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SITE
SUPERFUND INNOVATIVE
TECHNOLOGY EVALUATION

Technology Demonstration Summary

CF Systems Organics Extraction System, New Bedford Harbor, Massachusetts

The Site Program demonstration of CF Systems' organics extraction technology was conducted to obtain specific operating and cost information that could be used in evaluating the potential applicability of the technology to Superfund sites. The demonstration was conducted concurrently with pilot dredging studies managed by the U.S. Army Corps of Engineers at the New Bedford Harbor Superfund site in Massachuetts. Contaminated sediments were treated by CF Systems' Pit Cleanup Unit (PCU) that used liquified propane/butane as the extraction solvent. The PCU was a trailer-mounted system with a design capacity of 1.5 gpm (20 bbl/day). CF Systems claimed that the PCU would extract organics from contaminated soils based on solubility of organics in liquified propane/butane.

The objectives included an evaluation of (1) the unit's performance, (2) system operating conditions, (3) health and safety conditions, and (4) equipment and system materials handling problems. Extensive sampling and analyses were performed showing that polychlorinated biphenyl (PCB) extraction efficiencies of 90 percent were achieved for sediments containing PCBs ranging from 350 to 2,575 ppm. In Test 2, sediments containing 350 ppm were reduced to 40 ppm after 10 passes, or recycles, through the PCU. In Test 3, a 288 ppm feed was reduced to 82 ppm after 3 passes. In Test 4, a 2,575 ppm feed was reduced to 200 ppm after 6 passes. Some operating problems occurred, such as the intermittent retention of solids in system hardware and foaming in the treated sediment collection tanks. These problems did not affect extraction efficiency but could affect operation of a full-scale unit. Corrective measures will be addressed by the developer and EPA. A mass balance established over the entire demonstration showed excellent accountability for 96 percent of the total mass. Operation of the unit did not present any threats to the health and safety of the operators or the local community.

This Summary was developed by EPA's Risk Reduction Engineering Laboratory, Cincinnati, OH, to announce key findings of the SITE program demonstration that is fully documented in two separate reports (see ordering information at back).

Introduction

CF Systems Corp., developer of an organics extraction technology, was selected to demonstrate their system at the New Bedford Harbor, Massachusetts, Superfund site. The system demonstrated was CF Systems' Pit Cleanup Unit (PCU), a trailer-mounted system with a design capacity of 1.5 gpm (20 bbl/day). Successful application of the technology depends on the ability of organic pollutants to solubilize in the process solvent, a liquified gas. The process used a mixture of liquified propane and butane, at 240 psi and 69 degrees F, as a solvent for extracting organics from soils. As liquified solvent was mixed with the waste, organics were extracted into the solvent. The solvent-organics mixture was then decanted from the separated solids and water. The pressure of the solvent-organics mixture was reduced slightly to vaporize the solvent which allowed separation from the organics. The solvent was recovered by the system and compressed to a liquid for reuse.

The site is located on the Acushnet River Estuary north of Buzzard's Bay in the city of New Bedford, Massachusetts, where sediments contain pollutants discharged to the harbor from a variety of industrial sources. The pollutants include polychlorinated biphenyls (PCBs), polynuclear aromatic hydrocarbons, copper, chromium, zinc, and lead. PCBs present the greatest toxic threat and concentrations range up to 30,000 ppm.

The following technical criteria were used to evaluate the effectiveness of the CF Systems process for extracting PCBs from New Bedford Harbor sediments:

1. System Performance
   - Evaluate PCB concentration in sediments before and after treatment.
   - Evaluate PCB extraction efficiency with each pass, or recycle, of sediments through the unit.
   - Evaluate mass balances established for total mass, solids and PCBs.
2. Operating Conditions
   - Compare operating conditions to operating specifications for flow, temperature, pressure, and physical sediment characteristics of the sediment and assess the effect on extraction rate.
3. Health and Safety Considerations
   - Determine if significant amounts of propane/butane or PCBs are emitted to the air by the process.
   - Determine if staging area soils are contaminated by system spills or malfunctions.
   - Decontaminate the unit with toluene to levels less than 50 ppm in decontamination residues.
4. Equipment and Material Handling Problems
   - Observe equipment and material handling problems that would affect the performance of a full-scale site cleanup.

