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# SITE Emerging Technology Report
# Innovative Methods for Bioslurry Treatment

by:
Kandi Brown
**IT Corporation**
San Bernardino, California 92408

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Project Officer:
Brunilda Davila
National Risk Management Research Laboratory

**National Risk Management Research Laboratory**
**Office of Research and Development**
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16. ABSTRACT

IT Corporation conducted a pilot-scale testing of biological and chemical oxidation of slurry-phase polycyclic aromatic hydrocarbons (PAHs). The 7-month demonstration illustrated potential effectiveness of combined biological and chemical oxidation for the treatment of PAH-contaminated soils. The 80 percent CPAH destruction goal was achieved with CPAH transformation ranging up to 84 percent. A system hydraulic residence time (HRT) of approximately 37 days increased system performance. Due to the increased transformation of PAH in reactor 1 (R1) and reactor 2 (R2) during optimal performance, transformation rates in reactor 3 (R3) were significantly decreased. This result may indicate that R3 is not required for effective treatment. It should be noted that greater than 80 percent CPAH removal was achieved during the last 2 weeks of operation following modifications to the treatment process made during the previous weeks. These results are reflective of the effectiveness of the treatment system following achievement of steady state operation. The modification to the treatment system included increasing the system HRT from 18.5 to 37 days. This change resulted in a HRT in R1 equal to the previous system HRT. As a result, PAH and CPAH removal increased in R1 and R2, with a decreasing performance in R3. Overall, operation of R1 and R2 only was adequate for effective treatment following in increase in HRT. Continued investigation under the process set points maintained during the final month of system operation is recommended.

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## *Notice*

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## *Foreword*

The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, EPA's research program is providing data and technical support for solving environmental problems today and building a science knowledge base necessary to manage our ecological resources wisely, understand how pollutants affect our health, and prevent or reduce environmental risks in the future.

The National Risk Management Research Laboratory is the Agency's center for investigation of technological and management approaches for reducing risks from threats to human health and the environment. The focus of the Laboratory's research program is on methods for the prevention and control of pollution to air, land, water and subsurface resources; protection of water quality in public water systems; remediation of contaminated sites and ground water; and prevention and control of indoor air pollution. The goal of this research effort is to catalyze development and implementation of innovative, cost-effective environmental technologies; develop scientific and engineering information needed by EPA to support regulatory and policy decisions; and provide technical support and information transfer to ensure effective implementation of environmental regulations and strategies.

This publication has been produced as part of the Laboratory's strategic long-term research plan. It is published and made available by EPA's Office of Research and Development to assist the user community and to link researchers with their clients.

E. Timothy Oppelt, Director
National Risk Management Research Laboratory

## *Abstract*

IT Corporation (IT), Knoxville, Tennessee, in collaboration with U.S. Environmental protection Agency (EPA), investigated the feasibility of combined biological and chemical oxidation of polycyclic aromatic hydrocarbons (PAH). Bioslurry treatment of PAH-contaminated soils was demonstrated under the Superfund Innovative Technology Evaluation - Emerging Technology Program (SITE ETP) as an extension of research previously funded by IT Corporation (IT) (Brown and Sanseverino 1993) and additional investigations supported by the U.S. EPA (Davila et al. 1994). All testing was initiated in September, 1994.

During the demonstration, IT operated two 60-liter (L) TEKNO Associates bioslurry reactors (Salt Lake City, Utah) and a 10-L reactor in series under semicontinuous, plug-flow mode for a 7-month period. The first 60-L reactor received fresh feed daily and supplements of salicylate and succinate to enhance PAH biodegradation.

Slurry from the first reactor was fed to the second 10-L reactor, where Fenton's reagent ($Fe^{++}+H_2O_2$)was added to accelerate chemical oxidation of 4 to 6-ring PAHs. The third reactor in series was used to biologically oxidize contaminants remaining following addition of Fenton's reagent. This reactor received no additions of salicylate and succinate and was aerated, nutrient amended, and pH adjusted only.

During operation, the reactor system demonstrated total PAH and carcinogenic PAH (CPAH) transformation up to 95 and 84 percent, respectively.

This report was submitted in fulfillment of assistance agreement CR821186-01-0 by IT Corporation under the partial sponsorship of the United States Environmental Protection Agency. This report covers a period from September 1994 to April 1995, and work was completed as of December 1995.

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# List of Acronyms

| | |
|---|---|
| BAC | Biotechnology Applications Center |
| B(a)P | Benzo(a)pyrene |
| cfu/mL | colony forming units per milliliter |
| CHP | Chemical Hygiene plan |
| CPAH | carcinogenic polycyclic aromatic hydrocarbons |
| DCM | dichloromethane |
| DE | diatomaceous earth |
| EPA | U.S. Environmental Protection Agency |
| ETP | Emerging Technologies Program |
| GC/MS | gas chromatography/mass spectrography |
| $H_2SO_4$ | sulfuric acid |
| HPLC | high performance liquid chromatography |
| HRT | hydraulic retention time |
| IT | IT Corporation |
| L | liter |
| $LD_{50}$ | lethal dose |
| M | molar |
| mg/L | milligrams per liter |
| mg/kg | milligrams per kilogram |
| MGP | manufactured gas plant |
| mL | milliliters |
| mM | millimolar |
| mm | millimeter |
| N | Normal |
| nm | nanometer |
| PAH | polycyclic aromatic hydrocarbons |
| PCP | pentachlorophenol |
| PFD | process flow diagram |
| QAPP | quality assurance project plan |
| R1 | Reactor 1 |
| R2 | Reactor 2 |
| R3 | Reactor 3 |

## *List of Acronyms (continued)*

| | |
|---|---|
| rpm | revolutions per minute |
| SITE | Superfund Innovative Technology Evaluation |
| SOP | standard operating procedure |
| TC | total carbon |
| TOC | total organic carbon |
| TRPH | total recoverable petroleum hydrocarbon |
| TS | total solids |
| TSDF | treatment, storage, and disposal facility |
| UV | ultraviolet |
| VS | volatile solids |

## *Acknowledgment*

IT and U.S. EPA would like to acknowledge the contribution of Gunter Brox, TEKNO Associates, during the execution of this work.

# *1.0 Introduction*

IT Corporation (IT) was contracted by the U.S. Environmental Protection Agency (EPA) under a cost sharing contract (CR821186-01-01) in October 1993 to conduct pilot-scale testing of the biological and chemical oxidation of slurry-phase polycyclic aromatic hydrocarbons (PAH). Bioslurry treatment of PAH-impacted soils was demonstrated under the Superfund Innovative Technologies Evaluation - Emerging Technologies Program (SITE ETP) as an extension of research previously funded by IT (Brown and Sanseverino 1993) and additional investigations supported by the EPA (Davila et al. 1994).

All testing was initiated in September, 1994. Testing was conducted by IT personnel at IT's Biotechnology Applications Center (BAC) located in Knoxville, Tennessee.

## *1.1 Site Description*

Among the types of contaminants present in Superfund soils, complex PAHs constitute the more challenging class to treat. Sites that contain PAH contamination include manufactured gas plants (MGP), wood-treating facilities, petrochemical facilities, and coke plants. Soils employed during this investigation were collected from a wood-treating facility located in Arkansas.

## *1.2 Soil Characterization*

All soil collection and screening activities were conducted by IT personnel, with supervision by the wood-treating site health and safety officer during the week of September 12, 1994. For a complete review of soil screening activities see Section 3.3.1.

## *1.3 Waste Stream Description*

PAH and carcinogenic PAH (CPAH)-impacted soils, primarily sand (30 percent) and clay (70 percent), were wet-sieved on site through a 30 mesh screen and submitted to IT's BAC, for testing. All geotechnical analyses are presented in Appendix A. Oversized material was disposed on site. Blended slurry PAH and CPAH concentrations ranged up to 6,120 and 434 milligrams per kilogram (mg/kg), respectively. Wet sieving the soils increased the uniformity of the slurry, thereby, reducing the potential for sampling variability.

### *1.4 Remedial Technology Description*

PAHs are characterized by high organic partition coefficients, low aqueous solubility, and low vapor pressures (Table l-l). These characteristics result in the highly sorptive nature of PAHs and their subsequent limited availability to microbial populations. IT's past experience with PAH-contaminated soils indicated that contaminant desorption from soil is the rate limiting factor in bioremediation. Manipulation of parameters such as pH, agitation, and temperature, as well as the addition of surfactants or solvents, can be used to enhance the rate of desorption and, thereby, increase in the rate of biodegradation.

The optimum method of manipulating these parameters is in bioslurry reactors. Bioslurry reactors can provide rapid biodegradation of contaminants due to enhanced mass transfer rates and increased contaminant to microorganism contact. These units are capable of treating high concentrations of organic contaminants in soils and sludges, with demonstrated biodegradation of selected contaminant concentrations ranging from 2,500 to 250,000 mg/kg. In general, the percent removal of PAH in these systems ranges from 70 to 95 percent, with 30 to 80 percent reduction of the CPAH fraction (EPA, 1990).

Bioslurry reactors can aerobically biodegrade aqueous slurries created through the mixing of soils or sludges with water. Maximum contaminant reduction is accomplished in bioslurry reactors primarily through proper feed preparation. Preparation of the influent waste stream should produce the general characteristics presented in Table l-2.

The most common mode of bioslurry treatment is batch; however, continuous-flow operation can be achieved. Aeration is provided through floating or submerged aerators or compressors and spargers. Mixing may be achieved through aeration alone or in conjunction with mechanical mixers. Nutrient addition and pH adjustment are accomplished through metered chemical addition to the reactor. Following aeration, the treated slurry is dewatered via standard dewatering equipment, such as clarifiers or filter presses.

The residual streams created during bioslurry treatment include treated solids, process water, and possible air emissions. The process water collected during the solids/liquid separation phase is usually recycled for influent waste stream slurrying or discharged under permit. Air emissions may be controlled through air pollution control devices.

Full-scale commercial bioslurry units require approximately 0.5 to 1 acre per million gallons of reactor volume (EPA, 1990). Reactor size is determined based on the hydraulic retention time (HRT) required for treatment. Retention times are established based on the biodegradability of the waste, level of treatment required, influent contaminant concentration, and physical/chemical nature of the waste.

Major issues of concern during bioslurry treatment system design include reducing system HRT and increasing the rate and extent of contaminant biodegradation. These factors were addressed by IT during the SITE investigation. To reduce the operating HRT, thereby decreasing the size of the system, IT operated bioslurry reactors in series (i.e., plug flow) under semicontinuous mode and evaluated two HRT set points.

During the demonstration, IT operated two 60-liter (L) Tekno Associates bioslurry reactors (Salt Lake City, Utah) and a 10-L fermentation unit in semicontinuous, plug-flow mode for a 7-month period. The first 60-L reactor received fresh feed daily and supplements of salicylate and succinate to enhance PAH biodegradation.

Slurry from the first reactor was fed to the second 10-L reactor, where Fenton's reagent ($Fe^{++}+H_2O_2$) was added to accelerate chemical oxidation of 4 to 6-ring PAHs. The third reactor in series was used to biologically oxidize contaminants remaining following addition of Fenton's reagent. This reactor received no additions of salicylate and succinate and was aerated, nutrient amended, and pH adjusted only.

## *2.0 Conclusions and Recommendations*

### *2.1 Conclusions*

The 7-month demonstration illustrated the potential effectiveness of combined biological and chemical oxidation for the treatment of PAH-impacted soils. Overall, the following conclusions were made:

- The 80 percent CPAH destruction goal was achieved with CPAH transformation ranging up to 84 percent.
- A system HRT of approximately 37 days increased system performance.
- Due to the increased transformation of PAH in reactor 1 (Rl) and reactor 2 (R2) during optimal performance, transformation rates in reactor 3 (R3) were significantly decreased. This result may indicate that R3 is not required for effective treatment.

It should be noted that greater than 80 percent CPAH removal was achieved during the last 2 weeks of operation, following modifications to the treatment process made during the previous 2 weeks. These results are reflective of the effectiveness of the treatment system following achievement of steady state operation.

The modification to the treatment system included increasing the system HRT from 18.5 to 37 days. This change resulted in a HRT in Rl equal to the previous system HRT. As a result, PAH and CPAH removal increased in Rl and R2, with a decreasing performance in R3. Overall, operation of Rl and R2 only was adequate for effective treatment following an increase in HRT.

### *2.2 Recommendations*

Continued investigation under the process set points maintained during the final month of system operation is recommended. As demonstrated by the increase in PAH and CPAH transformation during this period, reduced solids loading, increased clay content, and extended HRT set points proved beneficial to the treatment process.

Modifications to the pilot-scale reactor design, decreasing the incidence of foaming should be investigated. Process foaming, particularly when operating on the full-scale, will result in poor system performance, reactor overflow, and the inability to effectively aerate the system.

# *3.0 Treatability Study Approach*

## *3.1 Test Objectives*

The primary objective of this SITE investigation was to document accelerated PAH removal rates using combined chemical and biological treatment techniques. The project used specific organic (succinate and salicylate) and inorganic nutrient (ammonia and phosphate) supplements, in combination with Fenton's reagent to achieve increased removal in a plug-flow treatment system.

The specific objectives of the pilot-scale demonstration were:

- Determine efficacy of achieving greater than 80 percent reduction in CPAH due to a combination of biological and chemical oxidation
- Estimate HRT required for operation of each reactor
- Determine CPAH reduction in each reactor to compare combined biological/chemical oxidation to biological treatment alone
- Determine the need for R3
- Generate performance data upon which full-scale design can be established
- Provide operating data from which full-scale cost estimates can be generated.

## *3.2 Experimental Design and Procedures*

Previous work by IT demonstrated that sodium salicylate and sodium succinate enhanced the levels of naphthalenedegrading bacteria in a slurry reactor. Previously published research has demonstrated that in MGP site soils naphthalene, phenanthrene, anthracene, and to a limited extent benzo(a)pyrene [B(a)P] were mineralized (Sanseverino et al., 1993). Biodegradability was confirmed with $^{14}C$-radiolabeled PAH. Further; the naphthalene-degrading pathway of *Pseudomonas purida* NAH7 and NAH7-like organisms mineralize phenanthrene and anthracene through the same genetic and biochemical pathway as naphthalene. Therefore, the presence of salicylate will keep the naphthalene pathway induced, promoting degradation of these 3-ring PAHs even when naphthalene levels diminish (Ogunseitan et al., 1991).

The primary objective of Rl operation was to increase the biological removal of organic carbon. Salicylate was used to induce the naphthalene degradation operon on NAH plasmids. It was assumed that NAH plasmids were naturally occurring in microbial populations indigenous to subject soils. Succinate, a by-product of naphthalene metabolism, served as a general carbon source in Rl which removed easily degradable carbon and increased biological activity against more recalcitrant PAH (i.e., 4-ring compounds and higher).

Slurry from Rl was fed to R2 where Fenton's reagent was continuously introduced, resulting in chemical oxidation being the primary mechanism for PAH transformation in this reactor. The pH in R2 was adjusted to 2.0 following the addition of Rl slurry. Fenton's reagent (hydrogen peroxide in the presence of reduced iron salts) produces free radicals, which have been shown effective in extensively oxidizing multiring aromatic hydrocarbons in both soil and water systems (Gauger et al. 1990; Elizardo 1991). The objective of Fenton's reagent addition was not PAH mineralization, but the hydroxylation of PAH, because hydroxylation of high-molecular-weight PAHs generally is the rate-limiting step in biological oxidation.

R3 was used for biological oxidation of R2 slurry. R3 received no additions of salicylate and succinate. The reactor was aerated, nutrient amended, and pH adjusted following the introduction of R2 feed. The system process flow diagram (PFD) is presented in Figure 3-l.

### *3.3 Equipment and Materials*

All treatability testing was completed at the BAC laboratory located in Knoxville, Tennessee. This facility holds a special exemption from the State of Tennessee that permits execution of treatability studies. The BAC laboratory operates in accordance with an approved Chemical Hygiene Plan (CHP). All project activities at the BAC conformed to the standards set forth in the CHP.

#### *3.3.1 Soil Collection and Preparation*

Soils were excavated by the on-site contractor using a rubber-tire backhoe and a Kamatso DC200 trackhoe. Lightly impacted soils were collected from the A-cell area during site preparation of a land treatment cell. Highly contaminated soils were collected from Catch All Pond Sediments in the area where the SB-5 sample had been collected. Appendix B contains all data obtained from the wood-treating facility prior to soil collection.

The objectives of field screening were:

- Prepare adequate volume of -30 mesh soil slurry to complete the pilot-scale investigation
- Dispose all oversized material on-site
- Prepare one 55-gallon drum of lightly impacted slurry
- Prepare four 55-gallon drums of a 50:50 blend of lightly and highly impacted material
- Collect additional volume of unscreened soil to support on-going project activities.

All objectives were met during field activities. Soils were screened in accordance with the approved Test Plan (IT, 1994). Site soils were excavated and staged on visqueen. Soils were transported to the wet-sieving area using a wheelbarrow. Three galvanized aluminum watering troughs with stainless-steel mesh sieves secured to the rim with lumber were used during soil screening. Each sieve was constructed using a -30 mesh U.S.A. Standard Testing Sieve.

During screening, the troughs were partially filled with tap water. Little Giant 2E Series submersible pumps (aluminum housing, epoxy coating, nylon pump head and impeller, and polypropylene screen) were placed on concrete blocks inside each trough. These pumps were used to recirculate the wash water and, thereby, increase the slurry density of the mixture. Evaporation of excess water could not be achieved during screening due to the limited equipment.

Soils were characterized as sand (30 percent) and clay (70 percent). Particle size distribution data are presented in Appendix A.

During screening, five drums of sieved material were generated - one drum of clean soil and four drums of blended material. Blended slurry was produced through the separate screening of lightly and highly impacted soils. The slurry produced during soil screening was then blended in a 1: 1 ratio. In addition to soil slurry, two drums of impacted soil were collected. All seven drums were shipped to the IT Bear Creek Facility located in Knoxville, Tennessee.

Following soil collection and screening, three drums of slurry (Drums 1, 2, and 3) were individually mixed and separated into smaller, more manageable containers. Cross-contamination of drum contents was avoided during mixing. A MQ Multiquip Whiteman cement mixer with a Honda GX240 8.0 motor operated at approximately 28 revolutions per

minute (rpm) was used to mix all slurry. The system was decontaminated using Mi-T-M pressure washer with a Honda GX160 5.5 motor.

### *3.3.2 Reactor Description and Operation*

Rl and R3 were 60-L, stainless-steel, Tekno™ (formerly Eimco) bioslurry reactors (Figure 3-2; Salt Lake City, Utah). R2 was a 30-L glass vessel fitted with an overhead impeller system. The system PFD is shown in Figure 3-l. Initial operational setpoints for R1, R2, and R3 are provided in Table 3-l. All portions of the reactor system that contacted the slurry mixture were stainless-steel, glass, or Viton tubing.

Agitation and aeration in Rl and R3 were accomplished using a combination of the Tekno™ reactor impeller, air lift, and diffuser systems. System pH was maintained through manual additions of 10 Normal (N) sodium hydroxide to the reaction vessels as necessary.

Fresh feed was manually introduced to Rl at an average daily flow rate of 6 L/day. For the first 4 months of operation, fresh feed was introduced to the system in 2-L batches three times per day to equalize the load of carbon into the system. At this daily flow rate and operating volume of 57 L, the Rl HRT was maintained at approximately 10 days.

