction from the water, and dry the hexane over 20 g Na_2SO_4.
3. Collect the hexane and pass through a 13 mm i.d. x 100 mm cleanup column packed with 2 g of Florisil. Elute the column with 30 mL hexane. Collect the eluate in a 100-mL volumetric flask. Add 1.00 mL 5 mg/mL TTBT stock solution. Dilute the entire mixture to 100 mL with hexane. Calculate the final concentrations (ca. 50 µg/mL), and label the flask appropriately.

Prepare individual solutions of derivatized tripropyltin and dipropyltin using the following procedure.

To a 25-mL pear-shaped flask, add 1.0 mL of the 5 mg/mL TPT (or DPT) stock to approximately 10 mL hexane, and derivatize following the procedure described above. Final volume is 100 mL, and the final concentration is ca. 50 µg/mL. Calculate the final concentration, and label the flask appropriately.

2.2.3 Preparation of Derivatized, Mixed Butyltin GC Standards

Prepare GC standards for analysis by making serial dilutions of the mixed, derivatized MBT, DBT, TBT, and TTBT solution. To each standard level, add the identical amount of derivatized TPT and DPT internal standards. Concentrations of the standards should range from about 0.1 µg/mL to 50 µg/mL. This standard concentration range covers the 0.5 µg/kg to 250 µg/kg analyte range in environmental samples. The concentration of the internal standard should be approximately 4 µg/mL in all standards, which corresponds to approximately the 20 µg/kg level in environmental samples. If higher concentrations are expected in environmental samples, the working range of the standards should be extended to bracket the expected analyte concentration.

2.2.4 Preparation of Tripropyltin Chloride Recovery Internal Standard

Tripropyltin is used as the recovery internal standard (RIS) for the analysis of butyltins in sediments. TPT chloride is added prior to extraction, derivatization and cleanup. A TPT chloride standard in acetone at approximately the 2 µg/mL level should be prepared by serial dilution of the TPT chloride stock solution prepared in Section 2.2.1.

2.2.5 Preparation of Derivatized Dipropyltin Quantitation Internal Standard

Derivatized dipropyltin is used as the quantitation internal standard (QIS) for the analysis of butyltins in sediments. Derivatized DPT is added just prior to GC/FPD analysis. A derivatized DPT standard in hexane should be prepared at approximately the 10 µg/mL level by serial dilution of the derivatized stock solution prepared in Section 2.2.2.

2.2.6 Preparation of Underivatized, Mixed Butyltin Fortification Standards

Fortification and recovery experiments in sediments may be carried out to test the method. Fortification spiking solutions are prepared by combining aliquots of monobutyltin trichloride, dibutyltin dichloride, tributyltin chloride, and tetrabutyltin stock solutions (preparation described in Section 2.2.1), and diluting in acetone. The concentration of the individual butyltins should be in the 1 µg/mL to 1000 µg/mL range, depending on the fortification level of interest. Spiking of 100-g sediment samples should be carried out with 0.1 to 1 mL of selected spiking solutions. Fortification should be carried out by spiking sediment after the overlying water has been removed, and mixing for 15-30 min prior to extraction.

2.3 ANALYTICAL PROCEDURES

Figure 1 presents a flowchart depicting the extraction, derivitization and cleanup steps for the analysis of butyltin species in sediments. The extraction, derivatization, and cleanup steps are presented in order below.

1. Remove extraneous material (biota, sticks, rocks, etc.) from sediment sample. Drain overlying water. Composite sample by stirring.
2. Transfer 200-300 g sediment to a Teflon jar. Centrifuge at 2500 rpm for 10 min to remove interstitial water. Drain off water.
3. Transfer 100 g (±0.1 g) of the sediment into a Teflon jar. Add the appropriate amount of RIS TPT to the sediment. Mix thoroughly for 5 min using shaker table.
4. Add 25 mL distilled water and 5 mL 48 percent hydrobromic acid. Mix on shaker table for 1 h.
5. Add 60 mL 0.05 percent tropolone in toluene to the mixture. Agitate on shaker table for 1 h.
6. Centrifuge sample at 2500 rpm for 5 min. Transfer liquid to a 500-mL separatory funnel. Drain off lower aqueous layer, and return to Teflon jar that contains the sediment. Transfer the organic layer to a 500-mL Erlenmeyer flask.

7. Repeat steps 5 and 6. Combine organic extracts from the two extractions.

8. Add ca. 40 g sodium sulfate to the toluene, and desiccate for 15-30 min.

9. Transfer the toluene, with rinsing, to a 500-mL roundbottom flask. Rotary evaporate the solvent to ca. 10 mL at a waterbath temperature of 60-70°C.

10. Exchange the toluene for hexane by adding ca. 20 mL hexane to the toluene, and rotary evaporating to ca. 10 mL at a water bath temperature of 60-70°C. Perform this step twice.

11. Add 5 mL of the derivatizing agent 1.9 M n-pentylmagnesium bromide directly into the roundbottom flask. Agitate the samples by hand every 5 min, and allow to react a minimum of 15 min at room temperature. A precipitate will form during the reaction.

12. Quench the reaction by adding ca. 25 mL distilled water followed by ca. 10 mL 10 N H_2SO_4 and mix well until the precipitate that formed during derivatization is dissolved. Transfer the mixture to a 250-mL separatory funnel. Allow layers to separate. Drain off lower aqueous layer and discard.

13. Transfer the hexane from the separatory funnel to a 40-mL vial, and reduce in concentration to ca. 1-2 mL by nitrogen gas evaporation.

14. Prepare the Florisil/silica gel column. Put a pre-fired glass wool plug in the bottom of a 22 mm i.d. x 300 mm chromatography column. Add 16 g Florisil (activated a minimum of 5 h in a 130°C oven), tapping the column while adding to facilitate a uniform pack. Add 7 g 1 percent deactivated silica gel. Top the column with approximately 2 g sodium sulfate. Prewet the column with a minimum amount of hexane. Add the hexane extract. Elute with 100 mL hexane, and collect the eluate in a 250-mL Kuderna-Danish receiver.

15. Fit the Kuderna-Danish receiver with a 3-ball Snyder column, prewet the balls with hexane, and reduce the volume to ca. 5 mL at a water bath temperature of 100°C. Transfer the solution to a 20-mL vial, and reduce to ca. 500 µL by successive nitrogen evaporations.

16. Add 100 µL of the appropriate concentration QIS DPT (previously derivatized).

17. Transfer the final extract volume to a 2-mL vial, and submit for GC/FPD analysis.

2.3.1 Optional GPC Cleanup

If sediments contain large amounts of hydrocarbons or biological organic matter that are not removed by the standard Florisil/silica gel column, gel permeation chromatography (GPC) may be employed to further clean the sample prior to analysis. Samples should be analyzed by GC/FPD after being carried through the standard extraction and cleanup procedure (Section 2.3). If serious interferences are seen in the GC/FPD analysis, the sample extract may be subjected to GPC cleanup and reanalyzed. The following steps describe the preparation of the GPC column and the procedure for processing sample extracts.

1. Column Preparation. A glass chromatography column is packed with pre-swelled Sephadex LH-20. Note that the column system is operated isocratically, using only 6:4:3 cyclohexane:methanol:methylene chloride.

   1.1. Add 10 mL 6:4:3 cyclohexane:methanol:methylene chloride to a 19-mm i.d. chromatography column equipped with a solvent reservoir. Add ca. 1 mL sand to the column. Tap the column to settle sand.

   1.2. Pour approximately 80 mL pre-swelled (12 h) Sephadex LH-20 into the column. Allow the Sephadex to settle for 10 min. Open the stopcock, and allow 80 mL of solvent to flow through the system. Leave ca. 30 mL of solvent in the column reservoir, and allow the gel to settle overnight (12 h).

   1.3. Elute 30 mL of solvent from the system. Remove excess gel packing from the top of the column so that the final bed length is 26.5 cm.

   1.4. Add ca. 1 mL sand to the top of the packing so it forms an even layer.

   1.5. Examine the column for entrained air bubbles. If bubbles are present, elute the column with ca. 250 mL of warm (30-35°C) 6:4:3 solvent. If the bubbles persist, repack the column.

2. Column calibration. The GPC column must be calibrated to ensure that it is packed properly and yields the proper elution pattern for sample cleanup.

   2.1. Prepare column calibration solution of 10 mg/mL azulene and 1 mg/mL perylene in 6:4:3 solvent.

2.2. Place a 100-mL graduated cylinder under the column to collect effluent.

2.3. Using a pipet, carefully remove any excess 6:4:3 solvent from the top of the packing.

2.4. Using a pipet, apply 2 mL of the azulene/perylene solution to the top of the column. Apply solution carefully so as not to disturb the packing.

2.5. Open the stopcock, drain so the liquid is at the top of the packing. Add ca. 0.5 mL of solvent, and again drain to top of packing. Repeat step 2.4 again.

2.6. Add 100 mL of solvent to the reservoir, and open the stopcock. Elute the solvent until all the perylene (second colored band) has emerged. Record the volume at which the azulene and perylene start and finish eluting.

2.7. If the azulene emerges in the 50-65 mL range, and the perylene emerges in the 60-80 mL range without any distinct tailing, the column is acceptable and may be used for sample cleanup.

2.8. Discard the eluate, and flush the column by passing 90 mL of solvent through the column.

3. Column conditioning. Active sites in the packing are deactivated by conditioning with lipoidal material.

3.1. Add 1 mL 6:4:3 solvent to 1 g Crisco vegetable oil in a 4-mL vial.

3.2. Charge the sample onto the column. Drain to top of packing.

3.3. Elute the column with 150 mL 6:4:3 solvent.
3.4. The column is now ready for sample processing.

4. Sample processing. Sample extracts previously derivatized and processed by Florisil/silica gel chromatography (Section 2.3) may be further processed by GPC using the following procedure.

4.1. The sample extract containing the derivatized butyltins and the QIS DPT should be applied to the GPC column in a maximum volume of 1 mL.

4.2. Elute the column with 80 mL 6:4:3 solvent. Collect the solvent in a Kuderna-Danish concentrator. Follow step 15 in Section 2.3 and reduce the eluate in volume to ca. 0.5 mL. Submit for GC/FPD analysis.

4.3. Flush the column with approximately 150 mL of additional 6:4:3 solvent. The column is now ready to be used for processing another sample.

2.3.2 Optional Sulfur Cleanup with Activated Copper

Experimental evidence suggests that ambient sulfur is removed from most sediments through the reaction of sulfur with the Grignard reagent. However, if ambient levels of sulfur are not removed from the sample during processing (as noted by characteristic GC/FPD interference; see Section 3.7), additional processing of sample extracts may be carried out using activated copper to remove low levels of sulfur. Samples should be analyzed by GC/FPD after being carried through the standard extraction and cleanup procedure (Section 2.3). If serious sulfur interferences are seen in the GC/FPD analysis, the sample extract may be subjected to activated copper cleanup and reanalyzed.

1. Activated copper is prepared no more than 1 h before sample cleanup by mixing approximately 20 g of copper turnings with 5 mL 6N HCl in a 50-mL beaker. The mixture is stirred until the copper turns pink. The copper is successively washed with 50-mL aliquots of distilled water, methanol, methylene chloride, and hexane.

2. Hexane extracts of samples (any time after derivitization) are brought to a volume of ca. 5 mL, and mixed with ca. 5 g activated copper. The solution is shaken gently for 2-3 min. If sulfur is present, the copper will turn black. Small amounts of copper are continually added until the copper remains pink. The extract is decanted quantitatively, and either subjected to further chromatographic cleanup (i.e., GPC), or concentrated by nitrogen evaporation and resubmitted for GC/FPD analysis.

2.4 INSTRUMENTATION

The instruments used and the conditions under which they were operated during the testing and validation study described in this document are presented in this section. Comparable instrumentation may be used and will produce similar results.

Quantitative analysis of butyltin compounds in the sediment extracts is accomplished by capillary gas chromatography followed by flame photometric detection. Figure 2 is a flowchart depicting the steps followed for GC/FPD analysis of butyltins in sediment samples. Detailed, step-by-step guidelines for GC/FPD analysis are presented below.

Optional confirmation of butyltin residues in sediment samples is performed by capillary gas chromatography/mass spectrometry (GC/MS) using selected ion monitoring (SIM) techniques.

2.4.1 GC/FPD

A Hewlett-Packard model 5890 capillary GC is fitted with a 30-m J&W Inc. DB-5 capillary column (0.25 mm i.d., 0.25 μm film thickness). Helium is used as the carrier gas (linear velocity of 30 cm/s) and as the detector make-up gas (flow of 40 mL/min). The injector and detector temperatures are maintained at 250°C. During analysis, the GC oven is temperature programmed from an initial temperature of 60°C (hold 1 min) to 250°C at 20°C/min, with a 4-min final hold time.

Flame photometric detection is carried out using a Hewlett-Packard model 19256A FPD. Tin-selective response is obtained by fitting the FPD photomultiplier tube with a 610-nm bandpass filter (Ealing Electroptics, Inc. #35-3847). The FPD flame is maintained with a hydrogen-air mixture. Hydrogen flow is set at 75 mL/min, and air flow is set at 100 mL/min.

Figure 3 shows a GC/FPD chromatogram of a standard containing the n-pentyl derivatives of TPT, DPT, TBT, DBT, MBT, as well as underivatized TTBT obtained using the GC conditions described above. Table 3 lists the gas chromatography retention times and relative retention times of the MBT, DBT, TBT, and TTBT relative to the QIS DPT and the RIS TPT. Gas chromatography retention times of individual butyltin compounds were obtained by analyzing individual derivatized butyltin compounds, determining their respective elution times, and confirming the structure of the compounds by mass spectrometry.

2.4.2 GC/MS

A Hewlett-Packard model 5970B MSD capillary GC/MS is fitted with a 30-m J&W Inc. DB-5 capillary column (0.25 mm i.d., 0.25 μm film thickness). Helium is used as the carrier gas (linear velocity of 30 cm/s). The injector temperature is maintained at 300°C. The mass spectrometer is operated with a source pressure of 5-7 x 10^{-5} torr. During analysis, the GC oven is temperature programmed from an initial temperature of 60°C (hold 1 min) to 250°C at 20°C/min, with a 4-min final hold time. Because the analytical column and GC temperature program used in GC/MS analysis of butyltins is the same as that used in GC/FPD analysis, the relative retention times of the butyltin analytes to the internal standard TPT are the same as those listed in Table 3.

The mass spectrometer is operated in selected ion monitoring mode, at a electron multiplier (EM) voltage 200 volts above that required to give an EM gain of 10^5 (normally 1700 - 1800 volts). Before operation in SIM, the spectrometer is calibrated with perfluorotributylamine (PFTBA). Selected ions used for analysis of butyltin compounds and the RIS TPT were determined from the full scan spectra obtained from analysis of the n-pentyl derivatives of the individual butyltin and propyltin compounds. Figures 4 through 9 show the full scan (50-500 amu) spectra for TTBT and the n-pentyl derivatives of MBT, DBT, TBT, TPT, and DPT. Note that the spectra are unique, by virtue of tin's 10 naturally occurring isotopes. The ions selected for SIM analysis from these spectra are both diagnostic and abundant. The following ions are scanned at 100 msec/ion:

Compound	Ions			
DPT	121	207	235	305
TPT	121	165	235	277
MBT	119	121	193	319
DBT	119	121	249	319
TBT	119	121	249	305
TTBT	119	121	235	291

If quantitation of butyltins in samples by SIM is necessary, the underlined ions in the above table may be extracted and used for quantitative analysis. The selection of the quantitation ions was based both on the sensitivity obtained using the ion signal and on the lack of interference observed in the extracted ion chromatograms of authentic water sample extracts (Uhler and Steinhauer, 1988).

2.4.3 GC/FPD Calibration Procedures

Retention time (RT) and RT windows are established prior to sample analysis by performing three analyses of a GC/FPD standard containing TTBT and the n-pentyl derivatives of TBT, DBT, MBT, TPT and DPT over a 72-hour period. This procedure defines the instrumental variability that will be encountered during normal operation of the gas chromatograph. The average retention time and the standard deviation (σ) of the average retention time for each compound is computed. The retention time window is $RT \pm 3\sigma$. During sample analysis, peaks that fall within the RT window are tentatively identified as the target analyte(s). This RT calibration procedure should be carried out initially, and repeated only if major maintenance or alterations (changes in flow rate, new column installation, etc.) are made to the chromatographic system.

Three calibration standards are analyzed by GC/FPD prior to analysis of a batch of samples. These standards are designated LOW, MID, and HIGH level, and ideally bracket the analyte concentration range. Internal standard response factors (RF) for each butyltin analyte are calculated relative to the internal standard TPT at each concentration level. For GC/FPD analysis, the peak height, rather than peak area, is found to be more accurate for butyltin analysis.

Internal standard RF are calculated using the following formula:

$$RF = \frac{H_i C_{is}}{H_{is} C_i} \qquad (7)$$

where H_i is the peak height of the analyte in the standard solution, H_{is} is the peak height of the internal standard in the standard solution, C_i is the concentration (or amount) of analyte in the standard solution, and C_{is} is the concentration (or amount) of internal standard in the standard solution.

The results of a calibration are considered acceptable if the RF percent relative standard deviation (%RSD) among the three RFs are within 20 percent. The %RSD is calculated using the relationship:

$$\%RSD = \frac{\sigma}{\bar{X}} \times 100 \qquad (8)$$

Where X is the mean of the three RF values, and σ is the standard deviation of the mean. If the %RSD exceeds 20 percent, a new calibration should be performed.

In this study, the concentration range covered by the three GC calibration standards was about a factor of 20. The %RSDs for the RF's calculated from these calibrations were generally 10 percent or less.

After the analysis of 12 samples, a MID level check calibration standard is analyzed. The original calibration is considered valid if the RF from this analysis is within ± 20 percent of the original mean RF. If the check MID meets the acceptance criterion, the next 12 samples are quantified using the original RFs. If the check MID differs from the original average RF by more than 20 percent, a new three-point calibration must be performed. As a matter of routine practice, a new three-point calibration curve should be run every 24 h.

2.5 QUALITY CONTROL

The quality of the analytical procedure and the data that result from the analysis of a set of samples is maintained by following quality assurance and quality control procedures. Every batch of samples (maximum of 12) should be accompanied by a procedural blank. A procedural blank is 25 mL of distilled water carried through the analytical procedure. Matrix spike samples should be prepared with every suite of samples. The minimum number of matrix spike samples should be 10 percent of the total number of samples to be analyzed in one project. A matrix spike is prepared by fortifying a sediment sample with approximately 10 times the expected background level of TBT. The matrix spike is carried through the analytical procedure, and the recovery of the analytes is determined.

The procedural blank must be free from butyltins as well as any other interferences in the chromatographic region where the analytes elute.

Recovery of TBT, DBT, and TTBT should fall in the range of 50 to 130 percent. Recovery of MBT should fall in the 10 to 100 percent range. Recovery of the RIS TPT, calculated relative to the QIS DPT, should fall in the range of 50 to 130 percent. If the recovery criteria are not met, remedial action and reanalysis are necessary.

The average response factor computed from the three-level calibration should have a percent RSD of $\leq$ 20 percent. The RFs of the MID level calibration check sample (run after the 12 samples) should be within ±20 percent of the mean RF of the original three-level calibration. If these calibration criteria are not met, the chromatographic system should be inspected, remedial action taken, and the calibration rerun.

2.6 CONFIRMATORY TECHNIQUES (GC/MS)

The method described here for the analysis of butyltins presents GC/FPD as the principal determinative technique. In practice, it may be necessary to confirm the presence of suspected butyltins in selected samples by GC/MS. Because of the very low levels of butyltins in sediments and the complex matrix in which they are usually found, selected ion monitoring (SIM) techniques are preferred over full mass scan techniques.

The confirmation of a butyltin analyte in a sample is based on the relative ratios of the mass fragments monitored relative to those ratios in a SIM spectrum of well-characterized butyltin standards. The expected ratios of the ions monitored for each n-pentyl butyltin species are presented in Table 4. The tolerance for the ratios, also presented in Table 4, are ± 20 percent of the expected ratios.

The confirmation process is carried out in the following fashion. A sample extract suspected of containing one or more butyltin residues (by virtue of GC/FPD results) is analyzed by GC/MS using SIM. If the suspected butyltin species is observed at the proper retention time in the extracted ion profile chromatogram, the relative ratios of the ions at the expected retention time of the butyltin in question are determined, and compared to the expected ratios presented in Table 4. If the ratios fall within the established tolerances, the presence of the butyltin in the sample extract is confirmed.

2.7 TIME REQUIRED FOR ANALYSIS

The complete analysis of butyltins in sediment samples (including extraction, cleanup, analysis, and calculations of final results) can be accomplished for 10 samples plus a procedural blank and spiked blank in roughly 28 man-hours:

o Sample extraction and derivitization	16 man-h
o Sample cleanup, concentration	6 man-h
o GC/FPD analysis. Include GC standard calibration check, visual inspection of chromatograms, and data compilation	6 man-h

Confirmatory analysis by GC/MS requires approximately the same time as that required by GC/FPD. GC/MS has the advantage of positive identification of all analytes, but, relative to GC/FPD, requires 1-2 more hours of data analysis by an experienced GC/MS operator. Additionally, the cost per sample will likely be higher because of greater GC/MS instrument use costs.

2.8 MODIFICATIONS OF POTENTIAL PROBLEMS TO THE ANALYTICAL METHOD

The method described in this report was tested with several estuarine sediments, representing a wide range in grain size and organic carbon content. No serious matrix interferences or matrix effects were observed. As described in Section 3.3.1, occasionally an early eluting, unidentified compound would coelute with TPT, making accurate TPT peak integration difficult. However, no interferences were noted in the region of the chromatograms where the analytes or the QIS DPT eluted. Some samples formed emulsions during extraction. As described in the Analytical Methods section (Section 2.3), emulsions can be reduced by addition of NaCl to the mixture. Any remaining emulsions are collected and easily broken down by mixing with sodium sulfate, and no significant loss of analyte (as judged from the results of the recovery experiments) was observed.

The FPD used in this study proved trouble free. Some investigators have suggested that during prolonged use, the optical windows of the FPD become coated with inorganic tin (breakdown product of the analytes), reducing overall sensitivity. During this study, approximately 500 samples were analyzed for butyltins without any noticeable loss of detector sensitivity. As a precaution, the sensitivity of the FPD should be monitored routinely by checking the absolute response of a mixed butyltin standard on a week-to-week basis. If noticeable degradation in sensitivity is observed between checks (reduction in sensitivity of > 20 percent), the FPD should be disassembled and the optical path cleaned according to procedures prescribed by the detector manufacturer.

The capillary column used in this study performed consistently throughout the study. Routine maintenance, consisting of changing the injection port liner at weekly intervals, and replacing the injection port septum approximately every 24 samples, conserved the GC system integrity. A capillary precolumn, consisting of 1-2 meters of deactivated fused silica capillary column installed between the injection port and the analytical column is recommended. If column degradation is suspected, the precolumn may be replaced while maintaining the integrity of the analytical column.

2.9 METHODS OF CALCULATION

Butyltin species in sediment sample extracts are quantified using the method of internal standards. The method of internal standards requires that prior to sample quantification, a response factor for an analyte relative to an internal standard compound be computed. As described in Section 2.4.3, response factors for each analyte are determined relative to the internal standard DPT from a three-level calibration. Samples are quantified using the following equation:

$$C_i\ (\mu g/kg) = \frac{(H_i) \times (C_{is})}{(H_{is}) \times (RF) \times (S)} \qquad (9)$$

where C_i is the concentration of the analyte in sample in µg/kg, H_i is the height of the analyte peak (arbitrary units), C_{is} is the amount of internal standard added to the sample extract (in µg), H_{is} is the peak height of the internal standard (arbitrary units), RF is the response factor for analyte i relative to the QIS DPT obtained from the three-point calibration, and S is the weight of the sediment sample in kg.

The following serves as an example calculation. A 0.1 kg sediment sample is extracted and analyzed for butyltin species. A total of 20 µg of dipropyltin quantitation internal standard is added to the extract. Prior to analysis, a three-point calibration is performed. Using the equation (7) presented in Section 2.4.3, a response factor of 0.75 for TBT is determined. Upon analysis, the GC/FPD chromatogram shows a response for TBT, with a measured peak height of 15 mv. The internal standard peak height is 10 mv. The variables required for calculation of the concentration of TBT in the original sample by using equation (9) are

$S = 0.1$ kg (original sample weight)

$H_i = 15$ mv (TBT response from analysis)

$H_{is} = 10$ mv (response for DPT internal standard from analysis)

$C_{is} = 20$ µg (amount of DPT added to extract)

$RF = 0.75$ (response factor for TBT relative to QIS DPT, determined from three-point calibration)

C_i = concentration of TBT in original sample

Substituting these values into equation (9) yields the following:

$$C_i = \frac{(15 \text{ mv}) \times (20\ \mu\text{g})}{(10 \text{ mv}) \times (0.75) \times (0.1 \text{ kg})} = 400\ \mu\text{g/kg TBT} \qquad (10)$$

The concentrations of other butyltin species in a sample are computed using equation (9) with the appropriate response factor and peak height data.

3.0 RESULTS AND DISCUSSION

3.1 TEST SEDIMENTS

Sediments from an estuarine area of Chesapeake Bay were collected and used in several phases of this work. One sample, CHB-1, was collected near the mouth of the South River in Maryland. A second, CHB-2, was collected from Love Point, Maryland, which is approximately mid-Bay on the Eastern Shore of Maryland. Both these sample sites were far removed from potential sources of TBT (marinas; drydocks), and were used to evaluate method performance during Phases 1, 2, and 4 of this study.

Two freshwater sediments were collected in Massachusetts. One sediment, designated DF-1, was collected from a freshwater lake in Duxbury, MA. The lake, Priest Pond, is located within a wildlife preserve. No boating activity is allowed on this lake. Thus, the sediments collected were free from any butyltin contamination.

The second freshwater sediment was collected from the Taunton River, just east of Taunton, MA. This river receives modest inputs of municipal runoff and sewer discharge, and is used for some recreational boating. For this study, the sediment was designated TR.

For Phase 3 work, several samples were collected from areas expected to have significant butyltin contamination. A sediment sample, designated HR, was collected from the Hampton Roads area of Virginia, near the mouth of the Chesapeake Bay. A sample, designated GB-1, was collected from the Galveston Bay ship channel in Texas. Finally, an estuarine sediment from Puget Sound, Washington, designated PS, was collected for testing.

Sediments were collected using a ponar grab sampler. A Teflon-coated scoop was used to sample the top 2 cm of the grab sample. Sediment was transferred

SAMPLE CROSS REFERENCE SHEET

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ATI #	CLIENT DESCRIPTION	DATE SAMPLED	MATRIX
9104-199-1	9A 0-0.3'	01/21/91	SEDIMENT
9104-199-2	8B 0-0.3'	01/22/91	SEDIMENT
9104-199-3	8C 0.3-2.0'	01/22/91	SEDIMENT
9104-199-4	2B 0-0.3'	01/24/91	SEDIMENT
9104-199-5	1A 0-0.3	01/24/91	SEDIMENT
9104-199-6	3A 0-0.3'	01/25/91	SEDIMENT
9104-199-7	4A 0-0.3'	01/28/91	SEDIMENT
9104-199-8	5A 0-0.3'	01/27/91	SEDIMENT
9104-199-9	6A 0-0.3'	01/30/91	SEDIMENT
9104-199-10	11 DUPLICATE	01/30/91	SEDIMENT
9104-199-11	7A 0-0.3'	01/30/91	SEDIMENT

----- TOTALS -----

MATRIX	# SAMPLES
SEDIMENT	11

ATI STANDARD DISPOSAL PRACTICE

The samples from this project will be disposed of in thirty (30) days from the date of this report. If an extended storage period is required, please contact our sample control department before the scheduled disposal date.

ATI I.D. # 9104-199

ANALYTICAL SCHEDULE

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

ANALYSIS	TECHNIQUE	REFERENCE	LAB
ARSENIC	ICAP	EPA 6010	R
BARIUM	ICAP	EPA 6010	R
CADMIUM	ICAP	EPA 6010	R
CHROMIUM	ICAP	EPA 6010	R
COPPER	ICAP	EPA 6010	R
LEAD	ICAP	EPA 6010	R
MERCURY	AA/COLD VAPOR	EPA 7470	R
NICKEL	ICAP	EPA 6010	R
SELENIUM	ICAP	EPA 6010	R
SILVER	AA/F	EPA 7760	R
ZINC	AA/F	EPA 7950	R

R = ATI - Renton
SD = ATI - San Diego
T = ATI - Tempe
PNR = ATI - Pensacola
FC = ATI - Fort Collins
SUB = Subcontract

TOXICITY CHARACTERISTIC
METALS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.
MATRIX : LEACHATE

PARAMETER	DATE PREPARED	DATE ANALYZED
ARSENIC	04/23/91	04/23/91
BARIUM	04/23/91	04/23/91
CADMIUM	04/23/91	04/23/91
CHROMIUM	04/23/91	04/23/91
COPPER	04/23/91	04/23/91
LEAD	04/23/91	04/23/91
MERCURY	04/23/91	04/30/91
NICKEL	04/23/91	04/23/91
SELENIUM	04/23/91	04/23/91
SILVER	04/23/91	04/25/91
ZINC	04/23/91	04/25/91

TOXICITY CHARACTERISTIC
METALS RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : LEACHATE

UNITS : mg/L

PARAMETER	9A 0-0.3′ -1	8B 0-0.3′ -2	8C 0.3-2.0′ -3	2B 0-0.3′ -4	1A 0-0.3′ -5
ARSENIC	<0.05	<0.05	<0.05	0.05	0.17
BARIUM	0.31	0.20	0.081	0.13	0.30
CADMIUM	0.004	0.008	0.018	0.046	0.017
CHROMIUM	0.006	<0.005	0.006	<0.005	0.006
COPPER	0.019	0.051	0.16	2.8	0.051
LEAD	<0.02	0.23	0.21	0.52	0.58
MERCURY	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
NICKEL	0.03	0.06	0.26	0.30	0.32
SELENIUM	<0.05	<0.05	<0.05	<0.05	<0.05
SILVER	<0.02	<0.02	<0.02	<0.02	<0.02
ZINC	0.38	2.0	3.2	83	59

TOXICITY CHARACTERISTIC METALS RESULTS

CLIENT : GEOENGINEERS, INC.
PROJECT # : 1299-003-B04
PROJECT NAME : UNIMAR, INC.

