

## **APPENDIX Y**

### **Summary of Pilot Study No. 3 Treatability Study (Phase IX)**

## APPENDIX Y

### SUMMARY OF PILOT STUDY NO. 3 TREATABILITY STUDY (Phase IX)

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Y3	Analytical Reports (Del Mar Analytical and Conti Testing Laboratories)

## APPENDIX Y

### SUMMARY OF PILOT STUDY NO. 3 TREATABILITY STUDY (Phase IX)

#### Y.1 Introduction

Treatability and pilot studies performed on impacted materials from the Ascon Landfill Site ("Site") can be divided between those conducted during the initial FS effort and those conducted during the recent Pilot Study No. 3 field activities (Phase IX). **Table X-1** of Appendix X of the RFS summarizes the parameters and results of the initial FS treatability and pilot studies. Detailed descriptions of these studies are presented in Appendix X of the RFS, which summarizes the presentation in the initial FS Chapters 8 and 9 (Environ, 2000). The Phase IX testing consisted of evaluating the performance of *Ex situ* Chemical Oxidation and Sludge Liquification on various waste types to reduce odors and emissions and render wastes pumpable, respectively.

#### Y.2 Pilot Study No. 3 Phase IX: Technology Bench Scale Testing

##### Y.2.1 Background

On October 15, 2004, PNL submitted on behalf of the RPs a technology bench scale testing workplan as an addendum to the Pilot Study No. 3 Workplan, referred to herein as the Phase IX Addendum and attached as Attachment Y1. Phases I through VIII of Pilot Study No. 3, as described in Section 3 of the RFS, focused on data collection to characterize the wastes present in specific areas of the Site and to assess emissions from these wastes when exposed. During the Phases I through VIII work, it was discovered that three waste types--impacted soils, drilling muds, and lagoon tarry liquids (found in Lagoons 1, 2, and 3)--present unique challenges for handling during excavation and removal activities associated with any contemplated remedial action program at the Site. These challenges, as defined in the Phase IX Addendum, are as follows:

- Drilling muds and impacted soils emit VOC (above 50 ppm) and odor emissions when disturbed during excavation. SCAQMD Rule 1166 specifies that materials that emit VOCs at or greater than 50 ppm require treatment (by an approved method) unless disposed of offsite at an appropriate disposal facility within 30 days of excavation.
- The lagoon tarry liquids and highly liquid drilling muds present in portions of the lagoons are semi-solid and may flow under their own weight. Liquification and pumping the lagoon tarry liquids and drilling muds may be an attractive alternative to *in situ* stabilization and/or standard excavation techniques to remove these materials.

Therefore, Phase IX of the Pilot Study No. 3 program was developed to gather data on the bench-scale performance of a few promising technologies that might assist in improving material handling efficiency and controlling VOC emissions of these materials during remedial activities at the Site. These technologies included:

- *Ex situ* Chemical Oxidation and *Ex situ* Thermal Desorption (evaluated qualitatively – see Section 8.2.4) to reduce TPH and VOCs in drilling muds and impacted soil, and
- Additives to enhance the pumpability of lagoon tarry liquids and drilling muds.

PNL conducted the treatability studies in cooperation with experienced technology vendors/suppliers and treatability testing subcontractors. The treatability studies were performed using the guidelines outlined in the U. S. Environmental Protection Agency (EPA) document "Guidance for Conducting Treatability Studies under CERCLA, EPA/540/R-92/071a, October 1992."

### Y.2.2 Objectives

The overall objectives for Phase IX were as follows:

Evaluate the effectiveness of *Ex situ* Chemical Oxidation treatment to reduce emissions (< 50 ppm) and TPH concentrations (below relevant standards) in impacted soils and drilling muds, because this technology may potentially be more cost effective than *Ex situ* Thermal Desorption treatment.

Explore the potential use of additives to facilitate pumping the lagoon tarry liquids and drilling muds without generating emissions above regulatory limits. Preliminary discussions and testing with vendors indicated it was feasible to liquify the drilling muds and lagoon tarry liquids by adding a 20% solution of an organic, water-based solvent. Chemical and physical testing needed to confirm the suitability of this material for handling and waste disposal.

### Y.2.3 Description of Waste Types

#### *Impacted Soil*

Impacted soil, which consists of both imported fill and native soil, appears to be impacted with TPH and metals (e.g., lead) from mixing with the drilling muds and lagoon materials during placement and subsequent movement of the waste and redistribution of fill materials over the Site during the operational life of the landfill. Based on the Pilot Study No. 3 fieldwork and EA activities, impacted soils exhibit the potential for VOC emissions greater than 50 ppm. The ability of *Ex situ* Chemical Oxidation treatment to mitigate emissions and remediate the hydrocarbons in impacted soil was evaluated in Phase IX.

#### *Drilling Muds*

Based on the Pilot Study No. 3 fieldwork and EA activities, because of the fine-grained, clayey nature of some of these materials, drilling muds may retain levels of VOCs that exceed the SCAQMD limit that could be released when these materials are disturbed. In addition, due to the semi-solid consistency of the drilling muds, the potential addition of amendments to further liquify the drilling muds so they could be removed by pumping was also explored. As discussed in Chapter 3 and the Phase IX addendum, the drilling muds found in the lagoon and former lagoon areas have distinct physical and chemical properties. The lagoon drilling muds are generally softer and have a relatively low strength compared to the drilling muds found in the former lagoon areas, though in many areas of Lagoons 4 and 5, the materials were relatively stiff and stood up during excavation during EA activities. The lagoon drilling muds also have TPH concentrations one to two orders of magnitude higher than those found in other areas of the Site. In contrast, drilling muds found in the former lagoons areas are generally stiff, relatively dry (unsaturated), and have a relatively high strength. Due to these distinct characteristics, both of these types of drilling muds were evaluated in both the emissions/hydrocarbons treatment and liquification testing during Phase IX.

#### *Lagoon Tarry Liquids*

Based on the Pilot Study No. 3 fieldwork, lagoon tarry liquids do not generate significant emissions when disturbed, but they flow under their own weight and could present a significant challenge for excavation. Excavation as a "solid" material may require pre-stabilization with soil or other amendments such as Portland Cement or Fly Ash applied *in situ* using special mechanical equipment. Since removal of semi-solid materials by pumping could be more efficient than conventional excavation and mixing, the potential of using additives to liquify the lagoon tarry liquids to facilitate pumping was evaluated during Phase IX. Additional characteristics of the lagoon tarry liquids are described in the Phase IX Addendum and Section 3.2.2 of this RFS.

## Y.2.4 Treatment Technology Descriptions

This section describes two candidate technologies—*Ex situ* Chemical Oxidation and Sludge Liquification--to improve the handling efficiency and/or mitigate the emissions associated with impacted soils, drilling muds, and lagoon tarry liquids. *Ex situ* Chemical Oxidation may also remediate petroleum hydrocarbons impacting these waste materials to levels below local and state standards. *Ex situ* Chemical Oxidation is a potentially more cost effective alternative to *Ex situ* Thermal Desorption for remediation of impacted soils and drilling muds to reduce TPH levels and emissions.

Results of a qualitative study of the *Ex situ* Thermal Desorption technology are presented in the Phase IX Addendum (Attachment Y1). This study determined that *Ex Situ* Thermal Desorption is a mature and effective technology for remediation of impacted soils and drilling muds, with unit treatment costs expected to range from \$70 to \$100 per ton.

### Y.2.4.1 *Ex Situ* Chemical Oxidation

*Ex situ* Chemical Oxidation is less widely implemented than *Ex situ* Thermal Desorption for remediation of impacted soils and drilling muds to reduce TPH levels and emissions. *Ex situ* Chemical Oxidation may be potentially more cost effective than *Ex situ* Thermal Desorption for a number of reasons: It does not require a heat source for treatment or product stream cooling; there are no liquid wastes (condensed product) to manage; and it is less energy intensive due to its mechanical simplicity.

In the *Ex situ* Chemical Oxidation process, a strong chemical oxidant such as permanganate in a water solution is mixed with the waste material for a period of time sufficient for the components to react. The mixture then cures over a period of hours to days as required to reduce the target contaminant(s) to design levels. If the process is effective, the target contaminant(s) are generally oxidized to carbon dioxide and water. The reactions are slightly exothermic, but generally do not impact the handling of the treated product (i.e., a minimal increase in temperature is anticipated).

In Phase IX, a third-party vendor, Environmental Technology Solutions' (ETS) accelerated *Ex situ* Chemical Oxidation process was tested. In ETS' approach, hydrocarbons in the waste matrix are oxidized by reacting the waste with an ionized water solution containing hydroxyl free radicals and permanganate using a proprietary reaction process. **Figure Y.2-1** shows the generalized treatment approach utilizing this process for treatment of hydrocarbon-impacted soil waste.

The process, as simulated in the Phase IX bench testing, is as follows. First, soil waste is screened as required to remove materials greater than one-inch size and/or excessive moisture. The soil waste is fed into a pug mill reactor or other *ex situ* mixer, where the process reagents are introduced. Mixing takes place typically for a few minutes, after which the waste is removed for curing for approximately 2 to 7 days depending on the design parameters.

The scope of the *Ex situ* Chemical Oxidation treatability study also included testing of ETS' proprietary reagent blend, Odor Pro®, to reduce odors and VOC emissions of the impacted soil and drilling mud samples submitted for remediation by ETS' *Ex situ* Chemical Oxidation process. The scope of this study was described in the Phase IX Addendum and involved applying the reagent to the waste, measuring the VOC emissions of the waste with a photoionization detector (PID) before and after addition (and repeating this process), and qualitatively assessing the odors before and after treatment.

