

PHASE IX ADDENDUM

PILOT STUDY NO. 3

**WASTE CHARACTERIZATION, EMISSIONS, AND
EXCAVATION TESTING PROGRAM WORKPLAN**

TECHNOLOGY BENCH SCALE TESTING

**ASCON LANDFILL SITE
HUNTINGTON BEACH, CALIFORNIA**

Submitted to:

**State of California Environmental Protection Agency
Department of Toxic Substances Control**
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1. INTRODUCTION

1.1 Preface

This Phase IX Addendum (Addendum) to the Pilot Study No. 3 Workplan (Workplan) contains the proposed final technical approach for Technology Assessment at the Ascon Landfill Site (the site) located in Huntington Beach, California. It provides additional details on bench scale testing procedures for four specific waste types identified at the site:

- The oily tars found on the surface in Lagoons 1, 2, and 3,
- The drilling muds found in the lagoons,
- The drilling muds found in the former lagoon areas, and
- Total petroleum hydrocarbon (TPH)-impacted soils.

This Addendum was prepared by Project Navigator, Ltd. (PNL) on behalf of the Ascon Landfill Site Responsible Parties (RPs) for submittal to the Department of Toxic Substances Control (DTSC).

1.2 Objectives

Phases I through VIII (Phase VII was not implemented) of the Workplan focused on data collection to define the nature and extent of the wastes present in specific areas of the site. To date, limited data have been collected to evaluate the efficacy of treatment methods designed to increase handling efficiency and control vapor emissions from waste materials. Therefore, Phase IX of the program has been developed to gather data on the bench scale performance of several promising technologies that might assist in the remedial activities at the site. These technologies include:

- Chemical oxidation to reduce volatile organic compounds (VOCs) in lagoon oils, drilling muds, or TPH-impacted soil; and
- Additives to enhance the pumpability of lagoon oils and drilling muds.

This Addendum describes the type of data to be collected and the technical approach that will be used for bench scale testing in the lab. Phase IX does not include any further collection of samples other than segregating already existing samples for use by the vendors described herein. Sufficient quantities of the lagoon tars, TPH-impacted

soils and the drilling muds were collected in 55-gallon drums and stored onsite during the implementation of previous phases.

Objectives for Phase IX include:

- Evaluating the effectiveness of chemical oxidation treatment to reduce emissions (< 50 ppm, see Section 2) and TPH concentrations (<500 to 1,000 ppm, see Section 4) in tars, soils and drilling muds. This technology may potentially be more cost-effective than thermal desorption treatment.
- Exploring the potential use of additives to facilitate pumping lagoon oils and drilling muds. Preliminary discussions and testing with vendors indicates it is feasible to fluidize the drilling muds and lagoon tars by adding a 20% solution of an organic, water-based solvent. Chemical and physical testing will need to be performed to confirm the suitability of this material for handling and waste disposal.

PNL will conduct the treatability studies in cooperation with experienced technology vendors/suppliers and treatability testing subcontractors. PNL has conducted extensive planning and discussions with several companies experienced in treatability testing. Treatability studies will be 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.”

1.3 Addendum Organization

The remainder of this Addendum is organized into the following sections:

Section 2.0 – Description of Waste Types: Provides descriptions of the physical properties of the four primary waste types to be tested during this phase.

Section 3.0 – Treatment Technology Descriptions: Provides overviews of the three treatment approaches to be evaluated, either qualitatively or quantitatively, during this phase of work.

Section 4.0 – Phase IX Treatability Testing Approach: Provides descriptions of the test objectives, general approach, test design and parameters, and residuals and data management techniques.

Section 5.0 – *Estimated Schedule*: Provides an estimated schedule for conducting the Phase IX bench studies.

Section 6.0 – *Reporting*: Summarizes the topics to be included in the final report.

Section 7.0 – *References*: Presents a listing of the references cited in this report.