MATRIX : LEACHATE

UNITS : mg/L

PARAMETER	3A 0-0.3′ -6	4A 0-0.3′ -7	5A 0-0.3′ -8	6A 0-0.3′ -9
ARSENIC	0.08	<0.05	<0.05	<0.05
BARIUM	0.19	0.060	0.067	0.33
CADMIUM	0.011	0.042	0.050	0.013
CHROMIUM	<0.005	<0.005	0.012	0.005
COPPER	0.61	3.7	1.8	0.064
LEAD	0.63	3.0	1.6	0.28
MERCURY	<0.0005	0.0005	<0.0005	<0.0005
NICKEL	0.14	0.38	0.41	0.10
SELENIUM	<0.05	<0.05	<0.05	<0.05
SILVER	<0.02	<0.02	<0.02	<0.02
ZINC	19	48	18	8.2

ATI I.D. # 9104-199

TOXICITY CHARACTERISTIC
METALS RESULTS

CLIENT	: GEOENGINEERS, INC.	MATRIX	: LEACHATE
PROJECT #	: 1299-003-B04		
PROJECT NAME	: UNIMAR, INC.	UNITS	: mg/L

PARAMETER	11 DUPLICATE -10	7A 0-0.3' -11	REAGENT BLANK
ARSENIC	<0.05	<0.05	<0.05
BARIUM	0.12	0.075	0.004
CADMIUM	0.012	0.028	<0.002
CHROMIUM	0.006	0.008	<0.005
COPPER	0.26	1.6	<0.002
LEAD	0.54	1.4	<0.02
MERCURY	<0.0005	<0.0005	<0.0005
NICKEL	0.16	0.41	<0.01
SELENIUM	<0.05	<0.05	<0.05
SILVER	<0.02	<0.02	<0.02
ZINC	10	15	<0.01

TOXICITY CHARACTERISTIC
METALS QUALITY CONTROL

CLIENT	: GEOENGINEERS, INC.	MATRIX	: LEACHATE
PROJECT #	: 1299-003-B04		
PROJECT NAME	: UNIMAR, INC.	UNITS	: mg/L

COMPOUND	ATI I.D.	SAMPLE RESULT	DUP RESULT	RPD	SPIKED SAMPLE	SPIKE CONC	% REC
ARSENIC	9104-199-9	<0.05	<0.05	NC	N/A	N/A	N/A
ARSENIC	BLANK SPIKE	<0.5	N/A	N/A	58.7	50.0	117
BARIUM	9104-199-9	0.33	0.33	0	9.20	10.0	N/A
CADMIUM	9104-199-9	0.013	0.012	8	N/A	N/A	N/A
CADMIUM	BLANK SPIKE	<0.02	N/A	N/A	52.2	50.0	104
CHROMIUM	9104-199-9	0.005	<0.005	NC	2.16	2.50	86
COPPER	9104-199-9	0.064	0.065	2	2.30	2.50	89
LEAD	9104-199-9	0.28	0.29	4	N/A	N/A	N/A
LEAD	BLANK SPIKE	<0.2	N/A	N/A	52.5	50.0	105
MERCURY	9104-199-11	<0.0005	<0.0005	NC	0.0022	0.0020	110
NICKEL	9104-199-9	0.10	0.10	0	2.37	2.50	91
SELENIUM	9104-199-9	<0.05	<0.05	NC	N/A	N/A	N/A
SELENIUM	BLANK SPIKE	<0.5	N/A	N/A	62.5	50.0	125
SILVER	9104-199-9	<0.02	<0.02	NC	0.94	1.0	94
ZINC	9104-199-9	8.2	8.2	0	11	2.5	112

NC = Not calculable.

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Concentration}} \times 100$$

$$\text{RPD (Relative \% Difference)} = \frac{(\text{Sample Result} - \text{Duplicate Result})}{\text{Average Result}} \times 100$$

Analytical Technologies, Inc.

560 Naches Avenue SW, Suite 101 Renton, WA 98055 (206)228-8335

DATE 1/21/91 PAGE 1 OF 1

Chain of Custody

LABORATORY NUMBER: ~~9101-148~~ 9104-199

PROJECT MANAGER: Paul Werner
COMPANY: Geo Engineers, Inc
ADDRESS: 140 7th Ave NE Suite 105
Bellevue, WA 98005
PHONE: 746-5200
SAMPLED BY: P. Werner

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

	SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13) *	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8) **			NUMBER OF CONTAINERS	
10C	VanVeen-Rinseate	1/21/91	1215	Water	1						X		X				X							X								8	✓
10A	Skelby Rinseate	1/21/91	1345	Water	2						X		X				X							X								8	✓
9A	Bottom Sediment 9A	1/21/91	1540	Soil	3					X			X				X							X					X			1	✓ 1 liter
9B	〃 9B	1/21/91	1610	Soil	4								X				X							X								1	✓ 1

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PROJECT INFORMATION

PROJECT NUMBER: 1299-003-B04
PROJECT NAME: Unimar, Inc
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	18
COC SEALS/INTACT? Y/N/NA	Y/Y
RECEIVED GOOD COND./COLD	Y/Y
RECEIVED VIA: Courier	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:
SEALS 01299-61 PCW * EPTOX TOTALS + Cu, Ni, Zn
01299-62 PCW ** TCLP METALS + Cu, Ni, Zn

RELINQUISHED BY: 1.
Signature: Paul Werner Time: [illegible]:45
Printed Name: Paul Werner Date: 1/21/91
Company: Geo Eng.

RELINQUISHED BY: 2.
Signature: Time:
Printed Name: Date:
Company:

RELINQUISHED BY: 3.
Signature: Time:
Printed Name: Date:
Company:

RECEIVED BY: 1.
Signature: Time:
Printed Name: Date:
Company:

RECEIVED BY: 2.
Signature: Time:
Printed Name: Date:
Company:

RECEIVED BY: (LAB) 3.
Signature: Time: 1000 A
Printed Name: V. PENNICK Date: 1/22/91
Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINA

AnalyticalTechnologies,Inc.

560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 1/22/91 PAGE 1 OF 2

LABORATORY NUMBER: ~~9101-167~~ 9104-199

PROJECT MANAGER: Paul Werner

COMPANY: Geo Engineers

ADDRESS: 140th Ave NE Bellevue WA 98005

PHONE: 746-5200 SAMPLED BY: P. Werner

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

	SAMPLE ID		DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	~~TOX 9020~~ * METALS SCREEN	% Moisture	EP TOX Metals (8) EP EXT	~~Priority Pollutant Metals (13)~~ *	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)	Tributyl Tin TBT	Hardness	NUMBER OF CONTAINERS
10E	BETA RINSEATE		1/22/91	1030	WATER	-1																			X						+		2
	8A WATER SAMPLE		1/22/91	1035	WATER	-2																			X						X	X	3
-2	8B Bottom Sediment 0-0.3'		↓	1320	Soil	-3					X	X		X				X							X					X			1
spilled	8C Bottom Sediment 0.3-2.0'			13:59	Soil	-4																			~~X~~	~~Sori~~				~~Sori~~	~~X~~		1
	8D	0.3-0.5'		14:00		-5						~~X~~ NO		~~X~~ NO				~~X~~ NO				X	YES		~~X~~ NO				NO	~~X~~	1/23/91		1
	8E	0.5-1.0'		13:27		-6																X											1
	8F	1.0-1.5'		1329		-7						~~X~~ NO	[illegible]					X				X											1
	8G	1.5-2.0'		1332		-8																X											1
	8H	2.0-2.5'		1334		-9																X											1
	8I	2.5-3.0'		1340		-10																X											1
	8J	3.0-3.5'		1350		-11																X											1
	8K	↓ 3.5-4.0'	↓	1353	↓	-12																X											1

C – 267

PROJECT INFORMATION

PROJECT NUMBER: 1299-003-B04

PROJECT NAME: Unimar

PURCHASE ORDER NUMBER:

ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS

COC SEALS/INTACT? Y/N/NA Y

RECEIVED GOOD COND./COLD Y

RECEIVED VIA: Courier

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK

GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:

Seals 1299-03 PGW
1299-04 PGW
* As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Se, Ag, Zn
** Cd, Cu, Pb, Zn

RELINQUISHED BY: 1.
Signature: Paul Werner Time: 15:30
Printed Name: Paul Werner Date: 1/22/91
Company: GEI

RELINQUISHED BY: 2.
Signature: Time:
Printed Name: Date:
Company:

RELINQUISHED BY: 3.
Signature: Time:
Printed Name: Date:
Company:

RECEIVED BY: 1.
Signature: D.L. Thomas Time: 4:40
Printed Name: D.L. Thomas Date: 1-23-91
Company: ATI

RECEIVED BY: 2.
Signature: Time:
Printed Name: Date:
Company:

RECEIVED BY: (LAB) 3.
Signature: Time:
Printed Name: Date:
Company: Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINATOR

Nº 145

AnalyticalTechnologies,Inc.

560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 1/21/91 PAGE 1 OF 1

LABORATORY NUMBER: ~~9101-148~~ 9104-199

PROJECT MANAGER: Paul Werner
COMPANY: Geo Engineers, Inc
ADDRESS: 140 Th Ave NE Suite 105
Bellevue, WA 98005
PHONE: 746-5200
SAMPLED BY: P. Werner

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

	SAMPLE ID	DATE	TIME	MATRIX	LAB ID
10C	VAN VEEN - RINSEATE	1/21/91	1215	Water	1
10A	Shelby Rinseate	1/21/91	1345	Water	2
9A	Bottom Sediment 9A	1/21/91	1540	Soil	3
9B	" 9B	1/21/91	1610	Soil	4

ANALYSIS REQUEST

LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13) *	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8) **			NUMBER OF CONTAINERS	
1						X		X				X							X								8	✓
2						X		X				X							X								8	✓
3					X			X				X							X					X			1	✓ 1 liter
4								X				X							X								1	✓ 1

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PROJECT INFORMATION

PROJECT NUMBER: 1299-003-B04
PROJECT NAME: Unimar, Inc
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	18
COC SEALS/INTACT? Y/N/NA	Y/Y
RECEIVED GOOD COND./COLD	Y/Y
RECEIVED VIA: Courier	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK

GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:
SEALS 01299-61 PLW * EPTOX TOTALS + Cu, Ni, Zn
01299-62 PLW ** TCLP METALS + Cu, Ni, Zn

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 1:45	Signature: Time:	Signature: Time:
Printed Name: PAUL WERNER Date: 1/21/91	Printed Name: Date:	Printed Name: Date:
Company: GEO ENG	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: Time:	Signature: Time:	Signature: Time: 1000A
Printed Name: Date:	Printed Name: Date:	Printed Name: U. PENNICK Date: 1/22/91
Company:	Company:	Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINA

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Chain of Custody

DATE 1/22/91 PAGE 1 OF 2

LABORATORY NUMBER: ~~9101-167~~ 9104-199

PROJECT MANAGER: Paul Werner
COMPANY: Geo Engineers
ADDRESS: 140th Ave NE Bellevue WA 98005
PHONE: 746-5200 SAMPLED BY: P. Werner

SAMPLE DISPOSAL INSTRUCTIONS
☒ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	~~TOX~~ 9020 *METAL SCREEN	% Moisture	EP TOX Metals (8) EP EXT	~~Priority Pollutant Metals (13)~~ *	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)	Tributyl Tin TBT	Hardness	NUMBER OF CONTAINERS
10E Beta Rinseate	1/22/91	10:30	Water	-1																			X						X		2
8A Water Sample	1/22/91	10:35	Water	-2																			X						X	X	3
8B Bottom Sediment 0-0.3'		13:20	Soil	-3					X	X		X				X							X					X			1
8C Bottom Sediment 0.3-2.0'		13:59	Soil ~~[illegible]~~	-4																			~~X~~ 20:i					20:i ~~X~~			1
8D 0.3-0.5'		14:00		-5						~~X~~ NO		~~X~~ NO				~~X~~ NO				X YES			~~X~~ NO					NO ~~X~~	1/23/91		1
8E 0.5-1.0'		13:27		-6																X											1
8F 1.0-1.5'		13:29		-7						NO ~~X~~	[illegible]					X				X											1
8G 1.5-2.0'		13:32		-8																X											1
8H 2.0-2.5'		13:34		-9																X											1
8I 2.5-3.0'		13:40		-10																X											1
8J 3.0-3.5		13:50		-11																X											1
8K 3.5-4.0	↓	13:53	↓	-12																X											1

-2 [illegible]

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PROJECT INFORMATION

PROJECT NUMBER: 1299-003-B04
PROJECT NAME: Unimar
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	
COC SEALS/INTACT? Y/N/NA	Y
RECEIVED GOOD COND./COLD	Y
RECEIVED VIA: Courier	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:
Seals 1299-03 PGW
1299-04 PGW
* As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Se, Ag, Zn
** Cd, Cu, Pb, Zn

RELINQUISHED BY: 1.	**RELINQUISHED BY: 2.**	**RELINQUISHED BY: 3.**
Signature: Paul Werner Time: 15:30	Signature: Time:	Signature: Time:
Printed Name: Paul Werner Date: 1/22/91	Printed Name: Date:	Printed Name: Date:
Company: GEI	Company:	Company:
RECEIVED BY: 1.	**RECEIVED BY: 2.**	**RECEIVED BY: (LAB) 3.**
Signature: D.L. Thomas Time: 4:40	Signature: Time:	Signature: Time:
Printed Name: D.L. Thomas Date: 1-23-91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINATOR

No. ..145

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DATE 1/22/91 PAGE 2 OF 3

Chain of Custody

LABORATORY NUMBER: ~~9101-169~~ 9104-199

PROJECT MANAGER: PAUL WERNER
COMPANY: GEO ENGINEERS
ADDRESS: SEE PAGE 1
PHONE: 746-5200 SAMPLED BY: P. WERNER

SAMPLE DISPOSAL INSTRUCTIONS

☐ ATI Disposal @ $5.00 each ☐ Return

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					ANALYSIS REQUEST																			TCLP ONLY							
SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) EP EXT	~~Priority Pollutant Metals (13)~~	8080 Pesticide (4)	8240 ZH-EXT	8270	8150 Herbicides (2)	Metals (8)	Metals Screen	Tributyl Tin	NUMBER OF CONTAINERS
Bottom SEDIMENT	/																														
8L ; 4.0 - 4.5'	1/22/91	1355	Soil	-13																									X		1
8M 4.5 - 4.8'	↓	1405	↓	-14																									X		1
8C	1/22/91	1359	H_2O	-15																			X							X	
-3 8C	↓	↓	sed	-16						X		X				X							X					X			

PROJECT INFORMATION

PROJECT NUMBER: 1299-003-B04
PROJECT NAME: Unimar, Inc
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	
COC SEALS/INTACT? Y/N/NA	y
RECEIVED GOOD COND./COLD	y
RECEIVED VIA: Courier	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)
SPECIAL INSTRUCTIONS:

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 15:30	Signature: Time:	Signature: Time:
Printed Name: PAUL WERNER Date: 1/22/91	Printed Name: Date:	Printed Name: Date:
Company:	Company:	Company:
RECEIVED BY: 1.	**RECEIVED BY: 2.**	**RECEIVED BY: (LAB) 3.**
Signature: DL Thomas Time: 4:40	Signature: Time:	Signature: Time:
Printed Name: DL Thomas Date: 1-23-91	Printed Name: Date:	Printed Name: Date:
Company:	Company:	Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 **DISTRIBUTION:** White, Canary - ATI • Pink - ORIGINATOR

Nº ,46

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560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

DATE 1/24/91 PAGE 1 OF 2

Chain of Custody

LABORATORY NUMBER: ~~9101-191~~ 9104-199

PROJECT MANAGER: Paul Werner
COMPANY: Geo Engineers
ADDRESS:
PHONE: 746-5200 SAMPLED BY: P. G. Werner

SAMPLE DISPOSAL INSTRUCTIONS
☒ ATI Disposal @ $5.00 each ☐ Return

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SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	~~TCLP 8000~~ Metals Screen *	TOX 9020	% Moisture	EP TOX Metals (8) ~~EP EXT~~ **	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)	TBT	Hardness	NUMBER OF CONTAINERS
Sample 2A @ 39'	1/24/90	1030	Water	1																		X							X	X	3
4 2B 0 - 0.3'	↓	1509	Sediment	2					X			X										X						X			1
see -13,14 2C 0.3 - 1.5' (Interstitial Water)		1500	↓	X																	2mi	X	—					2mi	X	—	1
2D 1.5 - 2.0'		1505		3															X												1
2E 2.0 - 2.5'		1515		4															X												1
2F 2.5 - 3.0'		1525		5															X												1
2G 3.0 - 3.5'		1535		6															X												1
2H 3.5 - 4.0'		1536		7															X												1
2I 4.0 - 4.5'		1538		8															X												1
2J 4.5 - 4.8'		1540		9															X												1
5 1A 0 - 0.3		900		10					X			X				X						X						X			1
	↓		↓	[illegible]					~~X~~			~~X~~				~~X~~		FCW		1/24/91		~~X~~						~~X~~			[illegible]

PROJECT INFORMATION	
PROJECT NUMBER:	1299-003-R04
PROJECT NAME:	Unimar Inc
PURCHASE ORDER NUMBER:	
ONGOING PROJECT?	YES ☒ NO ☐

SAMPLE RECEIPT	
TOTAL NUMBER OF CONTAINERS	14
COC SEALS/INTACT? Y/N/NA	Y
RECEIVED GOOD COND./COLD	Y
RECEIVED VIA:	Courier

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:
SEAL # 01299-05 FCW * Cd, Cu, Pb, Zn
SEAL # 01299-06 FCW ** EPTOX + Cu, Ni, Zn

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 10:00 1/24/91	Signature: Time:	Signature: Time:
Printed Name: Paul Werner Date: 1/24/91	Printed Name: Date:	Printed Name: Date:
Company: GEI	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2.	RECEIVED BY: (LAB) 3.
Signature: D L Thomas Time: 12:00	Signature: Time:	Signature: Time:
Printed Name: DL Thomas Date: 1-25-91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Analytical Technologies, Inc.

ATI s: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • nsacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGINA R

NO. 9149

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Chain of Custody

DATE: 1/24/91 PAGE 2 OF 2

LABORATORY NUMBER: ~~9101-191~~ 9104-19

PROJECT MANAGER: PAUL WERNER

COMPANY: GEO ENGINEERS

ADDRESS:

PHONE: 746-5200 SAMPLED BY: P.G. Werner

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

					ANALYSIS REQUEST																										
																							TCLP ONLY								
SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX ~~9020~~ Metals Scr	% Moisture	EP TOX Metals (8) EP EXT	Priority Pollutant Metals (13)	8080 Pesticide (4)	8240 ZH-EXT	8270	8150 Herbicides (2)	Metals (8)	METALS SCREEN	TBT	NUMBER OF CONTAINERS
*1B 0.3-1.0'	1/23/91	1652	Sediment	11																									X		
*1C 1.0-1.3'	1/23/91	1655	↓	12						X						X													X		
* 2C.			H2O	13																		X								X	
2C			Sed	14																		X								X	

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PROJECT INFORMATION	
PROJECT NUMBER:	1299-003-1304
PROJECT NAME:	Dana
PURCHASE ORDER NUMBER:	
ONGOING PROJECT?	YES ☐ NO ☐

SAMPLE RECEIPT	
TOTAL NUMBER OF CONTAINERS	
COC SEALS/INTACT? Y/N/NA	y
RECEIVED GOOD COND./COLD	y
RECEIVED VIA:	Courier

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☐ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK

GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:

HOLD Analysis *1B *1C Until Further Notice

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 1800	Signature: Time:	Signature: Time:
Printed Name: PAUL WERNER Date: 1/24/91	Printed Name: Date:	Printed Name: Date:
Company: GEI	Company:	Company:
RECEIVED BY: 1.	**RECEIVED BY: 2.**	**RECEIVED BY: (LAB) 3.**
Signature: DL Thomas Time: 12:02	Signature: Time:	Signature: Time:
Printed Name: DL THOMAS Date: 1-25-91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Analytical Technologies, Inc.

No. 0140

9104-144

AnalyticalTechnologies,Inc.
560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 1/25/91 PAGE 1 OF 1

LABORATORY NUMBER: ~~9101-203~~

PROJECT MANAGER: PAUL WERNER
COMPANY: GEO ENGINEERS
ADDRESS: Bellevue WA
PHONE: 746-5200 SAMPLED BY: P.G. WERNER

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 ~~Pesticides~~ & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	~~EP TOX Metals (8) EP EXT~~ * TCLP	Priority Pollutant Metals (13)	TCLP ONLY 8080 Pesticide (4)	TCLP ONLY 8240 ZH-EXT	TCLP ONLY 8270	TCLP ONLY 8150 Herbicides (2)	TCLP ONLY Metals (8)	METALS SCREEN **		NUMBER OF CONTAINERS
Bottom Sediment																															
1B 0.3'-1.0'	1/25/91	1400	SEDIMENT	-1																									X		1
1C 1.0'-1.5'		1405		-2							X					X													X		1
1D 1.5'-2.0'		1410		-3																									X		1
1E 2.0-2.5		1419		-4																									X		1
1F 2.5-3.0		1421		-5																									X		1
1G 3.0-3.5		1425		-6																									X		1
1H 3.5-4.0'		1430		-7																									X		1
		1555																													
3A 0-0.3'		~~1600~~		-8					X	~~X~~		X				X						X						X			1
3B 0.3-5.0		1600		-9						X	~~X~~					X						X									1

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PROJECT INFORMATION

PROJECT NUMBER: 1299-003-B04
PROJECT NAME: Unimar
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	9
COC SEALS/INTACT? Y/N/NA	Y
RECEIVED GOOD COND./COLD	Y/N0
RECEIVED VIA: Fedx	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:
SEAL # 01299-007 Rcw
* TEPMET + Cu, Ni, Zn
** Cd, Cu, Pb, Zn
Totals
not EPTOX
ICE Not frozen samples warm

RELINQUISHED BY: 1.	RELINQUISHED BY: 2.	RELINQUISHED BY: 3.
Signature: Paul Werner Time: 1700	Signature: Time:	Signature: Time:
Printed Name: PAUL WERNER Date: 1/25/91	Printed Name: Date:	Printed Name: Date:
Company: GEI	Company:	Company:

RECEIVED BY: 1.	RECEIVED BY: 2	RECEIVED BY: (LAB) 3.
Signature: DL Thomas Time: 1011	Signature: Time:	Signature: Time:
Printed Name: DL Thomas Date: 1-28-91	Printed Name: Date:	Printed Name: Date:
Company: ATI	Company:	Analytical Technologies, Inc.

ATI Labs: San Diego (619)458-9141 • Phoenix (602)438-1530 • Seattle (206)228-8335 • Pensacola (904)474-1001 DISTRIBUTION: White, Canary - ATI • Pink - ORIGIN

TCLP + Cu, Ni + Zn

NO

9104-147

AnalyticalTechnologies,Inc.

560 Naches Avenue SW, Suite 101 Renton,WA 98055 (206)228-8335

Chain of Custody

DATE 1/29/91 PAGE 1 OF 1

LABORATORY NUMBER: 9101-216

PROJECT MANAGER: Paul Werner
COMPANY: Geo Engineers
ADDRESS: Bellevue
PHONE: 746-5200
SAMPLED BY: P.G Werner

SAMPLE DISPOSAL INSTRUCTIONS

☒ ATI Disposal @ $5.00 each ☐ Return

ANALYSIS REQUEST

																								TCLP ONLY							
SAMPLE ID	DATE	TIME	MATRIX	LAB ID	8010 Halogenated Volatiles	8020 Aromatic Volatiles	BETX ONLY	8240 GCMS Volatiles	8270 GCMS BNA	8310 HPLC PNA	8080 Pesticides & PCB's	PCB's ONLY	8140 Phosphate Pesticides	8150 Herbicides	WDOE PAH/HH (WAC 173)	418.1 (TPH)	413.2 Grease & Oil	8015 (Modified)	TOC 9060	TOX 9020	% Moisture	EP TOX Metals (8) ~~EP EXT~~ *	Priority Pollutant Metals (13)	8080 Pesticide (4)	8240 ZH-EXT	8270	8150 Herbicides (2)	Metals (8)	TBT	Hardness	NUMBER OF CONTAINERS
Location # 12 Water	1/29/91	11.30	Water	-1																		X							X	X	3
Bottom Sediment			Soil																												
4A 0-0.3'		1500		-2					X			X				X						X						X			1
4B 0.3-4.2'		13:22		-3						X						X						X									1
~~5A~~ 5B 0.3-5.0		1625		-4						X						X						X									1
per Paul Werner on 1/31/91																															

PROJECT INFORMATION

PROJECT NUMBER: 1294-003-B41
PROJECT NAME: Unimar Inc
PURCHASE ORDER NUMBER:
ONGOING PROJECT? YES ☒ NO ☐

SAMPLE RECEIPT

TOTAL NUMBER OF CONTAINERS	6
COC SEALS/INTACT? Y/N/NA	y
RECEIVED GOOD COND./COLD	y
RECEIVED VIA: Courier	

PRIOR AUTHORIZATION IS REQUIRED FOR RUSH PROJECTS

TAT: (NORMAL) ☒ 2WKS (RUSH) ☐ 24HR ☐ 48 HRS ☐ 72 HRS ☐ 1 WK
GREATER THAN 24 HR. NOTICE? YES ☐ NO ☐ (LAB USE ONLY)

SPECIAL INSTRUCTIONS:
1294-008-B60
* TEPTOX + Cu, Ni, Zn TCP + Cu, Ni + Zn

RELINQUISHED BY: 1.	**RELINQUISHED BY: 2.**	**RELINQUISHED BY: 3.**
Signature: Paul Werner Time: 15:00	Signature: Time:	Signature: Time:
Printed Name: Paul Werner Date: 1/29/91	Printed Name: Date:	Printed Name: Date:
Company: GEI	Company:	Company:
RECEIVED BY: 1.	**RECEIVED BY: 2.**	**RECEIVED BY: (LAB) 3.**
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APPENDIX D

ANALYTICAL TEST METHOD

MEASUREMENT OF BUTYLTIN SPECIES IN SEDIMENTS

FINAL REPORT

Study Title

Measurement of Butyltin Species in Sediments
by n-Pentyl Derivatization with
Gas Chromatography/Flame Photometric Detection (GC/FPD)
and Optional Confirmation by
Gas Chromatography/Mass Spectrometry (GC/MS)

Data Requirement

Pesticide Assessment Guidelines,
Subdivision D: Product Chemistry

Submitted to

Consortium of Tributyltin Manufacturers
M&T Chemicals, Inc., Woodbridge, New Jersey
Sherex Chemicals Company, Inc., Dublin, Ohio

Authors

Allen D. Uhler and Gregory S. Durell

Study Completion Date

February 28, 1989

Performing Laboratory

Battelle Ocean Sciences
397 Washington Street
Duxbury, MA 02332

Laboratory Project Number

N-0519-6100

SIGNATURE PAGE

The data presented in this report is an accurate representation of the investigations conducted.

2-28-1989

Allen D. Uhler, Ph.D.
Study Director

Date

2-28-1989

Allen D. Uhler, Ph.D.
Lead Analytical Chemist

Date

2/28/89

Patricia D. Royal
Quality Assurance Manager

Date

Consortium of Tributyltin Manufacturers
Monitoring Scientist

Date

to Teflon jars, and the jars stored on ice. The samples were shipped on ice by overnight courier to Battelle Ocean Sciences, Duxbury, Massachusetts.

3.1.1 Test Sediment Characterization

The test sediments used in this study were characterized according to their grain size, and total organic carbon content (TOC). Select samples were analyzed for acid extractable, total tin content. Table 5 lists these physical and chemical parameters for the test sediments.

Grain size and TOC determinations were carried out following methods similar to those described by EPA (Plumb, 1981). Total organic carbon determinations were performed for Battelle by Global Geochemistry Company, Canoga Park, California. Extractable tin determinations were carried out using methods similar to those described by EPA procedure 7810 (EPA, 1986). Extractable tin determinations were carried out at Battelle's Sequim, Washington, laboratory.

The TOC content of the two Chesapeake Bay sediments bracket a wide range in TOC. Sediment CHB-1 has a low 0.33 percent TOC, whereas sediment CHB-2 has a relatively high 4.79 percent TOC content. The remaining sediments fall within the bracket defined by the CHB sediments.

The grain size distributions (gravel:sand:silt:clay) for the test sediments correlate reasonably well with the TOC content. The test sediments range from a high sand sediment (CHB-1) with low TOC to a high silt:clay sediment (CHB-2) with high TOC. In general, samples with higher percentages of silt tend to have higher levels of TOC.

Sediments CHB-1, CHB-2, HR, GB-1 and PS were analyzed for total extractable tin content. The freshwater sediments from Massachusetts were not analyzed for total extractable tin. The total tin analytical results for the selected sediments, reported on a wet weight basis, range from 601 μg/kg (CHB-1) to

3681 µg/kg (CHB-2). The extraction technique employed (aqua regia, 10 percent HCl/10 percent HNO_3) does not destroy the sediment matrix and tends to reflect the surficial or bound total tin concentration. The concentrations of total tin for these samples are typical tin values for sediments (Fairbridge, 1972). For the sediments characterized, the total butyltin concentration does not exceed 2 percent of the total extractable tin concentration.

3.1.2 Ambient Levels of Butyltins in Test Sediments

All test sediments were analyzed for ambient levels of butyltin species, either as part of Phase 1 (CHB-1 and CHB-2), or Phase 3 (HR, PS, GB-1, DF-1, TR). The method described in this study was used. The results of these finding are presented in Table 5. Total butyltin distribution in the test sediments is extremely varied. All butyltin species were identified in at least one or more sediment samples, except sediment DF-1, which was found to contain no detectable butyltin species at or above the 0.5 µg/kg level. The estuarine sediment collected from the Galveston Bay, Texas, area (GB-1) which had very low total butyltin concentration, and was selected for use in the method detection limit (MDL) determination (Section 3.4). The estuarine sediment collected from Hampton Roads, VA (HR) contained all butyltin species of interest, and was used in the storage stability experiments (Section 3.6).

3.2 CHEMICAL PURITY OF STANDARDS

The butyltin and propyltin compounds used for the preparation of standards in this study may be obtained commercially in high-purity form (≥ 95 percent). However, the organotin GC quantification standards are derivatized prior to use, as described in Section 2.2.3. In order to demonstrate that the organotin compounds retained their integrity, individual organotin compounds were carried through the GC standards preparation scheme (Section 2.2.3), and analyzed by GC/FPD and GC/MS. Figures 10 through 15 show the GC/FPD chromatograms of the individual organotin compounds after derivatization (except TTBT, which is used underivatized). Each compound was greater than 95 percent pure, by virtue of peak area measurements from the GC/FPD chromatograms. The GC/MS full scan spectra of each peak confirmed the structure of each individual organotin compound as the respective n-pentyl derivative (or the underivatized TTBT). Both the GC/FPD and GC/MS analysis demonstrated that the individual organotin compounds did not undergo rearrangement or degradation during preparation. Additionally, full scan GC/MS analysis showed that the individual organotin solutions contained no significant contaminants or interferants.

3.3 RECOVERY OF BUTYLTIN SPECIES FROM SEDIMENTS

The recovery of the butyltin species TBT, DBT, MBT, and TTBT from sediments, and the precision (reproducibility) of the method were evaluated as a function of analyte concentration and sediment characteristic. Table 2 summarizes the sediments and fortification levels used to evaluate method performance. The four sediments used to evaluate method performance (CHB-1, CHB-2, DF-1 and TR) were chosen because they were very different in their physical and chemical characteristics, and represented both estuarine and freshwater regimes. Sediment CHB-1 is a high sand:silt estuarine sediment with low TOC content (0.33 percent), whereas sediment CHB-2 is a high silt:clay estuarine sediment with a TOC content (4.79 percent) more than 10-fold higher than CHB-1. As an example, matrix effects, especially competitive binding, low recoveries, and interferences during GC/FPD analysis would be expected to be quite different for these two sediments.

The spiking levels used for the four test sediments in Phase 2 of this study were 2.5 μg/kg (DF-1), 25 μg/kg (DF-1), 50 μg/kg (CHB-1, CHB-2, TR) and 500 μg/kg (CHB-1, CHB-2, TR, DF-1). These levels are at least 5 times higher than the background levels determined in these sediments (Table 5). The freshwater sediment DF-1 contained no detectable ambient butyltin species, so it was spiked at the very low 2.5 μg/kg level. These fortification levels represent sediment concentrations that are within the range of environmental significance (Stephenson et al., 1987).

Method performance is evaluated in part by determining the recovery of fortified analytes and the precision, or reproducibility, of the method at different fortification levels. Recovery (percent) is defined using the formula

$$\text{Recovery (\%)} = \frac{X_d}{X_a} \times 100 \qquad (11)$$

where X_d is the average amount of analyte determined in a fortified sample, and X_a is the amount of analyte added to the sample. The amount of analyte in a sample is computed following the procedures described in Section 2.9. The concentrations determined are reported as absolute recoveries relative to the QIS DPT, and were not adjusted for recovery of the RIS TPT. The reproducibility, or precision, of the method is determined by evaluating the percent relative standard deviation (%RSD) of a series of replicate measurements. In this study, this factor is calculated as

$$\%\text{RSD} = \frac{\sigma}{X} \times 100 \qquad (12)$$

where σ is the standard deviation of replicate measurements at a given fortification level, and X is the average of the replicate determinations.

3.3.1 Recovery Results

The GC/FPD chromatograms for fortified sediment samples were generally interference-free in the region where the analytes eluted. Occasionally, an unidentified, coeluting compound partially obscured the RIS TPT peak, making integration and quantification of this peak difficult.