#### Y.2.4.2 Sludge Liquification

As described in the initial FS, J&W documented bench and pilot scale testing of an *Ex situ* Solvent Extraction approach using a hot water bath and proprietary surfactants to evaluate the pumpability of the tarry liquids in Lagoons 1 and 2 and to determine if any recoverable oil could be separated from the tars. Although the process appeared promising for liquifying the tars and phase separation, there were several drawbacks that inhibited the effectiveness of the process. Notable problems included the large quantity of water required, product heating and emissions, and cross-contamination of the separated phases. These adverse features led to the elimination of *Ex situ* Solvent Extraction as a viable process option during preliminary screening of this RFS (Section 8.5.2.3). In addition, no testing was performed on pumpability of the drilling muds.

Prior to developing the Phase IX Workplan, PNL identified a technology vendor, Petromax Technologies (Los Angeles, California) with experience in fluidizing processes to facilitate cleanup of crude oil tank bottoms. Petromax subsequently identified processes and products that may be applicable for emulsion breaking, phase separation and product recovery of lagoon tarry liquids and drilling muds at the Site through pre-test qualitative analysis. The feasibility of oil recovery from the tars/muds was expected to require additional products and time for evaluation and was beyond the scope of the test.

Petromax's products identified for application to the Site waste materials work by encapsulating and permanently modifying the opposing surface charges between hydrocarbon molecules and the inorganic solid particles to which they adhere. This process allows the hydrocarbons to flow freely in solution. The surface modification is accomplished by shearing the hydrocarbons from the inorganic particles. Treatment can be achieved by hydroblasting (at 3,000 to 5,000 psi or greater) Petromax's formulas into the waste material or by high-shear mixing<sup>1</sup> the waste while simultaneously injecting the product either *in situ* or *ex situ*. Several slides describing the theoretical mechanism of action for sludge fluidization utilizing Petromax's products are presented as **Figures Y.2-2a** through **f**.

On a bench scale, Petromax's products are mixed into the waste using a handheld electric mixer (see Attachment Y1). Mixing takes place until sufficient time has elapsed for the formula to contact with the inorganic soil particles/hydrocarbons to modify the surface charges and to create a permanent suspension. Two separate products were developed for testing the lagoon tars and drilling muds.

Subsequent to submitting the draft Phase IX Addendum, a second vendor was identified, Texas Envirochem Group, L.P. (TEC), as a marketer of products for liquifying and biotreating petroleum sludges and tars. On December 1, 2004, the RPs submitted their response to DTSC's comments (RTC) on the draft Phase IX Addendum report. In this RTC, the RPs proposed testing two of TEC's products on the drilling muds and lagoon tars following a similar protocol (see below) to the testing of Petromax's products. Specifically, testing using two TEC products was recommended:

1. For the drilling muds in the lagoons and former lagoon areas: Tx Chem HE-1000, a synthetic, water soluble, biodegradable, nontoxic, nonflammable surfactant. At an estimated application rate of only 1 to 1.5 gallons per cubic yard of waste material, this product works by emulsifying hydrocarbon contaminants and suspending them in microscopic droplets. The biodegradation feature of this product was not tested.
2. For the lagoon tarry liquids: Envirochem Asphaltic Crude Liquifier (ACL), an amber liquid containing terpene hydrocarbons, applied at a ratio of about 3 to 6% of the waste volume. Due to the volatile nature of ACL, small amounts of the HE-1000 product are added to waste streams treated with ACL to control emissions.

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<sup>1</sup> In high shear mixing/pumping, solid materials are mechanically shredded into smaller pieces by a sharp-edged propeller or impeller or in some other manner by the mixing apparatus. As the materials are shredded, they are dispersed throughout the liquifying formula, which improves contacting with the active ingredients in the formula. In hydroblasting, the high pressure water shears hydrocarbons from the soil or clay particles they coat, which allows penetration of the formula.

Texas Envirochem's products were to be applied and mixed into the waste in a similar manner as Petromax's (i.e., using a small mixer) for the bench test. On a field scale, pump suction from a Guzzler industrial vacuum unit would be utilized to generate the necessary agitation to mix the product with waste materials.

## **Y.2.5      Treatability Testing Approach**

### **Y.2.5.1      General Approach**

The Phase IX bench-scale treatability study was designed to evaluate performance of *Ex Situ* Chemical Oxidation and Sludge Fluidization to meet the following test objectives for possible full-scale application at the Site:

Evaluate the effectiveness of *Ex situ* Chemical Oxidation treatment in reducing VOC emissions (< 50 ppm) and TPH concentrations (to below relevant standards) in TPH-impacted soils and drilling muds to facilitate replacement of these materials at the Site.

Determine the potential for additives to be used to facilitate pumping lagoon tarry liquids and drilling muds and the suitability of this material for waste disposal, in terms of contaminant concentrations and fuel (BTU) content.

Key elements of the treatability study approach are described below. The *Ex situ* Chemical Oxidation tests were performed in the vendor's designated laboratory. The sludge fluidization technology tests were conducted at the Site with supervision from PNL personnel. **Table Y.2-1** presents a summary of the tests and key information for conducting the tests such as vendors, sample locations (note: some locations changed), chemical/physical analyses, and laboratory names.

**Samples of Waste Materials:** Samples of waste material collected from Lagoons 1 to 5 and the former lagoon areas during Phases I through VIII of Pilot Study No. 3 were placed in sealed 55-gallon drums that were located in an onsite drum storage area. Samples were collected from the drums for conducting the bench tests according to the following steps:

- The drums containing the target materials for treatability testing during Phase IX were retrieved from the onsite drum storage area.
- The drums were opened, and a pre-determined number of 5-gallon buckets were filled with the selected material using shovels and/or hand trowels.
- After sampling, the 55-gallon drums were re-sealed and returned to the onsite drum storage area.
- The 5-gallon bucket samples were properly labeled and sealed with chain-of-custody labels. As appropriate, split samples were collected from the 5-gallon buckets and shipped along with chain-of-custody documentation to the selected laboratories for pre-treatment waste characterization testing/bench testing.
- Some 5-gallon bucket samples were retained for onsite bench testing. Following testing, the buckets were sealed and placed into sealed 55-gallon drums.
- All non-disposable implements used in collecting the samples from a specific drum were decontaminated with a Liquinox® solution prior to collecting additional samples.
- The field workers adhered to the health and safety plan included as Appendix B in the Pilot Study No. 3 Workplan during the drum sampling.

**Pre-Treatment Waste Characterization:** Multiple sample splits were collected from the 5-gallon buckets as appropriate and shipped to the selected laboratories (see **Table Y.2-1**) for pre-treatment waste characterization analyses. These analyses were conducted to provide a baseline to assess the



effectiveness of the *In situ* Chemical Oxidation process. Pre-treatment samples were not collected for the sludge fluidization tests. The pre-treatment test parameters that were considered for *Ex situ* Chemical Oxidation are presented in **Table Y.2-2**. The analytical testing laboratories used during the previous Pilot Study No. 3 analyses were also used for chemical analyses of untreated and treated waste materials in the Phase IX bench studies.

**Sample Locations:** Sample locations are listed in **Table Y.2-1** and are shown on **Figure Y.2-3**. Sample locations for the drilling muds and impacted soils were chosen based on emissions data collected during Pilot Study No. 3, subject to field verification of representativeness.

**Treatability Testing:** Vendors or vendor-selected third-party treatability laboratories performed the treatability tests. The test parameters for the *Ex situ* Chemical Oxidation and Sludge Fluidization tests are shown in **Tables Y.2-2** and **Y.2-3**, respectively.

**Treatment Product Characterization:** Samples of treated materials and any by-products were collected for physical and chemical testing at laboratories shown in **Table Y.2-1**. These tests provided an indication of the effectiveness of the treatment approach to meet the specific goals for each of the materials tested. The test parameters for *Ex situ* Chemical Oxidation and Sludge Fluidization for post-test characterization are presented in **Table Y.2-2** and **Table Y.2-3**, respectively.

**Verification Data Analysis, and Interpretation:** Upon completion of the treatability studies, the data generated were analyzed, and an interpretation of the data was provided based on the treatability study objectives.

#### **Y.2.5.2 Test Design and Parameters**

To collect data that would assess the treatment options, analytical tests were identified and were completed during Phase IX. These tests are categorized into three areas:

- Pre-Treatment Waste Characterization,
- Characterization During Treatment, and
- Treatment Product Characterization.

A list of test parameters for the *Ex Situ* Chemical Oxidation and Sludge Fluidization treatability tests for each of these three categories are presented in **Tables Y.2-2** and **Y.2-3**, respectively.

#### **Y.2.5.3 Test Protocols**

The procedures outlined in the Phase IX Addendum were generally followed during the bench testing program except as noted below. These procedures also include an overview of the technology and purpose of the test and a list of the materials and equipment that were used (see Attachment Y1).

#### **Y.2.5.4 Residual Materials Management**

Residual materials consisting of unused waste samples, spent reagents and formulas, and product from the treatment process generated from the treatability studies were disposed of by the treatability subcontractors in accordance with appropriate state and federal regulations. These materials were



packaged, labeled, and manifested in accordance with the Code of Federal Regulations (CFR), 40 CFR Part 262, and applicable Department of Transportation (DOT) federal regulations under 49 CFR Part 172.

#### Y.2.5.5 Data Management, Analysis, and Interpretation

Raw data from the treatability studies were presented in tabular form by the vendors for review. These data have been analyzed to verify data quality. All data have been checked to assess precision, accuracy, and completeness, and no adverse findings were noted. Interpretation of the data is based on the study objectives presented in Section Y.2.2.

#### Y.2.6 Summary of Results and Conclusions from Phase IX Bench Scale Testing

##### Y.2.6.1 *Ex situ* Chemical Oxidation Testing

On December 13, 2005, PNL collected drum samples into 5-gallon pails according to the above procedures from the following locations for the *Ex situ* Chemical Oxidation treatability tests:

**Table Y-A. Sample Locations for *Ex Situ* Chemical Oxidation Testing**

Material	Drum (Sample) Location
Impacted Soil/Drilling Mud	PNL-03 (Former Lagoon Area Boring 03)
Drilling Mud – Lagoon	PNL-L4A (Lagoon 4-Sample A)
Drilling Mud – Lagoon	PNL-L5B (Lagoon 5-Sample B)
Drilling Muds – Former Lagoon	PNL-BA06

VOC emissions associated with the above samples to check representativeness were not measured due to calibration problems with the PID. These samples were instead chosen based on a visual assessment of material representativeness (i.e., hydrocarbon staining, odors).