2. DESCRIPTION OF WASTE TYPES

2.1 Background

Most of the materials present around or beneath the lagoon oily tar and/or the drilling mud consists of TPH-impacted soils, construction debris, or miscellaneous fill. The impacted soil, construction debris, and fill materials can be handled by standard construction excavation equipment, such as long reach excavators, backhoes or front-end loaders. However, the lagoon materials and the drilling muds pose two unique challenges for handling:

- Lagoon materials and drilling muds, along with the TPH-impacted soils, emit VOCs (above 50 ppm) when disturbed during excavation. South Coast Air Quality Management District (SCAQMD) Rule 1166 specifies that materials that emit VOCs at or greater than 50 ppm would require treatment unless disposed of offsite at an appropriate disposal facility within 30 days of excavation.
- The lagoon tars and drilling muds are semi-solid and flow under their own weight. Pumping the lagoon tars and muds may be an attractive alternative to *in situ* stabilization and/or standard excavation techniques to remove these materials.

Because the site in its early years was used primarily for disposal of drilling waste from oil production operations, a significant volume of the materials described above must be handled during any contemplated remedial action program. Mitigation of VOC levels during remedial action is therefore critical in any contemplated remedy. As such, the ability to partially or completely treat these materials onsite to reduce the VOC emissions to less than 50 ppm (when disturbed) will therefore be evaluated during Phase IX.

2.2 Lagoon Oily Tars

Flowable heavy oils and tars are present in the top 3 to 5 feet of Lagoons 1, 2, and 3, based on field investigations conducted to date, which have been limited to sampling around the perimeter within reach of standard construction equipment. The same materials also form an approximately 6-inch thick crust in places on top of Lagoons 4 and 5. The estimated volume of these materials is approximately 16,500

cubic yards. Based on the Pilot Study No. 3 fieldwork, though these materials do not generate significant emissions when disturbed, 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 fly ash/cement kiln dust applied *in situ* using special mechanical equipment. Since removal of semi-solid materials by pumping could be more efficient than conventional excavation, the potential of additives to fluidize the lagoon oily tars to facilitate pumping will be evaluated during this Phase of work.

2.3 TPH Impacted Soils

TPH-impacted soils represent a significant portion (approximately 325,000 cubic yards) of the potential waste types to be handled at the site. This soil, which consists of both imported fill and native soil, appears to be impacted with TPH 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, TPH-impacted soils exhibit the potential for VOC emissions greater than 50 ppm. During Phase IX, treatment of TPH-impacted soil to reduce VOCs below 50 ppm will be evaluated.

2.4 Drilling Muds

The drilling muds also represent a significant portion (approximately 550,000 cubic yards) of the potential waste types to be handled at the site. Because of the fine-grained, clayey nature of some of these materials, they may retain levels of VOCs that exceed the SCAQMD limit that could be released when these materials are disturbed. Phase IX will include evaluation of treatment of drilling muds to reduce VOCs to below 50 ppm. In addition, due to the liquid nature of the drilling muds, the potential addition of amendments to fluidize the drilling muds will also be explored in this Phase.

In general, the drilling muds found in the lagoon and former lagoon areas have distinct physical properties:

- 1) Drilling muds found in the current lagoons are soft, liquid-saturated and flow under their own weight. Based on measurements made during drilling, this material may be generally characterized as having relatively low strength (penetration test blow-counts of 3 blows-per-6-inches or less). TPH concentrations in lagoon areas are also one to two orders of magnitude higher than levels measured across the rest of the site.
- 2) Drilling muds found in the parts of the former lagoon areas (particularly on the west side of the site) are stiff, relatively dry and will support a

sidewall when excavated. This material has higher strength than lagoon material with standard penetration test blow counts greater than 3 blows-per-6-inches. This material is also mixed with coarser-grained drill cuttings and typically not noted as being “saturated”.