Figure 16 and 17 are GC/FPD chromatograms of the unfortified sediments CHB-1 and CHB-2. Figures 18 are 19 are chromatograms for sediments CHB-1 and CHB-2 fortified at the 50 µg/kg level. The chromatograms for the fortified sediments (Figures 18 and 19) appear to have less intense analyte signals than in the chromatograms for the ambient sediments (Figures 16 and 17). This is because the fortified samples were diluted by a factor of 10 prior to analysis in order to fall into the working range of the analytical standards. In all cases, the analyte peaks are clearly resolved, symmetrical, and readily interpretable.

Table 6 presents the recoveries of butyltin species from sediments CHB-1, CHB-2, DF-1 and TR at the 2.5 µg/Kg, 25 µg/Kg, 50 µg/kg and 500 µg/kg level. A spike level of 2.5 µg/kg was possible for freshwater sediment DF-1 because it came from a source completely free of ambient amounts of butyltin species.

Tributyltin recovery from sediment is good at all fortification levels. Recovery of TBT at the 25-50 µg/kg level averaged 125 percent for CHB-1, 86 percent for CHB-2, 107 percent for DF-1 and 104 percent for sediment TR. Recovery at the 500 µg/kg level averaged 110 percent for CHB-1, 87 percent for CHB-2, 67 percent for DF-1 and 83 percent for sediment TR.

Dibutyltin recovery from sediment is also good at both fortification levels, for both sediments. Dibutyltin recovery at the 25-50 µg/kg level averaged 127 percent for CHB-1, 70 percent for CHB-2, 85 percent for DF-1 and 93 percent for sediment TR. Recovery at the 500 µg/kg level averaged 127 percent for CHB-1, 85 percent for CHB-2, 60 percent for DF-1 and 70 percent for sediment TR.

Monobutyltin recovery from sediment was acceptable; recovery at the 25-50 µg/kg level was 64 percent for CHB-1, 34 percent for CHB-2, 100 percent for DF-1 and 98 percent for TR. Recovery at the 500 µg/kg fortification level averaged 62 percent for CHB-1, 50 percent for CHB-2, 75 percent for DF-1 and 63 percent for sediment TR. The recoveries of MBT at both fortification levels were significantly lower than DBT, TBT, or TTBT. MBT, a monoorganotin compound, is the most polar compound among the target analytes, and is presumably more strongly bound to sediment organic matter than the other butyltins, making the compound difficult to extract even with the use of the chelating agent tropolone. In general, recovery of MBT does appear to be somewhat better from the freshwater sediments tested. This may reflect differences in the binding strength of the organic matter associated with the estuarine sediments, relative to that of the freshwater sediments. However, since only a very small number of different sediments were used in this study, no definitive statements concerning recovery of butyltins from freshwater versus estuarine sediments can be made. Recovery of butyltin species from both sediment types are acceptable.

Tetrabutyltin recoveries were good at both fortification levels. Recovery of TTBT at the 25-50 µg/kg level was 97 percent for CHB-1, 77 percent for CHB-2, 97 percent for DF-1 and 74 percent for sediment TR. Recovery at the 500 µg/kg was 92 percent for CHB-1, 80 percent for CHB-2, 59 percent for DF-1 and 74 percent for sediment TR.

Recovery of the butyltin species at the 2.5 µg/kg level from the freshwater sediment DF-1 is presented in Table 6. Sediment DF-1 was the only test sediment with sufficiently low ambient butyltin levels to permit a series of very low spike and recovery experiments. The recovery results from these very low spike level experiments are very consistent with the results from the higher spike levels. TBT recovery averaged 119 percent, DBT recovery averaged 84 percent, MBT recovery averaged 106 percent and TTBT recovery averaged 92 percent. The very good recoveries of all butyltin species at this very low fortification level demonstrates that the method is dependable, even at these extremely low analyte concentrations.

For the estuarine sediments (CHB-1 and CHB-2) recoveries for all analytes at both fortification levels are somewhat lower (approximately 15 percent) from the CHB-2 sediment. This likely is due to the increased difficulty in extracting the compounds from the very high organic carbon content CHB-2 sediment. The organic matter associated with marine sediments is capable of binding organotins very strongly, with measured sediment-water distribution coefficients generally in the 10^3-10^4 range (Unger et al., 1987). Potentially, the higher the organic carbon content of a sediment, the stronger the binding of organotin compounds to the sediment surface. This hypothesis is consistent with the data presented in this study, because sediment CHB-2 has greater than 10-fold higher TOC than sediment CHB-1, and would be predicted to bind the organotins more strongly than CHB-1.

3.3.2 Reproducibility of Recovery

The reproducibility of recovery of the butyltin analytes from sediments is measured as the percent relative deviation (%RSD) of replicate measurements at a given fortification level. The %RSD for all measurements is presented in Table 6 as the value adjacent to the corresponding percent recovery value.

Reproducibility of measurements for the butyltin species at both fortification levels for all test sediments is excellent. At the 50 µg/kg level, the %RSD averaged 8 percent for all analytes in the CHB-1 sediment, 16 percent for all analytes in the CHB-2 sediment, 12 percent for all analytes in the DF-1 sediment, and 13 percent for all analytes in sediment TR. At the 500 µg/kg level, the %RSD averaged 3 percent for all analytes in the CHB-1 sediment, 4 percent for all analytes in the CHB-2 sediment, 3 percent for all analytes in the DF-1 sediment and 18 percent in sediment TR.

The recovery of butyltin species from the freshwater sediment TR at the 2.5 µg/kg level averaged 18 percent for all analytes.

The variability in the recovery of ambient butyltins for all the test sediments was in the 2.5 to 55 %RSD range for TBT, 3.4 to 54 %RSD range for DBT, 7.3 to 67 %RSD range for MBT and 1 to 93 %RSD range for TTBT. The observations presented in this study suggest that the analytical method is very reproducible, but that, in most cases, ambient levels of butyltins are not homogeneously distributed. This is consistent with a review of laboratory round-robin data for TBT findings in TBT-contaminated sediments (Stephenson et al., 1987). In that study, the average %RSD for the measurement of ambient TBT in a composited sediment sample at the 500 μg/kg level was 31 percent.

3.3.3 GC/MS Confirmation

The presence of incurred butyltin residues in test sediments (based on GC/FPD results) was confirmed by GC/MS operating in SIM mode, following instrumental operating procedures described in Section 2.4.2, and selected ion interpretation guidelines described in Section 2.6. Figure 20 is a representative GC/SIM extracted ion chromatogram of an extract of sediment collected from Puget Sound, Washington (PS), clearly showing the presence of TBT, DBT, MBT, and TTBT.

3.4 METHOD DETECTION LIMIT (MDL)

The Method Detection Limit (MDL) for the analysis of butyltins in sediments was determined according to EPA guidelines presented in the Federal Register (1984). The estuarine test sediment with the lowest ambient butyltin concentration, GB-1, from Galveston Bay, Texas, was used for this experiment. The MDL was determined by performing eight replicate analyses of a sediment fortified at approximately the 5 μg/kg level. This concentration was about five times higher than the ambient level of TBT in the test sediment. The MDL was calculated from the following equation:

$$\mathrm{MDL} = t \times \sigma \qquad (13)$$

where σ is the standard deviation of the eight replicate measurements, and t is the Student-t statistic at the 99 percent confidence level for eight measurements and seven degrees of freedom.

The upper confidence limit (UCL) and lower confidence limit (LCL) are calculated for use with the MDL. The UCL and LCL represent the upper and lower 95 percent confidence limits of the MDL. The UCL and LCL are calculated from the equations:

$$LCL = 0.66\ MDL \tag{14}$$
$$UCL = 2.04\ MDL \tag{15}$$

Table 7 is a summary of the MDL, LCL, and UCL for TBT, DBT, MBT, and TTBT, based on GC/FPD determinations.

In practice, analytes determined at and above the MDL should be reported in concentration units. Analytes detected below this level should be reported as "detected but below the MDL," with no concentration value reported.

3.5 ANALYSIS OF FIELD SAMPLES

The method for the analysis of butyltin species in sediments was used to evaluate the levels of butyltins in sediments from several estuarine regions in the United States. The objective of this work (Phase 3, Table 2) was to determine if randomly collected sediments posed any significant difficulties when using the analytical method described in this report. Three estuarine sediments were used for this phase of the study. A sediment sample, designated HR, was collected from the Hampton Roads area of Virginia, near the mouth of the Chesapeake Bay. The sampling site is in a heavily industrial area, with significant commercial, U.S. Naval, and recreational boat traffic. A sample, designated GB-1, was collected from the heavily plied Galveston Bay ship channel in Texas. This sampling site is located in the major ship channel just off Galveston Island. Finally, an estuarine sediment from Puget Sound, Washington, designated PS, was collected for testing. This sediment was collected off a pier in an active, recreational marina.

The results of the analysis of butyltins in the sediments are given in Table 5. The PS sediment, collected directly in a marina, has almost 10-fold higher levels of TBT (26.8 μg/kg) than do any of the other test sediments. The sediment from Hampton Roads (HR) had the next highest concentration of TBT (8.63 μg/kg), whereas the sediment from the very well flushed Galveston Bay site (GB-1) had the lowest level of TBT (1.15 μg/kg). The variability in the duplicate measurements ranged from 2.5 to 55 %RSD for TBT, 12 to 54 %RSD for DBT, 15 to 60 %RSD for MBT and 49 to 93 %RSD for TTBT. This degree of variability is consistent with literature findings, as discussed in Section 3.3.2.

None of the test sediments used in Phase 3 of this work presented any special problems during the sample workup or instrumental analysis. Especially encouraging was the fact that the PS sediment analysis was not plagued by any overt interferences. This sediment, obtained from a heavily used marina area where anthropogenic contaminants (i.e., fuel oil, grease, etc.) would be present (and with a rather high 2.43 percent TOC loading), would be predicted to present the most challenge to the analytical method.

The test sediments used in Phase 3 of this study were also analyzed by the method of standard additions (Skoog and West, 1976). The standard additions technique can provide insight into potential matrix interferences not accounted for by the conventional, or "single point," method. Aliquots of test sediment were fortified at levels approximately 100 percent, 200 percent, and 400 percent above ambient levels of the analyte targeted for standard additions analysis. Briefly, a plot of the ratio of analyte GC/FPD signal to internal standard GC/FPD signal versus amount of analyte added to the ambient sample was prepared. Extrapolation of the linear least-square fit to the data to the abscissa yielded the amount of analyte in the unfortified sample.

Because the TBT and DBT levels among sediments varied significantly (Table 5), only one analyte per sediment was selected for analysis by the method of standard additions. The sediments HR and GB-1 were selected for TBT analysis by standard additions; the sediment PS was selected for DBT analysis by standard additions.

The results of the standard additions technique with the ambient levels determined from single point analyses compare very favorably for the HR and GB sediments. TBT residues for the HR sediment by standard additions was found to be 7.77 µg/kg, which compares excellently with the 8.63 ± 2.8 µg/kg determined using the single point analytical method. Similarly, the TBT level in sediment GB-1 was found to 2.47 µg/kg by standard additions, compared to 1.15 ± 0.63 µg/kg by the single point method. Considering the variability in the ambient levels of TBT in the sediments (on the order of 30 to 60 percent), the agreement between the two methods is very good.

The comparison of DBT levels determined by standard additions and the single point method for sediment PS does not compare as favorably as the TBT determinations described above. The DBT concentration in sediment PS determined by the method of standard additions is 10.3 µg/kg, versus 3.84 ± 0.45 µg/kg determined by the single point technique. This discrepancy (a factor of 2.7) is probably due to the rather large scatter in the standard addition data, which in turn reflects the rather high variability in the ambient levels of DBT in the PS test sediment.

Overall, the results from the Phase 3 experiments reveal that the analytical method performs well for a wide range of sediment conditions, including those from heavily contaminated sites such as marinas and active shipping lanes.

3.6 STORAGE EXPERIMENTS

Phase 4 of the method validation strategy involved studying the effects of storage on the stability of butyltin species in sediments. Sediment samples are often collected in the field, returned to the laboratory, and stored for some period of time prior to extraction and analysis. In order to test whether the butyltin species TBT, DBT, MBT, and TTBT were stable over reasonable holding periods, two separate storage experiments were conducted. The test sediment designated HR was chosen for use in this Phase of the Study because it contained environmentally realistic, ambient levels of all four butyltin species.

The first experiment involved evaluating the stability of butyltin species in bulk sediment, by storing sediments with ambient levels of butyltin species in the dark, in a freezer maintained at -20°C, while monitoring the stability of the butyltin species by periodically analyzing the sediments. Ten separate Teflon jars were filled with 100 g of sediment HR. Initially (time = 0), and each week for one month, two sample jars were removed from the freezer, and the sediments analyzed for butyltin species following the procedures presented in Section 2.3.

The second experiment involved monitoring the stability of sediment-extracted, derivatized butyltin species stored in the dark, in a freezer maintained at -20°C. Ten 100-g sediment samples (HR) were extracted and derivatized as described in Section 2.3. The extracts were stored in the dark in at -20°C. Initially (time = 0) and each week for one month, two extracts were removed from the freezer and analyzed for butyltin species.

Figures 21 through 24 present stability plots of TBT, DBT, MBT and TTBT for the bulk sediment storage experiment. Figures 25 through 28 present stability plots of TBT, DBT, MBT, and TTBT for the sediment extract experiment. The data are presented as the measured concentration of the individual butyltin compound on a wet weight basis versus holding time. In both experiments, TBT, DBT, MBT and TTBT concentrations appear to remain stable over the course of the 4-week experiment. In general, the reproducibility of the duplicate measurements for TBT, DBT, and TTBT is in the 10-20 percent range. The variability in MBT measurements is somewhat higher--approximately 20-30 percent. This is a reflection of the very low MBT concentration (approximately 0.6 µg/kg) in the test sediment, which is approaching the MDL for this compound. Higher variability in the measurements is expected at this very low concentration.

There appears to be no advantage (in terms of butyltin stability) in storing sediment extracts instead of the bulk sediments. The results of this experiment demonstrate that sediments or sediment extracts may be stored over at least a 4 week period in a -20°C freezer in the dark without any compromise to analyte integrity.

3.7 INTERFERENCES

Interferences in the trace-level determination of substances can originate from numerous sources. Interferences (and contamination) can arise from the matrix in which the sample is found, the quality of reagents used in processing samples, the cleanness of labware and the care with which samples are handled by laboratory personnel. These issues are discussed below.

3.7.1 Sample Matrices

Sediments from both marine and freshwater environments potentially contain interfering materials. The most common naturally occurring interferants in sediments are the coextracted organic material associated with the sediment, and dissolved and colloidal sulfur (including elemental, polysulfidic, and sulfidic species). Additionally, sediment samples collected from near-coastal waters and from busy harbors may contain high levels of oils and/or other anthropogenic chemicals.

The only other class of organotin compounds reported to have been detected in the aquatic environment, the methyltins, have been shown to have different retention times from the butyltins, and do not pose an interference problem by coeluting with analytes of interest (Unger, 1986). When the method used in this study, inorganic tin is recovered with undetermined efficiency from water, and arises in extracts because it is naturally present in sediments. Inorganic tin is converted to tetrapentyltin during sample processing, and elutes as a peak approximately 1 min after the last analyte peak (MBT) elutes. The presence of inorganic tin does not constitute an interference.

3.7.1.1 Naturally Occurring Organic Matter

Naturally occurring organic matter coextracted with sediments used in this study did not pose a significant problem with the GC analysis of the butyltin analytes. Occasionally, a spurious coeluting peak very near to the RIS TPT was observed. This coeluting compound often obscured or made difficult the proper integration of the TPT. However, in no instance were any interferences noted in the region of the chromatogram where the butyltin analytes or the QIS DPT eluted.

3.7.1.2 Contamination of Sediments by Crude Oil

The effect of very high levels of crude oil on the analysis of butyltins in sediments was evaluated by spiking sediments with 10 mg/g Prudhoe Bay crude oil and with butyltin analytes at approximately the 5 µg/kg level prior to extraction and analysis. These samples were prepared in order to represent grossly contaminated harbor sediments. Two analytical strategies were evaluated and contrasted: (1) the crude-oil sediments were processed by the standard method (presented in Section 2.3) and analyzed, and (2) identical samples were processed by the standard method and then subjected to the auxiliary gel permeation chromatographic (GPC) cleanup process.

The results of these experiments showed that the standard Florisil/silica gel column removed the bulk of the oil added to the sediments (as determined by presence or lack of interferants in the GC/FPD chromatograms). The GC/FPD chromatograms of the sediment extracts processed through the standard method showed no obvious interferences with the analytes of interest. All analytes did exhibit a slight increase in their GC retention times and some moderate peak broadening, likely the result of a "keeper" effect of residual oil in the GC injection port. The percent difference between the determined amounts of butyltin analytes in uncontaminated sediments and oil-contaminated were MBT (-19%); DBT (-11%); TBT (+24%); TTBT (-10%). The differences between the determined amounts probably reflect the effect of peak broadening on accurate quantitation. However, considering that the oil contaminated sediments are an extreme worst-case example, these differences are acceptable.

Processing of the crude-oil contaminated sediments through GPC did not show a measurable difference in chromatographic quality relative to the standard Florisil/silica cleanup. This result suggests that even sediments with high hydrocarbon loadings will be sufficiently cleaned up using the standard sample processing technique. Additionally, the alternate GPC method is available to augment the standard Florisil/silica cleanup in the event of unusual contamination not removed by Florisil/silica.

3.7.1.3 Sulfur Contamination

Sulfur is often found in sediment samples, in either the elemental or reduced state. The FPD, although equipped with a selective 610-nm filter, can be affected by sulfur emissions if significant amounts of sulfur are present in a sample extract. The effect of gross amounts of sulfur on the FPD signal is probably related to changes in the emission flame temperature and to a lesser extent, direct emission due to sulfur. In certain cases, it may be necessary to chemically remove sulfur from extracts prior to successful analysis.

Sulfur interference is indicated by the presence of several large, unsymmetrical peaks, resulting from the degradation of elemental sulfur (S_8) in the injection port of the GC. Although several of the sediment samples used in this study contained detectable amounts of sulfur species (as determined by the distinct odor of sulfide during sample processing), no interference due to these ambient levels of sulfur was observed.

Experiments were carried out to determine if gross levels of sulfur could be removed effectively from sample extracts. As part of Phase 1 (Reconnaissance Experiments), sediment CHB-1 was intentionally contaminated with 10 mg/g elemental sulfur and fortified with 5 µg/kg butyltin species. These samples, carried through the analytical procedure, showed severe sulfur interference during GC/FPD analysis.

In this study, the n-pentyl derivatives of TBT, DBT, MBT, and the species TTBT were found to be stable in the presence of activated copper. Repeated treatment of the grossly sulfur contaminated extracts with activated elemental copper (EPA, 1986) removed much sulfur contamination, but did not remove enough of the sulfur to reduce the background interference for sufficiently successful GC/FPD analysis.

Natural levels of sulfur encountered in this study have not posed a problem in the analysis of butyltins in sediments. Low levels of sulfur react with the Grignard reagent and are probably trapped during sample cleanup. Moderate levels of sulfur may be removed by treatment of extracts with activated copper. However, extreme levels of sulfur in sediment specimens may result in samples that are very difficult to analyze by GC/FPD.

3.7.2 Presence of Other Pesticides in Sample Extracts

The presence of other pesticides in sediments, and their potential interference in the analysis of butyltins using this method, has not been directly investigated. However, due to the very selective nature of the determinative step (GC/FPD), interference due to the presence of low levels of pesticides is not expected. Although FPD is used as a detection technique for both phosphorus- and sulfur-containing pesticides, these classes of compounds do not pose an interference problem in the GC/FPD determination of organotin compounds. Sulfur and phosphorus compounds have unique flame emission characteristics that are centered below 550 nm in the electromagnetic spectrum. The optical filter used in the determination of butyltins allows only radiation in the window 610±20 nm to reach the detector. Hence, at the very low levels pesticide compounds may exist in sediments, the method is transparent to phosphorus-, sulfur-, nitrogen-, and halogen-containing compounds.

3.7.3 Solvents and Chemicals

The solvents used in this procedure were all of pesticide grade or better. No interferences or organotin contamination were experienced. The acid HBr used in the processing of samples often contains small, parts per million, (ppm) amounts of inorganic tin. However, the presence of this elemental form of tin does not constitute an interference.

All other reagents and chemicals were tested separately and found to be free of butyltin contamination. It is recommended that prior to analysis of samples in the laboratory, all solvents, reagents, and other chemicals be carried through the analytical procedure in an isolated fashion to test for potential butyltin contamination.

3.7.4 Labware

Laboratory glassware must be handled with extreme care prior to use in the analysis of butyltin compounds. Certain glassware may be treated with monobutyltin during manufacturing, which can result in an extremely large interference in the trace analysis of butyltin compounds. It is recommended that glassware that is to be used for butyltin analysis and especially glassware that is to be used for the collection and storage of water samples be subjected to the following cleaning procedure:

1. Soak glassware in glacial acetic acid for 4 h.
2. Rinse glassware three times with distilled water.
3. Soak glassware in concentrated HCl for 4 h.
4. Rinse glassware three times with distilled water.
5. Air dry.

The use of synthetic materials constructed of or containing PVC plastic should be avoided in any procedures related to trace organotin analysis. PVC is a potential sources of dibutyltin contamination because dibutyltin is used in the manufacturing of the polymer. Potentially serious contamination due to the presence of DBT can occur due to contact with these synthetic products during the processing of samples. It is also recommended that synthetic rubber and plastic materials not be used in the processing of samples. If it is necessary to wear gloves during glassware cleaning, those constructed of either Viton (preferred) or nitrile should be used.

4.0 ARCHIVING OF DATA

All raw data, the final report and protocol, and samples of the neat test materials used will be indefinitely archived at the Battelle Archive in Duxbury, Massachusetts.

5.0 CONCLUSIONS

The method presented in this report is a multiresidue technique for the analysis of TBT, its degradation products DBT and MBT, and the TBT manufacturing impurity TTBT in estuarine sediments. The tests described in this report demonstrate that the method provides good recoveries for TBT, DBT, MBT, and TTBT, with MDLs for the analytes less than 0.5 μg/kg on a wet weight basis. Recovery of the analytes at the 50 µg/kg level ranged from 70 to 127 percent for TBT, DBT, and TTBT in the matrices tested. Mean MBT recovery at the 50 µg/kg level was 74 percent. This lower recovery of MBT reflects the polarity and relatively affinity the trivalent cation MBT has for organic matter associated with sediments. Recovery of MBT will likely range from 30 to 100 percent, depending on the organic carbon loading of the sediments under study. No significant interferences were observed in the test sediments studied. Laboratory observations suggest that low, ambient levels of sulfur likely are removed during sample processing. Because the butyltins were found to be stable in the presence of activated copper, this material may be used to further remove sulfur should it prove to be a significant interferant.

Laboratory storage experiments using a sediment with ambient levels of butyltin species demonstrate that TBT, DBT, MBT and TTBT do not degrade when stored as bulk sediment or sediment extracts at -20°C in a darkened freezer. The storage experiments were conducted over a 4-week period. The results of this storage experiment show that prior to analysis field-collected sediment samples may be confidently stored for at least 4 weeks without any loss of analyte integrity.

The equipment used in this method is common to most analytical laboratories and is all commercially available. No specialized glassware or equipment is required to carry out the method. The only nonroutine hardware requirement is for the GC flame photometric detector. The FPD must be fitted with an optical filter that is commercially available and costs less than $100 (1988 price). The techniques (extraction, cleanup) are similar to other sediment extraction procedures. The derivatization step (prior to cleanup) is rapid and simple. Overall, the method should not present a serious challenge to the experienced analyst.

6.0 REFERENCES

Blunden, S.J. and A.H. Champman. 1982. A review of the degradation of organotin compounds in the environment and their determination in water. Int. Tin. Res. Inst. Publ. 62: 2-7.

Champ, M.A. and W.L. Pugh. 1987. Tributyltin antifouling paints: Introduction and overview. Oceans '87 Proceedings, Vol. 4: 1296-1308.

EPA (U.S. Environmental Protection Agency). 1982. Pesticide Assessment Guideline: Subdivision D, Product Chemistry. C.J. Beush (ed.). U.S. Environmental Protection Agency Office of Pesticides and Toxic Substances. U.S. EPA, Washington DC. EPA 540/9-82-018.

EPA (U.S. Environmental Protection Agency). 1986. Test methods for evaluating solid wastes. Office of Solid Waste and Emergency Response, Washington, DC.

Fairbridge, R.W. (ed). 1972. The Encyclopedia of Geochemistry and Environmental Chemistry. Van Nostrand Reinhold Co. New York, NY.

Federal Register. 1984. Definition and procedure for the determination of the Method Detection Limit. Vol 49 No. 209: 198-199.

Kaplia, S. and C.R. Vogt. 1980. Some aspects of organic tin analysis by gas chromatography with flame photometry. J. Chromatogr. Sci. 18: 144-147.

Meinema, H.A., T. Burger-Wiersma, G.V. Haan and E. Gevers. 1978. Determination of trace amounts of butyltin compounds in aqueous systems by gas chromatography/mass spectrometry. Env. Sci. Technol. 12: 288-293.

Plumb, R.H. 1981. Procedures for handling and chemical analysis of sediment and water samples. Technical Report EPA/CE-81-1, prepared by Great Lakes Laboratory, State University College at Buffalo, Buffalo, NY, for the U.S. Environmental Protection Agency/Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material. Published by the U.S. Army ENgineer Waterways Experiment Station, CE, Vicksburg, MI.

Skoog, D.A. and D.M. West. 1976. Fundamentals of Analytical Chemistry. Third Ed. Holt, Rinehart and Winston, New York, New York.

Stepenson, M.D., D.R. Smith, L.W. Hall, W.E. Johnson, P. Michel, J. Short, M. Waldock, R.J. Huggett, P. Seligman, and S. Kola. 1987. An international comparison of butyltin determinations in mussel tissue and sediments. Oceans '87 Proceedings, Vol. 4: 1334-1338.

Thain, J., M.J. Waldock, and M.E. Wait. 1987a. Toxicity and degradation of tributyltin and dibutyltin in the aquatic environment. Oceans '87 Proceedings, Vol. 4: 1398-1404.

Thain, J., M.J. Waldock, and M.E. Wait. 1987b. Changes in concentration of organotins in UK rivers and estuaries following legislation in 1986. Oceans '87 Proceedings, Vol. 4: 1352-1356.

Uhler, A.D. and W.G. Steinhauer. 1988. Measurement of butyltin species in water by n-pentyl derivitization with gas chromatography/flame photometric detection (GC/FPD) and gas chromatography/mass spectrometry (GC/MS). Final Report submitted to the Consortium of Tributyltin Manufacturers.

Unger, M.A., W.G. MacIntyre, J. Greaves, and R.J. Huggett. 1986. Determination of butyltins in natural waters by flame photometric detection of hexyl derivatives with mass spectrometric detection. Chemosphere 15: 461-470.

Unger, M.A., W.G. MacIntyre, and R.J. Huggett. 1987. Equilibrium Sorption of tributyltin chloride by Chesapeake Bay sediments. Oceans '87 Proceedings, Vol. 4: 1381-1385.

7.0 TABLES/FIGURES

The following section contains pertinent tables and figures referenced in the body of this report.

TABLE 1. BUTYLTIN COMPOUNDS REGISTERED FOR USE IN ANTIFOULANT PAINT FORMULATIONS

Compound Name	Chemical Abstract Services Registry Number
Bis(tributyltin) oxide	56-35-9
Bis(tributyltin) dodecenyl succinate	12379-54-3
Bis(tributyltin)sulfide	4804-30-4
Bis(tributyltin)acetate	56-36-0
Bis(tributyltin) adipate	7437-35-6
Tributyltin acrylate	1331-52-7
Tributyltin fluoride	1983-10-4
Tributyltin resinate	none assigned
Tributyltin methacrylate and copolymer	2155-70-6 26345-187

TABLE 2. TASKS PERFORMED FOR METHOD VALIDATION STUDY[a]

PHASE 1. Reconnaissance Experiments Effects of Sulfur and Crude Oil on Method Performance

Fortification Level (µg/kg)	Replicates CHB-1	CHB-1+PBC[b]	CHB-1+S[c]
5	4	4	4

PHASE 2. Precision, Accuracy, and Method Detection Limit (MDL) Determinations

Fortification Level (µg/kg)	Replicates				
	CHB-1	CHB-2	GB-1	DF-1	TR
Ambient	4	4	4	2	2
2.5	-	-	-	4	-
25	-	-	-	4	-
50	4	4	-	-	4
500	4	4	-	4	4
MDL Determination	-	-	8	-	-

PHASE 3. Field Sample Verification

Method	Replicates		
	HR	GB-1	PS
Conventional	2	2	2
Standard Additions[d]	6	6	6

PHASE 4. Storage Experiments

Sample Type	Number of Analyses
Sediment[e]	5-week x 2 duplicates
Extracts[f]	5-week x 2 duplicates

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[a] Sediment codes identified in the text, Section 3.1.
[b] Sample CHB-1 + 10 mg/g Prudhoe Bay Crude Oil.
[c] Sample CHB-1 + 10 mg/g elemental sulfur.
[d] Method of standard additions. 3-Level--duplicates at each level.
[e] 100 g sediment HR stored at -20°C.
[f] Extracts of sediment HR stored at -20°C.

TABLE 3. GAS CHROMATOGRAPHY RETENTION TIMES AND RELATIVE RETENTION TIMES OF BUTYLTIN COMPOUNDS

Compound[a]	Retention Time[b] (minutes)	Relative Retention Time[c] (minutes)
Dipropyltin (DPT)	9.61	1.00
Tripropyltin (TPT)	8.55	0.89
Tetrabutyltin (TTBT)	9.46	0.98
Tributyltin (TBT)	9.98	1.04
Dibutyltin (DBT)	10.48	1.09
Monobutyltin (MBT)	10.97	1.14

[a] Gas chromatography is performed on the n-pentyl derivatives of MBT, DBT, TBT, DPT and TPT. TTBT is not derivatized.

[b] Retention times relative to GC conditions given in text.

[c] Values calculated relative to the retention time of the quantitation internal standard DPT.

TABLE 4. SELECTED IONS AND EXPECTED RELATIVE ION ABUNDANCES USED IN MASS SPECTROMETRY CONFIRMATION OF BUTYLTIN SPECIES

Compound: Tetrabutyltin

Mass Fragments Monitored (amu):	119	121	235	291
Expected Relative Ratios:	36	47	100	66
± 20% Tolerance :	7.2	9.4	20	13.2

Compound: Tributyltin (n-pentyl derivative)

Mass Fragments Monitored (amu):	119	121	249	305
Expected Relative Ratios:	74	96	87	100
± 20% Tolerance :	15	19	17	20

Compound: Dibutyltin (n-pentyl derivative)

Mass Fragments Monitored (amu):	119	121	249	319
Expected Relative Ratios:	59	76	100	57
± 20% Tolerance :	12	15	20	11

Compound: Monobutyltin (n-pentyl derivative)

Mass Fragments Monitored (amu):	119	121	193	319
Expected Relative Ratios:	35	47	100	47
± 20% Tolerance :	7	9.4	20	9.4

TABLE 5. CHEMICAL AND PHYSICAL PROPERTIES OF TEST SEDIMENTS

						µg/kg[a]				
Sediment	%Gravel	%Sand	%Silt	%Clay	TOC (%)	ΣSn	TBT	DBT	MBT	TTBT
CHB-1	0.01	94.34	1.60	4.05	0.33	601 (113)	2.98 (0.91)	1.55 (0.43)	1.42 (0.3)	D
CHB-2	3.23	13.38	58.92	24.48	4.79	3681 (170)	4.88 (1.3)	0.30 (0.14)	0.48 (0.32)	D
HR	0.33	66.61	14.01	19.05	0.91	2045 (49)	8.63 (2.8)	3.65 (1.8)	1.15 (0.52)	3.87 (1.9)
GB-1	0.41	54.44	18.62	26.54	0.40	989 (223)	1.15 (0.63)	0.48 (0.26)	0.42 (0.25)	ND
PS	0.86	14.67	68.71	15.76	2.43	1831 (49)	26.83 (0.66)	3.84 (0.45)	0.68 (0.10)	7.55 (7.0)
DF-1	0.69	80.27	14.28	4.76	1.88	b	ND	ND	ND	ND
TR	0.63	55.18	38.36	5.83	2.24	b	11.04 (0.76)	9.39 (0.32)	17.50 (1.27)	1.11 (0.01)

[a] Chemical analyses reported as the mean of replicate determinations. Standard deviation of the replicate determinations presented as parenthetical values.