Facility and Process Description

Contaminated sediments from five harbor locations were processed by the PCU. The U.S. Army Corps of Engineers dredged sediments from the harbor and stored them in 55-gallon drums for processing by the PCU. The drummed sediments were blended to provide feedstocks for four tests. Each of the four tests was run similarly except that the number of passes and PCB concentrations were varied for each test. A pass was defined as one cycle of the feed through the PCU. A pass of feed results in a treated sediment product and an extract product. Collecting and recycling the treated sediment through the PCU constituted an additional pass. Recycling was conducted to simulate the operation of a full-scale commercial system. The PCU is only a two-stage system, whereas commercial designs include four stages, longer extractor residence times, and longer phase separation times.

1. Test 1 was run as a shakedown test to set pressure and flowrates in the PCU. The feed had a PCB concentration of 360 ppm. Three passes were run to gain experience with materials handling.
2. Ten passes were run in Test 2 to simulate a high-efficiency process and to achieve treated sediment levels less than 10 ppm. The feed had a PCB concentration of 350 ppm. A 350 ppm concentration was chosen for this test since this represents an average, or typical, PCB concentration in the harbor.
3. Test 3 was a 3-pass test that used a 288 ppm feed. The purpose of Test 3 was to reproduce the results of the first three passes of Test 2.
4. Test 4 was a 6-pass test. The purpose of this test was to reduce a high-level waste (2,575 ppm) to a lower level waste such as that used in Tests 1, 2, and 3. High-level wastes are found at several "hot spots" in the harbor.

Samples were taken of the feed at the commencement of each test. Treated sediment products and extracts were planned for sampling at each pass. Additional samples were taken of system filters and strainers, although the amount of PCB contained in these miscellaneous samples, later, proved to be small. PCU operating pressures, temperatures, and flow-rates were monitored throughout the tests. Field tests were conducted for feed viscosity, pH, and temperature. Decontamination of the system involved running toluene through the PCU as a solvent wash.

The PCU is a continuous processing unit that used a liquified propane/butane mix as the extraction solvent. The solvent mix was 70-percent propane and 30-percent butane. The PCU process flow diagram is shown in Figure 1. For each of the 3 demonstration tests, a batch of approximately 50 gallons of sediments was fed to the unit at a nominal rate of 0.9 gpm. Feed viscosity was maintained below 1,000 cp, by adding water in order to produce a pumpable slurry. Particles greater than one-eighth inch were screened from the feed to prevent damage to valves. Sediments were pumped to the extractors, which were typically operated at 240 psig and 70 degrees F. Liquified solvent was also pumped to the extractors at a rate of 2.3 gpm (10 lb/min) and mixed with the sediments.

The PCU was not designed for large-scale remedial actions. Therefore, treated sediments were recycled, or passed through the unit to simulate operation of a commercial-scale unit. CF Systems' commercial-scale designs do not include recycling. These designs feature 60 gpm flowrates, several extraction stages, and longer processing times.

The process steps included extraction, phase separation and solvent recovery. A simplified flowchart is shown in Figure 1. In step one, sediments were fed into the top of an extractor at a rate of 0.9 gpm. In step two, solvent was compressed to a liquid state and allowed to flow through the same extractor. In the extractor, the solvent was thoroughly mixed with the waste at a pressure of 240 psig. Following this extraction procedure, the residual mixture of water/solids was removed from the base of the extractor (step three). In step four, the mixture of solvent and organics left the top of the extractor and was expanded across a valve prior to passing to a separator. The reduction in pressure caused the solvent

Simplified Flow Chart

Here is the CF Systems unit operating cycle, for extracting and separating organics from liquid or solid waste:

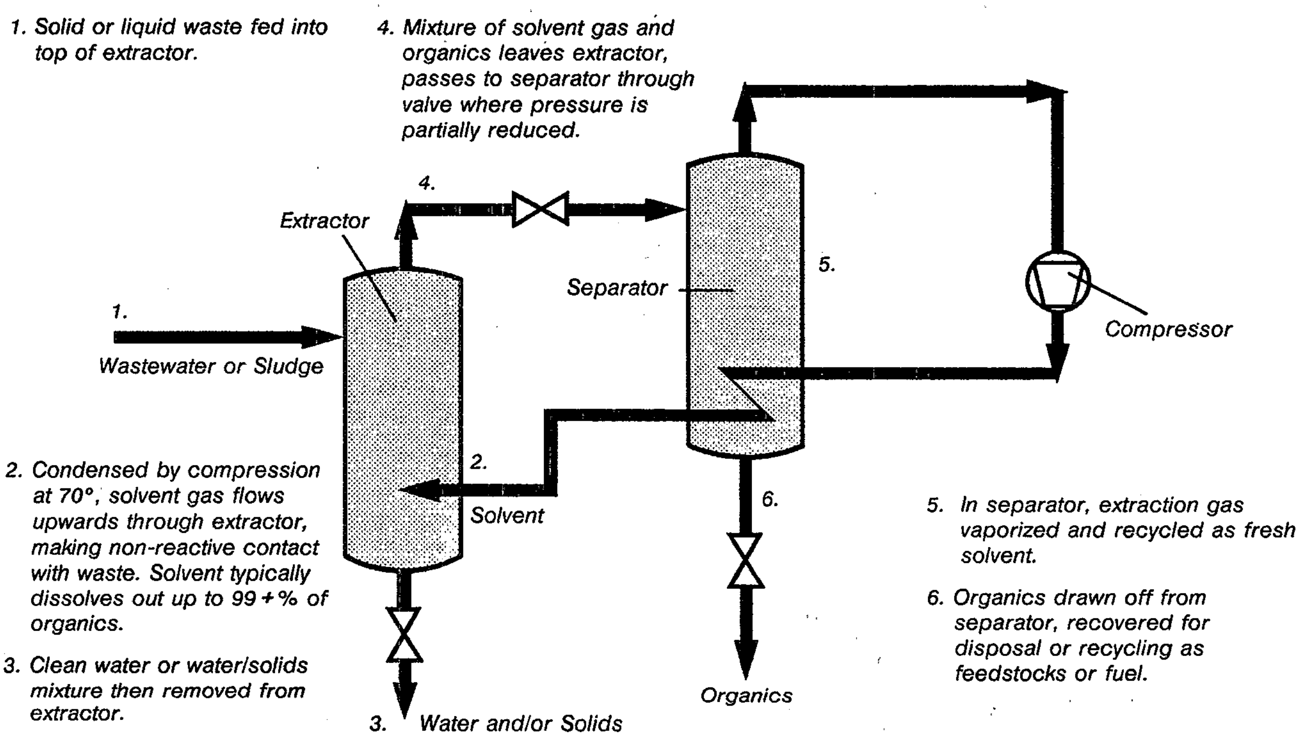

***Figure 1.** CF Systems organics extraction simplified flow chart.*

to vaporize through the top of the separator. It was then collected and recycled through the compressor as fresh solvent (step five). The liquid organics left behind were drawn off the separator and pumped to storage (step six). About 1 to 2 hours were required to run a feedstock through the PCU.

Results and Discussion

The program obtained a large amount of analytical and operating data for evaluating the effectiveness of the PCU for extracting PCBs from New Bedford Harbor sediments. The results are summarized below.

System Performance

The performance of the treatment unit was evaluated in terms of extraction efficiency and a mass balance. Extraction efficiency per pass is defined as the input PCB concentration minus the output PCB concentration divided by the input PCB concentration (multiplied by 100 percent). An inventory of system inputs and outputs was established and evaluated for total mass, total solids, and the total mass of PCBs.

PCB analyses for feed sediments and treated sediment, conducted for samples collected at each pass, are shown in Figures 2, 3, and 4. The data show that treated sediment concentrations below 10 ppm are achievable and that as much as 84 percent of the PCB contained a sediment that can be removed in a single pass. In Test 2, feed containing 350 ppm of PCB was reduced to 40 ppm after 10 passes through the PCU. In Test 3, a 288 ppm feed was reduced to 82 ppm after 3 passes. In Test 4, a 2,575 ppm feed was reduced to 200 ppm after 6 passes.

The data for each test show general reduction trends based on differences between initial feed and final treated sediment concentrations. However, these trends are not consistent on a pass-by-pass basis. For example, PCB concentrations in treated sediments increase at Test 2, passes 4 and 10, and at Test 3, passes 2 and 3. These anomolies are not related to the extraction process. Instead, they reflect cross-contamination within system hardware. Only 50 to 150 gallons per day were run through the unit, which was designed to handle up to 2,160 gallons per day. Therefore, some solids may have been retained in equipment dead spaces and intermittently discharged at later passes. Since the treated sediment collection tanks were under pressure, it was not possible to clean out collection hardware and piping.

