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PREDICTING THE BEHAVIOR OF ORIMULSION SPILLED ON WATER

VOLUME II

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FINAL REPORT
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NOTICE

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16. Abstract (MAXIMUM 200 WORDS)

Orimulsion is a fuel that is a bitumen-in-water emulsion made of approximately ~70% natural bitumen and ~30% fresh water. Orimulsion is of great interest to electric power utilities because of its competitive cost and pricing structure. Previous work on Orimulsion has determined it may largely float, remain suspended, or settle depending on the spill conditions. In this study, the mechanisms at work and the relative importance of different spill conditions (e.g., salinity, temperature, energy, Orimulsion concentration, particulate load, and particulate type) are investigated by (1) forty-seven benchtop scale (3L) experiments conducted and evaluated using design of experiment principles and (2) five flume scale (4000L) experiments. The insight gained from these laboratory experiments is interpreted in light of the limitations inherent in laboratory spills into confined vessels (e.g., vessel wall effects and absence of dilution) in order to predict the gross behavior that might be anticipated if Orimulsion were spilled in a 'real world,' open water setting. The most important environmental parameters identified, probably listed in order of their descending importance, were determined to be the receiving water salinity, receiving water energy, and presence/absence of suspended mineral matter. Interactions between these parameters determine whether most bitumen will ultimately float on or near the surface, remain suspended within the water column, or settle through the water column. The gross behavior of Orimulsion under different combinations of these parameters is predicted. These predictions provide the best available guidance to contingency planners and spill responders.

Appendices I through VI are included in Volume II of this report, and are available from the USCG Research and Development Center upon request.

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Orimulsion, oil spill, bitumen, PAH, petroleum, hydrocarbons, emulsion, design of experiment, flume test

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APPENDIX I

Detailed Workplan

Predicting the Behavior of Orimulsion Spilled on Water

EXECUTIVE SUMMARY

Orimulsion, an alternative fuel for power generation, is currently being used in countries such as Canada, Italy, Denmark, Japan and China and has been tested in the U.S. in Florida and Illinois. To each of these locations, Orimulsion is being shipped in ocean going tankers. In order to assess the required countermeasures in case of a spill, a thorough understanding of its behavior when released in water is required." Bench top and flume tank studies to date have demonstrated the complexities surrounding its behavior in the event of a release. For example, previous studies have shown that receiving water salinity will markedly effect Orimulsion's behavior. However, the impact and interactions of potentially important independent variables other than salinity, e.g., water temperature, Orimulsion concentration, particulate load, particulate type and turbulence of the water are poorly known. As a result, the need exists to rigorously evaluate the behavior of Orimulsion under a wide range of environmental conditions. This workplan describes a joint study devised by Battelle Memorial Institute, Bitor America Corp. Bitúmenes Orinoco, S.A., Intevep S.A., United States Coast Guard, and National Oceanographic and Atmospheric Administration and co-funded by Bitor America Corp. and the United States Coast Guard Research and Development Center.

The workplan consists of two principal phases – a benchtop study phase and a meso-scale flume tank study phase. The benchtop phase utilizes design of experiment (DOE) principles in order to reduce the number of experiments necessary to achieve a statistically valid result. These experiments will be conducted in 4L glass vessels. Initially, the benchtop experiments will include a 12-run Plackett-Burman screening design phase in which the gross behavior of Orimulsion will be evaluated under a wide range of environmental conditions represented by the six independent variables of interest (salinity, temperature, Orimulsion concentration, particulate loading, particulate type, and turbulence). The screening design will determine if any of the six independent variables has no impact on Orimulsion's behavior, as measured by particle growth and its 'compartmentalization' (i.e., does it float, sink, remain suspended, dissolve, or stick to walls) after 24 hours. Four high Orimulsion concentration (50,000 mg/L) experiments will also be performed during this phase. Those variables that influence Orimulsion's behavior will subsequently be evaluated and quantified in a 3-level factorial, face-centered cube design phase that will include up to 47 additional experiments. The objectives of the 3-level design are to (1) quantify the influence of the individual independent variables and interaction between independent variables on Orimulsion's behavior and (2) help focus the conditions under which the meso-scale flume tank studies will be conducted in the second phase of the study.

In the meso-scale flume tank study phase, a limited number of experiments will be conducted in Battelle's 4000L flume tank under environmental conditions identified as critical in the benchtop phase. The results of the flume studies will be used to ground truth the benchtop studies under conditions that more closely mimic open water conditions. Prior to the Orimulsion release experiments, an attempt will be made to quantify the turbulent diffusive energy within the flume tank under varying wind speed, wave height, and wave frequency conditions. This information is necessary in order to project the results from the flume experiments to open water conditions. Realizing the shortcomings of any laboratory experiments in mimicking nature, the ultimate objectives of this study are to improve the understanding of Orimulsion's behavior when spilled on water and to provide practical guidelines for responders in the event of an Orimulsion spill under a variety of conditions.

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1. INTRODUCTION

In the event of an oil spill at sea, most oils will float and rapidly begin to undergo fairly predictable modifications to their physical and chemical properties collectively known as weathering. Most oil spill contingency measures currently available have been developed for floating oils and their respective emulsions. Recently, however, there is increased interest in predicting the behavior of a relatively new, petroleum-derived fuel known as Orimulsion.

Orimulsion is a fuel that is an emulsion made of approximately 73% natural bitumen and 27% fresh water that is of great interest to electric power utilities because of its competitive cost and pricing structure. Orimulsion is in commercial use in other countries (e.g., Denmark, Canada, Japan, Italy, China, Lithuania, Barbados) but is not in use in the United States. At the present time, Florida Power and Light Co. has proposed to transport Orimulsion through Tampa Bay for their Manatee plant. The permit for that operation is being reviewed by the Governor of Florida and his Cabinet. If approved, FPL expects to bring Orimulsion into Tampa Bay in two years. In addition to FPL there are thought to be several other East Coast oil-fired power plants considering conversion to Orimulsion. As the potential for transport of Orimulsion in U.S. water is eminent, the need exists to better understand the fate of Orimulsion if it were to be accidentally spilled.

2. SCOPE OF STUDY

Orimulsion is classified as a Group V product along with other heavy oils called low API oils (LAPIO). Various benchtop scale studies of Orimulsion and other LAPIO fuels, in particular fuel oil #6, have been performed (Brown et al., 1995; Febres et al., 1995; Potter, 1995). Battelle's 1997 meso-scale (i.e., flume tank) study of LAPIO fuels included Orimulsion (Ostazeski et al., 1997a). During this study 14.2 L of Orimulsion was released into a 4000 L flume tank (3542 mg Orimulsion/L water) containing, on separate occasions, freshwater (0 ‰) and salt water (32 ‰). It was determined that the behavior of Orimulsion 100 when spilled in water is very different from other Group V products and as a result, it presents a variety of very challenging response scenarios.

The receiving water salinity was determined to be a critical factor effecting Orimulsion's behavior. In the fresh water flume test, the Orimulsion was observed to disperse in a black cloud of fine droplets that remained in suspension throughout the 120-hour experiment. In the sea water flume test, Orimulsion initially dispersed throughout the water column but within 15 minutes, a heavy viscous product composed of coalesced bitumen particles began to float on the surface. The surface slick continued to 'grow' by coalescence throughout the 120-hour experiment. The complex interaction of bitumen particle buoyancy, turbulence, and reduced effectiveness and concentration of the polyethoxylated nonylphenol surfactant in sea water was believed to allow the bitumen particles to either (1) coalesce and rise to the surface forming the viscous surface slick, (2) rise to the surface and then coalesce, or (3) a combination of these processes.

At this point, the behavior of Orimulsion in salinities representative of brackish estuaries is uncertain. Similarly, the effect that other potentially important variables, e.g., water temperature, Orimulsion concentration, particulate load, particulate type, and turbulence of the water (i.e., energetics), have on Orimulsion's behavior are not well known. Therefore in the event of a release, Orimulsion could be found to predominantly float, or sink, or remain suspended in the water column. The need to predict the fate of Orimulsion under a variety of environmental conditions is of critical importance for containment, recovery and spill modeling.

This workplan outlines Battelle's proposed approach to gaining an understanding of the important parameters that affect the behavior of Orimulsion when spilled on water. The objective of the study is to investigate the weathering characteristics of Orimulsion in water, particularly as it relates to the ultimate fate of spilled Orimulsion. The project was proposed to consist of three phases, namely;

- ☐ Phase I - Design of Behavior Characterization Experiments
- ☐ Phase II - Evaluation of Factors Effecting Orimulsion's Fate in Water
    - Phase IIA – design and conduct an appropriate number of benchtop experiments to evaluate the numerous independent variables effecting Orimulsion's fate based on design of experiment (DOE) principles
    - Phase IIB – evaluate bench-top data using DOE principles to identify the most significant factors effecting the fate of Orimulsion.
    - Phase IIC – further investigate and quantify flume tank energetics so that Phase III can be conducted under more representative wave and wind conditions (than previous flume experiments).
- ☐ Phase III – Perform meso-scale flume experiments that focus on conditions which have the greatest effect on Orimulsion's fate in water (as based on the results of Phase II) and prepare a detailed comprehensive report of the entire study.

This report comprises the deliverable for Phase I of this study. The objective of this phase is to propose a detailed course of study for Phases II and III. Upon review and concurrence of this course of study by both the U.S. Coast Guard (USCG) and BITOR America Corp. (BITOR), Phase II will be initiated.

3. PHASE II - EVALUATION OF FACTORS EFFECTING THE FATE OF ORIMULSION

3.1 Introduction to Experimental Design

The independent variables identified as potentially impacting the fate of Orimulsion spilled in water were discussed and agreed upon at a meeting held at our Duxbury facility on Oct. 31, 1997 which was attended by BITOR, NOAA, and USCG representatives. These variables and their ranges of interest included:

Independent Variables	Range	Selected Values of Interest
water salinity	0-35 ppt	0, 17, 35 ppt
water temperature	0-30 °C	3, 15, 25 °C
Orimulsion conc.	1000-17000 mg/L	1000-16000 mg/L; 50,000 mg/L[a]
particulate loading	none to heavy	none, 20-120 mg/L
particulate type	calcite/kaolinite	calcite/kaolinite
water energetics	calm to heavy seas	static to dynamic

With six variables and 19 degrees of freedom an *ad hoc* approach (i.e., changing only one variable at a time while holding the others constant) would require 648 separate experiments. The costs and interpretation of such an approach would be impractical. Furthermore, this 'one variable at a time approach' ignores the interaction(s) that may exist among the individual variables. Therefore, an alternative approach was discussed, namely design of experiment.

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[a] It was recently suggested to include at least a couple of benchtop experiments with a very high concentration.

Design of experiment (DOE) is a discipline that incorporates statistics into the process of experimentation. DOE principles can be used to systematically vary a number of independent variables in order to evaluate their effect on dependent variables (Hicks, 1982). In the case of Orimulsion behavior in water, DOE can be used to establish the minimum number of bench-top experiments that can be used to evaluate the independent variables of interest in a statistically valid manner. DOE will also enable one to determine the existence of, and even quantify, interaction between variables.

3.2 Evaluation of Variables

It is important to distinguish between independent and dependent variables. Independent variables are those parameters that will be deliberately controlled while dependent variables are those parameters that are to be measured. In preparation for this study there were six independent variables identified as having a role in bitumen particle growth (see Section 3.1). It is appropriate to ask at this point, are these independent variables appropriate for the purposes of this study? In the Section 3.2.1, data and observations from Battelle's previous experiments on Orimulsion and those by other researchers are reviewed with the goal being able to justify including all of the variables selected for study.

3.2.1 Independent Variables

3.2.1.1 Water Salinity and Temperature

Most oceanic waters occur over a relatively narrow range of salinity (33-37 ppt). However, the salinity of coastal waters can vary widely due the competing influences of evaporation, precipitation and freshwater river discharge. Therefore, consideration of widely varying salinity (0-35 ppt) is certainly justified.

Temperature variation due to unequal heating with latitude and seasonality produces a significant range of surface water temperatures. The ocean is warmest (25-30°C) in the tropical and subtropical regions and coldest (down to –1.7°C) near the poles. Given the latitude variation of the continental U.S., the potential for Orimulsion shipping into continental U.S. waters limits the temperature range to between approximately 10 and 25°C. Therefore, our plan to consider temperature over the range of 4°C to 25°C is more than adequate.

The most important influence of salinity and temperature on Orimulsion's behavior is probably related to their effect on density. It is well known that the density of seawater varies largely with salinity and is further impacted by temperature (see Knudson's Hydrographic tables, e.g., in Riley and Chester, 1971). Direct comparison of Orimulsion's measured density at different temperatures (e.g., 1.0176 at 0°C to 1.0048 at 25°C; Ostazeski et al., 1998) to a seawater density table reveal that bitumen particles are more dense than seawater with salinity < 20 ppt between 0°C and 20°C, and less dense than seawater with salinity > 25 ppt between 25°C and 30°C. Thus considering no other variables, this relationship would indicate that bitumen should sink when salinity < 20 ppt (between 0-20°C) and < 25 ppt (between 25-30°C). Oppositely, it should float when salinity > 20 ppt (between 0-20°C) and > 25 ppt (between 25-30°C).

This float/sink relationship was confirmed in the static vessel experiments by Febres et al. (1995) in which bitumen particles sank and floated when salinity was below and above 20 ppt, respectively. However, similar static vessel tests conducted by Brown et al. (1995) at 24.3°C showed that after an initial period of stirring the bitumen floated in all instances where the salinity exceeded 10 ppt. Thus, there is still some discrepancy in what to expect under different salinity conditions and water salinity and temperature are certainly important variables worthy of further study herein.

An important effect of salinity may lie in the influence that dissolved salts have on the effectiveness of the surfactant. This influence is evidenced by the coalescence of bitumen in seawater and lack of coalescence in freshwater (Ostazeski et al., 1997a). It can be envisioned that the dielectric potential of water varies with salinity and that charged ions in the water (i.e., salts) may reduce the effectiveness of the surfactant bound to bitumen particles.

3.2.1.2 Orimulsion Concentration

In addition to the float/sink behavior of Orimulsion, there is the issue of coalescence of bitumen particles. Among other factors, coalescence must be related to the effectiveness of the surfactant after being spilled. Orimulsion originally contains approximately 30% water (Jokury et al., 1995; Febres et al., 1995). When it is spilt into water the bitumen particles readily disperse, in other words, the water content increases drastically. A substantial proportion of surfactant is thought disassociate from the bitumen particles into the water (Potter, 1995). This process would seem to promote coalescence of bitumen particles if they collide. Theoretically speaking, the collision frequency of bitumen particles should be a function of (1) bitumen concentration, (2) energetics in the system, and (3) the buoyancy forces resulting from the salinity-temperature driven density differences between the water and the bitumen (as described above). Therefore, our experiments must include each of these factors.[b]

Experiments have shown that when the Orimulsion concentration in water is high (>20,000 mg/L) it retains the properties of the emulsion (Febres et al., 1995). One can assume that under such conditions the surfactant concentration in the water and on the bitumen particle surfaces is high enough to prevent coalescence of bitumen particles. However, if the concentration is low (<10,000 mg/L) the Orimulsion looses the properties of an emulsion and bitumen particles become widely dispersed (Febres et al., 1995). Under these conditions the surfactant concentration in the water is too low to prevent coalescence of any colliding bitumen particles (i.e., the critical micelle concentration is not exceeded). Theoretically speaking, if the concentration of Orimulsion spilled is very low (<1000 mg/L) the extremely low concentration of bitumen particles in the water may preclude coalescence due to the scarcity of collisions between particles.

As noted above, collision frequency is undoubtedly also related to the turbulent energy of the system. (This is discussed further below.) However, if energy alone were responsible for collisions and the resulting coalescence of bitumen particles, then one would expect the same degree of coalescence to occur in freshwater as occurs in saltwater. However, the flume experiments of Ostazeski et al. (1997a; 1997b; 1998) have shown that under the same bitumen concentration and energetic conditions, the particle growth (particularly surface patty formation) is much greater in saline conditions.

The reason for the different responses in freshwater and saltwater has been theorized to be the result of a chemical 'denaturing' of the surfactant in saltwater (Ostazeski et al., 1998). However, this theory remains untested as there is no evidence of the chemical change. It could also be theorized that the degree of disassociation/partitioning of the surfactant from the bitumen may be greater in saltwater than in freshwater. However, Ostazeski et al. (1998) showed that the dissolved surfactant concentration remained the same and stable throughout both the freshwater and saltwater flume experiments. Therefore, the third factor controlling collision frequency may well lie in the density-float/sink

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[b] However, based upon experiments involving clay particles in water, other factors that undoubtedly play at least minor roles in collision frequency of bitumen particles are; (1) the particle distribution and shape (i.e., polydispersed populations and non-spherical particles generally exhibit greater collision potential than uniform particles) and (2) orthokinetic coagulation (i.e., the rate of collision as smaller particles are 'captured' by rising or falling larger particles).

relationship discussed above. One can envision how the number of collisions might be increased in saltwater where the buoyant bitumen particles will tend to rise, become more concentrated in the surface layer, and thereby increase the frequency of their collisions. On the other hand, in freshwater, where the bitumen particles remain dispersed (or even sink) the collision frequency is greatly reduced. Perhaps, under real spill conditions in freshwater the bitumen particles would sink to the bottom and, perhaps, even coalesced there (i.e., form a sinking patty). Notably, in the Ostazeski et al. (1997a; 1997b; 1998) freshwater flume tank experiments (with a finite and relatively shallow water depth) the tank's energetics kept the bitumen particles dispersed and thereby may have prevented their sinking (and perhaps their coalescence). Undoubtedly, a range of Orimulsion concentrations must be included in our experiments. The proposed range of 17,000 to 1000 mg Orimulsion/L water seems appropriate to test the effect of concentration on coalescence. However, it was recently suggested that a much higher concentration, *ca.* 50,000, also be included for observational purposes only. Therefore, at least a couple of tests will be performed at this concentration.

3.2.1.3 Particulate Amount and Type

Particulate matter in oceanic waters includes both organic (living and dead) and inorganic moieties. The concentration of particulates is typically reported as the total suspended solids (TSS). For our purposes, the importance of TSS in the water into which Orimulsion is spilled probably lies with their potential to increase coalescence of bitumen particles by increasing the potential for collisions. This phenomenon was observed by Brown et al. (1995) who showed that when excessively large concentrations of (Tampa Bay) sediment was added to agitated vessels significant quantities of bitumen became adhered to the finer particles and rapidly sank (due to the relatively high specific gravity of the mineral-bitumen particles). It was envisioned that suspended sediments in water provided a surfactant-free surface to which bitumen particles could adhere upon a collision.

The concentration of TSS in water will be a function of the particle size and the turbulent energy in the system (i.e., energetics). In addition, in the case of freshwater the concentration of some particulates will also be dependent upon their solubility (e.g., calcite added to freshwater will be subject to some dissolution). Furthermore, since energetic conditions in our experiments will vary we can expect the TSS concentration to vary. Therefore, we propose to add a given amount of particulate matter to each experiment that will represent the 'high' particulate load. The 'low' load amount to be added will be zero. The combined effect of the energetics and particle size of this material will then determine the amount of particulate in suspension. What amount is appropriate to add?

Open ocean concentrations of TSS are generally in the 0.5-250 µg/L range (Riley and Chester, 1971). Concentrations in areas of deltaic sedimentation are much higher (e.g., Mississippi River average 270 mg/L; Milliman, 1990). We propose to select a 'high' concentration of around 270 mg/L.[c] We envision only a certain amount of this will stay in suspension, especially under the low energetic conditions.

What kind of material should be added? Theoretically speaking, smaller-sized particulate matter would seem to favor collisions and adherence of bitumen due to the increased surface area and ease of suspension of smaller particulates, e.g., clay- and silt-sized particles (< 2 and 2-62.5 µm, respectively). This was also confirmed in the study by Brown et al. (1995) who showed that these fine grained particulates (< 75 µm) demonstrated approximately three orders of magnitude greater adhesion potential than coarser grained particulates (> 75 µm). Small particulates also comprise the majority of the lithogenous sediment load carried to coastal waters by rivers and these particles also have the longest residence time in seawater. Thus, only clay-to-silt size particles will be considered in these experiments.

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[c] Note that the 35,710 to 142,860 mg/L sediment concentrations used in the experiments by Brown et al. (1995) far exceeded the typical suspended solid (TSS) load in Tampa Bay waters which ranges from 20-120 mg/L.

What kind of clay-silt size particles will be used? It had been suggested that two types of particulate material be used in these experiments, kaolinite and calcite. Kaolinite's selection is reasonable, as it comprises a significant proportion (10-20%) of the clay-sized materials in U.S. coastal marine sediments. The impact of additional particles (other than bitumen) in our experiments will confound the measurement of bitumen particle size. Kaolinite, like all clays, are platy minerals whose crystal structure consists of layers or sheets. Thus, the size of an individual kaolinite particle varies significantly depending on the orientation of the plates and the number of plates in a 'stack'. Our preliminary investigations have revealed that the particle size distribution of a kaolinite slurry exhibits maxima around 2 μm and 25 μm. We believe these maxima represent the 'side' and 'top' views of kaolinite particles. These populations of particles cannot be distinguished from bitumen; therefore, it must be realized that for the experiments in which kaolinite is added the particle diameters being measured will be a composite of bitumen, kaolinite, and potentially bitumen/kaolinite particles.

It had also been suggested that we consider the influence of a non-lithogenous particulate material, e.g., a precipitated material. Calcite is the most common carbonate mineral which forms from the precipitation of $CaCO_3$ from seawater. Since calcite is not carried by rivers to larger water bodies and deposited, it typically does not occur as TSS in most settings. In areas where carbonate sedimentation predominates (e.g., coastal Florida) it is possible to get calcite particles into suspension through disturbing of the bottom sediments. Therefore, we will still consider including calcite. However, one important factor that has not been considered in our earlier discussions is the fact that if we were to use a second type of particulate material, the particle size of it would be different from the other (e.g., kaolinite). Calcite will also partially dissolve in our freshwater experiments. Since particle size measurements are already going to be confounded by the presence of kaolinite, we will further confound the particle size results by using a second type of particulate material. Nonetheless, it is considered valuable to include calcite in order to address the issue of whether or not the type of particulate matter may influence the coalescence phenomenon. As will be evident in the Section 3.3, the use of both kaolinite and calcite introduces a discrete variable into the experimental design. In doing so, the options for the design become limited, therefore, we expect that the addition of calcite will be limited to the screening phase of the study.

3.2.1.4 Energetics

Previous benchtop experiments involving Orimulsion have been performed under static conditions or under gentle agitation by magnetic stirrers. No attempt was made to quantify the energy in the latter systems. In our study we intend to introduce some degree of turbulence to all of our benchtop experiments (i.e., no static tests). The objective of evaluating this variable is to determine the impact that turbulent energy has on the frequency of collisions between bitumen particles and between bitumen and clay particles. Therefore, the method used need only increase the propensity for collisions.

Naturally, there are a variety of ways in which turbulence can be introduced. We have considered many different options. We intentionally have not considered any method that required putting some type of agitator into the vessels (e.g., oscillating rings, propellers, etc.) due to the 'stickiness' of Orimulsion. We have also considered rotating flasks and based on earlier experience have eliminated this option. (Previous experiments with Orimulsion in seawater have produced a bitumen 'plug' in these vessels.) We have also had to consider the need to conduct a fairly large number of experiments on moderate volumes of water. Therefore, from a practical standpoint we propose to introduce turbulence to our benchtop experiments by using an orbital shaker table apparatus (described in Section 4.1). A brief discussion here is directed at understanding the turbulence within such a system.

A turbulence spectrum will include eddies of different amounts of energy and sizes. According to turbulence theory the largest eddies will cascade into smaller eddies and the smallest will dissipate by being converted into heat by the viscosity of the water (Delvigne, 1993). In our proposed shaker table experiments the larger eddies created will be of the same order of magnitude as the disturbance, i.e., the diameter of the container. Recently, Intervep researchers (Masciangioli et al., personal communication, 1998) have conducted orbital shaker table studies in which the cascade process is believed to occur and the kinetic energy (K) of the water due to its rotation (i.e., not its oscillation due to the surface wave) could be estimated by the following equation:

$$K = \pi \rho \omega^2 H R^4/4$$

Where ρ=density of fluid, ω=mean angular velocity of the fluid, H=water depth and R=radius of vessel. They demonstrated that in a 1L vessel with H=12.7 and R=5 cm, rotated at 120 rpm, the water had an oscillation frequency of 25 rpm (reduced from the table's rpm's due to inertia, friction and other effects) and the K could be calculated from the equation above to be 0.52 Joules/m^2. The mean tangential velocity of a water particle 5 cm from the center of the vessel was determined to be 13 cm/sec. This was considered comparable to a 1.44 cm amplitude progressive wave having an orbital velocity of 16.5 cm/sec.

We have conducted some preliminary experiments using our prototype benchtop apparatus (described in Section 4.1). We propose that the following operating conditions be used: diameter of oscillation of 1.9 cm, shaker table frequencies of 10 and 100 rpm, shaker table oscillation periods of 6.0 and 0.60 sec, radius of 3L water of 9 cm, height of 3L of water is 14 cm. Calculations like those described above were performed on these operating conditions. These resulted in a K of 1.096 J/m^2. The mean tangential velocity of a water particle 8 cm from the center of the vessel was determined to be 17.5 cm/sec. This was considered comparable to a 2.1 cm amplitude progressive wave having an orbital velocity of 12.0 cm/sec.

3.2.1.5 Summary of Independent Variables to be used in Benchtop Experiments

Table 3-1 summarizes the evaluation of the independent variables that we propose to include in our benchtop experiments. All but one of the variables to be included are continuous variables, i.e., they can have any value over some numerical range. The complicating factors introduced by the inclusion of the discrete variable, particulate type, have been discussed. It was nonetheless decided to include this variable in the screening phase of our study (See below).

Table 3-1: Summary of Independent Variables to be use in our Benchtop Experiments

		EXPERIMENTAL RANGE			
INDEPENDENT VARIABLES	FACTOR TYPE	LOW	HIGH	CENTER	UNITS
Salinity	continuous	0	35	17.5	ppt
Water temperature	continuous	4	25	14.5	°C
Orimulsion concentration	continuous	1000	17000	9000	mg/L
Particulate loading	continuous	0	270	135	mg/L
Particulate Type	discrete	kaolinite	calcite	----	----
Water energetics	continuous	10	100	45	rpm

3.2.2 Dependent Variables

3.2.2.1 Bitumen Particle Size

The primary dependent variable that was to be measured is bitumen particle size. The method for collection and measurement of bitumen from the benchtop vessels is described in Section 5.2. As mentioned above, bitumen particle size measurements will be complicated by the addition of kaolinite and calcite in some of the experiments. Our preliminary experiments have shown that it will be extremely difficult to describe bitumen particles separately from kaolinite or calcite, therefore, any particle size measurements will be a composite of bitumen, kaolinite (or calcite), and bitumen/kaolinite (or bitumen/calcite) particles. Nonetheless, with careful interpretation and knowledge of kaolinite's and calcite's particle size, it should still be possible to qualitatively recognize changes in overall particle size. However, absolute bitumen particle size cannot be determined in the experiments that will include kaolinite. Those experiments that will not include any kaolinite or calcite will allow for bitumen particle growth to be quantified.

The need to measure changes in particle size over the course of the experiments requires that either the measurements be performed at discrete intervals throughout the experiments or that experiment duration become another independent variable. The former seems like a more cost-effective approach since far fewer experiments will need to be conducted. We propose to measure particle size (bitumen, clay/calcite, or bitumen/clay/calcite) of the suspended material in intervals of 1, 3, 8 and 24 hours following the release of Orimulsion in our benchtop studies. These four intervals should provide the information necessary to determine if particle growth is occurring and at what relative rate for the variable conditions of our experiments. One potential problem with this approach is that after 24 hours it is possible that there will be too few suspended particles to provide a reliable particle size measurement.

Another confounding aspect of particle size measurements will be the formation of a wide range of particle sizes (e.g., from 2 micron clay particles to 10 cm diameter patties). Measurement of particles over such a wide dynamic range is virtually impossible. Only via techniques such as microscopy or sedimentation can this wide range of particles be measured. However, these techniques cannot be used in the case of bitumen since they would undoubtedly change the particles' environment to the point where the particle size would continue to change. Thus, the measurement technique must be relatively fast. The most appropriate technique is laser diffraction but this technique is unable to 'see' particles over such a wide dynamic range. (Even the most expensive units can only see from 0.4 to 2000 microns). The widest dynamic range available on our instrument is 1.5 and 600 microns (see Section 5.1.4). Our experience tells us that particles well above 600 microns can be formed in these experiments. Nonetheless, we will measure particle size throughout our experiments with the intention being to recognize changes rather than absolute particle diameters.

3.2.2.2 Mass Balance Measurements

We believe that bitumen particle size should not be the only dependent variable to be measured in these experiments. A variety of chemical and physical measurements can be made that will provide additional insight into the fate of Orimulsion when spilled under a variety of conditions. After all, this is the primary goal of these studies. First and foremost among these is an attempt to 'mass balance' the end products.

A mass balance of bitumen's fate was attempted in the previous flume experiments conducted in Battelle's 4000L tank. In the seawater study it was estimated that at the end of the 168 hour test that 65% of the bitumen occurred in the surface patty, ~35% was adhered to the walls near the water level, and ~1% was still within the water column. We intend to attempt a more thorough reconciliation of the fate of Orimulsion in our benchtop experiments. Our preliminary experiments using the prototype apparatus have shown that Orimulsion's bitumen will either

(1) form a floating patty,
(2) form a sinking patty,

(3) coalesce with kaolinite and sink to form non-cohesive grains of bitumen/kaolinite,

(4) dissolve into the water,

(5) remain suspended in the water, or

(6) stick to the vessel walls in the vicinity of the water line (i.e., form a "bathtub ring").

We propose to quantify the bitumen that ends up in each of these six 'compartments' (see Section 5.2.6).

The weight of the floating patty will be measured at the conclusion of the experiment. It's moisture content will be determined in order to 'back out' the mass of bitumen in the patty. If a sinking patty has formed it will be weighed and analyzed in the same manner. Any sediment that accumulates in the bottom of the vessels after 24 hours will be collected and analyzed to determine the concentration of total petroleum hydrocarbons (TPH) that occurs in the 'sink' fraction. The TPH that occurs in suspension will be determined after 1 and 8 hours. The concentration of TPH that is dissolved (filtered water) and remains suspended (filtered material) in the water column after 24 hours will be determined on an aliquot of water. Finally, the amount of bitumen that has adhered to the vessel will be determined gravimetrically.

The fact that some bitumen will be removed from the vessels in the course of the experiments for particle size and TPH measurements implies that some small percentage of the bitumen will not be accounted for in the mass balance. Nonetheless, the results will provide a semi-quantitative and relative sense of just where the organic matter is going. These semi-quantitative results will provide the basic information necessary to predict Orimulsion's behavior under the wide range of conditions to be studied and also help to focus the meso-scale experiments. We believe that herein may lie the greatest practical yield of information from the benchtop experiments proposed herein.

3.2.2.3 Fate of Polycyclic Aromatic Hydrocarbons

Polycyclic aromatic hydrocarbons are perhaps the most important chemicals of concern in terms of their potential impact to marine and coastal biota. These experiments provide the opportunity to investigate the influence of several variables on determining where the PAH's would end up in the event of an Orimulsion spill. We propose to determine the concentration of PAH that occur dissolved in the water and those which occur in the sediment at the conclusion of the benchtop experiments.

3.2.2.4 Summary of Dependent Variables to be Measured in Benchtop Experiments

In summary of the discussion above, Table 3-2 lists the dependent variables that will be measured in the course of the benchtop experiments.

Table 3-2: Inventory of proposed independent and dependent variables.