3.0 TREATMENT TECHNOLOGY DESCRIPTIONS

The following sections include descriptions of three technologies, *ex situ* thermal desorption, *ex situ* chemical oxidation, and sludge fluidization. Those technologies are considered potential candidates for remediation of petroleum hydrocarbons and emissions control of heavy oils/tars, impacted soils and drilling muds at the site. Bench scale testing of the *ex situ* chemical oxidation and sludge fluidization technologies will be performed on various wastes from the site during this phase. The effectiveness of thermal desorption for the treatment of hydrocarbon-impacted soils and drilling muds is well demonstrated and documented. Bench scale testing of this technology is therefore not planned. Instead, the technology and its application to the site have been evaluated based on a review of applicable literature. This evaluation is summarized below.

3.1 Ex Situ Thermal Desorption

A qualitative study was performed by PNL on the efficacy of *ex situ* thermal desorption (ESTD) treatment for remediation of hydrocarbon-impacted soils and oil-based drilling muds to reduce TPH levels and emissions. ESTD works by heating the soil to a target temperature causing volatile constituents (petroleum hydrocarbons, water, and other target compounds) to volatilize and separate from the soil. The volatilized organic vapors and water off-gas components are collected and generally treated by one or more off-gas treatment technologies. Types of off-gas treatment technologies include filtration, wet-scrubbing, vapor-phase granular activated carbon adsorption, and thermal oxidation.

Two common ESTD designs are the rotary dryer and thermal screw. Rotary dryers (kilns) are horizontal cylinders that can be direct or indirect fired/heated (meaning heat is applied either directly or indirectly to the surface of the contaminated medium). A process schematic for an indirect heated rotary kiln ESTD system sold by On-Site Technology, LLC, is shown in Figure 3-1. Contaminated soil is conveyed into the kiln, which consists of a rotating, closed heat-jacketed barrel. The unit is electrically powered and fired with natural gas or diesel fuel. The material is fed into one end of the unit and as it travels the length of the heated drum, organics and water are volatilized. The off-gas vapor is captured in a vapor-liquid separator, with the non-condensable vapors recycled back to the unit for recombustion. The condensed liquid is separated out into water and organic phases. The treated soil is cooled by water spray in the discharge end of the drum and discharged through an auger. Processing rates for ESTD systems vary widely and are a function of the moisture/oil content of the

impacted material, the grain size of the soil particles, and the residence time required to meet the performance objectives. Expected unit treatment costs for ESTD for the Ascon Site waste types, based on vendor input (see below), would be on the order of \$70 to \$100 per ton.

The effectiveness of the ESTD approach for remediation of hydrocarbon impacted soil and drilling muds has been well documented in the available literature. For instance, there are seven ESTD projects involving treatment of petroleum hydrocarbon impacted soil identified in EPA's year 2000 "Remediation Technology Cost Compendium" (EPA, 2001) report. These seven sites involved treatment of between 5,000 and 105,000 tons of impacted soil. The EPA report also presented cost versus cleanup volume statistics based on the projects reviewed and included in the database. Abstracts of two of the case studies presented in EPA's report are presented on the Federal Remediation Technology Roundtable's website at <http://www.frtr.gov>.

Several vendors were contacted regarding documented experience in treating oil-based drilling muds via ESTD. One vendor, On-Site Technologies, LLC (Houston, Texas), has documented experience at 11 sites, with up to 350,000 tons processed, where drilling muds containing 10 to 30 percent oil were treated down to below 300 to 1,000 mg/kg TPH as required to meet treatment objectives. A summary table listing the main parameters of interest for these 11 projects is provided in Appendix A.

3.2 Ex Situ Chemical Oxidation

While ESTD is a well documented and widely used technology for treatment of hydrocarbon waste, use of chemical oxidation technology is less widely applied for treatment of hydrocarbons in soil and drilling mud. Nevertheless, because of its relative simplicity and low cost of treatment, this technology may be potentially more cost-effective than ESTD for achieving the treatment objectives. As described below, in addition to mechanical simplicity, chemical oxidation has several advantages over ESTD in that it does not require emissions treatment, product stream cooling, or potential for management of liquid wastes. It is also less energy intensive due to far fewer moving parts and processes and does not require a heat source for treatment.