##### *Experimental Procedures -- Ex situ Chemical Oxidation Testing*

The *Ex situ* Chemical Oxidation tests were performed according to the steps shown in the ETS Treatability Test protocol shown in Attachment Y1. Tests were performed only on impacted soil from Boring PNL-03 due to time limitations and possible incompatibilities that tarry samples could have had with the technology. VOC emissions were not quantified during the *Ex situ* Chemical Oxidation testing, but instead during a separate Odor Control study performed on the drilling muds and impacted soil samples using ETS' Odor Pro technology. This was due to the fact that VOC emissions on the untreated PNL-03 sample were too low to distinguish from background.

The *Ex situ* Chemical Oxidation tests were performed by PRIMA Environmental (PRIMA) on behalf of ETS at their laboratory in Sacramento, California. PRIMA also prepared a report documenting the procedures and results of the testing (Attachment Y2).

At the Site, samples from 5-gallon buckets collected from the locations listed in **Table Y-A** were sent to Del Mar Analytical for pre-test chemical analyses per **Table Y.2-1/****Table Y.2-2**. Split samples from these locations were sent to PRIMA.

Prior to conducting testing, PRIMA homogenized approximately 5 kilograms of sample from the PNL-03 sample shipped to their lab. The sample was prepared by kneading by hand until a visually uniform color was obtained. A subsample of the untreated, homogenized soil (control) was sent to Del Mar Analytical

for analysis per **Table Y.2-1/Table Y.2-2**. In addition, geotechnical testing was performed by PRIMA and Sierra Testing Labs (El Dorado Hills, California). Results of the pre-treatment samples (Site sample and PRIMA homogenized sample) are shown in **Table Y.2-4**. Analytical results and chain-of-custody forms are attached (Attachment Y3).

Bench testing generally followed the protocol described in Section Y.2.5 with exceptions noted herein. Five treatment conditions, shown in **Table Y.2-4**, were assessed in determining the ability of ETS technology to remove the target compounds. The number of compound concentrations tested was greater than proposed in the test protocol (see Attachment Y1) due to the results of the initial 3 recipes tested. For each treatment test, a known amount of the homogenized control sample was placed into a Pyrex baking dish. Reagent water (10-20 ml) was combined with different amounts of solid reagent and kneaded into the soil.<sup>2</sup> The temperature of the samples was then monitored until a maximum was reached (maximum temperatures are noted in **Table Y.2-4**). The treated samples were subsequently covered with foil to minimize release of odors into the laboratory and allowed to cure for approximately 24 to 48 hours undisturbed. Following curing, the samples were analyzed per **Table Y.2-1/Table Y.2-2**. The control sample was handled in the same manner as the treated samples; it was allowed to stand for 24 to 48 hours and was then submitted for laboratory analysis to assess whether target contaminants were lost due to volatilization.

#### *Experimental Procedures – ETS Odor Control Study*

PRIMA conducted a separate study to assess the effectiveness of an ETS reagent blend known as Odor Pro® to reduce emissions on samples of impacted soil (PNL-03) and lagoon drilling mud (PNL-L5B). The testing generally followed the procedures presented in the test protocol (Attachment Y1) with the exception of the number of test iterations. PID readings were taken immediately prior to and after applying reagent solution of 50 g/l concentration. The sample was then redisturbed and retested. The effectiveness of the ETS odor control technology was also evaluated qualitatively by PRIMA staff.

#### *Results – ETS Ex situ Chemical Oxidation Testing*

The results of the ETS bench testing are shown in **Table Y.2-4**. As shown in the table, the following five reagent blends were tested: Dosages of 7.5 g/kg, 15 g/kg, and 30 g/kg (initial round); and 30 g/kg at pH 11 and 60 g/kg at pH 11 (final round). The reagent concentrations are higher than those proposed in the initial test protocol submitted (1.5 g/kg, 2.5 g/kg, and 5 g/kg). Additional reagent blends were tested based on the performance of the initial three blends run (7.5, 15, and 30 g/kg) at neutral pH. The pH was increased at the 30 g/kg dosage and for the 60 g/kg based on input from ETS following the additional round of tests.

As shown in **Table Y.2-4**, the ETS technology effectively decreased the concentration of TRPH, with removal to less than 30 mg/kg using the highest dose of reagent tested, 60 g/kg of soil. At the lowest dosage (7.5 g/kg), nearly two-thirds of the TRPH was removed. At 30 g/kg–pH 11, TRPH was reduced by nearly 88%. Except for the 60 g/kg test in which extractable fuel hydrocarbons (EFH) increased, the technology did not appear to have significant impact on EFH or VOCs. PRIMA speculated that the poor performance with respect to EFH/VOC reduction was due to the fact that the technology was not tuned properly for treating soils with a relatively high clay/silt content (57.5%, as shown in **Table Y.2-4**). In addition, specifically for the 60 g/kg test, PRIMA speculates that the increase in EFH and GRO was most likely due to the breakdown of larger compounds into smaller ones. EFH is measured using gas chromatography with a flame ionization detector (GC/FID), which measures the amount of carbon. Thus,

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<sup>2</sup> Mechanical mixing of the reagent water with the samples was not performed, as the purpose of the test was to develop proof of concept rather than test specific field application techniques.

the breaking of larger compounds into smaller ones would not change the number of carbon molecules, which in turn would not effect EFH<sup>3</sup>. The TRPH test, by contrast, measures carbon-carbon bonds, and the breakdown of larger compounds into smaller ones would decrease the number of carbon bonds, thus reducing TRPH. EFH can also increase due to smaller molecules derived from larger molecules that are not easily quantified by GC/FID (e.g., those greater than C40).

**Table Y.2-4** also shows the ETS technology did not significantly impact chromium or hexavalent chromium concentrations and that sample moisture content varied as high as 7% higher and 8% lower than the control for the various tests. Process temperatures expectedly increased with increasing reagent concentration, up to a maximum of 53°C at the highest dosage.

ETS did not provide economic data for their *Ex situ* Chemical Oxidation study. However, an ETS representative commented that at concentrations above 30 g/kg, the cost of their product would likely be prohibitive.

Strongly based on the results of the ETS bench study, *Ex situ* Chemical Oxidation was rejected as a process option in preliminary screening (Section 8). The primary test objective, to remediate the light end hydrocarbons (C6 to C12), was not met, and TRPH concentrations were effectively reduced only at reagent concentrations that would be cost prohibitive. In addition, the objective of evaluating VOC emissions reduction with the ETS technology was not assessed for impacted soil due to sample characteristics and not assessed for drilling muds because these materials were not tested.

#### *Results - ETS Odor Control Testing*

ETS' Odor Pro product was applied to two samples, PNL-03 (impacted soil/drilling mud) and PNL-L5B (lagoon drilling mud). PID measurements were not recorded for PNL-03 since levels were too low to distinguish from background. PID readings for PNL-L5B before and after treatment were 66 ppm and 28 ppm, respectively, a reduction of about 56%. Odors were evaluated qualitatively by PRIMA staff after application of Odor Pro. PRIMA staff agreed that the treated sample of PNL-L5B still had a strong, unpleasant odor, whereas the petroleum odor was eliminated from PNL-03 after two applications of the product.

ETS' Odor Pro product was not included in the evaluation of Suppression Agents in Section 8 due to its poor performance in controlling VOC emissions from the lagoon muds and the fact that the foam products tested during Pilot Study No. 3 effectively controlled both VOC and odor emissions.

### **Y2.6.2 Sludge Fluidization Testing**

Sludge Fluidization testing was performed at the Site on December 13 and December 14, 2004, under the direction of PNL on behalf of the RPs. On December 13, 2004, with vendor assistance, PNL collected samples from drums into 5-gallon pails according to the above procedures from the following locations for the sludge fluidization tests:

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<sup>3</sup> A review of the EFH output chromatograms would be needed to confirm breakdown products to see if target compounds are reconfigured.

**Table Y-B. Sample Locations for Sludge Fluidization Testing Material**

Waste Type	Drum (Sample) Location
Tarry Liquids – Lagoons	PNL-L1A (Lagoon 1-Sample A)
Drilling Muds – Lagoon	PNL-L4A and PNL-L5B (Lagoon 4 -Sample A and Lagoon 5-Sample B)
Drilling Muds – Former Lagoon	PNL-BA6

Drilling muds from Lagoon 4 (PNL-L4A) and Lagoon 5 (PNL-L5B) were composited from the 5-gallon pails for the bench testing. VOC emissions associated with the above samples to check representativeness were not measured due to calibration problems with the PID. These locations were chosen based on a visual assessment of material representativeness, as well as presence of hydrocarbon staining, odor level, and physical consistency.

*Experimental Procedures - Petromax Sludge Fluidization Tests:*

The Sludge Fluidization tests were performed according to the steps shown in the Petromax Treatability Test protocol shown in Attachment Y1. With the exception of the field viscosity tests (using a Marsh Funnel viscometer, due to material thickness), all of the tests were run according to the protocol. The samples were measured into glass Mason jars, and liquid product was added and mixed into the waste using a handheld electric mixers until satisfactory fluid properties were achieved (i.e., the waste was in a pourable state, with the waste suspended in the liquid).