DEPENDENT VARIABLES
particle size in suspension after 1 hour - μm
particle size in suspension after 3 hours - μm
particle size in suspension after 8 hours - μm
particle size in suspension after 24 hours - μm
Mass of floating bitumen/patty after 24 hours - g
Mass of sinking bitumen/patty after 24 hours - g
Mass of bitumen adhered to glass walls after 24 hours - g
TPH in sediment after 24 hours – mg/kg
TPH in suspension after 1 hours – mg/L
TPH in suspension after 8 hours – mg/L
TPH in suspension after 24 hours – mg/L
Dissolved TPH in water after 24 hours – μg/L
Dissolved PAH in water after 24 hours – μg/L

3.3 Design of Experiment Principles

There are three basic levels to DOE, (1) screening level, (2) two-level factorial design and (3) three-level response surface design. The screening level experiments are intended to identify those variables that have the greatest effect on the outcome and thereby reduce the number of variables for a more sophisticated two- or three-level design study. In our case, screening would help identify which of the five independent variables (Table 3-1) have the greatest effect on the dependent variables (Table 3-2). Because we are monitoring multiple dependent variables some judgement may have to be made regarding the 'most important' ones if it is found that they are controlled by different independent variables. In this study it will be prudent to perform a set screening level experiments in order to potentially reduce the number of independent variables in some additional two- or three-level experimental designs.

3.3.1 Screening Designs

There are two types of screening techniques Plackett-Burman (PB) and fractional factorial (FF), with the former being much more widely used do to its ease of interpretation. PB designs are multi-variable, two-level designs that can be used to evaluate any number of variables up to four less than the number of experiments. Given the six independent variables determined for these experiments (Table 3-1) we propose to screen using a 12-run PB design as shown in Table 3-3. In Table 3-3 the '+' and '-' signs represent the high and low values for each of the dependent variables. Assigning these values to our list of five variables yields the design shown in Table 3-4.

Table 3-3: Twelve-run Plackett-Burman design

Trial	Value	X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11
1	+	+	+	-	+	+	+	-	-	-	+	-
2	+	+	-	+	+	+	-	-	-	+	-	+
3	+	-	+	+	+	-	-	-	+	-	+	+
4	+	+	+	+	-	-	-	+	-	+	+	-
5	+	+	+	-	-	-	+	-	+	+	-	+
6	+	+	-	-	-	+	-	+	+	-	+	+
7	+	-	-	-	+	-	+	+	-	+	+	+
8	+	-	-	+	-	+	+	-	+	+	+	-
9	+	-	+	-	+	+	-	+	+	+	-	-
10	+	+	-	+	+	-	+	+	+	-	-	-
11	+	-	+	+	-	+	+	+	-	-	-	+
12	+	-	-	-	-	-	-	-	-	-	-	-

Table 3-4: Factors assigned to the first six columns of Table 3-3 and sorted by temperature.

Trial	Dependent Variable	S (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Energetics (rpm)	Part. Type	X7	X8	X9	X10	X11
2		35	4	17000	270	10	Kaol.	-	-	+	-	+
6		35	4	1000	0	10	Kaol.	+	+	-	+	+
7		0	4	1000	270	100	Calc.	+	-	+	+	+
8		0	4	17000	0	100	Calc.	-	+	+	+	-
10		35	4	17000	270	100	Calc.	+	+	-	-	-
12		0	4	1000	0	10	Kaol.	-	-	-	-	-
1		35	25	1000	270	100	Calc.	-	-	-	+	-
3		0	25	17000	270	10	Kaol.	-	+	-	+	+
4		35	25	17000	0	10	Kaol.	+	-	+	+	-
5		35	25	1000	0	100	Calc.	-	+	+	-	+
9		0	25	1000	270	10	Kaol.	+	+	+	-	-
11		0	25	17000	0	100	Calc.	+	-	-	-	+

In Table 3-4 the first six columns contain the assigned 'extreme' values for the independent variables. (The extreme Orimulsion concentration, 50,000 mg/L, is not included in this design-see below). Using these extremes is considered to be an important part of any DOE as it will make it easier to recognize the variables which have the greatest effect. Examination of Table 3-3 reveals that there is a balance among all of the variables so that each variable is paired with each other variable's extreme values. While it would be most appropriate to conduct the experiments (trials) sequentially and in a random order this is considered impractical given the need to minimize costs. Since temperature is the most difficult parameter to control, we propose to perform two sets of 6 experiments to be conducted at 4°C and 25°C.

The last five columns in Table 3-4, the unassigned columns, will be used to measure the "noise" (or standard error of the factor effect; S_{FE}) within the system. The effect of these unassigned factors will also provide a means of assessing the presence of a large interaction between variables.

The dependent variables that will be quantified in the course of the 12 PB design experiments include all of those listed in Table 3-2. Calculation of the t-value and 95% confidence limits of the S_{FE} will provide a means of identifying and ranking those independent variables that have a significant effect on each of the dependent variables being measured. Interpreting which independent variables lead to the highest or lowest concentrations of TPH or PAH, or greatest patty weight after 24 hours will be straightforward.

However, an evaluation of the particle growth, i.e., particle size at different times in the course of the experiments (1, 3, 8 and 24 hours) will be more difficult since it requires qualitative analysis of four different results. We intend to synthesize the particle size results for each individual time period in order to identify/rank the most important variables leading to increased particle size throughout the entire experiment. Interpretation of these variables on particle growth will be further complicated by the presence of kaolinite/calcite in some of these runs.

It can be anticipated that the PB screening process will help to identify the three to five most significant independent variables that effect each of the dependent variables over 24 hours. However, as mentioned above, because we are monitoring multiple dependent variables some judgement will have to be made regarding the 'most important' dependent variables, particularly if it is found that they are controlled by different independent variables. In any case, in having eliminated some variables from further consideration, we have two options by which to proceed, namely a two-level factorial design experiment or a three-level response surface design (i.e., a face-centered cube design or a Box-Behnken spherical design). These two scenarios are described below for completeness. However, for reasons described at the beginning of Section 3.3.3, we believe that we will perform only the three-level response surface design.

In addition to the PB screening design shown in Table 3-4, it is proposed that four additional experiments be conducted at extremely high concentrations of Orimulsion, namely 50,000 mg/L. Four experiments are proposed in which the primary independent variables to be investigated are salinity, temperature and energetics (Table 3-4'). The dependent variable results of these investigation will not be include in the statistical results of the screening study but rather they will be investigated and described qualitatively. Experiments at this high concentration of Orimulsion are intended to provide some basis upon which to predict its response in the immediate vicinity of a release or spill, i.e., where concentrations would be their highest.

Table 3-4': High Orimulsion concentration experiments to be conducted concurrently with those in Table 3-4.

Trial	Dependent Variable	S (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Energetics (rpm)	Part. Type
13		35	4	50,000	0	10	--
14		0	4	50,000	0	100	--
15		35	25	50,000	0	100	--
16		0	25	50,000	0	10	--

3.3.2 Two-level Factorial Design

A two-level factorial design approach is used to estimate all factor effects in linear interaction effects. (This approach must be used if we were to change our approach to include any discrete variables, e.g., particulate type.) The variables with significant effects identified by the PB screening experiments will be used to construct either a three-, four-, five- or six-factor two-level factorial design leading to a total of 8, 16, 32 or 64 additional experiments. We fully anticipate that the PB screening can be used to limit the number of significant variables to three or four, thus limiting the number of additional experiments to 8 or 16. As an example to this approach, Table 3-5 shows the four-factor two-level factorial design assuming salinity, temperature, Orimulsion concentration, and energetics were identified as being significant in the PB screening.

Table 3-5: Example of a four-factor two level factorial design.

	X1	X2	X3	X4	X1	X2	X3	X4	
Run #	**Salinity**	**Temp**	**Conc.**	**Energetics**	**Salinity**	**Temp**	**Conc.**	**Energetics**	**Batch**
1	-1	-1	-1	-1	0	4	1000	10	1
2	1	-1	-1	-1	35	4	1000	10	1
3	-1	1	-1	-1	0	25	1000	10	2
4	1	1	-1	-1	35	25	1000	10	2
5	-1	-1	1	-1	0	4	17000	10	1
6	1	-1	1	-1	35	4	17000	10	1
7	-1	1	1	-1	0	25	17000	10	2
8	1	1	1	-1	35	25	17000	10	2
9	-1	-1	-1	1	0	4	1000	100	1
10	1	-1	-1	1	35	4	1000	100	1
11	-1	1	-1	1	0	25	1000	100	2
12	1	1	-1	1	35	25	1000	100	2
13	-1	-1	1	1	0	4	17000	100	1
14	1	-1	1	1	35	4	17000	100	1
15	-1	1	1	1	0	25	17000	100	2
16	1	1	1	1	35	25	17000	100	2

No blocking or randomization would be used but rather the experiments would be conducted in two 'batches' depending on the temperature (4 or 25°C), again, in order to reduce costs. As was the case in the PB screening the same set of dependent variables (Table 3-2) would be measured for each experiment. The factor effect and interaction effects of these four (or other number of) factors will be calculated according to standard methods that would produce a measure of the effect. The unassigned factor effects would be used to calculate random error in the system and 95% confidence limits. Evaluation of the results would proceed as described above for the PB screening. If the two-level factorial design is performed, the most important independent variables identified would be used to select a set of conditions that would be used in the meso-scale flume experiments (see below).

3.3.3 Three-level Response Surface Design

3.3.3.1 Overview and Justification for this Approach

A three-level response surface design is preferred over the two-level factorial design because the former will allow for the recognition and quantification of the non-linear (i.e., curvature) effects. In other words, two-level factorial design can only recognize changes over a linear response curve. However, our preliminary experiments and previous work by others give us reason to believe that the properties of Orimulsion will cause it to not necessarily respond linearly. For example, at some salinity value between 32 and 4 $^{o}/_{oo}$ it might be anticipated that particle growth and patty formation will occur but at only a slightly lower salinity particle growth and patty formation will not occur. Thus, the response may be non-linear.

In order recognize this and other non-linear effects there can be no discrete variables among the independent variables being evaluated in a three-level response surface design. (This, therefore, is another good reason to not complicate the study by including both kaolinite and calcite.) This is because if curvature effects are to be estimated, each independent variable must have at least three levels. In our opinion, there is no realistic intermediate level in the case of particulate type (i.e., 50:50 kaolinite:calcite is not a real world situation). It is for these reasons that we propose to perform the three-level response surface design on the most critical continuous independent variables identified in the PB screening.

As a result of considering intermediate values for continuous variables, three-level response surface designs are better able to describe the responses throughout the entire experimental ranges. This is accomplished by the use of quadratic polynomials to match the response surfaces. This will permit more accurate prediction of the responses even at factor combinations not actually run. For example if, as we believe, salinity will play an important role in particle growth or floating patty weight, the response surface design will permit us to identify the 'key' salinity value at which these responses are most effected. Furthermore, this approach will allow us to narrow the range over which each variable has a significant effect on the dependent variables of interest. These results will permit an identification and narrowing of the conditions to be used during the subsequent meso-scale studies of Phase III.

3.3.3.2 Types of Three-level Response Surface Designs

There are two types of three-level response surface designs, namely a face-centered cube design or a Box-Behnken spherical design. The former considers a cubical experimental region and the latter considers a spherical experimental region. The best way to consider this is to imagine a three variable problem. The shape describing the range for each variable would be a cube, with the high and low values of each of the three variables occurring at the corners of the cube. This cube would have 8 corners, 6 face centers, and one center point. This design would include all values within the ranges for each variable and is the basis for the face-center cube design. Connecting only the midpoints of these same three variable ranges would form a polyhedron that approximates a sphere (within the original cube). This polyhedron would have 12 edge points and one center point. This design would exclude the corners of the cube, i.e., the high and low values for each variable and is the basis for the Box-Behnken spherical design.

The face-centered cube design is considered more 'bold' than the Box-Behnken design. The face-centered cube design may be more appropriate for the problem at hand since it includes the entire range for each independent variable. It seems prudent to not ignore the 'extreme' conditions given that they may occur in the 'real world'. For example, it is possible that a situation exists were Orimulsion is released into 35 °/oo seawater, at 25°C, with no inorganic particulates, and 'mild' energetics. Therefore, we recommend that a face-centered cube design be used in this part of Phase II. The number of significant variables identified in the PB screening will determine the number of experiments necessary in a face-centered cube design according to Table 3-6.

Table 3-6: Size of response surface model necessary for 2 to 5 independent variables.

Number of Variables	Experiments required - Face-centered cube design
2	11
3	17
4	31
5	45

If all five of the independent variables (Table 3-1) are identified as important then 45 additional experiments will be necessary. If any can be eliminated then 31 or fewer experiments will be necessary. This number will still provide a modest number of degrees of freedom for calculating error.

Let us assume that the PB screening has determined that only salinity, temperature, and bitumen concentration are important in determining most of the dependent variables (Table 3-2). Evaluating these three independent variables would require 17 experiments performed according to the three-factor face-centered cube design shown in Table 3-7.

Table 3-7: Hypothetical three-factor face-centered cube design.

Run #	**Cube Location**	**X1**	**X2**	**X3**	**Salinity**	**Temp**	**Conc.**	**Batch**
					°/oo	**°C**	**mg/L**	
1	Corner	-	-	-	0	4	1000	1
2	Corner	+	-	-	35	4	1000	1
3	Corner	-	+	-	0	25	1000	3
4	Corner	+	+	-	35	25	1000	3
5	Corner	-	-	+	0	4	17000	1
6	Corner	+	-	+	35	4	17000	1
7	Corner	-	+	+	0	25	17000	3
8	Corner	+	+	+	35	25	17000	3
9	Face Center	-	0	0	0	14.5	9000	2
10	Face Center	+	0	0	35	14.5	9000	2
11	Face Center	0	-	0	17.5	4	9000	1
12	Face Center	0	+	0	17.5	25	9000	3
13	Face Center	0	0	-	17.5	14.5	1000	2
14	Face Center	0	0	+	17.5	14.5	17000	2
15	Center	0	0	0	17.5	14.5	9000	2
16	Center	0	0	0	17.5	14.5	9000	2
17	Center	0	0	0	17.5	14.5	9000	2
18	Center	0	0	0	17.5	14.5	9000	2

As was the case previously, the maximum (+) and minimum (-) of each variable are used as well as the arithmetic mean of these values (0) to design the experiments. If temperature turns out to be important then the experiments will be conducted in 3 batches that correspond to the temperature of the experiments in order to save time and reduce costs. This will eliminate the ability to block or randomize the results but is considered a necessary step given the large impact on costs. In the hypothetical case described here, that would require 3 batches of 5, 11, and 5 experiments. Analysis of four center point replicates (runs 15-18) will provide a means to determine the pure error within the system.

As in the previous experimental designs discussed, the dependent variables to be measured in the course of these experiments are listed in Table 3-2. Once again, only those dependent variables that can be quantified can be analyzed statistically. That is to say the particle growth <u>rate</u> cannot be quantified.

While simple 'cube' plots or X-Y scatter plots can provide some means of interpreting the results of a face-centered cube design, generally speaking interpretation will require computer assistance. We propose to use JMP Software Version 3.1 from SAS Institute, Inc. to perform our analysis. This package allows for the determination of the b coefficients, standard error in b, and t-values for the quadratic solution for each dependent variable being considered. The results can be graphically depicted using 2-dimensional contour plots.

In the case of our hypothetical 3-factor face-centered cube design depicted in Table 3-7, the output would include calculation of each of the b-coefficients for each factor in the quadratic formula:

$$Y = b_0+b_1X1+b_2X2+b_3X3+b_{12}X1X2+b_{13}X1X3+b_{23}X2X3+b_{11}X1^2+b_{22}X2^2+b_{33}X3^2$$

Where:

Y = dependent variable of interest (Table 3-2)
$X1$ = temperature (linear effect)
$X2$ = salinity (linear effect)
$X3$ = bitumen concentration (linear effect)
$X1X2$ = temperature-salinity (two-way interaction effect)
$X1X3$ = temperature-bitumen concentration (two-way interaction effect)
$X2X3$ = salinity-bitumen concentration (two-way interaction effect)
$X1^2$ = temperature (curvature effect)
$X2^2$ = salinity (curvature effect)
$X3^2$ = bitumen concentration (curvature effect)

The solution to this equation provides a means of predicting the outcome for any "Y" for any setting of "X". The fit of the equations for each "Y" is determined by standard least squares regression analysis.

3.4 Utility of the DOE Phase of Experimentation

The outcome of the benchtop experiments will result in

(1) identification and ranking of the independent variables (Table 3-1) which have the greatest impact on each of the dependent variables of interest (Table 3-2),

(2) a narrowing/focussing of the ranges over which changes in the independent variables have the greatest impact, and

(3) qualitative understanding of the fate of Orimulsion when present in extremely high concentrations (50,000 mg/L).

For example, if salinity is determined to be the most important variable in determining bitumen particle size, TPH in sediment, or dissolved PAH, our benchtop results should help to narrow the range over which salinity is most critical, e.g., 15 to 20 ‰. This result would allow us to design a focussed set of meso-scale (flume) experiments to address particle growth over this narrow range of salinity.

4. DESIGN OF BENCHTOP EXPERIMENTS

4.1 Overview and Description of Prototype Benchtop Apparatus

As described in Section 3.2, the fate of Orimulsion spilled onto natural waters will depend on the complex interaction of physical, chemical, and biological processes. Reproducing the effects of these processes in the laboratory is extraordinarily difficult, essentially impossible. Keeping this in mind, the benchtop experiments to be performed in the course of this study must be kept relatively simple and thereby allowing for some reasonable level of understanding of the effects of the five independent variables (Table 3-1) to result. The design of the experiments is intended to provide insight into the expected behavior of Orimulsion when spilt under a wide range of conditions. The objectives of the benchtop study is to help focus the meso-scale experiments that will follow and also to aid in the design of contingency actions under a wide variety of conditions.

In this section we describe our proposed benchtop design. We have drawn upon our first-hand experience in designing and testing of a prototype apparatus (Fig. 4-1). Our design has attempted to incorporate practical knowledge obtained in previous studies on Orimulsion by other labs (e.g., Intevep, S.A., Univ. of Mass., and Univ. of Miami). Some key elements related to each step in the benchscale experimental design are discussed in the following sections.

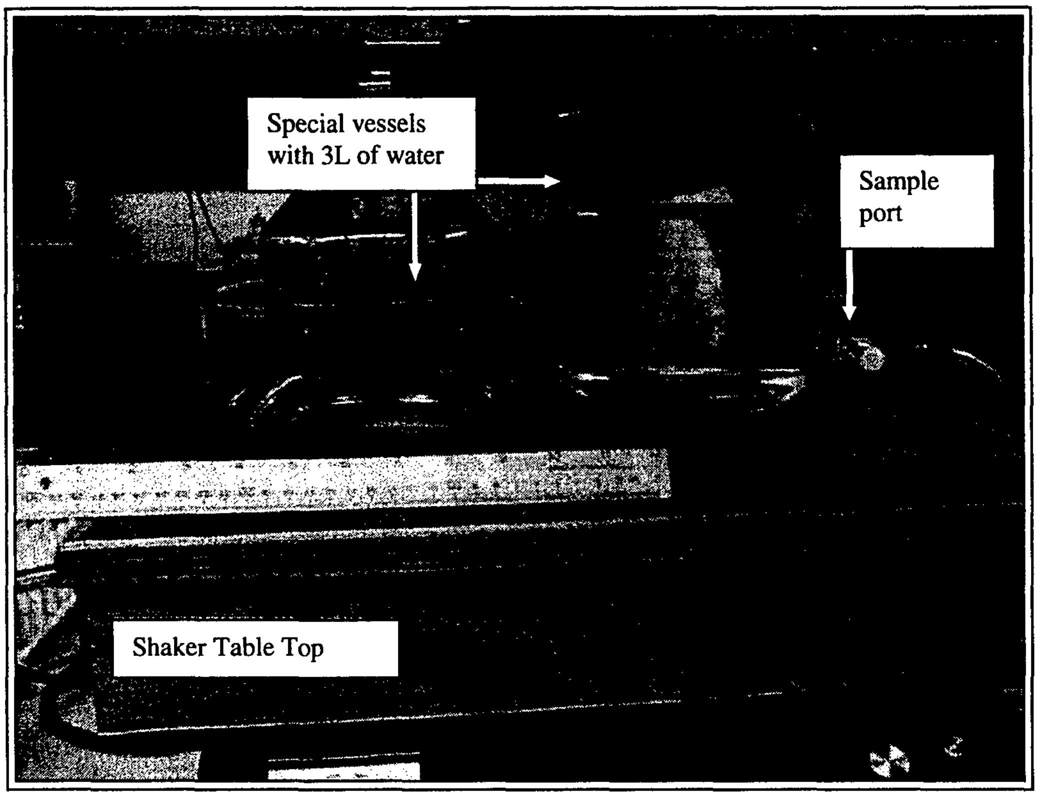

Figure 4-1: Photograph showing prototype vessels resting on and seated in a custom made orbital shaker tabletop capable of holding four vessels.

4.2 Vessel Construction

Four-liter, open top, clear glass vessels will be fabricated from 5 L bottles by removing their tops and polishing the cut edges. The final vessel dimensions will be 24 cm high by 18 cm diameter. A small sampling port consisting of a glass neck containing a Teflon stopcock will be added to each vessel. This sampling port will be located approximately 4 cm from the bottom of the vessels (Fig. 4-1). We find this location for the sampling port provides a good head pressure so that a representative water sample can be drawn. Samples for particle size analysis (1, 3, 8, and 24 hours), TPH in water (1, 3, 8 and 24 hours) will be collected through these ports. Attached to each port will be a flexible Tygon tube that will permit collection of these samples without interruption of the orbital shaking motion. During the experiments the vessels will be loosely covered with aluminum foil to minimize evaporation and contamination.

4.3 Salinity Determination and Maintenance

In the experiments to be conducted the salinity of the water will be determined with the aid of a conductivity meter. Seawater from Duxbury Bay (~23 ppt) will be adjusted accordingly using Instant Ocean sea salts or freshwater in order to achieve the desired salinity for each experiment. During the high temperature experiments (20-25°C) there may be some change in the salinity of the water due to evaporative losses. However, we believe that this change will be minimized by the use of aluminum foil and the relatively short duration of these experiments (24 hours).

4.4 Temperature

The benchtop experiments will be conducted in Battelle's temperature controlled rooms. One of these rooms contains benches for the benchtop experiments and the other contains the 4000 L flume tank. Both rooms must be operated at the same temperature. The range of temperatures is from 0°C to 30°C.

4.5 Orimulsion Concentration

A new shipment of Orimulsion will be used in these experiments (see below). The concentration of Orimulsion to be added to the benchtop experiments will be determined based upon the measured density of this material. With a maximum and minimum concentrations of Orimulsion in these experiments being 17,000 mg/L and 1000 mg/L, respectively (Table 3-1), and a density of approximately 1.01 g/ml, we can expect the need to add approximately 50.5 ml and 2.97 ml, respectively, to the 3 L of water to obtain the desired max. and min. concentrations.[d] These volumes of Orimulsion will be precisely added to the vessels using pre-weighed 50 ml plastic syringes. Our preliminary experiments have shown that this method of measuring and adding Orimulsion can be very precise. The exact volume of the Orimulsion added to each vessel will be determined by weighing the syringes after squirting out as much Orimulsion as possible (as some will stick to the outside of the syringe). Orimulsion will be added to the vessels after the shaker tables have been turned on in order to assure a more realistic mixing of the product in the water column.

4.6 Particulate Load

Pure kaolinite clay has been obtained from the Southeaster Clay Co., Aiken, South Carolina (803-648-3246). The density of this material has been determined to be 2.6 g/ml. The particle size distribution of a kaolinite slurry has been measured using the Malvern MasterSizer X (Section 5.2.2) and the clay was determined to have a bi-modal particle size distribution. The first population was centered around 2 microns and the second was centered around 25 microns. It is our impression that these populations represent the thickness (2 microns) and width (25 microns) of these platy minerals. The total particle size ranged from less than 1.4 microns to 100 microns. The source and characteristics of the calcite to be used has not yet been identified.

The mass of kaolinite or calcite to be added to each vessel will range up to 270 mg/L which, for the 3 L benchtop experiments, indicates 810 mg of kaolinite or calcite will be added. We propose to add the kaolinite/calcite to the vessels and vigorously stir the water to achieve a complete slurry. Following this the orbital shaker table will be turned on (see Section 4.7) and undoubtedly some of the clay will settle out, certainly under the 'mild' agitation conditions. However, some will remain suspended and be in suspension at the time when the Orimulsion is introduced. We believe this is the most appropriate manner by which to treat all the experiments equally. The initial particle size distribution of the clay slurry will be determined in order to help interpret the particle size results once the Orimulsion is added to the system.

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[d] The four extremely high concentration experiments (50,000 mg/L) will require approximately 150 ml Orimulsion be added tot he vessels (Table 3-4').

4.7 Energetics

The energetics of the benchtop apparatus will be introduced with the aid of a modified orbital shaker table (Figure 4-1). Two of these will be constructed which will allow for 8 experiments to be performed at any given time. The proposed 'mild' and 'harsh' agitation conditions are 10 and 100 rpm, respectively. Our preliminary experiments conducted under these conditions indicate that these rpm levels should provide an adequate range of turbulent energy in the 3L vessels. Higher rpm's tended to create a considerable vortex within the vessel which acted to draw Orimulsion down from the surface near the center of the vessel. A discussion of the turbulent energy introduced by orbital shaking was presented in Section 3.2.1.4.

5. ANALYTICAL METHODS DURING BENCHTOP EXPERIMENTS

5.1 Mixing and Characterization of Fresh Orimulsion

Prior to the initiation of benchtop testing, the new shipment of Orimulsion currently en route to Battelle will be thoroughly mixed using an air powered stainless steel drum mixing apparatus. Aliquots of approximately 4-L will be drawn off into one gallon paint cans, where they will be stored for future use throughout these benchtop and subsequent meso-scale flume experiments. The new "1998" batch of Orimulsion will be put through an abbreviated suite of physical and chemical measurements to ensure the exact makeup of the material relative to the "1997" Orimulsion previously analyzed at Battelle (Ostazeski et al., 1998), as well as, any specification or data provided by Bitor. These measurements will include density, viscosity, Iatroscan chemical analysis of saturate, aromatic, resin, and asphaltene (SARA) fractions, and bitumen particle size.

5.1.1 Density

The density of the fresh Orimulsion®, and water-in-oil emulsions was determined using an Anton Paar DMA 48 Density Meter oscillating cell digital density meter. This method is based on ASTM D4052-91, *Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter* (ASTM, 1993). Where the density or viscosity of the bitumen or emulsions formed exceeded the practical operational limits of the density meter, the density will be obtained gravimetrically using a 3 or 5 mL plastic syringe. The mass of the syringe and the emulsion is then made and the mass of the emulsion determined by difference (equation 2.1).

$$\text{Mass emulsion (g)} = [\text{Mass emulsion (g)} + \text{Mass syringe (g)}] - \text{Mass syringe (g)}$$

5.1.2 Viscosity

The viscosity of the fresh Orimulsion, resulting bitumen and any water-in-oil emulsions will be determined using a Haake RV-20 Rotational Viscometer. The viscosities will be determined using the viscometer, operated at the lowest shear rate necessary for accurate measurement of viscosity (Daling and Almås 1988). Typically, viscosities for fresh and weathered oils are measured at shear rates of 1 s^{-1}, 10 s^{-1}, 100 s^{-1}, and 1000 s^{-1}. Based on our past experiences, we expect the viscosity of the fresh Orimulsion will be measured at 100 s^{-1}.

5.1.3 SARA Analysis

The relative concentrations of saturated hydrocarbons, aromatic hydrocarbons, polars/resins and asphaltenes (SARA) will be determined by Iatroscan TLC/FID. Approximately 10 μg of Orimulsion or concentrated water extract is spotted onto a silica rod, and the saturated, aromatic, and resin fractions of the oil separated by development of the rods in a series of increasingly polar solvents (*n*-hexane, toluene, dichloromethane:methanol). The relative distribution of the saturated, aromatic, and resin fractions on each rod is then determined.

5.1.4 Particle Size Analysis

Particle size analysis of the fresh Orimulsion will conducted using a Malvern Mastersizer® X laser diffraction particle size analyzer (Fig. 5-1). The Mastersizer® will be calibrated initially using Duke Scientific Corporation (Palo Alto, CA) NIST particle size standards. It is well known that the diameters of non-spherical particles in a population of particles can be calculated and reported by numerous methods. These methods are simply different ways of describing the diameters of non-spherical particles, such as the bitumen particles in Orimulsion. We propose to use the most commonly referenced type of diameter, the volume mean diameter (D[4,3]), throughout this study. The volume mean diameter is defined as the diameter of a sphere having the same volume as the particles being measured.

Reporting a single diameter for a broad range of particles can be misleading. For example, if multiple populations of particles are observed the volume mean diameter for the entire range of particles is meaningless. If appropriate the raw data of these analyses will be provided so that the particle size distribution is available.

Bitumen particle diameters for the fresh Orimulsion® will determined by adding small, well-stirred quantity of Orimulsion to a circulating volume of milli-Q ultrapure water following a background measurements on the water alone. An obscuration of 10-30% will be obtained. A series of measurements will be obtained and averaged. The Malvern MasterSizer will be equipped with the internal flow through sample cell (Fig. 5-1A) and the 300 mm focusing lens. This lens allows for measurement of particles in the 1.5 to 600 micron range. Processing of the data will be performed using the polydispersion model and the 2$$D presentation code. (Some results may be re-processed and reported using the 40HD presentation code, as per Intevep's method. We are unable to use a 118 mm focal lens as per Intevep's method).

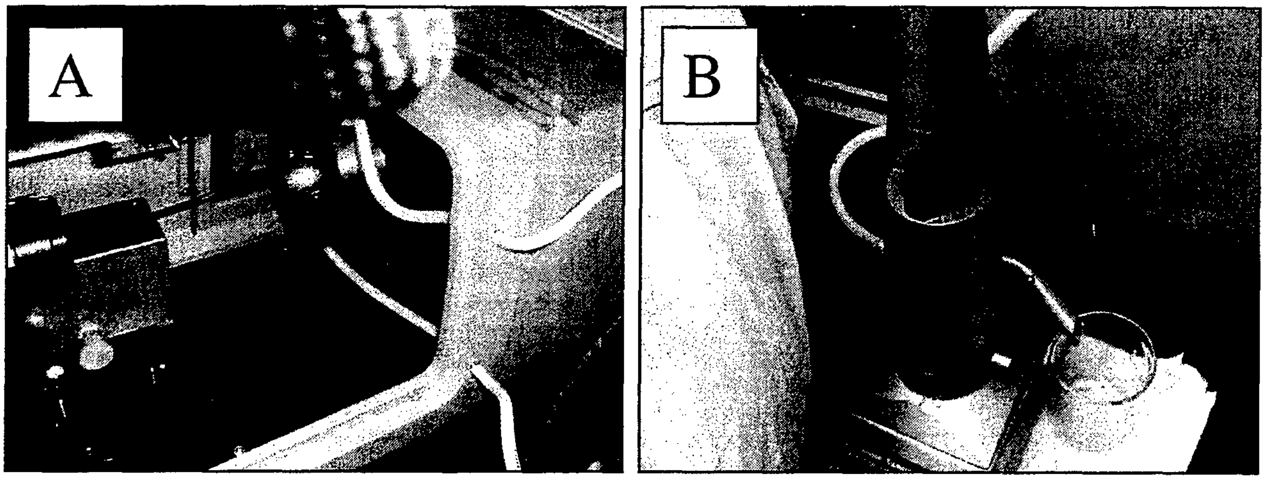

Figure 5-1: Close-up of Mastersizer X internal flow through cell (A) and circulating unit (B). **Orimulsion will be added to circulating (background) Milli-Q water sample through the opening in the circulating unit until an appropriate obscuration is achieved. Multiple measurements will immediately follow as bitumen particles are circulated through the internal flow through cell, thus passing through the laser beam.**

5.1.5 Gas Chromatographic Analysis of Fresh Orimulsion

Analysis of the fresh Orimulsion® for C_8 to C_{36} *n*-alkanes, isoprenoid hydrocarbons, and overall boiling range of selected samples will be performed according to modifications of EPA Methods 8015. The total gas chromatographic hydrocarbon signature of fresh Orimulsion® will be determined using a Hewlett-Packard 5890 Series II capillary gas chromatograph with flame ionization detection (GC/FID). A 3 µL aliquot of a 5 mg/mL dichloromethane dilution of the Orimulsion will be injected using splitless techniques onto a 30-m DB-5 capillary column (J&W fused silica DB-5, 30-m, 0.32-mm internal diameter, and 0.25-µm film thickness), and the oven will be programmed from 35EC to 320°C at 6°C/min. Prior to sample analysis, a multi-point calibration curve containing *n*-C_8 to *n*-C_{36} *n*-alkanes, pristane, and phytane will be generated to demonstrate instrument calibration and performance. Based on previous experience we do not expect any resolved alkanes or isoprenoids to be present. If they are, however, they will be quantified by the method of internal standards using the internal standard 5α-androstane.