In the *ex situ* chemical oxidation process, a strong chemical oxidant such as permanganate in a water solution is mixed with the waste matrix 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. The target

contaminants are generally oxidized to hydrogen and oxygen. The reactions are slightly exothermic, but do not impact the handling of the treated product (e.g., there is no increase in temperature).

In an accelerated *ex situ* oxidation process promoted by Environmental Technology Solutions (ETS), hydrocarbons in the soil/clay matrix are oxidized by reacting the soil/clay with an ionized water solution containing hydroxyl free radicals and permanganate using a proprietary reaction process. Figure 3-2 shows the generalized treatment approach utilizing ETS' process for treatment of hydrocarbon impacted soil waste. The ETS process is described 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* mixing process, where it is mixed with the process reagents. Mixing takes place typically for a few minutes, after which the waste is removed to stockpiles for curing, which can take from a few to several days.

The specific design parameters for the bench testing proposed by ETS for this Phase are presented in Section 4 and Appendix B. As shown in these sections, the *ex situ* chemical oxidation treatability test will measure the effectiveness of the process in reducing TPH and VOC concentrations in soil, as well as VOC emissions [measured by a photoionization detector (PID) before, during and following treatment].

3.3 Sludge Fluidization

3.3.1 Summary of Previous Testing

In the 2000 Ascon Feasibility Study report, J&W Engineering, LTD., conducted bench and field pilot scale studies of *ex situ* solvent extraction on the heavy tars in Lagoons 1 and 2. For the bench testing, J&W used a hot water bath and proprietary surfactants to evaluate the pumpability of the heavy tars in Lagoons 1 and 2 and to determine if any recoverable oil could be separated from the tars (J&W, 1998). For the test, the tars were added to a 140°F to 180°F hot water bath, the surfactants mixed in for several minutes and the phases were allowed to separate. The mixtures were tested in the ratio of approximately 75% water, 24% tar and 1% additives. Laboratory analyses were performed on the mixtures where phase separation was successful. The results of the tests were as follows:

- Separation of the oil phase from water and sediment occurred for 2 of the 3 samples tested and the viscosity of the mixture was reduced and the fluid properties of the mixture were improved while heated.
- Small amounts of VOCs, SVOCs, and metals from the oil moved into the water phase.
- Emissions measured with a PID were minor during mixing.
- Mixing times were less than 10 minutes; separation times were less than 30 minutes (when successful); and pumping rates of at least 3 gallons per minute were used.

A follow-up pilot test was performed by J&W on an undetermined volume of tar from Lagoon 2 (J&W, 1999). The purpose of the field program was to evaluate the air emissions associated with the solvent extraction process and the physical and chemical characteristics of the recovered oil and other products. The test was conducted using 2,700 gallons of water, 1% biosurfactant (identical in volume and type to bench test) and a centrifuge to separate the oil, water and sediment. The process rate used was 25 gpm. A summary of the test results is as follows:

- Cross-contamination was observed in the water and sediment phases, indicating that both phases would require treatment (analytical results of the water were similar to the oil, and the sediment contained high TPH and lead).
- Excessive emissions were not generated.
- The characteristics of the recovered oil (low BTU due to high liquid content, high viscosity) appeared to suggest that resale was unlikely.

3.3.2 Petromax Technologies Fluidization Process

The above description of the *ex situ* solvent extraction processes indicates that the lagoon tars are pumpable through a heated water bath process without generating significant emissions, but there are major drawbacks (such as cross contamination of the added water, and the shear volume of water required) to the process that would inhibit implementability. In addition, no testing was performed on the drilling muds, which are a significant waste stream that may be suitable for fluidization.

PNL contacted vendors to investigate the efficacy of 1) adding amendments to fluidize the lagoon tars and drilling muds without using a hot water bath approach; and 2) recovering product from a fluidized waste stream. Petromax Technologies Inc. (Petromax) responded as a firm with experience fluidizing crude oil tank bottoms to

facilitate easy cleanup. Petromax identified processes and products that may be applicable for emulsion breaking, phase separation and product recovery of lagoon tars and drilling muds at the Ascon Landfill Site through pre-test analysis (see Appendix B).