Slurry from Rl was manually fed to R2. The working volume of this 10-L reactor was approximately 6 L. With an influent flow rate of 6 L/day, the HRT in R2 was 1 day. In addition to the influent slurry, Fenton's reagent was added to this reactor at a rate of 2 L/day. Overall, 8 L/day of slurry was removed from R2 and introduced to R3.

Slurry from R2 was manually fed to R3. The working volume of this reactor was equal to Rl. The resulting HRT at 8 L/day was 7.5 days. Volume loss due to evaporation was checked daily and adjusted as needed. Effluent from R3 was collected and stored for disposal at a licensed treatment storage and disposal facility (TSDF).

After 4 months of operation, the HRT was doubled to 20 days in Rl, 2 days in R2, and 15 days in R3 to reduce the loading of organic carbon on the system. The total system HRT was increased from 18.5 to 37 days. In addition, influent feed (3 L) was introduced once per day.

For the first 4 months of operation, Fenton's reagent was prepared by mixing a 1:1 ratio of 35 percent hydrogen peroxide and 1.5 molar (M) ferrous sulfate heptahydrate solution. For the

remainder of the study, Fenton's reagent was prepared using 8.8 milliMolar (mM) ferrous sulfate heptahydrate. The change in the concentration of Fenton's reagent addition was initiated to increase the efficiency of the chemical reaction. A reduced concentration prevented the occurrence of competing side reactions.

Initially, ferrous sulfate and hydrogen peroxide were added by dripping each solution into R2 at a rate of 1.0 L each per day. This system was modified such that each solution was introduced simultaneously below the slurry surface. This was thought to provide for better mixing, less splashing of each reagent, and reduced foam production.

Mixing efficiency of the reactor solids was verified periodically during the course of the investigation. Verification was accomplished through analysis of total solids (TS) concentrations in samples extracted from sample ports located on the side of the bioslurry reactors. If nonuniform mixing was evident, agitation speed, rake speed, airlift system, or solids content was adjusted. Due to the combined application of a 1:1 hydrogen peroxide:iron sulfate solution in R2, only the impeller system was used to maintain complete mixing of the soil slurry.

### *3.4 Sampling and Analysis*

The reactors were charged on September 23, 1994. All reactors were operated in batch through October 10, 1994. Initial analytical data was colkcted prior to reactor batch operation, during batch operation, at the initiation of semi-continuous operation, and routinely throughout the remainder of the study. Sampling dates are presented in Section 4.0.

No steps were taken to reduce biological activity in soil samples prior to testing due to a shortage of refrigerated storage area. However, each influent batch was analyzed prior to introduction into the reactor system to accurately determine initial concentrations.

Slurry samples were collected from sample port S2 on. Rl and R3 (Figure 3-l). Grab samples were collected from R2. The sampling and analytical schedule for the slurry phase, as well as volumes required for each analysis is shown in Table 3-2. In addition to the slurry phase analyses, the headspace of Rl was sampled monthly for PAHs The influent slurry was sampled each time a new batch was introduced into the reactors. Influent slurry was analyzed for PAH, total organic carbon (TOC), TS/volatile solids (VS), and density.

### *3.4.1 Physical Analyses*

A summary of all analytical methods used during this investigation is presented in Table ***3-3.*** Physical measurements included dissolved oxygen, total and volatile solids, pH, ammonia, and phosphate.

### *3.4.2 PAH Analyses*

PAHs were measured in the slurry phase of each reactor, as well as the headspace of Rl. PAH concentrations in the slurry were determined using modified EPA Method 8310. For PAH analysis of solids, air-dried slurry samples (10 grams) were mixed with anhydrous sodium sulfate, placed in an extraction thimble and extracted using dichloromethane (DCM) in a Soxhlet extractor for 16 hours. (The dry weight of the solid phase was analyzed for weight loss in a 105°C drying oven.) The DCM extract was concentrated to 1 milliliters (mL) using a Snyder column and solvent exchanged to 100 mL acetonitrile. Following extraction the sample was analyzed using a Dionex high performance liquid chromatograph (HPLC) equipped with an ultraviolet (UV) detector at 254 nanometer (nm). The elution profile was acetonitrile:water (35:65) for 1 minute followed by a gradient to 100 percent acetonitrile over 15 minutes and held for 10 minutes.

Aqueous phase PAH analysis was conducted for the first 3 months of operation. Aqueous phase PAH were quantified by direct injection of the aqueous phase into a Perk&Elmer HPLC equipped Vydac $C_{18}$ column (Model 201TP54; Hesperia, California) with a variable wavelength programmable fluorescence detector (LC-240). The elution profile was acetonitrile: water (50:50) for 2 minutes followed by a gradient to 100 percent acetonitrile over 12 minutes and held for 5 minutes.

Headspace semivolatile constituents were measured through air sampling at port Z-2 on Rl (Figure 3-l). The air sampling train consisted of a Teflon™ probe, a 47-millimeters (mm) Teflon™ membrane filter, and an XAD-2 sorbent sampling tube. Headspace gases were pulled through the XAD-2 tube for 24 hours. The XAD tube was extracted with 5 mL of acetonitrile and analyzed by HPLC.

Reactor samples were shipped to the EPA (Cincinnati, Ohio) for extraction and analysis by gas chromatography/mass spectrography (GC/MS) for confirmation of the HPLC analysis.

### 3.4.3 *Microbial Enumerations*

Total heterotrophic bacteria were quantified using BAC standard operating procedure (SOP) 009. Naphthalene degrading bacteria were quantified by DNA:DNA colony hybridization using the NAH gene as a probe (Sayler et al., 1985; Sanseverino et al., 1993). The NAH encodes the naphthalene dioxygenase enzyme which is the first step in naphthalene metabolism. Enumeration of naphthalene-degrading bacteria occurred on weekly samples for the first three months of operation at which time it was discontinued.

### 3.4.4 *$^{14}C$ Mineralization Assays*

$^{14}C$-PAH mineralization assays were performed to estimate *in situ* microbial degradative capacity for specific compounds. Two mL of slurry were placed in a 40-mL vial (Pierce, Rockford, Ii.). Labeled l-$^{14}C$-naphthalene, 9-$^{14}C$-phenanthrene, UL-$^{14}C$-anthracene, or 7, 10-$^{14}C$-benzo(a)pyrene (Sigma, St. Louis, Mo; specific activity 8.0, 10.4, 10.4, and 60.0 mCi/mmol, respectively) were individually added to triplicate vials. Slurries were incubated at 26°C with shaking (100 rpm). Naphthalene and phenanthrene mineralization ($^{14}CO_2$ production) was analyzed at regular intervals over a 7-day period. Anthracene mineralization was analyzed at intervals up to 10 days. Benzo(a)pyrene mineralization was analyzed at intervals up to 2 weeks. Biological control samples were inhibited by acidification with 0.5 mL of 2 N sulfuric acid ($H_2SO_4$) and metabolism assays were also terminated by $H_2SO_4$ addition. $^{14}CO_2$ was trapped in 0.5 mL of 0.5 N NaOH. The NaOH was added to 1 mL of water and 10 mL of Beckman ReadySafe$^{Tm}$ scintillation fluid and counted in a Beckman liquid scintillation counter (Model LS380 1).

### 3.4.5 *Toxicity Screening*

The aqueous phases of the influent feed, Rl, and R3 were tested for toxicity using *Terrahymena* as a test organism. Dilutions of the aqueous phase (1:200, 1: 100, 1:40) were added to *Tetrahymena* cells (approximately 1,000 cells/ml) and incubated for 16 hours. Acute toxicity was assessed by observing each tube for cell lysis and/or lack of ciliary movement as determined with the aid of a dissecting microscope. A lethal dose ($LD_{50}$) was determined for each sample.

### 3.4.6 *Chemicals*

Chemicals, analytical grade, and sources are listed in Table 3-4.

### *3.5 Data Management*

All data was generated and stored as specified in the approved Test Plan

### *3.6 Deviations from the Test Plan and QAPP*

Per the project contract, work progress and quality were monitored through audits of the laboratory and project files. Audits indicated minor nonconformances and technical changes from the Test Plan, however, nothing was identified which adversely impacted the project quality of work. All project technical changes and nonconformances are listed in Tables 3-5 and 3-6.

# 4.0 Results and Discussion

## 4.1 Data Analysis and Interpretation

The results of the 7-month demonstration are presented in the following text.

### 4.1.1 Reactor Operation

Daily reactor operation was described in Section 3.3.2. Operational difficulties were encountered, during the demonstration. First, tar balls were formed following reactor charging and aeration. These tar balls were physically removed from the system and their mass determined. Following removal, PAH concentrations and mass within all reactors were recalculated to assure that physical removal was not accounted for as biological/chemical removal.

Second, foaming of the reactor contents was a routine occurrence. R1 and R3 were fitted with mechanical foam breakers but these were inadequate to contain the foam. Antifoam 289 (Sigma, St. Louis, Missouri) was used as required to contain the foam inside the reactors.

The third problem was clogging of the airlifts. This problem usually appeared due to disruption of the air flow to the reactors and unusually high settling due to decreased clay content of the soil. Periodic purging and/or dismantling of the air lifts were necessary to restore air flow.

Table 4-l summarizes significant upsets and changes which effected reactor operations.

### 4.1.2 Physical Analyses

Results of physical analyses are presented below

#### 4.1.2.1 pH

The pH of R1 and R3 were maintained at 7.0 (Figure 4-l) through the addition of NaOH on an as needed basis. R1 required minor periodic adjustments in pH through the addition of 1 N NaOH. The material transferred from R2 into R3 required pH adjustment with every transfer using 10 N NaOH. The Fenton's reagent dictated the pH in R2. When 1.5 M

ferrous sulfate was used, the pH ranged from 2.0 to 2.5. When 8.8 mM ferrous sulfate was used, the pH reached a new steady state at 3.5 to 4.0.

#### *4.1.2.2 Dissolved Oxygen*

For the first 3 months of operation, the dissolved oxygen concentration fluctuated between 0 and 10 milligrams per liter (mg/L) (Figure 4-2). Due to foaming within the reactors, it was not always possible to obtain a "pure" liquid sample for dissolved oxygen analysis. The last 3 months of operation showed a steady dissolved oxygen in Rl ranging from 2.0 to 7.0 mg/L. The dissolved oxygen in R3 ranged from 4 .0 to 9.0 mg/L over the same period.

#### *4.1.2.3 Reactor Solids*

The reactors were initially charged at 40 percent solids using the highly contaminated site soil. This loading was necessary for the airlifts to operate properly and keep the solids in suspension due to high sand content. Due to increased foaming problems, the impacted TS content was reduced to 20 percent and Celatom™ diatomaceous earth (DE) was used to increase the TS concentration to 35 percent. This switch occurred on December 13, 1995. Use of DE was discontinued on December 29, 1995 and locally obtained clean clay was used to reduce the influent organic content. The solids loading for the final 3 months of operation was 30 percent contaminated soil plus 10 percent clay for a 40 percent total solids loading.

Figure 4-3 shows the TS for each reactor. For the final 3 months of operation, the TS in R1 was steady at 30 to 35 percent. R2 showed a decrease in TS relative to R1. This decrease was due to dilution of the reactor contents with Fenton's reagent. R2 and R3 showed a steady decline in TS over time. On January 4, 1995, the TS was 29 percent. On April 19, 1995, the TS in R3 reached a low of 9 percent.

Volatile solids for each reactor are shown in Figure 4-4. Rl VS remained steady for the last 2 months of operation ranging from 8 to 9 percent. R2 displayed wide fluctuations in VS ranging from 5 to 14 percent. R3 showed an increase in VS with values ranging from 10 to 13 percent over the last 2 months of operation. The increase in VS may represent increased biological growth possibly due to the metabolism of recalcitrant hydrocarbons oxidized in R2.

The VS/TS ratio (Figure 4-5) remained steady for Rl ranging from 0.2 to 0.3 while R3 showed a steadily increasing VS/TS ratio during the final 2 months of operation. R3 values

ranged from 0.8 during early March to 1.3 on April 19, 1995. R2 VS/TS values fluctuated, but centered around 0.4 to 0.5.

Solids distribution for Rl and R3 is shown in Figures 4-6 and 4-7, respectively. A uniform solids suspension was dependent on the airlift operating properly, maintaining an adequate solids loading within the reactor, and maintaining the proper volume. Stratification was a minor problem in Rl (Figure 4-6) but was a more persistent problem in R3 (Figure 4-7). The DE promoted stratification by binding to the clay present in the contaminated soil. This did not pose a significant problem in Rl due to the high organics present, however, it may have been a problem in R3 as seen in the solids distribution during the months of December, 1994 and January, 1995.

#### *4.1.2.4 Nutrients*

Ammonia was added initially once per week for the first 2 months followed by 3 times per week for the remainder of the study. Ammonia addition to R3 was discontinued after 3 months due to carry over from Rl and R2. Ammonia values ranged from 0 to 240 mg/L over the course of the study (Figure 4-8).

The large increase in ammonia concentrations resulted from increased addition rather than reduced utilization. During the mid project review meeting held in December it was decided that a possible nitrogen deficiency may have resulted in system foaming. As a result, the nitrogen addition rate to the system was increased.

Phosphate was initially added for the first month of operation at which time it was discontinued. The phosphate concentration in the contaminated soil was significant and ranged from 100 to 200 mg/L in each reactor during the course of the investigation (Figure 4-9).

#### ***4.1.2.5 Microbial Enumerations***

The total heterotrophic bacterial populations in Rl ranged from 1.4 x 10' colony forming units per mL of slurry (cfu/mL) to 4.0 x $10^8$ cfu/mL slurry over the course of the study (Figure 4-10). Bacterial populations in R3 were similar to Rl except during the last 2 months of the study. Total populations reached 2.2 x $10^9$ cfu/mL slurry on March 2, 1995 and ranged from 3.0 to 5.0 x $10^8$ cfu/mL slurry for the remainder of the study. This increase coincided with an increase in the HRT set points.

The naphthalene-degrading bacterial populations were 3.6 x $10^5$ cfu/mL slurry at the time the reactors were charged. This population dropped below the method detection limit (2.0 x $10^5$ cfu/mL) after onset of continuous reactor operation. Naphthalene-degrading bacteria were enumerated by colony hybridization in the April 13, 1995 samples. The influent feed material, Rl, and R3 contained 1.5 x$10^4$, 1.1 x $10^6$, and 1.1 x $10^8$ NAH positive cfu/mL slurry. The large increase in NAH positive cells in R3 was surprising since there was no detectable naphthalene in R3.

#### *4.1.2.6 Total Recovemble Petroleum Hydrocarbons*

Total recoverable petroleum hydrocarbons (TRPH) were measured in March 16, 1995 samples. The data is summarized in Table 4-2. R2 had no apparent effect on TRPH while R3 showed a 86 percent reduction. Even though R2 had the same TRPH concentration as Rl, this does not imply that the TRPH is in the same form as in Rl. ERA Method 418.1 does not discriminate differences in hydrocarbon chain length or possible side chain modifications (such as hydroxylations). Therefore, the Fenton's reagent could have broken down longer chain hydrocarbons into shorter chains which were more susceptible to bacterial degradation in R3.

#### *4.1.2.7 Total Organic Carbon Analysis*

Solid and aqueous phase TOC concentrations are shown in Figures 4-l1 and 4-12, respectively. The average influent solid phase total carbon (TC) concentration was 19,000 mg/kg. On March 16, 1995, influent solid phase TC was 17,500 mg/kg. After treatment, there was a 55 percent reduction in TC. R2 showed a 48 percent reduction relative to Rl. Although this reduction was not consistent with the TRPH removals, there may have been shorter chained organic compounds not measured in the TRPH analyses which were mineralized in R2.

For the 4-week period from March 16 through April 19, 1995, the average solid phase TC for Rl, R2, and R3 was 13,700; 7,900; and 6,400 mg/kg, respectively. In comparison, for the first 6 months of the study, the average solid phase TC for Rl, R2, and R3 was 14,500; 11,900; and 10,000 mg/kg, respectively.

The average aqueous phase TOC for the 4-week period from March 16 through April 19, 1995 in Rl, R2, and R3 was 570; 1,680; and 740 mg/L, respectively. In comparison, for the first 6 months of the study, the average aqueous phase TOC in Rl, R2, and R3, was 690; 1,680; and

340 mg/L, respectively. By observation of Figure 4-12, the production of aqueous phase TOC was erratic in R2 while Rl was consistent over the 7-month period.

#### *4.1.2.8 PAH and Mass Balance*

Physical removal of PAHs during treatment were corrected during the determination of mass removal. All tar ball removal was corrected through the recalculation of initial PAH concentrations and mass in all reactors following physical removal. Significant wall losses were not recognized following the dismantling of the reactors due to operation at all the reactor's full working volume. Dilution with the addition of Fenton's reagent was corrected by using real-time TS concentration data from each reactor to determine PAH mass.

Additionally the difference in sampling location for Rl and R3 as compared to R2 did not create a sampling bias in PAH results. All reactors were tested for adequate mixing and PAH mass determinations were corrected for the TS concentration of the sample. Reduced performance in R3 during the final stages of the testing program were due to the increased performance of Rl and R2 and reduced influent carbon concentrations in the reactor.

It should also be noted that PAH percent removal perturbations were due to operational conditions rather than a decrease in PAH recovery efficiencies. See Section 4.2 for a detailed discussion.

Slurry samples from each reactor were dried and analyzed by modified EPA method 8310. This method accounted for any recoverable PAH in the soil and aqueous phases. The air phase was monitored once per month and no substantial volatilization of PAH was observed in Rl.

The bioslurry reactor system demonstrated 95 and 84 percent removal of PAH and CPAH, respectively, as of April 19, 1995. Figures 4-13 through 4-16 illustrate PAH and CPAH reductions in Rl, R2, R3, and overall. Overall, the biologically active reactors (Rl and R3) illustrated a decreasing effectiveness in PAH transformation as a function of compound molecular weight. This is indicated in Figures 4-17 and 4-18 which present the concentrations of fluorene and B(a)P throughout the system.

Prior to operational changes initiated in March (following 5 months of treatment), Rl demonstrated 62 percent transformation of PAH, with approximately 28 percent transformation

of CPAH. R2 demonstrated comparable destruction of PAH and CPAH (approximately 30 percent), as expected during chemical oxidation. R3 CPAH and PAH transformations averaged approximately 14 and 33 percent, respectively. The total system PAH and CPAH transformations averaged 85 and 65 percent, respectively. All PAH and CPAH removal efficiency data are presented in Tables 4-3 and 4-4, respectively. No significant volatilization of PAH was evident in Rl.

Following operational changes initiated in March, overall PAH and CPAH transformation rates increased up to 95 and 84 percent. Rl demonstrated 87 percent transformation of PAH, with 65 percent transformation of CPAH. R2 demonstrated comparable destruction of PAH and CPAH (greater than 45 percent), as expected during chemical oxidation. R3 CPAH and PAH transformations were decreased averaging -31.6 and -26 (0) percent, respectively. The total system PAH and CPAH transformations increased to 91 and 75 percent, respectively. All PAH and CPAH mass removal efficiency data are presented in Tables 4-5 and 4-6, respectively.