5.1.6 Polycyclic Aromatic Hydrocarbons in Fresh Orimulsion

The analysis of target parent polycyclic aromatic hydrocarbons (PAH's), alkylated PAHs, and dibenzothiophenes (Table 5-1) will performed on the fresh Orimulsion® using HRGC/MS methods modified from EPA Method 8270. Basically, prior to sample analysis, the GC/MS will be tuned with perfluorotributylamine (PFTBA), and a multi-point initial calibration consisting of parent and alkylated PAH compounds will be established to demonstrate the linear range of the analysis. A calibration check standard will be analyzed in order to monitor instrument response relative to the initial calibration. Quantification of individual components will be performed by the method of internal standards using relative response factors (RRF) for individual components relative to the internal standards acenaphthene-d_{10}, phenanthrene-d_{10}, and benzo[*a*]pyrene-d_{12}. PAH alkyl homologues will be quantified using the straight baseline integration of each level of alkylation and the RRF of the respective parent PAH compound (Table 5-1).

The fresh Orimulsion sample will be analyzed by GC/MS using a Hewlett-Packard 5890 gas chromatograph equipped with a Hewlett-Packard 5970 mass selective detector operating in the selected ion monitoring (SIM) mode. A 2 µL aliquot of the sample will be injected into a gas chromatograph equipped with a high-resolution capillary column (J&W fused silica DB-5-MS, 30-m, 0.25-mm i.d., and 0.25-µm film thickness) operated in the splitless mode.

5.1.7 Volatile Organic Compounds (VOC)

The fresh Orimulsion will also be analyzed for target volatile organic compounds (VOC; Table 5-1) using modified EPA 8260 purge and trap techniques, utilizing a GC/MS system equipped with a 50-m Petrocol® capillary column. Prior to sample analysis, the instrument will be tuned with PFTBA and a minimum three-point calibration will be performed to demonstrate the linear range of the analysis. The calibration solution is composed of selected VOCs identified in Table 5-1.

Table 5-1. Target analyte list for characterization of fresh Orimulsion.

GC/MS Target Analytes

Naphthalene
C_1-naphthalenes
C_2-naphthalenes
C_3-naphthalenes
C_4-naphthalenes
Biphenyl
Acenaphthylene
Dibenzofuran
Acenaphthene
Fluorene
C_1-fluorenes
C_2-fluorenes
C_3-fluorenes
Anthracene
Phenanthrene
C_1-phenanthrenes/anthracenes
C_2-phenanthrenes/anthracenes
C_3-phenanthrenes/anthracenes
C_4-phenanthrenes/anthracenes
Dibenzothiophene
C_1-dibenzothiophenes
C_2-dibenzothiophenes
C_3-dibenzothiophenes
C_4-dibenzothiophenes
Fluoranthene
Pyrene
C_1-fluoranthenes/pyrenes
C_2-fluoranthenes/pyrenes
C_3-fluoranthenes/pyrenes
Benz[*a*]anthracene
Chrysene
C_1-chrysenes
C_2-chrysenes
C_3-chrysenes
C_4-chrysenes
Benzo[*b*]fluoranthene
Benzo[*k*]fluoranthene
Benzo[*e*]pyrene
Benzo[*a*]pyrene
Perylene
Indeno[1,2,3-*c,d*]pyrene
Dibenz[*a,h*]anthracene
Benzo[*g,h,i*]perylene

GC/FID Target Analytes

n-C_8 through *n*-C_{36}
Pristane
Phytane
Isoprenoid 1380
Isoprenoid 1470
Isoprenoid 1650
TPH

Monoaromatic Hydrocarbons (VOCs)

Benzene
Toluene
Ethylbenzene
m-Xylene
p-Xylene
o-Xylene
Isopropylbenzene
n-Propylbenzene
1-Methyl-3-ethylbenzene
1-Methyl-4-ethylbenzene
1-Methyl-2-ethylbenzene
1,3,5-Trimethylbenzene
1,2,4/1,2,3-Trimethylbenzene
sec-Butylbenzene
1-Methyl-3-isopropylbenzene
1-Methyl-4-isopropylbenzene
1-Methyl-2-isopropylbenzene
1-Methyl-3-n-propylbenzene
1-Methyl-4-n-propylbenzene
1,3,dimethyl-5-ethylbenzene
1,2-Diethylbenzene
1,3-Diethylbenzene
1,4-Diethylbenzene
1-Methyl-2-n-propylbenzene
1,4-Dimethyl-2-ethylbenzene
1,2-Dimethyl-4-ethylbenzene
1,3-Dimethyl-2-ethylbenzene
1,3-Dimethyl-4-ethylbenzene
1,2-Dimethyl-3-ethylbenzene
1,2,4,5-Tetramethylbenzene
1,2,3,5-Tetramethylbenzene
1,2,3,4-Tetramethylbenzene

Spiking Compounds and Reporting Limits

GC/MS SIS Compounds

Naphthalene-d_8
Fluorene-d_{10}
Chrysene-d_{12}
Phenol -d_5

GC/FID SIS Compound

o-terphenyl

GC/MS RIS Compounds

Acenaphthene-d_{10}
Phenanthrene-d_{10}
Benzo[*a*]pyrene-d_{12}

Reporting Limits

PAH: 5 mg/kg
VOC: 10 mg/kg
alkanes: 100 mg/kg
TPH: 100,000 mg/kg

5.2 Collection and Analysis of Benchtop Samples

5.2.1 Collection of Samples during Benchtop Experiments

In the course of the benchtop experiments, water samples will be collected for TPH analysis from the vessel sampling ports (Fig. 4-1) at intervals of 1 and 8 hours. The orbital shaker tables will not be turned off during sampling in order to maintain a constant degree of turbulence in the water. Approximately 40 ml water samples will be drawn at each interval from each sample vessel for TPH analysis. Given our experience with some preliminary experiments the reduced quantity of water collected should be sufficient for TPH analysis by gravimetric of GC-FID methods. Some difficulties may occur during the 8-hour sampling when under some circumstances too little bitumen might be still suspended in the water. In these cases the TPH may be below the detection limit. The water sample drawn at the end of the experiments (24 hr) will be larger, consisting of between 1 and 2 L. This volume of sample will permit the sample to be split and still provide enough sample to obtain good concentration data. One half of the water will be used for TPH and total PAH analysis and one half will be used for dissolve PAH analysis. The latter aliquot will be filtered through a 0.45 micron filter prior to analysis in order to remove all but the dissolved materials.

At the 1, 3, 8 and 24-hour intervals, samples will also drawn for bitumen particle size analysis. It should be noted (as is described in greater detail in Section 5.2.2) that for those experiments that include kaolinite particulates, it will not be possible to distinguish <u>bitumen</u> particle size from <u>particulate</u> particle size. In the latter case, particulates will include bitumen, clay, and bitumen/clay conglomerates. The volume of water drawn during each of these sampling events will vary slightly depending upon the concentration of suspended particulates in the water. Our preliminary experiments have shown that early in the experiments as little as 10 ml is sufficient to achieve appropriate obscuration levels during particle size measurement. However, later in the experiment, when very little suspended particulate is present, a larger volume 100 ml may be necessary. Under some circumstances we can imagine that not enough suspended matter will remain in the water to achieve a reliable particle size measurement. In such instances, no values will be reported.

5.2.2 Measurement of Particle Size

Measuring particle size in a mixture can be done by a variety of methods. These include (1) microscopy, (2) sieving and weighing, (3) settling experiments, and (4) laser diffraction. We believe that because of the potential to change bitumen particle size upon changing the environment of the sample, only laser diffraction is appropriate for the bitumen/clay particles that will be formed in the course of these experiments. However, because our preliminary experiments have indicted that particles beyond the range of the Mastersizer X will be formed (> 600 microns), we propose to supplement the laser particle size analysis by qualitative microscopy.

The parameters for the measurement of particulates in the water from the benchtop experiments using the MasterSizer X will be the same as described for fresh Orimulsion (Section 5.1.4). Diameters will be reported as the volume mean diameter (D[4,3]), which refers to the diameter of a sphere having the same volume as the particles being measured. Our preliminary experiments have indicated that there may be multiple populations present in any given sample. If this is the case, then the diameter of each population will be determined separately. Qualitative light microscopy will be used to describe the populations (e.g., clay/calcite-dominant, bitumen-dominant, mixed, etc.) and this may aid in determining which population is most representative of the bitumen that is present.

As noted previously, a complication in the measurement of bitumen particle size lies in the inability to differentiate bitumen particles from clay/calcite particles in the samples. This will be problematic as particulate load is considered to be an important independent variable. We believe this problem can be managed as the real goal of the experiments to be performed is to recognize changes in the size of the bitumen particles. Our preliminary experiments have shown that even with kaolinite in the system the change in the overall particle diameter can be recognized by the change in the distribution of all of the particles in a sample. With microscopy to supplement the laser diffraction analysis we believe a reasonable interpretation of the total distributions (i.e., bitumen and inorganic particles) can be made. However, we believe that it would be inappropriate to use the average diameters of these mixed populations in any attempt to 'model' bitumen particle growth rate. They could only be used to model a total particle growth rate. (Of course, the data from the experiments in which no kaolinite or calcite is added will provide 'bitumen-only' particle diameters, and these results could be used to model a bitumen particle growth rate).

5.2.3 Extraction of Water Samples for TPH and PAH

Water samples collected for TPH and PAH analysis will be extracted according to BOS SOP 5-200. Each batch of samples will include a procedural blank and a blank spike. Samples will be spiked with surrogate internal standards (SIS) prior to extraction according to Table 5-2. Final extracts will be concentrated to a volume of 0.5 mL. All extracts will then be spiked with recovery internal standard (RIS) according to Table 5-3, split, and transferred to the GC/FID and GC/MS task leaders for TPH and PAH analysis, respectively.

Table 5-2. Surrogate Internal Standard (SIS) spiking information.

Standard ID	Contents	Volume to Spike (μL)
EM82	PAH/SHC SIS	50
EM85 (BS only)	Alkanes	50
EM84 (BS only)	PAHs	50

Table 5-3. Recovery Internal Standard (RIS) spiking information.

Standard ID	Contents	Volume to Spike (μL)
EM83	PAH/SHC RIS	50

5.2.4 Measurement of total and dissolved TPH

Analysis of water sample extracts for TPH will be determined by HRGC methods modified from EPA Method 8015 and defined by Battelle SOP 5-202, *Determination of Low Level Total Petroleum Hydrocarbon and Individual Hydrocarbon Concentrations in Environmental Samples*. Prior to sample analysis, a minimum of a five-point calibration will be performed to demonstrate the linear range of the analysis. The calibration solution will be composed of selected normal and iso-alkanes ranging in molecular weight from C_8 through C_{36}. Calibration check standards will be analyzed at least every 10 samples to monitor instrument response relative to the initial calibration. Instrument calibration and other data quality objectives are detailed in SOP 202.

The GC/FID condition for the analysis will be:

Initial Column temperature:	35°C
Initial hold time:	5 minutes
Program rate:	6°C/minute
Final column temperature:	320°C
Final hold time:	10 minutes
Injector temperature:	275°C
Detector temperature:	325°C
Column flow rate:	1 mL/min

The reporting limit for TPH in water will be 20.0 µg/L (ppb).

5.2.5 Measurement of total and dissolved PAH

The analysis of the target PAH compounds will be performed using HRGC/MS methods modified from EPA Method 8270 and defined by Battelle SOP 5-157, *Identification and Quantitation of Polynuclear Aromatic Hydrocarbons (PAH) by Gas Chromatography/Mass Spectrometry*. Prior to sample analysis, the GC/MS will be tuned with perfluorotributylamine (PFTBA) and a minimum of a 5-point initial calibration consisting of the target compounds will be established to demonstrate the linear range of the analysis.

Analyte concentrations in the standard solutions will range from approximately 0.02 to 10 ng/µL. Quantification of individual components will be performed by the method of internal standards using the relative response factors (RRFs) of the parent PAH compounds in the calibration solutions. Final data will be reported vs. the appropriate surrogate compound, following our standard IS designation procedures (only the PAH data will be surrogate corrected). Quantification at the instrument may be vs. the RIS acenaphthene-d_{10} and/or fluorene-d_{10}, followed by surrogate correction of the data in a spreadsheet, or by direct quantification vs. the surrogate compound in the instrument method whichever the analyst deems most efficient. Instrument calibration and other data quality objectives are detailed in SOP 5-157.

The GC/MS condition for the analysis will be:

Initial column temperature:	40°C
Initial hold time	1 minute
Program rate:	6°/minutes
Final column temperature:	290°C
Final hold time:	10 minutes
Injector temperature:	300°C
Detector temperature:	280°C
Column flow rate:	1 mL/min

Electronic pressure control (EPC) conditions:

Vacuum compensation:	On
Pressure at injection:	15 psi
Hold time:	1.50 min.
Pressure program ramp:	99 psi/min.
Final pressure	7.7 psi

Reporting limits for individual PAHs in water will be 1.0 ng/L (ppt).

5.2.6 Measurements for Mass Balance

As described in Section 3.2.2.2, considerable knowledge regarding the fate of Orimulsion can be obtained by determining which five 'compartments' the bitumen ends up in at the end of our benchtop experiments:

(1) floating bitumen/patty,
(2) sunk bitumen/patty,
(3) dissolved in water,
(4) suspended in the water, or
(5) stuck to the vessel walls ("bathtub ring").

There will be a known volume of Orimulsion added to each vessel (determined by the desired concentration). Approximately 73% of this volume will be bitumen and the density of bitumen can be used to determine the mass of bitumen added to each experiment. Each vessel will originally contain 3 L of water and immediately following the addition of the Orimulsion all of the bitumen will be dispersed into the water. After 24 hours the bitumen will occur in one of the five compartments.

After 24 hours any floating patty (e.g., Fig. 5-2) will be recovered using pre-weighed wooden tongs and placed in a hood to dry for a minimum of 24 hours. The weight of the floating bitumen will then be recorded.

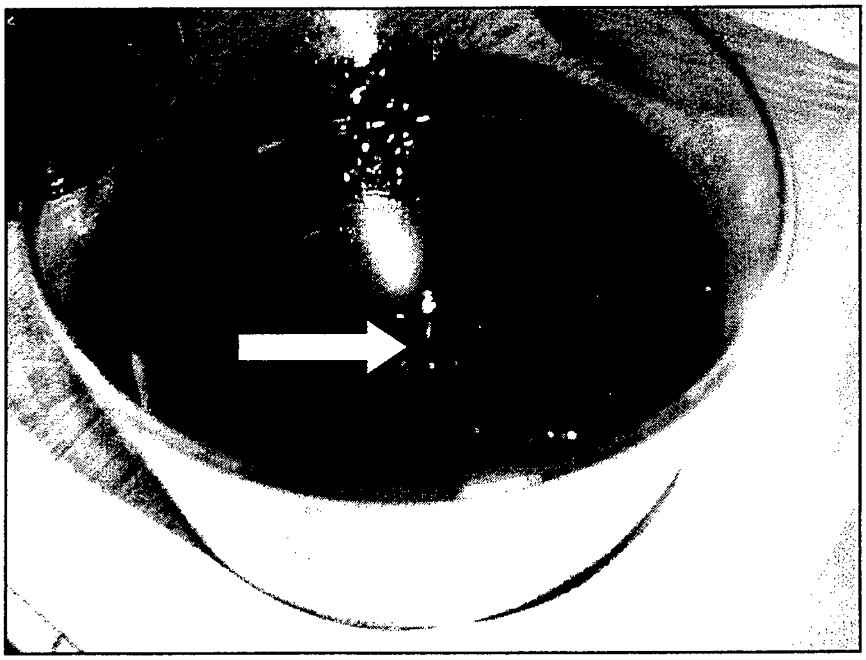

Figure 5-2: Example of floating surface patty (arrow) formed after 24 hours in a preliminary experiment involving 17,000 mg/L Orimulsion in seawater. Note formation of the 'bathtub ring' of bitumen on the glass walls of the vessel.

After 24 hours the water in the vessel, a slightly reduced volume from the original 3L due to the 1, 3 and 8 hours samplings, will be drained through the sampling port into an appropriate container in preparation for TPH and PAH analysis. The volume of water drained will be recorded. The thoroughly mixed water sample will be split for dissolved and total TPH and PAH analysis as described in Sections 5.2.3 and 5.2.4. The "dissolved" split will be filtered through pre-weighed 0.45 micron filter and analyzed to determine the concentration of dissolved TPH in the water (Compartment 3). The mass of dissolved material (Compartment 4) will be calculated given the total volume of water drained. The filter will be dried and weighed in order to determine the weight of total (bitumen and perhaps clay) suspended material in the water. The "total" water split will be extracted and analyzed as described above. The concentration of suspended and dissolved TPH will be determined and the total mass will be calculated given the total volume of water drained. The mass of suspended TPH (Compartment 4) will be determined by the difference between the total and dissolved masses.

After draining the water, if a sinking patty is present it will be collected, dried, and weighed as described above. If no patty is present, only bitumen particles these will be recovered. The method of recovering this may vary but our preliminary experiments have shown that this material, mostly a mixture of bitumen and clay, can be rinsed from the bottom of the vessel with a squirt bottle into a pre-weighed beaker. This bitumen/sediment mixture will be dried and weighed. The calculated weight of the sediment (mineral matter) will be subtracted from the total "sunk" weight to yield the mass of the sunk bitumen (Compartment 2).

Finally the now "empty" vessel, having been pre-weighed prior to the experiment, can be re-weighed in order to determine the mass of bitumen that occurs stuck to the vessel (Compartment 5). Our preliminary experiments have indicated that this material will only occur at the water-air interface in the form of a 'bathtub ring'.

The collective total of the masses of the bitumen in each compartment should approach the total mass of bitumen spilled in each experiment. These results will be compared for the given experimental conditions in order to determine the influence of the independent variables on the fate of Orimulsion.

5.2.7 Reporting of Results

All analytical data generated as part of this study will be reviewed by Battelle's Quality Assurance Unit (QAU). Data will be subjected to a targeted audit prior to delivery to the client. Data will be reviewed by the Project Managers for chemical reasonableness. All project deliverables will be subjected to peer review for technical and editorial content. Prior to issuance the final Phase II and Phase III reports and supporting documentation (e.g. relevant figures, tables and data) will undergo a QA audit.

A report summarizing the results of the benchtop experiments will be issued at the completion of Phases II and III. The Phase II report will include a discussion of the effects of the five independent variables (Table 3-1) on the various dependent variables (Table 3-2) and on the mass balance results as determined from the benchtop studies. This report will also include the results of our flume energetic study and our recommendations for Phase III's meso-scale flume experiments generally described below.

6. PHASE III - MESO-SCALE FLUME TANK EXPERIMENTS

6.1 Introduction

The meso-scale experiments comprising Phase III of this study will be conducted in Battelle's 4000 L flume tank under appropriate energy conditions (see below). The specifications for the tank have been described elsewhere (Fredriksson et al., 1996). The tank will be prepared for the weathering experiments by filling it with the desired amount and type of water. The initial water to be used will be (1) seawater from Duxbury Bay, MA that will be acquired with Battelle's seawater system and/or (2) fresh water obtained from Battelle's fresh water well. The target salinity will be obtained by mixing these two water sources and/or by the addition of Instant Ocean^{TM} sea salts. The salinity will be monitored with a conductivity meter calibrated for the temperature of the water. Both seawater and freshwater will be filtered to 5 microns prior to its use in the flume experiments.

The temperature conditions that are ultimately decided for the flume experiments will be set for the flume room. The temperature of the water added to the tank will be monitored until it reaches the temperature set for the room.

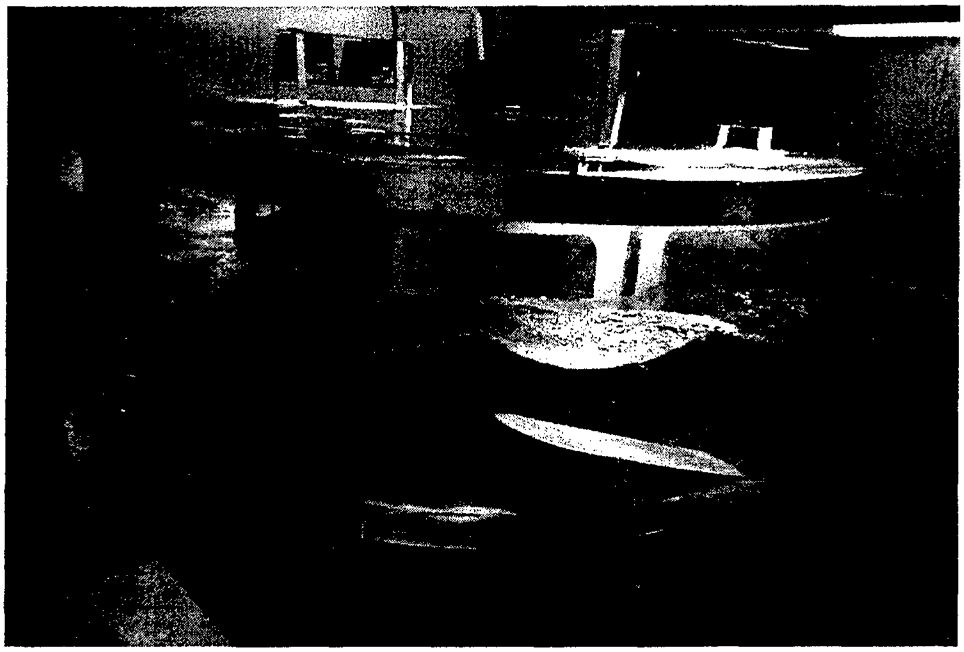

Figure 6-1: Battelle's 4000 L flume tank showing conditions producing spilling breaking waves. The flume has a track length of 9.1 m and a depth of 0.8 m. Fifteen wind vanes can generate up to 25 m/sec wind speed and the wedge-shaped plunger (foreground) stroke ranges from 2-30 cm at rates up to 100 cpm.

6.2 Sample Collection and Preparation

Once the flume experiments are initiated, samples of Orimulsion within the water column will be taken at pre-set times for analysis according to the schedule and analyses summarized in Table 6-1. Naturally, the schedule and analysis may be changed after considering the Phase II results. The parameters to be measured in the course of these experiments will likely parallel those made at the benchtop scale (Table 3-2) and those from previous flume experiments on Orimulsion (Ostazeski et al., 1998).

At the indicated sampling times following introduction of the Orimulsion, water samples will be collected using a stainless steel hand pump. The samples will be collected from a depth of approximately 35 cm below the water surface and mid channel in the flume track. The water column samples will be prepared for the appropriate analyses according to standard operating procedures described in Section 5-2.

Table 6-1. Possible Sampling and Testing Scheme for Orimulsion® during Phase III Flume Testing

	Floating/Sinking Bitumen			Unfiltered Water				Filtered Water	
Time (hours)	Viscosity	Density	TPH Analysis (sediment)	SARA Analysis (Iatroscan)	TPH Analysis	PAH Analysis	Particle Size	TPH Analysis	PAH Analysis
0	—	—	—	✓	✓	✓	✓	✓	✓
15 min	—	—	—	✓	✓	✓	✓	✓	✓
30 min	—	—	—	✓	✓	✓	✓	✓	✓
1	—	—	—	✓	✓	✓	✓	✓	✓
2	—	—	—	✓	✓	✓	✓	✓	✓
4	—	—	—	✓	✓	✓	✓	✓	✓
8	—	—	—	✓	✓	✓	✓	✓	✓
12	—	—	—	✓	✓	✓	✓	✓	✓
24	—	—	—	✓	✓	✓	✓	✓	✓
48	—	—	—	✓	✓	✓	✓	✓	✓
72	—	—	—	✓	✓	✓	✓	✓	✓
96	—	—	—	✓	✓	✓	✓	✓	✓
120	—	—	—	✓	✓	✓	✓	✓	✓
144	—	—	—	✓	✓	✓	✓	✓	✓
168	✓	✓	✓	✓	✓	✓	✓	✓	✓

6.3 Phase IIC – Evaluation and Quantification of Energy within the Flume Tank

The quantification of the energy conditions prior to the spilling of any Orimulsion in the flume tank is a separate and integral part of this study. This will be particularly true if the benchtop studies reveal that energetics plays a very important role in the fate of Orimulsion spilled on water. The problem remains separate, however, because translating the kinetic energy levels from the benchtop to the meso-scale will be essentially impossible.

The conditions of the earlier Orimulsion studies (Ostazeski et al., 1997a; 1997b; 1998) were arbitrarily set to match those previously used by IKU (Singsaas et al., 1995). The 'energetics' of the previous studies conducted in Battelle's flume tank were all performed under these conditions which were described by Fredriksson et. al. (1996). In that study the following data were generated; (1) wave height measurements using a capacitance wave probe and (2) current velocity measurements at the center of the channel at six depths using acoustic Doppler velocimeters (ADV) and neutrally-buoyant styrene beads. These measurements were made with the plunger and wind vane operating at the same levels as in the previous Orimulsion spill experiments, i.e., wind vanes at 80 cpm and plunger at 50 cpm with a 12 cm stroke (Ostazeski et al., 1997a; 1997b; 1998). The energy density under these conditions was determined to be 22.39 Joules/m^2, which is well within the energy density estimates for open ocean sea-state 2 conditions (10.48 to 298.61 Joules/m^2).[e] Stokes second order wave theories were used to determine the mean vertical, radial and tangential current, wave and turbulent velocity components under these conditions.

Concern was recently expressed that the earlier work performed on Orimulsion, in fact, on all previously spilled oils, were conducted at energies that were too high. The basis for this concern was the fact that in the event of a real spill, there is normally a decrease in the turbulent mixing with depth (P. Masciangioli, 1997, personal communication). However, it was observed that in the flume comparable turbulence

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[e] Note that without the plunger operating the energy was only 7.06 Joules/m^2.

existed throughout the entire flume water depth leading to the complete mixing of the water mass. This was suggested to be the reason that, during the freshwater experiments, the bitumen was not observed to settle out of the water. Perhaps the simplest solution to this concern would be to eliminate the use of the plunger apparatus since it is presumably responsible for introducing most of the energy into the deeper parts of the flume tank.

The "flume energetics" aspect of the proposed study undoubtedly requires additional discussions between Bitor, NOAA, USCG and Battelle personnel. We anticipate the scope of this phase of the study to be determined collectively during our meeting March 19, 1998 in Duxbury. Both the technical and financial limitations of the study must be kept in mind throughout these discussions.

It is our original proposal to perform a series of measurements in the flume tank under a variety of wind vane and plunger settings. We propose to measure:

(1) wave height,

(2) wave period, and

(3) wind speed.

It is our opinion that these gross measurements may allow for an easier 'translation' of real world conditions and if particle velocities were measured and modeled. Therefore, we do not intend to measure current velocity nor calculate current, wave or turbulence component velocities. Instead, we would simply use the matrix of wind vane/plunger conditions *versus* wave height, wave period and wind sped data to select the appropriate wind vane and plunger settings to be used for the meso-scale studies. We would propose to acquire this data at the same time that the benchtop studies would be being conducted. We would seek agreement from Bitor and USGC representative on the conditions under which the meso-scale studies should be conducted in order to more closely match real world conditions.

After receiving additional comments on this workplan, we have agreed to consider including a study by which the turbulent diffusity through the flume can be investigated for a small number of wind/wave conditions or settings. The data from these investigations could be used by NOAA and Intevep representatives to aid in determining the wind/wave conditions under which to conduct the flume experiments and allow for modeling the results in open water conditions. This study will involve the use of inert particles of a known diameter and density. (The nature of these materials is yet to be determined and we expect to receive some guidance from NOAA on an appropriate product to use). Let's assume these are slightly buoyant particles. The buoyant particles will be added to the flume tank for a given wind/wave condition. The turbulent energy within the tank will drive them down into the tank and distribute the particles throughout the water column. Buoyant forces will attempt to drive them back up, but turbulence will keep some down. These forces should result in a fairly "steady state" at which the concentration of particles decreases with increasing depth. Using video or photography, the number of particles at a given depth range will be determined as accurately as possible. The number density of particles at discrete depths within the tank can be used to calculate the turbulent diffusivity according to the following equation:

$$dN/dt = D\ (d^2N/dz^2) - U\ (dN/dz)$$

Where D=turbulent diffusivity, N = number density of particles, n = number of particles, U = Stokes velocity, z = depth.

If still considered to be important enough to justify the additional costs, and some technical guidance becomes available regarding the selection of 'inert particles', we will attempt to collect number density data for a small number of wind/wave conditions that will allow some capacity to translate the flume energetics to open water energetics. Battelle's role in this phase of the study is limited to collection of data under guidance from NOAA/USGC and Bitor/Intevep representatives. The costs for this additional component of the study are not included in Section 7.

7. TIME CONSIDERATIONS

This workplan constitutes the deliverable for Phase I of this project. Table 7-1 summarizes the tasks that will be performed in both Phases II and III. Because of the dynamic character of Phases II and III Battelle will provide monthly updates regarding the progress and costs accrued throughout the project. This will allow constant re-assessment of the direction and scope of subsequent phases.

Table 7-1: Inventory of Tasks to be Performed in each Phase of the Study

PHASES AND TASKS
Phase I - Design of Behavior Characterization Experiments
Planning Meeting
Literature Review
Technical Meetings
Evaluation of particle size analyzers
Development of Design of Experiment
Development Benchtop Prototype
Conducting Preliminary Benchtop Experiments
Preparation of Phase I deliverable
Phase II - Evaluate Fate and Determine Conditions for Meso-Scale Testing
Phase IIA - Conduct Benchtop Experiments
analytical costs - fresh Orimulsion
construct benchtop apparatus, purchase materials
perform 12 screening design experiments
Report screening results
perform up to 47 three-level response surface design experiments
analytical costs - dependent variables and mass balance
Phase IIB - Data Interpretation
DOE evaluation using JMP software
Phase IIC - Energetic Study
Construct wind speed/wave height/wave frequency matrix
Phase II Report Preparation with Recommendations for Phase III
Review Meeting - Duxbury
Phase III - Meso-Scale Flume Tank Tests
Conduct 4 or 5 meso-scale flume tests
Analytical Costs - dependent variables of interest in Phase II
Phase III Report with Final Section on Synthesis of Project

8. REFERENCES

Brown, J.W., Fuentes, H.R., Jaffe, R. and Tsihrintzis, V.A., 1995, Chapter 4 - Comparative evaluation of physical and chemical fate processes of Orimulsion and fuel oil no. 6 in the Tampa Bay marine environment. In, Comparative Ecological Risk Assessment – Final Report. June 1995. Center for Marine and Environmental Analysis, Univ. of Miami.

Delvigne, G.A.L., 1993, Natural dispersion of oil by different sources of turbulence. Proc. 1993 Oil Spill Conference, p. 415.

Febres, G.A., Goncalves, J.A., Masciangioli, P, and Vilas, J., 1995, Fate and behavior of Orimulsion spilt in sea water. Internal Report, Intervep, S.A.

Fredriksson, D.W., Albro, C.S., and Ostazeski, S.A., 1996, Hydrodynamic calibration of a meso-scale, wind-wave flume basin for the study of spilled oil. Proc. 19th Annual Artic and Marine Oil Spill Program, Calgary, Alberta. Pp. 1541-1553.

Hicks, C.R., 1982, Fundamental Concepts in the Design of Experiments. 3rd Edition., Holt, Rinehart and Winston, New York.

Marshall, C.F. 1964. The Physical Chemistry and Mineralogy of Soils, Vol. 1: Soil Materials. J. Wiley & Sons, Inc., New York.

Milliman, J.D., 1990, Fluvial sediment in coastal seas: flux and fate. Nature and Resources 26(4):10-26.

Ostazeski, S.A., Macomber, S.C., Roberts, L.G. and Uhler, A.D., 1997a, Micro- and meso-scale methods for predicting the behavior of low API gravity oils (LAPIO) spilled on water. Report to VOLPE Nat'l. Transportation Systems.