As applied to fluidization of drilling muds and heavy oils, Petromax's products work by encapsulating and permanently modifying the opposing surface charges between hydrocarbon molecules and the inorganic particles (i.e., solids) to which they adhere (see Appendix C for details). 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 mixing¹ the waste while simultaneously injecting the product either *in situ* or *ex situ*.

A complete description of the Petromax Fluidization Bench Test is presented in Appendix B. The test will include evaluation of VOC emissions during mixing (product application) and the chemical characteristics of the treated product. Appendix C provides a few slides illustrating the Petromax sludge conditioning mechanism of action described above.

¹ 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 fluidizing 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.

4. PHASE IX TREATABILITY TESTING APPROACH

4.1 Introduction

The basic scope of the Phase IX treatability study is to conduct a bench-scale, vendor- and technology-specific testing program for the following two treatment technologies that are considered applicable to treat different waste materials in the lagoons and former lagoon areas at the Ascon Landfill Site:

- *Ex Situ* chemical oxidation
- Sludge fluidization

The bench-scale treatability study will evaluate performance of the above-mentioned treatment technologies for possible full-scale application at the site. The objectives of the Phase IX treatability study are to:

- Ascertain the effectiveness of a chemical oxidation treatment in reducing VOC emissions (< 50 ppm) and TPH concentrations (<500 to 1,000 ppm) in tars, soils and drilling muds. This technology may potentially be more cost-effective than thermal desorption treatment, whose success in achieving this objective is well documented.
- Determine the potential for additives to be used to facilitate pumping lagoon tars and drilling muds and the suitability of this material for waste disposal, in terms of chemical concentrations and fuel (BTU) content.

4.2 General Approach

Upon approval of this workplan addendum from the DTSC, PNL will conduct the studies described herein in cooperation with experienced technology vendors/suppliers and subcontractors. Key elements of the treatability study approach are described below. The *ex situ* chemical oxidation tests will be performed in the vendor's designated laboratory. The sludge fluidization technology tests are planned to be conducted at the Ascon Landfill Site, with supervision from PNL personnel. Table 4-1 presents a summary of the proposed tests and key parameters.

Samples of Waste Materials: Samples of waste material have been previously collected in sealed 55-gallon drums from Lagoons 1 to 5 and the former lagoon areas and are currently staged at the site. These samples will be provided to each of the

technology vendors completing the treatability testing. Samples will be 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 will be retrieved from the onsite drum storage area.
- The drums will be opened and a pre-determined number of 5-gallon buckets will be filled with the selected material using shovels and/or hand trowels.
- After sampling, the 55-gallon drums will be re-sealed and returned to the onsite drum storage area.
- The 5-gallon bucket samples will be properly labeled and sealed with chain-of-custody labels. As appropriate, split samples will be collected from the 5-gallon buckets and shipped along with chain-of-custody documentation to the selected laboratories for pre-treatment waste characterization testing.
- All non-disposable implements used in collecting the samples from a specific drum will be decontaminated with a Liquinox® solution, prior to collecting additional samples.
- The field workers will adhere 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: As described above, split-samples will be collected from the 5-gallon buckets as appropriate and shipped to the selected laboratories (see Table 4-1) for pre-treatment waste characterization analyses. These analyses will be conducted to provide a basis to compare the effectiveness of *in situ* chemical oxidation (pre-treatment samples will not be collected for the sludge fluidization test – see Table 4-3). The test parameters that will be considered for *ex situ* chemical oxidation are presented in Tables 4-2. Note that the same analytical testing laboratories used during the previous Pilot Study No. 3 analyses will be used for chemical analyses of untreated and treated waste materials.