During optimal operation, the influent PAH concentration was decreased from 6,210 mg/kg to 325 mg/kg. Influent CPAH concentrations were decreased from 422 mg/kg to 65 mg/kg. CPAH and PAH concentrations throughout the system are presented in Figures 4-19 and 4-20, respectively. All PAH data is presented in Appendix C.

Mass balance data is presented in Appendix C. All mass removals were calculated per the specifications of the approved Test Plan Table 4-3. This table has been included in Appendix C for reference. PAH and CPAH mass balance data has been summarized in Tables 4-5 and 4-6. Over the 7-month operational period, Rl demonstrated the highest transformation of PAH, averaging 10.6 grams/day (g/day). Following March 2, 1995, the PAH mass removal in Rl was increased to 7.7 g/day.

R2 and R3 demonstrated 3.2 and 3.2 g/day PAH removal, respectively, during the entire operational period (Table 4-5). R2 and R3 PAH removal efficiencies also decreased following March 2, 1995. R2 and R3 demonstrated 1.17 and 0.48 g/day PAH removal, respectively, from March 2 to April 19, 1995.

CPAH mass removal is summarized in Table 4-6. The same trends in PAH removal were also evident when analyzing the CPAH removal data. CPAH mass removal in Rl, R2, and R3

during the total operational period averaged 0.93,0.44, and 0.24 g/day, respectively. Rl performance following March 2, 1995 indicated an average CPAH removal of 0.63 R2 and R3 CPAH removal was decreased following March 2, 1995 to an average of 0.22 and 0.08 g/day, respectively.

Since February 2, 1995, R3 showed little to no transformation of PAH. In fact, there was an increase in PAH observed in the slurry. This observation was preceded by the change in addition of Fenton's reagent to R2. It is also known (at least for March 16, 1995 samples) that 62 percent of the total TRPH was removed by R3. It is hypothesized that if R.2 was breaking down the larger chain aliphatic hydrocarbons to smaller chain aliphatic hydrocarbons and these hydrocarbons were subsequently metabolized in R3, then PAH could be released from these aliphatic hydrocarbons. The tar phase, not the soil, is considered to be the dominant phase in manufactured gas plant sites and creosote-contaminated sites (Lane and Loehr, 1993). Therefore, all PAH will be found sorbed to the tar. If the tar phase breaks down, the sorbed PAH will be released. The build-up in R3 may reflect PAH being released from the tar phase and accumulating in the reactor. Whether PAHs were being metabolized in R3 is not clear although $^{14}C$-mineralization data discussed in Section 4.1.2.9 suggests that at least the 2- and 3-ring PAHs were metabolized.

### *4.1.2.9 $^{14}C$-Mineralization Assays*

First-order mineralization rates were determined by calculating the available (soluble) PAH for degradation and calculating specific activities for each $^{14}C$-PAH in each soil. To estimate the distribution of each PAH in the aqueous phase, the tar-water partition coefficient ($K_{tw}$) was estimated (Lane and Loehr 1992). The tar phase, rather than the particulate phase, is the dominant force in determining partition in these soils due to the high organic carbon content. Lane and Loehr (1992), using MGP soils in their experimental design, determined that a relationship existed between the octanol-water partition coefficient ($K_{ow}$) and $K_{tw}$.

$$\log K_{tw} = 1.13 \log K_{ow} + 0.33 \qquad \text{(Equation 1)}$$

The log $K_{ow}$ values for naphthalene, phenanthrene and anthracene were 3.37, 4.46, and 4.45, respectively (Sims and Overcash 1983). Specific activities of PAH were determined by dividing the $\mu$Ci of specific radiolabeled PAH added to the slurry by the calculated number of $\mu$moles of specific PAH present in the aqueous phase. Mineralization rates were determined

by plotting the number of **μmoles** of $^{14}CO_2$ produced vs time. The slope of the initial linear portion ($r^2 \geq 0.95$) of each curve was used as an estimate of the mineralization rate.

#### *4.1.2.10 Toxicity Screening*

To estimate the reduction in toxicity of the aqueous phase, an assay using the protozoan *Tetrahymena* was performed. Table 4-7 summarizes the results with an estimate of the lethal dose-50 ($LD_{50}$) for the aqueous phases of the influent feed, Rl, and R3. Rl and R3 had $LD_{50}$ at 1: 100 dilution of the aqueous phase in comparison to an $LD_{50}$ at the 1:40 dilution for the aqueous phase of the influent feed. No definitive conclusions should be drawn from this assay. While there was a substantial reduction in PAH and TRPH, pentachlorophenol (PCP) and arsenic were also present in this soil. Although PCP was not measured directly, past experience has shown that there is little to no reduction in this type of treatment system. After passage through R2, Fenton's reagent should be in its most oxidized form ($As^{+5}$).

### *4.2 Quality Assurance/Quality Control*

GC/MS analyses confirmed the correct identification of all PAH peaks measured using the HPLC. In addition, an approximate 20 percent variance comparing the GC/MS and HPLC analyses was noted. All other analytical measurements were made within the specifications defined in the approved quality assurance project plan (QAPP) unless specified in Table 3-6.

With regard to accuracy criteria for matrix spikes using modified Method 8310, the stipulated acceptance criteria provided in Table 6-l of the QAPP was a minimum recovery of 80 percent for each of the 16 compounds. The corrective action for those matrix spikes which did not meet criteria was reanalysis of the sample extract. Upon analysis and reanalysis, where necessary, 95 percent of the matrix spike recoveries met this criteria, and nonconformances were generated for any outlying data points.

### *4.3 Costs/Schedule for Performing the Treatability Study*

The overall budget for project execution was $209,751, All project activities were conducted between September 1994 and October 1995.

### *4.4 Key Contacts*

The EPA Project Manager, Brunilida Davila, can be contacted at (513-569-7849). IT contacts including the project manager (Kandi Brown) and principal investigator (John Sanseverino) who can be reached at 909-799-6869 and 615-690-3211 respectively.

## *5.0 References*


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Table l-l

Physical/Chemical Properties of CPAH Constituents

IT Project No. 408250

| Carcinogenic Polynuclear Aromatic Hydrocarbons | Kow (log) | Aqueous Solubility (µg/l) | V.P at 20°C (torr) |
|---|---|---|---|
| benz(a)anthracene | 5.61 | 14 | $5.0x10^{-9}$ |
| benzo(a)pyrene | 6.04 | 3.8 | $5.0x10^{-7}$ |
| benzo(b)fluoranthene | 6.57 | 1.2 | $5.0x10^{-7}$ |
| benzo(k)fluoranthene | 6.84 | 0.55 | $5.0x10^{-7}$ |
| chrysene | 5.61 | 2 | $6.3x10^{-7}$ |
| dibenz(a,h)anthracene | 5.97 | .50 | $1.0x10^{-10}$ |
| benzo(g,h,i)perylene | 7.23 | 0.26 | $1.0x10^{-10}$ |
| indeno(1,2,3-c,d)pyrene | 7.66 | 62 | $1.0x10^{-10}$ |

Sims, R. C. and M. R. Overcash "Fate of Polynuclear Aromatic Compounds (PNAs) in Soil - Plant Systems," *Residue Reviews, 1983.*

µg/l - micrograms per liter
V.P. - vapor pressure

Table l-2
General Influent Feed Characteristics
for Bioslurry Treatment

IT Project No. 408250

| Parameter | Target |
|---|---|
| Organics | 0.025 - 25 percent by weight |
| Solids | 10 - 40 percent by weight |
| Water | 60 - 90 percent by weight |
| Solids Particle Size | Less than 1/4 inch |
| Temperature | 15 - 35$^{o}$C |
| pH | 4.5 - 8.8 |

EPA, 1990, "Slurry Biodegradation,' EPA/540/290/016

**Table 3-1**
**Initial Operational Setpoints**

**IT Project No. 408250**

| Parameter | R1 | R2 | R3 |
|---|---|---|---|
| Feed Flow | 6 L/day | 6 L/day | 8 L/day |
| Hydraulic Retention Time | 10 days | 1 day | 7.5 days |
| Temperature | 25°C ± 5°C | 24°C ± 5°C | 25°C ± 5°C |
| Dissolved Oxygen | 3 mg/L | --- | 3 mg/L |
| pH | 7.0 ± 0.5 | <5.0 | 7.0 ± 0.5 |
| Agitation | 500 rpm | 250 rpm | 500 rpm |
| Working Volume | 57 L | 6 L | 57 L |
| Ammoniacal Nitrogen | 50 mg/L | --- | --- |
| *o*-phosphate | 10 mg/L | --- | 10 mg/L |
| Sodium Salicylate | 100 mg/L | --- | --- |
| Sodium Succinate | 10 mg/L | --- | --- |
| Fenton's Reagent Addition | --- | 2 L/day | --- |

**Table 3-2**
**Sampling and Analytical Schedule for R1, R2, and R3 Slurry Phase**

**IT Project No. 408250**

| | Analysis | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | PAH | TOC | N & P | DO | pH | TS/VS | Density | Microbial Enumerations | Particle Size |
| | First Four Months of Operation | | | | | | | | |
| Volume (mL) | 100 | 100 | 100 | 1 | - | 20 | - | 10 | 500 |
| Frequency | 1/wk | 1/wk | 1/wk | 2/wk | daily | 2/wk | daily | 1/wk | 1/month (R1 only) |
| | Last Two Months of Operation | | | | | | | | |
| Volume (mL) | 100 | 100 | 100 | 1 | -- | 20 | -- | 10 | 500 |
| Frequency | Biweekly | biweekly | 1/wk | 2/wk | daily | 2/wk | daily | biweekly | 1/month (R1 only) |

Table 3-3
Summary of Analytical Methods

IT Project No. 408250

| Parameter | Sample Type | Method Number | Method Title | Method Type | Reference |
|---|---|---|---|---|---|
| PAH | soil/water | modified EPA Standard Method 8310 | Polynuclear Aromatic Hydrocarbons | HPLC | SW-846 |
| PAH | soil | EPA Method 8270 | Polynuclear Aromatic Hydrocarbons | GC/MS | SW-846 |
| TOC | waler | BAC008 | Carbon Analysis Using the Dohrmann Total Carbon Analyzer | TOC Analyzer | BAC Proprietary |
| TC | soil | BAC031 | Total Carbon Analysis | Persulfate Oxidation | BAC Proprietary |
| TS | slurry | Standard Methods 2540G or 2540B | Total, Fixed, and Volatile Solids in Solid and Semisolid Samples or Total Solidr Dried at 103-105$^{o}$C, respectively | Drying oven | Standard Method |
| VS | slurry | Standard Methods 2540G or 2540B | Total, Fixed, and Volatile Solidr in Solid and Semisolid Samples or Total Solids Dried at 103-105$^{o}$C, respectively | Drying oven | Standard Method |
| $NH_3$ | slurry | BAC022 | Electronic Ammonia Analysis | Ion Probe | BAC Proprietary |
| o-$PO_4$ | slurry | BAC015 | Phosphate Analysis | Colorimetric | BAC Proprietary |
| Total Heterotrophs | slurry | BAC009 | Microbial Enumeration Analysis | Spread Plate | BAC Proprietary |
| Naphthalene Degraders | slurry | NA | Application of DNA-DNA Colony Hybridization to the Detection of Catabolic Genotypes in Environmental Samples | Colony Hybridization | Saylcr, et al., 1985 |
| pH | slurry | BAC014 | pH Analysis | Membrane probe | BAC Proprietary |
| DO | slurry | BAC021 | Oxygen Analysis | Galvanic cell | BAC Proprietary |
| Particle Size | slurry | ASTM Method D422 | Particle Size Analysis | Sieve and Hydrometer | ASTM |
| PAH | headspace | modified NIOSH 5506 | Polynuclcar Aromatic Hydrocarbons | HPLC | Standard Methods |

Table 3-4
Summary of Chemicals

IT Project No. 408250

| Chemical | Grade | Source |
|---|---|---|
| Sodium salicylic | Reagent | J.T. Baker, Phillipsburg, N.J. |
| Sodium succinic | Reagent | Mallinckrodt. Paris, KE. |
| Ammonium chloride | Reagent | Mallinckrodt, Paris, KE. |
| Potassium phosphate dibasic | Reagent | J.T. Baker, Phillipsburg, N.J. |
| Hydrogen peroxide (35%) | Reagent | PB&S Chem. Co., Hendersonville, KE. |
| Sodium Hydroxide | Reagent | J.T. Baker. Phillipsburg. N.J. |
| Ferrous sulfate • 7H2O | Reagent | Sigma Chemical Co., St. Louis, MO. |
| Sodium sulfate anhydrous | Reagent | Mallinckrodt, Paris, Kentucky |
| Methylene chloride | Nanograde | Burdick & Jackson, Muskegon, WI. |
| PAH standards | | Supelco, Inc., Bellefonte. PA. |
| Acetonitrile | Nanograde | Burdick & Jackson, Muskegon, WI. |

*Table* ***3-5***
***List of Technical Changes***

***IT Project No. 408250***

| CHANGE NUMBER | TECHNICAL CHANGE | EPA APPROVED[a] |
|---|---|---|
| 1 | BAC completion of initial EIMCO soil evaluation. | 10/7/94 |
| 2 | Use of 1.3 M ferrous sulfate and 30 percent peroxide solution to prepare Fenton's reagent. | 10/7/94 |
| 3 | Only reagent grade chemicals used are nutrients. | 10/7/94 |
| 4 | Reactor 2 equipment change. | 10/7/94 |
| 5 | No influent air filtration. | 10/7/94 |
| 6 | All reactors sampled from middle port. | 10/7/94 |
| 7 | Calibration of thermocouple by manufacturer. | 10/7/94 |
| 8 | Change in components of air sampling tram. | 10/7/94 |
| 9 | Placement of anhydrous sodium sulfate during sample extraction. | 10/7/94 |
| 10 | Vessel volume change during initial batch study. | 10/7/94 |
| 11 | Correction of QAPP concerning surrogate additions. | 10/7/94 |
| 12 | Addition of GC/MS confirmation for PAH and PCP. | 10/7/94 |
| 13 | Fenton's reagent addition at 2.0 L/day. | 10/7/94 |
| 14 | PAH extract exchange and dilution. | 10/7/94 |
| 15 | Preparation of surrogates through l-gram vial additions and analysis. | 10/7/94 |
| 16 | QA/QC analyses for PAH at 10 percent of ail samples collected. | 10/7/94 |
| 17 | Surrogate switched to 1-Fluoronapthaiene. | Verbal on 10/7/94 |

## *LIST OF TECHNICAL CHANGES (CONTINUED)*

## *IT Project No. 408250*

| CHANGE NUMBER | TECHNICAL CHANGE | EPA APPROVED[a] |
|---|---|---|
| 18 | Increase reactor and influent feed TS concentration to 40 percent. | Verbal on 10/7/94 |
| 19 | Change in salicylate and succinate concentration additions. | Verbal on 10/7/94 |
| 20 | Discontinued use of the clarifier. | Verbal on 10/7/94 |
| 21 | Increased daily TS and density measurements and reduced DO measurements during period of varying reactor solids distribution. | Verbal on 10/7/94 |
| 22 | Rerun samples when matrix is out of ± 20 percent | Verbal on 10/7/94 |
| 23 | Recalculate MDLs | Verbal on 10/7/94 |
| 24 | Changes in the analytical schedule for TS/VS, DO, aqueous PAH, nitrogen, gene probe, and pH | 12/20/94 |
| 25 | Increased impeller speed to control DO | 12/20/94 |
| 26 | Adjust volume of Rl | 12/20/94 |
| 27 | Decrease influent organic loading by one-half through the mixing of clean clay | 12/20/94 |
| 28 | Reduction in Rl and R3 HRT from 10 to 20 days | 12/24/95 |
| 29 | Reduce feed introduction to each reactor from twice/day to once/day | 12/24/95 |
| 30 | Discontinued addition of salicylate and succinate | 4/6/95 |

a Date indicates date of EPA correspondence documenting technical variance approval.

Table *3-6*
*List of Technical Nonconformances*

*IT Project No. 408250*

| NONCONFORMANCE NO. | NONCONFORMANCE | DATE REPORTED |
|---|---|---|
| 1 | Influent feed added as 2, 3L transfers instead of 3, 2L transfers. | 10/19/94 |
| 2 | Bottle study recoveries | 11/8/94 |
| 3 | Influent $T_0$ MS and MSD | 1l/8/94 |
| 4 | SRM acceptance criteria | 12/12/94 |
| 5 | PAH not identified during aqueous analyses | 12/12/94 |
| 6 | Method calibration for chrysene 10/14/94 | 12/12/94 |
| 7 | Missing check standards | 12/12/94 |

Table 4-1
Summary of Reactor Upsets and Operational Modifications

IT Project No. 408250

| Date | Upsets/Modifications |
|---|---|
| 29 November, 1994 | Rl airlifts clogged |
| 5 December, 1994 | R1 dismantled to remove clogs from airlifts; tar balls responsible for clog |
| 13 December, 1994 | In order to reduce organic loading in R1, solids were reduced to 20% and 15% diatomaceous earth was added |
| 29 December, 1994 | Discontinued use of diatomaceous earth as a solid supplement; caused air lifts to clog |
| 11 January, 1995 | Supplemented solids with a local, clean clay |
| 24 January, 1995 | Switched to 8.8 mM $FeSO_4.7H_2O$ |
| 27 February, 1995 | Increased HRT to 20 days for R1, 2 days for R2, and 20 days for R3 |
| 22 March, 1995 | Discontinued salicylate and succinate addition to R1 |

Table 4-2
Summary of Total Recoverable Petroleum Hydrocarbons (TRPH), PAH, and CPAH for March 16 Samples

IT Project No. 408250

| Sample | TRPH | PAH | CPAH |
|---|---|---|---|
| Influent Feed | 27,000 | 5860 | 430 |
| Reactor 1 | 14,000 (48%)[1] | 710 (88%) | 130 (70%) |
| Reactor 2 | 14,000 (48%) | 430 (93%) | 70 (84%) |
| Reactor 3 | 3,700 (86 %) | 560 (90%) | 110 (74%) |

[1]Number in parenthesis represents the percent reduction relative to the influent feed TRPH concentration.

Table 4-3
PAH Removal Efficiencies (Percent)

IT Project No. 408250

| Date | Reactor 1 | Reactor 2 | Reactor 3 | Overall |
|---|---|---|---|---|
| 10/10 | 85 | -180 | 36.1 | 72.6 |
| 10/19 | 65 | 58.2 | 67 | 95.2 |
| 10/26 | 29 | 45.2 | 59.3 | 84.2 |
| 11/2 | 55.5 | 37.7 | 42 | 83.9 |
| 11/9 | 59.2 | 26.7 | 44.8 | 83.5 |
| 11/16 | 61.7 | 41 | 59.6 | 90.9 |
| 11/22 | 55.3 | 42.9 | 69.5 | 92.2 |
| 12/2 | 30.4 | 62 | 28.9 | 81 |
| 12/8 | 56.9 | 44.1 | 5.7 | 77.2 |
| 12/15 | 68.6 | 22 | 31.7 | 83.3 |
| 12/30 | 71.6 | 51.1 | 6.5 | 87 |
| 1/5 | 63.6 | 64 | 44.8 | 92.8 |
| 1/26 | 69.3 | 44.8 | 12.1 | 85.1 |
| 2/2 | 71.6 | 38 | 1.2 | 82.6 |
| 2/16 | 79.8 | 22.3 | -19 | 81.3 |
| 3/2 | 85.9 | 33 | -82 | 82.8 |
| 3/9 | 87.9 | 39.1 | -31 | 90.4 |
| 3/16 | 86 | 37 | 10.3 | 92 |
| 3/30 | 85.8 | 65.9 | -66 | 91.9 |
| 4/13 | 88.6 | 37 | 12.9 | 93.8 |
| 4/19 | 88.3 | 55.4 | -1.1 | 94.7 |
| Total Operational Period Average | 69.17 ± 17.9 | 32.7 ± 50.3 | 15.87 ± 40.57 | 86.59 ± 6.17 |
| Average Prior to 3/2 | 61.5 ± 15.45 | 28 ± 58.95 | 32.68 ± 26.56 | 84.85 ± 6.05 |
| Average Following 3/2 | 87.08 ± 1.32 | 44.6 ± 13.04 | -26.2 ± 40.53 | 90.93 ± 4.26 |

± Indicates standard deviation.