Ostazeski, S.A., Macomber, S.C., Roberts, L.G., and Uhler, A.D., 1997b, The environmental behavior of Orimulsion spilled on water. Proc. 1997 Int'l. Oil Spill Conf., API Publ. No. 4651, Washington, D.C., p. 469-477.

Ostazeski, S.A., Stout, S.A., and Uhler, A.D., 1998, Testing and characterization of Orimulsion 400. Report to Bitor America.

Potter, T.L., 1995, Chapter 14 - Chemistry of fuel oil #6 and Orimulsion. In, Comparative Ecological Risk Assessment – Final Report. June 1995. Center for Marine and Environmental Analysis, Univ. of Miami.

Riley, J.P. and Chester, R., 1971, Introduction to Marine Chemistry. Acad. Press, Inc., New York.

Singsaas, I., Reed, M., Daling, P.S and Lewis, A., 1995, Mesoscale flume calibration and testing. Report submitted to Esso Norge by IKU Sintef Group.

APPENDIX II

Plackett-Burman Design Factor Effect Tables

Trial	Mean Dependent Variable Mean	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					Particle Size 1 h
		X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	NA
2		35	4	17000	270	kaol	10	-	-	+	-	+	46.37
3		0	25	17000	270	calc	10	-	+	-	+	+	20.36
4		35	25	17000	0	kaol	10	+	-	+	+	-	NA
5		35	25	1000	0	calc	100	-	+	+	-	+	20.40
6		35	4	1000	0	kaol	10	+	+	-	+	+	NA
7		0	4	1000	270	calc	100	+	-	+	+	+	48.31
8		0	4	17000	0	calc	100	-	+	+	+	-	23.23
9		0	25	1000	270	kaol	10	+	+	+	-	-	22.80
10		35	4	17000	270	calc	100	+	+	-	-	-	98.34
11		0	25	17000	0	calc	100	+	-	-	-	+	19.76
12		0	4	1000	0	kaol	10	-	-	-	-	-	16.57
Sum +	316.14	165.11	83.32	208.06	236.18	230.40	210.04	189.21	185.13	161.11	91.90	155.20	
Sum -	0.00	151.03	232.82	108.08	79.96	85.74	106.10	126.93	131.01	155.03	224.24	160.94	
Check Sum	316.14	316.14	316.14	316.14	316.14	316.14	316.14	316.14	316.14	316.14	316.14	316.14	
Difference	316.14	14.08	-149.50	99.98	156.22	144.66	103.94	62.28	54.12	6.08	-132.34	-5.74	
Factor Effect	26.35	2.35	-24.92	16.66	26.04	24.11	17.32	10.38	9.02	1.01	-22.06	-0.96	
Lower CL		-27.57	-54.83	-13.25	-3.88	-5.81	-12.59	-19.54	-20.90	-28.90	-51.97	-30.87	
Upper CL		32.26	5.00	46.58	55.95	54.03	47.24	40.30	38.94	30.93	7.86	28.96	

S_{FE} = 11.64

tS_{FE} = 29.92

Trial	Mean Dependent Variable Mean	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					Particle Size 3 h
		X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	NA
2		35	4	17000	270	kaol	10	-	-	+	-	+	54.17
3		0	25	17000	270	calc	10	-	+	-	+	+	17.85
4		35	25	17000	0	kaol	10	+	-	+	+	-	NA
5		35	25	1000	0	calc	100	-	+	+	-	+	18.35
6		35	4	1000	0	kaol	10	+	+	-	+	+	NA
7		0	4	1000	270	calc	100	+	-	+	+	+	15.78
8		0	4	17000	0	calc	100	-	+	+	+	-	17.00
9		0	25	1000	270	kaol	10	+	+	+	-	-	17.37
10		35	4	17000	270	calc	100	+	+	-	-	-	58.29
11		0	25	17000	0	calc	100	+	-	-	-	+	22.92
12		0	4	1000	0	kaol	10	-	-	-	-	-	11.87
Sum +	233.60	130.81	76.49	170.23	163.46	150.19	132.34	114.36	128.86	122.67	50.63	129.07	
Sum -	0.00	102.79	157.11	63.37	70.14	83.41	101.26	119.24	104.74	110.93	182.97	104.53	
Check Sum	233.60	233.60	233.60	233.60	233.60	233.60	233.60	233.60	233.60	233.60	233.60	233.60	
Difference	233.60	28.02	-80.62	106.86	93.32	66.78	31.08	-4.88	24.12	11.74	-132.34	24.54	
Factor Effect	19.47	4.67	-13.44	17.81	15.55	11.13	5.18	-0.81	4.02	1.96	-22.06	4.09	
Lower CL		-21.64	-39.74	-8.50	-10.75	-15.18	-21.13	-27.12	-22.29	-24.35	-48.36	-22.22	
Upper CL		30.98	12.87	44.12	41.86	37.44	31.49	25.49	30.33	28.26	4.25	30.40	

S_{FE} = 10.24

tS_{FE} = 26.31

Trial	Mean Dependent Variable Mean	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					Particle Size 24 h
		X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	NA
2		35	4	17000	270	kaol	10	-	-	+	-	+	365.17
3		0	25	17000	270	calc	10	-	+	-	+	+	7.85
4		35	25	17000	0	kaol	10	+	-	+	+	-	NA
5		35	25	1000	0	calc	100	-	+	+	-	+	NA
6		35	4	1000	0	kaol	10	+	+	-	+	+	NA
7		0	4	1000	270	calc	100	+	-	+	+	+	2.01
8		0	4	17000	0	calc	100	-	+	+	+	-	15.62
9		0	25	1000	270	kaol	10	+	+	+	-	-	9.21
10		35	4	17000	270	calc	100	+	+	-	-	-	4.73
11		0	25	17000	0	calc	100	+	-	-	-	+	12.61
12		0	4	1000	0	kaol	10	-	-	-	-	-	7.76
Sum +	424.96	369.90	29.67	405.98	388.97	42.82	34.97	28.56	37.41	392.01	25.48	387.64	
Sum -	0.00	55.06	395.29	18.98	35.99	382.14	389.99	396.40	387.55	32.95	399.48	37.32	
Check Sum	424.96	424.96	424.96	424.96	424.96	424.96	424.96	424.96	424.96	424.96	424.96	424.96	
Difference	424.96	314.84	-365.62	387.00	352.98	-339.32	-355.02	-367.84	-350.14	359.06	-374.00	350.32	
Factor Effect	35.41	52.47	-60.94	64.50	58.83	-56.55	-59.17	-61.31	-58.36	59.84	-62.33	58.39	
Lower CL		-101.90	-215.31	-89.87	-95.54	-210.92	-213.54	-215.68	-212.73	-94.53	-216.70	-95.98	
Upper CL		206.84	93.43	218.87	213.20	97.82	95.20	93.06	96.01	214.21	92.04	212.76	

S_{FE} = 60.07

tS_{FE} = 154.37

Trial	Mean Dependent Variable Mean	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					Particle Size Size 8 h
		X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	NA
2		35	4	17000	270	kaol	10	-	-	+	-	+	293.80
3		0	25	17000	270	calc	10	-	+	-	+	+	11.10
4		35	25	17000	0	kaol	10	+	-	+	+	-	NA
5		35	25	1000	0	calc	100	-	+	+	-	+	NA
6		35	4	1000	0	kaol	10	+	+	-	+	+	NA
7		0	4	1000	270	calc	100	+	-	+	+	+	5.12
8		0	4	17000	0	calc	100	-	+	+	+	-	20.30
9		0	25	1000	270	kaol	10	+	+	+	-	-	13.25
10		35	4	17000	270	calc	100	+	+	-	-	-	9.10
11		0	25	17000	0	calc	100	+	-	-	-	+	17.96
12		0	4	1000	0	kaol	10	-	-	-	-	-	7.13
Sum +	377.76	302.90	42.31	352.26	332.37	63.58	52.48	45.43	53.75	332.47	36.52	327.98	
Sum -	0.00	74.86	335.45	25.50	45.39	314.18	325.28	332.33	324.01	45.29	341.24	49.78	
Check Sum	377.76	377.76	377.76	377.76	377.76	377.76	377.76	377.76	377.76	377.76	377.76	377.76	
Difference	377.76	228.04	-293.14	326.76	286.98	-250.60	-272.80	-286.90	-270.26	287.18	-304.72	278.20	
Factor Effect	31.48	38.01	-48.86	54.46	47.83	-41.77	-45.47	-47.82	-45.04	47.86	-50.79	46.37	
Lower CL		-84.36	-171.22	-67.91	-74.54	-164.13	-167.83	-170.18	-167.41	-74.50	-173.15	-76.00	
Upper CL		160.37	73.51	176.83	170.20	80.60	76.90	74.55	77.32	170.23	71.58	168.73	

S_{FE} = 47.61

tS_{FE} = 122.37

Trial	Mean Dependent Variable Mean	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					TPH in water 1 hr (mg/L)
		X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	204.76
2		35	4	17000	270	kaol	10	-	-	+	-	+	1227.27
3		0	25	17000	270	calc	10	-	+	-	+	+	2045.45
4		35	25	17000	0	kaol	10	+	-	+	+	-	0.95
5		35	25	1000	0	calc	100	-	+	+	-	+	290.00
6		35	4	1000	0	kaol	10	+	+	-	+	+	0.91
7		0	4	1000	270	calc	100	+	-	+	+	+	243.90
8		0	4	17000	0	calc	100	-	+	+	+	-	8625.00
9		0	25	1000	270	kaol	10	+	+	+	-	-	1850.00
10		35	4	17000	270	calc	100	+	+	-	-	-	1350.00
11		0	25	17000	0	calc	100	+	-	-	-	+	6232.56
12		0	4	1000	0	kaol	10	-	-	-	-	-	168.89
Sum +	22239.70	3073.90	10623.73	19481.24	6921.39	18786.92	16946.22	9678.32	14161.36	12237.13	11120.98	10040.10	
Sum -	0.00	19165.80	11615.97	2758.46	15318.31	3452.78	5293.48	12561.38	8078.34	10002.57	11118.72	12199.60	
Check Sum	22239.70	22239.70	22239.70	22239.70	22239.70	22239.70	22239.70	22239.70	22239.70	22239.70	22239.70	22239.70	
Difference	22239.70	-16091.91	-992.25	16722.78	-8396.92	15334.13	11652.74	-2883.06	6083.03	2234.55	2.26	-2159.51	
Factor Effect	1853.31	-2681.98	-165.37	2787.13	-1399.49	2555.69	1942.12	-480.51	1013.84	372.43	0.38	-359.92	
Lower CL		-4102.24	-1585.63	1366.87	-2819.74	1135.43	521.87	-1900.77	-406.42	-1047.83	-1419.88	-1780.18	
Upper CL		-1261.73	1254.88	4207.39	20.77	3975.95	3362.38	939.75	2434.10	1792.68	1420.64	1060.34	

S_{FE} = 552.63

tS_{FE} = 1420.26

Trial	Mean Dependent Variable Mean	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					TPH in water 8 hr (mg/L)
		X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	2.00
2		35	4	17000	270	kaol	10	-	-	+	-	+	737.50
3		0	25	17000	270	calc	10	-	+	-	+	+	452.38
4		35	25	17000	0	kaol	10	+	-	+	+	-	1.05
5		35	25	1000	0	calc	100	-	+	+	-	+	23.81
6		35	4	1000	0	kaol	10	+	+	-	+	+	2.73
7		0	4	1000	270	calc	100	+	-	+	+	+	48.65
8		0	4	17000	0	calc	100	-	+	+	+	-	10581.40
9		0	25	1000	270	kaol	10	+	+	+	-	-	245.00
10		35	4	17000	270	calc	100	+	+	-	-	-	305.00
11		0	25	17000	0	calc	100	+	-	-	-	+	6047.62
12		0	4	1000	0	kaol	10	-	-	-	-	-	79.55
Sum +	18526.68	1072.09	6771.86	18124.95	1790.53	17458.85	17008.47	6650.05	11610.31	11637.41	11088.20	7312.69	
Sum -	0.00	17454.59	11754.82	401.73	16736.15	1067.83	1518.21	11876.63	6916.37	6889.27	7438.47	11213.99	
Check Sum	18526.68	18526.68	18526.68	18526.68	18526.68	18526.68	18526.68	18526.68	18526.68	18526.68	18526.68	18526.68	
Difference	18526.68	-16382.50	-4982.95	17723.22	-14945.62	16391.03	15490.27	-5226.58	4693.95	4748.13	3649.73	-3901.31	
Factor Effect	1543.89	-2730.42	-830.49	2953.87	-2490.94	2731.84	2581.71	-871.10	782.32	791.36	608.29	-650.22	
Lower CL		-4650.15	-2750.23	1034.13	-4410.67	812.10	661.98	-2790.83	-1137.41	-1128.38	-1311.45	-2569.95	
Upper CL		-810.68	1089.24	4873.60	-571.20	4651.57	4501.45	1048.64	2702.06	2711.09	2528.02	1269.52	

S_{FE} = 746.98

tS_{FE} = 1919.73

Trial	Mean Dependent Variable Mean	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					TPH in water 24 hr (mg/L)
		X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	0.64
2		35	4	17000	270	kaol	10	-	-	+	-	+	2159.66
3		0	25	17000	270	calc	10	-	+	-	+	+	629.61
4		35	25	17000	0	kaol	10	+	-	+	+	-	NA
5		35	25	1000	0	calc	100	-	+	+	-	+	0.90
6		35	4	1000	0	kaol	10	+	+	-	+	+	18.13
7		0	4	1000	270	calc	100	+	-	+	+	+	6.35
8		0	4	17000	0	calc	100	-	+	+	+	-	1764.97
9		0	25	1000	270	kaol	10	+	+	+	-	-	77.20
10		35	4	17000	270	calc	100	+	+	-	-	-	26.78
11		0	25	17000	0	calc	100	+	-	-	-	+	647.76
12		0	4	1000	0	kaol	10	-	-	-	-	-	12.31
Sum +	5344	2206	1356	5229	2900	3076	2447	776	2518	4009	2420	3462	
Sum -	0	3138	3988	116	2444	2268	2897	4568	2826	1335	2925	1881	
Check Sum	5344	5344	5344	5344	5344	5344	5344	5344	5344	5344	5344	5344	
Difference	5344	-932	-2632	5113	456	808	-450	-3792	-309	2674	-505	1581	
Factor Effect	445	-155	-439	852	76	135	-75	-632	-51	446	-84	264	
Lower CL		-1101	-1384	-94	-870	-811	-1021	-1578	-997	-500	-1030	-682	
Upper CL		790	507	1798	1022	1081	871	314	894	1391	862	1209	

S_{FE} = 368

tS_{FE} = 946

Trial	Mean Dependent Variable Mean	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					TPH dissolved in water 24 hr
		X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	0.64
2		35	4	17000	270	kaol	10	-	-	+	-	+	2.65
3		0	25	17000	270	calc	10	-	+	-	+	+	19.78
4		35	25	17000	0	kaol	10	+	-	+	+	-	NA
5		35	25	1000	0	calc	100	-	+	+	-	+	0.59
6		35	4	1000	0	kaol	10	+	+	-	+	+	0.48
7		0	4	1000	270	calc	100	+	-	+	+	+	0.46
8		0	4	17000	0	calc	100	-	+	+	+	-	1.99
9		0	25	1000	270	kaol	10	+	+	+	-	-	0.76
10		35	4	17000	270	calc	100	+	+	-	-	-	3.27
11		0	25	17000	0	calc	100	+	-	-	-	+	5.38
12		0	4	1000	0	kaol	10	-	-	-	-	-	0.41
Sum +	36.42	7.64	27.15	33.07	27.56	31.47	12.33	10.35	26.87	6.46	23.36	29.35	
Sum -	0.00	28.78	9.26	3.35	8.86	4.94	24.08	26.07	8.90	29.96	13.06	6.42	
Check Sum	36.42	36.42	36.42	36.42	36.42	36.42	36.42	36.42	35.78	36.42	36.42	35.78	
Difference	36.42	-21.15	17.89	29.73	18.70	26.53	-11.75	-15.71	17.97	-23.50	10.30	22.93	
Factor Effect	3.03	-3.52	2.98	4.95	3.12	4.42	-1.96	-2.62	2.99	-3.92	1.72	3.82	
Lower CL		-11.55	-5.04	-3.07	-4.91	-3.60	-9.98	-10.64	-5.03	-11.94	-6.31	-4.20	
Upper CL		4.50	11.00	12.98	11.14	12.44	6.06	5.40	11.02	4.11	9.74	11.84	

S_{FE} = 3.12

tS_{FE} = 8.02

Trial	Mean Dependent Variable Mean	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					% sunk after 24 hr
		X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	0.25
2		35	4	17000	270	kaol	10	-	-	+	-	+	0.07
3		0	25	17000	270	calc	10	-	+	-	+	+	0.62
4		35	25	17000	0	kaol	10	+	-	+	+	-	NA
5		35	25	1000	0	calc	100	-	+	+	-	+	0.19
6		35	4	1000	0	kaol	10	+	+	-	+	+	0.20
7		0	4	1000	270	calc	100	+	-	+	+	+	0.59
8		0	4	17000	0	calc	100	-	+	+	+	-	0.05
9		0	25	1000	270	kaol	10	+	+	+	-	-	0.92
10		35	4	17000	270	calc	100	+	+	-	-	-	0.05
11		0	25	17000	0	calc	100	+	-	-	-	+	0.25
12		0	4	1000	0	kaol	10	-	-	-	-	-	0.98
Sum +	4.17	0.76	2.23	1.04	2.50	1.75	1.38	2.02	2.04	1.82	1.71	1.93	
Sum -	0.00	3.41	1.94	3.14	1.67	2.42	2.79	2.16	1.89	2.35	2.46	2.00	
Check Sum	4.17	4.17	4.17	4.17	4.17	4.17	4.17	4.17	3.93	4.17	4.17	3.93	
Difference	4.17	-2.65	0.29	-2.10	0.83	-0.66	-1.41	-0.14	0.14	-0.52	-0.76	-0.07	
Factor Effect	0.35	-0.44	0.05	-0.35	0.14	-0.11	-0.23	-0.02	0.02	-0.09	-0.13	-0.01	
Lower CL		-0.62	-0.13	-0.53	-0.04	-0.29	-0.42	-0.20	-0.16	-0.27	-0.31	-0.19	
Upper CL		-0.26	0.23	-0.17	0.32	0.07	-0.05	0.16	0.20	0.09	0.05	0.17	

S_{FE} = 0.07

tS_{FE} = 0.18

Trial	Mean Dependent Variable Mean	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					% float after 24 hr
		X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	0.00
2		35	4	17000	270	kaol	10	-	-	+	-	+	0.40
3		0	25	17000	270	calc	10	-	+	-	+	+	0.27
4		35	25	17000	0	kaol	10	+	-	+	+	-	NA
5		35	25	1000	0	calc	100	-	+	+	-	+	0.00
6		35	4	1000	0	kaol	10	+	+	-	+	+	0.66
7		0	4	1000	270	calc	100	+	-	+	+	+	0.00
8		0	4	17000	0	calc	100	-	+	+	+	-	0.00
9		0	25	1000	270	kaol	10	+	+	+	-	-	0.00
10		35	4	17000	270	calc	100	+	+	-	-	-	0.00
11		0	25	17000	0	calc	100	+	-	-	-	+	0.21
12		0	4	1000	0	kaol	10	-	-	-	-	-	0.00
Sum +	1.53	1.06	0.47	0.88	0.67	0.47	0.21	0.87	0.92	0.40	0.92	1.53	
Sum -	0.00	0.47	1.06	0.66	0.87	1.06	1.33	0.67	0.61	1.13	0.61	0.00	
Check Sum	1.53	1.53	1.53	1.53	1.53	1.53	1.53	1.53	1.53	1.53	1.53	1.53	
Difference	1.53	0.59	-0.59	0.22	-0.20	-0.59	-1.12	0.20	0.32	-0.73	0.32	1.53	
Factor Effect	0.13	0.10	-0.10	0.04	-0.03	-0.10	-0.19	0.03	0.05	-0.12	0.05	0.26	
Lower CL		-0.24	-0.44	-0.30	-0.37	-0.44	-0.52	-0.31	-0.29	-0.46	-0.29	-0.08	
Upper CL		0.44	0.24	0.37	0.31	0.24	0.15	0.37	0.39	0.22	0.39	0.59	

S_{FE} = 0.13

tS_{FE} = 0.34

Trial	Mean Dependent Variable Mean	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					% stuck to sides after 24 hr
		X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	0.75
2		35	4	17000	270	kaol	10	-	-	+	-	+	0.21
3		0	25	17000	270	calc	10	-	+	-	+	+	0.01
4		35	25	17000	0	kaol	10	+	-	+	+	-	NA
5		35	25	1000	0	calc	100	-	+	+	-	+	0.80
6		35	4	1000	0	kaol	10	+	+	-	+	+	0.12
7		0	4	1000	270	calc	100	+	-	+	+	+	0.40
8		0	4	17000	0	calc	100	-	+	+	+	-	0.77
9		0	25	1000	270	kaol	10	+	+	+	-	-	0.00
10		35	4	17000	270	calc	100	+	+	-	-	-	0.95
11		0	25	17000	0	calc	100	+	-	-	-	+	0.47
12		0	4	1000	0	kaol	10	-	-	-	-	-	0.00
Sum +	4.48	2.84	2.04	2.40	2.32	3.40	4.14	1.94	2.66	2.18	2.06	2.02	
Sum -	0.00	1.65	2.45	2.08	2.16	1.08	0.34	2.54	1.07	2.30	2.43	1.71	
Check Sum	4.48	4.48	4.48	4.48	4.48	4.48	4.48	4.48	3.73	4.48	4.48	3.73	
Difference	4.48	1.19	-0.41	0.32	0.15	2.32	3.80	-0.60	1.58	-0.13	-0.37	0.30	
Factor Effect	0.37	0.20	-0.07	0.05	0.03	0.39	0.63	-0.10	0.26	-0.02	-0.06	0.05	
Lower CL		-0.14	-0.41	-0.28	-0.31	0.05	0.30	-0.44	-0.07	-0.36	-0.40	-0.29	
Upper CL		0.54	0.27	0.39	0.36	0.72	0.97	0.24	0.60	0.32	0.28	0.39	

S_{FE} = 0.13

tS_{FE} = 0.34

Trial	Mean Dependent Variable Mean	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					% suspended in water 24 hr
		X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	0.00
2		35	4	17000	270	kaol	10	-	-	+	-	+	0.32
3		0	25	17000	270	calc	10	-	+	-	+	+	0.10
4		35	25	17000	0	kaol	10	+	-	+	+	-	NA
5		35	25	1000	0	calc	100	-	+	+	-	+	0.00
6		35	4	1000	0	kaol	10	+	+	-	+	+	0.02
7		0	4	1000	270	calc	100	+	-	+	+	+	0.01
8		0	4	17000	0	calc	100	-	+	+	+	-	0.18
9		0	25	1000	270	kaol	10	+	+	+	-	-	0.08
10		35	4	17000	270	calc	100	+	+	-	-	-	0.00
11		0	25	17000	0	calc	100	+	-	-	-	+	0.07
12		0	4	1000	0	kaol	10	-	-	-	-	-	0.02
Sum +	0.80	0.34	0.25	0.68	0.51	0.37	0.27	0.18	0.38	0.59	0.31	0.52	
Sum -	0.00	0.46	0.55	0.12	0.29	0.44	0.53	0.63	0.42	0.21	0.50	0.28	
Check Sum	0.80	0.80	0.80	0.80	0.80	0.80	0.80	0.80	0.80	0.80	0.80	0.80	
Difference	0.80	-0.12	-0.30	0.57	0.21	-0.07	-0.27	-0.45	-0.04	0.38	-0.19	0.24	
Factor Effect	0.07	-0.02	-0.05	0.09	0.04	-0.01	-0.04	-0.08	-0.01	0.06	-0.03	0.04	
Lower CL		-0.15	-0.18	-0.03	-0.09	-0.14	-0.17	-0.20	-0.13	-0.06	-0.16	-0.09	
Upper CL		0.11	0.08	0.22	0.16	0.12	0.08	0.05	0.12	0.19	0.10	0.17	
S_{FE} =	0.05												
tS_{FE} =	0.13												

Trial	Mean Dependent Variable	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					% Dissolved (total) after 24 hr
	Mean	X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	0.0007
2		35	4	17000	270	kaol	10	-	-	+	-	+	0.0004
3		0	25	17000	270	calc	10	-	+	-	+	+	0.0032
4		35	25	17000	0	kaol	10	+	-	+	+	-	NA
5		35	25	1000	0	calc	100	-	+	+	-	+	0.0005
6		35	4	1000	0	kaol	10	+	+	-	+	+	0.0004
7		0	4	1000	270	calc	100	+	-	+	+	+	0.0005
8		0	4	17000	0	calc	100	-	+	+	+	-	0.0002
9		0	25	1000	270	kaol	10	+	+	+	-	-	0.0008
10		35	4	17000	270	calc	100	+	+	-	-	-	0.0002
11		0	25	17000	0	calc	100	+	-	-	-	+	0.0006
12		0	4	1000	0	kaol	10	-	-	-	-	-	0.0006
Sum +	0.0083	0.0023	0.0059	0.0047	0.0058	0.0053	0.0028	0.0025	0.0054	0.0024	0.0051	0.0057	
Sum -	0.0000	0.0059	0.0023	0.0036	0.0024	0.0030	0.0055	0.0058	0.0021	0.0059	0.0032	0.0018	
Check Sum	0.0083	0.0083	0.0083	0.0083	0.0083	0.0083	0.0083	0.0083	0.0075	0.0083	0.0083	0.0075	
Difference	0.0083	-0.0036	0.0036	0.0011	0.0034	0.0023	-0.0027	-0.0033	0.0033	-0.0035	0.0018	0.0038	
Factor Effect	0.0007	-0.0006	0.0006	0.0002	0.0006	0.0004	-0.0004	-0.0005	0.0005	-0.0006	0.0003	0.0006	
Lower CL		-0.0020	-0.0008	-0.0012	-0.0008	-0.0010	-0.0018	-0.0019	-0.0008	-0.0020	-0.0011	-0.0007	
Upper CL		0.0008	0.0020	0.0016	0.0019	0.0018	0.0009	0.0008	0.0019	0.0008	0.0017	0.0020	

S_{FE} = 0.0005

tS_{FE} = 0.0014

Trial	Mean Dependent Variable	Salinity (ppt)	Temp. (°C)	Orim. conc. (mg/L)	Part. Loading (mg/L)	Part. Type	Energetics (rpm)	UNASSIGNED					Dissolved PAH (ug/L) after 24 hr
	Mean	X1	X2	X3	X4	X5	X6	X7	X8	X9	X10	X11	
1		35	25	1000	270	kaol	100	-	-	-	+	-	315.75
2		35	4	17000	270	kaol	10	-	-	+	-	+	2997.78
3		0	25	17000	270	calc	10	-	+	-	+	+	86418.26
4		35	25	17000	0	kaol	10	+	-	+	+	-	NA
5		35	25	1000	0	calc	100	-	+	+	-	+	287.64
6		35	4	1000	0	kaol	10	+	+	-	+	+	4149.12
7		0	4	1000	270	calc	100	+	-	+	+	+	5104.11
8		0	4	17000	0	calc	100	-	+	+	+	-	12008.47
9		0	25	1000	270	kaol	10	+	+	+	-	-	8712.17
10		35	4	17000	270	calc	100	+	+	-	-	-	4389.93
11		0	25	17000	0	calc	100	+	-	-	-	+	26192.70
12		0	4	1000	0	kaol	10	-	-	-	-	-	5722.73
Sum +	156299	12140	121927	132007	107938	134401	48299	48548	115966	29110	107996	125150	
Sum -	0	144158	34372	24292	48361	21898	108000	107751	40017	127188	48303	30833	
Check Sum	156299	156299	156299	156299	156299	156299	156299	156299	155983	156299	156299	155983	
Difference	156299	-132018	87554	107716	59577	112504	-59701	-59203	75948	-98078	59693	94316	
Factor Effect	13025	-22003	14592	17953	9930	18751	-9950	-9867	12658	-16346	9949	15719	
Lower CL		-55921	-19325	-15965	-23988	-15167	-43868	-43785	-21259	-50264	-23969	-18198	
Upper CL		11914	48510	51870	43847	52668	23967	24050	46576	17571	43866	49637	

S_{FE} = 13197.48

tS_{FE} = 33917.53

APPENDIX III

Response Surface Design Model Calculations

ɜcreening Fit

PS 1

Summary of Fit

RSquare	0.759198
RSquare Adj	0.548497
Root Mean Square Error	42.63504
Mean of Response	73.49548
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	14	91695.78	6549.70	3.6032
Error	16	29083.95	1817.75	Prob>F
C Total	30	120779.73		0.0081

Lack of Fit

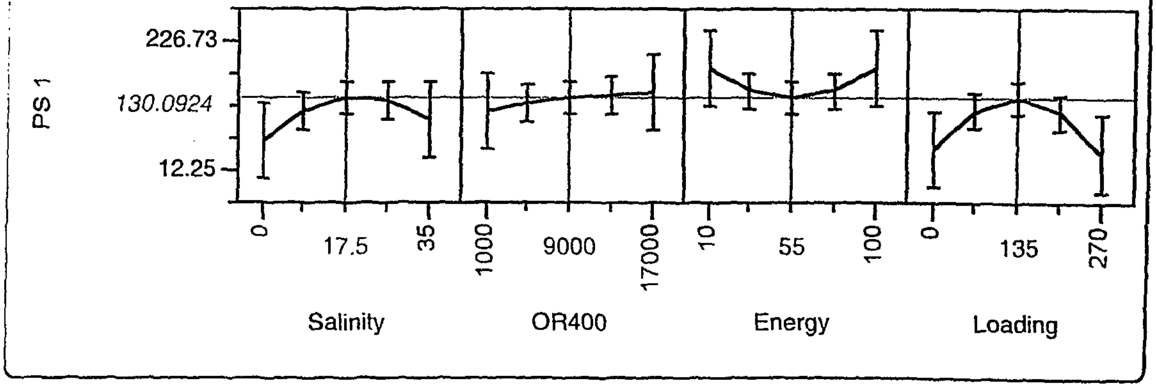
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	40.498531	41.96349	0.97	0.3489
Salinity	6.2048185	3.297419	1.88	0.0782
OR400	0.0023283	0.007949	0.29	0.7734
Energy	-2.864445	1.516547	-1.89	0.0772
Loading	1.297812	0.427443	3.04	0.0079
Salinity*Salinity	-0.170765	0.086419	-1.98	0.0657
OR400*Salinity	0.0000758	0.000076	1.00	0.3340
OR400*OR400	-1e-7	4.135e-7	-0.24	0.8119
Energy*Salinity	0.0073556	0.013535	0.54	0.5943
Energy*OR400	0.0000081	0.00003	0.27	0.7877
Energy*Energy	0.0238164	0.01307	1.82	0.0872
Loading*Salinity	-0.001951	0.004512	-0.43	0.6711
Loading*OR400	-0.000003	0.00001	-0.30	0.7683
Loading*Energy	0.0003928	0.001755	0.22	0.8257
Loading*Loading	-0.004774	0.001452	-3.29	0.0046

Effect Test

Response Surface

Prediction Profile



creening Fit

PS 3

Summary of Fit

RSquare	0.743712
RSquare Adj	0.519461
Root Mean Square Error	46.06573
Mean of Response	65.05903
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	14	98526.52	7037.61	3.3164
Error	16	33952.83	2122.05	Prob>F
C Total	30	132479.34		0.0121