**Table Y-C** lists the parameters measured in the Petromax tests:

**Table Y-C. Sludge Fluidization Testing Parameters**

Sample	PID – Pre-Test (ppm)	PID – Mixing (ppm)	Quantity of Waste (ml)	Quantity of Product (ml)	Sample Temp. (°F)	Product Used
PNL-L1A	NA	NA	300	100	NA	Petromax PRR-C
PNL-L4A/L5B	25-30	15-30	400	500	64	NA
PNL-BA6	30	5-6	300	350	64	NA

NA – Not Available

After preparing each test batch, the treated product was in a pourable state and did not stick to the sides of the glass jar. The pourability and lack of adhesion to the sample container was considered an important parameter, since this indicates the material will not stick to the inside of a vacuum truck or tanks during pumping. For the testing of PNL-L4A/L5B and PNL-BA6, the product was added in excess to qualitatively measure viscosity changes. Petromax estimates the material was pumpable at a ratio of about 1:4 to 1:6 product to waste. In addition, heated water (at temperatures of 90°F and 105°F) was added to the mixtures to qualitatively test viscosity changes. By increasing the amount of Petromax product and heat, the viscosity of the material was observed to decrease. Emissions were noted both prior to and during mixing of the Petromax product and waste and were observed to be below 50 ppm, as shown in the table. Addition of the Petromax product appeared to mitigate the sample odors somewhat. However, odors were still observed after treatment.

Enough liquified waste material was generated for chemical and fuel analyses. The results of these analyses and comparison to the test objectives are discussed below.

### *Experimental Procedures - TEC's Fluidization Tests:*

TEC performed fluidization testing using their own products on the same waste streams using a protocol similar to the Petromax tests.

#### *HE-1000 – Drilling Muds*

As described above, TEC's HE-1000 product was used for fluidizing the drilling muds (Lagoon and Non-Lagoon), and their ACL product was used for fluidizing the lagoon tarry liquids. For the drilling muds, according to the vendor, HE-1000 should theoretically be applied at a rate of about 1 ml per 4 ounces (118 ml) of waste to achieve pumpability, although in the field, much greater portions (not quantified) were added. Following treatment, the viscosity of the materials appeared to be greater than the material tested by Petromax. Product evaluation was hindered by the unavailability of a mixing device during the tests. Emissions measured during product application were low, less than 10 ppm; however, odors associated with the waste were observed during and following testing. It should be noted that HE-1000 also has a secondary bioremediation component which was not evaluated during this test.

#### *ACL – Lagoon Tarry Liquids*

According to the vendor, about 6% ACL product by volume was required to make the lagoon tars pumpable. In the field tests at the Site, a greater volume (not quantified) was added, and after treatment, the viscosity of the tars appeared to be higher than that observed during the Petromax tests. As expected with the ACL product, VOC emissions were in the few hundred ppm range during product addition and mixing. This required mitigation with HE-1000, which reduced VOCs to less than 10 ppm after several additions.. The ACL product also had a persistent strong chemical odor that overwhelmed the waste odors following testing.

Enough liquified waste material was generated (**Table Y.2-1**) for chemical and physical (fuel) analyses. The results of these analyses and comparison to the test objectives is discussed below.

### *Analytical Results – Chemical Analyses:*

As shown in **Tables Y.2-1** and **Y.2-2**, treated samples from the Petromax and TEC bench tests were submitted for the following laboratory chemical analyses: TRPH by EPA Method 418.1, VOCs by EPA method 8260B, and Fish Bioassay. These tests were performed to determine the chemical characteristics of the waste material following treatment to enable assessment of the changes in chemical characteristics from the untreated materials due to product application. Pre-treatment waste concentrations were established by analyses conducted in conjunction with the *Ex situ* Chemical Oxidation study, as well as prior Pilot Study No. 3 testing.

Analytical results are presented in Attachment Y3 and summarized in **Table Y.2-5**. Analytical tests were performed on treated samples from Lagoon 1A (PNL-L1A), Lagoons 4A/5B composite (PNL-L4A/5B), and the Former Lagoon Areas (i.e., PNL-BA06). The sample IDs were modified slightly from above for these tests and are defined in the table.

**Table Y.2-5** shows the TRPH concentrations ranged from about 3% in the treated former lagoon area samples to between about 30% and 50% in the treated Lagoon 1A samples. Concentrations were the same order of magnitude in the Petromax- and TEC-treated samples and higher in each case in the TEC samples. The highest result of 50% TRPH in the Lagoon 1A TEC-treated sample (PNL-L1-TE) is likely due to the terpene hydrocarbon additive of the TEC-ACL product. Also shown in **Table Y.2-5**, VOCs detected were primarily BTEX compounds ranging from low ppm up to the few percent range, except for

a detection of toluene of 15% in PNL-L1-TE. This detection is likely attributable to the ACL product. The detection of 1.7% styrene in PNL-L1-TE is also likely due to the ACL product, since this analyte was not detected in the pre-treatment samples (see below). VOC concentrations appear to be in the same range for both the Petromax- and TEC-treated samples. Finally, **Table Y.2-5** shows the treated material passed the California Title 22 fish bioassay hazardous waste screening, indicating the material would not be classified as California Hazardous Material for waste disposal purposes in California due to toxicity to aquatic organisms.

Pre-treatment waste samples were collected from Lagoons 4 and 5 and the former lagoon areas during the *Ex situ* Chemical Oxidation bench testing and analyzed for VOCs and TRPH, along with other analytes pertinent to evaluation of the *Ex situ* Chemical Oxidation test. In addition, samples collected from Lagoons 1 and 2 were evaluated for the same analytes during Phase IV of Pilot Study No. 3. These sample results are used for general comparison with the treated results described above to see if the fluidization treatments had any measurable effects on the chemical concentrations in the materials.

A subset of the pre-treatment results associated with the *Ex situ* Chemical Oxidation bench testing and the results from the Lagoon 1 sampling during Phase IV are shown in **Table Y.2-6**. **Table Y.2-6** shows that the TRPH concentrations in Lagoons 4 and 5 and the Former Lagoon areas are comparable after product addition, for both Petromax and TEC products. **Table Y.2-6** also shows the same VOC compounds were detected in the pre-treatment and post-treatment samples, but concentrations were up to one to two orders of magnitude higher in the post-treatment samples, apparently due to product addition. A few analytes--styrene, toluene (except for a slight detection in PNL-L1B) and 1,1,2,2-tetrachloroethane--were not detected in the pre-treatment samples. The table also shows the TRPH concentrations in Lagoon 1A/1B are the same order of magnitude but about 2 and 5 times higher in the Petromax- and TEC-treated samples, respectively. Fish bioassay analyses were not conducted for the pre-treatment samples.

#### *Analytical Results – Physical (Fuel) Analyses:*

Treated samples from the Petromax and TEC tests were submitted to Conti Testing Laboratories (Bethel Park, Pennsylvania) for heat value and viscosity tests, as indicated in **Table Y.2-1**. Analytical reports are presented in Attachment Y3. The results of these tests are presented in **Table Y-D**:

**Table Y-D. Post-Fluidization Analytical Results**

Sample ID	Heat Value (ASTM D-3286) (BTU/lb)	Viscosity at 70°F (Brookfield Test) (cps)
PNL-L1A (Petromax)	7,502	2,850
PNL-L1A (TEC)	12,964	38,667
PNL-L4A/5B (Petromax)	2,564	200
PNL-L4A/5B (TEC)	2,368	175,000
PNL-BA6 (Petromax)	348	540
PNL-BA6 (TEC)	1,012	405,000

It is apparent from the above table that 1) the Petromax products were superior to the TEC products in reducing viscosity and hence enhancing pumpability, confirming the qualitative assessments from the field tests, and 2) Petromax's products appeared to depress the BTU value of these materials more than TEC's products, which is not unexpected given the nature of the additives (particularly TEC's ACL product).



In Pilot Study No. 3 Phase IV, discussed in Section 3.2, heat value (BTU/lb) and Brookfield viscosity were measured for various lagoon materials—tarry liquids and drilling muds. Heat value was about 13,000 BTU/lb for Lagoons 1 and 2 (3 of 4 samples) and 5,000 BTU/lb for Lagoon 3. Heat value was not measured in drilling muds from Lagoons 4 and 5 or from samples collected in the former lagoon areas. Based on the data from Lagoon 1, it appears the Petromax product decreased heat value by about 40% due to dilution, and TEC's ACL product maintained heat value of the material. This is significant, because costs may increase for fuel blending materials that are below a certain heat value threshold (e.g., 10,000 BTU/lb).

In Pilot Study No. 3 Phase IV, Brookfield viscosity was measured on Lagoon 1A/1B samples and Lagoon 2A/2B samples at various temperatures. At 100°F (the closest temperature to the above post-treatment tests), Brookfield viscosity was >400,000 centiPoise (cP) for Lagoon 1A/B and Lagoon 2A/B samples. Comparing this to the post-treatment test results for the Lagoon 1A samples, it is apparent that both Petromax and TEC products achieved significant reductions in viscosity, with the Petromax products reduction more than an order of magnitude greater. For reference, at the highest temperature measured in the pre-treatment tests (300°F), Brookfield viscosities ranged between approximately 1,000 and 2,500 cP.

Based on the results of the Sludge Fluidization bench studies described above, it is apparent that this technology is feasible for making certain drilling muds and tarry liquids from the Site pumpable, without generating significant emissions (or with application of certain mitigating steps). The above results were taken into consideration in evaluating the feasibility of Sludge Fluidization for treatment and removal of drilling muds and lagoon tarry liquids during the initial and final screening of this remedial technology (Fluidization and Pumping, see Section 9).