[b] Total tin determinations not performed.

D detected, but below the Method Detection Limit (MDL).

ND Not detected in sample.

TABLE 6. RECOVERY OF BUTYLTIN SPECIES FROM ESTUARINE SEDIMENTS DETERMINED BY GAS CHROMATOGRAPHY WITH FLAME PHOTOMETRIC DETECTION[a]

Spike Level[b] -- 2.5 μg/kg

Sediment	TBT %Recovery	TBT %RSD	DBT %Recovery	DBT %RSD	MBT %Recovery	MBT %RSD	TTBT %Recovery	TTBT %RSD
DF-1	119	14	84	19	106	15	92	22

Spike Level[b] -- 50 μg/kg

Sediment	TBT %Recovery	TBT %RSD	DBT %Recovery	DBT %RSD	MBT %Recovery	MBT %RSD	TTBT %Recovery	TTBT %RSD
CHB-1	125	14	127	5	64	6	97	6
CHB-2	86	12	70	17	34	26	77	8
DF-1[c] Fw lake	107	5	85	19	100	17	97	8
TR River	104	12	93	9	98	18	74	11

Spike Level[b] -- 500 μg/kg

Sediment	TBT %Recovery	TBT %RSD	DBT %Recovery	DBT %RSD	MBT %Recovery	MBT %RSD	TTBT %Recovery	TTBT %RSD
CHB-1	110	3	127	2	62	4	92	4
CHB-2	87	2	85	2	50	8	80	3
DF-1	67	7	60	1	75	1	59	3
TR	83	25	70	7	63	19	74	20

---

[a] Recovery is reported as the average of four replicate determinations. Recovery is calculated from the formula Xd/Xa, where Xd is the amount of analyte determined in the sample, and Xa is the amount of analyte added to the sample. %RSD is the relative standard deviation of the replicate determinations, defined as $\sigma/X * 100$, where X is the mean of the determinations and σ is the standard deviation of the mean.

[b] Spike level is expressed in μg/kg wet weight.

[c] Spike level is 25 μg/kg.

TABLE 7. METHOD DETECTION LIMIT (MDL) FOR BUTYLTIN SPECIES IN SEDIMENT WHEN USING GAS CHROMATOGRAPHY WITH FLAME PHOTOMETRIC DETECTION

	µg/kg			
	TBT	DBT	MBT	TTBT
σ[a]	0.084	0.190	0.096	0.042
MDL[b]	0.252	0.570	0.288	0.126
LCL[c]	0.166	0.376	0.190	0.083
UCL[d]	0.512	1.163	0.588	0.255

---

[a] Standard deviation of the mean of eight determinations, in µg/kg. Concentrations expressed on a wet weight basis.

[b] Method detection limit, in µg/kg wet weight. Defined as t$\times\sigma$, where t is the Student's-t statistic at the 99 percent confidence limit for the number of measurements at n-1 degrees of freedom, and σ is the standard deviation of the mean for the determinations. For 8 measurements, t = 2.998.

[c] LCL Lower confidence limit, in µg/kg wet weight. Lower 95 percent confidence limit, defined as 0.66 x MDL.

[d] UCL Upper confidence limit, in µg/kg wet weight. Upper 95 percent confidence limit, defined as 2.04 x MDL.

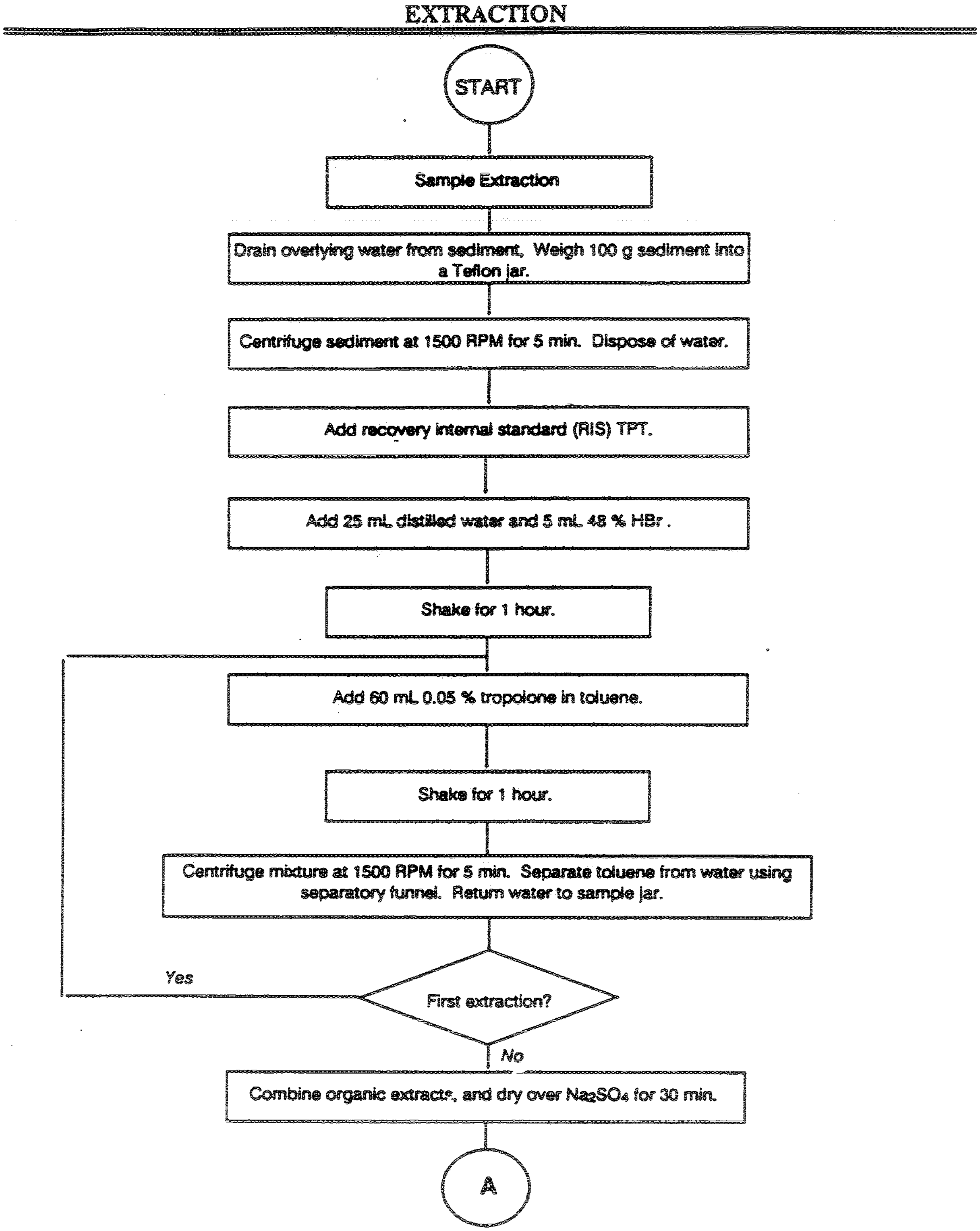

FIGURE 1. FLOWCHART FOR THE ANALYSIS OF BUTYLTIN SPECIES IN SEDIMENTS.

DERIVATIZATION AND CLEANUP

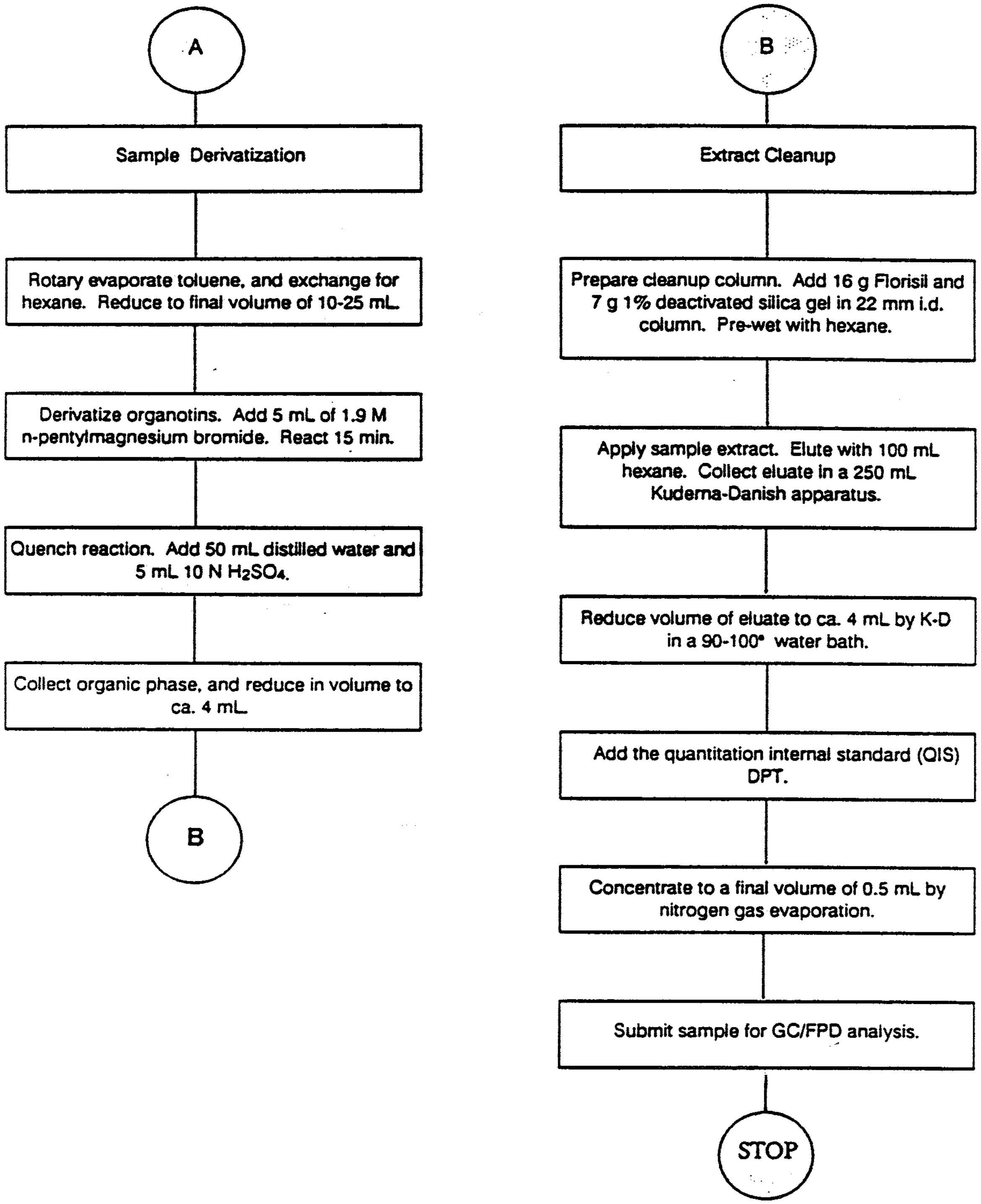

FIGURE 1 (cont). FLOWCHART FOR THE ANALYSIS OF BUTYLTIN SPECIES IN SEDIMENTS.

GC/FPD ANALYSIS

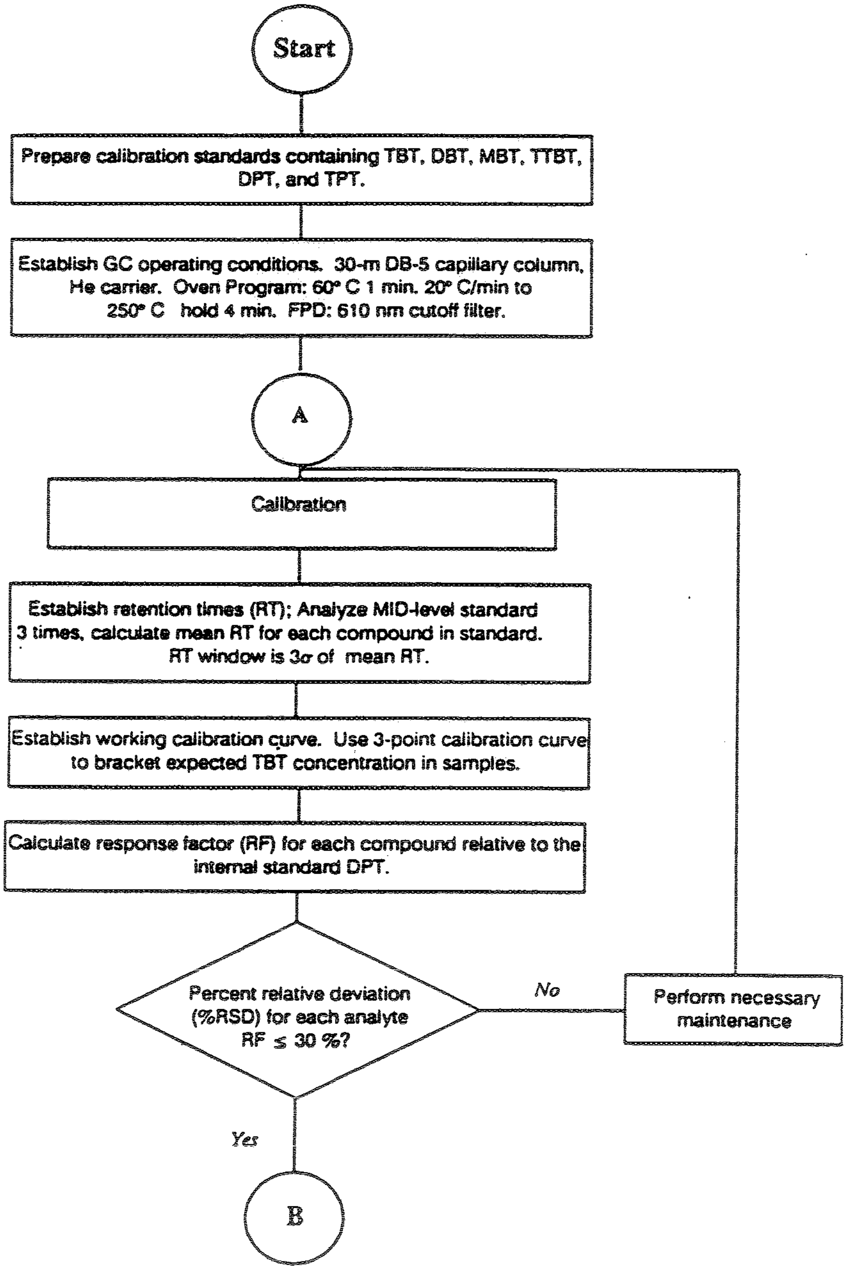

FIGURE 2. FLOWCHART FOR THE GAS CHROMATOGRAPHIC ANALYSIS OF BUTYLTIN SPECIES IN SEDIMENTS.

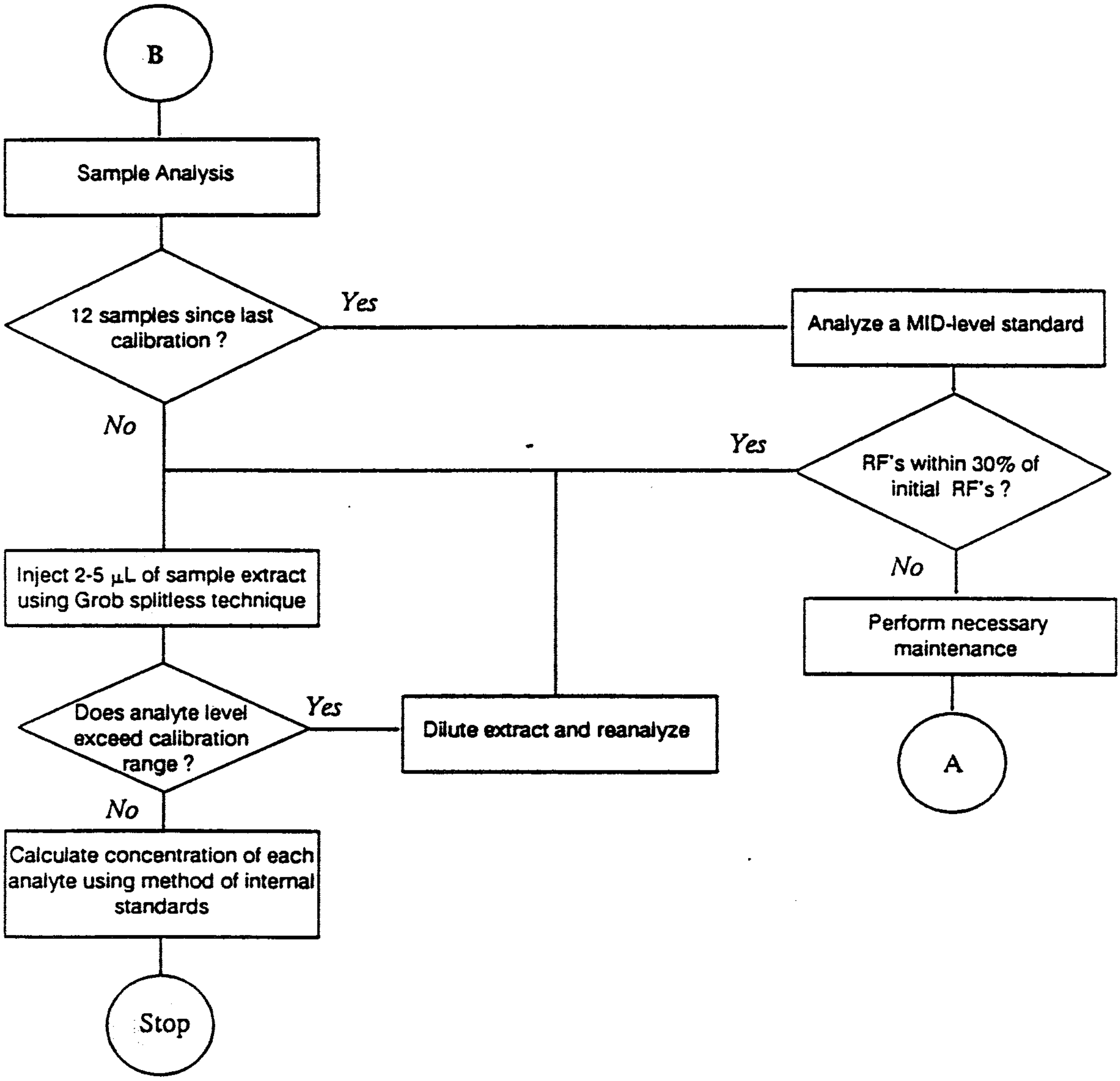

FIGURE 2 (cont). FLOWCHART FOR THE GAS CHROMATOGRAPHIC ANALYSIS OF BUTYLTIN SPECIES IN SEDIMENTS.

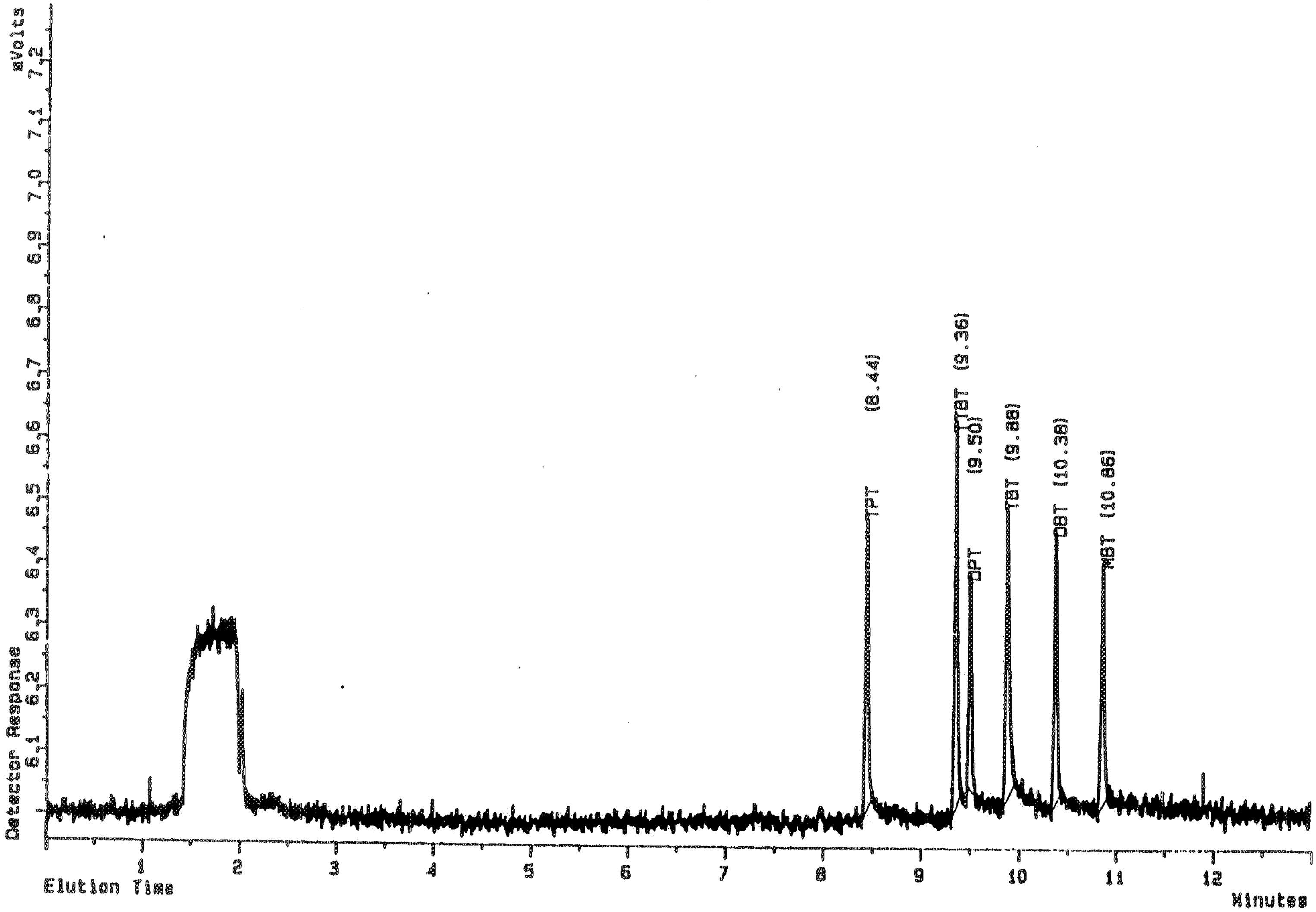

FIGURE 3. GC/FPD CHROMATOGRAM OF A MIXED GC ORGANOTIN STANDARD CONTAINING TTBT AND THE n-PENTYL DERIVATIVES OF TBT, DBT, MBT, TPT, AND DPT APPROXIMATELY THE 0.2 NG/μL PER COMPONENT LEVEL.

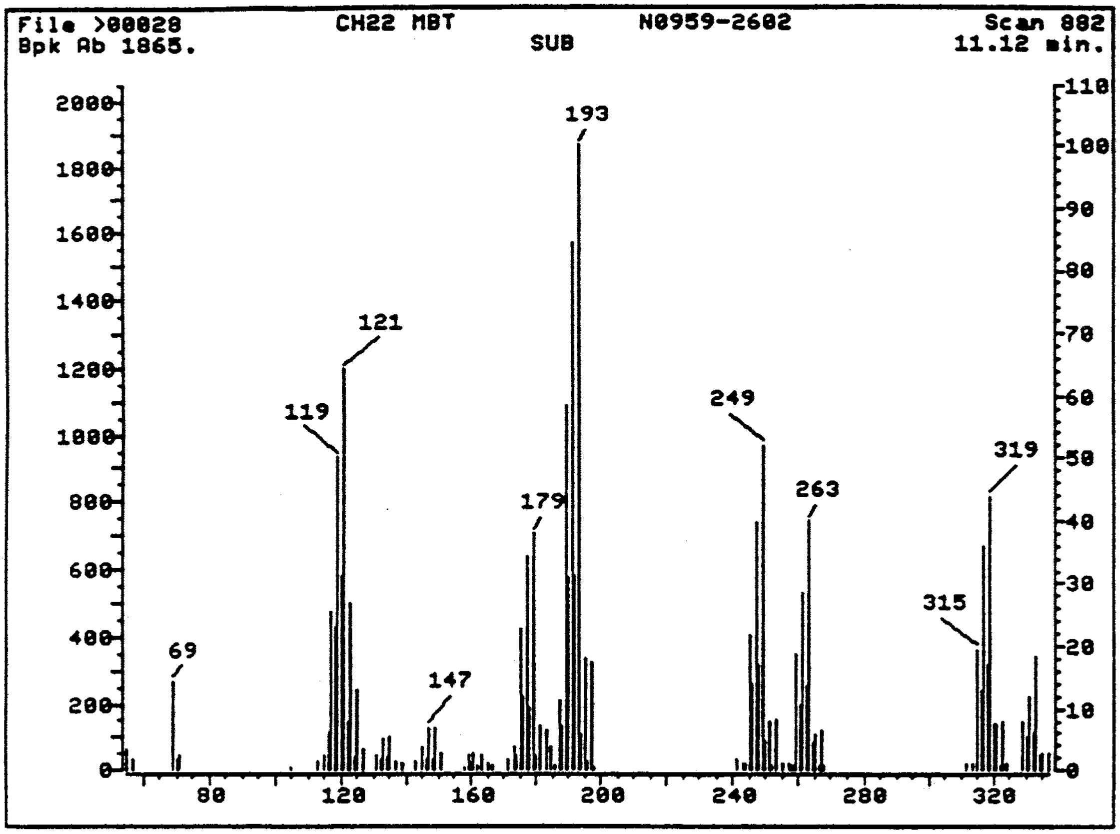

FIGURE 4. FULL SCAN MASS SPECTRUM OF THE n-PENTYL DERIVATIVE OF MONOBUTYLTIN.

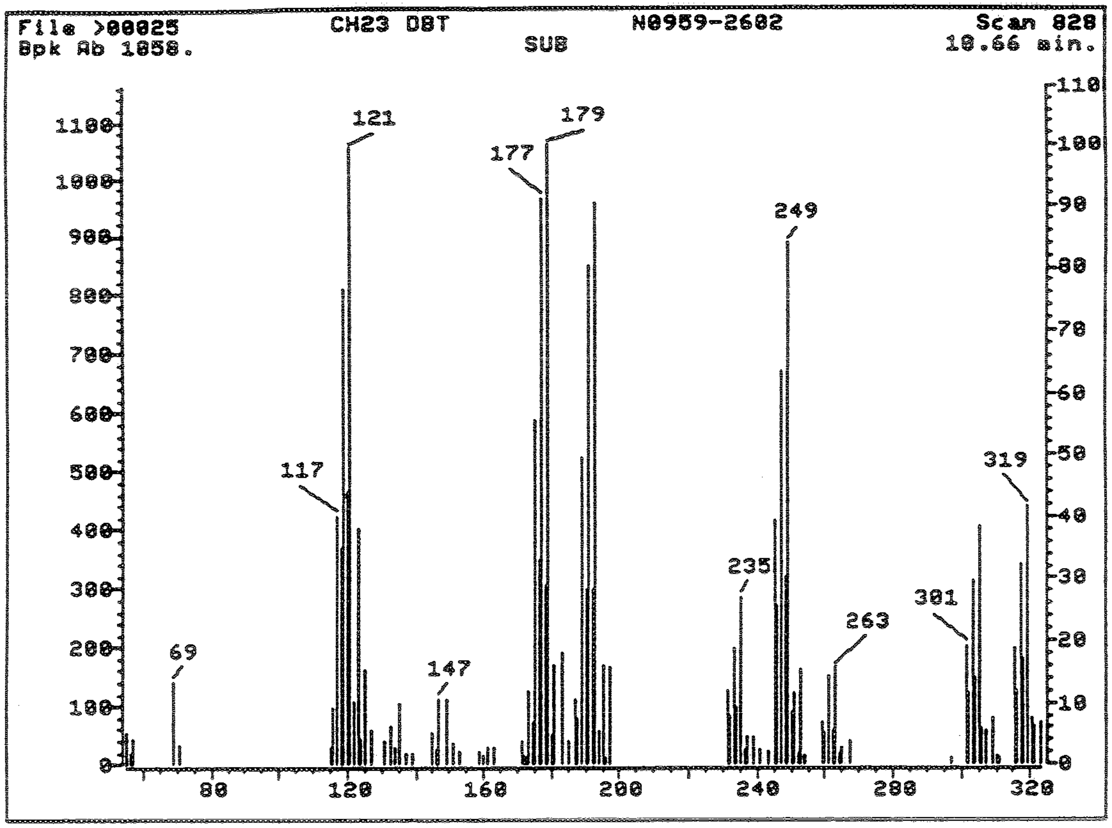

FIGURE 5. FULL SCAN MASS SPECTRUM OF THE n-PENTYL DERIVATIVE OF DIBUTYLTIN.

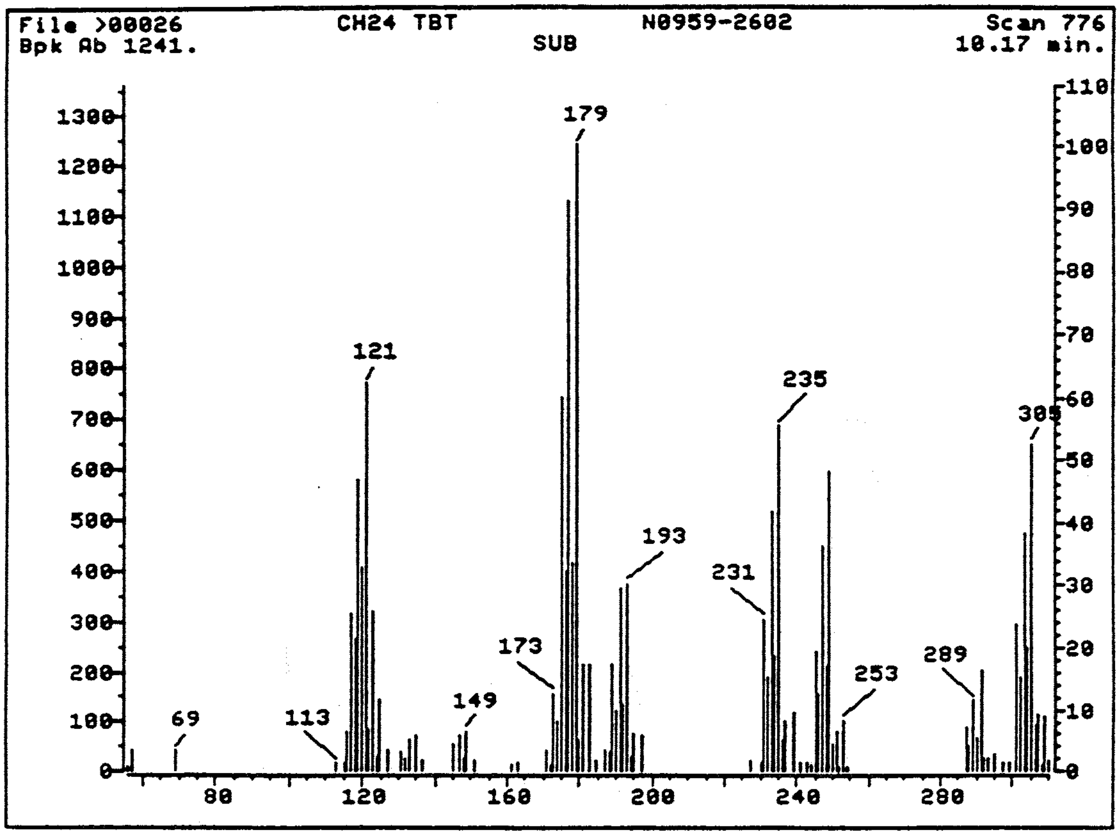

FIGURE 6. FULL SCAN MASS SPECTRUM OF THE n-PENTYL DERIVATIVE OF TRIBUTYLTIN.