Table 4-4
CPAH Removal Efficiencies (Percent)

IT Project No. 408250

| Date | Reactor 1 | Reactor 2 | Reactor 3 | Overall |
|---|---|---|---|---|
| 10/10 | 60 | -58.2 | 40.9 | 62.2 |
| 10/19 | 20.2 | 56.9 | 49 | 84.3 |
| 10/26 | -16 | 48.5 | 33.9 | 60.5 |
| 11/2 | 2.5 | 40.5 | 26 | 56.9 |
| 11/9 | -1.1 | 23.9 | 44 | 56.9 |
| 11/16 | 9.3 | 50 | 44.5 | 74.7 |
| 11/22 | 30.4 | 33.4 | 51.2 | 77.4 |
| 12/2 | 10.4 | 51 | 26.3 | 67.7 |
| 12/8 | 30.9 | 48.3 | -7.1 | 61.8 |
| 12/15 | 32.7 | 15 | 36.1 | 63.4 |
| 12/30 | 43.5 | 43.3 | 12.9 | 72.1 |
| 1/5 | 38 | 59 | -26 | 67.7 |
| 1/26 | 30.7 | 35.3 | 14.4 | 61.6 |
| 2/2 | 37.6 | 40 | -8.4 | 59.4 |
| 2/16 | 44.4 | 23.3 | -5 | 55.2 |
| 3/2 | 66.4 | 34 | -105 | 54.2 |
| 3/9 | 71 | 46.3 | -63 | 74.7 |
| 3/16 | 59.9 | 44 | 2.5 | 78 |
| 3/30 | 60.9 | 69 | -93 | 76.6 |
| 4/13 | 69 | 44 | -1.2 | 82.6 |
| 4/19 | 64.8 | 55.9 | -3.1 | 84 |
| Total Operational Period Average | 36.83 ± 25.18 | 38.26 ± 25.52 | 3.3 ± 44.19 | 68.19 ± 9.92 |
| Average Prior to 3/2 | 27.99 ± 22.11 | 34.01 ± 27.51 | 14.23 ± 39.51 | 65.45 ± 8.42 |
| Average Following 3/2 | 65.33 ± 4.39 | 48.87 ± 12.08 | -31.56 ± 43.75 | 75.02 ± 10.79 |

± Indicates standard deviation.

Table 4-5
PAH Mass Removals (Grams/Day)

IT Project No. 408250

| Date | Reactor 1 | Reactor 2 | Reactor 3 |
|---|---|---|---|
| 10/10 | 35.5 | 0 | 17.50 |
| 10/19 | 0 | 1.78 | 11.49 |
| 10/26 | 0 | 3.68 | 0 |
| 11/2 | 15.6 | 4.00 | 0 |
| 11/9 | 19.7 | 4.90 | 0 |
| 11/16 | 7.13 | 2.78 | 5.95 |
| 11/22 | 2.65 | 5.35 | 4.24 |
| 12/2 | 7.86 | 4.14 | 0 |
| 12/8 | 9.57 | 11.23 | 1.43 |
| 12/15 | 12.67 | 4.30 | 6.75 |
| 12/30 | 13.82 | 4.96 | 10.55 |
| 1/5 | 12.48 | 1.26 | 5.61 |
| 1/26 | 12.52 | 3.15 | 0 |
| 2/2 | 13.02 | 4.06 | 0 |
| 2/16 | 14.01 | 3.32 | 0.90 |
| 3/2 | 7.62 | 2.57 | 1.10 |
| 3/9 | 7.70 | 1.26 | 0 |
| 3/16 | 7.85 | 0 | 0.29 |
| 3/30 | 7.54 | 1.75 | 0.58 |
| 4/13 | 7.90 | 1.47 | 0.47 |
| 4/19 | 7.71 | 1.38 | 1.06 |
| Total Operational Period Average | 10.61 ± 7.5 | 3.21 ± 2.43 | 3.23 ± 4.83 |
| Average Prior to 3/2 | 11.51 ± 8.45 | 3.93 ± 2.49 | 4.29 ± 5.39 |
| Average Following 3/2 | 7.74 ± 0.14 | 1.17 ± 0.68 | 0.48 ± 0.39 |

± Indicates standard deviation.

Table 4-6
CPAH Mass Removal (Grams/Day)

IT Project No. 408250

| Date | Reactor 1 | Reactor 2 | Reactor 3 |
|---|---|---|---|
| 10/10 | 0.44 | 0 | 0.29 |
| 10/19 | 0 | 0.31 | 0.56 |
| 10/26 | 0 | 0.52 | 0 |
| 11/2 | 0.65 | 0.47 | 0 |
| 11/9 | 1.61 | 0.76 | 0 |
| 11/16 | 0.18 | 0.59 | 0.97 |
| 11/22 | 0 | 0.73 | 0.44 |
| 12/2 | 0 | 0.43 | 0.28 |
| 12/8 | 2.33 | 1.00 | 0.18 |
| 12/15 | 0.24 | 0.24 | 0 |
| 12/30 | 4.02 | 0.76 | 1.82 |
| 1/5 | 0.50 | 0.19 | 0 |
| 1/26 | 0.32 | 0.34 | 0 |
| 2/2 | 1.90 | 0.69 | 0.12 |
| 2/16 | 0.95 | 0.49 | 0.06 |
| 3/2 | 3.33 | 0.55 | 0.01 |
| 3/9 | 1.06 | 0.23 | 0 |
| 3/16 | 0 | 0 | 0.08 |
| 3/30 | 0.44 | 0.34 | 0.07 |
| 4/13 | 1.31 | 0.29 | 0.10 |
| 4/19 | 0.33 | 0.25 | 0.16 |
| Total Operational Period Average | 0.93 ± 1.13 | 0.44 ± 0.26 | 0.24 ± 0.43 |
| Average Prior to 3/2 | 0.88 ± 1.14 | 0.50 ± 0.26 | 0.29 ± 0.49 |
| Average Following 3/2 | 0.63 ± 0.54 | 0.22 ± 0.13 | 0.08 ± 0.06 |

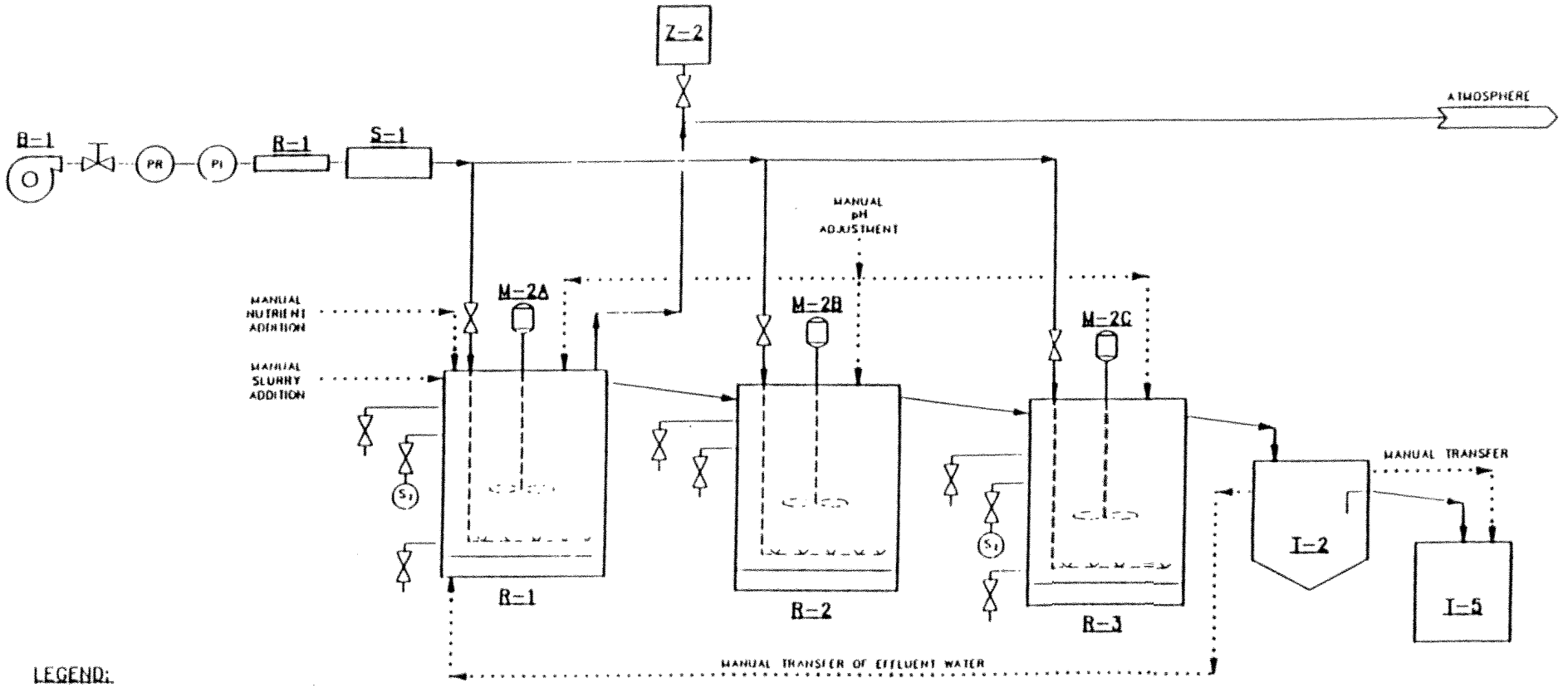
± Indicates standard deviation.

Table 4-7
16-Hour Toxicity Determination Using Tetrahymena

IT Project No. 408250

| Sample | Sample Dilution | Percent Mortality |
|---|---|---|
| Influent Feed | 1:10<br>1:20<br>1:40<br>1:100 | 100<br>90<br>50<br>NOEC[1] |
| Reactor 1 | 1:40<br>1:100<br>1:200 | 100<br>50<br>NOEC |
| Reactor 3 | 1:40<br>1:100<br>1:200 | 100<br>50<br>NOEC |

[1]NOEC - No observable effect concentration




LEGEND:

- $S_1$ SAMPLE PORT
- PR PRESSURE REGULATOR
- PI PRESSURE INDICATOR

| | | | | |
|---|---|---|---|---|
| B-1 AIR BLOWER | S-1 AIR FILTER | R-1 BIO REACTOR 1 (60L) | R-3 BIO REACTOR 3 (60L) | Z-2 AIR SAMPLING DEVICE |
| B-1 AIR ROTAMETER | M-2A,B,C BIOREACTOR MIXER | R-2 BIO REACTOR 2 (60L) | I-2 CLARIFIER | I-5 EFFLUENT CONTAINER |