**Table Y.2-1**  
Phase IX Bench Scale Treatability Test Summary  
Ascon Landfill Site

	Vendor	Testing Location	Technology Type	Proposed Sample Locations <sup>(1)(2)</sup>	Tests/Analyses <sup>(3)</sup>	Proposed Laboratories for Analytical Tests
<b>TPH-Impacted Soil</b>	Environmental Technology Solutions	Sacramento, CA	<i>Ex situ</i> Chemical Oxidation	PNL-4, PNL-5, PNL-6	Grain size (ASTM D1140) (untreated samples only). Moisture content (ASTM D2216), TRPH (EPA 418.1), EFH/GRO (EPA 8015B MOD), and VOCs (EPA 8260B) in untreated and treated samples. VOC emissions (by PID) in untreated and treated samples, and during treatment.	Del Mar Analytical (Irvine, CA); PRIMA Environmental (Sacramento, CA); Sierra Testing Labs (El Dorado, CA)
	Various (OnSite Technology, LLC; Mobile Enviromental Technologies, Inc.)	N/A - Qualitative Evaluation	<i>Ex situ</i> Thermal Desorption	N/A - Qualitative Evaluation	N/A - Qualitative Evaluation	N/A - Qualitative Evaluation
<b>Drilling Muds - Lagoons</b>	Environmental Technology Solutions	Oakland, CA	<i>Ex situ</i> Chemical Oxidation	PNL-L4A; PNL-L5B	Grain size (ASTM D1140) (untreated samples only). Moisture content (ASTM D2216), TRPH (EPA 418.1), EFH/GRO (EPA 8015B MOD), and VOCs (EPA 8260B) in untreated and treated samples. VOC emissions (by PID) in untreated and treated samples, and during treatment.	Del Mar Analytical (Irvine, CA); PRIMA Environmental (Sacramento, CA); Sierra Testing Labs (El Dorado, CA)
	Petromax Technologies, Texas EnviroChem Group, LLC	Sacramento, CA	<i>Ex situ</i> Sludge Fluidization	PNL-L4A; PNL-L5B	TRPH (EPA 418.1), VOCs (EPA 8260B), Fish Bioassay (See Notes), BTU/lb (ASTM D3286), and viscosity (Brookfield test and Marsh Funnel) of treated sample. Also VOC emissions (by PID) during treatment.	Del Mar Analytical (Irvine, CA); Conti Testing Laboratories (Bethel Park, PA)
	Various (OnSite Technology, LLC; Mobile Enviromental Technologies, Inc.)	N/A - Qualitative Evaluation	<i>Ex situ</i> Thermal Desorption	N/A - Qualitative Evaluation	N/A - Qualitative Evaluation	N/A - Qualitative Evaluation
<b>Drilling Muds - Former Lagoon Areas</b>	Environmental Technology Solutions	Sacramento, CA	<i>Ex situ</i> Chemical Oxidation	PNL-3; PNL-4; PNL-5; PNL-6	Grain size (ASTM D1140) (untreated samples only). Moisture content (ASTM D2216), TRPH (EPA 418.1), EFH/GRO (EPA 8015B MOD), and VOCs (EPA 8260B) in untreated and treated samples. VOC emissions (by PID) in untreated and treated samples, and during treatment.	Del Mar Analytical (Irvine, CA); Prima Environmental (Sacramento, CA)
	Petromax Technologies, Texas EnviroChem Group, LLC	Huntington Beach, CA	<i>Ex situ</i> Sludge Fluidization	PNL-3; PNL-4; PNL-5; PNL-6	TRPH (EPA 418.1), VOCs (EPA 8260B), Fish Bioassay (See Notes), BTU/lb (ASTM D3286), and viscosity (Brookfield test and Marsh Funnel) of treated sample. Also VOC emissions (by PID) during treatment.	Del Mar Analytical (Irvine, CA); Conti Testing Laboratories (Bethel Park, PA)
	Various (OnSite Technology, LLC; Mobile Enviromental Technologies, Inc.)	N/A - Qualitative Evaluation	<i>Ex situ</i> Thermal Desorption	N/A - Qualitative Evaluation	N/A - Qualitative Evaluation	N/A - Qualitative Evaluation
<b>Lagoon Tar</b>	Petromax Technologies, Texas EnviroChem Group, LLC	Huntington Beach, CA	<i>Ex situ</i> Sludge Fluidization	PNL-L1A; PNL-L2A	TRPH (EPA 418.1), VOCs (EPA 8260B), Fish Bioassay (See Notes), BTU/lb (ASTM D3286), and viscosity (Brookfield test and Marsh Funnel) of treated sample. Also VOC emissions (by PID) during treatment.	Del Mar Analytical (Irvine, CA)/ Conti Testing Laboratories (Bethel Park, PA)

**Notes:**

TRPH - total recoverable petroleum hydrocarbons

EFH/GRO - extractable fuel hydrocarbons/gasoline range organics

BTU - British Thermal Unit

VOCs - volatile organic compounds

PNL - Project Navigator, Ltd.

(1) Material characteristics (including emissions potential) will be verified in the field prior to sample collection. Alternative sample locations, if more representative, may be substituted for these locations.

(2) See Figure 8.2-3 for sample locations.

(3) From 22CFR22 CCR 66261.24 (a)(6): "Static Acute Bioassay Procedures for Hazardous Waste Samples," California Department of Fish and Game, Water Pollution Control Laboratory, November 1988.

**Table Y.2-2**  
*Ex Situ* Chemical Oxidation Treatability Test Parameters  
 Ascon Landfill Site

<b>Pre-Treatment Waste Characterization</b>			
Matrix	Parameter	Purpose	Methodology
Soil	Physical (grain size, moisture content)	To determine reagent selection and residence time requirements.	ASTM D1140; ASTM D2216
	TRPH	To determine total petroleum hydrocarbon concentration of waste versus treated waste.	EPA Method 418.1
	EFH/GRO	To determine carbon speciation and proportion of lighter end hydrocarbons in the untreated waste.	EPA Method 8015B
	VOCs	To determine VOC concentration of waste versus treated waste.	EPA Method 8260B
	VOCs (vapor)	To compare emissions of untreated versus treated material and to air quality standard.	Calibrated PID meter
<b>Characterization During Treatment</b>			
Matrix	Parameter	Purpose	Methodology
Soil with Reagents	Cure Time	To determine total reaction time required per batch.	Bench Test
	VOCs (vapor)	Quantify potential emissions during treatment.	Calibrated PID meter
<b>Treatment Product Characterization</b>			
Matrix	Parameter	Purpose	Methodology
Treated Soil	Moisture Content	To determine how the treatment process impacts moisture content of the native materials, since this may affect final handling.	ASTM D2216
	TRPH	To determine degree of TRPH reduction in treated product and to compare to potential treatment standard.	EPA Method 418.1
	EFH/GRO	To determine the proportion of lighter end hydrocarbons remaining in the waste following treatment, and (for EFH), to compare to treatment standard.	EPA Method 8015B
	VOCs	To determine degree of VOC reduction in treated product and to compare to potential treatment standard.	EPA Method 8260B
	VOC (vapor)	To compare emissions of untreated versus treated material and to air quality standard.	Calibrated PID meter

**Notes:**

TRPH - total recoverable petroleum hydrocarbons

EFH/GRO - extractable fuel hydrocarbons/gasoline range organics

VOCs - volatile organic compounds

PID - photoionization detector

**Table Y.2-3**  
**Sludge Fluidization Treatability Test Parameters**  
**Ascon Landfill Site**

<b>Pre-Treatment Waste Characterization</b>			
Matrix	Parameter	Purpose	Methodology
Note: Physical and chemical characteristics (including emissions) of TPH-Impacted Soil and Drilling Muds (Lagoon/Non-Lagoon) will be established during the ETS treatability test and will not be repeated here, as they are not pertinent to assessing product performance or waste receiver requirements.			
<b>Characterization During Treatment</b>			
Matrix	Parameter	Purpose	Methodology
Tar or Drilling Mud with Additives	Mixing Time	To determine material application rate.	Bench Test
	VOCs (vapor)	Quantify potential emissions during treatment.	Calibrated PID meter
<b>Treatment Product Characterization</b>			
Matrix	Parameter	Purpose	Methodology
Treated Tar or Drilling Mud	TRPH	To determine total petroleum hydrocarbon concentration of treated product - for waste receiver acceptance.	EPA Method 418.1
	VOCs	To determine VOC content of treated product - for waste receiver acceptance/characterization.	EPA Method 8260B
	Fish Bioassay	To determine toxicity of fluidized waste material - for waste receiver acceptance/characterization.	See Table 8.2-1
	Viscosity (lab)	To quantify the pumpability of the fluidized waste material.	Brookfield Test
	Viscosity (field)	To quantify the pumpability of the fluidized waste material, to compare to standard used in oil industry.	Marsh Funnel Viscometer Test
	Heat value (BTU/lb)	To estimate fuel value of treated product.	ASTM D240

**Notes:**

TRPH - total recoverable petroleum hydrocarbons

VOCs- volatile organic compounds

BTU - British Thermal Unit

ASTM - American Society of Testing Materials

**Table Y.2-4**  
Summary of Experimental and Analytical Results  
*Ex Situ* Chemical Oxidation Treatability Study  
Ascon Landfill Site

Analyte	Units	Untreated Soil	Process Control	Reagent Addition Rate				
				7.5 g/kg	15 g/kg	30 g/kg	30 g/kg-pH 11	60 g/kg-pH 11
GRO	mg/kg	84	680	690	610	490	840	950
DRO/ORO	mg/kg	700	5600	5200	4500	3600	6100	7100
EFH	mg/kg	780	6200	5900	5100	4100	7000	8000
TRPH	mg/kg	11000	2100	700	910	830	260	< 30
VOCs								
sec butylbenzene	µg/kg	37	78	100	93	41	52	65
ethylbenzene	µg/kg	30	35	46	65	32	41	42
isopropylbenzene	µg/kg	61	77	110	110	48	81	90
p-isopropylbenzene	µg/kg	67	120	160	150	67	87	110
napthalene	µg/kg	50	83	90	110	67	58	75
n-proylbenzene	µg/kg	80	120	170	170	71	120	130
toluene	µg/kg	ND < 1.8	ND < 9.6	ND < 10	ND < 10	ND < 10	2.9	ND < 4
1,2,4-trimethylbenzene	µg/kg	160	220	260	420	190	100	310
1,3,5-trimethylbenzene	µg/kg	85	140	170	170	73	41	130
o-xylene	µg/kg	5	ND < 9.6	ND < 10	ND < 10	ND < 10	2.4	ND < 4
m,p-xylenes	µg/kg	15	13	17	31	17	19	23
Total Cr	mg/kg	24	23	21	21	23	n.m.	n.m.
Cr(VI)	mg/kg	ND < 0.2	ND < 0.2	ND < 0.2	0.38	0.69	n.m.	n.m.
Maximum Temperature during treatment	°C	not applicable	18	26	32	41	40	53
% Moisture	%	27.2	22	23.1	29.7	26.1	17.5	14.1
% Passing #200 Sieve	%	57.5	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.