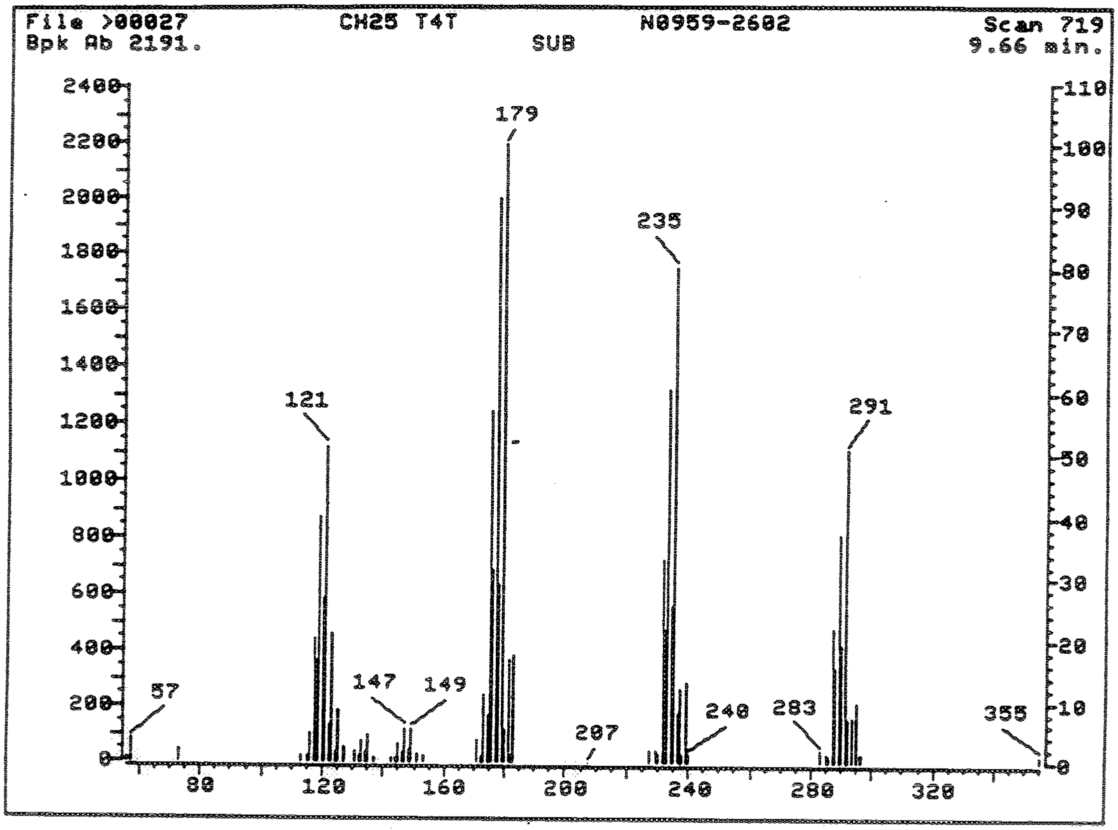

FIGURE 7. FULL SCAN MASS SPECTRUM OF TETRABUTYLTIN.

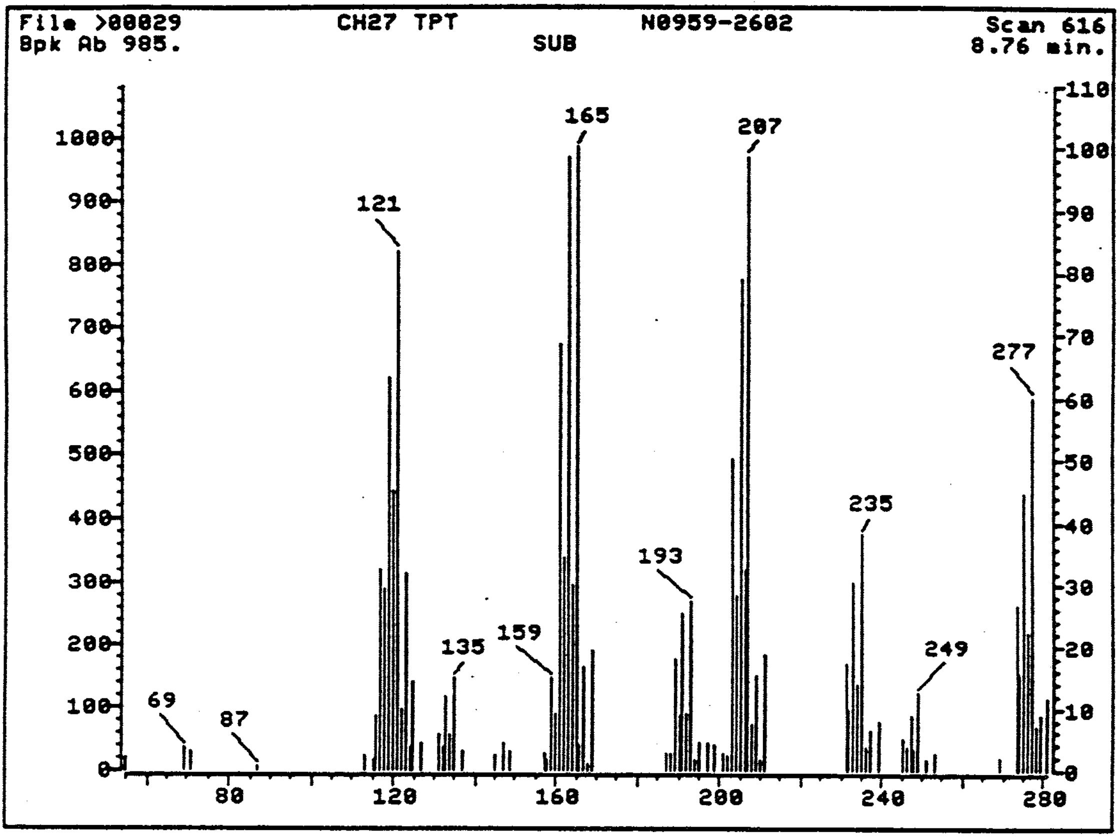

FIGURE 8. FULL SCAN MASS SPECTRUM OF THE n-PENTYL DERIVATIVE OF TRIPROPYLTIN.

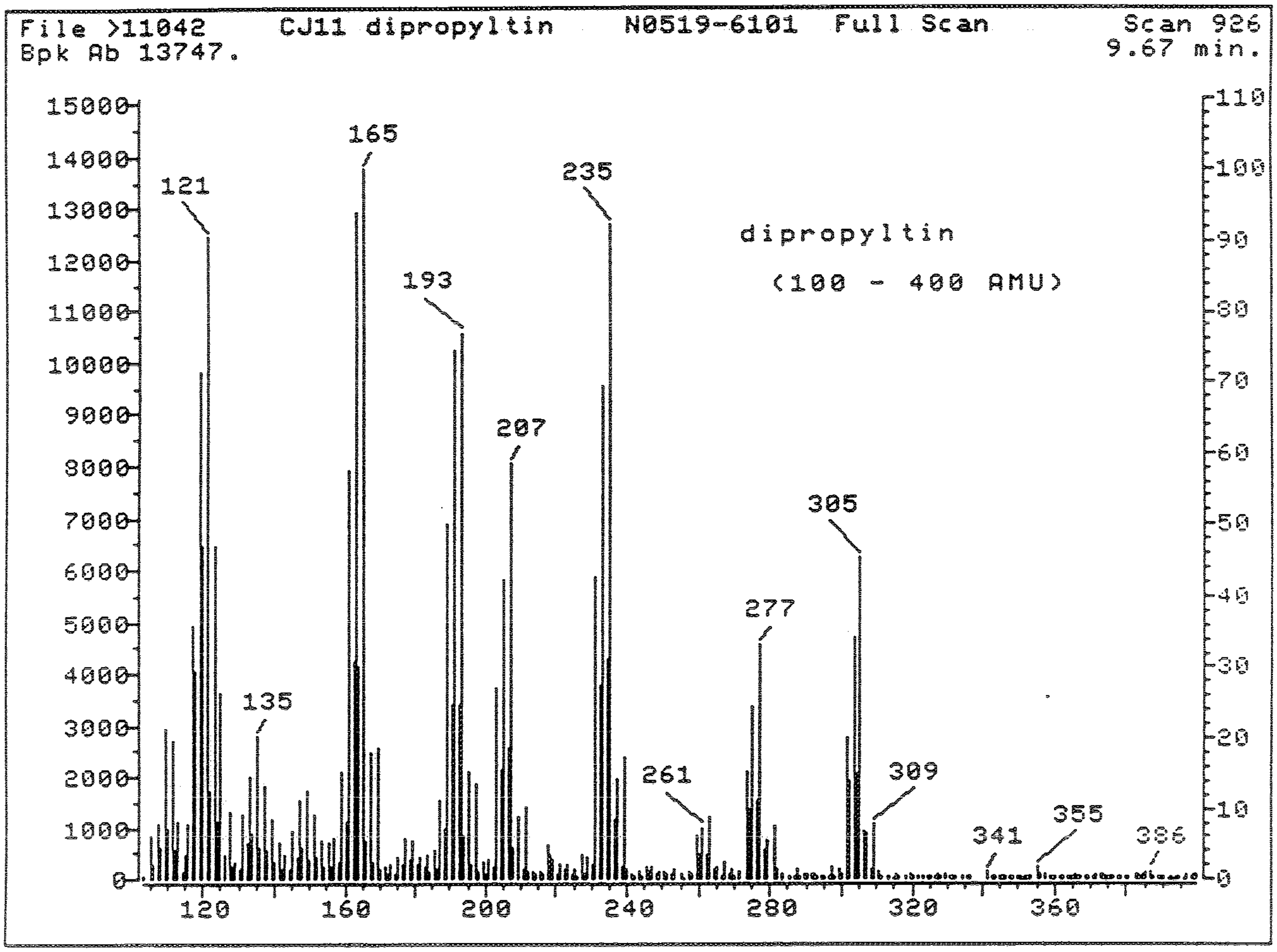

FIGURE 9. FULL SCAN MASS SPECTRUM OF THE n-PENTYL DERIVATIVE OF DIPROPYLTIN.

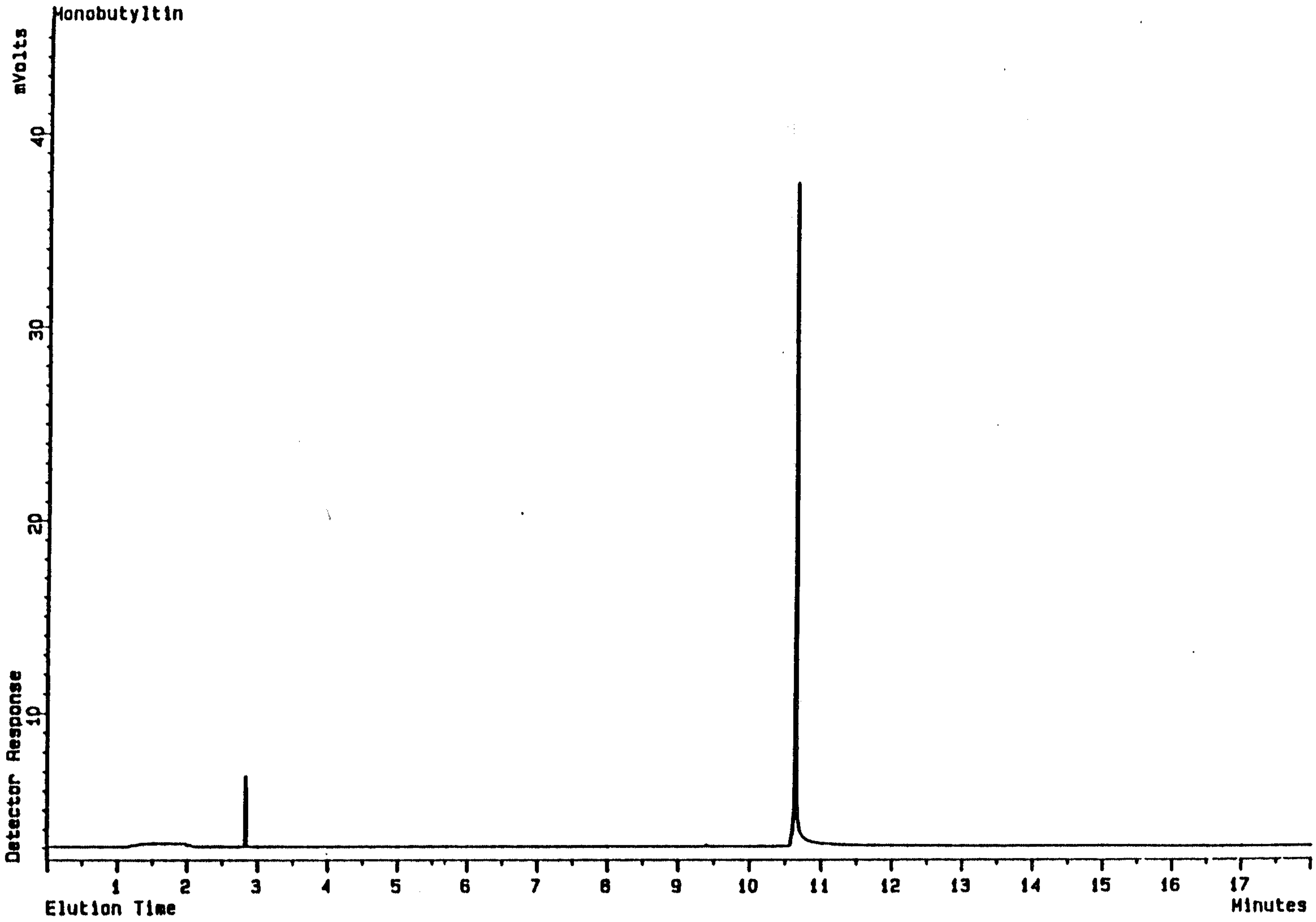

FIGURE 10. GC/FPD CHROMATOGRAM OF THE n-PENTYL DERIVATIVE OF MONOBUTYLTIN.

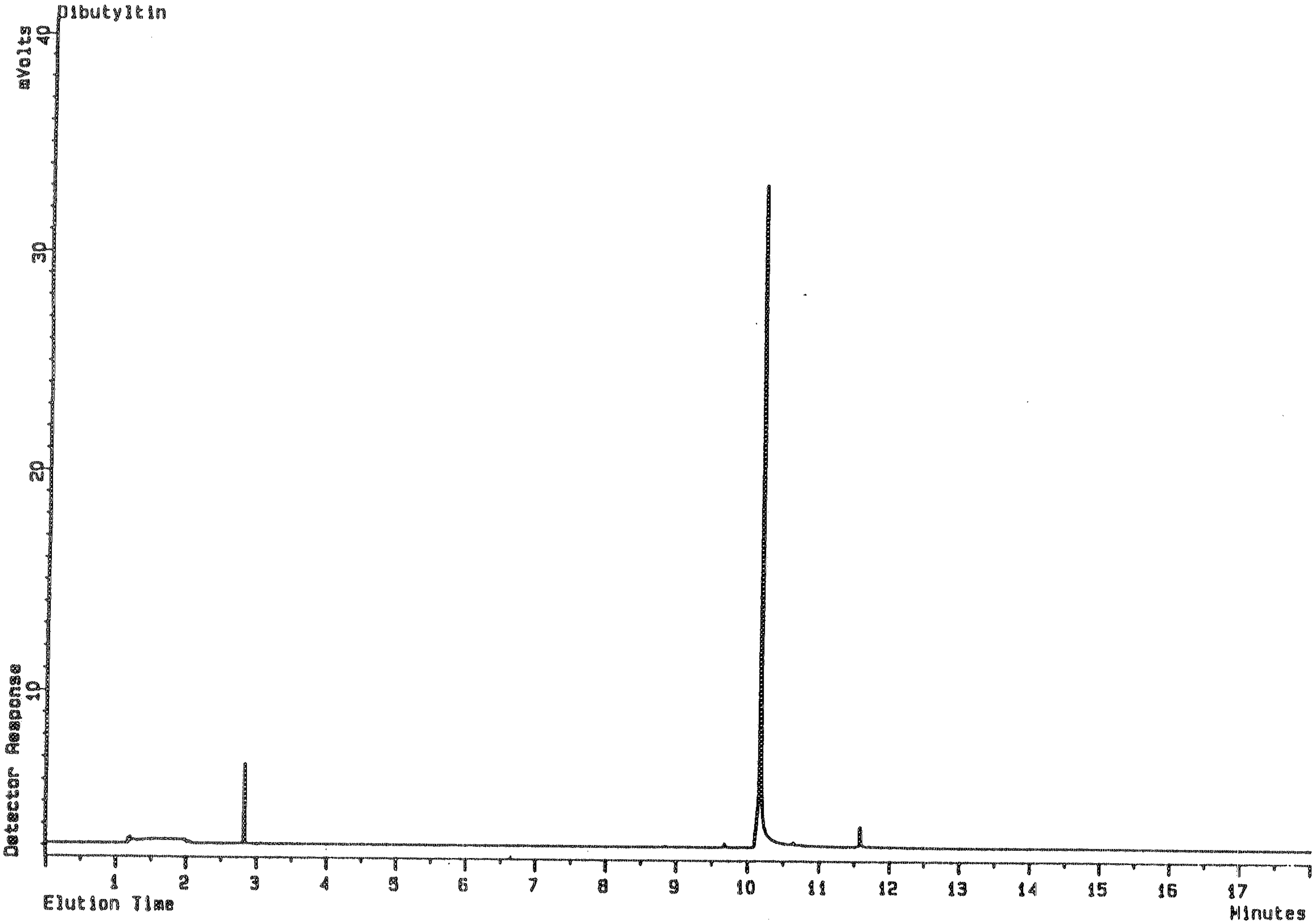

FIGURE 11. GC/FPD CHROMATOGRAM OF THE n-PENTYL DERIVATIVE OF DIBUTYLTIN.

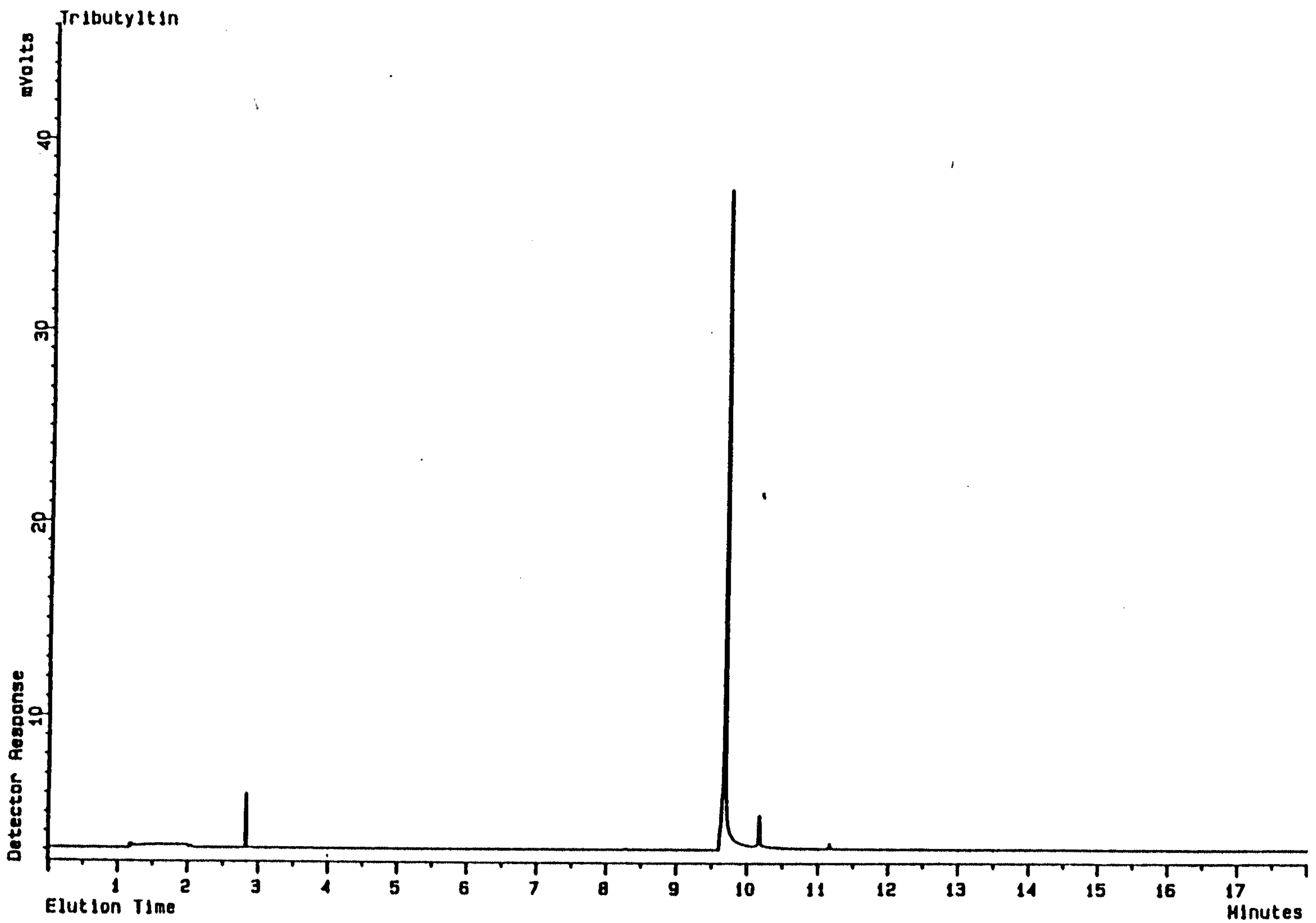

FIGURE 12. GC/FPD CHROMATOGRAM OF THE n-PENTYL DERIVATIVE OF TRIBUTYLTIN.

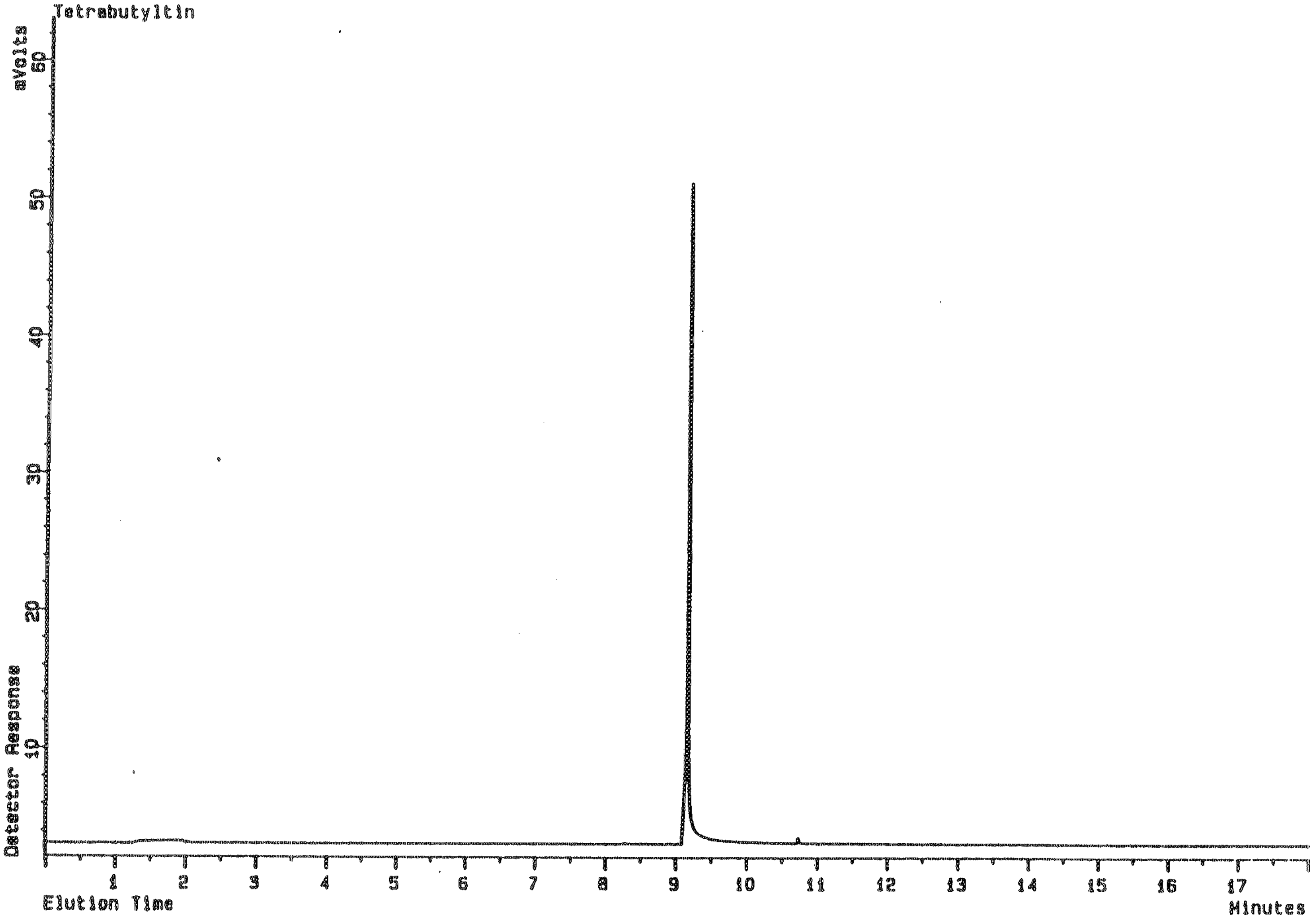

FIGURE 13. GC/FPD CHROMATOGRAM OF TETRABUTYLTIN.

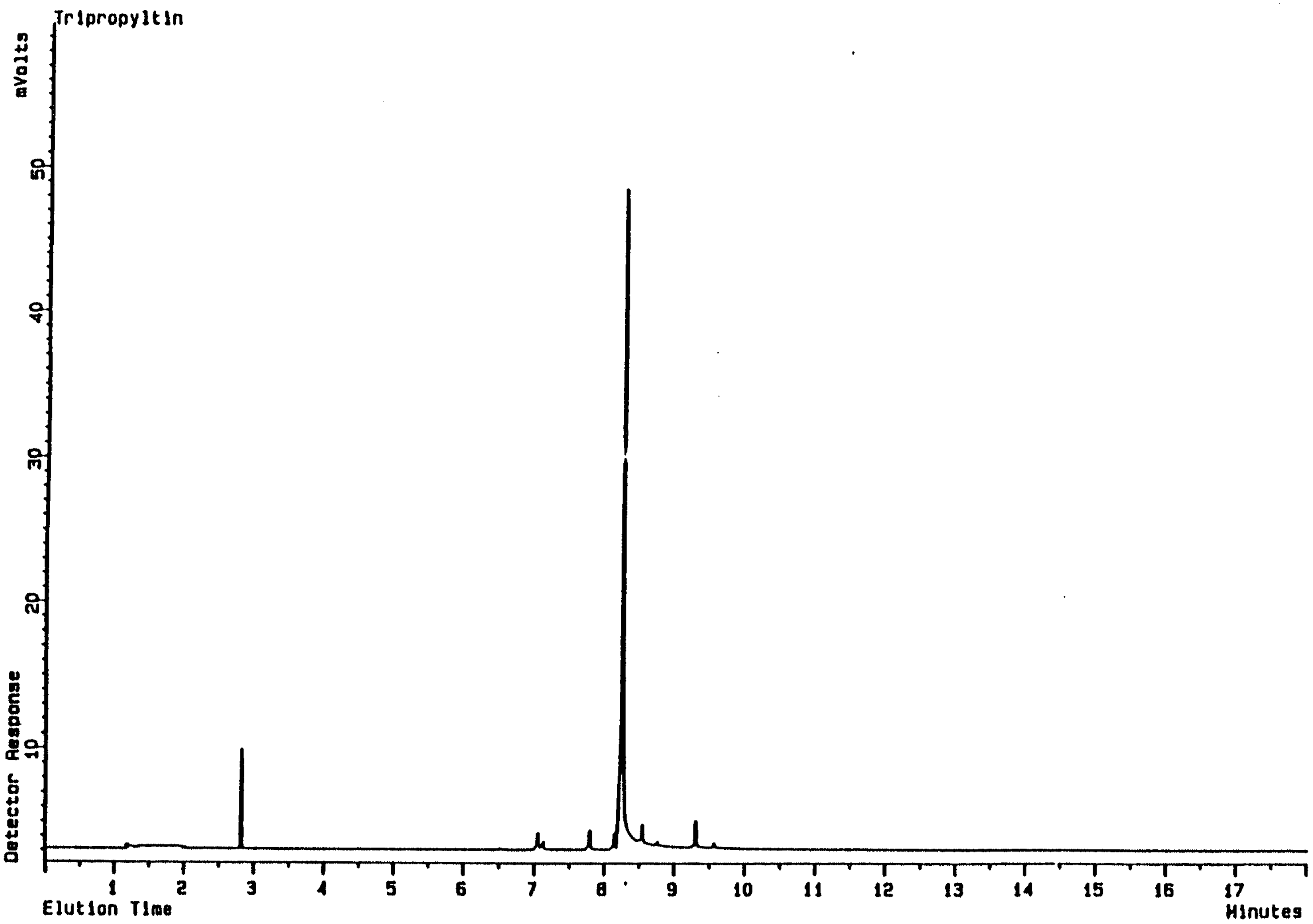

FIGURE 14. GC/FPD CHROMATOGRAM OF THE n-PENTYL DERIVATIVE OF TRIPROPYLTIN.

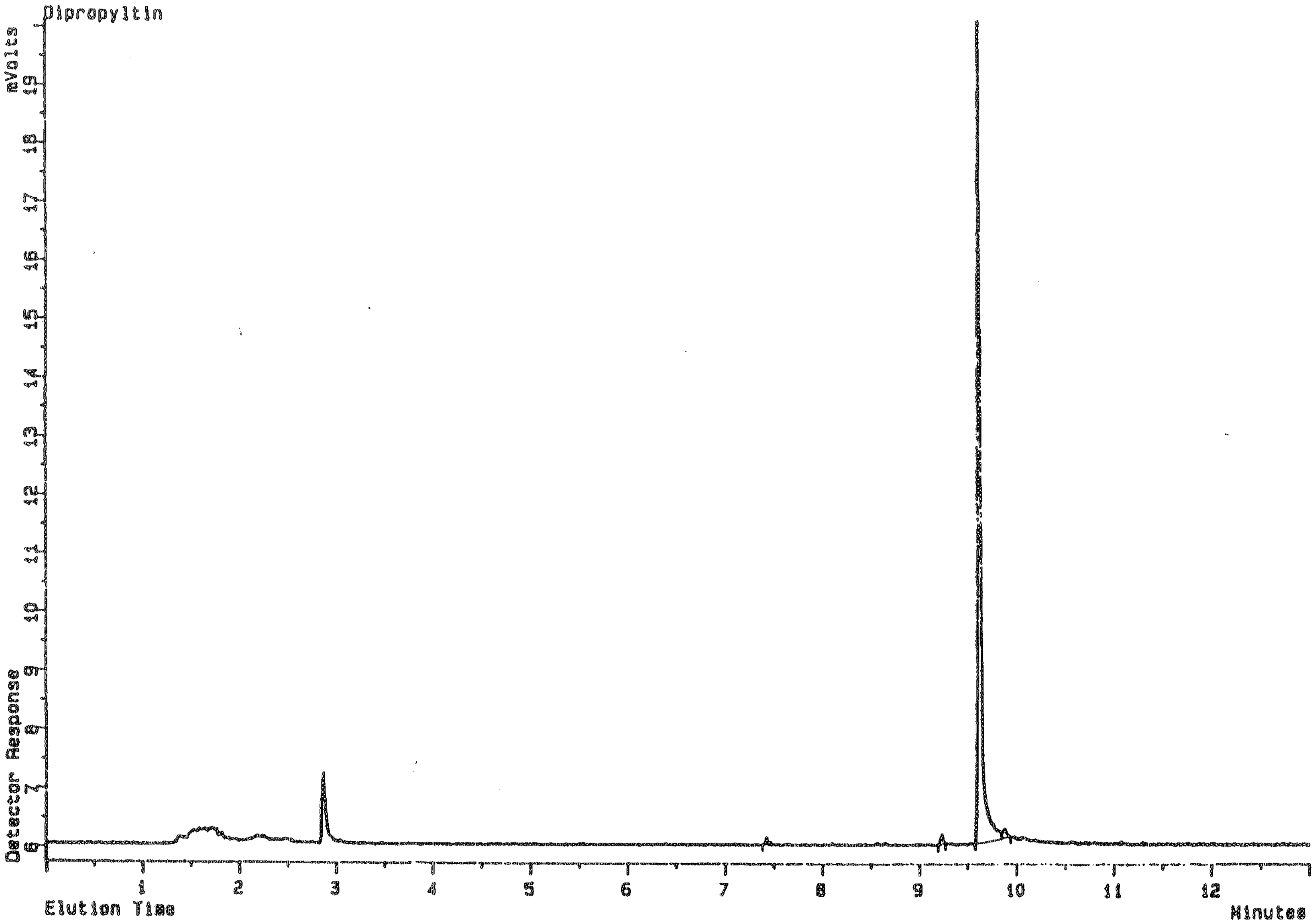

FIGURE 15. GC/FPD CHROMATOGRAM OF THE n-PENTYL DERIVATIVE OF DIPROPYLTIN.