Figure 3-1

PROCESS FLOW DIAGRAM
BIOSLURRY REACTOR

*SITE ETP06*
*IT PROPOSAL #408582*

INTERNATIONAL TECHNOLOGY CORPORATION

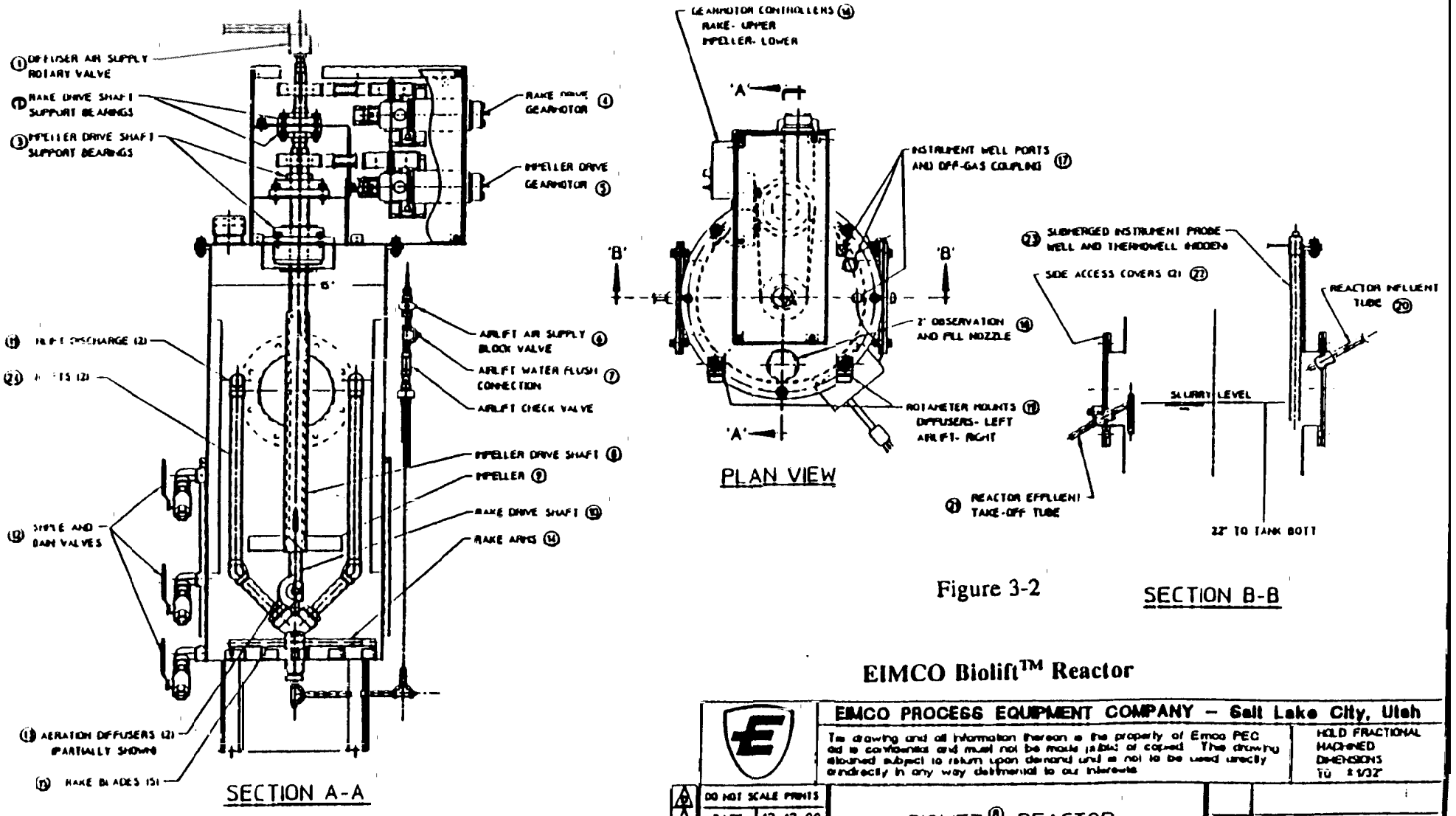



Figure 3-2

**EIMCO Biolift™ Reactor**

FIGURE 41
REACTOR pH DATA

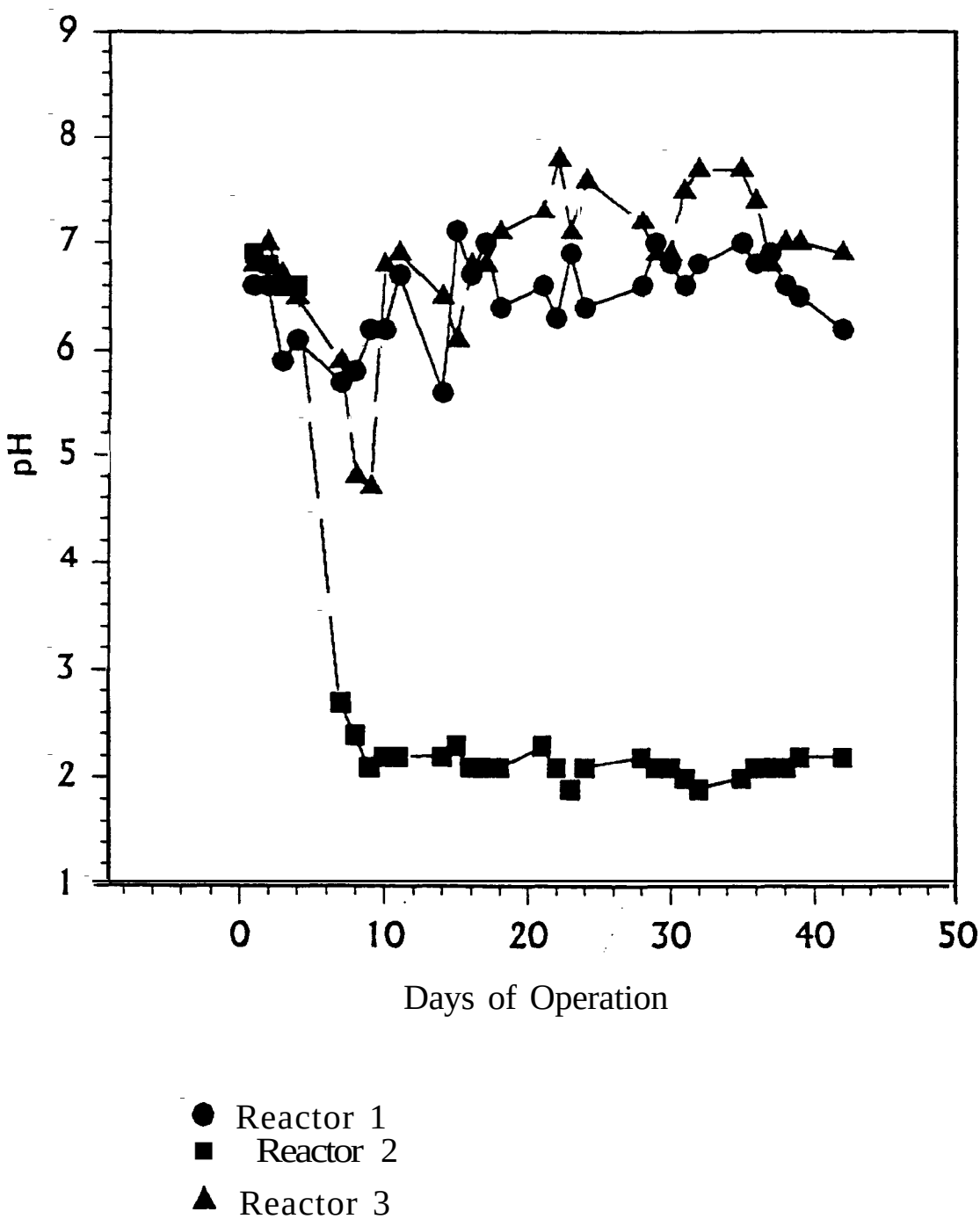
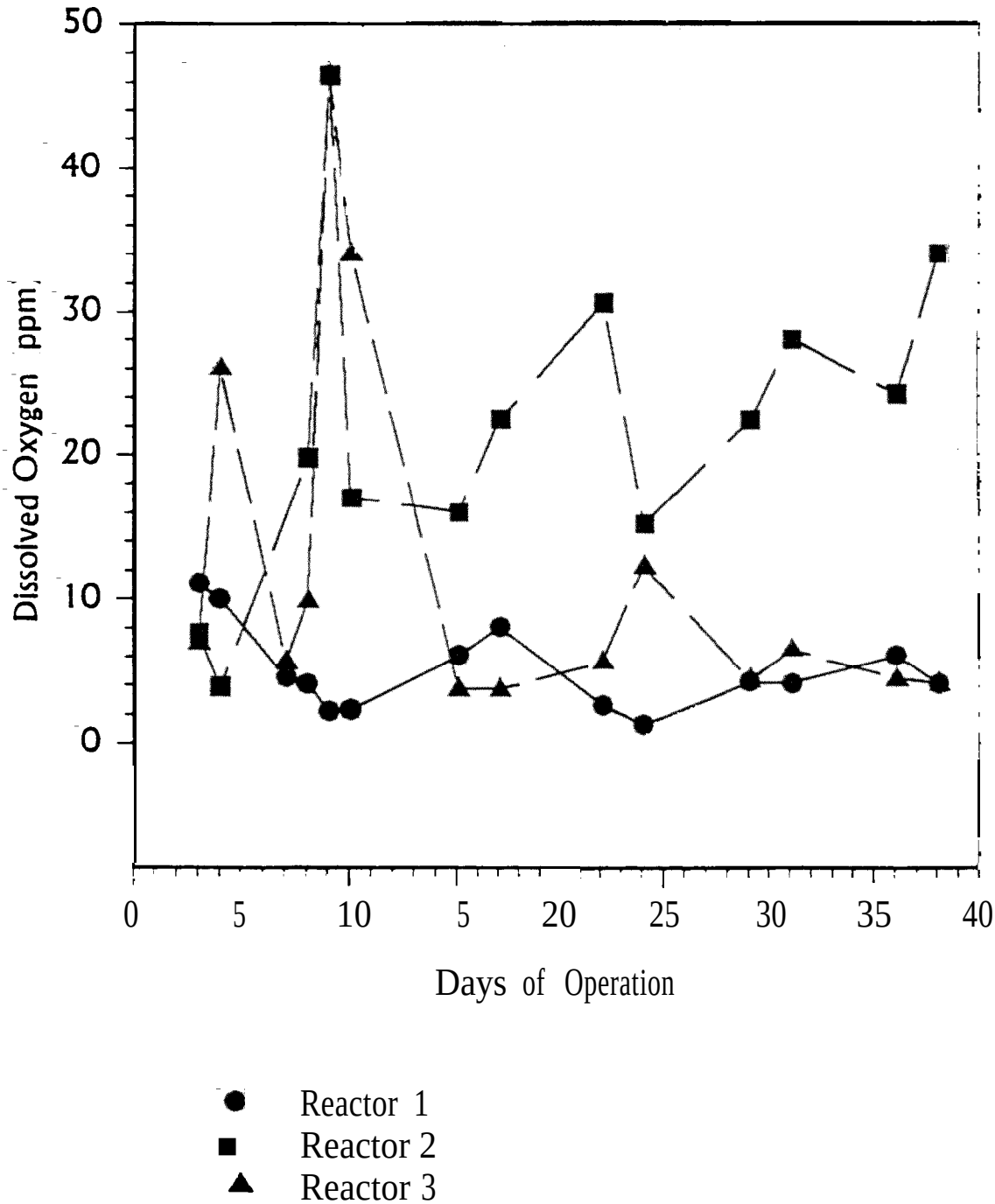
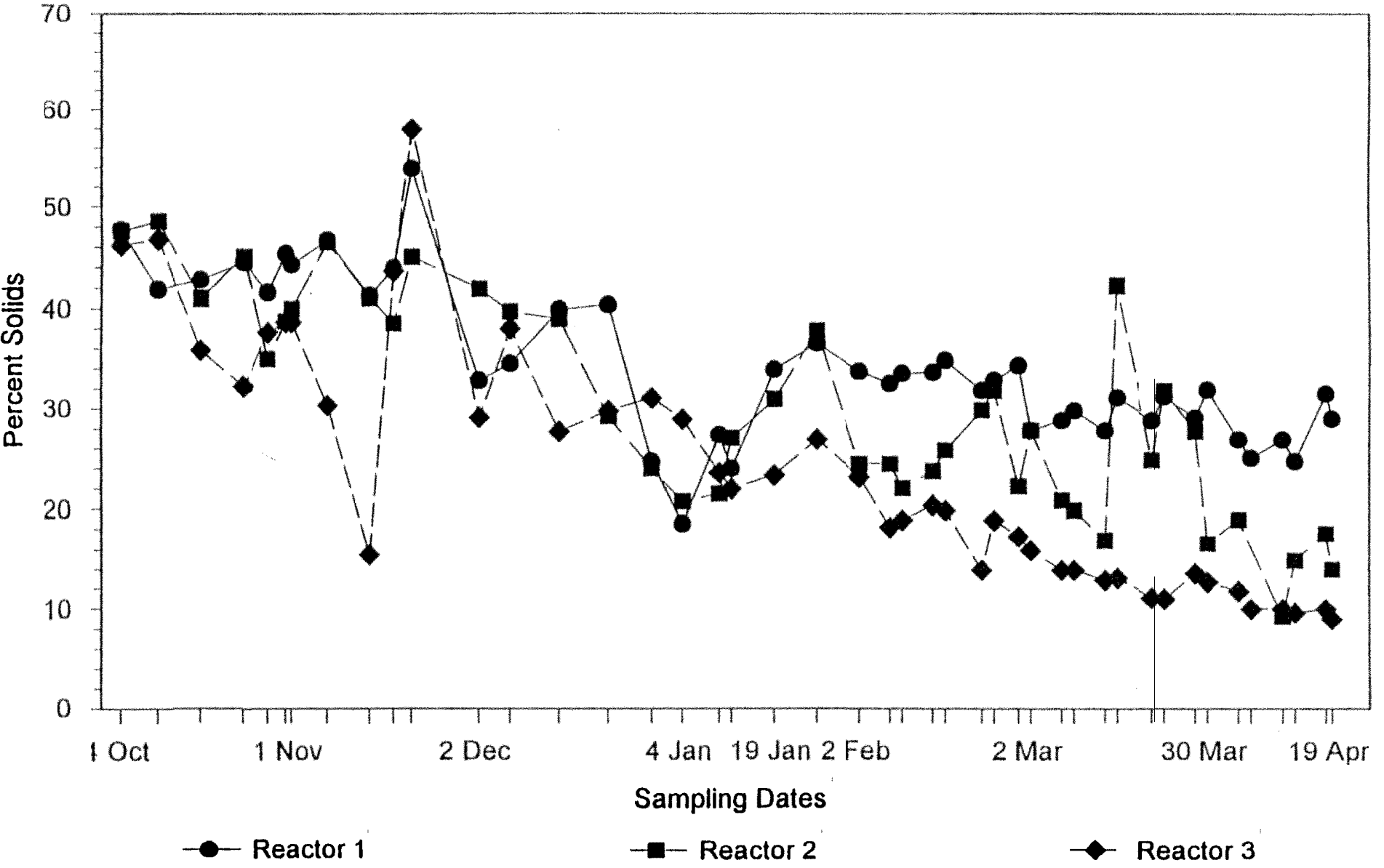