**Notes:**

Untreated soil is field sample PNL-03

Process Control is soil that was handled in the same manner as the treated samples, but to which no reagents were added.

For clarity, only detected VOCs are listed.

Numbers in *italics* are qualified due to matrix effects

"n.m." = not measured

**Table Y.2-5**  
Summary of Post-Treatment Sludge Fluidization Study Analytical Results  
Ascon Landfill Site

Analyte	Units	PNL-L1-P	PNL-L45-P	PNL-FLA-P	PNL-L1-TE	PNL-L45-TE	PNL-FLA-TE
<b>TRPH - EPA 418.1</b>	mg/kg	280,000	74,000	27,000	510,000	85,000	29,000
<b>VOCs - EPA 8260B</b>							
Benzene	µg/kg	1,000	ND < 1,000	ND < 190	1,000	ND < 200	ND < 200
n-Butylbenzene	µg/kg	6,100	8,800	ND < 460	ND < 2,500	ND < 500	ND < 500
sec-Butylbenzene	µg/kg	5,700	10,000	3,300	ND < 2,500	ND < 1,800	1,400
Ethylbenzene	µg/kg	13,000	11,000	7,500	39,000	4,200	200
Isopropylbenzene	µg/kg	5,800	26,000	6,900	22,000	6,500	2,400
p-Isopropyltoluene	µg/kg	67,000	5,000	3,600	ND < 1,000	3,400	1,800
Naphthalene	µg/kg	24,000	20,000	15,000	6,900	4,300	3,400
n-Propylbenzene	µg/kg	14,000	15,000	8,100	ND < 1,000	3,800	2,700
Styrene	µg/kg	ND < 1,000	ND < 1,000	ND < 190	17,000	ND < 200	ND < 200
1,1,2,2-Tetrachloroethane	µg/kg	1,000	ND < 1,000	ND < 190	ND < 1,000	ND < 200	ND < 200
Toluene	µg/kg	1,100	ND < 1,000	ND < 190	150,000	ND < 200	ND < 200
1,2,4-Trimethylbenzene	µg/kg	11,000	5,300	11,000	7,100	2,700	3,600
1,3,5-Trimethylbenzene	µg/kg	1,600	1,300	3,00	1,300	910	1,000
o-Xylene	µg/kg	1,800	ND < 1,000	410	12,000	270	ND < 200
m,p-Xylenes	µg/kg	2,700	1,800	2,500	16,000	670	700
<b>Fish Bioassay</b>	N/A	Passed	Passed	Passed	Passed	Passed	Passed

Notes/Legend:

Only detected VOCs are shown.

ND = Not Detected (Below Laboratory Reporting Limit)

PNL-XX-P - Petromax samples

PNL-XX-TE - TEC samples

FLA = Former Lagoon Areas (corresponds to drum sample PNL-BA6)

PNL-L45-X is a composite sample from Lagoon 4A/5B (see text section Y.2.6.2)

Fish Bioassay - CCR Title 22 Fathead Minnow HW Screen Bioassay

Passed means <40% of fish dead at 750 mg/L waste concentration (corresponding to LC50 < 750 mg/l).

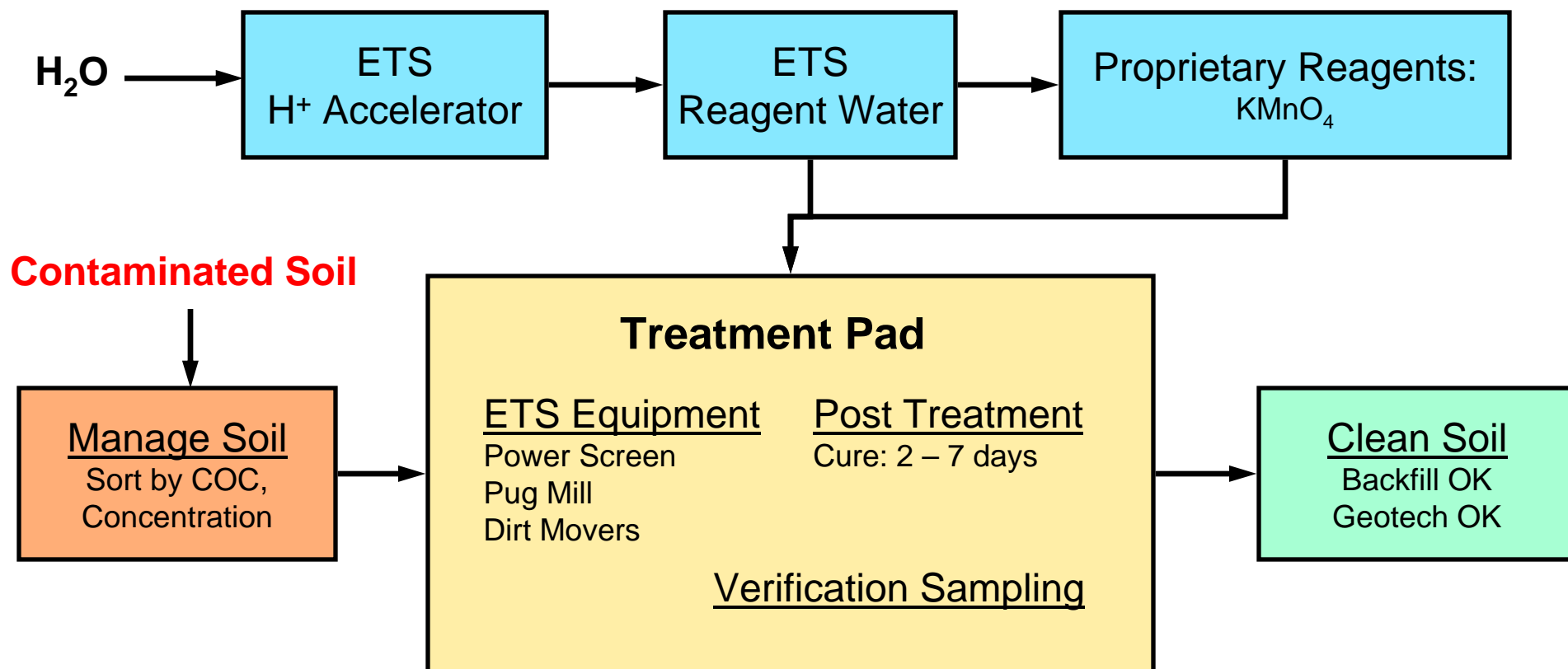
**Table Y.2-6**  
Pre-Treatment Waste Concentrations for Phase IX Bench Testing  
Ascon Landfill Site

Analyte	Units	PNL-L4A	PNL-L5B	PNL-BA06	PNL-L1A (from Phase IV)	PNL-L1B (from Phase IV)
<b>TRPH - EPA 418.1</b>	mg/kg	290,000	86,000	35,000	110,000	120,000
<b>VOCs - EPA 8260B</b>						
Benzene	µg/kg	110	ND < 100	120	640	140
n-Butylbenzene	µg/kg	ND < 250	730	ND < 250	ND < 250	ND < 250
sec-Butylbenzene	µg/kg	400	1,100	770	920	330
Ethylbenzene	µg/kg	880	2,100	2,000	3,600	880
Isopropylbenzene	µg/kg	570	4,700	1,200	1,500	360
p-Isopropyltoluene	µg/kg	420	ND < 100	800	580	240
Naphthalene	µg/kg	2,400	2,300	2,700	5,500	1,900
n-Propylbenzene	µg/kg	870	2,300	1,600	2,700	670
Styrene	µg/kg	ND < 100	ND < 100	ND < 100	ND < 100	ND < 100
1,1,2,2-Tetrachloroethane	µg/kg	ND < 100	ND < 100	ND < 250	ND < 100	ND < 100
Toluene	µg/kg	ND < 100	ND < 100	ND < 100	ND < 100	110
1,2,4-Trimethylbenzene	µg/kg	1,800	ND < 100	1,900	2,000	770
1,3,5-Trimethylbenzene	µg/kg	580	ND < 100	480	350	170
o-Xylene	µg/kg	380	ND < 100	230	130	130
m,p-Xylenes	µg/kg	700	ND < 100	290	310	240
<b>Fish Bioassay</b>	NA	NA	NA	NA	NA	NA

Notes/Legend:

Only detected VOCs are shown.

NA = Not Available

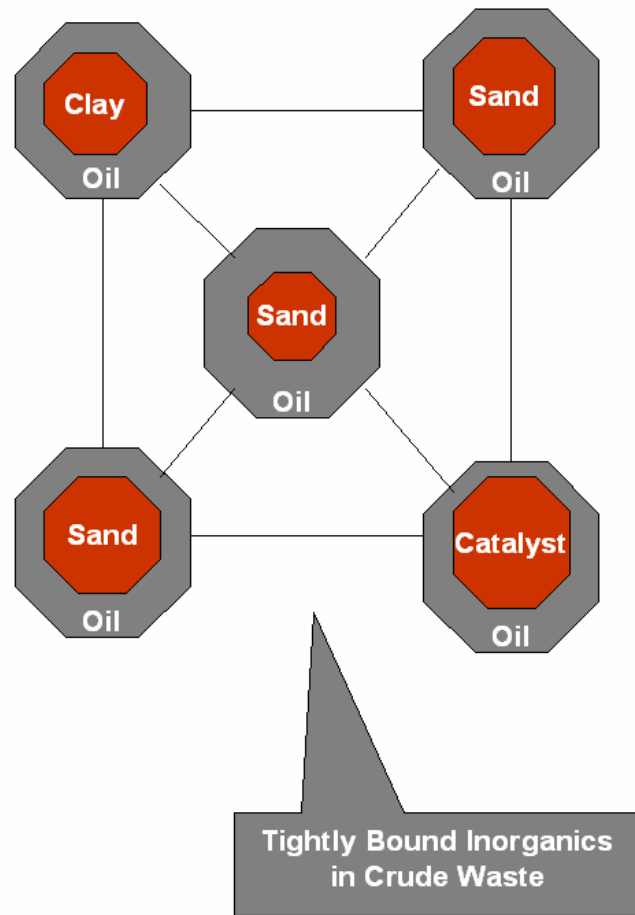


*Ex situ* Chemical Oxidation - Process Flow Diagram

Figure Y.2-1



# Petromax Sludge Conditioning – Mechanism of Action



**The Problem:** Crude oil tank bottoms contain settled inorganic particles which create a very tacky sludge at the bottom of storage tanks. The inorganics and crude are tightly bound by the balance of negative and positive charges in this non-polar environment throughout the sludge.