Sample Locations: Sample locations listed in Table 4-1 are shown on Figure 4-1. Sample locations listed in Table 4-1 were chosen (for the drilling muds and TPH-impacted soils) based on emissions data collected during Pilot Study No. 3. As noted in Table 4-1, there are several sample locations chosen as candidates for each test and waste type. Sample locations will be verified in the field for representativeness prior to collection. For instance, VOC emissions from samples will be measured using a photoionization detector (PID). The final “sample” collected for each waste type may

consist of a composite from one to several sampling locations, depending on the results of these field determinations.

Treatability Test: Vendors or vendor-selected third party treatability laboratories (see ETS test protocol, Appendix B) will perform the treatability tests. PNL or other independent third parties will have the option of providing oversight during testing (when conducted at an offsite laboratory). Various treatment technology parameters such as the mixing or cure time will be modified to optimize the process and to meet performance criteria. The test parameters that will be considered for each of the technologies during the test are shown in Tables 4-2 and 4-3.

Treatment Product Characterization: Samples of treated materials and any by-products will be collected and physical and chemical testing will be performed. These tests will provide an indication of the effectiveness of the treatment approach to meet the specific goals for each of the materials tested. The test parameters that will be considered for each of the technologies for post-test characterization are presented in Table 4-2 and 4-3.

Verification, Data Analysis, and Interpretation: To verify the treatment of the wastes by the technology vendors (or their designated testing laboratories), a representative from PNL or an independent third party may witness and document treatment of the wastes sent to the technology vendors (for those tests conducted at offsite laboratories). In addition, PNL or the third party representative may elect to collect samples of the treated material (product) for independent analysis. Documentation from these activities will be included in the Second FS report.

Upon completion of the treatability studies, data generated will be analyzed and interpretation of the data will be provided based on the treatability study objectives.

4.3 Test Design and Parameters

PNL and the treatability subcontractors have identified various tests that will be completed during Phase IX. These tests can be categorized into three areas:

- Pre-Treatment Waste Characterization
- Characterization During Treatment
- Treatment Product Characterization

A list of test parameters for each technology is presented in Table 4-2 and 4-3. During the testing, additional parameters may be identified based on the treatment being completed.

4.4 Test Protocols

PNL and the treatability test vendors have developed a summary of the procedures that will be followed during the bench testing program. These procedures are presented in Appendix B. These procedures also include an overview of the technology and test, and a list of the materials and equipment that will be used.

4.5 Residual Materials Management

Residual materials (unused samples of the wastes, spent reagents and formulas, and product from the treatment process) generated from the treatability studies will be disposed of by the treatability subcontractors in accordance with appropriate state and federal regulations. These materials will be 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.

4.6 Data Management, Analysis and Interpretation

Raw data from the treatability studies will be presented in tabular or graphic form by the vendors for PNL review. These data will be analyzed to verify data quality. All data will be checked to assess precision, accuracy, and completeness. Interpretation of the data will be based on the study objectives presented in Section 4.1.

5. ESTIMATED SCHEDULE

The duration of Phase IX Technology Bench Scale Testing is anticipated to be approximately 1 month. The timeframe for completion of this work will be incorporated into the master schedule for the Second Feasibility Study (SFS).

6. REPORTING

The findings of this study will be incorporated into the Second Feasibility Study Report. The findings will include, but not be limited to, the following topics:

- Overview: Waste type descriptions, treatment technology descriptions
- Treatability study approach: Test objectives and rationale; test parameters; test procedures; materials and equipment; sampling and analysis and data management
- Results and discussion: Data analysis and interpretation; comparison to test objectives; quality assurance/quality control
- References and appendices: References of guidance and other documents; raw data and laboratory reports; and standard operating procedures

7. REFERENCES

- EPA, 1992, Final, Guide for Conducting Treatability Studies under CERCLA, Risk Reduction Engineering Laboratory, Office of Research and Development and Office of Emergency and Remedial Response, Office of Solid Waste and Emergency Response.
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TABLES