FIGURE 4-2

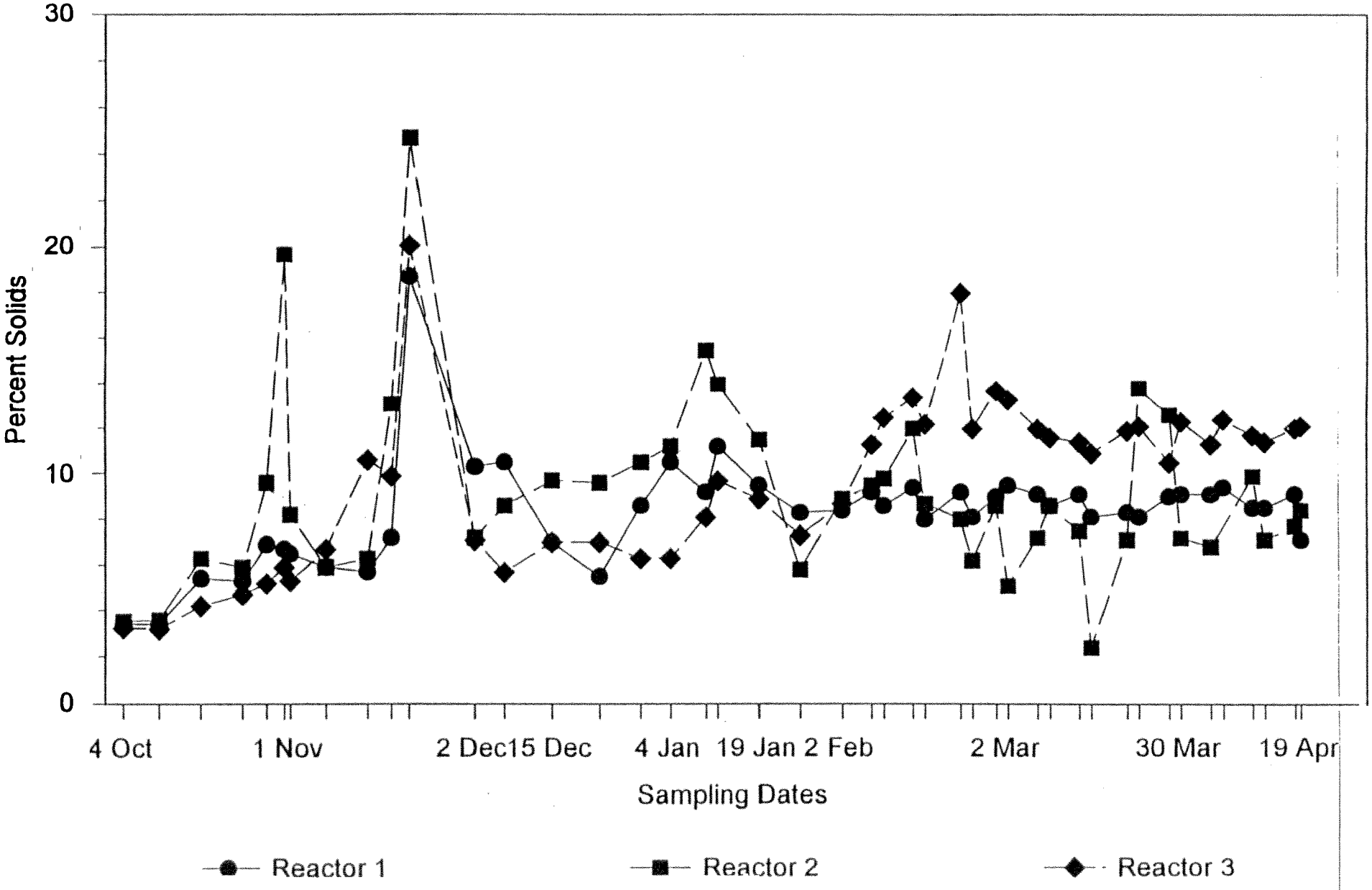
DISSOLVED OXYGEN DATA



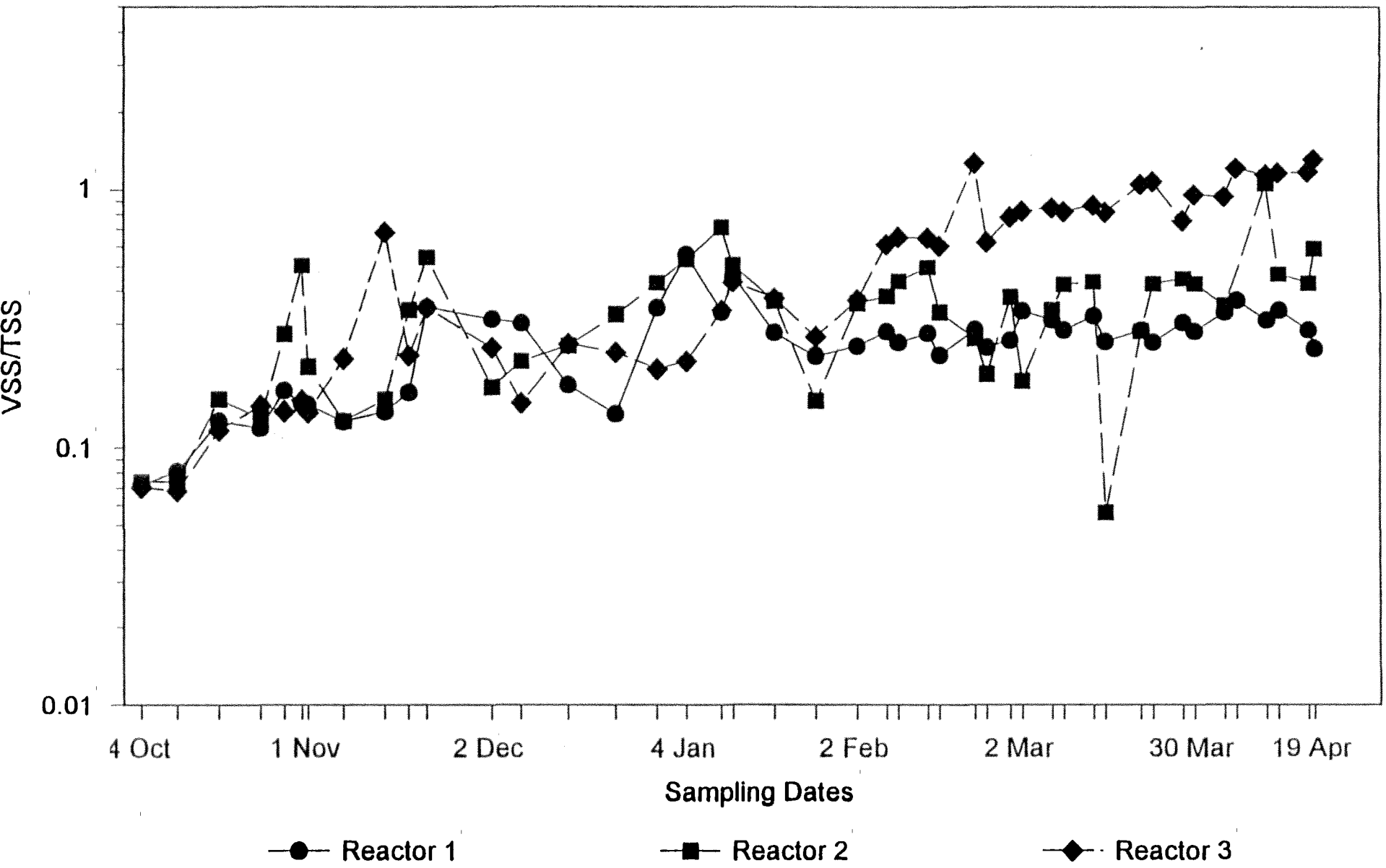
# FIGURE 4-3
# REACTOR TOTAL SOLIDS



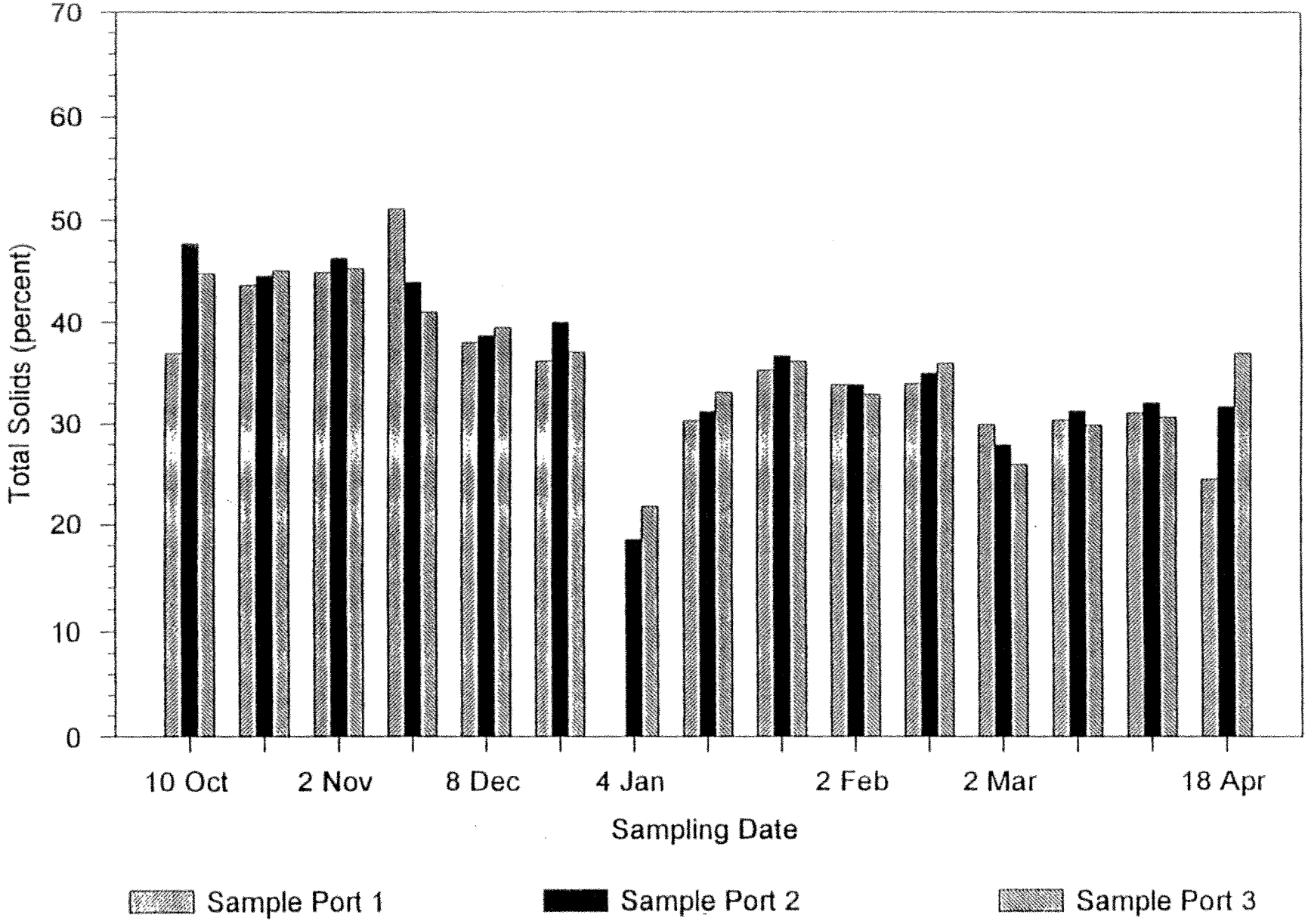
# FIGURE 4-4
# REACTOR VOLATILE SOLIDS



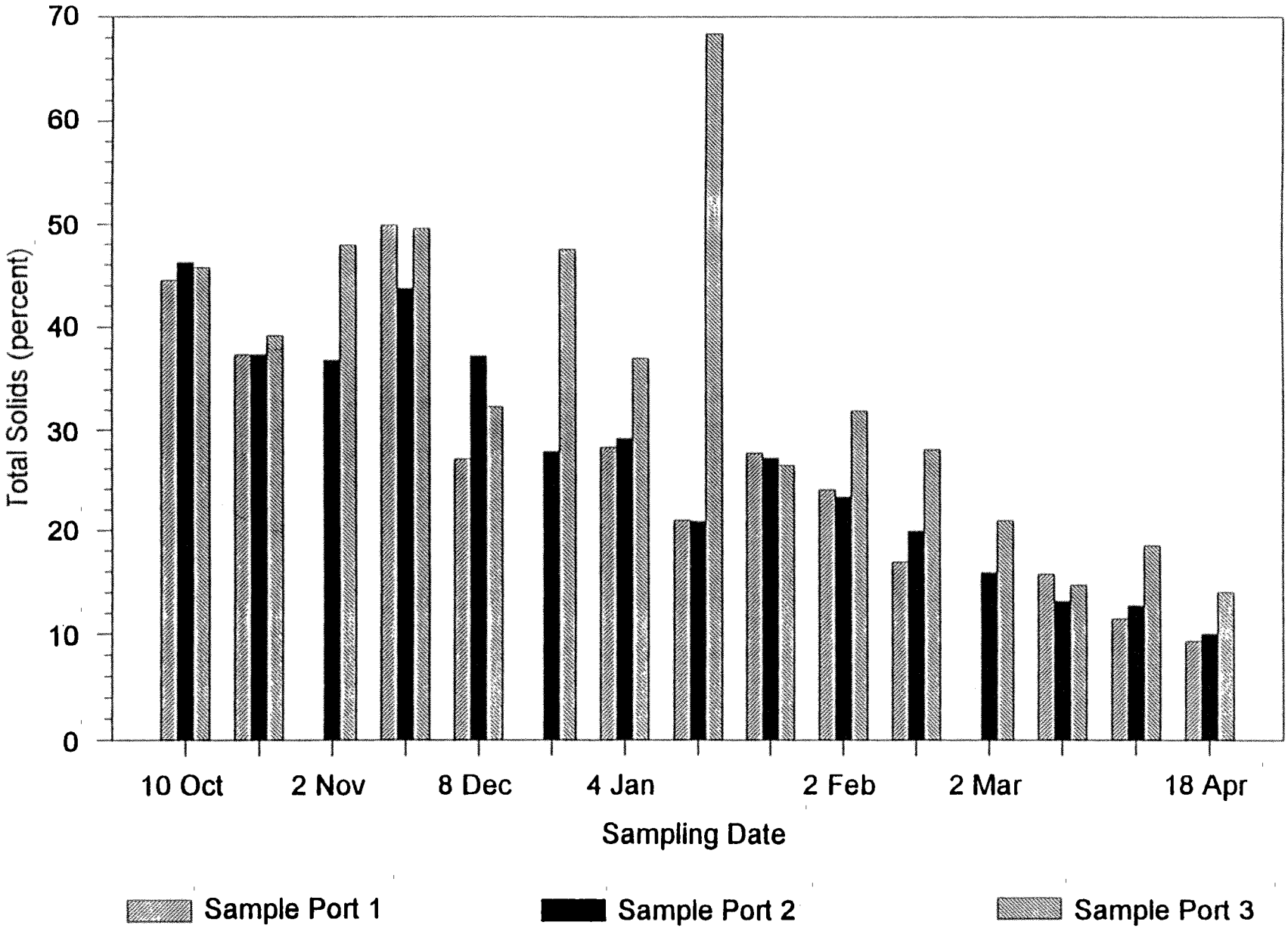
# FIGURE 4-5
# RATIO OF VOLATILE SOLIDS TO TOTAL SOLIDS



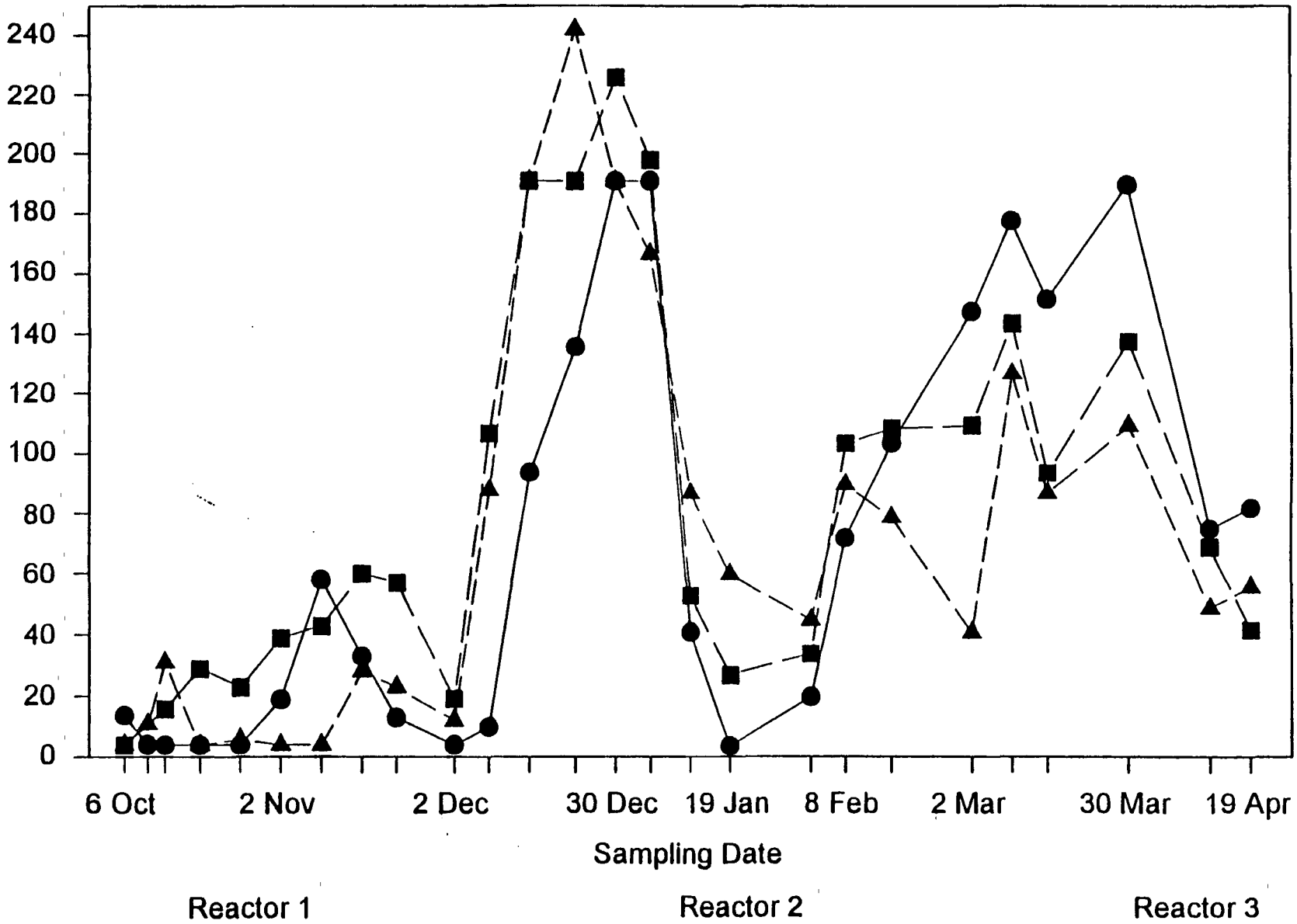
# FIGURE 4-6
# SOLIDS DISTRIBUTION IN REACTOR 1



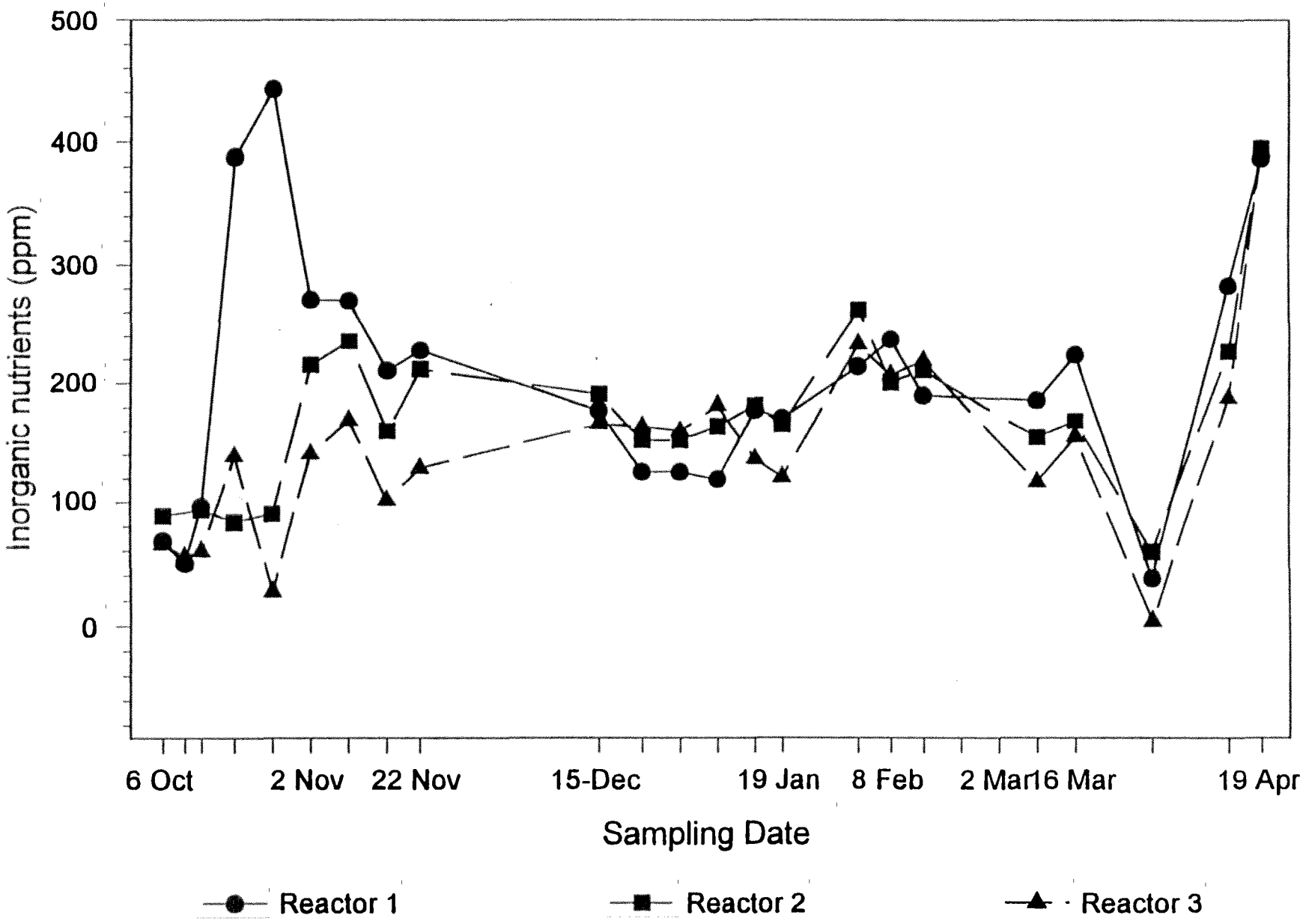
# FIGURE 4-7
# SOLIDS DISTRIBUTION IN REACTOR 3



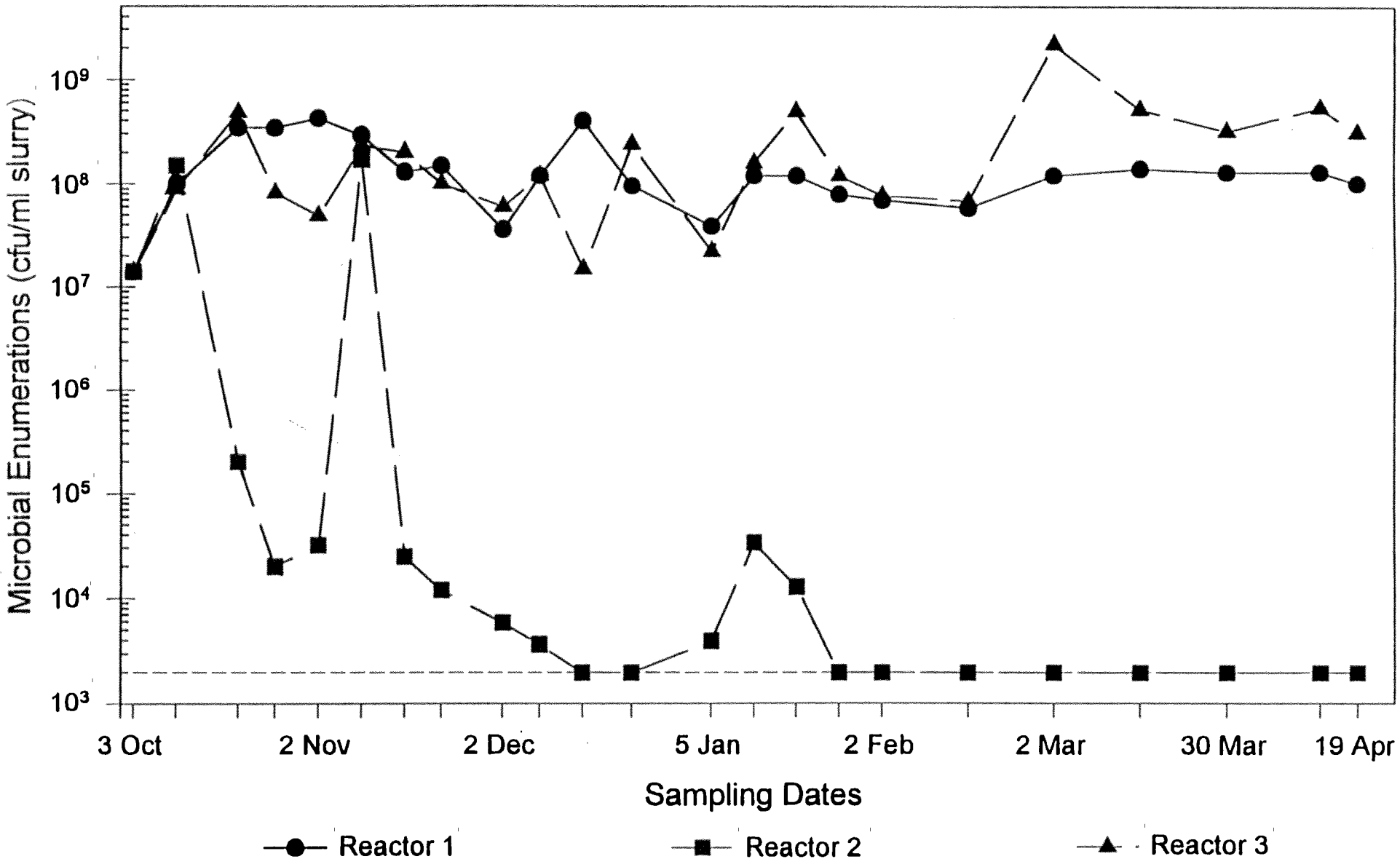
# FIGURE 4-8
# REACTOR AMMONIA CONCENTRATION



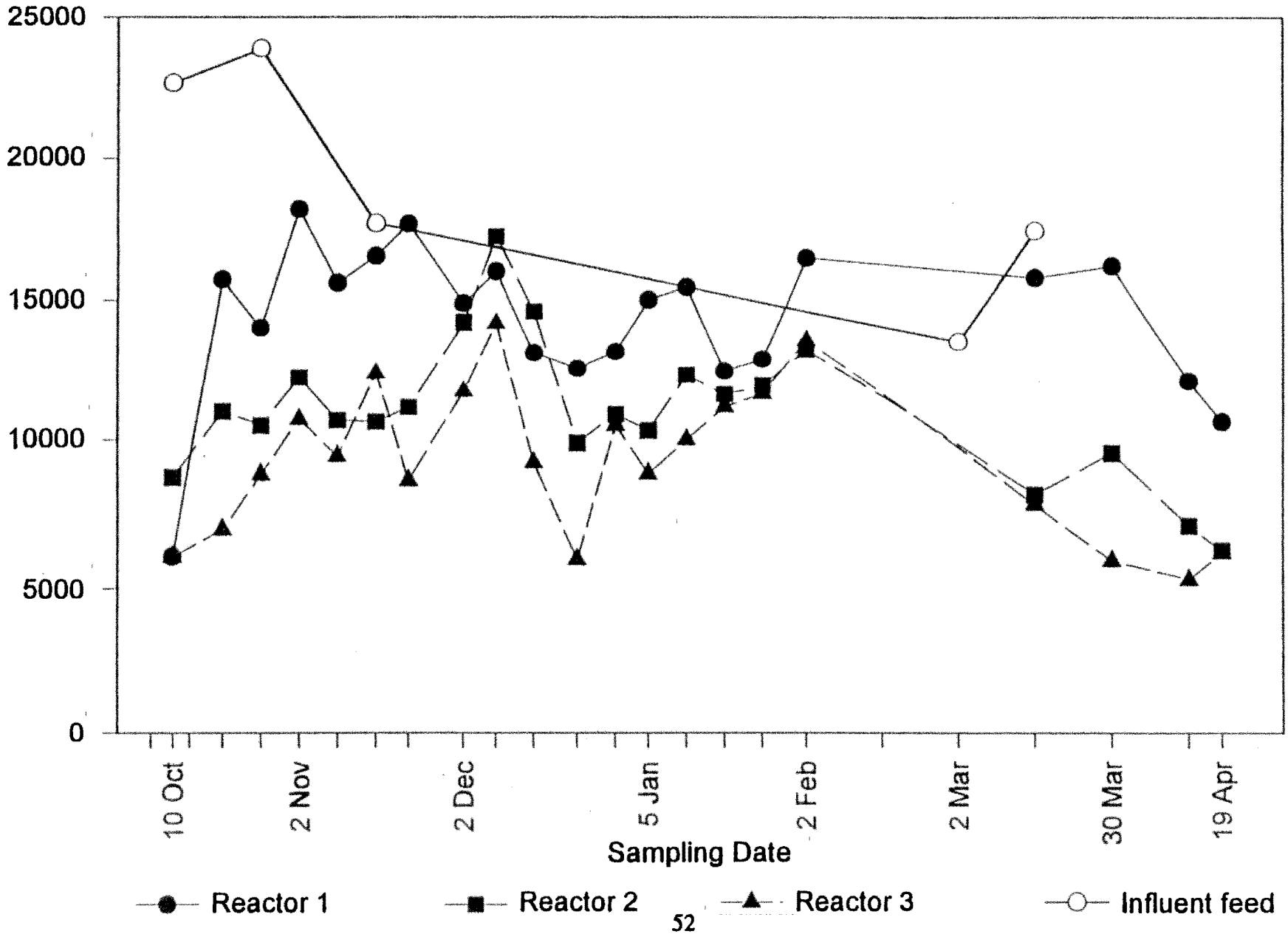
# FIGURE 4-9
# REACTOR PHOSPHATE CONCENTRATION