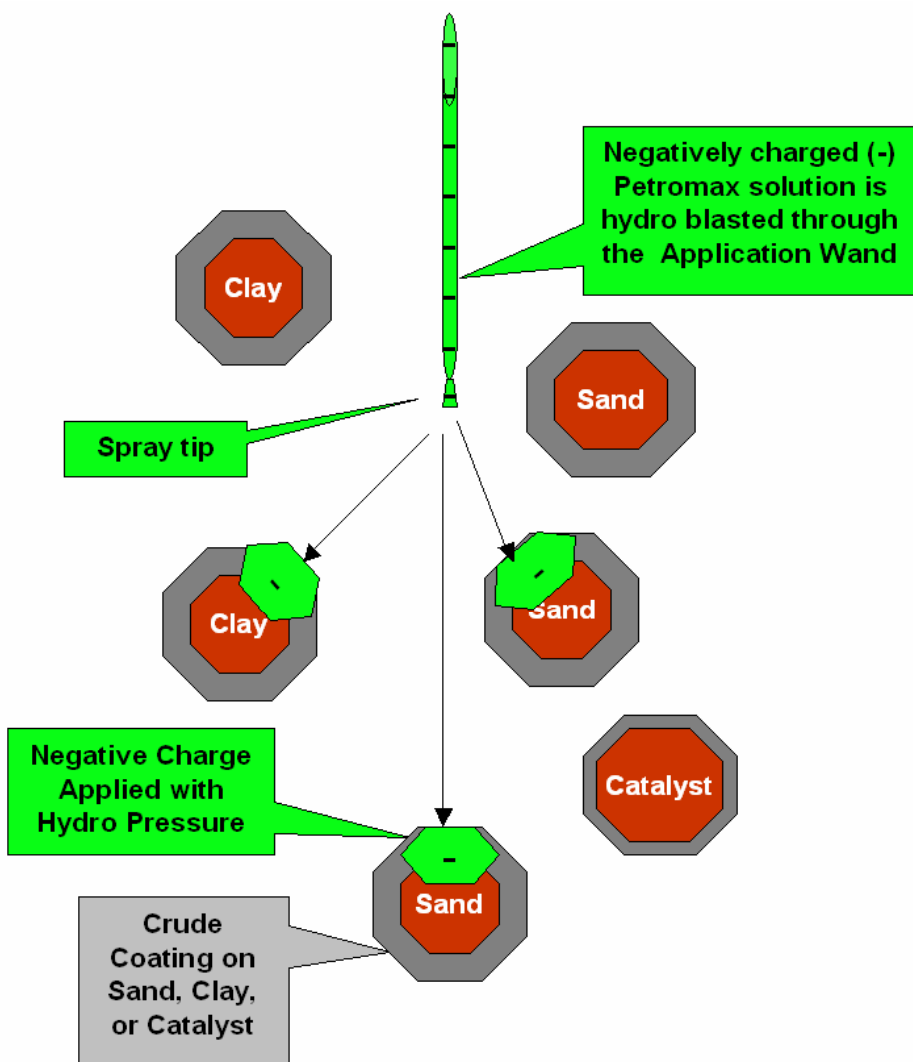
This makes for a very unpumpable waste that is difficult to remove from the tank. In addition, conventional methods for removal, recovery, and disposal are expensive when all costs are taken into consideration.



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**Petromax Technologies: Sludge Conditioning Mechanism of Action**

**Figure Y.2-2a**



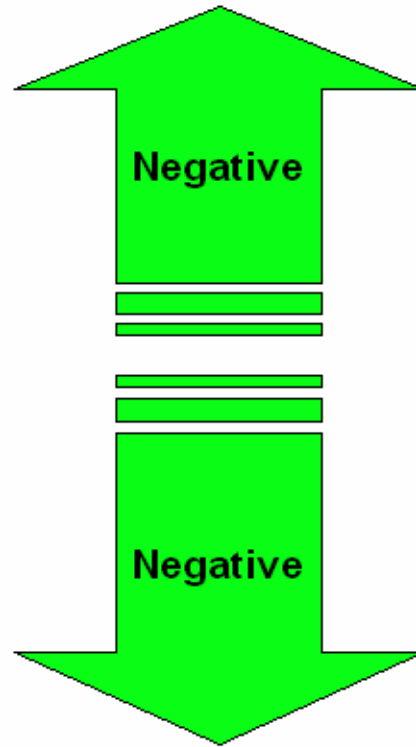
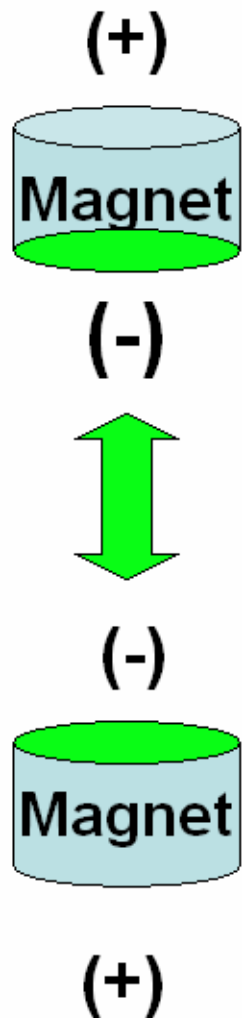
**The Solution:** The solution is to break the bond between the inorganic particle and the oil. Once the balance of “charges” have shifted to a negative state, the inorganic particles, the waste oil, and the Petromax solution instantly begin to repel from each other.

**The Application:** This diagram depicts our Petromax Conditioning Process hydro-blasting Petromax product into a crude sludge at 3000 psi. This physical application requires close proximity of a spray tip/wand to the inorganics (sand, clay, catalyst fines, etc.) in order to shear off the attached hydrocarbon and apply the negative charge to sand, clay, catalyst, etc.

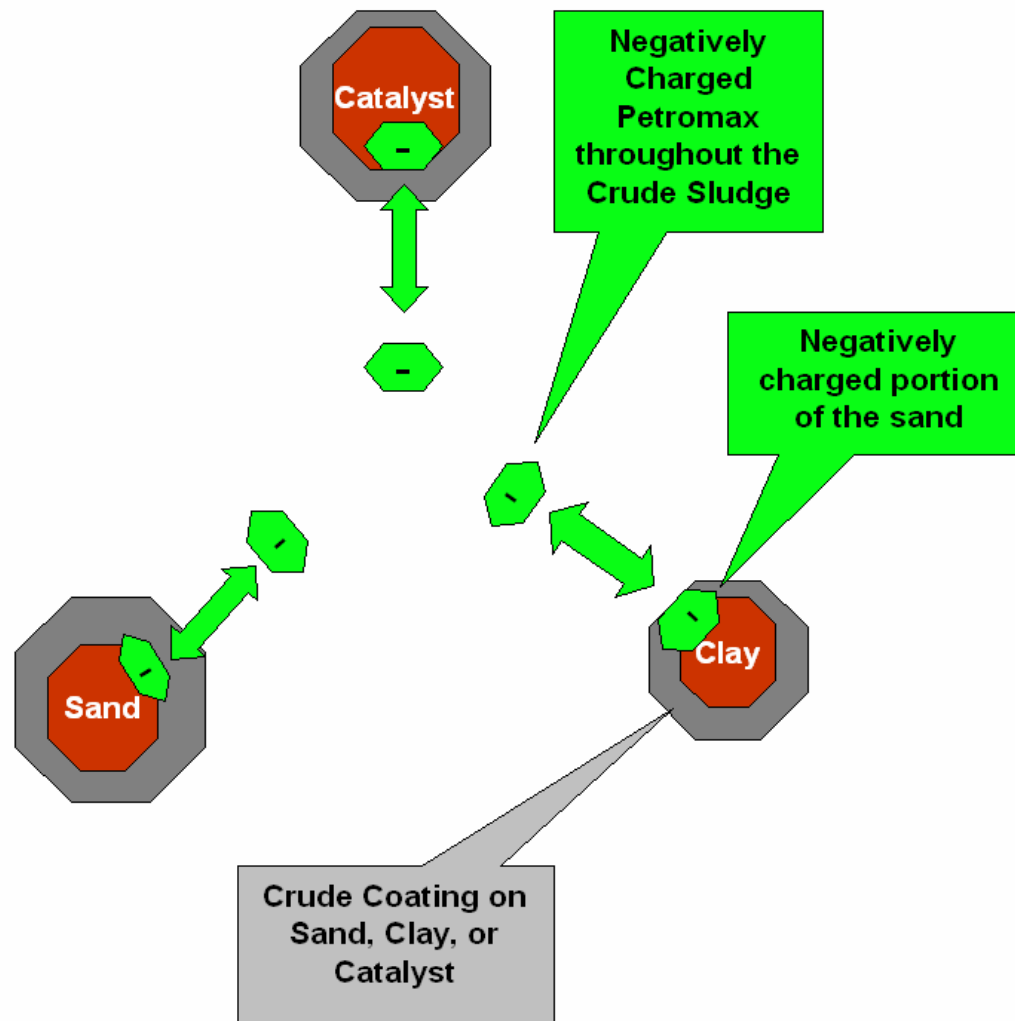
**The Effect:** The diagram shows a partial cleaning or releasing of the hydrocarbon from the inorganic particulate. This application is designed to create a solids suspended slurry or a very pumpable sludge that will not stick to the interior of hoses or downstream containment once it is removed from a tank.