Extraction efficiencies greater than 60 percent were achieved on the first pass of each test. Later passes, or recycles, of treated sediments through the unit resulted in efficiencies that ranged from

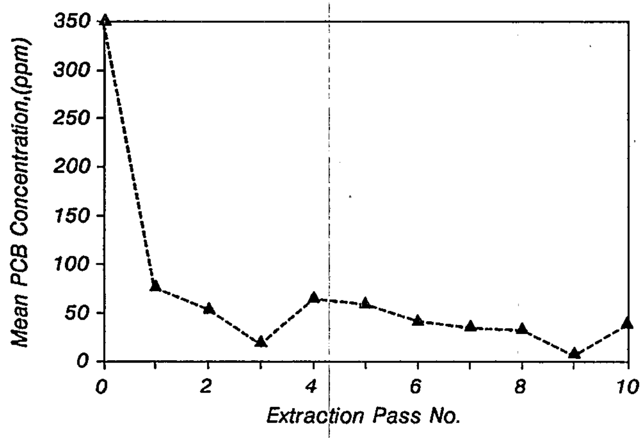

Figure 2. *Test PCB reduction.*

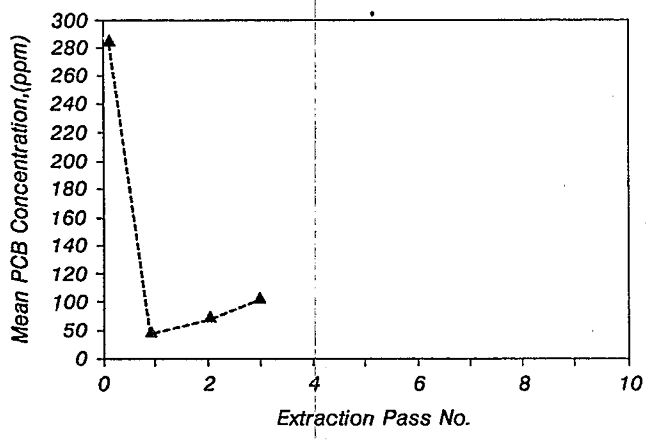

Figure 3. *Test 3 PCB Reduction.*

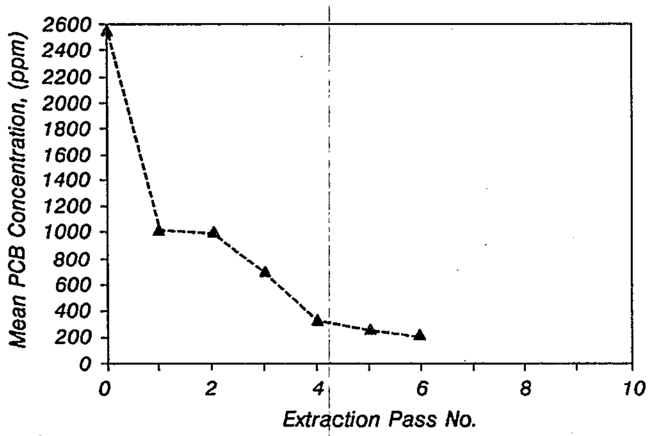

Figure 4. *Test 4 PCB Reduction.*

zero to 84 percent. This wide range was due to solids retention in the system. Solids retained in the system cross-contaminated treated sediments that were recycled. Recycling was necessary to simulate the peformance of a full-scale commercial system. CF Systems' full-scale designs do not include recycling since more extraction stages and longer processing times are involved.

A good mass balance was established for total mass and solids through the system. A total of 3-1/2 tons of solids and water were fed to the unit during Tests 2, 3, and 4. Of the total, 96 percent was accounted for in effluent streams. A total of 789 pounds of solids were processed during Tests 2, 3, and 4. Of the total, 93 percent was accounted for in effluent streams. The slight imbalances, 4 and 7 percent, are attributed to the inaccuracy of the weighing device (1 percent), sample error, and accumulation of mass in system hardware.

A mass balance was not established for PCBs. A total of 157 grams were fed to the unit during system shakedown and Tests 2, 3, and 4. Of the total, 80 grams were accounted for in system effluents. Decontamination washes produced an additional 169 grams. The sum of effluents and decontamination washes was, therefore, 101 grams greater than that fed to the unit. This large difference may be due, in part, to limitations of the analytical method. PCB analytical Method 8080 precision criteria established for this project were plus or minus 20 percent and accuracy criteria were plus or minus 50 percent. In addition, the mass balance calculation was dominated by the Test 4 feed concentration. Therefore, error associated with the Test 4 feed sample could also be a source of the PCB mass imbalance. Another possibility is contamination of the PCU from prior use at other sites.

Metals were not expected to be removed from the sediments, and were not removed during the extraction. EP Tox test results indicate that metals did not leach from either treated or untreated sediments. Characteristics of the sediments, with respect to the EP Tox test, were not changed by the treatment process.