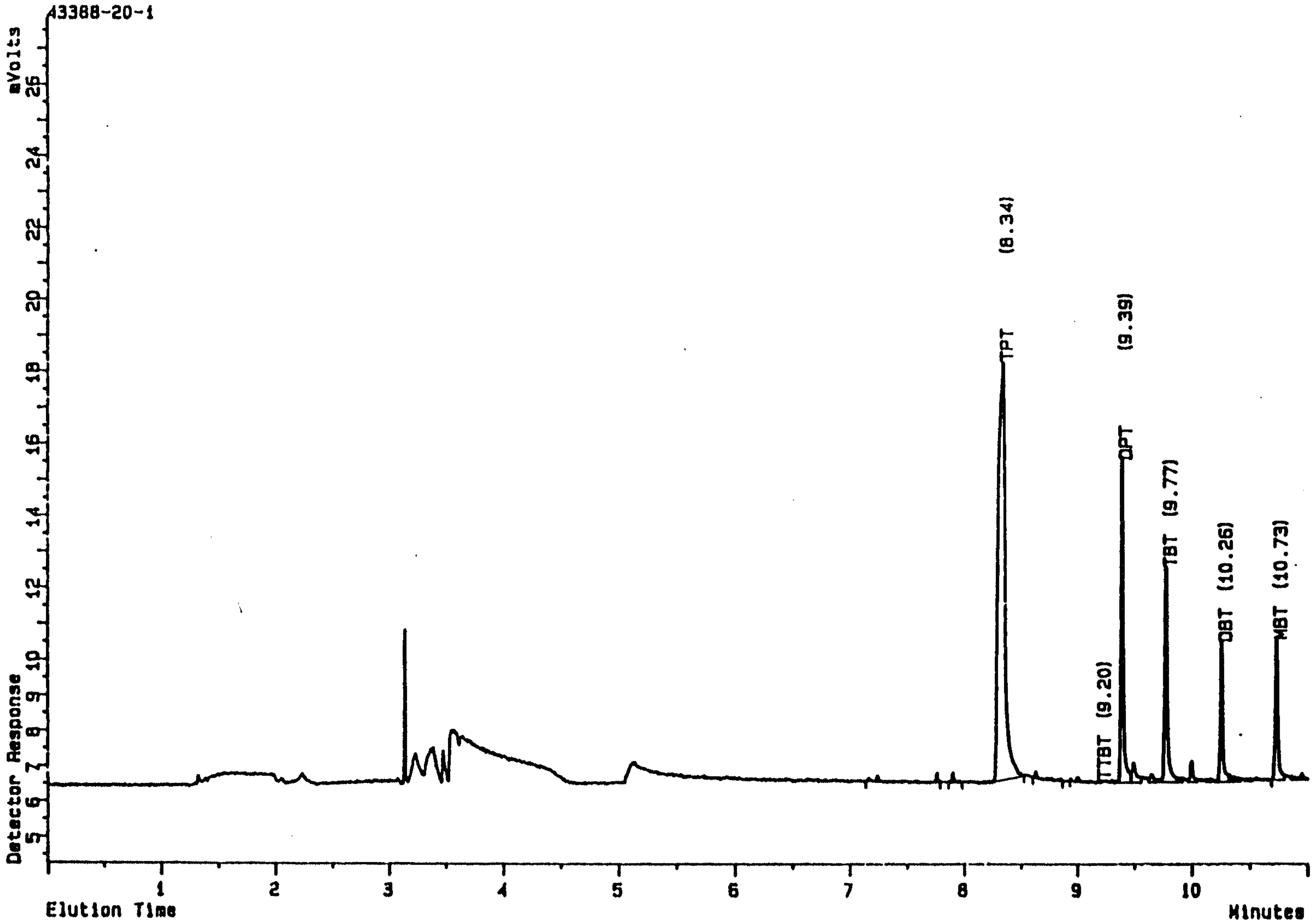

FIGURE 16. GC/FPD CHROMATOGRAM OF TEST SEDIMENT CHB-1.

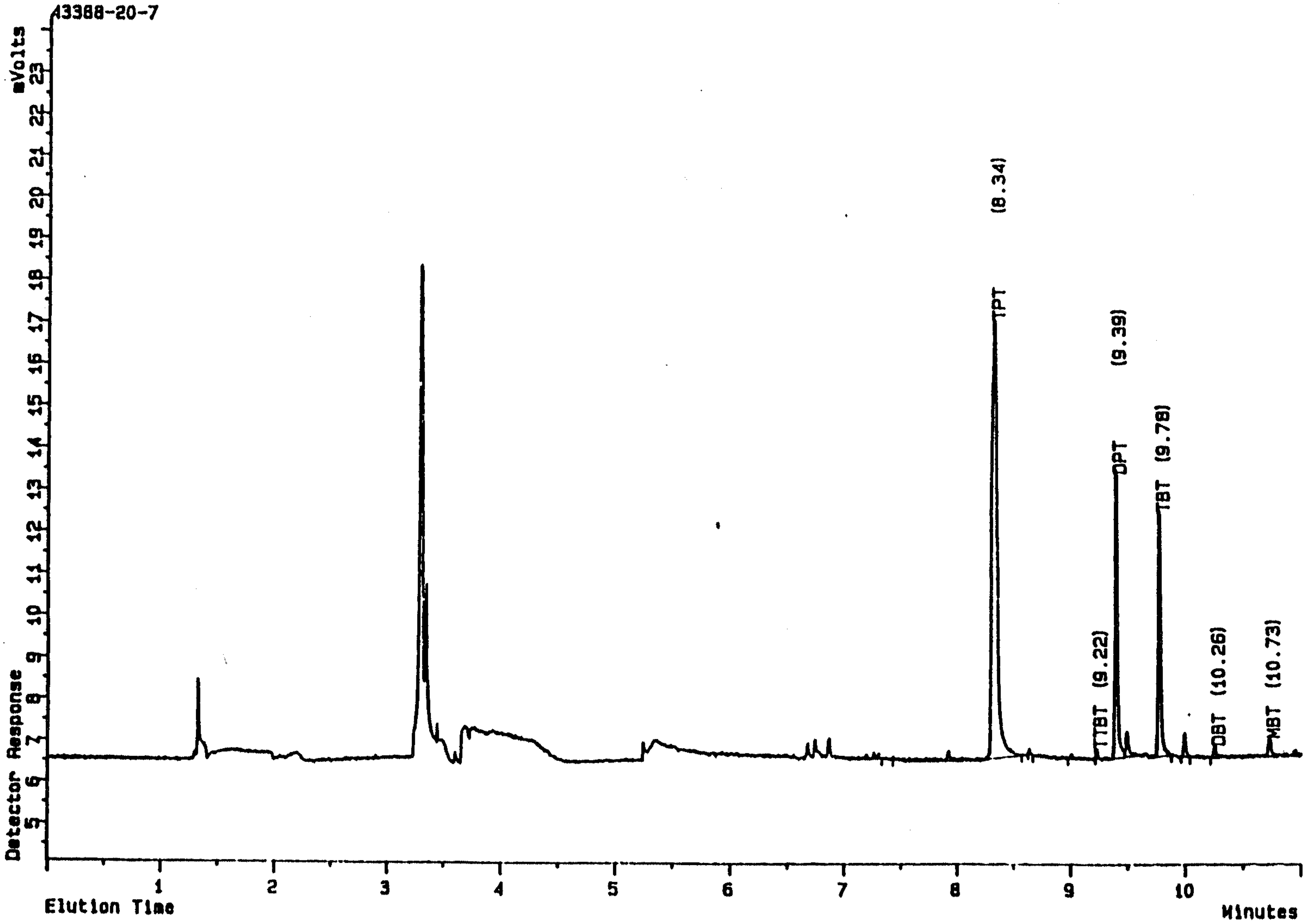

FIGURE 17. GC/FPD CHROMATOGRAM OF TEST SEDIMENT CHB-2.

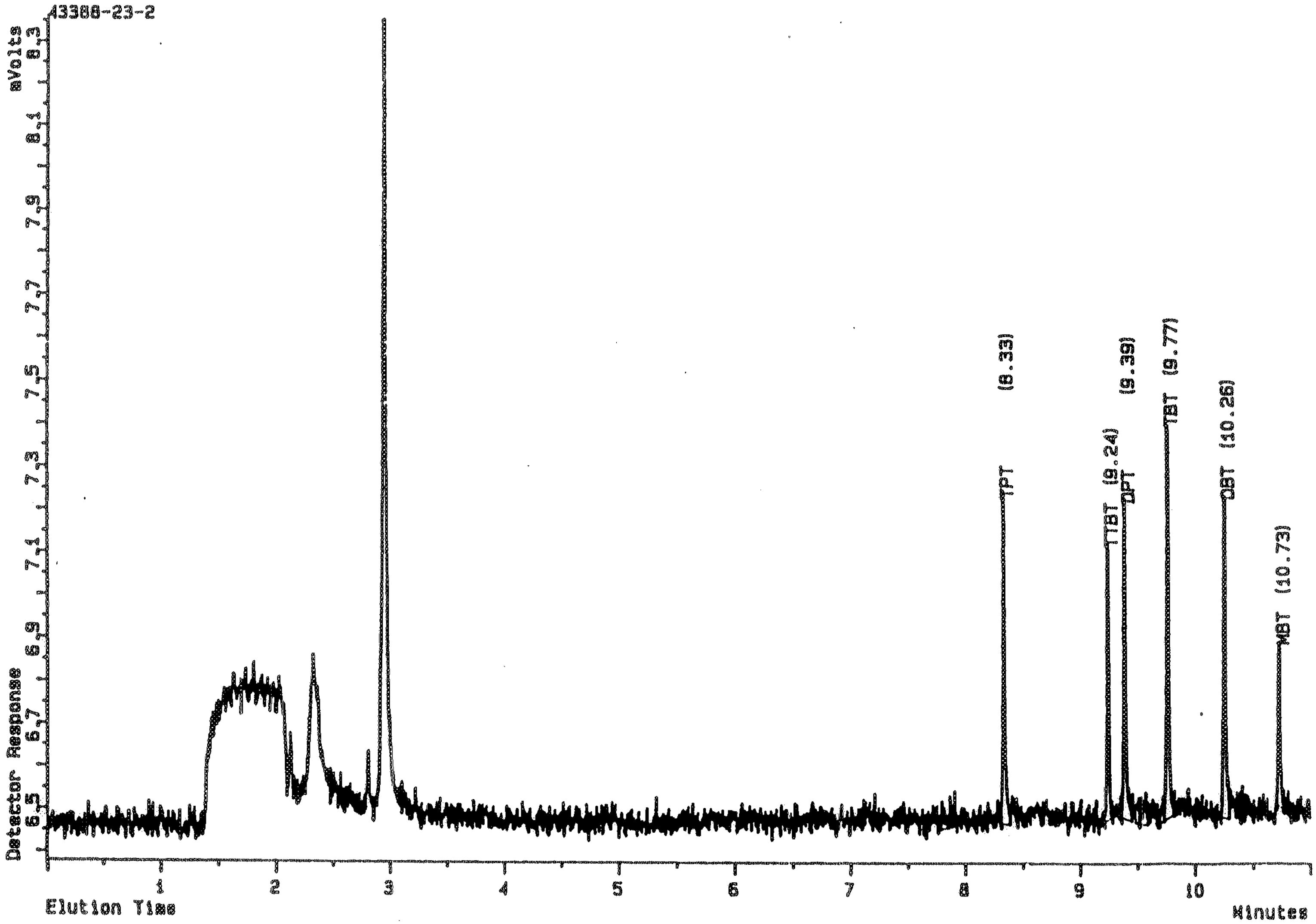

FIGURE 18. GC/FPD CHROMATOGRAM OF TEST SEDIMENT CHB-1 FORTIFIED WITH MBT, DBT, TBT AND TTBT AT THE 50 µg/Kg PER COMPONENT LEVEL.

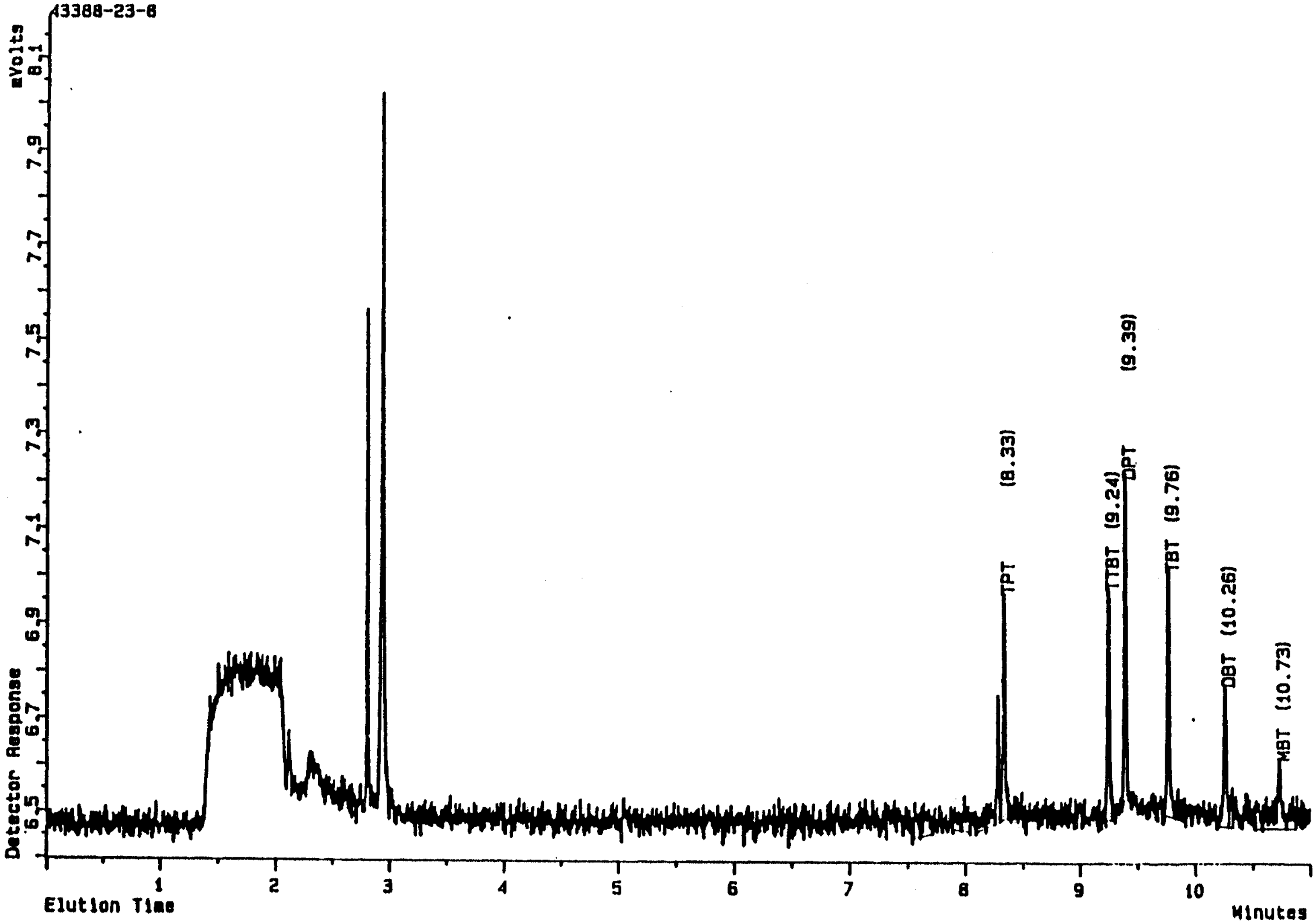

FIGURE 19. GC/FPD CHROMATOGRAM OF TEST SEDIMENT CHB-2 FORTIFIED WITH MBT, DBT, TBT AND TTBT AT THE 50 µg/Kg PER COMPONENT LEVEL.

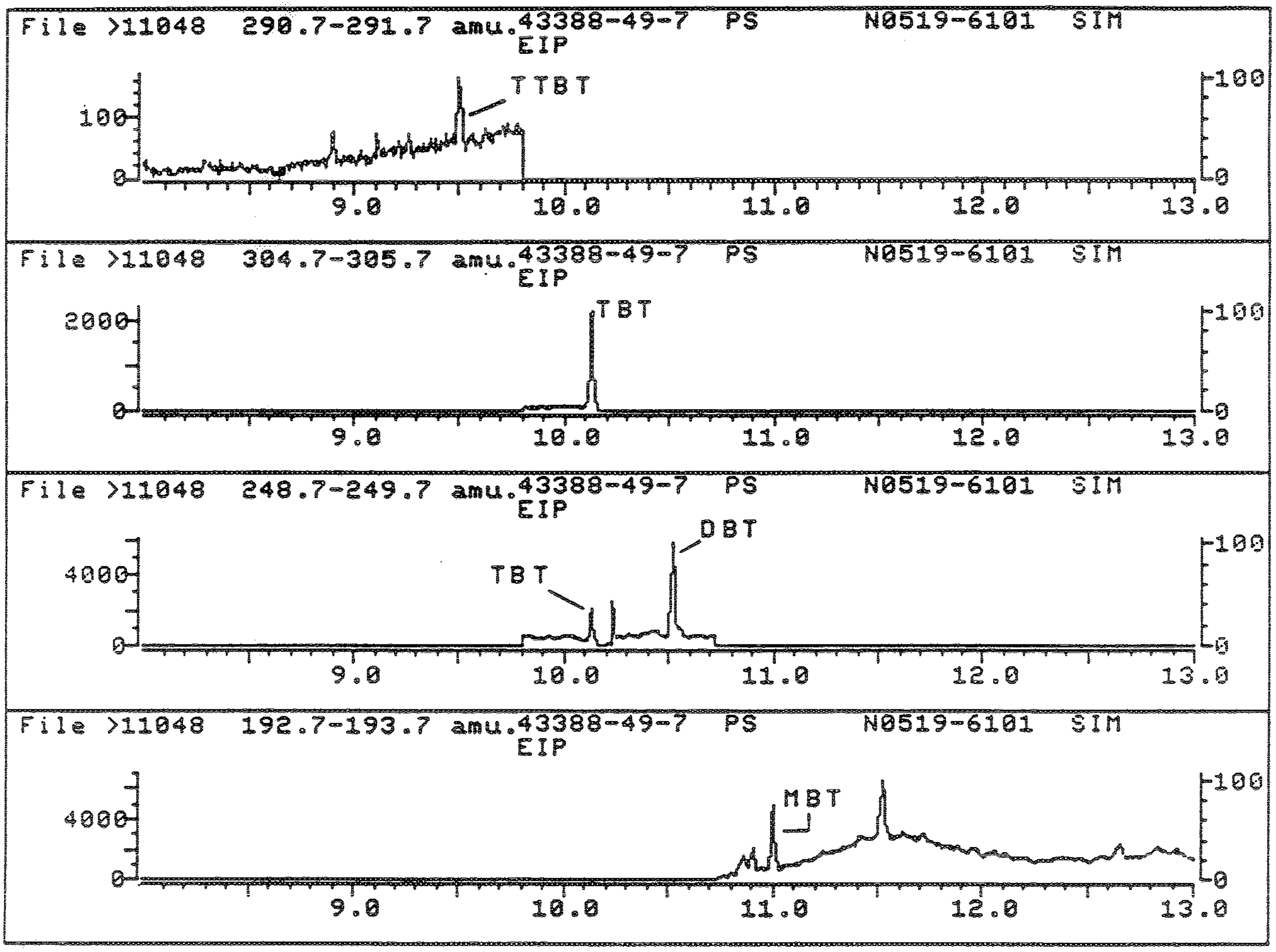

FIGURE 20. GC/SIM EXTRACTED ION CHROMATOGRAM FOR SEDIMENT PS. ION TRACE ONE (TOP) IS TTBT, ION TRACE TWO IS TBT, ION TRACE THREE IS DBT, AND ION TRACE FOUR (BOTTOM) TRACE IS MBT.

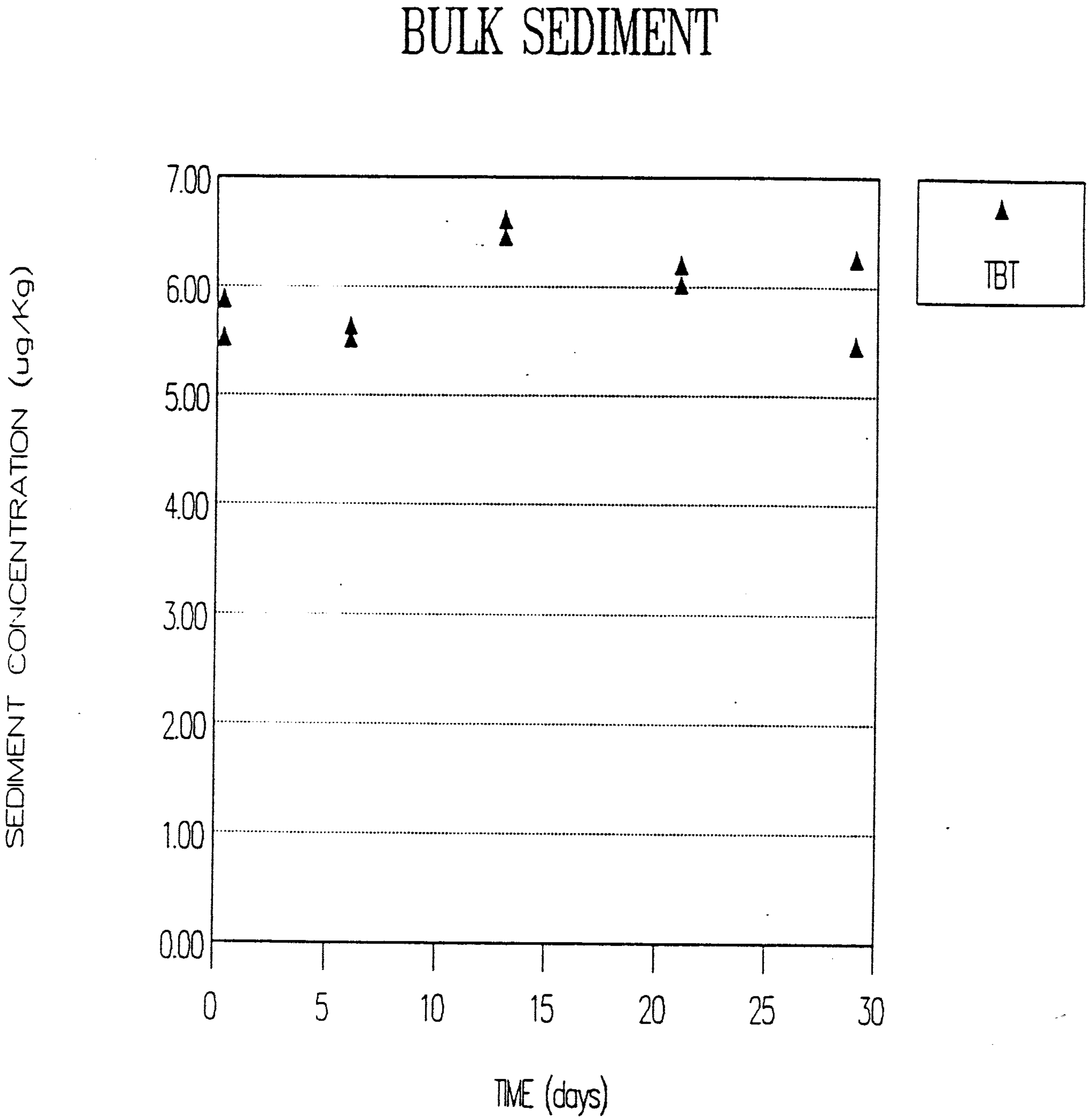

FIGURE 21. BULK SEDIMENT STORAGE EXPERIMENTS: RELATIVE STABILITY PLOT FOR TBT.

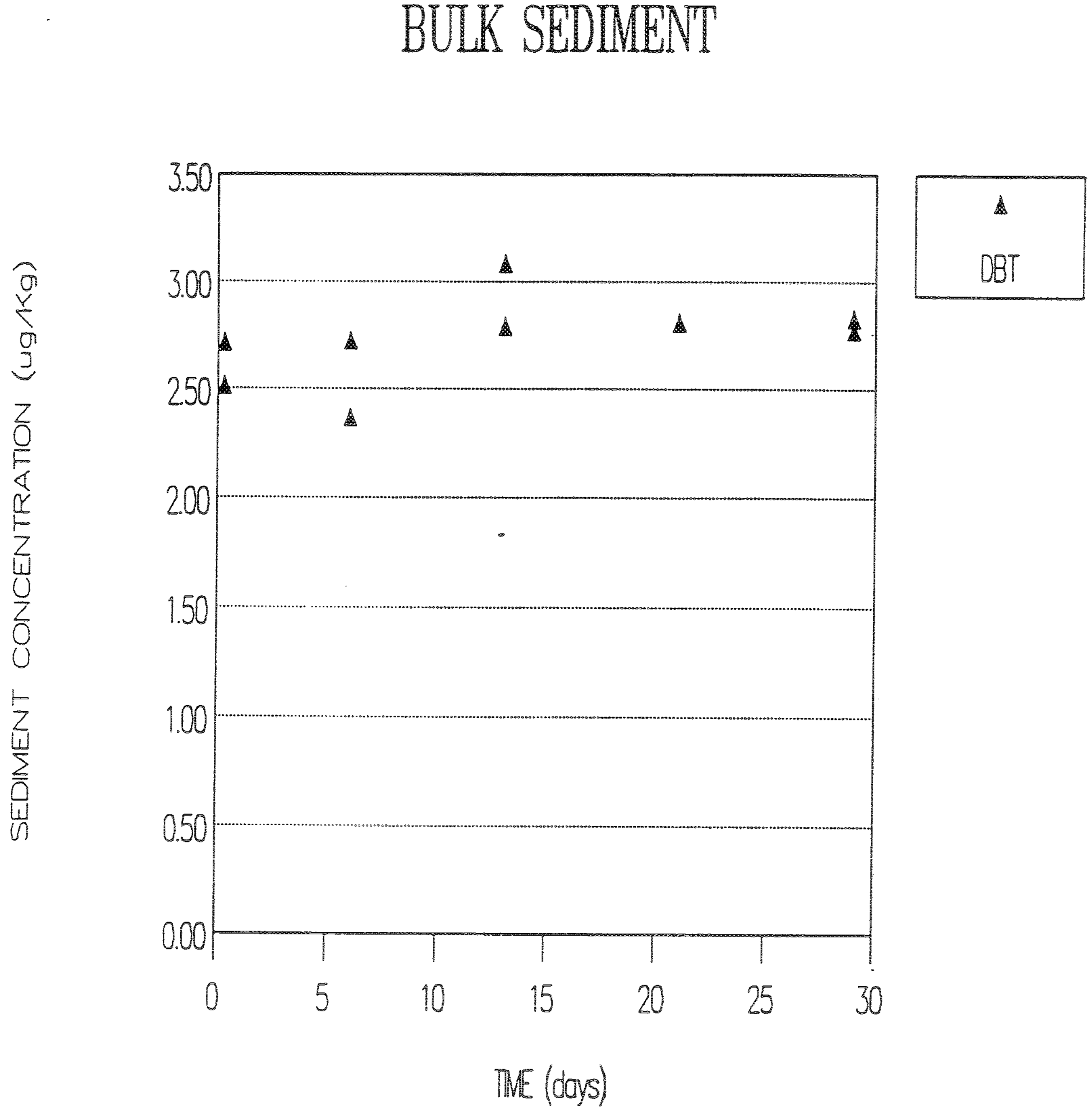

FIGURE 22. BULK SEDIMENT STORAGE EXPERIMENTS: RELATIVE STABILITY PLOT FOR DBT.

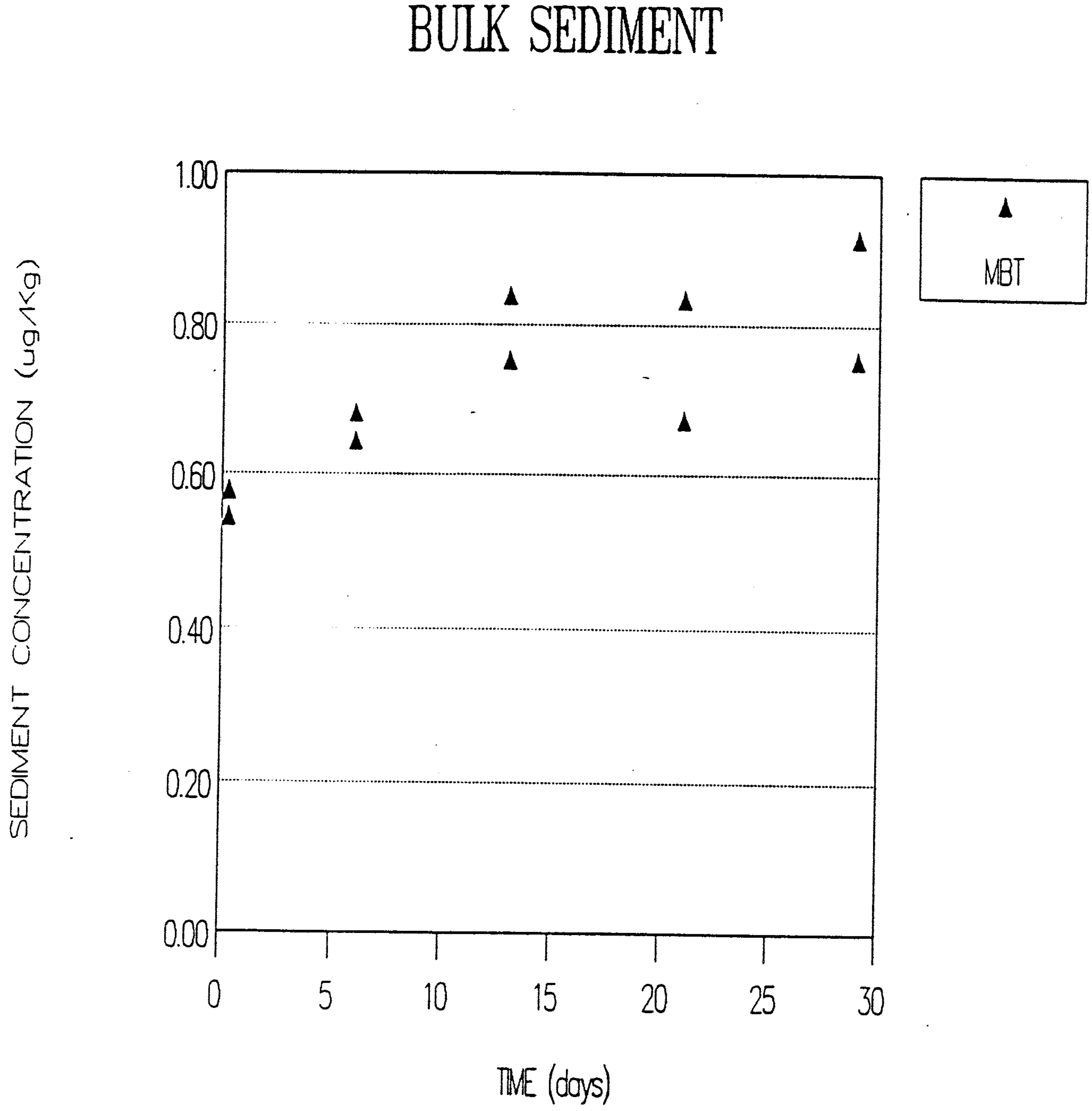

FIGURE 23. BULK SEDIMENT STORAGE EXPERIMENTS: RELATIVE STABILITY PLOT FOR MBT.