# FIGURE 4-10
# REACTOR TOTAL HETEROTROPHIC BACTERIAL POPULATIONS



# FIGURE 4-11
# SOLID PHASE TOTAL CARBON



# FIGURE 4-12
# AQUEOUS PHASE TOTAL ORGANIC CARBON

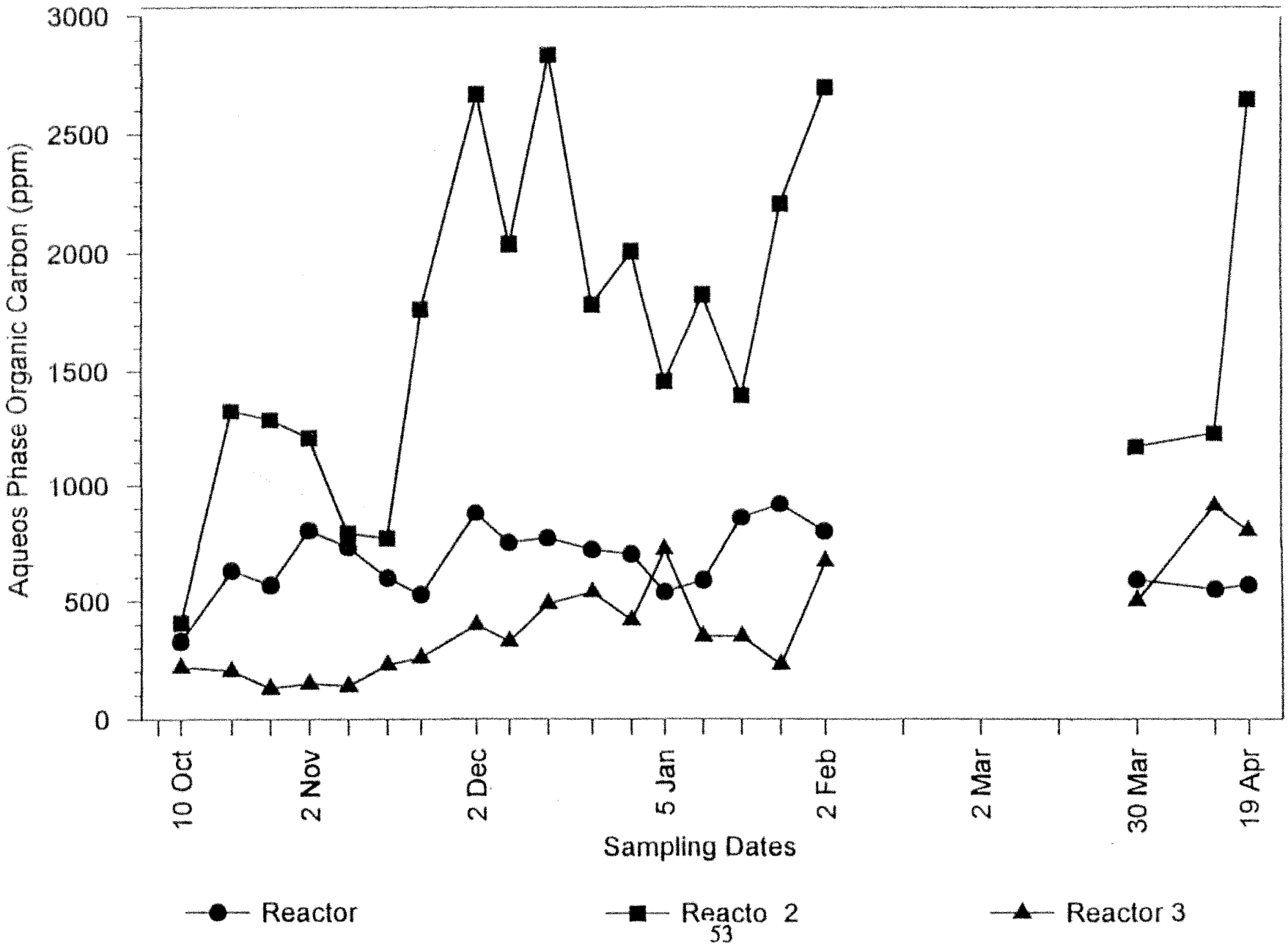


**Figure 4-13**

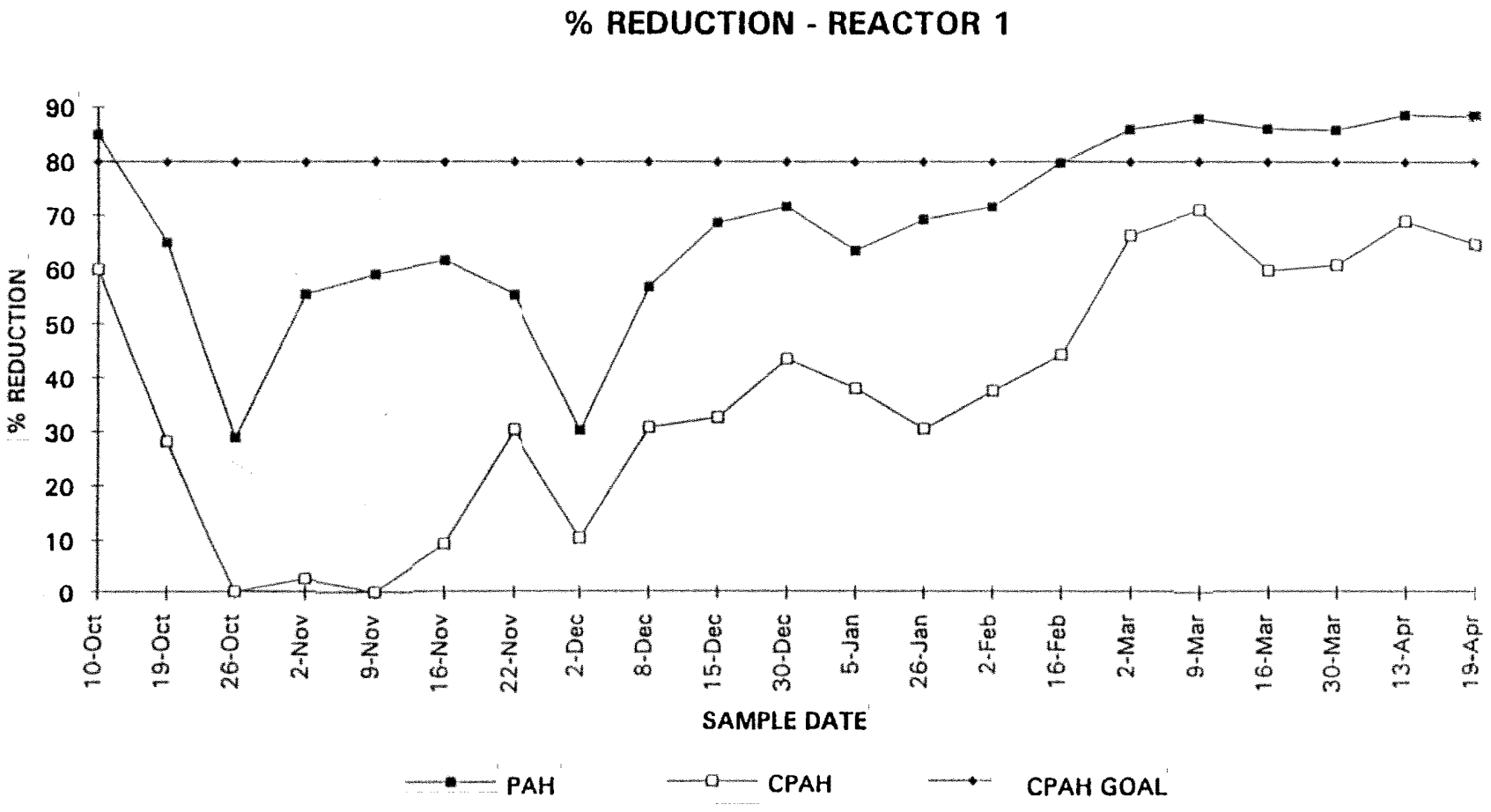

% REDUCTION - REACTOR 1
% REDUCTION
90
80
70
60
50
40
30
20
10
0
10-Oct
19-Oct
26-Oct
2-Nov
9-Nov
16-Nov
22-Nov
2-Dec
8-Dec
15-Dec
30-Dec
5-Jan
26-Jan
2-Feb
16-Feb
2-Mar
9-Mar
16-Mar
30-Mar
13-Apr
19-Apr
SAMPLE DATE
PAH
CPAH
CPAH GOAL

**Figure 4-14**

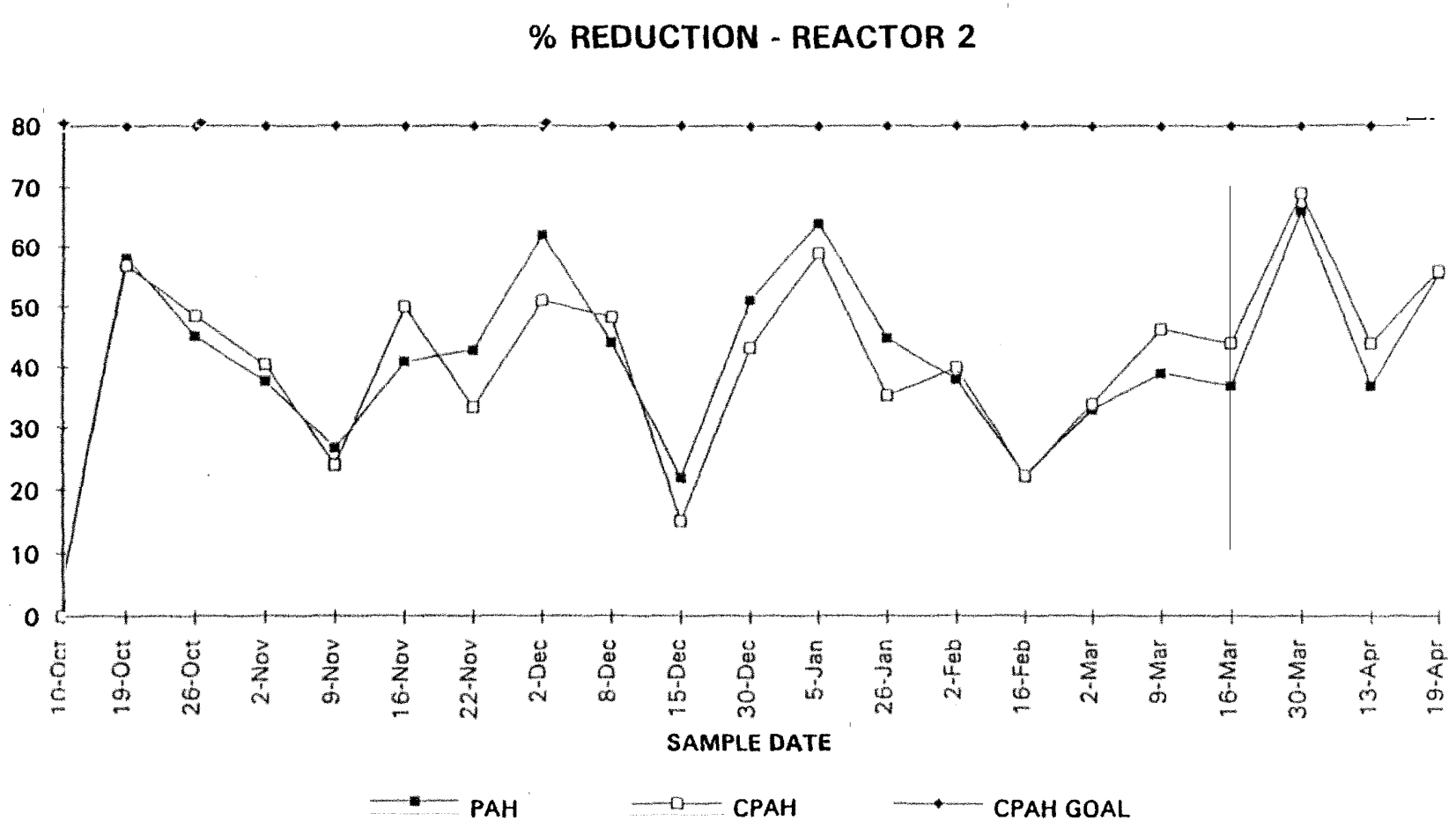

% REDUCTION - REACTOR 2
80
70
60
50
40
30
20
10
0
10-Oct
19-Oct
26-Oct
2-Nov
9-Nov
16-Nov
22-Nov
2-Dec
8-Dec
15-Dec
30-Dec
5-Jan
26-Jan
2-Feb
16-Feb
2-Mar
9-Mar
16-Mar
30-Mar
13-Apr
19-Apr
SAMPLE DATE
PAH
CPAH
CPAH GOAL

**Figure 4-15**

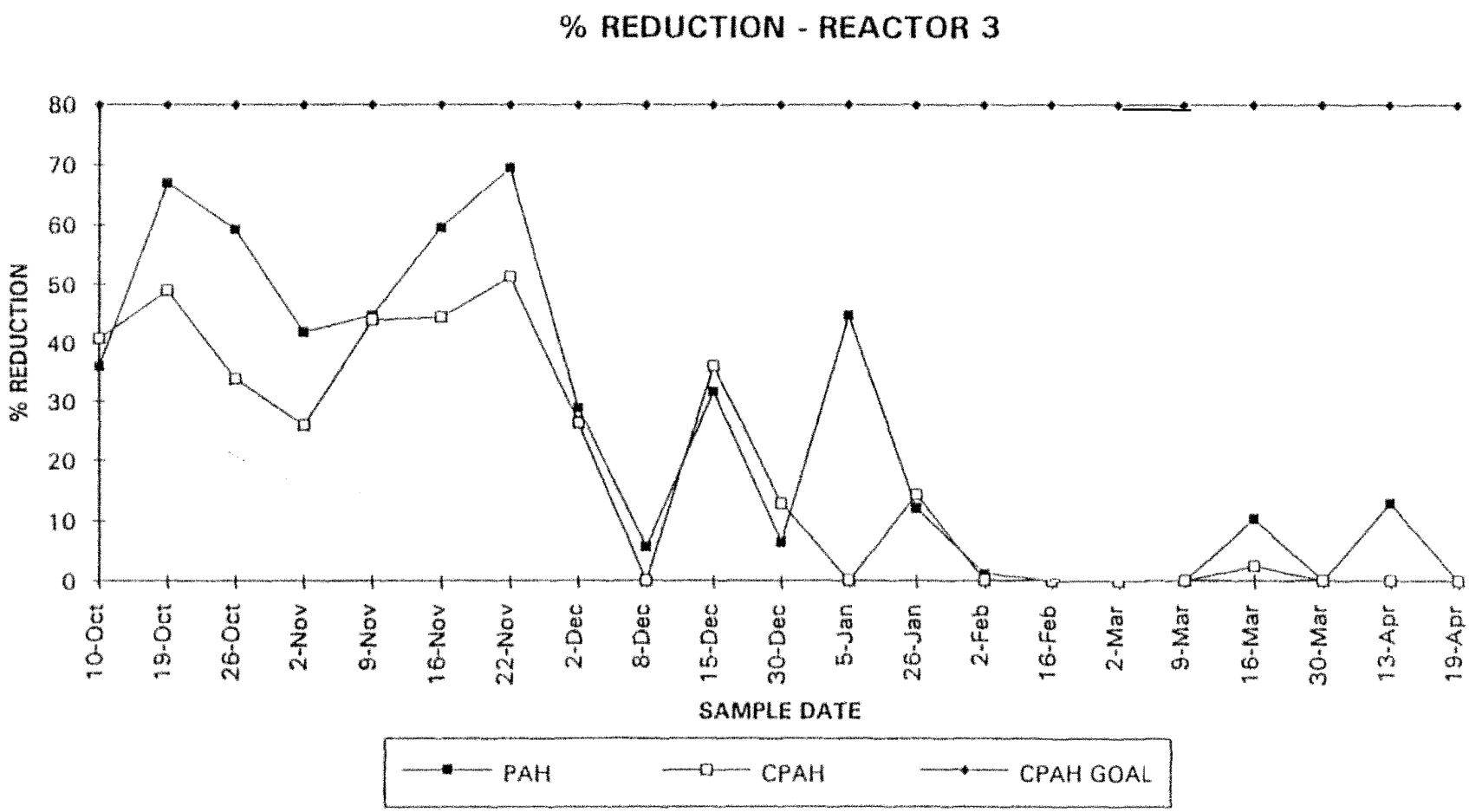

% REDUCTION - REACTOR 3
% REDUCTION
80
70
60
50
40
30
20
10
0
10-Oct
19-Oct
26-Oct
2-Nov
9-Nov
16-Nov
22-Nov
2-Dec
8-Dec
15-Dec
30-Dec
5-Jan
26-Jan
2-Feb
16-Feb
2-Mar
9-Mar
16-Mar
30-Mar
13-Apr
19-Apr
SAMPLE DATE
PAH
CPAH
CPAH GOAL