The decontamination procedure showed that PCBs were separated from the sediment during the tests since nearly all, 88 percent, of the PCBs were contained in extract subsystem hardware. Of the 81 grams of PCB fed to the unit during Tests 2, 3, and 4, only 4 grams remained in the final treated sediments. This indicated an overall PCB separation efficiency of 95 percent. Subsequent decontamination of the PCU with a toluene wash showed that some PCB had accumulated in system hardware. However, 91 percent of the PCBs contained in decontamination residues were found in extract subsystem hardware.

Operating Conditions

Operating conditions that were essential to the efficient performance of the PCU were manually controlled and monitored during Tests 2, 3, and 4. These included (1) feed temperature, particle size, flow rate, pH, and solids content, (2) solvent flow rate and solvent/feed mass ratio, and (3) extractor pressure and temperature. The unit generally performed as predicted by the developer, although some deviations from the planned specifications occurred.

During Test 2, feed temperatures for the last 4 passes were 10 degrees F lower than the minimum specification, 60 degrees F. This may have contributed to decreased extraction efficiency that was apparent during this test. Sustained low temperatures could have the effect of seriously reducing extraction efficiency of a full-scale commercial system.

Solvent flow fluctuated as much as 75 percent above and below the nominal flow rate, 12 lb/min. This may have affected the solvent-to-feed ratio in Test 2 Pass 1. Low solvent-to-feed ratios could directly affect extraction efficiency in a full-scale system, since less solvent would be available to extract organic pollutants from the feed soil.

Specifications for maximum particle size, one-eighth inch, were met by sieving sediments through a screen. This was necessary to prevent damage to system valves. Less than 1 percent of the sediment particles were greater than the one-eighth inch.

Specifications for maximum viscosity, 1,000 centipoise, were met by adding water to form a pumpable feed mixture. Feed viscosities ranged from 25 to 180 centipoise. However, added water increased the mass of waste by about 33 percent.

Solids contents ranged from 6 to 23 percent and fell below the minimum specification, 10 percent, after the fourth pass of Tests 2 and 4. A 10-percent minimum spec was set merely to ensure that the technology would be demonstrated for high solids content feeds.

Health and Safety Considerations

The Health and Safety Plan established procedures and policies to protect workers and the public from potential hazards during the demonstration. Air emissions from the unit did not affect operating personnel or the local community. Combustible gas meters indicated that the unit did not leak significant amounts of propane. Therefore, operation of the unit does not present an explosion threat much different than that associated with domestic propane usage. Background air sampling and personnel monitoring results indicate that organic vapors and PCB levels were present at levels below the detection limit for the analytical methods. The unit did not cause a sudden release of propane/butane or liquids. Only minor leaks occurred and staging area soils were not affected. The treated sediment subsystem was successfully decontaminated before leaving the site. The extract subsystem was decontaminated with toluene, however, the decontamination goal of 50 ppm was not achieved since the final wash contained 60 ppm of PCB.

Equipment and Material Handling Problems

Equipment and material handling problems occurred throughout the demonstration. While these problems did not impede achievement of the developer's treatment goals, they could impact the economic performance of a full-scale commercial system. Some problems were anticipated since relatively small volumes of sediments were batch-fed to a unit that was designed for continuous operation. The nominal capacity of the unit is 2,160 gallons per day, but only 50 to 100 gallons per day were batch-fed during shakedown and Tests 2, 3, and 4. Consequently, the unit intermittently discharged and retained solids with each pass.

Previous use of the unit affected interpretation of semivolatiles data. Internal surfaces of extract collection hardware collected PCBs as evidenced by mass balances. In addition, Test 3 was interrupted and viscous oils were found accumulating in extract subsystem hardware. PCBs are soluble in oil, which coated the internal surfaces of system hardware. As a result of this demonstration, CF Systems now requires more rigorous decontamination procedures for the PCU.

Solids were observed in extract samples that were expected to be solids-free. This indicates poor performance or failure of the pleated paper cartridge filter. An alternative type of filter should be investigated by the developer.

Low-pressure dissolved propane caused foaming to occur in the treated sediment product tanks. This hindered sample collection and caused frequent overflow of treated sediment to a secondary treated sediment product tank. CF Systems states that design of a commercial-scale unit will allow release of propane entrained in the treated sediment and elimination of the foaming problem.