Lack of Fit

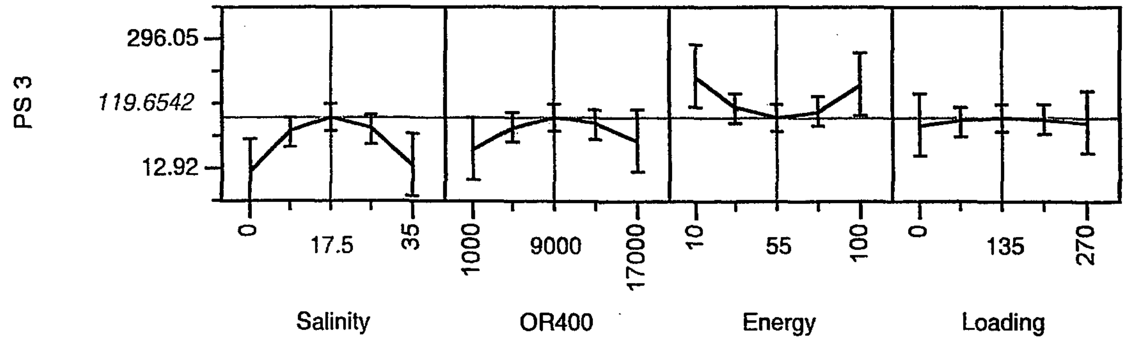
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	48.721588	45.34014	1.07	0.2985
Salinity	12.502851	3.562751	3.51	0.0029
OR400	0.0170416	0.008589	1.98	0.0647
Energy	-4.833287	1.638578	-2.95	0.0094
Loading	0.2018765	0.461838	0.44	0.6679
Salinity*Salinity	-0.353135	0.093373	-3.78	0.0016
OR400*Salinity	0.0000479	0.000082	0.58	0.5682
OR400*OR400	-9.132e-7	4.468e-7	-2.04	0.0578
Energy*Salinity	-0.000777	0.014624	-0.05	0.9583
Energy*OR400	0.0000005	0.000032	0.02	0.9869
Energy*Energy	0.041263	0.014121	2.92	0.0100
Loading*Salinity	-0.001479	0.004875	-0.30	0.7656
Loading*OR400	-0.000003	0.000011	-0.29	0.7770
Loading*Energy	0.0007787	0.001896	0.41	0.6867
Loading*Loading	-0.000603	0.001569	-0.38	0.7059

Effect Test

Response Surface

Prediction Profile



Screening Fit

PS 8

Summary of Fit

RSquare	0.712097
RSquare Adj	0.460183
Root Mean Square Error	51.90606
Mean of Response	63.57742
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	14	106622.78	7615.91	2.8267
Error	16	43107.83	2694.24	Prob>F
C Total	30	149730.61		0.0246

Lack of Fit

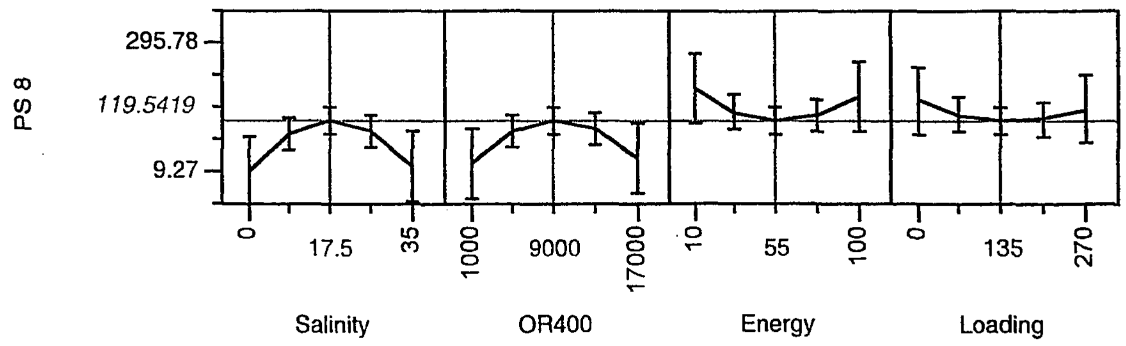
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	32.095051	51.08847	0.63	0.5387
Salinity	12.773623	4.014445	3.18	0.0058
OR400	0.0263109	0.009677	2.72	0.0152
Energy	-3.596207	1.846321	-1.95	0.0692
Loading	-0.58055	0.520391	-1.12	0.2811
Salinity*Salinity	-0.348884	0.105211	-3.32	0.0044
OR400*Salinity	0.0000195	0.000093	0.21	0.8364
OR400*OR400	-0.000001	5.035e-7	-2.75	0.0142
Energy*Salinity	-0.002492	0.016478	-0.15	0.8817
Energy*OR400	-0.000011	0.000036	-0.30	0.7650
Energy*Energy	0.0312045	0.015912	1.96	0.0675
Loading*Salinity	-0.001763	0.005493	-0.32	0.7523
Loading*OR400	-0.000003	0.000012	-0.25	0.8036
Loading*Energy	0.0006202	0.002136	0.29	0.7753
Loading*Loading	0.0019681	0.001768	1.11	0.2821

Effect Test

Response Surface

Prediction Profile



creening Fit

PS 24

Summary of Fit

RSquare	0.54186
RSquare Adj	0.140987
Root Mean Square Error	67.30239
Mean of Response	60.17194
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	14	85717.49	6122.68	1.3517
Error	16	72473.78	4529.61	Prob>F
C Total	30	158191.28		0.2792

Lack of Fit

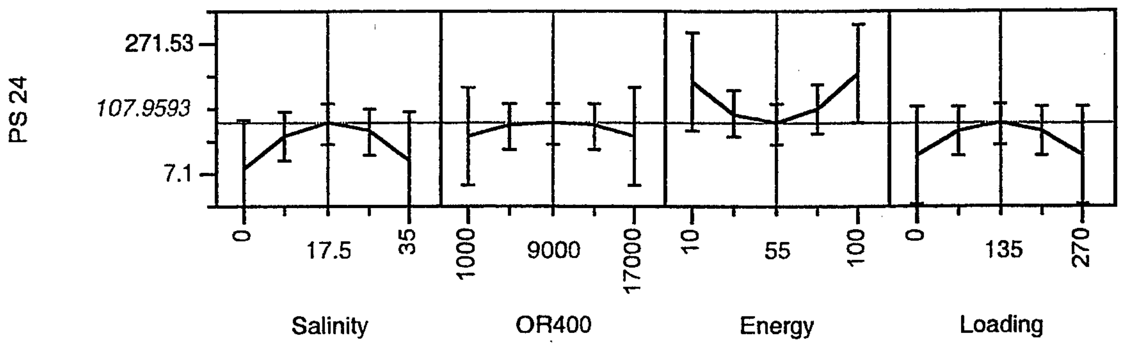
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	26.35642	66.24229	0.40	0.6960
Salinity	10.659205	5.205206	2.05	0.0574
OR400	0.0103664	0.012548	0.83	0.4209
Energy	-4.831103	2.393975	-2.02	0.0607
Loading	0.9624622	0.674749	1.43	0.1730
Salinity*Salinity	-0.277094	0.136418	-2.03	0.0592
OR400*Salinity	-0.00003	0.00012	-0.25	0.8073
OR400*OR400	-4.154e-7	6.528e-7	-0.64	0.5336
Energy*Salinity	-0.000165	0.021366	-0.01	0.9939
Energy*OR400	-0.00003	0.000047	-0.65	0.5245
Energy*Energy	0.0462938	0.020631	2.24	0.0393
Loading*Salinity	-0.001607	0.007122	-0.23	0.8244
Loading*OR400	-0.000005	0.000016	-0.32	0.7512
Loading*Energy	0.0014041	0.00277	0.51	0.6191
Loading*Loading	-0.003545	0.002292	-1.55	0.1416

Effect Test

Response Surface

Prediction Profile



Screening Fit

TPH 24HR Susp

```
Summary of Fit
RSquare                          0.740479
RSquare Adj                      0.513399
Root Mean Square Error           748.9078
Mean of Response                 469.8548
Observations (or Sum Wgts)             31
```

```
Analysis of Variance
Source     DF   Sum of Squares   Mean Square    F Ratio
Model      14         25604573       1828898     3.2609
Error      16          8973806        560863     Prob>F
C Total    30         34578379                   0.0130
```

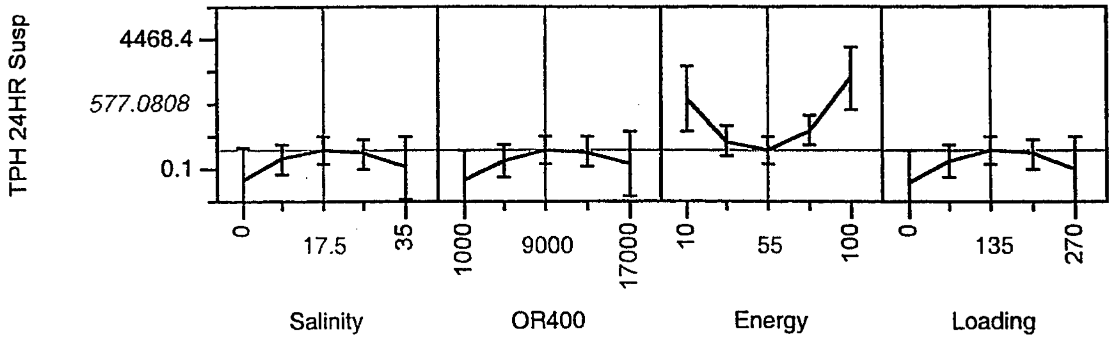
```
Lack of Fit
```

```
Parameter Estimates
Term                   Estimate   Std Error   t Ratio   Prob>|t|
Intercept              1942.768    737.1115      2.64     0.0180
Salinity              46.758148    57.92097      0.81     0.4313
OR400                 0.1343998    0.139628      0.96     0.3501
Energy                -126.9313    26.63897     -4.76     0.0002
Loading               7.5091246    7.508274      1.00     0.3321
Salinity*Salinity      -2.47083    1.517996     -1.63     0.1231
OR400*Salinity        0.0017653    0.001337      1.32     0.2054
OR400*OR400           -0.000011    0.000007     -1.57     0.1353
Energy*Salinity       0.3208968    0.237749      1.35     0.1959
Energy*OR400           0.000824     0.00052      1.58     0.1327
Energy*Energy         1.0586954    0.229573      4.61     0.0003
Loading*Salinity      0.1275317     0.07925      1.61     0.1271
Loading*OR400         0.0002399    0.000173      1.38     0.1854
Loading*Energy        0.0412891    0.030819      1.34     0.1991
Loading*Loading       -0.046123    0.025508     -1.81     0.0894
```

```
Effect Test
```

```
Response Surface
```

Prediction Profile



creening Fit

TPH 24HR Diss

Summary of Fit

RSquare	0.793133
RSquare Adj	0.612124
Root Mean Square Error	1.805823
Mean of Response	2.874194
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	14	200.04338	14.2888	4.3817
Error	16	52.17597	3.2610	Prob>F
C Total	30	252.21935		0.0030

Lack of Fit

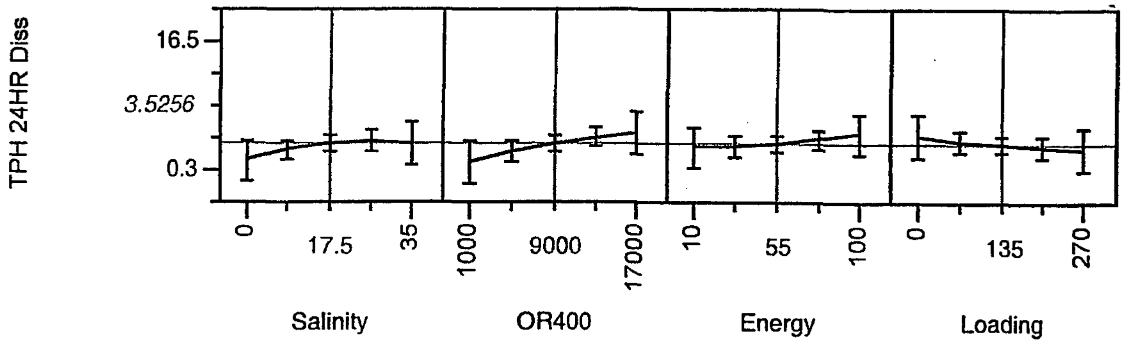
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0374578	1.777379	0.02	0.9834
Salinity	0.0861456	0.139663	0.62	0.5460
OR400	0.0001886	0.000337	0.56	0.5831
Energy	-0.024585	0.064234	-0.38	0.7069
Loading	0.0119271	0.018105	0.66	0.5194
Salinity*Salinity	-0.00361	0.00366	-0.99	0.3387
OR400*Salinity	0.0000089	0.000003	2.75	0.0141
OR400*OR400	-7.898e-9	1.752e-8	-0.45	0.6581
Energy*Salinity	0.0011349	0.000573	1.98	0.0652
Energy*OR400	0.0000026	0.000001	2.04	0.0588
Energy*Energy	0.0001701	0.000554	0.31	0.7625
Loading*Salinity	-0.000294	0.000191	-1.54	0.1439
Loading*OR400	-7.813e-7	4.18e-7	-1.87	0.0800
Loading*Energy	-0.000135	0.000074	-1.81	0.0885
Loading*Loading	0.0000079	0.000062	0.13	0.8990

Effect Test

Response Surface

Prediction Profile



creening Fit

%Float

Summary of Fit

RSquare	0.913948
RSquare Adj	0.838653
Root Mean Square Error	0.127845
Mean of Response	0.384694
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	14	2.7774886	0.198392	12.1382
Error	16	0.2615111	0.016344	Prob>F
C Total	30	3.0389997		<.0001

Lack of Fit

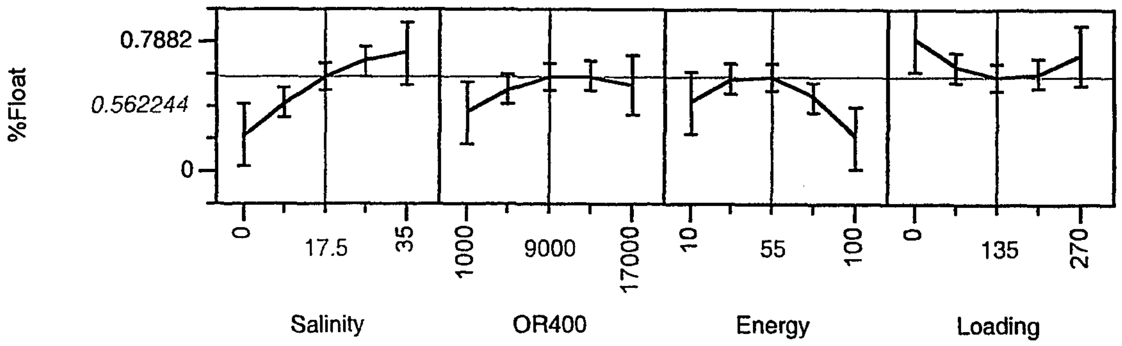
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.155355	0.125832	-1.23	0.2348
Salinity	0.033053	0.009888	3.34	0.0041
OR400	0.0000322	0.000024	1.35	0.1953
Energy	0.0131156	0.004548	2.88	0.0108
Loading	-0.002399	0.001282	-1.87	0.0796
Salinity*Salinity	-0.000335	0.000259	-1.29	0.2147
OR400*Salinity	0.0000004	2.283e-7	1.70	0.1082
OR400*OR400	-2.038e-9	1.24e-9	-1.64	0.1197
Energy*Salinity	-0.000173	0.000041	-4.27	0.0006
Energy*OR400	0.0000002	8.878e-8	2.48	0.0247
Energy*Energy	-0.000126	0.000039	-3.21	0.0055
Loading*Salinity	-0.000007	0.000014	-0.49	0.6341
Loading*OR400	-2.764e-8	2.959e-8	-0.93	0.3641
Loading*Energy	-0.000005	0.000005	-0.93	0.3653
Loading*Loading	0.00001	0.000004	2.29	0.0359

Effect Test

Response Surface

Prediction Profile



creening Fit

%Sunk

Summary of Fit

RSquare	0.881017
RSquare Adj	0.776907
Root Mean Square Error	0.149496
Mean of Response	0.284797
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	14	2.6477745	0.189127	8.4624
Error	16	0.3575858	0.022349	Prob>F
C Total	30	3.0053603		<.0001

Lack of Fit

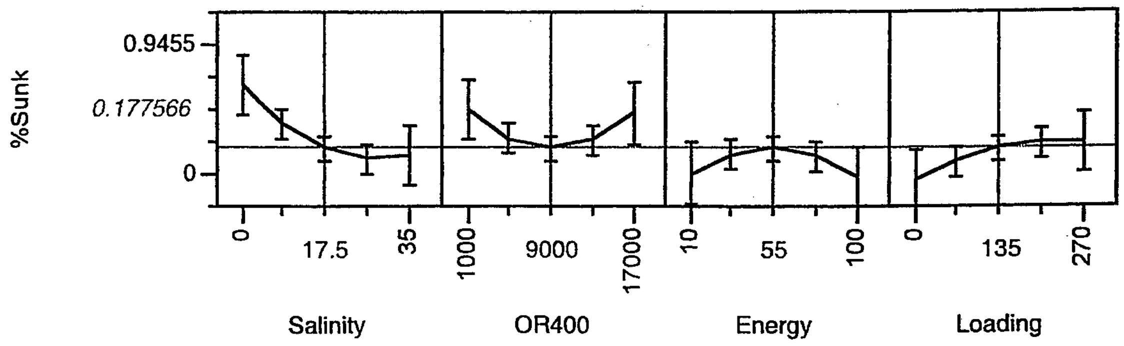
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.5019611	0.147141	3.41	0.0036
Salinity	-0.03787	0.011562	-3.28	0.0048
OR400	-0.000051	0.000028	-1.84	0.0848
Energy	0.0064977	0.005318	1.22	0.2394
Loading	0.0020299	0.001499	1.35	0.1944
Salinity*Salinity	0.0006596	0.000303	2.18	0.0448
OR400*Salinity	-6.663e-7	2.67e-7	-2.50	0.0239
OR400*OR400	4.2415e-9	1.45e-9	2.93	0.0099
Energy*Salinity	0.0001224	0.000047	2.58	0.0202
Energy*OR400	-1.057e-7	1.038e-7	-1.02	0.3237
Energy*Energy	-0.000097	0.000046	-2.11	0.0505
Loading*Salinity	-0.000004	0.000016	-0.26	0.7971
Loading*OR400	-6.907e-8	3.461e-8	-2.00	0.0633
Loading*Energy	0.000019	0.000006	3.09	0.0070
Loading*Loading	-0.000005	0.000005	-1.00	0.3335

Effect Test

Response Surface

Prediction Profile



Screening Fit

%Ring

Summary of Fit

RSquare	0.855077
RSquare Adj	0.728269
Root Mean Square Error	0.107847
Mean of Response	0.268977
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	14	1.0980061	0.078429	6.7431
Error	16	0.1860966	0.011631	Prob>F
C Total	30	1.2841027		0.0003

Lack of Fit

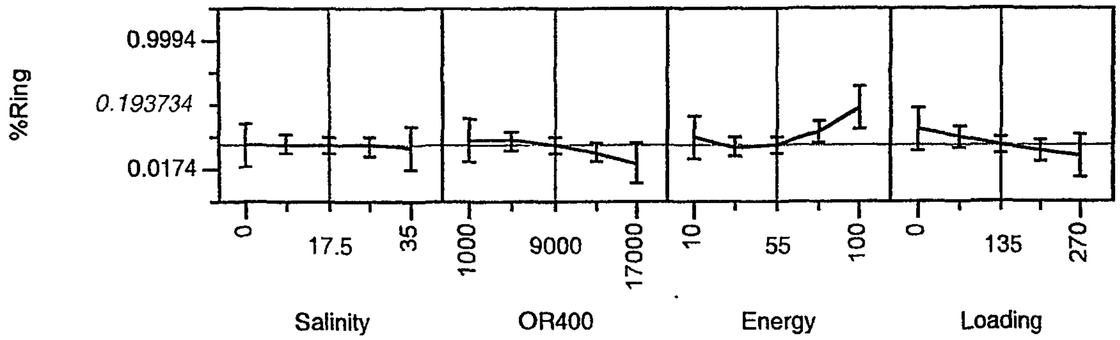
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.4306283	0.106149	4.06	0.0009
Salinity	-0.002011	0.008341	-0.24	0.8125
OR400	-0.000001	0.00002	-0.05	0.9596
Energy	-0.004101	0.003836	-1.07	0.3009
Loading	-0.000951	0.001081	-0.88	0.3921
Salinity*Salinity	-0.000043	0.000219	-0.20	0.8463
OR400*Salinity	-5.116e-8	1.926e-7	-0.27	0.7939
OR400*OR400	-7.73e-10	1.046e-9	-0.74	0.4709
Energy*Salinity	0.0000437	0.000034	1.28	0.2196
Energy*OR400	-1.282e-7	7.489e-8	-1.71	0.1062
Energy*Energy	0.0000853	0.000033	2.58	0.0201
Loading*Salinity	0.0000062	0.000011	0.54	0.5956
Loading*OR400	8.6285e-8	2.496e-8	3.46	0.0033
Loading*Energy	-0.000018	0.000004	-3.95	0.0011
Loading*Loading	0.0000011	0.000004	0.29	0.7745

Effect Test

Response Surface

Prediction Profile



creening Fit

%Susp.

Summary of Fit

RSquare	0.724263
RSquare Adj	0.482992
Root Mean Square Error	0.080382
Mean of Response	0.061119
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	14	0.27154148	0.019396	3.0019
Error	16	0.10337987	0.006461	Prob>F
C Total	30	0.37492135		0.0190

Lack of Fit

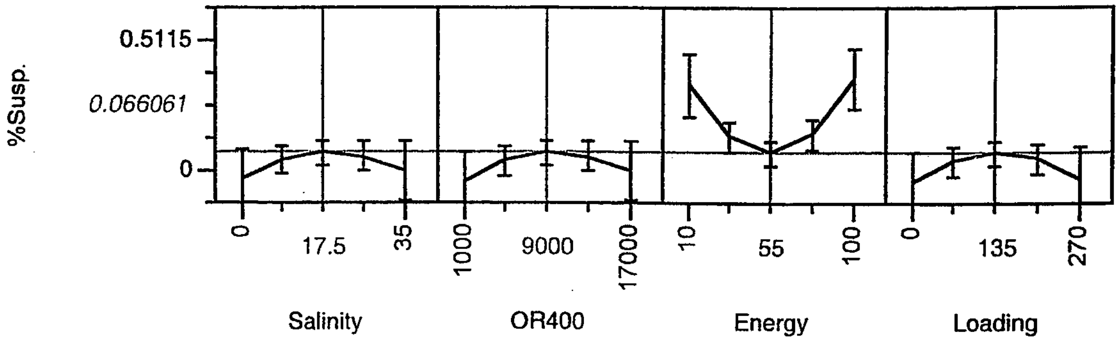
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.2218206	0.079116	2.80	0.0127
Salinity	0.0068229	0.006217	1.10	0.2887
OR400	0.0000202	0.000015	1.35	0.1961
Energy	-0.015513	0.002859	-5.43	<.0001
Loading	0.0013159	0.000806	1.63	0.1220
Salinity*Salinity	-0.000281	0.000163	-1.73	0.1036
OR400*Salinity	0.0000003	1.435e-7	2.29	0.0362
OR400*OR400	-1.439e-9	7.8e-10	-1.85	0.0836
Energy*Salinity	0.0000069	0.000026	0.27	0.7906
Energy*OR400	1.3576e-8	5.582e-8	0.24	0.8109
Energy*Energy	0.0001374	0.000025	5.58	<.0001
Loading*Salinity	0.0000045	0.000009	0.53	0.6017
Loading*OR400	1.0451e-8	1.861e-8	0.56	0.5821
Loading*Energy	0.0000034	0.000003	1.04	0.3129
Loading*Loading	-0.000006	0.000003	-2.17	0.0450

Effect Test

Response Surface

Prediction Profile



Screening Fit

%Diss

Summary of Fit

RSquare	0.702738
RSquare Adj	0.442635
Root Mean Square Error	0.000238
Mean of Response	0.000394
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	14	0.00000215	1.535e-7	2.7018
Error	16	0.00000091	5.683e-8	Prob>F
C Total	30	0.00000306		0.0298

Lack of Fit

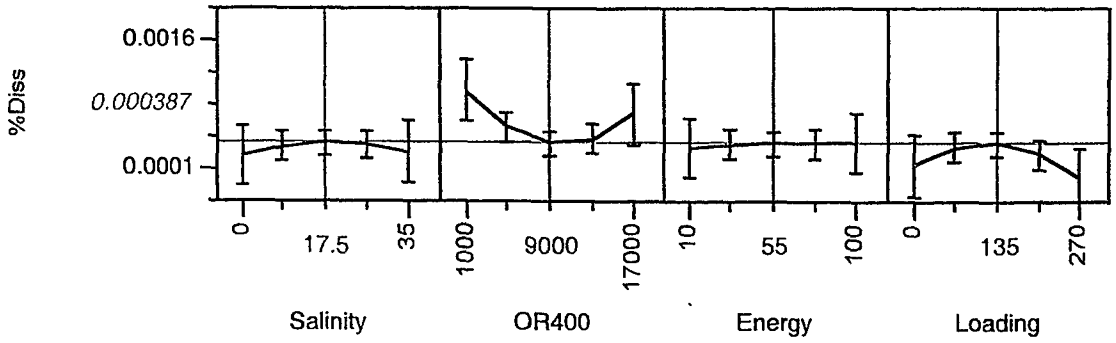
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0009368	0.000235	3.99	0.0010
Salinity	0.0000001	0.000018	0.01	0.9941
OR400	-1.827e-7	4.444e-8	-4.11	0.0008
Energy	-0.000001	0.000008	-0.14	0.8914
Loading	0.0000058	0.000002	2.43	0.0270
Salinity*Salinity	-3.992e-7	4.832e-7	-0.83	0.4208
OR400*Salinity	9.375e-10	4.26e-10	2.20	0.0427
OR400*OR400	7.465e-12	2.31e-12	3.23	0.0053
Energy*Salinity	0.0000001	7.568e-8	1.78	0.0936
Energy*OR400	3.646e-10	1.66e-10	2.20	0.0427
Energy*Energy	-1.1e-8	7.308e-8	-0.15	0.8823
Loading*Salinity	-7.937e-9	2.523e-8	-0.31	0.7571
Loading*OR400	-4.05e-11	5.52e-11	-0.73	0.4735
Loading*Energy	-1.955e-8	9.81e-9	-1.99	0.0637
Loading*Loading	-1.768e-8	8.119e-9	-2.18	0.0447

Effect Test

Response Surface

Prediction Profile



Screening Fit

PAH diss

Summary of Fit

RSquare	0.68452
RSquare Adj	0.408476
Root Mean Square Error	8375.271
Mean of Response	5796.961
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	14	2435188423	1.7394e8	2.4797
Error	16	1122322623	70145164	Prob>F
C Total	30	3557511046		0.0422

Lack of Fit

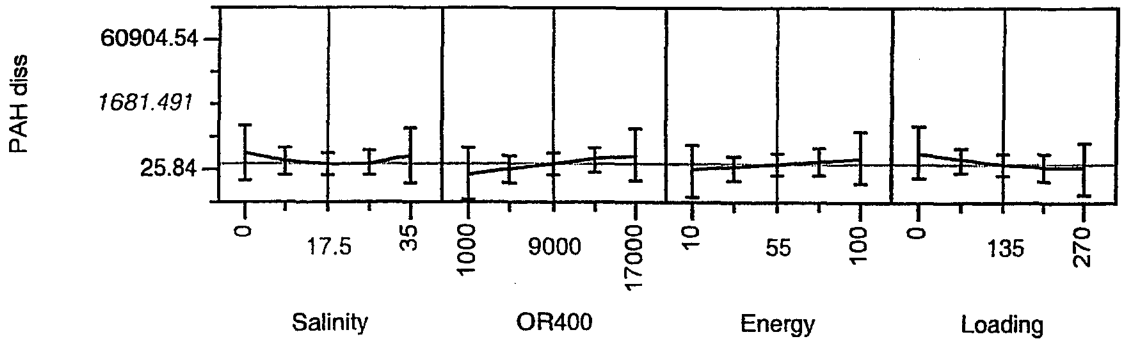
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	6293.3322	8243.35	0.76	0.4563
Salinity	-875.3955	647.7484	-1.35	0.1954
OR400	0.0170025	1.561499	0.01	0.9914
Energy	-56.02128	297.912	-0.19	0.8532
Loading	34.982893	83.96738	0.42	0.6825
Salinity*Salinity	15.107808	16.97622	0.89	0.3867
OR400*Salinity	0.0250849	0.014956	1.68	0.1129
OR400*OR400	0.0000002	0.000081	0.00	0.9983
Energy*Salinity	5.0158159	2.658816	1.89	0.0775
Energy*OR400	0.0109507	0.005816	1.88	0.0780
Energy*Energy	0.1415734	2.567392	0.06	0.9567
Loading*Salinity	-1.494825	0.886272	-1.69	0.1111
Loading*OR400	-0.003833	0.001939	-1.98	0.0655
Loading*Energy	-0.583095	0.344661	-1.69	0.1101
Loading*Loading	0.118695	0.285266	0.42	0.6829

Effect Test

Response Surface

Prediction Profile



APPENDIX IV

Polycyclic Aromatic Hydrocarbon Data

Response Surface Experiments

PAH Concentrations in Filtered Water (ng/L)