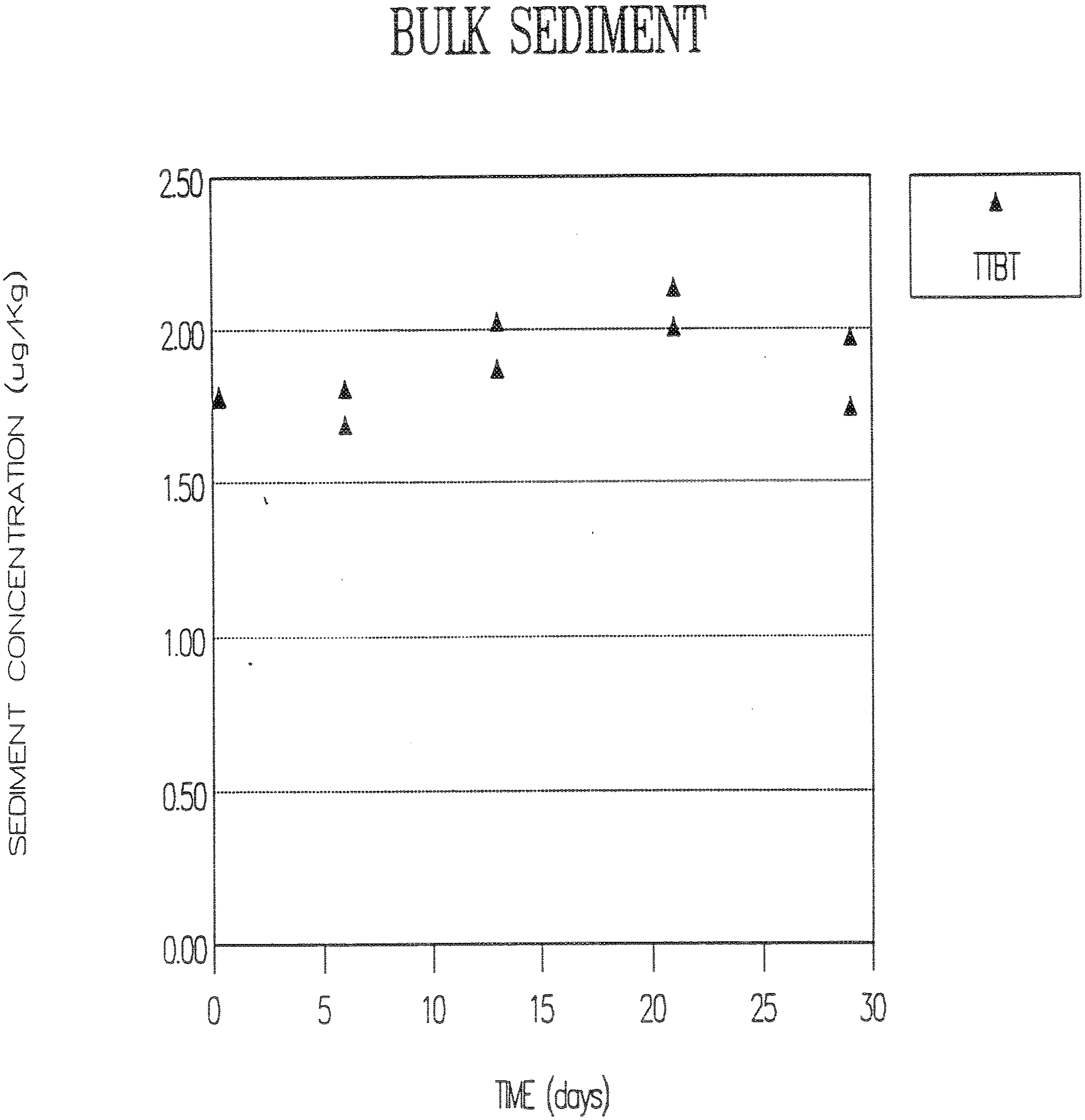

FIGURE 24. BULK SEDIMENT STORAGE EXPERIMENTS: RELATIVE STABILITY PLOT FOR TTBT.

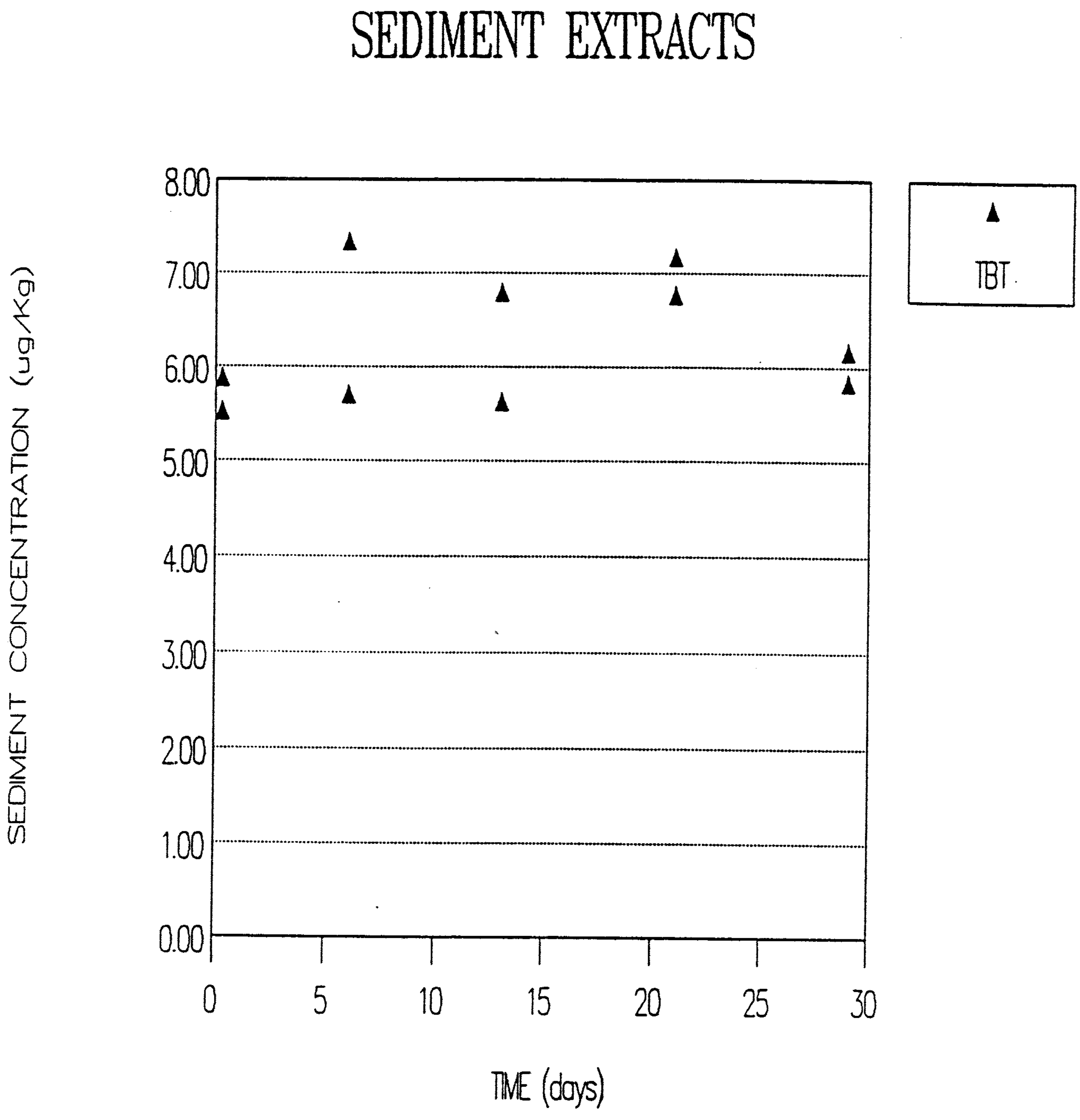

FIGURE 25. SEDIMENT EXTRACT STORAGE EXPERIMENTS: RELATIVE STABILITY PLOT FOR TBT.

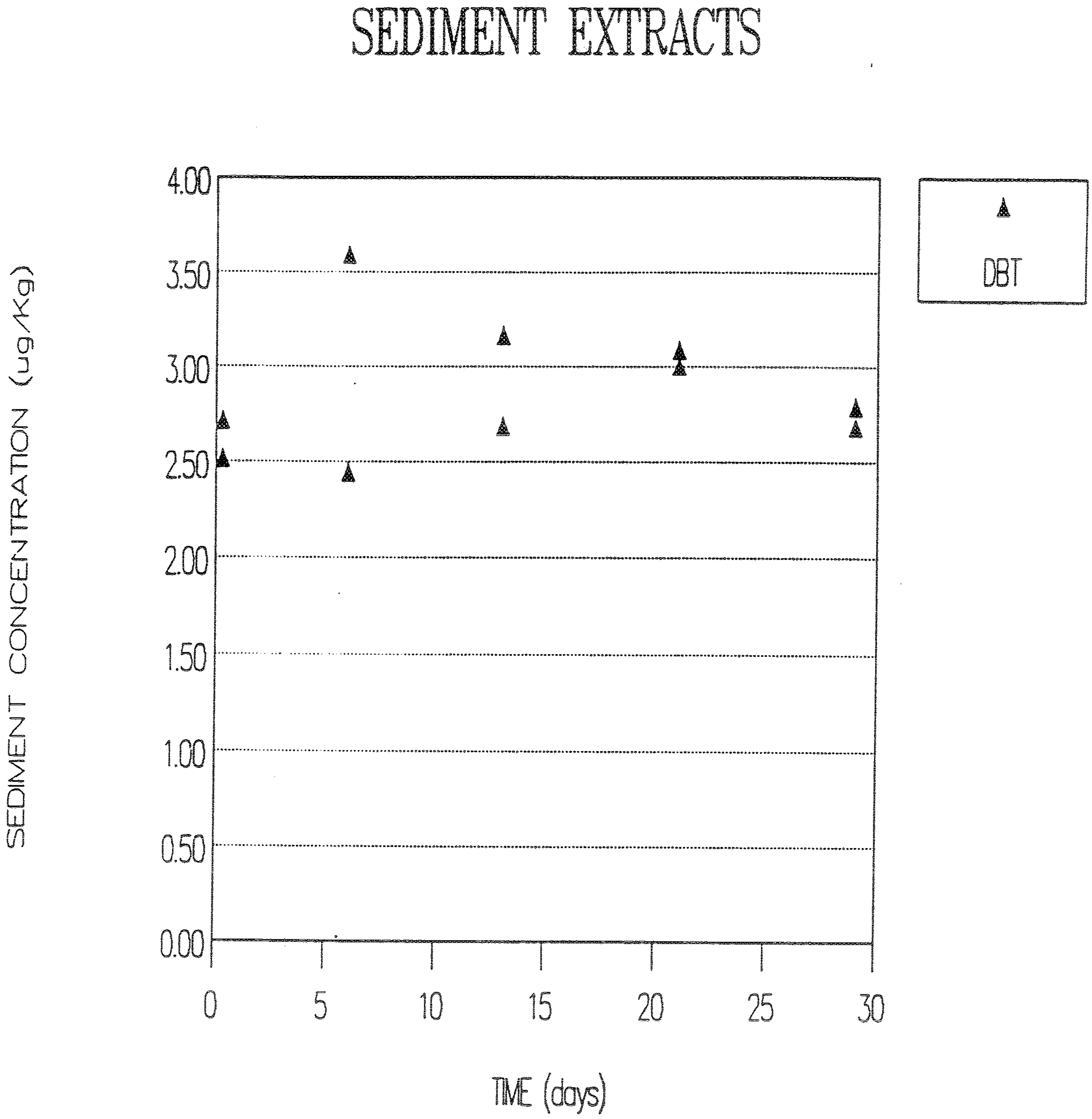

FIGURE 26. SEDIMENT EXTRACT STORAGE EXPERIMENTS: RELATIVE STABILITY PLOT FOR DBT.

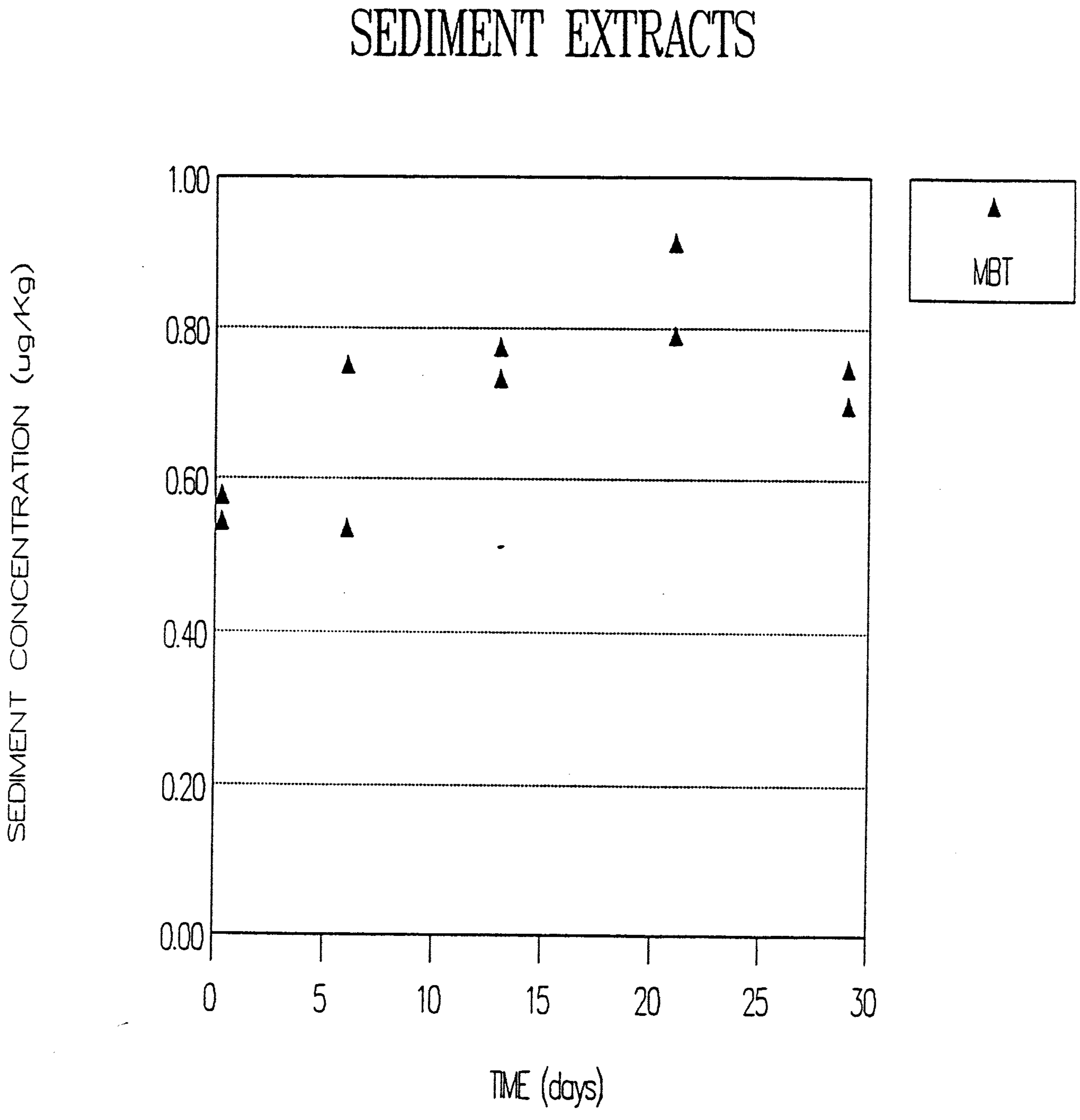

FIGURE 27. SEDIMENT EXTRACT STORAGE EXPERIMENTS: RELATIVE STABILITY PLOT FOR MBT.

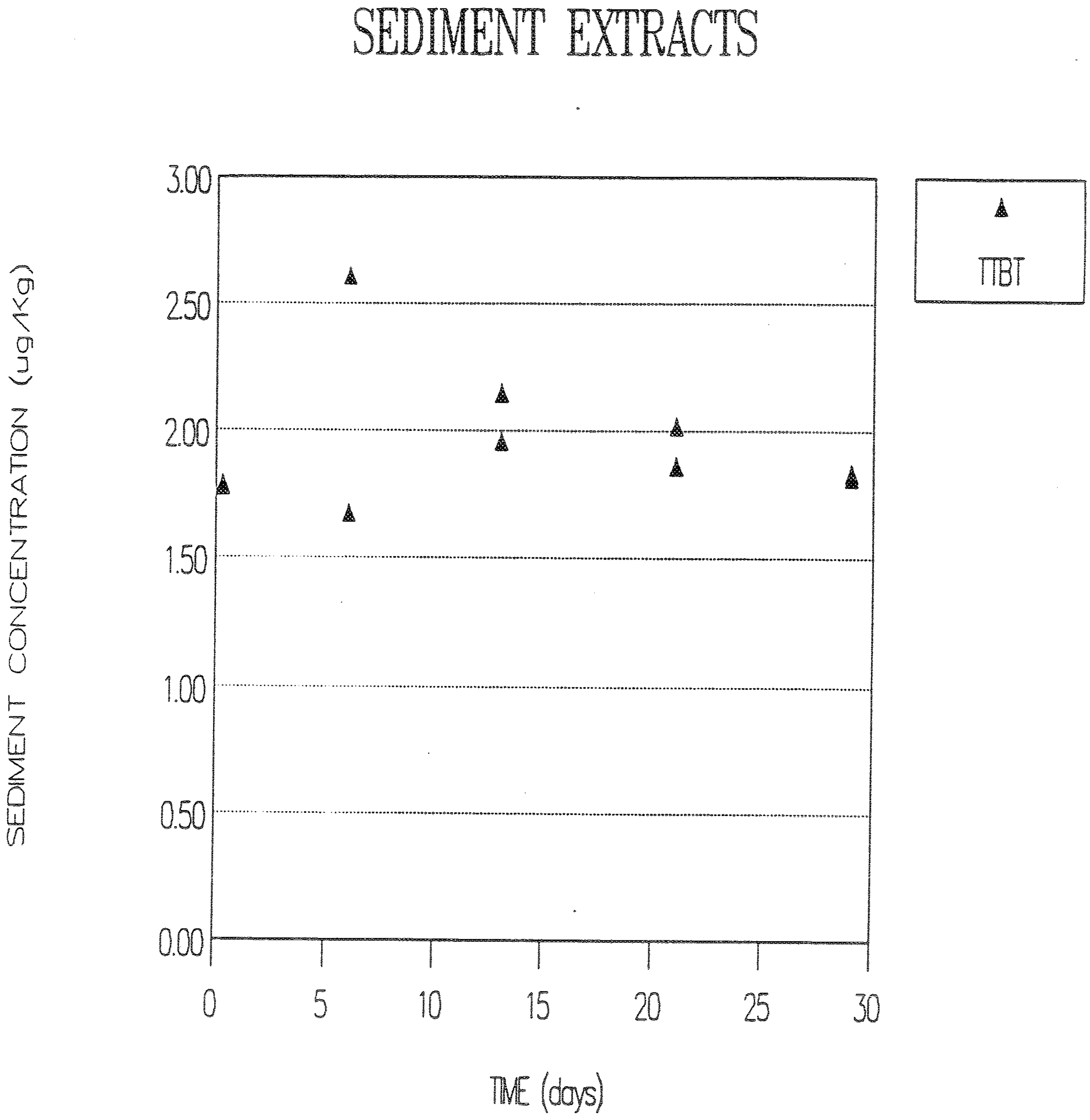

FIGURE 28. SEDIMENT EXTRACT STORAGE EXPERIMENTS: RELATIVE STABILITY PLOT FOR TTBT.

Appendix A. PROTOCOL, AMENDEMENTS TO PROTOCOL, AND PROTOCOL DEVIATIONS.

PROTOCOL

Title: "Measurement of Butyltin Species in Sediments by n-Pentyl Derivatization with Gas Chromatography/Flame Photometric Detection (GC/FPD) and Optional Confirmation by Gas Chromatography/Mass Spectrometry (GC/MS)"

Sponsor: Consortium of Tributyltin Manufacturers
M&T Chemicals, Inc., Woodbridge, New Jersey
Sherex Chemicals Company, Inc., Dublin, Ohio

Testing Facility: Battelle Ocean Sciences
397 Washington Street
Duxbury, Massachusetts 02332

Project Number: N-0519-6101

Proposed Experimental Start Date: May 16, 1988
Proposed Experimental Completion Date: September 16, 1988

Signatures:

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Envirosphere Company

1.0 OBJECTIVES

The Consortium of Tributyltin Manufacturers has requested Battelle to refine and validate an analytical method for the determination of trace levels of butyltin species in sediments. The method will be amenable to the analysis of tributyltin (TBT), and its degradation products dibutyltin (DBT) and monobutyltin (MBT), as well as the TBT manufacturing impurity tetrabutyltin (TET). The method must meet all criteria of the Pesticide Assessment Guidelines-Subsection D (1982) of the U.S. Environmental Protection Agency (EPA) Pesticides Programs, and development and validation activities must adhere to EPA Good Laboratory Practices (40 CFR Part 160).

2.0 TECHNICAL APPROACH

The development of the method for analysis of butyltin species in sediments will be based on solvent extraction of the butyltins from sediment, followed by n-pentyl derivatization of the extracted butyltin species, Florisil column cleanup of the extract, and quantitative determination of the butyltins by gas chromatography/flame photometry (GC/FPD). An optional analyte confirmation step by gas chromatography/mass spectrometry (GC/MS) is presented.

The method development will be carried out in four phases:

- Reconnaissance Experiments
- Method Detection Limit, Recovery, and Precision Determinations
- Field Sample Verification
- Storage Experiments.

Below, the analytical method is presented, followed by a description of each phase of work.

2.1 TEST SUBSTANCE IDENTIFICATION

Substance	CAS Number	Abbreviation
Tributyltin chloride	1461-22-9	TBT
Dibutyltin dichloride	683-18-1	DBT
Monobutyltin trichloride	1118-46-3	MBT
Tetrabutyltin	1461-25-2	TET

2.2 ANALYTICAL METHOD

The proposed method for the determination of butyltins in sediments is based on a procedure presented by Rice et al. (1987), with modifications that will allow for implementation in a monitoring laboratory environment. The method is described below. Modifications to this method may be made during the reconnaissance phase of this study.

2.2.1 Extraction

1. Approximately 100 g of sediment are separated from any overlying water by decantation. The sediment is transferred to a tared Teflon jar and weighed to the nearest 0.1 g by using a top loading balance. The sample is centrifuged for 5 min at 1500 rpm, any resulting water decanted, and the jar weighed to determine the final sediment weight.

2. Next, 150 mL 0.05 percent tropolone in 1:1 methylene chloride: acetone is added to the sample. One mL of the appropriate concentration quantitation internal standard (QIS) tripropyltin chloride (TPT) is added. The bottle is capped, and agitated on a shaker table for a minimum of 12 h.

3. After this period, the bottle is removed from the shaker table, and centrifuged at 1500 rpm for 5 min to separate the sediment from the solvent. The solvent is decanted into an Erlenmeyer flask. Another 150 mL of 0.05 percent tropolone in 1:1 methylene chloride:acetone is added to the sediment, and the extraction is repeated for a minimum of 4 h.

4. After this period, the mixture is again centrifuged at 1500 rpm for 5 min, and the solvent decanted and added to the Erlenmeyer flask. Next, the sediment is extracted with 150 mL 0.05 percent tropolone in hexane. This mixture is extracted on the shaker table for a minimum of 8 hours.

5. After this period, the mixture is centrifuged at 1500 rpm for 5 min, and the solvent decanted and combined with the methylene chloride:acetone in the Erlenmeyer flask.

6. This solvent mixture is transferred to a round-bottom flask, and reduced in concentration to approximately 20 mL. The resulting solution is dried over approximately 40 g of sodium sulfate for about 30 min.

2.2.2 Derivatization

1. The extracted TBT, DBT, MBT, and the QIS TPT are converted to the corresponding n-pentyl derivatives by adding 2 mL of n-pentylmagnesium bromide to the mixture, and allowing the reaction to proceed at room temperature for a minimum of 15 min.

2. The reaction is quenched by adding 10-20 mL 10 N sulfuric acid to the flask, and mixing until any precipitate has dissolved. The mixture is transferred to a 250-mL separatory funnel, and the phases allowed to separate. The lower aqueous phase is drawn off and discarded.

3. In preparation for column cleanup, the hexane phase is collected and reduced in concentration to approximately 1-2 mL by nitrogen gas evaporation.

2.2.3. Florisil-Silica Gel Column Cleanup

If gel permeation chromatography is required, procedures presented in Section 2.2.4 are followed.

1. A Florisil-silica cleanup column is prepared by dry packing a 22-mm i.d. chromatography column with 16 g PR-grade Florisil, topping with 7 g 1 percent deactivated silica gel and 2 g sodium sulfate. The column is prewet with a minimum amount of hexane.

2. The sample is applied to the top of the column, eluted with 150 mL hexane, and collected at a flow rate of 1-4 mL/min.

3. The eluate is transferred to a Kuderna-Danish apparatus, and reduced in concentration to < 4 mL. The final extract is reduced in volume to approximately 0.5 mL by nitrogen gas evaporation, and 100 µL of the recovery internal standard (RIS)

dipropyldipentyltin (PPT) is added. The sample is transferred to a gas chromatography autosampler vial, and submitted for analysis.

2.2.4 Optional Gel Permeation Cleanup

If sediments contain large quantities of hydrocarbons or other organic matter, gel permeation chromatography (GPC) may be used to cleanup extracts prior to Florisil chromatography. Gel permeation chromatography is proposed as a cleanup step if the extracted lipid weight of the sediment exceeds approximately 5 mg/g.

1. A GPC column is prepared by adding 80 mL swelled Sepahdex LH-20 (swelled overnight in 6:4:3 cyclohexane:methanol:methylene chloride) to a 19-mm i.d. chromatography column.
2. The column is calibrated when made by applying a mixed butyltin standard (n-pentyl derivatives of MBT, DBT, TBT as well as TET) to the column, collecting 1-mL eluate fractions, and analyzing the fractions by GC/FPD. The elution volume for the butyltins is determined from these calibration data.
3. The volume of the hexane sediment extract is reduced to 1-2 mL by nitrogen gas evaporation. The extract is then filtered through a 0.4-μM glass fiber filter and applied to the GPC column.
4. The column is eluted with 6:4:3 cyclohexane:methanol:methylene chloride, and the fraction that contains the butyltin compounds collected.
5. The eluate is transferred to a Kuderna-Danish apparatus and reduced in volume to < 4 mL.
6. The sample is reduced in volume to approximately 0.5 mL by nitrogen gas evaporation, transferred to a gas chromatography autosampler vial, and submitted for Florisil cleanup.

2.2.5 Optional Sulfur Cleanup

Sulfur will be removed from sediment extracts by using activated copper prior to Florisil or GPC column cleanup.

1. Activated copper is prepared no more than 1 hour before sample cleanup by mixing approximately 20 g of copper turnings with 5 mL 6N HCl in a 50-mL beaker. The mixture is stirred until the copper turns pink. The copper is successively washed with 50-mL aliquots of reagent water, methanol, methylene chloride, and hexane.

2. Prior to chromatographic cleanup, approximately 5 g activated copper is added to sample extracts. The solution is shaken gently for 2-3 min. If sulfur is present, the copper will turn black. Small amounts of copper are continually added until the copper remains pink. The extract is decanted from the copper and submitted for chromatographic cleanup.

2.2.6 Gas Chromatography/Flame Photometry (GC/FPD)

1. A Hewlett-Packard 5890 gas chromatograph (GC) is fitted with a 30-m DB-5 capillary column (J&W Scientific, Inc.), using Helium as the carrier gas at 30±2 cm/sec linear velocity. The injection port temperature is set at 250°C. The flame photometric detector (FPD) is fitted with a 610-nm band-pass filter for tin-selective detection. The FPD is operated at 250°C, with an air flow of 40 mL/min and a hydrogen flow of 100 mL/min. During analysis, the GC oven is held at 60°C for 1 min after injection, then temperature programmed to 250°C at 20°C/min, with a final hold time of 5 min. Data from the GC/FPD will be acquired using a chromatography data acquisition system from Beckman Inc.

2. The GC/FPD is calibrated prior to analysis of samples by means of three-point calibration curves. Response factors for each analyte will be calculated using the method of internal standards, with TPT as the internal standard. These calculations will be performed by computer algorithms that are part of the chromatography data acquisition/reduction system.

3. Samples are analyzed and quantified relative to the internal standard TPT. Results are reported in terms of nanograms butyltin per kilogram sediment (ng/Kg).

2.2.7 Gas Chromatography/Mass Spectrometry Confirmation

1. A Hewlett-Packard 5970B quadrupole mass spectrometer is fitted with a 30-m DB-5 capillary column (J&W Scientific, Inc.). The mass spectrometer is operated with a source pressure of 5-7 x 10^{-5} torr. During analysis, the GC oven temperature is held for 1 min at 60°C then programmed to 250°C at 20°C/min, with a 4-min final hold time. The mass spectrometer is operated in

selected ion monitoring mode (SIM). The following ions are scanned at 100 msec/ion:

Compound	Ions			
TPT	121	165	235	277
MBT	119	121	193	319
DBT	119	121	249	319
TBT	119	121	249	305
TET	119	121	235	291

The ions that are underlined are extracted and used for quantitation. The GC/MS is calibrated prior to analysis of samples by using three-point calibration curves. Response factors for each analyte will be calculated using the method of internal standards. These calculations will be performed by computer algorithms that are part of the mass spectrometry data acquisition/reduction system.

2. Samples are analyzed and quantified relative to the internal standard TPT. Results are reported in terms of nanograms butyltin per kilogram sediment (ng/Kg).

2.2.8 Sediment Characterization

Sediments used in this study will be characterized in terms of grain size, total organic carbon content (TOC), and total extractable tin content. The analysis for grain size and TOC will follow general procedures as presented by EPA (Plumb, 1981). Extractable tin will be determined following EPA procedure 7810 (EPA, 1986). Sediment dry weights will be determined for all test sediments.

2.3 PHASE 1: RECONNAISSANCE EXPERIMENTS

The performance of the analytical method will be tested and refined as necessary by performing a series of experiments with sediments collected from the Chesapeake Bay area. The objective of these experiments is to address the potential interferences in the analysis of butyltins due the presence of hydrocarbons (oil) and elemental sulfur. Phase 1 of Table 1 summarizes the reconnaissance experiments to be performed.