Two analysis methods for PCBs were used and results were compared. Reviewers suggested the use of EPA Method 680, since the CF Systems technology could have selectively extracted higher molecular weight PCB congeners as opposed to lower weight PCB congeners. Method 680 would reveal any selective extraction, since Method 680 is used to analyze individual PCB congeners. Method 8080, a less expensive analysis method, would not reveal selective extraction since it is used to analyze mixtures of PCBs called Alaclors, instead of individual congeners. EPA Method 8080 was chosen over Method 680 since selective extraction was minor and since it analyzes for the classes of congeners that compose the majority of PCB contaminants (Aroclors 1242 and 1254) in the harbor sediments.

Methods 680 and 8080 produced similar relative results, but very different absolute results. Use of Method 680 in Test 4 showed a PCB extraction efficiency of 96 percent and Method 8080 showed a similar efficiency, 87 percent. However, Method 680 showed an untreated sediment PCB concentration of 8,700 ppm while Method 8080 showed 2,575 ppm. Data quality objectives were met for each measurement.

Conclusions and Recommendations

Based on the above data and discussions, the following conclusions and recommendations can be made concerning the operation and performance of the CF Systems organics extraction process.

1. Even though solids retention caused cross-contamination of treated sediments, significant PCB removal occurred. For example, in Test 2 after Pass 9, treated sediments contained 8 ppm of PCB.Compared with a Test 2 feed concentration of 350 ppm, this represents an extraction efficiency of 98 percent.
2. System decontaminated procedures showed that PCBs were separated from the sediment since nearly all, 88 percent, of the PCBs were contained in extract subsystem hardware. Of the 81 grams of PCB fed to the unit duing Tests 2, 3, and 4, only 4 grams remained in the final treated sediments. This indicates an overall PCB separation efficiency of 95 percent.
3. Bench-scale tests are useful for determining whether or not organics contained in a soil will be extracted by a liquified solvent such as a propane-butane mixture. Bench-scale tests may also be used to determine if a liquified solvent selectively extracts specific classes of organica such as high or low molecular weight PCBs. Bench-scale tests, however, do not yield information relating to operational and material handling issues such a pumpability, foaming, and temperature, for example.
4. Commercial-scale designs for application of the technology should ensure that operating specifications are maintained. Wide fluctuations in the feed-to-solvent ratio should be minimized, since extraction efficiency may be directly related to this parameter.
5. Feed materials are likely to be well below 60 degrees F throughout winter months and this could affect performance. Therefore, heat must be added to sediments fed to a commercial-scale unit.
6. Pretreatment technology will be required to condition feed materials. Coarse solids removal will be required to maintain feed sediment particle sizes below one-eighth inch and water must be added to ensure pumpability.
7. Health and safety monitoring showed that OSHA level B protection will be necessary for personnel that will handle input and output. However, only OSHA level C protection will be necessary for unit operators.
8. Regulatory or engineering interpretation of PCB analysis should include consideration of the analysis methods used.
9. Operations, materials handling, and safety issues are addressed in the Application Analysis Report. Costs are estimated for several cases involving the New Bedford Harbor Superfund site. A significant cost element for a full-scale system is extraction process equipment which must be scaled to handle much higher throughputs (60 gpm) than the PCU (0.9pgm). Full-scale extractors have 4 to 6 foot diameters as compared with the 18 inch diameter of the PCU extractors. Recommended treatment technology includes conveyors, screening, heat and water addition, mixing and holding tanks. Post treatment technology includes treated sediment dewatering, wastewater treatment and reuse, holding tanks, conveyors and disposal of treated sediments and extracted organics. Onsite analytical capabilities and health and safety program implementation are additional cost elements.
10. EPA and the developer will address corrective measures for operational controls and material handling issues. However, these measures are not the subject of this report.

*The EPA Project Manager, **Richard Valentinetti**, was with the Risk Reduction Engineering Laboratory, Cincinnati, OH 45268 (see below).*

The complete report consists of two volumes, entitled "Technology Evaluation Report, SITE Program Demonstration Test, CF Systems Organics Extraction System, New Bedford Harbor, Massachusetts."

"Volume I (Order No. PB 90-186 495/AS; Cost: $21.95, subject to change) discusses the results of the SITE demonstration

"Volume II (Order No. PB 90-116 024/AS;Cost: $42.95, subject to change) contains the technical operating data logs, the sampling and analytical report, and the quality assurance project plan/test plan

These two reports will be available only from:

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
Telephone: 703-487-4650

A related report, entitled "Applications Analysis Report; CF Systems Organics Extraction System" which discusses application and costs, is under development.

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