Client ID		**NA**	**NA**	**NA**	**Trial 1'**	**Trial 2'**	**Trial 3'**	**Trial 4'**	**Trial 5'**	**Trial 6'**	**Trial 7'**	**Trial 8'**	**Trial 9'**	**Trial 10'**	**Trial 11'**	**Trial 12'**
Battelle ID		VU66PB	VV44PB	VV53PB	UU24FIL	UU25FIL	UT54FIL	UT55FIL	UU26FIL	UU27FIL	UT56FIL	UT57FIL	UU11FIL	UU12FIL	UT68FIL	UT69FIL
Sample Volume (L)		1.000	1.000	1.000	0.870	0.870	0.800	0.700	0.840	0.910	0.720	0.790	0.880	0.870	0.990	0.995
Naphthalene	**N**	2.97	4.18	4.67	1124.13	1436.03	271.78	519.99	2056.90	1677.05	1479.97	1057.57	14.11	14.84	11.85	13.55
C1-naphthalenes	**N1**	2.44	3.72	3.39	1685.90	2126.25	1258.22	1244.01	2495.88	2041.41	1871.75	1497.60	14.05	11.00	5.28	0.00
C2-naphthalenes	**N2**	0.00	0.00	0.00	1395.36	1449.48	835.91	1308.99	1634.74	1522.60	1686.74	1433.73	0.00	0.00	0.00	0.00
C3-naphthalenes	**N3**	0.00	0.00	0.00	768.99	713.68	563.63	703.41	747.79	686.96	954.19	863.40	0.00	0.00	0.00	0.00
C4-naphthalenes	**N4**	0.00	0.00	0.00	530.18	510.43	484.95	667.72	554.57	496.49	559.84	544.51	0.00	0.00	0.00	0.00
Biphenyl	**BI**	0.00	0.00	0.00	83.96	87.53	41.43	64.84	111.27	91.75	88.33	78.63	0.00	0.00	0.00	0.00
Acenaphthylene	**ACY**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Acenaphthene	**ACE**	0.00	0.00	0.00	244.27	251.48	192.51	243.60	283.40	260.60	289.71	279.42	0.00	0.00	0.00	9.66
Dibenzofuran	**DI**	0.00	0.00	0.00	97.26	96.60	58.02	68.12	109.12	93.50	82.69	79.25	0.00	0.00	0.00	0.00
Fluorene	**F**	0.00	0.00	0.00	170.11	167.86	109.19	134.81	190.77	171.76	159.15	151.87	0.00	0.00	0.00	0.00
C1-fluorenes	**F1**	0.00	0.00	0.00	253.47	242.05	180.82	210.85	262.18	221.38	245.45	217.30	0.00	0.00	0.00	0.00
C2-fluorenes	**F2**	0.00	0.00	0.00	333.67	258.28	239.64	308.64	316.13	232.07	367.71	237.64	0.00	0.00	0.00	0.00
C3-fluorenes	**F3**	0.00	0.00	0.00	99.67	78.13	98.21	165.36	123.12	108.62	208.20	149.50	0.00	0.00	0.00	0.00
Anthracene	**A**	0.00	0.00	0.00	9.21	8.32	8.81	9.79	16.33	15.15	26.02	16.67	0.00	0.00	0.00	0.00
Phenanthrene	**P**	0.00	0.00	0.00	391.44	380.39	281.92	324.01	442.32	337.33	333.63	300.04	0.00	0.00	0.00	0.00
C1-phenanthrenes/anthracenes	**P1**	0.00	0.00	0.00	201.25	192.46	161.99	177.63	254.98	168.61	284.13	196.48	0.00	0.00	0.00	0.00
C2-phenanthrenes/anthracenes	**P2**	0.00	0.00	0.00	139.03	123.78	193.50	206.47	248.94	131.51	430.39	266.97	0.00	0.00	0.00	0.00
C3-phenanthrenes/anthracenes	**P3**	0.00	0.00	0.00	63.73	0.00	94.54	164.26	124.23	68.78	330.63	178.47	0.00	0.00	0.00	0.00
C4-phenanthrenes/anthracenes	**P4**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	163.22	0.00	0.00	0.00	0.00	0.00
Dibenzothiophene	**D**	0.00	0.00	0.00	165.86	160.84	111.31	128.57	192.23	159.98	137.02	131.92	0.00	0.00	20.19	22.21
C1-dibenzothiophenes	**D1**	0.00	0.00	0.00	223.83	214.89	227.58	234.11	287.38	240.26	436.00	353.69	0.00	0.00	16.89	33.95
C2-dibenzothiophenes	**D2**	0.00	0.00	0.00	272.35	233.29	241.18	352.18	344.96	247.20	790.83	523.18	0.00	0.00	71.53	96.49
C3-dibenzothiophenes	**D3**	0.00	0.00	0.00	248.90	226.19	241.68	388.21	445.81	282.78	714.78	490.52	0.00	0.00	103.02	165.01
C4-dibenzothiophenes	**D4**	0.00	0.00	0.00	82.56	84.89	117.40	161.68	174.19	104.86	303.58	255.19	0.00	0.00	0.00	66.67
Fluoranthene	**FL**	0.00	0.00	0.00	3.84	2.95	3.88	3.08	8.95	0.00	6.17	7.00	0.00	0.00	0.00	0.00
Pyrene	**PY**	0.00	0.00	0.00	6.86	5.67	8.66	8.47	10.44	0.00	16.21	16.26	0.00	0.00	0.00	0.00
C1-fluoranthenes/pyrenes	**FP1**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C2-fluoranthenes/pyrenes	**FP2**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C3-fluoranthenes/pyrenes	**FP3**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Benz(a)anthracene	**BA**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.91	0.00	0.00	0.00	0.00
Chrysene	**C**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	7.12	0.00	0.00	0.00	0.00
C1-chrysenes	**C1**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	14.75	0.00	0.00	0.00	0.00
C2-chrysenes	**C2**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C3-chrysenes	**C3**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C4-chrysenes	**C4**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Benzo(b)fluoranthene	**BB**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Benzo(k)fluoranthene	**BK**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Benzo(e)pyrene	**BE**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Benzo(a)pyrene	**BAP**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Perylene	**PER**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Indeno(1,2,3-c,d)pyrene	**IP**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Dibenz(a,h)anthracene	**DA**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Benzo(g,h,i)perylene	**GHI**	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Total PAH's	**TOTAL**	**5.41**	**7.90**	**8.06**	**8595.81**	**9051.46**	**6026.74**	**7798.76**	**11436.63**	**9360.62**	**11966.33**	**9351.58**	**28.15**	**25.84**	**228.76**	**407.53**
Surrogate Recoveries																
Naphthalene-d8		68	80	76	67	58	29	54	54	64	66	62	66	62	51	47
Phenanthrene-d10		78	96	88	95	97	82	78	83	94	82	78	98	99	77	76
Chrysene-d12		77	93	90	89	93	87	79	74	81	74	69	93	93	82	81

PAH Concentrations in Filtered Water (ng/L)

	Trial 13'	Trial 14'	Trial 15'	Trial 16'	Trial 17'	Trial 18'	Trial 19'	Trial 20'	Trial 21'	Trial 22'	Trial 23'	Trial 24'	Trial 25'	Trial 26'	Trial 27'	Trial 28'	Trial 29'	Trial 30'
	UU13FIL	UU14FIL	UT70FIL	UT71FIL	UT42FIL	UT43FIL	UT44FIL	UT45FIL	UU15FIL	UT72FIL	UT81FIL	UT82FIL	UT83FIL	UT84FIL	UT95FIL	UT96FIL	UT97FIL	UT98FIL
	0.855	0.900	1.000	0.900	0.905	0.925	0.940	0.945	0.920	0.875	0.970	0.980	0.900	0.930	0.900	0.920	0.880	0.880
N	46.44	31.83	30.12	123.38	2055.04	24.30	15.84	29.09	45.14	26.55	46.48	64.38	57.17	49.69	83.45	91.68	63.28	85.30
N1	52.14	46.55	27.88	118.01	2149.75	49.68	17.56	51.00	66.49	48.27	134.84	90.96	53.86	52.20	174.73	238.88	149.67	168.14
N2	99.07	96.89	633.68	233.52	1366.66	38.72	0.00	137.56	90.93	50.91	170.23	140.10	106.95	130.44	178.86	216.55	199.16	155.99
N3	77.26	84.12	2098.03	226.24	765.45	0.00	0.00	137.73	58.11	0.00	110.39	119.74	100.48	87.34	320.09	190.58	193.31	190.48
N4	0.00	57.14	3888.50	168.85	594.86	0.00	0.00	0.00	23.71	0.00	60.73	90.60	23.42	0.00	0.00	132.44	135.71	86.33
BI	0.00	0.00	0.00	7.15	81.66	0.00	0.00	14.96	0.00	0.00	3.31	0.00	0.00	0.00	5.66	0.00	0.00	0.00
ACY	0.00	0.00	0.00	10.20	0.00	34.69	10.75	20.00	19.10	0.00	256.23	202.00	170.89	109.42	217.77	227.41	241.14	224.42
ACE	23.87	0.00	190.64	133.59	276.52	0.00	0.00	80.84	18.75	0.00	73.16	48.85	29.04	15.43	64.42	67.96	80.39	58.44
DI	0.00	0.00	38.13	44.94	83.95	13.02	0.00	27.25	8.65	0.00	17.01	9.70	6.10	10.79	15.27	0.00	19.36	17.25
F	5.90	0.00	157.22	78.68	162.03	0.00	0.00	54.29	23.16	0.00	103.24	89.53	74.60	78.91	93.65	105.60	105.57	72.04
F1	42.00	0.00	584.76	110.84	270.62	0.00	0.00	107.48	74.91	0.00	164.02	98.41	75.18	89.92	80.91	89.82	134.33	100.92
F2	252.37	0.00	2197.43	136.94	403.72	0.00	0.00	0.00	221.16	0.00	259.05	309.45	236.31	196.57	397.97	359.22	277.41	237.69
F3	76.68	0.00	3755.71	0.00	163.52	0.00	0.00	0.00	0.00	0.00	135.85	180.15	0.00	119.05	0.00	0.00	122.76	71.71
A	21.45	24.78	0.00	0.00	13.69	0.00	0.00	0.00	15.58	0.00	22.08	0.00	0.00	0.00	23.43	22.08	15.61	13.98
P	29.68	48.96	475.85	52.02	356.97	0.00	0.00	49.56	64.62	0.00	46.59	25.01	26.01	19.43	68.04	64.90	64.71	41.49
P1	101.00	103.49	1407.04	54.18	240.33	0.00	0.00	0.00	69.20	0.00	103.21	72.41	74.67	80.65	52.30	77.28	69.59	52.23
P2	106.80	113.50	4788.72	142.69	250.44	0.00	0.00	0.00	82.17	0.00	174.86	95.07	133.69	84.66	224.68	115.38	133.96	140.90
P3	0.00	0.00	5832.14	0.00	0.00	0.00	0.00	0.00	0.00	0.00	87.44	0.00	0.00	0.00	0.00	0.00	0.00	0.00
P4	0.00	0.00	3187.95	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
D	312.81	492.37	275.68	61.16	172.42	245.24	59.30	224.43	620.96	0.00	280.23	209.73	170.82	132.25	324.87	350.45	316.35	265.70
D1	146.69	161.89	1818.18	86.72	446.70	234.20	161.87	229.81	237.12	0.00	218.31	184.03	183.87	149.10	150.86	137.82	171.59	199.12
D2	363.19	413.12	5387.25	223.35	367.44	239.88	248.86	205.60	346.94	0.00	293.96	215.57	127.57	143.57	154.60	166.06	301.84	221.81
D3	473.87	0.00	10155.38	295.73	0.00	0.00	0.00	0.00	230.80	0.00	489.08	344.05	0.00	0.00	0.00	406.84	0.00	0.00
D4	197.59	0.00	9632.99	206.63	0.00	0.00	0.00	0.00	0.00	0.00	293.40	0.00	0.00	0.00	0.00	0.00	0.00	0.00
FL	0.00	0.00	0.00	0.00	8.32	0.00	0.00	0.00	0.00	0.00	10.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00
PY	0.00	0.00	159.31	0.00	13.58	0.00	8.28	0.00	0.00	0.00	10.74	0.00	0.00	0.00	0.00	0.00	0.00	0.00
FP1	0.00	0.00	553.53	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
FP2	0.00	0.00	940.35	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
FP3	0.00	0.00	1049.87	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
BA	0.00	0.00	28.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.67	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C	0.00	0.00	171.76	0.00	0.00	0.00	0.00	0.00	0.00	0.00	10.21	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C1	0.00	0.00	451.77	0.00	0.00	0.00	0.00	0.00	0.00	0.00	9.37	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C2	0.00	0.00	629.52	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C3	0.00	0.00	357.10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C4	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
BB	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	3.47	0.00	0.00	0.00	0.00	0.00	0.00	0.00
BK	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.88	0.00	0.00	0.00	0.00	0.00	0.00	0.00
BE	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.93	0.00	0.00	0.00	0.00	0.00	0.00	0.00
BAP	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.58	0.00	0.00	0.00	0.00	0.00	0.00	0.00
PER	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
IP	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
DA	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
GHI	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
TOTAL	2428.83	1674.64	60904.54	2514.80	10243.67	879.75	522.45	1369.61	2317.53	125.73	3606.57	2589.75	1650.66	1549.44	2631.54	3060.95	2795.73	2403.93
	76	76	56	56	51	54	53	59	73	56	44	50	52	54	53	44	62	43
	105	106	87	86	77	85	78	87	101	85	92	98	99	100	99	96	96	100
	96	99	73	80	69	83	82	85	94	82	91	91	92	92	88	87	85	90

PAH Concentrations in Filtered Water (ng/L)

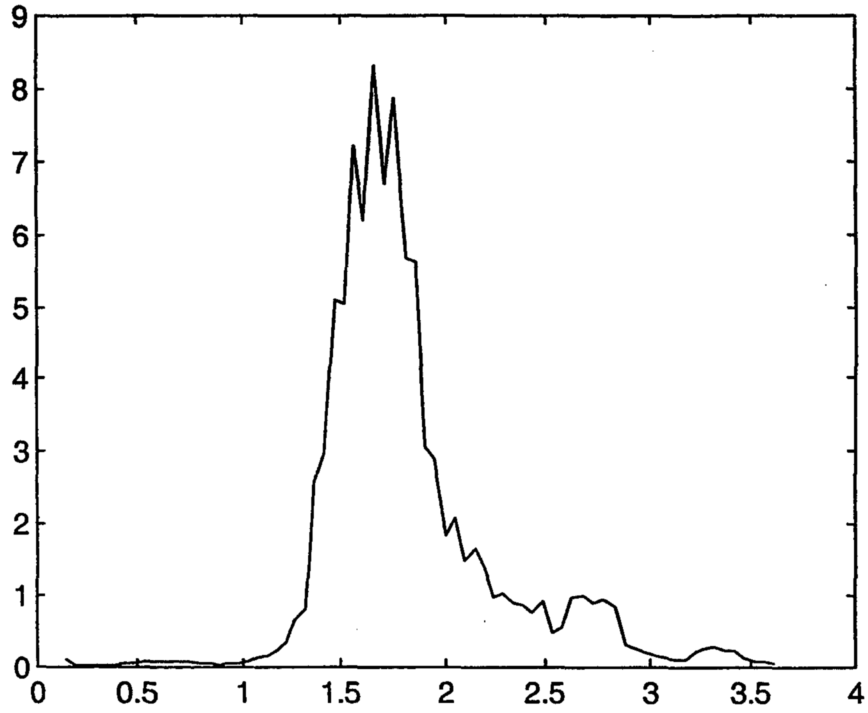
	Trial 31' UT99FIL 0.845
N	60.87
N1	147.45
N2	155.95
N3	129.18
N4	92.07
BI	0.00
ACY	228.69
ACE	54.16
DI	12.25
F	81.82
F1	79.75
F2	247.14
F3	79.57
A	14.33
P	34.90
P1	55.25
P2	131.72
P3	0.00
P4	0.00
D	259.64
D1	102.43
D2	190.30
D3	0.00
D4	0.00
FL	0.00
PY	0.00
FP1	0.00
FP2	0.00
FP3	0.00
BA	0.00
C	0.00
C1	0.00
C2	0.00
C3	0.00
C4	0.00
BB	0.00
BK	0.00
BE	0.00
BAP	0.00
PER	0.00
IP	0.00
DA	0.00
GHI	0.00
TOTAL	**2157.47**
	55
	105
	90

APPENDIX V

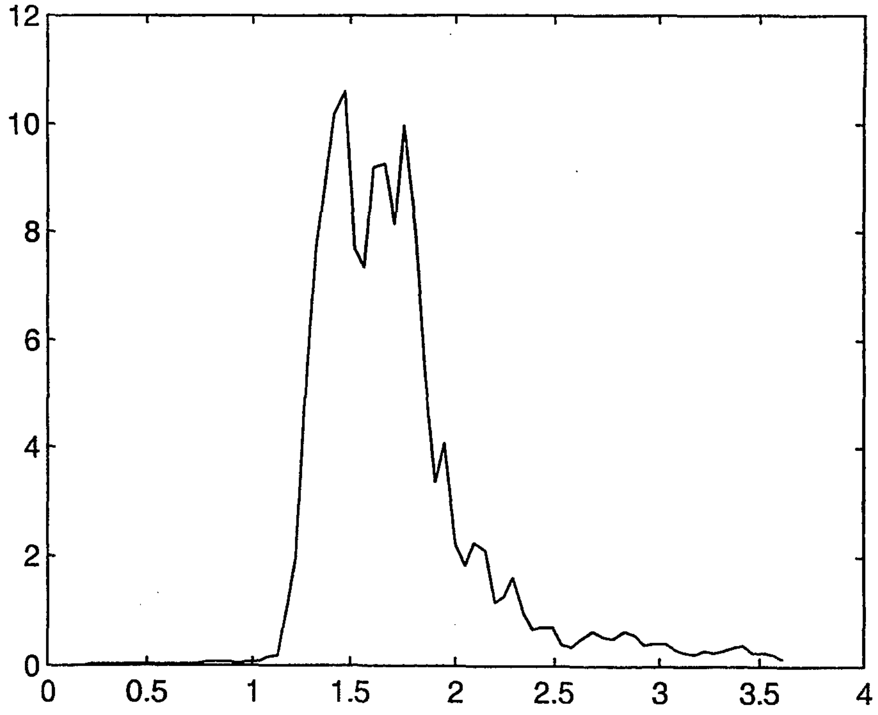
Energy Density Spectra for Flume Wind-Wave Settings