**Petromax Technologies: Sludge Conditioning Mechanism of Action**

**Figure Y.2-2b**



**The Physical Phenomena:** The effect of this repelling/cleaning application would be like two magnets that are brought close to one another and are immediately repelled by like polarities.



### The Conditioning Process:

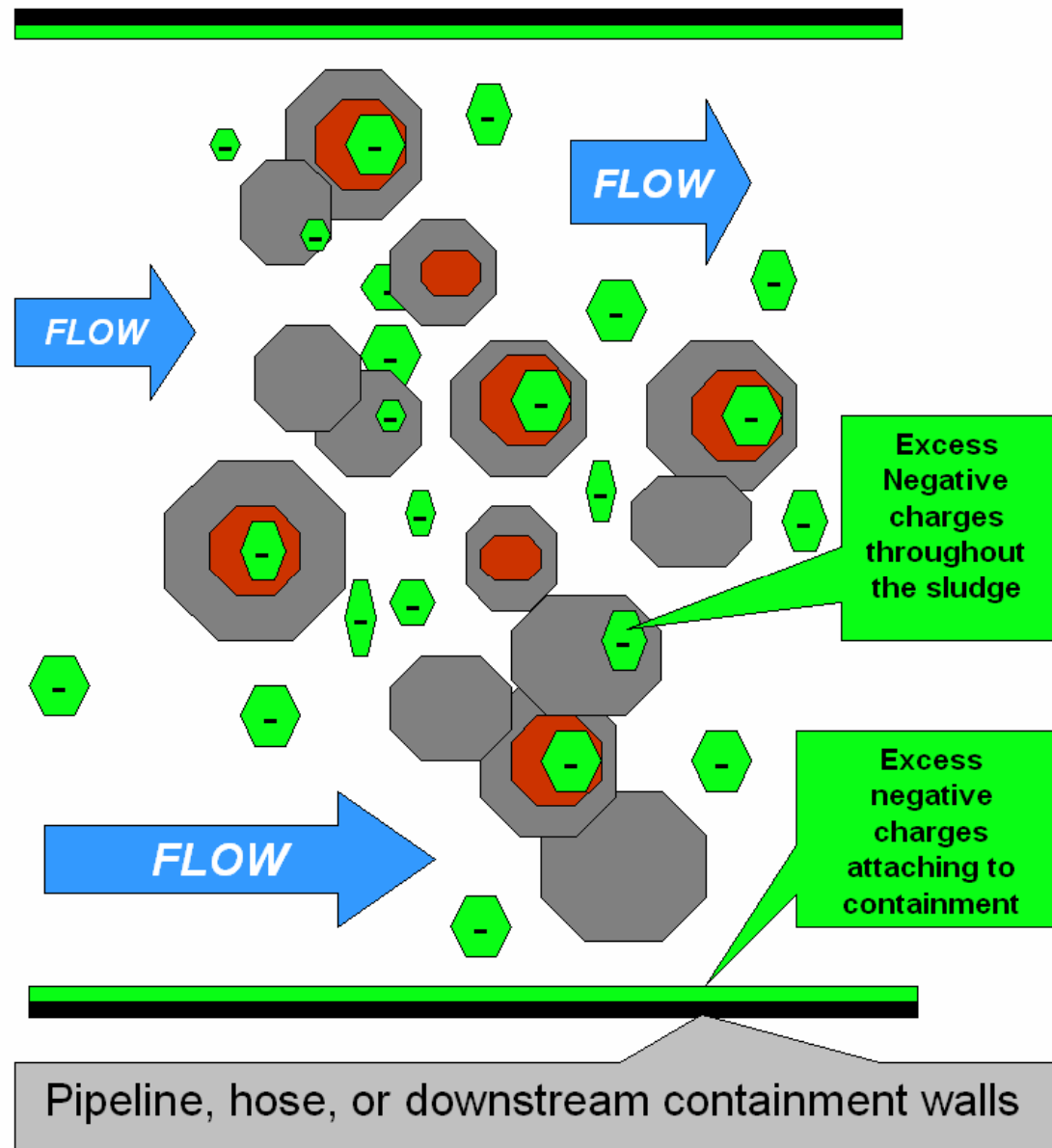
The water-based Petromax solution that has been hydro blasted throughout the crude sludge has created a pumpable slurry.

The *uncharged* portion of the heavy crude is essential to suspend the solids within the "conditioned sludge".

This suspension / conditioning process enables the waste stream to be transferred easily from point A to point B.

Petromax Technologies: Sludge Conditioning Mechanism of Action

Figure Y.2-2d



**The Result:** The conditioning process carries an excess of negative charges throughout the sludge.

*Although we cannot see this activity with the naked eye, the sludge molecules are actively repelling. At this point, the sludge will very quickly take the path of least resistance as it begins to flow.*

Visibly we see a very homogeneous pumpable mix much like a thick dark colored milk shake.


## Sludge Conditioning in Process

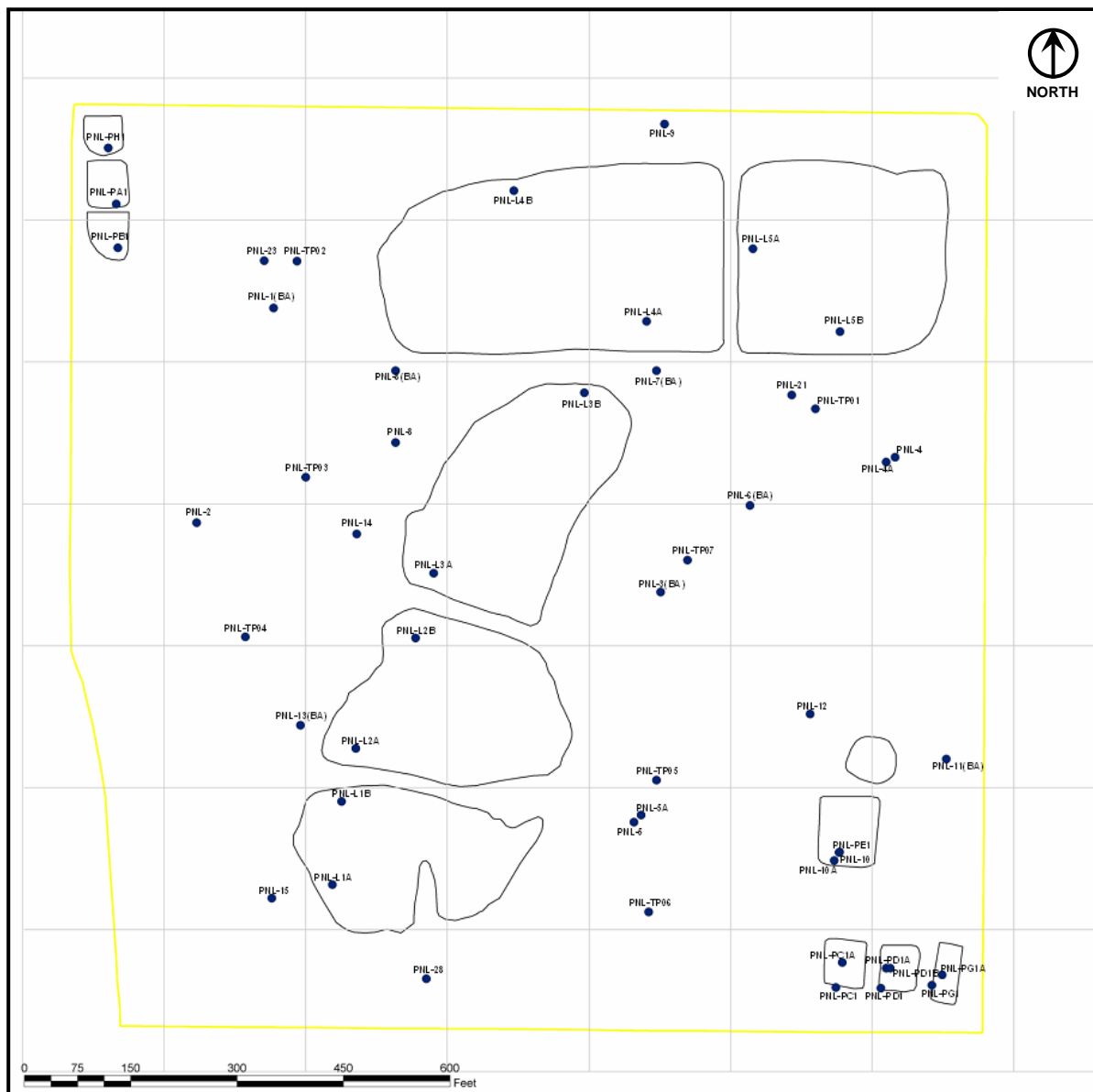


The hand held hydro blasting wand is buried in the sludge as an important part of the Petromax Process

Notice the spray tip buried into the sludge during the conditioning process. Shearing the crude from the inorganics must occur as part of the mechanism of action. Because our process occurs rapidly, on average our Petromax Process adds only 15 - 20 % to the waste stream.

## Production Crude Tank Sludge Conditioning Process

Petromax Technologies: Sludge Conditioning Mechanism of Action		Figure Y.2-2f
Revised Feasibility Study Ascon Landfill Site, Huntington Beach, California	September 2007	 PROJECT NAVIGATOR, LTD.®



## LEGEND

- Pilot Study No. 3 Location
- Pit and Lagoon Boundary
- Site Boundary
- 200-foot Grid

NOTE: Phase VIII (Pit F) sampling locations not shown for clarity.

Phase IX Technology Bench Scale Testing Sampling Locations (Includes Additional PS3 Locations)

Figure Y.2-3