TABLE 1. SUMMARY OF TASKS FOR METHOD VALIDATION

Phase 1. Reconnaissance Experiments

Level (ng/kg)	CHB-1	CHB+PBC	CHB+S
Ambient	4	0	0
5,000	8^{a}	8^{a}	4
Blanks	2	2	1

Phase 2. Method Detection Limit, Recovery, and Precision

Level (ng/kg)	CHB-1	CHB-2	DR-1	Blanks
Ambient	-	4^{b}	4^{b}	1
500	4	4	4	1
5,000	-	4	4	1
50,000	4	4	4	1
500,000	4	4	4	1

Phase 3. Field Sample Verification

Level (ng/kg)	HR	PS	GB	BI
Ambient	8^{b,c}	8^{b,c}	8^{b,c}	8^{b,c}
Blanks	2	2	2	2

Phase 4. Storage Experiments

Sample Type	Number of Analyses
Authentic sediment	5 x 2 duplicates
Sediment extracts	5 x 2 duplicates

CHB+PBC=CHB-1 + 10 mg/g crude oil
CHB+S=CHB-1 + 10 mg/g elemental sulfur
CHB-1 = Chesapeake Bay sediment (ca. 10,000 μg/g TOC)
CHB-2 = Chesapeake Bay sediment (ca. 30,000 μg/g TOC)
DR = sediment from Detroit River, MI
HR = sediment from Hampton Roads, VA
PS = sediment to be collected by Envirosphere Co. from Puget Sound, WA and sent to Battelle
GB = sediment from Galveston Bay, TX
BI = sediment from Belle Isle, MI

[a] =4 florisil, 4 GPC+florisil
[b] =grain size, TOC, and Sn by CVAA (hydride)
[c] =method of standard addition, 3 level addition

To evaluate the effect of high levels of hydrocarbons on the performance of the analytical method, Chesapeake Bay sediments will be fortified with butyltins at approximately the 5000-ng/Kg level, and then contaminated with crude oil at a level of approximately 10 mg oil per gram of sediment. The effectiveness of two steps--Florisil and sequential GPC-Florisil--for the cleanup of these heavily contaminated sediments will be evaluated by subjecting contaminated sediment extracts to Florisil column cleanup only, as well as combined GPC-Florisil cleanup. The results from these two cleanup techniques will be contrasted and evaluated in terms of the quality of gas chromatography analysis and the overall recovery of the butyltins of sediment extracts subjected to the two cleanup procedures.

To evaluate the effect of high levels of sulfur on the performance of the analytical method, Chesapeake Bay sediments will be fortified with butyltins at approximately the 5000-ng/Kg level, and contaminated with elemental sulfur at a level of about 10 mg sulfur per gram of sediment. The effectiveness of elemental copper for the removal of sulfur from contaminated sediments will be evaluated. Sulfur-contaminated sediment extracts will be subjected to Florisil column cleanup as well as to pre-column cleanup sulfur removal using activate copper. The results from these two cleanup techniques will be contrasted. If the copper cleanup is unsatisfactory, elemental mercury and tetrabutylammonium sulfite will be evaluated as alternative sulfur removal procedures.

2.4 PHASE 2: METHOD DETECTION LIMIT, RECOVERY, AND PRECISION DETERMINATIONS

To evaluate method performance, a series of experiments, designated as Phase 2 in Table 1, will be conducted to evaluate method performance using three different sediments at four butyltin spike levels. Replicate experiments (four per level) at each spike level for each sediment type will be performed. Based on these data, method performance will be determined. At each level, the recovery of the analytes will be reported. At each spike level, reproducibility of the method will be determined and reported as the percent relative standard deviation of the mean of the replicate determinations.

The method detection limit (MDL) for each butyltin compound will be determined using the recovery data at the lowest spike level (500 ng/Kg). The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The MDL is calculated using the equation:

$$MDL = t \times \sigma$$

where σ is the standard deviation of the results of replicate measurements for an analyte, and t is the student-t statistic for the number of measurements.

2.5 PHASE 3: FIELD SAMPLE VERIFICATION

The analytical method will be tested by analyzing field-collected sediment samples from areas with suspected butyltin contamination. Table 1, Phase 3, lists the sediments to be tested. Each sediment will be analyzed using the proposed method (single-point analysis), and using the method of standard additions to evaluate if matrix effects are systematically biasing the results of single-point analyses. For each sediment type, two four-level standard addition analyses will be performed. The results of the standard addition analyses will be compared to the single-point analyses and will provide verification of the method performance. If a butyltin compound is suspected to be present in a sediment (by virtue of the GC/FPD results), a GC/MS analysis will be performed to provide structural confirmation of the residue.

2.6 PHASE 4: STORAGE EXPERIMENTS

The stability of butyltins in archived sediments and sediment extracts will evaluated over a 4-week period. Sediment subsamples (100 g) will be fortified at the 5000-ng/Kg level and stored in a freezer. At the time of the initial spiking, and at weekly intervals for 4 weeks, the sediments will be extracted and analyzed.

The stability of butyltins in archived sediment extracts will be evaluated. Sediment samples will be fortified at the 5000-ng/Kg level, extracted, derivatized, and stored in a freezer for 4 weeks. At the time of the initial spiking, and at weekly intervals for 4 weeks, the sediment extracts will be analyzed.

The stability of the butyltin analytes in both experiments will be determined by measuring the absolute and relative changes in concentration of the butyltin compounds.

2.7 SEDIMENTS AND SEDIMENT SAMPLING

During the course of this study, three sediments from the Chesapeake Bay, one from the Belle Isle region of the Detroit River, Detroit, Michigan, one from Galveston Harbor, Galveston, Texas, and one from Puget Sound, Washington, will be collected (Table 1).

The Chesapeake Bay sediments to be used in Phase 1 and Phase 2 of this study (CHB-1 and CHB-2) will have two different organic carbon contents. Sediment CHB-1 will have an organic carbon content of about 1 percent, whereas CHB-2 will have an organic carbon content of about 3 percent. Selection of sampling sites in Chesapeake Bay for these two sediments will be based on chemical and physical reconnaissance data from the National Oceanic and Atmospheric Administration (NOAA) National Status and Trends Mussel Watch Program.

In all cases, sediments will be obtained using either a Teflon scoop or a van Veen grab sampler. At each sampling location, the top 2 cm of sediment will be collected and transferred to Teflon sampling jars. Sampling jars will be labeled with a project number, sampling date, location, and the name of the person who collected the sediment. Samples will be shipped on ice and kept frozen until used. Sample collection log sheets containing this information will be stored as a permanent record in the study data log book. Battelle will collect sediments in Chesapeake Bay, Detroit River and Galveston Harbor. Envirosphere will collect sediment from Puget Sound.

3.0 QUALITY ASSURANCE APPROACH

The method development and validation work described in this protocol will be conducted under EPA Good Laboratory Practices (GLP) rules and regulations, and monitored by a Quality Assurance Unit (QAU) experienced in environmental programs. The Battelle program currently meets the requirements for Good Laboratory Practices compliance under the Toxic Substance Control Act (TSCA) and the Federal Insecticide, Fungicide, and Rodenticide (FIFRA) GLPs including environmental and chemical fate studies.

During this program the QAU will monitor and review protocols and standard operating procedures (SOPs) to assure compliance with appropriate regulatory specifications, audit the conduct of the study and the final reports to ensure data integrity and accurate reporting, and manage on-site archival of all raw data generated during the course of this study. QAU record-keeping and management activities such as maintenance of the Master Schedule Sheets and SOPs (both required under EPA GLP) are routine activities.

In the laboratory, all reagents will be logged into a record book. Lot number, purity, and chemical descriptions will be recorded. Standard preparation activities will be logged and the records maintained in separate three-ring binders. All chromatograms generated will be labeled and archived both as hard copy and on magnetic tape.

Access to all chemicals, calibration solutions, and reference materials will be controlled. Chemicals will be stored in locked cabinets, and solutions will be kept in a locked refrigerator. Balances used to weigh standard materials will be calibrated according to standard procedures. All purchase orders related to this task will be retained for reference.

Permanent archival of all raw data generated during this study is mandatory. At the conclusion of the study, these data will be archived indefinitely in Battelle's access-restricted facility according to the requirements of Battelle's GLP program. Test substances used in this study will be archived at Battelle Ocean Sciences.

4.0 REPORTING OF RESULTS

Monthly progress reports will be submitted to the client, describing activities completed, problems encountered and their solutions, and a projection of the next month's work.

At the completion of the study, a Draft Final Report will be submitted to the client. The report will contain, but not be limited to, the following information:

1. Dates on which the study began and ended.
2. Name and address of the testing laboratory.
3. Location where tests were conducted.
4. Name of Study Director and other supervisory personnel.
5. Signatures of senior personnel responsible for the study.
6. A full description of the experimental design and procedures, including the test equipment used, and descriptions of any deviations from protocol and their impact on the study.
7. Identification of the test substances used.
8. Test sediment sampling information and data.
9. Characterization of the test sediments (percent organic carbon, grain size analysis).
10. Results of reconnaissance experiments.
11. Results of MDL, recovery, and precision experiments.
12. Results of the field sample verification experiments.
13. Results of the storage experiments.
14. Detailed, step-by-step description of the analytical method.
15. Mathematical equations used in evaluation of the data.
16. Summary, conclusions, and recommendations of the study.
17. List of personnel involved with the study.
18. Quality Assurance Statement.
19. GLP Compliance Statement.

20. Location where all data, reports, and chemicals are archived.

The Draft Final Report will be reviewed by the client, and the comments returned to the Study Director. Based on the review comments of the Draft Final Report, a Final Report will be produced and submitted to the client.

5.0 PROJECT SCHEDULE

Table 2 presents a milestone and time schedule for this study. The time frame for this study is 4 months from initiation of the study to submission of the Draft Final Report.

6.0 PROGRAM ORGANIZATION

Oversight of this study will be provided by Mr. William Steinhauer, Associate Section Manager of Battelle's Marine Chemistry and Geochemistry Section. Dr. Allen Uhler will be the Study Director, overseeing day-to-day activities in the laboratory and reporting project progress directly to Mr. Steinhauer. Laboratory work, which includes standard and sample preparation, analyses, log book maintenance, and other elements of laboratory quality control, will be the responsibility of appointed laboratory technicians. Ms. Debra McGrath of the Quality Assurance Unit will monitor the study and report findings to the Study Director, Laboratory Management, and Ms. Patricia D. Royal, Manager of the Quality Assurance Unit.

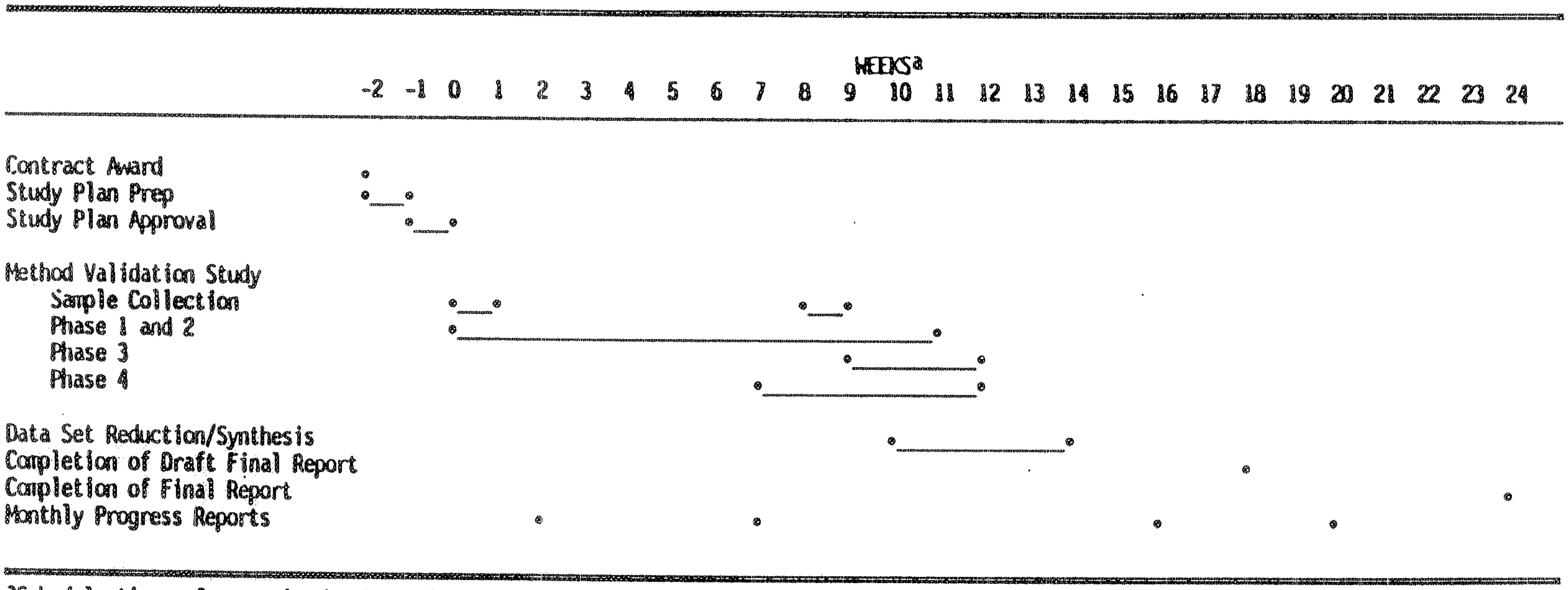
7.0 REFERENCES

Plumb, R.H. 1981. Environmental Protection Agency/Corps of Engineers technical committee on criteria for dredged and fill material. Procedures for handling and chemical analysis of sediment and water

Rice, C., F. Espourteille, and R.J. Huggett. 1987. A method for the analysis of tributyltin in estuarine sediments and oyster tissue, Crassostrea virginica. App. Organomet. Chem. 1:541-544.

United States Environmental Protection Agency. 1986. Test methods for evaluating solid wastes. Office of Solid Waste and Emergency Response, Washington, DC.

TABLE 2. MILESTONES AND SCHEDULE FOR SEDIMENT METHOD VALIDATION STUDY



[a] Schedule time reference is the date of study plan approval by the Consortium for Tributyltin Manufacturers.

Date: July 21, 1988

Subject: Amendment 1 to "Measurement of Butyltin Species in Sediments by n-Pentyl Derivatization with Gas Chromatography/Flame Photometric Detection (GC/FPD) and Optional Confirmation by Gas Chromatography/Mass Spectrometry (GC/MS)"

ORIGINAL METHOD

See Analytical Method, section 2.2, in Protocol. The subsections which are altered are:

2.2.1 Extraction
2.2.2 Derivatization
2.2.3 Florisil-Silica Gel Column Cleanup
2.2.4 Optional Gel Permeation Cleanup

NEW METHOD (CHANGES)

2.2.1 Extraction

1. Approximately 100 g of sediment is separated from any overlying water by decantation. The sediment is transferred to a tared Teflon jar and weighed to the nearest 0.01 g by using a top loading balance. The sample is centrifuged for 5 min. at 1500 rpm, any resulting water decanted, and the jar weighed to determine the final sediment weight.

2. Add the appropriate amount of quantitation internal standard (QIS) tripropyltin chloride (TPT) to the sediment sample. Next, add 25 mL of DI water, and enough HBr to make the solution 10 % in HBr (approximately 5 mL of 48% HBr). The bottle is capped, and agitated on a shaker table for 30 min.

3. Add 60 mL toluene:0.05% tropolone to the slurry and extract by shaking on a shaker table for 30 min.

4. After this period, the bottle is removed from the shaker table, and centrifuged at 1500 rpm for 5 min. to separate the sediment from the aqueous and organic phases. The aqueous and organic phases are poured

into a separatory funnel, and separated. If phase separation is complicated by emulsion formation, then add 10 to 20 mL hexane, after removing as much organic phase extract as possible to an Erlenmeyer flask, shake, centrifuge, and separate phases again. The aqueous phase (and any emulsion) is poured back into the sediment, and the organic phase is saved in an Erlenmeyer flask.

5. The extraction is repeated with 60 mL toluene:0.05% tropolone by shaking on a shaker table for 30 min. The sample is centrifuged, and the phases separated. The two organic extract portions, and any residual emulsion, are combined, and dried over approximately 40 g sodium sulfate for about 30 min.

6. The extract is transferred to a round-bottom flask, and reduced in concentration to approximately 10 mL by rotary evaporation. The extract is then solvent exchanged by adding approximately 30 mL of hexane and concentrating to between 10 and 15 mL by rotary evaporation.

2.2.2 Derivatization

1. The extracted TBT, DBT, MBT, and the QIS TPT are converted to the corresponding n-pentyl derivatives by adding 5 mL n-pentylmagnesium bromide solution (1.9 M in ether) to the mixture, and allowing the reaction to proceed at room temperature for a minimum of 15 min.

2. The reaction is quenched by slowly adding 50 mL DI water, and 5 mL of 10 N sulfuric acid, and swirling the flask until any precipitate has dissolved. The mixture is then transferred to a 250 mL separatory funnel, and the phases allowed to separate. The lower aqueous phase is drawn off and discarded.

3. In preparation for column cleanup, the hexane phase is collected and reduced in concentration to approximately 1 to 2 mL by evaporation under a stream of nitrogen.

2.2.3 Florisil-Silica Gel Column Cleanup

The only change is the use of 100 mL (rather than 150 mL) of eluant.

2.2.4 Optional Gel Permeation Cleanup

Gel Permeation Cleanup (GPC) is not performed on a routine basis. GPC is only done on samples which need this additional cleanup, and is performed following Florisil-Silica Gel cleanup.

A. Column Preparation

1. Solvent rinse a 19-mm i.d. chromatography column once with acetone, twice with methylene chloride, and once with hexane.
2. Add 10 mL 6:4:3 cyclohexane:methanol:methylene chloride and a 5 to 10 mm glass-wool plug. Tap the plug with a glass rod to remove any bubbles.
3. Add about 1 mL of sand to the column, and tap the column gently so that the sand forms a smooth layer on top of the glass wool.
4. Pour about 80 mL swelled Sephadex LH-20 (swelled overnight in 6:4:3 cyclohexane:methanol:methylene chloride) into the column, until the gel fills the column to about 1/4 of the top, rounded reservoir.
5. Allow the Sephadex gel to settle for 10 minutes. Open the stopcock and elute 80 mL of solvent to ensure firm packing. Add more solvent as needed. Leave approximately 30 mL of solvent in the column reservoir, cover with aluminum foil and allow the packing to settle overnight.
6. Elute 10 mL of solvent. Remove the excess Sephadex gel from the top with a pipet until the height of the gel in the column is 26.5 cm.
7. Add about 1 mL of sand onto the gel so that it forms an even layer on top.
8. Examine the packing for air bubbles. If bubbles are present, elute with about 250 mL of warm solvent. If bubbles persist, repack the column.

B. Column Calibration with Azulene/Perylene

1. Place a 100 mL graduated cylinder beneath the column.
2. Using a pipet, carefully remove any excess solvent from the top of the column gel packing.

3. With a pipet carefully apply 2 mL of the azulene/perylene calibration solution down the column wall so as not to disturb the packing.

4. Open the stopcock and drain to the top of the packing.

5. Add about 0.5 mL 6:4:3 cyclohexane:methanol:methylene chloride solvent to the top of the column. Drain to the packing top. Repeat with an additional 0.5 mL solvent.

6. Add 100 mL solvent, and open the stopcock.

7. Elute the column until all of the perylene has emerged. Azulene will elute first and then perylene, both of which are clearly visible as colored bands on the column. Record the volumes at which the azulene and perylene begin and finish eluting.

8. If the azulene emerges in the 50-65 mL range, and the perylene elutes in the 60-80 mL range without distinct tailing on the packing, the column passes the azulene/perylene calibration.

9. Discard the eluate. Flush the column by eluting 50 mL of the 6:4:3 solvent through the column. Discard the eluate.

C. Column Conditioning

1. Add 1 mL 6:4:3 solvent to 1 g Crisco vegetable oil in a 4 mL vial. Carefully add the oil onto the column and drain to the top of the gel packing.

2. Rinse the vial with 0.5 mL 6:4:3 solvent, add to the column, and drain to the top of the gel packing. Repeat with an additional 0.5 ml of 6:4:3 solvent.

3. Elute 150 mL 6:4:3 solvent through the column.

4. The column is now ready for a sample.

D. Sample Loading and Cleanup Procedure

1. Carefully add the sediment extract sample, which has been reduced in volume to between 1 and 2 mL by concentrating it under a stream of nitrogen, to the calibrated and conditioned column. Drain to the top

of the column packing.

2. Elute the column with 100 mL 6:4:3 cyclohexane:methanol:methylene chloride. This eluate includes the butyltin compounds

3. Transfer the eluate to a Kuderna-Danish apparatus and concentrated to < 4 mL. Reduce the sample to a final volume of approximately 0.5 mL by nitrogen gas evaporation. The recovery internal standard (RIS) dipropyldipentyltin (DPT) is added, and the sample is transferred to a gas chromatography (GC) autosampler vial, and submitted for GC analysis.

IMPACT ON STUDY

The procedural changes described in this amendment were made after carefully evaluating them, the originally proposed procedures, and other possible procedures (e.g. extraction solvents). These new procedures will improve the recovery of monobutyltin and the cleanup of sediment samples for the analysis of butyltins, and will thus provide higher quality data for this study than if the original methods were used.

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Allen D. Uhler, Ph.D Date
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Paul D. Boehm, Ph.D Date
Section Manager
Battelle Ocean Sciences

Project Number N-0519-6101

Date: 7 September, 1987

From: AD Uhler

Deviation to Protocol

Subject: Dipropyltin as Quantitation Internal Standard

In the original protocol, tripropyltin (TFT) was designated as the quantitation internal standard. Our experience has shown that TPT often coelutes with interfering compounds, making accurate measurements difficult when based on this compound. We have found that dipropyltin (DPT) is a much more reliable internal standard upon which to base measurements.

Therefore, dipropyltin (DPT) is recommended for use as the quantitation internal standard (QIS). Tripropyltin should still be added to samples and used as the recovery internal standard (RIS), if desired. Note that TPT can be used effectively as the QIS if there is no interfering compounds in the same region of the chromatogram. Essentially, the analyst has 2 internal standards to choose from for quantitation purposes. It is important that the analyst identify which internal standard is used for quantitation.

Battelle Project Number N-0519-6100

: Number N-0519-6101

: 7 September, 1987

. AD Uhler ADUhler

tion to Protocol

ct: Total Organic Carbon (TOC) and Total extractable tin analyses

e analyses of test sediments for TOC will be carried out by the contract oratory Global Geochemistry Corporation, Canoga Park, CA. Sample analyses to be coordinated by Ms. Mo Rice of Global Geochemistry Corp. [illegible]

he instrumental analyses of test sediments for total extractable tin will be rried out by Dr. Eric Crecelius of Battelle, ~~Squeim~~ WA. Sediment preparation will be carried out at Battelle, Duxbury, MA.

e TOC determinations are being carried out by Global Geochemistry Corp. because Battelle, Duxbury MA does not have the specialized equipment to erform the assays.

Project Number N-0519-6101

Date: 21 September, 1987

From: AD Uhler

Deviation to Protocol

Subject: Sediment Spiking Scheme

Phase 2 of the protocol calls for spiking sediments at the 500, 5000, 50,000 and 500,000 ng/Kg levels. Because ambient levels of butyltins are in the 2000-5000 ng/Kg range, the fortification levels of 500, and 5000 ng/Kg were omitted from the project, since these levels are too close in concentration to the ambient levels, and little meaningful information would be derived from the results.

Project Number N-0519-6101

Date: 10 October, 1987

From: AD Uhler

Deviation to Protocol

Subject: Method for Freshwater Sediment Analyses

Recovery of butyltins from the freshwater sediment from the Detroit River (DYC-1) were very good for TBT (>90 percent). However, recovery of DBT and MBT were < 50 percent. Thus, the method presented in the Final Report will not be used for freshwater sediment. Further work on the analysis of butyltins in freshwater sediments is being carried out, and the data and the modified method will be reported in a supplemental FInal Report.

Deviation to Protocol

PROJECT NUMBER: N-0519-6101
DATE: January 4, 1989
FROM: AD Uhler

SUBJECT: Storage Experiments Using Sediments with Ambient Levels of Butyltins

1.0 OBJECTIVE

Storage experiments using sediments and sediment extracts spiked with butyltin compounds have shown that TBT degrades during storage. Dr. Peter Seligman, of the US Navy, at a recent conference (OECD Conference on Monitoring of Butyltin, Paris, France Nov 28-Dec 1, 1988) explained that he too has observed degradation of spiked butyltins in sediment. However, he has also observed that incurred butyltins did not degrade upon storage. The implication is that spiked butyltins probably degraded due to reaction with active sites in the sediment matrix. Incurred butyltins, however, have already equilibrated with passive sites and are relatively nonreactive. Therefore, storage experiments using sediments with incurred butyltin contamination represents a more realistic storage scenario.

A storage experiment using sediments and sediment extracts will be carried out over a 1 month period, with duplicate analyses performed at weekly intervals. A sediment with incurred butyltin residues will be used in order to more accurately simulate actual storage conditions.

2.0 PROCEDURE

A marine sediment from the Hampton Roads area of Virginia (designated HR) will be used for the storage experiment. This sediment has been selected because it contains detectable amounts of TBT, DBT, MBT and TTBT (see Table 5, Draft Final Report).

2.1 PREPARATION OF SEDIMENT

1. Remove sediment HR from freezer. Thaw at room temperature.
2. Transfer approximately 2000 g of sediment to a large nalgene vessel. Composite the sediment by shaking and/or tumbling for 4 hours.
3. Remove approximately 100 g aliquots of sediment and transfer to a clean 250 mL pre-weighed teflon jar. Prepare 18 jars in this fashion.

4. Centrifuge the water off the sediments at 2500 rpm for 10 min. Decant the water off the sediments. Determine the weight of the sediment in the jars by difference.

2.2 FORTIFICATION OF THE SEDIMENTS WITH RIS TPT

Each sediment will be fortified with the recovery internal standard (RIS) tripropyltin chloride (TPT) at the beginning of the experiment.

1. Obtain a spiking solution that contains approximately 1 µg/mL TPT.
2. Add 1 mL of the spiking solution to each sample jar. Thus, approximately 1 µg of TPT is added to each sample.
3. Mix the sediment thoroughly on a shaker table for 10 min.

2.2.1 Preparation of Sediment Extract Storage Samples

1. Extract and derivatize 10 sediment samples by following the procedures for extraction and derivitization of sediment samples presented in the Draft Final Report "Measurement of Butyltin Species in Sediments by n-Pentyl Derivitization with Gas Chromatography/Flame Photometric Detection (GC/FPD) and Optional Confirmation by Gas Chromatography/Mass Spectrometry (GC/MS).
2. Store the samples in an approximately -20°C freezer at a volume of approximately 40 mL until ready for analysis.

2.3 STORAGE AND EXTRACT EXPERIMENT CONDITIONS

Ten of the prepared sediment sample extracts will be stored in an approximately -20°C freezer. At time=0 and at weekly (7-day) intervals, duplicate extract samples will be removed from the freezer, the quantitation internal standard dipropyldipentyltin (DPT) added to them, and the extracts analyzed by GC/FPD.

Eight of the sediments will be stored in an approximately -20°C freezer as the bulk sediments. At t=7 days and at weekly (7-day) intervals, duplicate sediments will be removed from the freezer, extracted, derivatized, the QIS DPT added, and the extracts analyzed by GC/FPD. Extraction, derivitization and analysis will be accomplished by following the procedures for extraction and derivitization of sediment samples presented in the Draft Final Report "Measurement of Butyltin Species in Sediments by n-Pentyl Derivitization with Gas Chromatography/Flame Photometric Detection (GC/FPD) and Optional Confirmation by Gas Chromatography/Mass Spectrometry (GC/MS). At weekly (7-day) intervals, duplicate extract samples will be removed from the freezer, the quantitation internal standard dipropyldipentyltin (DPT) added to them, and the extracts analyzed by GC/FPD.

2.3.1 Analysis

In all cases, prior to analysis, approximately 1 µg of DPT will be added to the extracts prior to analysis. The extracts should be concentrated to approximately 1 mL and submitted for GC/FPD analysis. The concentration of the butyltin analytes will be computed versus the QIS DPT.

3.0 DOCUMENTATION

Laboratory gravimetric measurements, amounts of internal standards added, dates of analysis, etc., should be recorded in the Laboratory Record Book and/or on the forms supplied for this project. All entries should be initialed and dated by the person entering the information. The sample IDs, and dates of analyses should be recorded on the attached sheet and stored with the data books.

DEVIATION TO PROTOCOL

Project Number: N-0519-6101

Date: February 2, 1989 2-2-89 AW

Subject: Recovery of Butyltin Species from Fresh Water River Sediment and Fresh Water Lake Sediment

INTRODUCTION

Initial fresh water sediment work (spike and recovery experiments with sediments from the Detroit River, MI) showed poor recovery of butyltins from the matrix. Additional fresh water sediments are to be collected from a fresh water lake and a fresh water river. Spike and recovery experiments will be conducted, and the recovery of butyltin species assessed.

FRESH WATER SEDIMENT SOURCES

Fresh water river sediments will be collected from the Taunton River, MA. Fresh water lake sediments will be collected from the pristine Priest Pond, in Duxbury, MA.

OBJECTIVES

The two fresh water sediments will be characterized for TOC and grain size. The sediments will not be characterized for total extractable tin, since all work to date, as well as literature evidence, shows that the total extractable tin is at least 1-2 orders of magnitude higher in concentration than the total organotin concentrations.

The sediments will initially be screened for ambient levels of butyltin concentration. Depending on the results, the sediments will be fortified with approximately 5 times the background level of TBT.

Nominally, the sediments will be fortified at approximately the 5 µg/Kg, 50 µg/Kg and 500 µg/Kg level with butyltin species, extracted and analyzed by GC/FPD. The objective of this experiment is to evaluate recoveries of the butyltin species from the fresh water sediment using the methods developed during the course of the methods validation study.

The following schedule describes spike level and the number of analyses to be conducted at each level:

Butyltin Spike Level (µg/Kg)	Replicates Performed per sediment
Ambient	2 (no spiking)
1-5	4
25-50	4
300-500	4

EXPERIMENTAL

Extraction and analysis of samples prepared for this study will be carried out following the methods described in the Draft Final Report "Measurement of Butyltin Species in Sediments by n-Pentyl Derivatization with Gas Chromatography/ Flame Photometric Detection (GC/FPD) and Optional Confirmation by Gas Chromatography/Mass Spectrometry (GC/MS)".

These experiments will be carried out using 100 g (wet weight) sediment samples. Weight out samples and record weights. Sediments will be fortified before extraction with TPT and the butyltin species MBT, DBT, TBT and TTBT. After extraction and prior to GC/FPD analysis, the sediments will be fortified with the quantitation internal standard DPT. The following spiking scheme should be followed:

Spike Level (µg/Kg)	------------ added to sample ---------------		
	TPT Std	BuxSn Std	DPT Std
1 - 5	0.5 µg	0.1 - 0.5 µg	0.5 µg
25 - 50	5 µg	2.5 - 5 µg	5 µg
300 - 500	50 µg	30 - 50 µg	50 µg

The following pre-injection volumes (final extract volumes) should be used for the corresponding spike levels:

Spike Level (µg/Kg)	Final Volume
5	1 mL
50	10 mL
500	1 mL then dilute ca. 50 times prior to analysis

APPENDIX E

PSDDA
SCREENING LEVEL (SL) AND
MAXIMUM LEVEL (ML) GUIDELINE
CHEMISTRY VALVES

TABLE A.7 (con.)

Chemical	EPTA (1988) SL	EPTA (1988) ML	CURRENT (1989) SL	CURRENT (1989) ML
Pesticides				
Total DDT	6.9	69	6.9	69
Aldrin 5/	10		10	
Chlordane 5/	10		10	
Dieldrin 5/	10		10	
Heptachlor 5/	10		10	
Lindane 5/	10		10	
Total PCB's	130	2,500	130	2,500

5/No ML is established for these compounds.

- A potential for remaining in a toxic form for a long time in the environment.

- A potential for entering the food web.

The list was pared down from the 129 priority pollutants and 30+ hazardous substances, plus the many anthropogenic chemicals found by NOAA in a study of Commencement Bay sediments.

In addition to the standard chemicals of concern, there is a limited list of chemicals of concern that need to be measured for dredging projects located near specific pollution sources. These chemicals include:

- Guaiacols.

- Chlorinated guaiacols.

- Chromium.

- Tri-, tetra-, and pentachlorobutadienes.

- Butyltins

Butyltin testing is indicated in areas near boat and vessel maintenance and construction. (See also chapter 5.c.(7) of draft Phase II MPR.) An interim SL of 30 ppb has been established for tributyltin (TBT).

Chromium appears to derive largely from the natural erosion of crustal rocks into Puget Sound, but localized sources of chromium also exist (e.g., plating industries and some chemical manufacturing facilities).