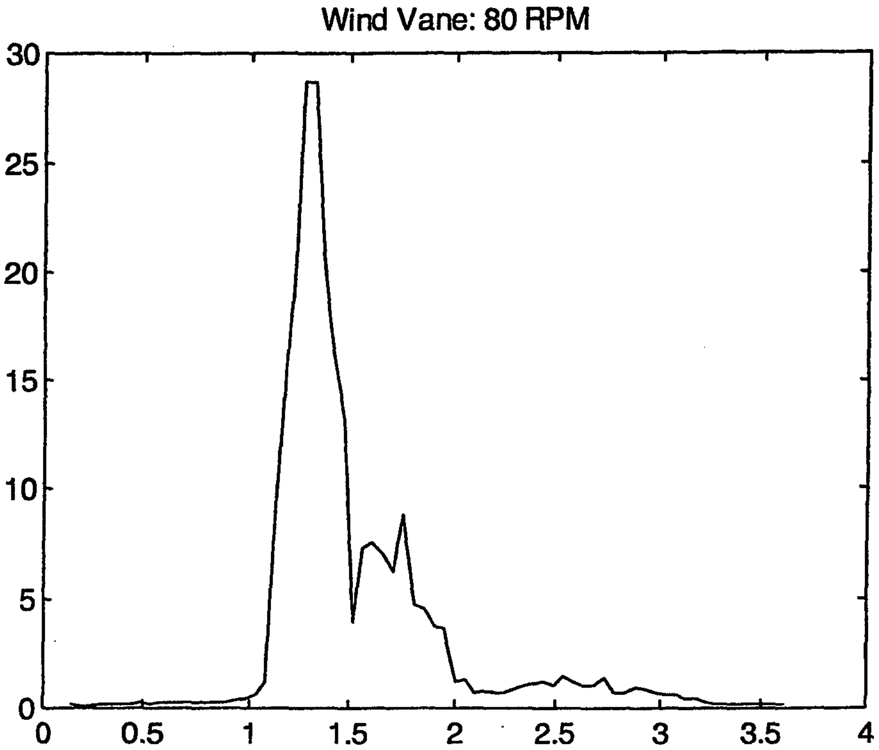
Appendix I: Wave Spectra

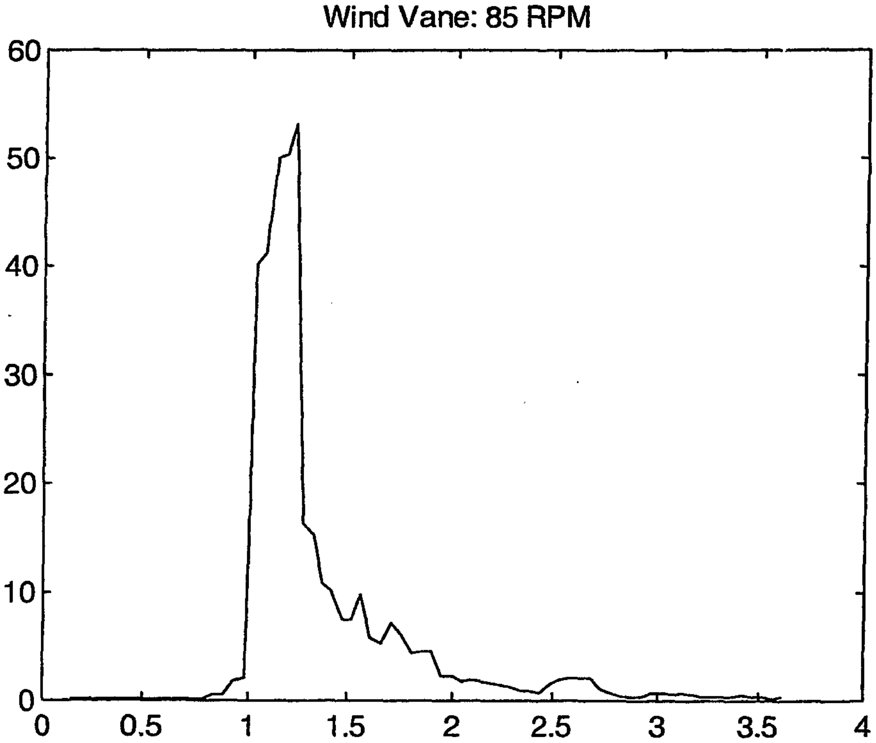
Wind Vane: 70 RPM

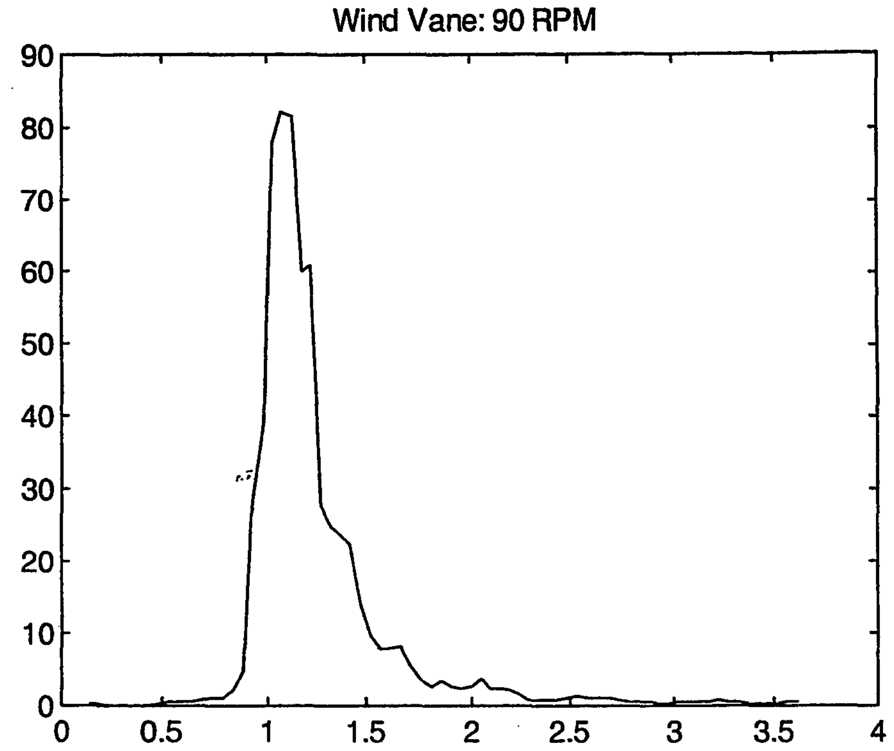


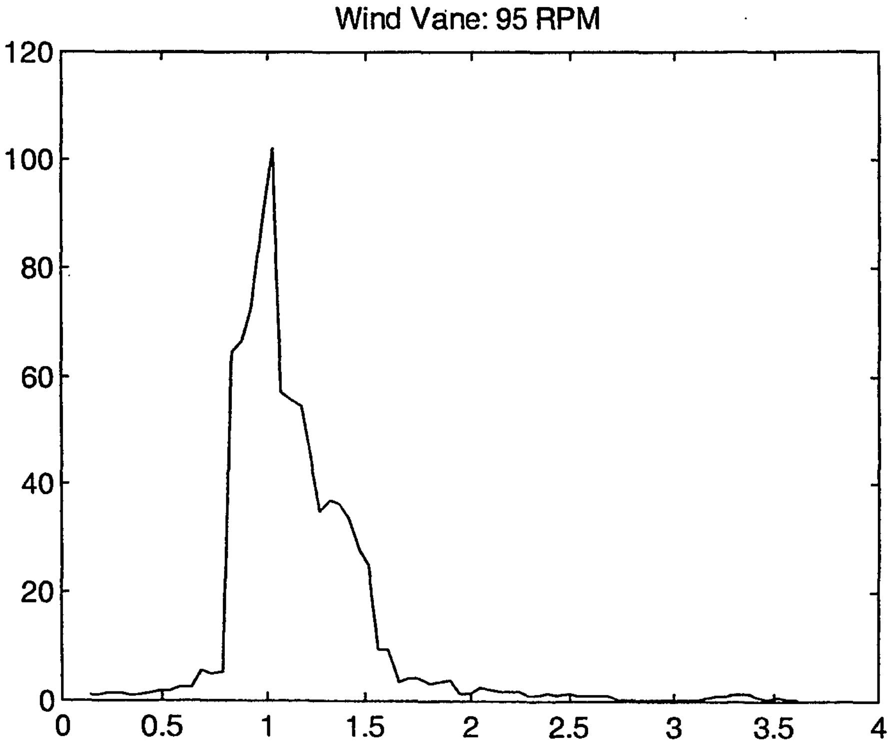
Wind Vane: 75 RPM

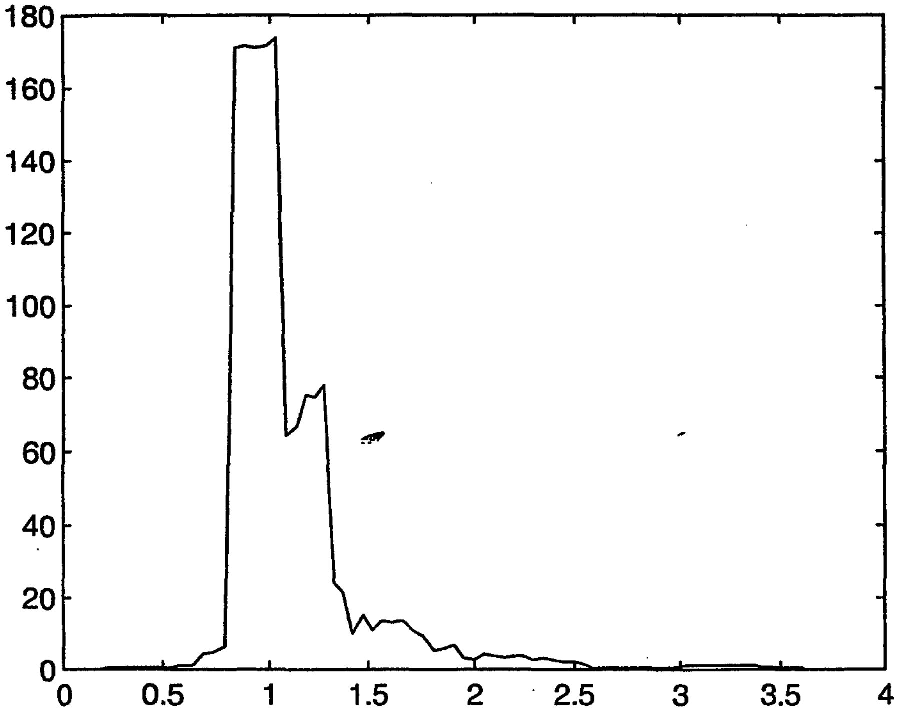



Wind Vane: 80 RPM
30
25
20
15
10
5
0
0
0.5
1
1.5
2
2.5
3
3.5
4

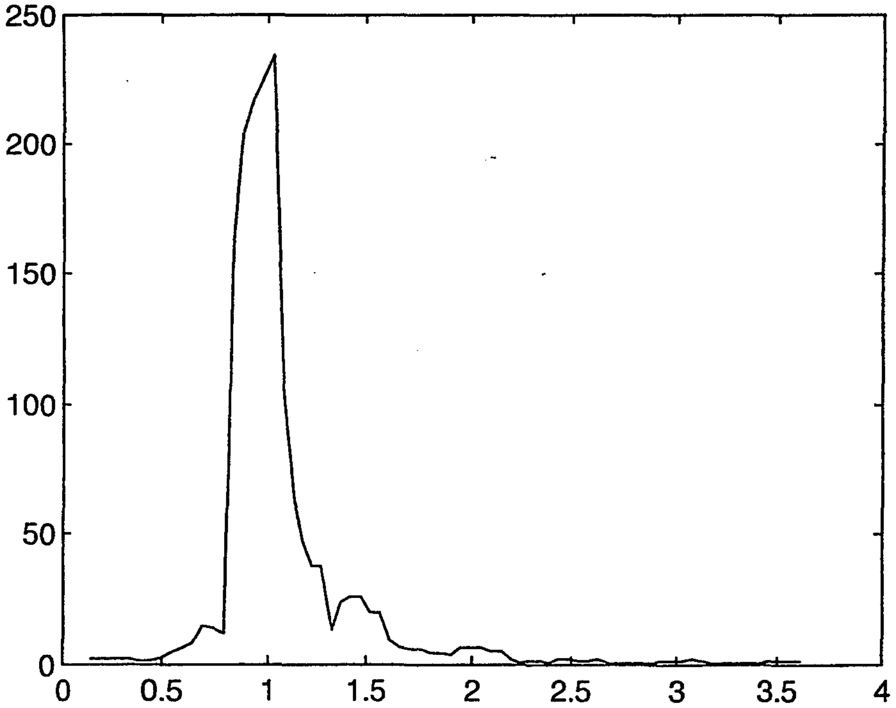

Wind Vane: 85 RPM
60
50
40
30
20
10
0
0
0.5
1
1.5
2
2.5
3
3.5
4


Wind Vane: 90 RPM

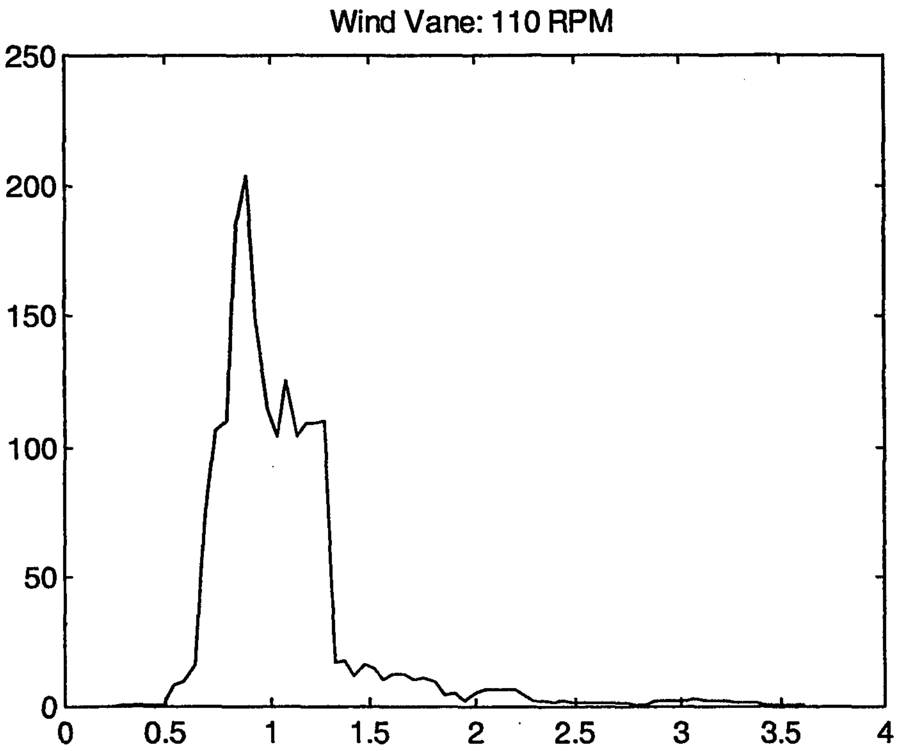

Wind Vane: 95 RPM

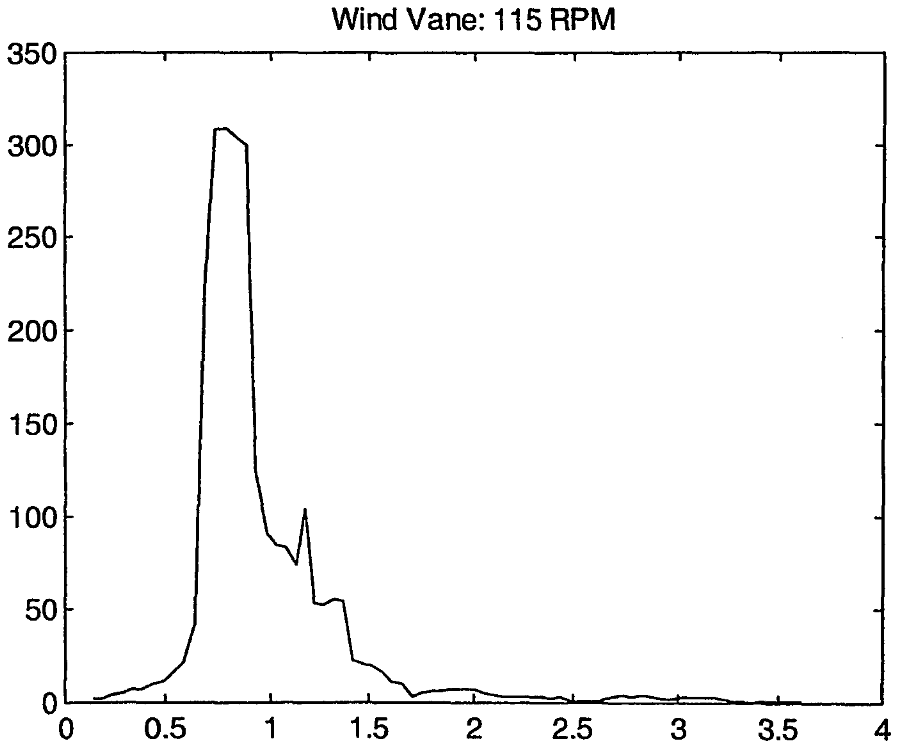


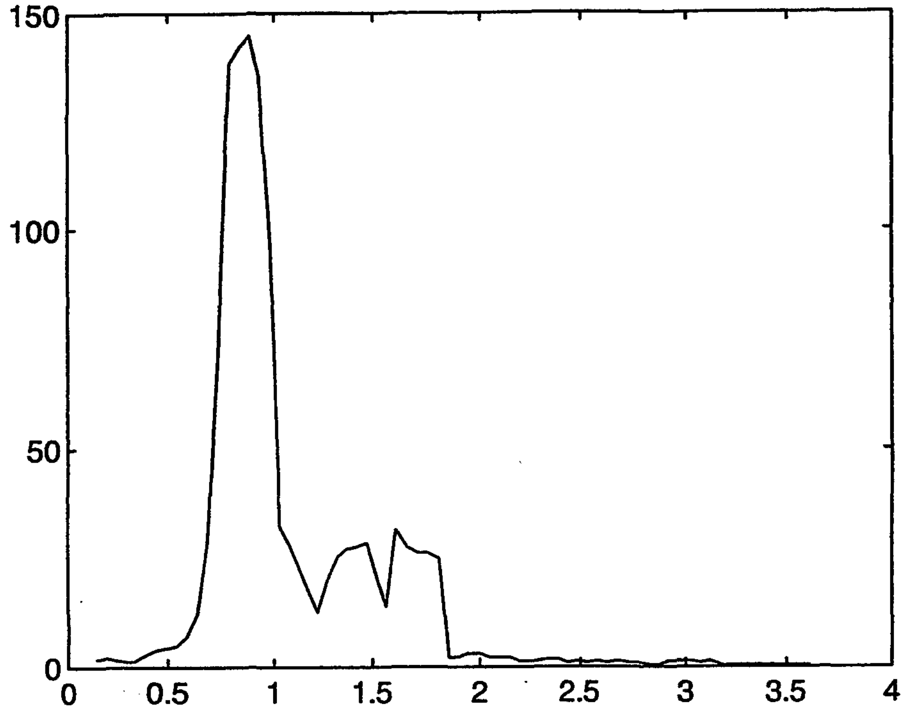
Wind Vane: 100 RPM



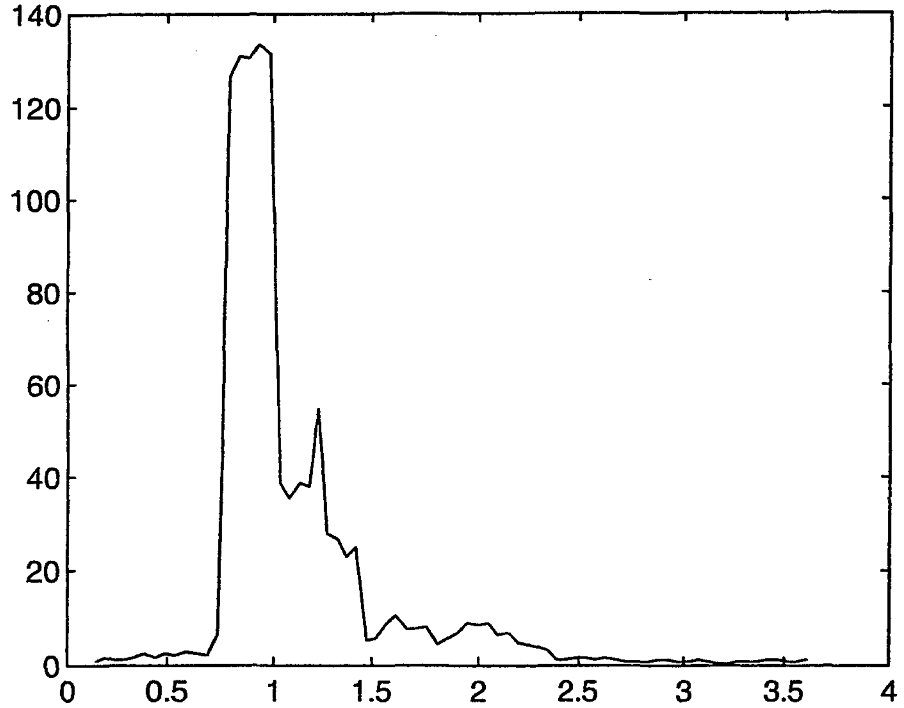
Wind Vane: 105 RPM


Wind Vane: 110 RPM
250
200
150
100
50
0
0
0.5
1
1.5
2
2.5
3
3.5
4


Wind Vane: 115 RPM
350
300
250
200
150
100
50
0
0
0.5
1
1.5
2
2.5
3
3.5
4



Wind Vane: 80 RPM, Wave: 50 cpm, Stroke: 6 cm



Wind Vane: 95 RPM, Wave: 50 cpm, Stroke: 6 cm

APPENDIX VI

Data from Flume Tests

pH of water used in flume tests

Instrument Calibration:

Buffer:	4.0	7.0	10.0
Results:	4.0	7.0	10.0

Battelle Freshwater (0 $^{o}/_{oo}$):	7.4	7.5	7.5	1
Mixed Brackish water (17.5 $^{o}/_{oo}$):	7.6	7.6	7.6	2,3
Duxbury Bay Seawater (32 $^{o}/_{oo}$) w/ Kaol:	7.8	7.8	7.8	4
Battelle Freshwater w/ Kaol:	6.8	6.9	6.9	5

TOTAL SUSPENDED SOLIDS DATA - FLUME TESTS 4& 5
EPA METHOD 160.2M

Sample ID	Filter Tare Wt (g)	Filter Final Wt (g)	Volume Filtered (ml)	Concentration (mg/L)	Average	Stdev
Blank	0.01615	0.01659	250	1.760		
Test#4 Rep1	0.01565	0.04493	300	97.600		
Test#4 Rep2	0.01574	0.04435	300	95.367		
Test#4 Rep3	0.01614	0.04365	250	110.040	**101.00**	**7.91**
Test#5 Rep1	0.01603	0.02853	300	41.667		
Test#5 Rep2	0.01568	0.02974	300	46.867		
Test#5 Rep3	0.01539	0.02972	300	47.767	**45.43**	**3.29**

FLUME TESTS 1 THROUGH 5

AVERAGE PARTICLE SIZE RESULTS

Battelle Flume Tank Particle Size Results (1998)

	Flume Test #1				Flume Test #2				Flume Test #3				Flume Test #4				Flume Test #5			
	Rep 1	Rep 2	Rep 3	Avg	Rep 1	Rep 2	Rep 3	Avg	Rep 1	Rep 2	Rep 3	Avg	Rep 1	Rep 2	Rep 3	Avg	Rep 1	Rep 2	Rep 3	Avg
Kaol.Conc.	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	13.3	20.5	15.9	16.6	10.7	10.7	10.6	10.6
t=0	21.9	23.3	22.1	22.4	24.6	24.1	27.5	25.4	23.0	21.5	NA	22.2	23.2	23.1	NA	23.2	22.7	22.5	22.5	22.6
t=15min	23.0	22.8	23.6	23.2	73.7	73.5	70.6	72.6	113.9	102.7	109.3	108.6	37.9	52.8	74.5	55.0	22.7	22.7	22.7	22.7
t-30min	22.2	23.8	22.1	22.7	80.5	81.3	83.1	81.6	131.4	116.3	107.9	118.5	64.7	55.5	73.9	64.7	23.0	22.0	21.8	22.3
t=1hr	22.2	23.0	22.4	22.5	120.2	97.0	139.4	118.9	116.6	139.9	109.6	122.0	88.8	125.2	78.6	97.5	22.6	21.6	22.0	22.1
t=2hr	24.6	23.8	22.6	23.7	155.8	126.2	121.8	134.6	154.9	119.3	101.9	125.4	104.1	128.5	114.4	115.7	22.6	22.9	21.0	22.2
t=4hr	23.0	23.2	22.0	22.8	185.2	181.2	200.3	188.9	98.3	113.7	117.9	110.0	122.7	104.0	166.0	130.9	22.3	22.4	22.6	22.4
t=6hr	22.4	22.9	22.6	22.7	236.8	190.6	229.4	218.9	118.4	104.7	104.0	109.1	156.8	90.1	163.0	136.6	23.3	22.2	22.7	22.7
t=12hr	21.5	20.9	21.7	21.4	258.8	248.2	263.2	256.7	112.9	110.3	110.0	111.0	124.4	102.9	133.5	120.3	21.4	28.4	24.1	24.6
t=24hr	20.5	18.8	19.0	19.4	286.8	255.0	222.3	254.7	133.8	88.0	79.2	100.3	46.2	60.4	179.5	95.4	26.8	27.7	25.0	26.5
t=48hr	16.2	14.9	NA	15.6	283.8	283.8	209.1	258.9	82.0	76.5	116.7	91.7	38.3	52.7	46.6	45.8	27.3	25.1	26.8	26.4
t=72hr	13.3	11.2	12.0	12.2	219.7	246.7	200.3	222.2	148.7	96.9	84.8	110.1	84.7	55.9	90.1	76.9	23.2	24.1	23.6	23.6

SHADED CELLS = data become suspect due to limitations in measureing particles > 600 microns

FLUME TESTS 1 THROUGH 5

TPH RESULTS IN FILTERED AND UNFILTER WATERS

Laboratory Name: Battelle Duxbury Operations
Project Name:Bitor Flumw Tests 1& 2
Project Number: G003477-0002
File Created by: RMU 11-23-98
File Name: C:\Active\GCProjects\Fingerprinting\Bitor\Bitor 11-98.xls

Battelle ID:	WA41BS	WB71BS		WA41BS	WB71BS
Client/Field ID:	NA	NA			
Batch ID:	98-355	98-386			
Analyte	Amt Rec'd (ug)	Amt Rec'd (ug)	EN04 50 ul (ug)	% Recovered	% Recovered
C10	2.23	1.85	2.49	90	74
C12	1.58	1.38	2.50	63	55
C14	1.66	1.37	2.48	67	55
C16	1.88	1.88	2.49	76	75
Pristane	2.08	2.26	2.60	80	87
C18	1.80	1.91	2.50	72	77
Phytane	2.15	2.26	2.35	91	96
C19	2.33	2.42	2.50	93	97
C20	2.01	2.10	2.49	81	84
C22	2.02	2.17	2.49	81	87
C24	1.98	2.18	2.50	79	87
C26	1.87	2.10	2.50	75	84
C28	1.73	2.26	2.50	69	90
C30	1.74	2.16	2.48	70	87
C36	1.63	2.66	2.50	65	107
OTP % Recovery:				78	88

FLUME TEST 1

Laboratory Name: Battelle Duxbury Operations
Project Name:Bitor Flumw Tests 1& 2
Project Number: G003477-0002
File Created by: RMU 11-23-98
File Name: C:\Active\GCProjects\Fingerprinting\Bitor\Bitor 11-98.xls
Data reported in ug/l

BDO Sample ID:	WA40PB	WA41BS	UU31FIL	UU36FIL	UU40FIL-REP1	UU40FIL-REP2
Client Field ID:	Procdural Blank	Blank Spike	t= 1 hr Filtered	t=24 hr Filtered	t=72 hr d=35 cm Filtered	t=72 hr d=35 cm Filtered
Batch ID:	98-355	98-355	98-355	98-355	98-355	98-355
Matrix:	Water	Water	Water	Water	Water	Water
SampleVolumet (l):	1.00	1.00	0.67	0.55	0.61	0.65
Reporting Units:	ug/l	ug/l	ug/l	ug/l	ug/l	ug/l
Total Sample Area:	28441.57	NA	40241.23	41293.64	40156.63	40523.48
Total DCM:	28785.70	NA	28785.70	28785.70	28785.70	28785.70
Surrogate Area:	52.06	65.74	68.71	60.48	59.99	56.31
Internal Std. Area:	163.88	165.36	164.57	165.19	157.40	158.12
Total Area:	-560.07	NA	11222.25	12282.27	11153.54	11523.35
Internal Std Amount (ug):	20.660	20.660	20.660	20.660	20.660	20.660
Surrogate Amount (ug):	9.839	9.839	9.839	9.839	9.839	9.839
PAH Internal Std. Amt (ug):	3.101	3.101	3.101	3.101	3.101	3.101
PAH Surrogate Amount (ug):	2.020	2.020	2.020	2.020	2.020	2.020
Dilution Factor:	1	1	1	1	1	1
Average Response Factor:	0.913	0.913	0.913	0.913	0.913	0.913
OTP RF	1.072	1.072	1.072	1.072	1.072	1.072
Surrogate Recovery:	62	78	82	72	75	70
Total THC (ug/l):	ND	NA	2295.5	3049.8	2620.3	2529.2

FLUME TEST 1

Laboratory Name: Battelle Duxbury Operations
Project Name:Bitor Flumw Tests 1& 2
Project Number: G003477-0002
File Created by: RMU 11-23-98
File Name: C:\Active\GCProjects\Fingerprinting\Bitor\Bitor 11-98.xls
Data reported in ug/l

BDO Sample ID:	UU40FIL-REP3	UU31-D	UU36-D	UU40-REP1-D	UU40-REP2-D	UU40-REP3-D
Client Field ID:	t=72 hr d=35 cm	t=1 hr	t=24 hr	t=72 hr d=35 cm	t=72 hr d=35 cm	t=72 hr d=35 cm
	Filtered	Unfiltered	Unfiltered	Unfiltered	Unfiltered	Unfiltered
Batch ID:	98-355	98-355	98-355	98-355	98-355	98-355
Matrix:	Water	Water	Water	Water	Water	Water
SampleVolumet (l):	0.65	0.79	0.70	0.74	0.80	0.85
Reporting Units:	ug/l	ug/l	ug/l	ug/l	ug/l	ug/l
Total Sample Area:	41898.97	55391.3	53418.2	54614.2	50817.1	56527.2
Total DCM:	28785.70	28785.70	28785.70	28785.70	28785.70	28785.70
Surrogate Area:	69.79	7.03	13.6	24.0	19.2	27.0
Internal Std. Area:	183.21	183.08	177.9	167.0	169.2	185.9
Total Area:	12860.27	26415.48	24441.07	25637.49	21842.92	27528.62
Internal Std Amount (ug):	20.660	20.660	20.660	20.660	20.660	20.660
Surrogate Amount (ug):	9.839	614.940	614.940	614.940	614.940	614.940
PAH Internal Std. Amt (ug):	3.101	3.101	3.101	3.101	3.101	3.101
PAH Surrogate Amount (ug):	2.020	126.240	126.240	126.240	126.240	126.240
Dilution Factor:	1	500	400	200	250	200
Average Response Factor:	0.913	0.913	0.913	0.913	0.913	0.913
OTP RF	1.072	1.072	1.072	1.072	1.072	1.072
Surrogate Recovery:	75	60	96	90	89	91
Total THC (ug/l):	2435.8	1984565.8	1703092.5	903937.8	872258.4	758019.8

FLUMETEST 2

Laboratory Name: Battelle Duxbury Operations
Project Name:Bitor Flumw Tests 1& 2
Project Number: G003477-0002
File Created by: RMU 11-23-98
File Name: C:\Active\GCProjects\Fingerprinting\Bitor\Bitor 11-98.xls
Data reported in ug/l

BDO Sample ID:	WB70PB	WB71BS	UU43FIL	UU48FIL	UU51FIL-REP1	UU51FIL-REP2
Client Field ID:	Procdural Blank	Blank Spike	t= 1 hr Filtered	t=24 hr Filtered	t=72 hr d=35 cm Filtered	t=72 hr d=35 cm Filtered
Batch ID:	98-386	98-386	98-386	98-386	98-386	98-386
Matrix:	Water	Water	Water	Water	Water	Water
SampleVolumet (l):	1.00	1.00	0.95	0.89	0.92	0.85
Reporting Units:	ug/l	ug/l	ug/l	ug/l	ug/l	ug/l
Total Sample Area:	21533.05	NA	47401.80	41931.68	40287.27	39225.81
Total DCM:	27722.98	NA	27722.98	27722.98	27722.98	27722.98
Surrogate Area:	87.16	104.67	104.97	100.83	106.35	94.44
Internal Std. Area:	221.82	235.12	247.15	243.39	255.43	236.75
Total Area:	-6498.91	NA	19326.70	13864.48	12202.51	11171.64
Internal Std Amount (ug):	20.660	20.660	20.660	20.660	20.660	20.660
Surrogate Amount (ug):	9.839	9.839	9.839	9.839	9.839	9.839
PAH Internal Std. Amt (ug):	3.101	3.101	3.101	3.101	3.101	3.101
PAH Surrogate Amount (ug):	2.020	2.020	2.020	2.020	2.020	2.020
Dilution Factor:	1	1	1	1	1	1
Average Response Factor:	0.896	0.896	0.896	0.896	0.896	0.896
OTP RF	1.061	1.061	1.061	1.061	1.061	1.061
Surrogate Recovery:	78	88	84	82	82	79
Total THC (ug/l):	ND	NA	1892.6	1470.1	1191.8	1274.0

FLUMETEST 2

Laboratory Name: Battelle Duxbury Operations
Project Name:Bitor Flumw Tests 1& 2
Project Number: G003477-0002
File Created by: RMU 11-23-98
File Name: C:\Active\GCProjects\Fingerprinting\Bitor\Bitor 11-98.xls
Data reported in ug/l

BDO Sample ID:	UU51FIL-REP3	UU43-D	UU48-D	UU51-REP1-D	UU51-REP2-D	UU51-REP3-D
Client Field ID:	t=72 hr d=35 cm	t=1 hr	t=24 hr	t=72 hr d=35 cm	t=72 hr d=35 cm	t=72 hr d=35 cm
	Filtered	Unfiltered	Unfiltered	Unfiltered	Unfiltered	Unfiltered
Batch ID:	98-386	98-386	98-386	98-386	98-386	98-386
Matrix:	Water	Water	Water	Water	Water	Water
SampleVolumet (l):	0.8	0.95	0.8700	0.8600	0.8600	0.8500
Reporting Units:	ug/l	ug/l	ug/l	ug/l	ug/l	ug/l
Total Sample Area:	39053.79	51936.29	56017.8	70731.8	66638.5	68491.7
Total DCM:	27722.98	27722.98	27722.98	27722.98	27722.98	27722.98
Surrogate Area:	92.39	2.10	23.2	50.5	52.5	52.3
Internal Std. Area:	223.17	239.17	252.2	241.3	250.0	247.1
Total Area:	11015.25	23972.04	28019.42	42716.99	38613.06	40469.35
Internal Std Amount (ug):	20.660	20.660	20.660	20.660	20.660	20.660
Surrogate Amount (ug):	9.839	49.195	49.195	9.839	9.839	9.839
PAH Internal Std. Amt (ug):	3.101	3.101	3.101	3.101	3.101	3.101
PAH Surrogate Amount (ug):	2.020	10.099	10.099	2.020	2.020	2.020
Dilution Factor:	1	250	25	2	2	2
Average Response Factor:	0.896	0.896	0.896	0.896	0.896	0.896
OTP RF	1.061	1.061	1.061	1.061	1.061	1.061
Surrogate Recovery:	82	87	91	83	83	84
Total THC (ug/l):	1416.2	604713.2	73222.5	9479.8	8272.0	8874.3

Laboratory Name: Battelle
Project Name: Bitor 98 - Flume Test #3-5
Batch 98-393 G003477-0002
File Name: C:\Active\Bitor\TblsTest3-5.xls
Entered By: LGR, 01-18-99
Data reported in ug

Client/Field ID: Blank Spike
BOS Sample ID: WB94BS
Batch ID: 98-393

Analyte	Amt Rec'd (ug)	EN04 100 ul (ug)	% Recovered
C9	0.10	4.95	2 &
C10	0.24	4.98	5 &
C12	0.95	5.00	19 &
C14	2.12	4.95	43
C16	2.82	4.97	57
Pristane	4.31	5.20	83
C18	3.26	4.99	65
Phytane	3.79	4.70	81
C19	4.33	5.00	87
C20	3.95	4.98	79
C22	4.46	4.98	89
C24	5.05	5.00	101
C26	5.48	5.00	110
C28	7.65	4.99	153 &
C30	5.82	4.95	118
C36	5.21	4.99	104
OTP % Recovery:			83

& Analyte recovery outside of program criteria

Laboratory Name: Battelle
Project Name: Bitor 98 - Flume Test #3-5
Batch 98-393 G003477-0002
File Name: C:\Active\Bitor\TblsTest3-5.xls
Entered By: LGR, 01-18-99
Data reported in ug/L

FLUME TEST 3

Client/Field ID:	Procedural Blank	Blank Spike	US59, t = 1hr.	US59, t = 24hr.	US59, t = 72hr. @ 35 cm	US59, t = 72hr. @ 35 cm	US59, t = 72hr. @ 35 cm
BOS Sample ID:	WB93PB	WB94BS	UU55-D	UU60-D	UU63REP1-D	UU63REP2-D	UU63REP3-D
Batch ID:	98-393	98-393	98-393	98-393	98-393	98-393	98-393
Matrix:	Water	Water	Water	Water	Water	Water	Water
Sample Volume (L):	1.000	1.000	0.780	0.890	0.860	0.860	0.870
Sample Area:	10996.99	NA	100370.45	88464.57	83656.61	81591.70	88435.19
DCM Area:	11378.77	NA	11378.77	11378.77	11378.77	11378.77	11378.77
Surrogate Area:	234.09	212.57	85.58	742.75	54.50	52.73	55.88
Internal Std. Area:	268.79	244.49	282.38	252.68	270.77	266.81	284.22
Std. Corrected Total Area:	ND	NA	88623.72	76090.37	71952.56	69893.38	76716.31
Internal Std Amount (ug):	20.66	20.66	20.66	20.66	20.66	20.66	20.66
Surrogate Amount (ug):	19.68	19.68	614.94	614.94	19.68	19.68	19.68
PAH Int. Std. Amt (ug):	3.10	3.10	3.10	3.10	3.10	3.10	3.10
PAH Surrogate Amount (ug):	4.04	4.04	126.24	126.24	4.04	4.04	4.04
Dilution Factor:	1.00	1.00	100.00	10.00	5.00	5.00	5.00
Average Response Factor:	1.002	1.002	1.002	1.002	1.002	1.002	1.002
OTP RF	1.101	1.101	1.101	1.101	1.101	1.101	1.101
Surrogate Recovery:	83	83	92	90	96	94	94
Total Hydrocarbons (ug/L):	ND	NA	829076	69587	31832	31380	31962

NA-Not Applicable
ND-Not Detected

FLUME TEST 3

Laboratory Name: Battelle
Project Name: Bitor 98 - Flume Test #3-5
Batch 98-393 G003477-0002
File Name: C:\Active\Bitor\TblsTest3-5.xls
Entered By: LGR, 01-18-99
Data reported in ug/L

Client/Field ID:	US59, t = 1hr. Filtered	US59, t = 24hr. Filtered	US59, t = 72hr. Filtered @ 35 cm	US59, t = 72hr. Filtered @ 35 cm	US59, t = 72hr. Filtered @ 35 cm
BOS Sample ID:	UU55FIL	UU60FIL	UU63FILREP1	UU63FILREP2	UU63FILREP3
Batch ID:	98-393	98-393	98-393	98-393	98-393
Matrix:	Water	Water	Water	Water	Water
Sample Volume (L):	0.900	0.830	0.870	0.800	0.870
Sample Area:	28085.82	25049.71	31101.95	29289.53	31036.85
DCM Area:	11378.77	11378.77	11378.77	11378.77	11378.77
Surrogate Area:	238.26	229.73	269.09	249.91	254.46
Internal Std. Area:	269.44	253.59	291.37	265.32	274.55
Std. Corrected Total Area:	16199.35	13187.61	19162.72	17395.53	19129.06
Internal Std Amount (ug):	20.66	20.66	20.66	20.66	20.66
Surrogate Amount (ug):	19.68	19.68	19.68	19.68	19.68
PAH Int. Std. Amt (ug):	3.10	3.10	3.10	3.10	3.10
PAH Surrogate Amount (ug):	4.04	4.04	4.04	4.04	4.04
Dilution Factor:	1.00	1.00	1.00	1.00	1.00
Average Response Factor:	1.002	1.002	1.002	1.002	1.002
OTP RF	1.101	1.101	1.101	1.101	1.101
Surrogate Recovery:	84	86	88	90	88
Total Hydrocarbons (ug/L):	1369	1283	1550	1681	1643

NA-Not Applicable
ND-Not Detected

Laboratory Name: Battelle
Project Name: Bitor 98 - Flume Test #3-5
Batch 98-398 G003477-0002
File Name: C:\Active\Bitor\TblsTest3-5.xls
Entered By: LGR, 01-18-99
Data reported in ug

Client/Field ID: Blank Spike
BOS Sample ID: WC11BS
Batch ID: 98-398

Analyte	Amt Rec'd (ug)	EN04 100 ul (ug)	% Recovered
C9	1.12	4.95	23 &
C10	1.36	4.98	27 &
C12	1.82	5.00	36 &
C14	2.44	4.95	49
C16	3.02	4.97	61
Pristane	4.08	5.20	78
C18	3.14	4.99	63
Phytane	3.40	4.70	72
C19	3.90	5.00	78
C20	3.35	4.98	67
C22	3.34	4.98	67
C24	3.37	5.00	67
C26	3.37	5.00	67
C28	3.86	4.99	77
C30	3.57	4.95	72
C36	3.80	4.99	76
OTP % Recovery:			73

& Analyte recovery outside of program criteria

Laboratory Name: Battelle
Project Name: Bitor 98 - Flume Test #3-5
Batch 98-398 G003477-0002
File Name: C:\Active\Bitor\TblsTest3-5.xls
Entered By: LGR, 01-18-99
Data reported in ug/L

FLUME TEST 4

Client/Field ID:	Procedural Blank	Blank Spike	US59, t = 1hr.	US59, t = 24hr.	US59, t = 72hr. @ 35 cm	US59, t = 72hr. @ 35 cm	US59, t = 72hr. @ 35 cm
BOS Sample ID:	WC10PB	WC11BS	UU67-D	UU72-D	UU75REP1-D	UU75REP2-D	UU75REP3-D
Batch ID:	98-398	98-398	98-398	98-398	98-398	98-398	98-398
Matrix:	Water	Water	Water	Water	Water	Water	Water
Sample Volume (L):	1.000	1.000	0.830	0.880	0.870	0.830	0.840
Sample Area:	10179.42	NA	64734.54	45238.35	64943.31	45264.43	64186.78
DCM Area:	9396.82	NA	9396.82	9396.82	9396.82	9396.82	9396.82
Surrogate Area:	167.11	126.25	314.71	402.27	706.51	388.81	668.76
Internal Std. Area:	195.99	174.00	176.58	188.42	161.38	172.47	147.84
Std. Corrected Total Area:	419.50	NA	54846.43	35250.84	54678.60	35306.34	53973.35
Internal Std Amount (ug):	20.66	20.66	20.66	20.66	20.66	20.66	20.66
Surrogate Amount (ug):	19.68	19.68	922.41	922.41	922.41	922.41	922.41
PAH Int. Std. Amt (ug):	3.10	3.10	3.10	3.10	3.10	3.10	3.10
PAH Surrogate Amount (ug):	4.04	4.04	189.36	189.36	189.36	189.36	189.36
Dilution Factor:	1.00	1.00	25.00	20.00	10.00	20.00	10.00
Average Response Factor:	0.943	0.943	0.943	0.943	0.943	0.943	0.943
OTP RF	1.041	1.041	1.041	1.041	1.041	1.041	1.041
Surrogate Recovery:	86	73	96	92	94	97	97
Total Hydrocarbons (ug/L):	40	NA	204642	92868	85068	107770	94955

NA-Not Applicable
ND-Not Detected

Laboratory Name: Battelle
Project Name: Bitor 98 - Flume Test #3-5
Batch 98-398 G003477-0002
File Name: C:\Active\Bitor\TblsTest3-5.xls
Entered By: LGR, 01-18-99
Data reported in ug/L

FLUME TEST 4

Client/Field ID:	US59, t = 1hr. Filtered	US59, t = 24hr. Filtered	US59, t = 72hr. Filtered @ 35 cm	US59, t = 72hr. Filtered @ 35 cm	US59, t = 72hr. Filtered @ 35 cm
BOS Sample ID:	UU67FIL	UU72FIL	UU75FILREP1	UU75REP2FIL	UU75REP3FIL
Batch ID:	98-398	98-398	98-398	98-398	98-398
Matrix:	Water	Water	Water	Water	Water
Sample Volume (L):	0.910	0.880	0.830	0.850	0.870
Sample Area:	21210.04	18994.21	19722.25	20950.27	19986.81
DCM Area:	9396.82	9396.82	9396.82	9396.82	9396.82
Surrogate Area:	172.87	138.77	144.83	138.81	131.11
Internal Std. Area:	201.73	168.66	183.86	164.40	155.71
Std. Corrected Total Area:	11438.62	9289.96	9996.74	11250.24	10303.18
Internal Std Amount (ug):	20.66	20.66	20.66	20.66	20.66
Surrogate Amount (ug):	19.68	19.68	19.68	19.68	19.68
PAH Int. Std. Amt (ug):	3.10	3.10	3.10	3.10	3.10
PAH Surrogate Amount (ug):	4.04	4.04	4.04	4.04	4.04
Dilution Factor:	1.00	1.00	1.00	1.00	1.00
Average Response Factor:	0.943	0.943	0.943	0.943	0.943
OTP RF	1.041	1.041	1.041	1.041	1.041
Surrogate Recovery:	86	83	79	85	85
Total Hydrocarbons (ug/L):	1357	1363	1427	1755	1658

NA-Not Applicable
ND-Not Detected

Laboratory Name: Battelle
Project Name: Bitor 98 - Flume Test #3-5
Batch 98-446 G003477-0002
File Name: C:\Active\Bitor\TblsTest3-5.xls
Entered By: LGR, 01-18-99
Data reported in ug

Client/Field ID:	Blank Spike
BOS Sample ID:	WD77BS
Batch ID:	98-446

Analyte	Amt Rec'd (ug)	EN04 100 ul (ug)	% Recovered
C9	2.73	4.95	55
C10	2.79	4.98	56
C12	3.26	5.00	65
C14	3.53	4.95	71
C16	3.77	4.97	76
Pristane	5.31	5.20	102
C18	4.07	4.99	82
Phytane	4.55	4.70	97
C19	5.21	5.00	104
C20	4.74	4.98	95
C22	5.10	4.98	102
C24	5.44	5.00	109
C26	5.71	5.00	114
C28	8.29	4.99	166 &
C30	6.27	4.95	127 &
C36	6.73	4.99	135 &
OTP % Recovery:			87

& - Analyte recovery outside of program criteria
NA - Not Applicable

FLUME TEST 5

Laboratory Name: Battelle
Project Name: Bitor 98 - Flume Test #3-5
Batch 98-446 G003477-0002
File Name: C:\Active\Bitor Tbls8-98.xls
Entered By: LGR, 01-18-99, updated 1-21-99 LGR
Data reported in ug/L

Client/Field ID:	Procedural Blank	Blank Spike	US59, t = 1hr.	US59, t = 24hr.	US59, t = 72hr. @ 35 cm	US59, t = 72hr. @ 35 cm	US59, t = 72hr. @ 35 cm
BOS Sample ID:	WD76PB	WD77BS	UU79-D	UU84-D	UU87REP1-D	UU87REP2-D	UU87REP3-D
Batch ID:	98-446	98-446	98-446	98-446	98-446	98-446	98-446
Matrix:	Water	Water	Water	Water	Water	Water	Water
Sample Volume (L):	1.000	1.000	0.930	0.940	0.910	0.900	0.8900
Sample Area:	14648.84	NA	139224.09	125837.47	142328.42	143364.94	139717.13
DCM Area:	14484.20	NA	14484.20	14484.20	14484.20	14484.20	14484.20
Surrogate Area:	227.68	233.00	127.08	242.22	1215.73	1126.03	1191.77
Internal Std. Area:	260.03	254.75	254.89	238.71	250.81	255.68	249.55
Std. Corrected Total Area:	ND	NA	124357.92	110872.34	126377.68	127499.03	123791.60
Internal Std Amount (ug):	20.66	20.66	20.66	20.66	20.66	20.66	20.66
Surrogate Amount (ug):	19.68	19.68	922.41	922.41	922.41	922.41	922.41
PAH Int. Std. Amt (ug):	3.10	3.10	3.10	3.10	3.10	3.10	3.10
PAH Surrogate Amount (ug):	4.04	4.04	189.36	189.36	189.36	189.36	189.36
Dilution Factor:	1.00	1.00	100.00	50.00	10.00	10.00	10.00
Average Response Factor:	1.002	1.002	1.002	1.002	1.002	1.002	1.002
OTP RF	1.101	1.101	1.101	1.101	1.101	1.101	1.101
Surrogate Recovery:	83	87	101	103	99	90	97
Total Hydrocarbons (ug/L):	ND	NA	1081147	509025	113928	114000	114676