**Figure 4-16**

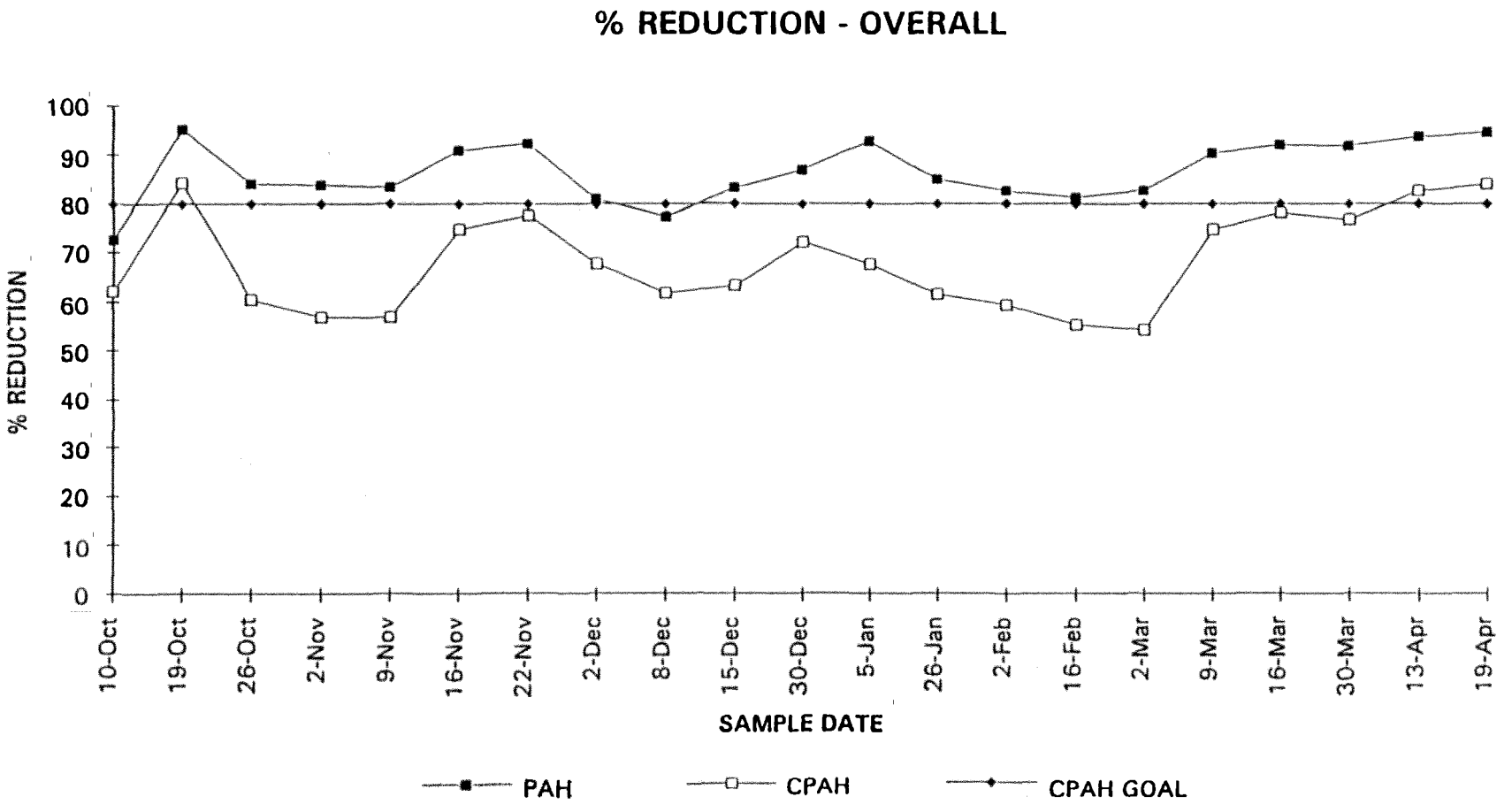
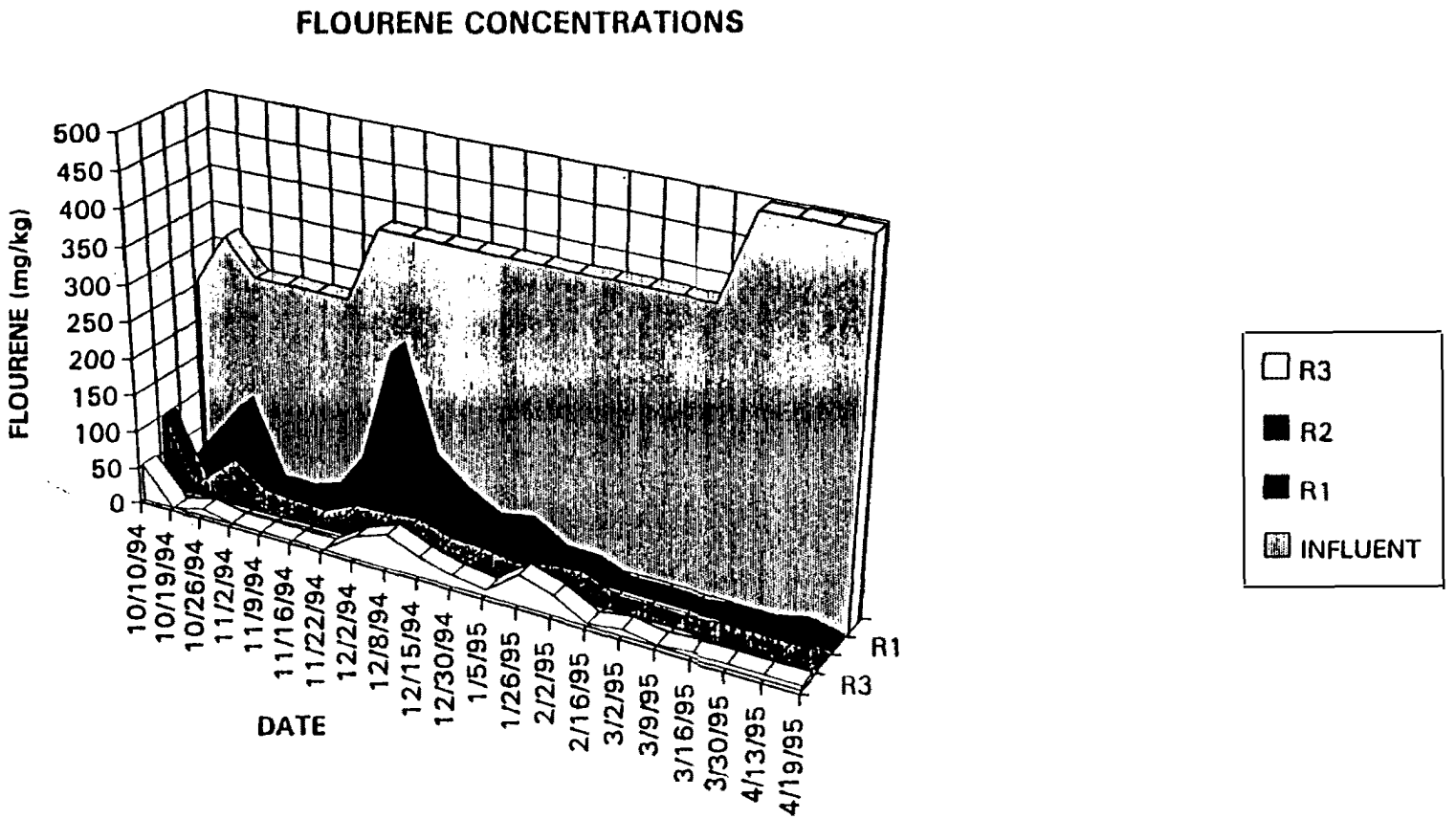

% REDUCTION - OVERALL
% REDUCTION
100
90
80
70
60
50
40
30
20
10
0
10-Oct
19-Oct
26-Oct
2-Nov
9-Nov
16-Nov
22-Nov
2-Dec
8-Dec
15-Dec
30-Dec
5-Jan
26-Jan
2-Feb
16-Feb
2-Mar
9-Mar
16-Mar
30-Mar
13-Apr
19-Apr
SAMPLE DATE
PAH
CPAH
CPAH GOAL

Figure 4-17


FLOURENE CONCENTRATIONS
FLOURENE (mg/kg)
500
450
400
350
300
250
200
150
100
50
0
10/10/94
10/19/94
10/26/94
11/2/94
11/9/94
11/16/94
11/22/94
12/2/94
12/8/94
12/15/94
12/30/94
1/5/95
1/26/95
2/2/95
2/16/95
3/2/95
3/9/95
3/16/95
3/30/95
4/13/95
4/19/95
DATE
R1
R3
R3
R2
R1
INFLUENT

## Figure 4-18

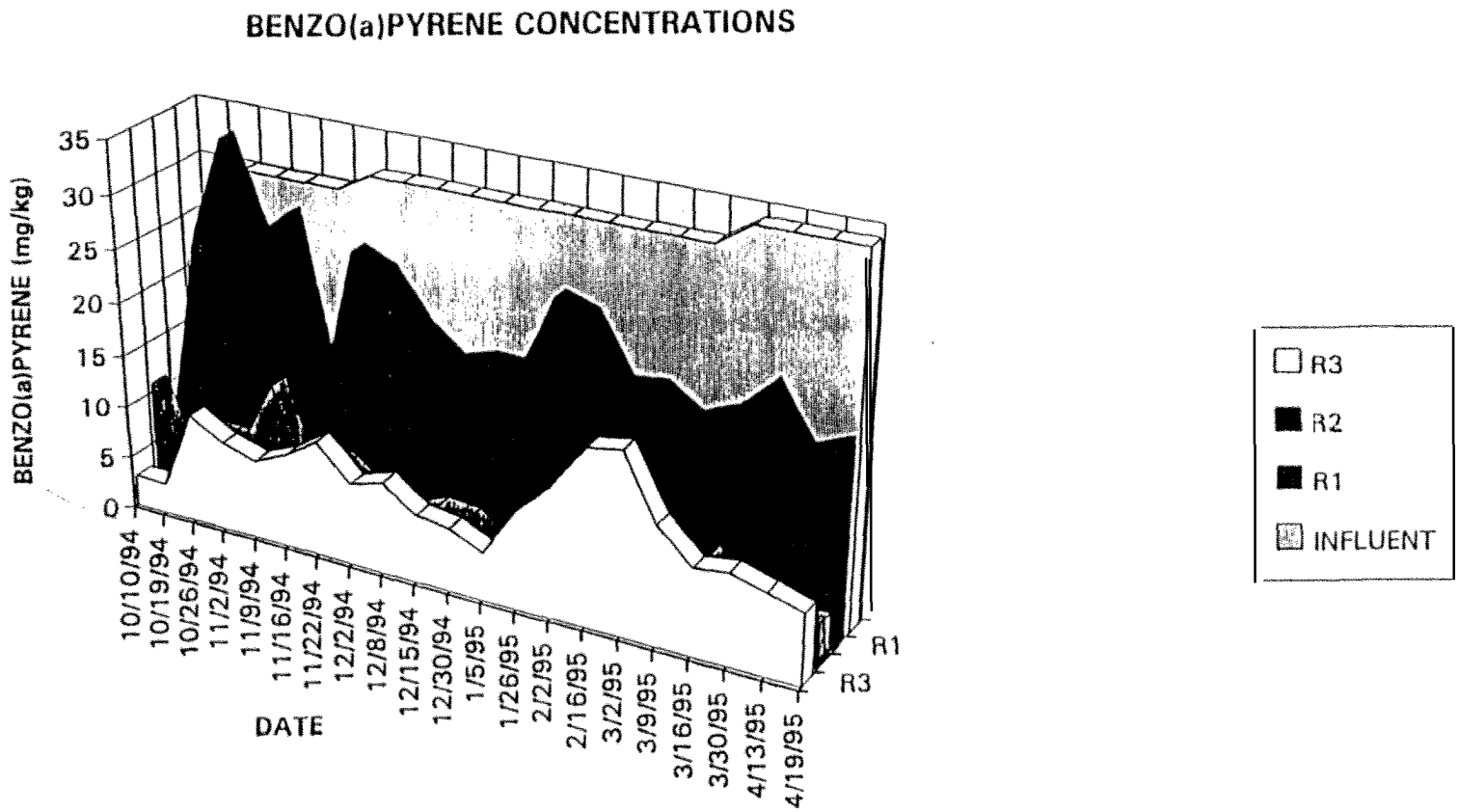
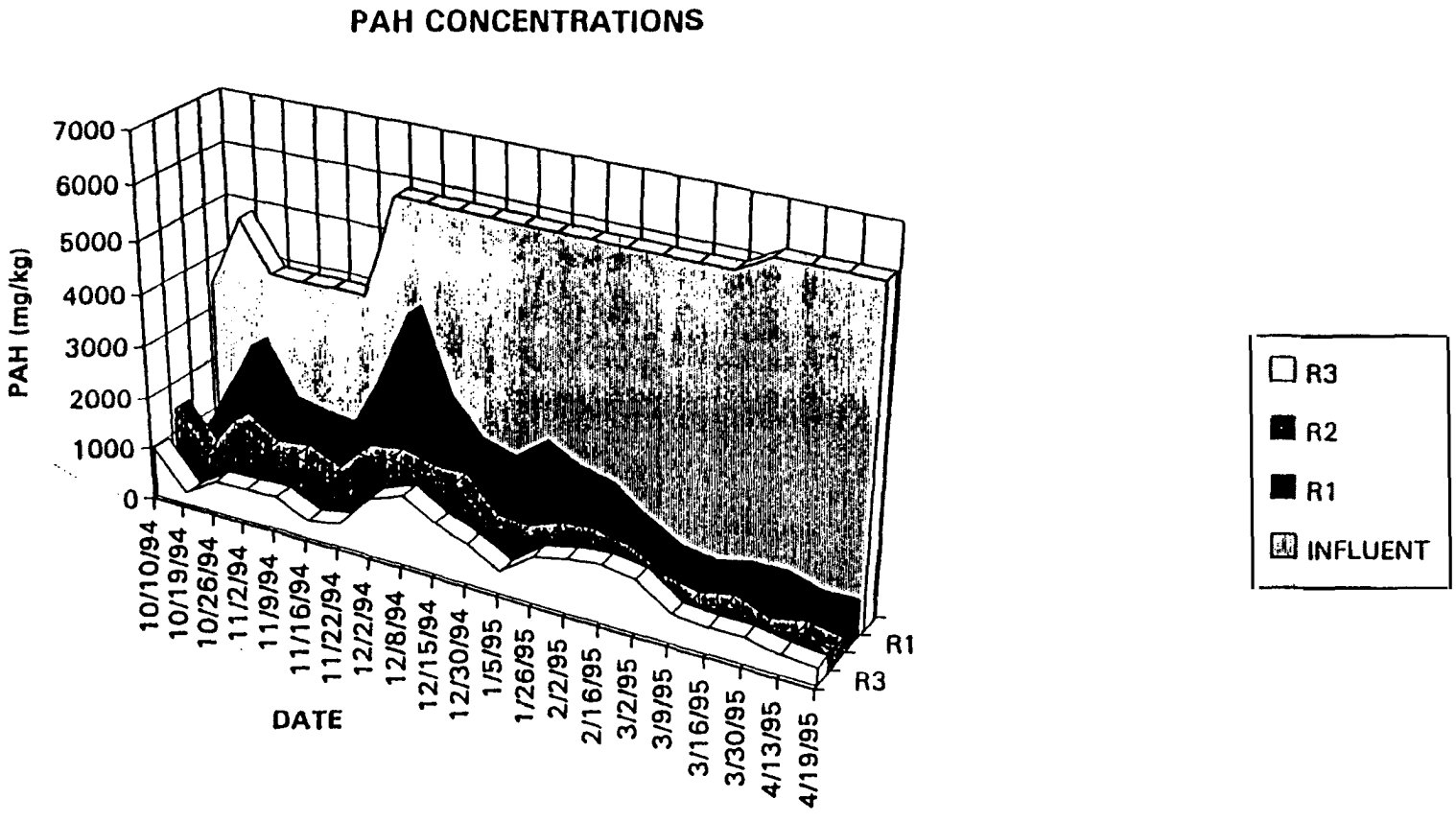
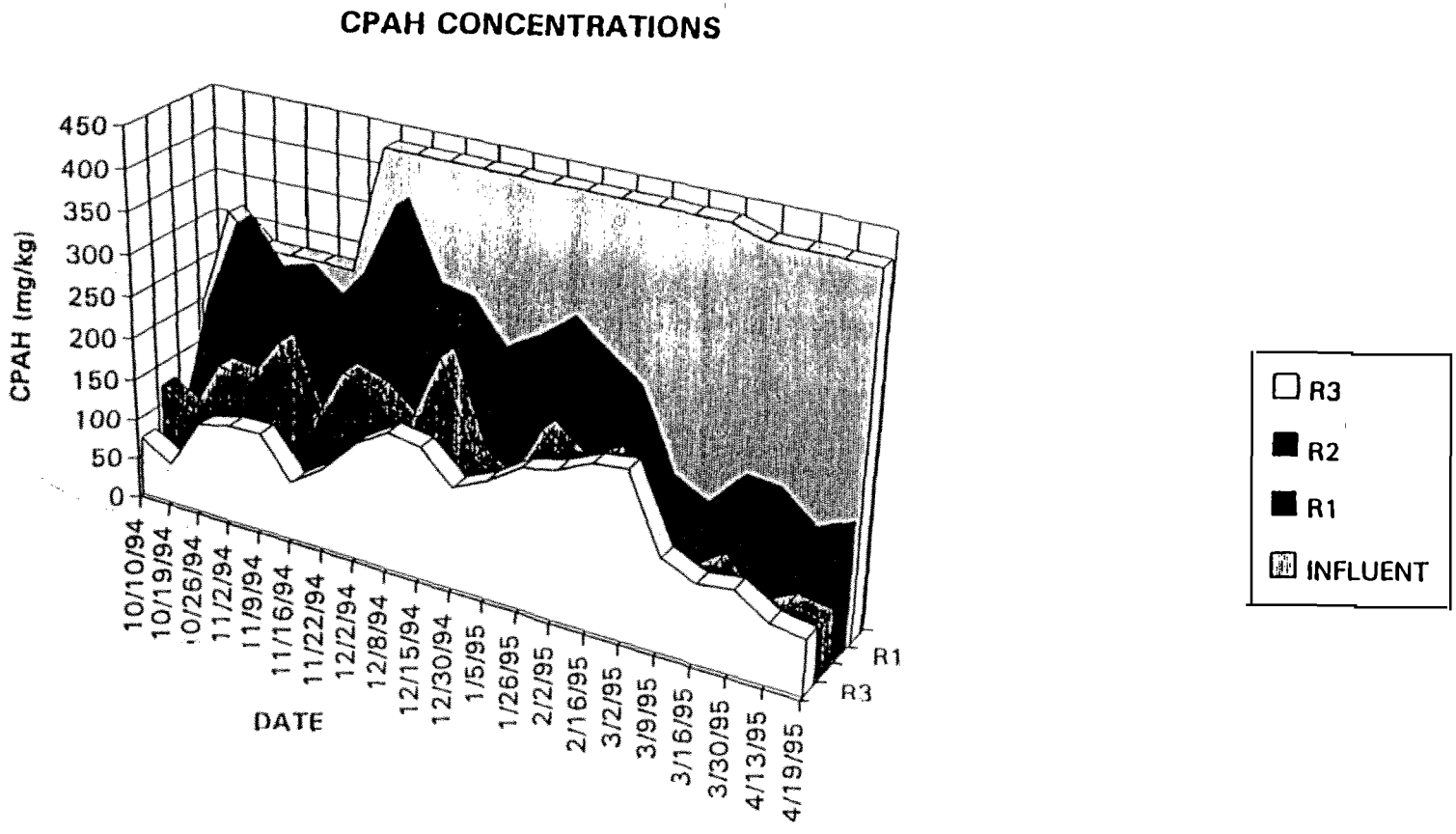

BENZO(a)PYRENE CONCENTRATIONS
BENZO(a)PYRENE (mg/kg)
35
30
25
20
15
10
5
0
10/10/94
10/19/94
10/26/94
11/2/94
11/9/94
11/16/94
11/22/94
12/2/94
12/8/94
12/15/94
12/30/94
1/5/95
1/26/95
2/2/95
2/16/95
3/2/95
3/9/95
3/16/95
3/30/95
4/13/95
4/19/95
DATE
R1
R3
R3
R2
R1
INFLUENT

Figure 4-19


PAH CONCENTRATIONS
PAH (mg/kg)
7000
6000
5000
4000
3000
2000
1000
0
10/10/94
10/19/94
10/26/94
11/2/94
11/9/94
11/16/94
11/22/94
12/2/94
12/8/94
12/15/94
12/30/94
1/5/95
1/26/95
2/2/95
2/16/95
3/2/95
3/9/95
3/16/95
3/30/95
4/13/95
4/19/95
DATE
R1
R3
R3
R2
R1
INFLUENT

# Figure 4-20


CPAH CONCENTRATIONS
CPAH (mg/kg)
450
400
350
300
250
200
150
100
50
0
10/10/94
10/19/94
0/26/94
11/2/94
11/9/94
11/16/94
11/22/94
12/2/94
12/8/94
12/15/94
12/30/94
1/5/95
1/26/95
2/2/95
2/16/95
3/2/95
3/9/95
3/16/95
3/30/95
4/13/95
4/19/95
DATE
R1
R3
R3
R2
R1
INFLUENT