NA-Not Applicable
ND-Not Detected

FLUME TEST 5

Laboratory Name: Battelle
Project Name: Bitor 98 - Flume Test #3-5
Batch 98-446 G003477-0002
File Name: C:\Active\Bitor Tbls8-98.xls
Entered By: LGR, 01-18-99
Data reported in ug/L

Client/Field ID:	US59, t = 1hr. Filtered	US59, t = 24hr. Filtered	US59, t = 72hr. Filtered @ 35 cm	US59, t = 72hr. Filtered @ 35 cm	US59, t = 72hr. Filtered @ 35 cm
BOS Sample ID:	UU79FIL	UU84FIL	UU87FILREP1	UU87FILREP2	UU87FILREP3
Batch ID:	98-446	98-446	98-446	98-446	98-446
Matrix:	Water	Water	Water	Water	Water
Sample Volume (L):	0.860	0.890	0.860	0.750	0.870
Sample Area:	34764.34	37744.23	49095.65	43648.95	44358.05
DCM Area:	14484.20	14484.20	14484.20	14484.20	14484.20
Surrogate Area:	256.57	189.35	259.28	251.78	250.20
Internal Std. Area:	247.50	248.79	251.35	246.11	264.45
Std. Corrected Total Area:	19776.06	22821.88	34100.81	28666.86	29359.19
Internal Std Amount (ug):	20.66	20.66	20.66	20.66	20.66
Surrogate Amount (ug):	19.68	19.68	19.68	19.68	19.68
PAH Int. Std. Amt (ug):	3.10	3.10	3.10	3.10	3.10
PAH Surrogate Amount (ug):	4.04	4.04	4.04	4.04	4.04
Dilution Factor:	1.00	1.00	1.00	1.00	1.00
Average Response Factor:	1.002	1.002	1.002	1.002	1.002
OTP RF	1.101	1.101	1.101	1.101	1.101
Surrogate Recovery:	99	73	98	98	90
Total Hydrocarbons (ug/L):	1907	2117	3245	3193	2623

NA-Not Applicable
ND-Not Detected

FLUME TESTS 1 THROUGH 5

PAH RESULTS IN FILTERED AND UNFILTER WATERS

Laboratory Name: Battelle - Duxbury
Project Name: Bitor America - Flume Testing and Characterization of Orimulsion 400 (1998)

Project Number G003477-0002
FILE: D:\Active\Bitor\98355.xls CREATED WN 12/21/98

FLUME TEST 1 -FILTERED & UNFILTERED WATERS

Table 1. Surrogate Corrected Filtered Freshwater PAH concentrations in ug/L

Battelle ID	WA40PB	UU31FIL	UU36FIL	UU40FIL-REP1	UU40FIL-REP3	UU31-D	UU36-D	UU40REP3-D
Client ID	NA	t = 1hr filtered water	t = 24hr filtered water	t = 72hr d = 35cm filtered water rep 1	t = 72hr d = 35cm filtered water rep 3	t = 1hr unfiltered water	t = 24hr unfiltered water	t = 72hr d = 35cm unfiltered water rep 3
Matrix	Freshwater	Freshwater	Freshwater	Freshwater	Freshwater	Freshwater	Freshwater	Freshwater
Sample Volume (L)	1.000	0.670	0.550	0.610	0.650	0.790	0.700	0.850
Naphthalene	0.048	0.524	0.459	0.179	0.208	31.106	13.224	2.365
C1-naphthalenes	0.031	0.591	0.190	0.157	0.292	90.376	38.393	4.137
C2-naphthalenes	0.012	0.497	0.151	0.206	0.307	321.240	182.611	27.278
C3-naphthalenes	0.000 ND	0.395	0.232	0.217	0.260	612.553	435.181	108.302
C4-naphthalenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	1200.965	941.178	417.929
Biphenyl	0.000 ND	0.026	0.000 ND	0.010	0.014	7.241	0.000 ND	0.636
Acenaphthylene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Acenaphthene	0.000 ND	0.103	0.000 ND	0.000 ND	0.055	28.631	16.685	0.000 ND
Dibenzofuran	0.000 ND	0.043	0.000 ND	0.021	0.025	14.168	11.921	3.913
Fluorene	0.000 ND	0.070	0.000 ND	0.000 ND	0.043	27.682	17.683	2.072
C1-fluorenes	0.000 ND	0.181	0.000 ND	0.000 ND	0.000 ND	174.870	131.832	37.569
C2-fluorenes	0.000 ND	0.719	0.000 ND	0.000 ND	0.000 ND	649.951	463.401	176.874
C3-fluorenes	0.000 ND	0.433	0.000 ND	0.000 ND	0.000 ND	1121.918	921.659	350.657
Anthracene	0.000 ND	0.037	0.070	0.034	0.038	0.000 ND	13.887	4.133
Phenanthrene	0.000 ND	0.114	0.060	0.052	0.075	146.905	88.979	13.549
C1-phenanthrenes/anthracenes	0.000 ND	0.118	0.103	0.105	0.072	395.340	288.349	71.982
C2-phenanthrenes/anthracenes	0.000 ND	0.167	0.207	0.208	0.144	1343.784	1081.259	423.206
C3-phenanthrenes/anthracenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	1927.901	1594.126	628.254
C4-phenanthrenes/anthracenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	1124.059	954.254	413.238
Dibenzothiophene	0.000 ND	0.869	1.539	1.377	1.089	87.437	81.206	16.206
C1-dibenzothiophenes	0.000 ND	0.140	0.263	0.237	0.108	524.873	405.607	126.065
C2-dibenzothiophenes	0.000 ND	1.032	1.647	1.364	0.844	1823.144	1517.301	530.861
C3-dibenzothiophenes	0.000 ND	0.560	0.860	0.803	0.541	3570.778	3041.287	1195.196
C4-dibenzothiophenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	3037.234	2336.407	1032.728
Fluoranthene	0.000 ND	0.007	0.000 ND	0.000 ND	0.000 ND	17.438	13.650	6.016
Pyrene	0.000 ND	0.012	0.000 ND	0.000 ND	0.000 ND	49.510	39.029	16.371
C1-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	209.817	174.491	67.467
C2-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	417.204	331.884	140.540
C3-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	590.010	469.901	199.660
Benz(a)anthracene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	17.487	14.303	6.333
Chrysene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	69.769	46.805	20.368
C1-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	153.037	125.032	64.837
C2-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	209.605	174.541	74.684
C3-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	169.982	127.258	54.769
C4-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	79.365	49.546	31.454
Benzo(b)fluoranthene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	9.890	7.298	3.412
Benzo(k)fluoranthene	0.000 ND	0.000 ND	0.000 ND	0.002	0.000 ND	2.024	0.000 ND	0.000 ND
Benzo(e)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	14.897	9.099	6.336
Benzo(a)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	10.710	11.079	4.244
Perylene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	33.962	25.656	11.767
Indeno(1,2,3-c,d)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Dibenz(a,h)anthracene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Benzo(g,h,i)perylene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	6.395	4.792	2.651
Diagnostic Ratios								
Total PAH's	0.09	6.64	5.78	4.97	4.11	20303.67	16198.69	6285.27
D2/P2	NA	6.18	7.95	6.56	5.87	1.36	1.40	1.25
D3/P3	NA	NA	NA	NA	NA	1.90	1.91	1.90
Surrogate Recoveries								
Naphthalene-d8	49	61	60	73	61	105	100	101
Phenanthrene-d10	63	89	78	84	83	104	106	103
Chrysene-d12	67	87	77	83	77	104	105	100

Laboratory Name: Battelle - Duxbury
Project Name: Bitor America - Flume Testing and Characterization of Orimulsion 400 (1998)

Project Number: G003477-0002
FILE: D:\Active\Bitor\98386.xls CREATED: WN 12/21/98

FLUME TEST 2 -FILTERED & UNFILTERED WATERS

Table 1. Surrogate Corrected Filtered Brackishwater PAH concentrations in ug/L.

Battelle ID	WB70PB	UU43FIL	UU48FIL	UU51FILREP2	UU43-D	UU48-D	UU51REP2-D
Client ID	NA	US59, t = 1hr - Filtered	US59, t = 24hr - Filtered	US59, t = 72hr - Filtered @ 35cm	US59, t = 1hr	US59, t = 24hr	US59, t = 72hr @ 35cm
Matrix	Brackishwater	Brackishwater	Brackishwater	Brackishwater	Brackishwater	Brackishwater	Brackishwater
Sample Volume (L)	1.000	0.950	0.890	0.850	0.950	0.870	0.860
Naphthalene	0.056	0.077	0.047	0.025	2.130	0.216	0.041
C1-naphthalenes	0.137	0.128	0.051	0.069	2.498	0.358	0.091
C2-naphthalenes	0.000 ND	0.209	0.057	0.024	12.072	0.581	0.012
C3-naphthalenes	0.000 ND	0.326	0.036	0.000 ND	82.571	1.033	0.004
C4-naphthalenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	262.595	14.498	0.000 ND
Biphenyl	0.000 ND	0.006	0.000 ND	0.000 ND	0.615	0.000 ND	0.004
Acenaphthylene	0.000 ND	0.278	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.017
Acenaphthene	0.000 ND	0.067	0.000 ND	0.000 ND	3.428	0.000 ND	0.007
Dibenzofuran	0.000 ND	0.051	0.000 ND	0.000 ND	4.011	0.145	0.006
Fluorene	0.000 ND	0.281	0.000 ND	0.000 ND	4.272	0.158	0.016
C1-fluorenes	0.000 ND	0.516	0.000 ND	0.000 ND	40.703	1.172	0.024
C2-fluorenes	0.000 ND	0.487	0.000 ND	0.000 ND	157.190	11.990	0.000 ND
C3-fluorenes	0.000 ND	0.150	0.000 ND	0.000 ND	327.893	32.420	0.000 ND
Anthracene	0.000 ND	0.025	0.009	0.007	4.181	0.000 ND	0.043
Phenanthrene	0.000 ND	0.062	0.020	0.014	14.544	0.371	0.037
C1-phenanthrenes/anthracenes	0.000 ND	0.207	0.054	0.000 ND	80.095	3.333	0.009
C2-phenanthrenes/anthracenes	0.000 ND	0.161	0.000 ND	0.000 ND	370.670	38.943	0.067
C3-phenanthrenes/anthracenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	524.143	71.472	0.000 ND
C4-phenanthrenes/anthracenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	305.082	45.393	0.000 ND
Dibenzothiophene	0.000 ND	0.608	0.000 ND	0.000 ND	15.033	0.415	0.036
C1-dibenzothiophenes	0.000 ND	0.165	0.000 ND	0.000 ND	129.680	8.634	0.023
C2-dibenzothiophenes	0.000 ND	0.333	0.000 ND	0.000 ND	506.333	49.765	0.034
C3-dibenzothiophenes	0.000 ND	0.414	0.000 ND	0.000 ND	971.236	131.014	0.000 ND
C4-dibenzothiophenes	0.000 ND	0.239	0.000 ND	0.000 ND	811.493	117.215	0.000 ND
Fluoranthene	0.000 ND	0.007	0.000 ND	0.000 ND	4.477	0.534	0.033
Pyrene	0.000 ND	0.003	0.000 ND	0.000 ND	14.748	2.048	0.153
C1-fluoranthenes/pyrenes	0.000 ND	0.077	0.000 ND	0.000 ND	58.844	8.054	0.000 ND
C2-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	117.448	16.908	0.000 ND
C3-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	166.893	23.470	0.000 ND
Benz(a)anthracene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	4.144	0.569	0.034
Chrysene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	13.828	2.011	0.137
C1-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	38.049	5.135	0.365
C2-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	51.750	7.776	0.518
C3-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	36.842	5.716	0.460
C4-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	21.807	2.523	0.237
Benzo(b)fluoranthene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	2.503	0.374	0.030
Benzo(k)fluoranthene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.008
Benzo(e)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	3.557	0.539	0.051
Benzo(a)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	3.523	0.747	0.054
Perylene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	7.711	1.091	0.095
Indeno(1,2,3-c,d)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.006
Dibenz(a,h)anthracene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.002
Benzo(g,h,i)perylene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	1.490	0.251	0.020
Diagnostic Ratios							
Total PAH's	0.19	4.88	0.27	0.14	5178.09	606.87	2.67
D2/P2	NA	2.07	NA	NA	1.37	1.28	0.51
D3/P3	NA	NA	NA	NA	1.85	1.83	NA
Surrogate Recoveries							
Naphthalene-d8	2 &	39 &	55	61	94	73	42
Phenanthrene-d10	72	92	89	85	96	82	97
Chrysene-d12	90	90	92	87	109	97	105

Laboratory Name: Battelle - Duxbury

Project Name: *Bitor America - Flume Testing and Characterization of Orimulsion 400 (1998)*
Project Number: G003477-0002
FILE: D:\Active\Bitor\98393.xls CREATED: WN 1/6/99

FLUME TEST 3 -FILTERED & UNFILTERED WATERS

Table 1. Surrogate Corrected Saltwater PAH concentrations in ug/L

Battelle ID	WB93PB	UU55FIL	UU60FIL	UU63FILREP1	UU55-D	UU60-D	UU63REP1-D
Client ID	NA	US59, t = 1hr - Filtered	US59, t = 24hr - Filtered	US59, t = 72hr - Filtered @ 35cm	US59, t = 1hr	US59, t = 24hr	US59, t = 72hr @ 35cm
Matrix	Saltwater	Saltwater	Saltwater	Saltwater	Saltwater	Saltwater	Saltwater
Sample Volume (L)	1.000	0.900	0.830	0.870	0.780	0.890	0.860
Naphthalene	0.000 ND	0.038	0.046	0.030	2.505	0.176	0.093
C1-naphthalenes	0.000 ND	0.051	0.127	0.066	4.290	0.000 ND	0.000 ND
C2-naphthalenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	36.812	0.000 ND	0.000 ND
C3-naphthalenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	138.413	3.360	0.000 ND
C4-naphthalenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	512.063	32.951	4.999
Biphenyl	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Acenaphthylene	0.000 ND	0.015	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Acenaphthene	0.000 ND	0.026	0.000 ND	0.000 ND	8.118	0.000 ND	0.000 ND
Dibenzofuran	0.000 ND	0.000 ND	0.000 ND	0.000 ND	4.700	0.082	0.000 ND
Fluorene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	8.451	0.140	0.000 ND
C1-fluorenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	71.468	3.051	0.000 ND
C2-fluorenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	250.037	17.663	3.373
C3-fluorenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	450.931	36.474	12.754
Anthracene	0.000 ND	0.031	0.012	0.005	6.502	0.240	0.093
Phenanthrene	0.000 ND	0.119	0.016	0.013	40.138	0.560	0.105
C1-phenanthrenes/anthracen	0.000 ND	0.194	0.036	0.024	140.457	6.442	1.286
C2-phenanthrenes/anthracen	0.000 ND	0.148	0.000 ND	0.000 ND	514.480	36.341	10.263
C3-phenanthrenes/anthracen	0.000 ND	0.000 ND	0.000 ND	0.000 ND	685.211	50.213	23.779
C4-phenanthrenes/anthracen	0.000 ND	0.000 ND	0.000 ND	0.000 ND	457.976	27.285	12.803
Dibenzothiophene	0.000 ND	0.246	0.060	0.042	26.321	0.939	0.173
C1-dibenzothiophenes	0.000 ND	0.228	0.046	0.030	136.548	10.084	1.362
C2-dibenzothiophenes	0.000 ND	0.166	0.000 ND	0.000 ND	704.322	48.605	16.138
C3-dibenzothiophenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	1284.415	97.599	40.263
C4-dibenzothiophenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	1029.008	73.853	35.404
Fluoranthene	0.000 ND	0.004	0.000 ND	0.000 ND	5.482	0.449	0.200
Pyrene	0.000 ND	0.011	0.000 ND	0.004	17.702	1.673	0.663
C1-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	71.645	6.965	2.731
C2-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	134.396	10.603	5.289
C3-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	167.642	13.626	7.506
Benz(a)anthracene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	5.194	0.461	0.235
Chrysene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	19.667	1.760	0.926
C1-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	42.684	4.067	1.700
C2-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	55.984	3.408	2.110
C3-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	46.424	2.089	1.185
C4-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Benzo(b)fluoranthene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	3.774	0.255	0.113
Benzo(k)fluoranthene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Benzo(e)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	3.492	0.269	0.133
Benzo(a)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	2.874	0.227	0.050
Perylene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Indeno(1,2,3-c,d)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Dibenz(a,h)anthracene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Benzo(g,h,i)perylene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Diagnostic Ratios							
Total PAH's	NA	1.28	0.34	0.21	7090.13	491.91	185.75
D2/P2	NA	1.12	NA	NA	1.37	1.34	1.57
D3/P3	NA	NA	NA	NA	1.87	1.94	1.69
Surrogate Recoveries							
Naphthalene-d8	4 &	64	13 &	17 &	87	78	64
Phenanthrene-d10	76	92	91	92	95	97	97
Chrysene-d12	89	90	90	88	102	93	99

Laboratory Name: Battelle - Duxbury

Project Name: Bitor America - Flume Testing and Characterization of Orimulsion 400 (1998)
Project Number: G003477-0002
FILE: D:\Active\Bitor\98398.xls CREATED: WN 1/6/99

FLUME TEST 4 -FILTERED & UNFILTERED WATERS

Table 1. Surrogate Corrected Saltwater PAH concentrations in ug/L.

Battelle ID	WC10PB	UU67FIL	UU72FIL	UU75FILREP3	UU67-D	UU72-D	UU75REP3-D
Client ID	NA	US59, t = 1hr - Filtered	US59, t = 24hr - Filtered	US59, t = 72hr - Filtered @ 35cm	US59, t = 1hr	US59, t = 24hr	US59, t = 72hr @ 35cm
Matrix	Saltwater	Saltwater	Saltwater	Saltwater	Saltwater	Saltwater	Saltwater
Sample Volume (L)	1.000	0.910	0.880	0.870	0.830	0.880	0.840
Naphthalene	0.007	0.047	0.035	0.032	0.855	0.244	0.173
C1-naphthalenes	0.008	0.075	0.066	0.069	0.737	0.000 ND	0.000 ND
C2-naphthalenes	0.000 ND	0.000 ND	0.131	0.101	13.213	0.000 ND	3.627
C3-naphthalenes	0.000 ND	0.000 ND	0.169	0.000 ND	38.229	12.142	11.381
C4-naphthalenes	0.000 ND	0.000 ND	0.295	0.000 ND	114.284	47.277	38.454
Biphenyl	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Acenaphthylene	0.000 ND	0.246	0.208	0.258	0.782	0.000 ND	0.641
Acenaphthene	0.000 ND	0.092	0.076	0.067	2.001	0.000 ND	0.575
Dibenzofuran	0.000 ND	0.033	0.026	0.000 ND	0.948	0.171	0.260
Fluorene	0.000 ND	0.124	0.128	0.059	1.681	0.372	0.578
C1-fluorenes	0.000 ND	0.195	0.185	0.125	14.837	5.610	4.727
C2-fluorenes	0.000 ND	0.139	0.155	0.155	61.234	26.327	21.635
C3-fluorenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	102.563	50.578	42.172
Anthracene	0.000 ND	0.018	0.015	0.019	1.161	0.813	0.337
Phenanthrene	0.000 ND	0.050	0.041	0.030	9.498	2.105	3.406
C1-phenanthrenes/anthracenes	0.000 ND	0.092	0.069	0.065	30.754	11.557	11.260
C2-phenanthrenes/anthracenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	116.936	55.729	46.256
C3-phenanthrenes/anthracenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	156.708	71.007	61.930
C4-phenanthrenes/anthracenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	65.441	42.025	36.665
Dibenzothiophene	0.000 ND	0.258	0.222	0.289	5.918	1.912	2.136
C1-dibenzothiophenes	0.000 ND	0.120	0.094	0.181	29.971	12.031	10.698
C2-dibenzothiophenes	0.000 ND	0.170	0.209	0.231	163.320	76.082	62.327
C3-dibenzothiophenes	0.000 ND	0.000 ND	0.139	0.168	286.579	143.542	115.309
C4-dibenzothiophenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	221.927	110.613	88.085
Fluoranthene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	1.086	0.626	0.478
Pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	4.214	2.015	1.702
C1-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	15.486	8.710	8.066
C2-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	31.410	13.913	11.541
C3-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	36.049	18.975	15.102
Benz(a)anthracene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	1.426	0.764	0.578
Chrysene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	4.767	2.255	1.891
C1-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	10.431	5.516	4.143
C2-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	11.382	6.122	3.184
C3-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	4.658	2.499	0.849
C4-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Benzo(b)fluoranthene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.365	0.400	0.245
Benzo(k)fluoranthene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Benzo(e)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.593	0.432	0.236
Benzo(a)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Perylene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Indeno(1,2,3-c,d)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Dibenz(a,h)anthracene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Benzo(g,h,i)perylene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Diagnostic Ratios							
Total PAH's	0.01	1.66	2.26	1.85	1561.46	732.36	610.65
D2/P2	NA	NA	NA	NA	1.40	1.37	1.35
D3/P3	NA	NA	NA	NA	1.83	2.02	1.86
Surrogate Recoveries							
Naphthalene-d8	75	50	48	63	88	82	86
Phenanthrene-d10	84	90	83	91	99	94	98
Chrysene-d12	88	87	79	86	88	85	83

Project Name: Bitor America - Flume Testing and Characterization of Orimulsion 400 (1998)
Project Number: G003477-0002
FILE: D:\Active\Bitor\98446.xls CREATED: WN 1/7/99

FLUME TEST 5 - FILTERED & UNFLITERED WATERS

Table 1. Surrogate Corrected Freshwater PAH concentrations in ug/L.

Battelle ID	WD76PB	UU79FIL	UU84FIL	UU87FILREP1	UU79-D	UU84-D	UU87REP1-D
Client ID	NA	US59, t = 1hr - Filtered	US59, t = 24hr - Filtered	US59, t = 72hr - Filtered @ 35cm	US59, t = 1hr	US59, t = 24hr	US59, t = 72hr @ 35cm
Matrix	Freshwater	Freshwater	Freshwater	Freshwater	Freshwater	Freshwater	Freshwater
Sample Volume (L)	1.000	0.860	0.890	0.860	0.930	0.940	0.910
Naphthalene	0.008	0.151	0.076	0.034	2.949	1.463	0.395
C1-naphthalenes	0.029	0.062	0.060	0.048	0.000 ND	0.000 ND	0.000 ND
C2-naphthalenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
C3-naphthalenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	108.356	14.752	0.000 ND
C4-naphthalenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	527.307	151.470	0.000 ND
Biphenyl	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Acenaphthylene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Acenaphthene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Dibenzofuran	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Fluorene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	3.813	0.546	0.000 ND
C1-fluorenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	31.839	7.609	0.000 ND
C2-fluorenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	246.959	62.678	0.000 ND
C3-fluorenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	463.502	179.305	0.000 ND
Anthracene	0.000 ND	0.023	0.000 ND	0.000 ND	3.050	1.439	0.242
Phenanthrene	0.004	0.062	0.000 ND	0.000 ND	4.798	1.947	0.424
C1-phenanthrenes/anthracenes	0.010	0.000 ND	0.000 ND	0.000 ND	54.427	15.963	0.880
C2-phenanthrenes/anthracenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	463.211	134.699	5.707
C3-phenanthrenes/anthracenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	713.503	302.406	33.145
C4-phenanthrenes/anthracenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	397.070	178.818	31.530
Dibenzothiophene	0.000 ND	0.903	0.308	0.366	7.269	2.332	0.425
C1-dibenzothiophenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	30.584	0.000 ND	0.000 ND
C2-dibenzothiophenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	575.403	181.197	0.000 ND
C3-dibenzothiophenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	1304.771	532.929	57.251
C4-dibenzothiophenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	1110.849	454.268	84.168
Fluoranthene	0.002	0.000 ND	0.000 ND	0.000 ND	8.185	1.668	0.486
Pyrene	0.003	0.014	0.000 ND	0.000 ND	22.547	8.528	1.711
C1-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	77.649	28.067	6.585
C2-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	170.796	68.043	15.560
C3-fluoranthenes/pyrenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	211.064	79.647	18.991
Benz(a)anthracene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	7.545	2.752	0.492
Chrysene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	20.650	10.183	1.941
C1-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	47.352	20.386	4.800
C2-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	55.433	26.754	6.692
C3-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	37.941	11.255	1.831
C4-chrysenes	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Benzo(b)fluoranthene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	2.304	1.268	0.310
Benzo(k)fluoranthene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Benzo(e)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	3.468	1.392	0.300
Benzo(a)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	3.631	0.000 ND	0.000 ND
Perylene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Indeno(1,2,3-c,d)pyrene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Dibenz(a,h)anthracene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Benzo(g,h,i)perylene	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND	0.000 ND
Diagnostic Ratios							
Total PAH's	0.06	1.22	0.44	0.45	6718.22	2483.76	273.87
D2/P2	NA	NA	NA	NA	1.24	1.35	NA
D3/P3	NA	NA	NA	NA	1.83	1.76	1.73
Surrogate Recoveries							
Naphthalene-d8	67	69	46	69	92	100	96
Phenanthrene-d10	82	91	69	91	97	105	104
Chrysene-d12	93	93	69	91	103	102	91

FLUME TESTS 1 THROUGH 5

MASS BALANCE RESULTS

FINAL MASS BALANCE TABLES

	Mass Bitumen Added (g)	Floating (g)	Sunk (g)	Bathtub Ring (g)	Suspended (g)	Dissolved (g)
TEST 1	26280	45	17211	5	9009	10
TEST 2	10366	9453	-27	861	74	5
TEST 3	10366	8000	2004	29	326	6
TEST 4	10366	7030	1192	1407	730	6
TEST 5	10366	3630	-157	1212	5668	12

	Spill Conditions	Floating (%)	Sunk (%)	Bathtub Ring (%)	Suspended (%)	Dissolved (%)
TEST 1	low energy, freshwater	0.2	65.5	0.0	34.3	0.0
TEST 2	high energy, brackish water	91.2	-0.3	8.3	0.7	0.0
TEST 3	low energy, brackish water	77.2	19.3	0.3	3.1	0.1
TEST 4	high energy saltwater	67.8	11.5	13.6	7.0	0.1
TEST 5	high energy, freshwater	35.0	-1.5	11.7	54.7	0.1

Battelle - Iatroscan Data Flume Tests 1 through 5

TEST 1

Percent Area Response

Time					Sample
(hr)	Saturates	Aromatics	Resins	Asphaltenes	ID
1.0	8.5	43.2	33.2	15.1	UU31
24.0	8.8	47.0	30.3	14.0	UU36
72.0	8.9	44.7	31.7	14.7	UU40

TEST 2

Percent Area Response

Time					Sample
(hr)	Saturates	Aromatics	Resins	Asphaltenes	ID
1.0	9.7	47.3	29.3	13.7	UU43
24.0	7.7	29.7	42.7	19.9	UU48
72.0	3.8	9.9	60.3	26.1	UU51

TEST 3

Percent Area Response

Time					Sample
(hr)	Saturates	Aromatics	Resins	Asphaltenes	ID
1.0	7.7	41.0	34.7	16.6	UU55
24.0	7.4	33.3	40.2	19.1	UU60
72.0	3.9	24.5	45.4	26.3	UU63

TEST 4

Percent Area Response

Time					Sample
(hr)	Saturates	Aromatics	Resins	Asphaltene	ID
1.0	8.4	41.1	33.0	17.5	UU67
24.0	9.2	32.4	38.4	20.0	UU72
72.0	8.2	38.1	36.3	17.4	UU75

TEST 5

Percent Area Response

Time					Sample
(hr)	Saturates	Aromatics	Resins	Asphaltene	ID
1.0	8.2	47.9	27.4	16.5	UU79
24.0	7.9	47.4	27.8	16.9	UU84
72.0	4.1	38.1	38.7	19.1	UU87

TPH and PAH Data - Battelle Flume Tests 1 through 5

TEST 1 TIME (hrs)	TPH (TOTAL) (mg/L) GRAV	TPH (DISSOLVED) (mg/L) INST	PAH (TOTAL) (ug/L)	PAH (DISSOLVED) (ug/L)
1	5063.29	2.30	20303.6	6.6
2	5328.95	NA	NA	NA
4	5064.94	NA	NA	NA
6	5197.37	NA	NA	NA
12	5031.85	NA	NA	NA
24	4200.00	3.05	16198.9	5.8
48	3184.00	NA	NA	NA
72Rep1(15cm)	2081.08	NA	NA	NA
72Rep2(15cm)	2062.50	NA	NA	NA
72Rep3(15cm)	1776.47	NA	NA	NA
72Rep1(35cm)	2187.50	2.62	NA	5.0
72Rep2(35cm)	2211.76	2.53	NA	NA
72Rep3(35cm)	2280.00	2.44	6285.3	4.1
72Rep1(55cm)	2617.45	NA	NA	NA
72Rep2(55cm)	2535.21	NA	NA	NA
72Rep3(55cm)	2518.52	NA	NA	NA

NA - not analyzed

TPH and PAH Data - Battelle Flume Tests 1 through 5

TEST 3

TIME (hrs)	TPH (TOTAL) (mg/L) GRAV	TPH(DISSOLVED) (mg/L) INST	PAH (TOTAL) (ug/L)	PAH (DISSOLVED) (ug/L)
1	1878.21	1.37	7090.1	1.3
2	1549.67	NA	NA	NA
4	741.75	NA	NA	NA
6	630.00	NA	NA	NA
12	361.14	NA	NA	NA
24	157.30	1.28	491.9	0.3
48	155.57	NA	NA	NA
72Rep1(15cm)	73.45	NA	NA	NA
72Rep2(15cm)	72.50	NA	NA	NA
72Rep3(15cm)	78.19	NA	NA	NA
72Rep1(35cm)	81.40	1.55	185.75	0.2
72Rep2(35cm)	79.07	1.68	NA	NA
72Rep3(35cm)	80.46	1.64	NA	NA
72Rep1(55cm)	77.53	NA	NA	NA
72Rep2(55cm)	107.47	NA	NA	NA
72Rep3(55cm)	83.72	NA	NA	NA

NA - not analyzed

TPH and PAH Data - Battelle Flume Tests 1 through 5

TEST 5 TIME (hrs)	TPH (TOTAL) (mg/L) GRAV	TPH(DISSOLVED) (mg/L) INST	PAH (TOTAL) (ug/L)	PAH (DISSOLVED) (ug/L)
1	2381.72	1.91	6718.2	1.2
2	2396.74	NA	NA	NA
4	2144.44	NA	NA	NA
6	2135.87	NA	NA	NA
12	2134.41	NA	NA	NA
24	2122.34	2.12	2483.8	0.4
48	1552.69	NA	NA	NA
72Rep1(15cm)	1289.47	NA	NA	NA
72Rep2(15cm)	1364.13	NA	NA	NA
72Rep3(15cm)	1472.22	NA	NA	NA
72Rep1(35cm)	1366.48	3.25	273.87	0.5
72Rep2(35cm)	1381.11	3.19	NA	NA
72Rep3(35cm)	1385.96	2.62	NA	NA
72Rep1(55cm)	1467.74	NA	NA	NA
72Rep2(55cm)	1478.02	NA	NA	NA
72Rep3(55cm)	1548.94	NA	NA	NA

NA - not analyzed