TABLE 4-1
BENCH SCALE TREATABILITY TEST SUMMARY
Ascon Landfill Site

	Vendor	Testing Location	Technology Type	Sample Locations ⁽¹⁾⁽²⁾	Tests/Analyses ⁽³⁾	Proposed Laboratories for Analytical Tests
TPH-Impacted Soil	Environmental Technology Solutions	Oakland, CA	Chemical Oxidation	PNL-4, PNL-5, PNL-6	Grain size (ASTM D1140), Moisture content (ASTM D2216), TRPH (EPA 418.1), EFH/GRO (EPA 8015B), and VOCs (EPA 8260B) in untreated samples; TRPH and VOCs in treated samples; VOC emissions (by PID) in untreated and treated samples, and during treatment.	Del Mar Analytical (Irvine, CA); Prima Environmental (Sacramento, CA)
	Various (OnSite Technology, LLC; Mobile Enviromental Technologies, Inc.)	N/A - Qualitative Evaluation	Thermal Desorption	N/A - Qualitative Evaluation	N/A - Qualitative Evaluation	N/A - Qualitative Evaluation
Drilling Muds - Lagoons	Environmental Technology Solutions	Oakland, CA	Chemical Oxidation	PNL-L4A; PNL-L5B	Grain size (ASTM D1140), Moisture content (ASTM D2216), TRPH (EPA 418.1), EFH/GRO (EPA 8015B), and VOCs (EPA 8260B) in untreated samples; TRPH and VOCs in 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	Los Angeles, CA	Sludge Fluidization	PNL-L4A; PNL-L5B	TRPH (EPA 418.1), VOCs (EPA 8260B), Fish Bioassay (See Notes), BTU/lb (ASTM D240), 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	Thermal Desorption	N/A - Qualitative Evaluation	N/A - Qualitative Evaluation	N/A - Qualitative Evaluation
Drilling Muds - Former Lagoon Areas	Environmental Technology Solutions	Oakland, CA	Chemical Oxidation	PNL-3; PNL-4; PNL-5; PNL-6	Grain size (ASTM D1140), Moisture content (ASTM D2216), TRPH (EPA 418.1), EFH/GRO (EPA 8015B), and VOCs (EPA 8260B) in untreated samples; TRPH and VOCs in 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	Los Angeles, CA	Sludge Fluidization	PNL-3; PNL-4; PNL-5; PNL-6	TRPH (EPA 418.1), VOCs (EPA 8260B), Fish Bioassay (See Notes), BTU/lb (ASTM D240), 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	Thermal Desorption	N/A - Qualitative Evaluation	N/A - Qualitative Evaluation	N/A - Qualitative Evaluation
Lagoon Tar	Petromax Technologies	Los Angeles, CA	Sludge Fluidization	PNL-L1A; PNL-L2A	TRPH (EPA 418.1), VOCs (EPA 8260B), Fish Bioassay (See Notes), BTU/lb (ASTM D240), 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) Candidates for collection - 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 4-1 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 4-2
ETS CHEMICAL OXIDATION TREATABILITY TEST PARAMETERS
Ascon Landfill Site

Pre-Treatment Waste Characterization			
Matrix	Parameter	Purpose	Methodology
Soil/Drilling Mud	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
Characterization During Treatment			
Matrix	Parameter	Purpose	Methodology
Soil/Drilling Mud with Reagents	Cure Time	To determine total reaction time required per batch.	Bench Test
	VOCs (vapor)	Quantify potential emissions during treatment.	Calibrated PID meter
Treatment Product Characterization			
Matrix	Parameter	Purpose	Methodology
Treated Soil or Drilling Mud	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 4-3
PETROMAX SLUDGE FLUIDIZATION TREATABILITY TEST PARAMETERS
Ascon Landfill Site

Pre-Treatment Waste Characterization			
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.			
Characterization During Treatment			
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
Treatment Product Characterization			
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 4-1
	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
	BTU/lb	To estimate fuel value of treated product.	ASTM D240

Notes:

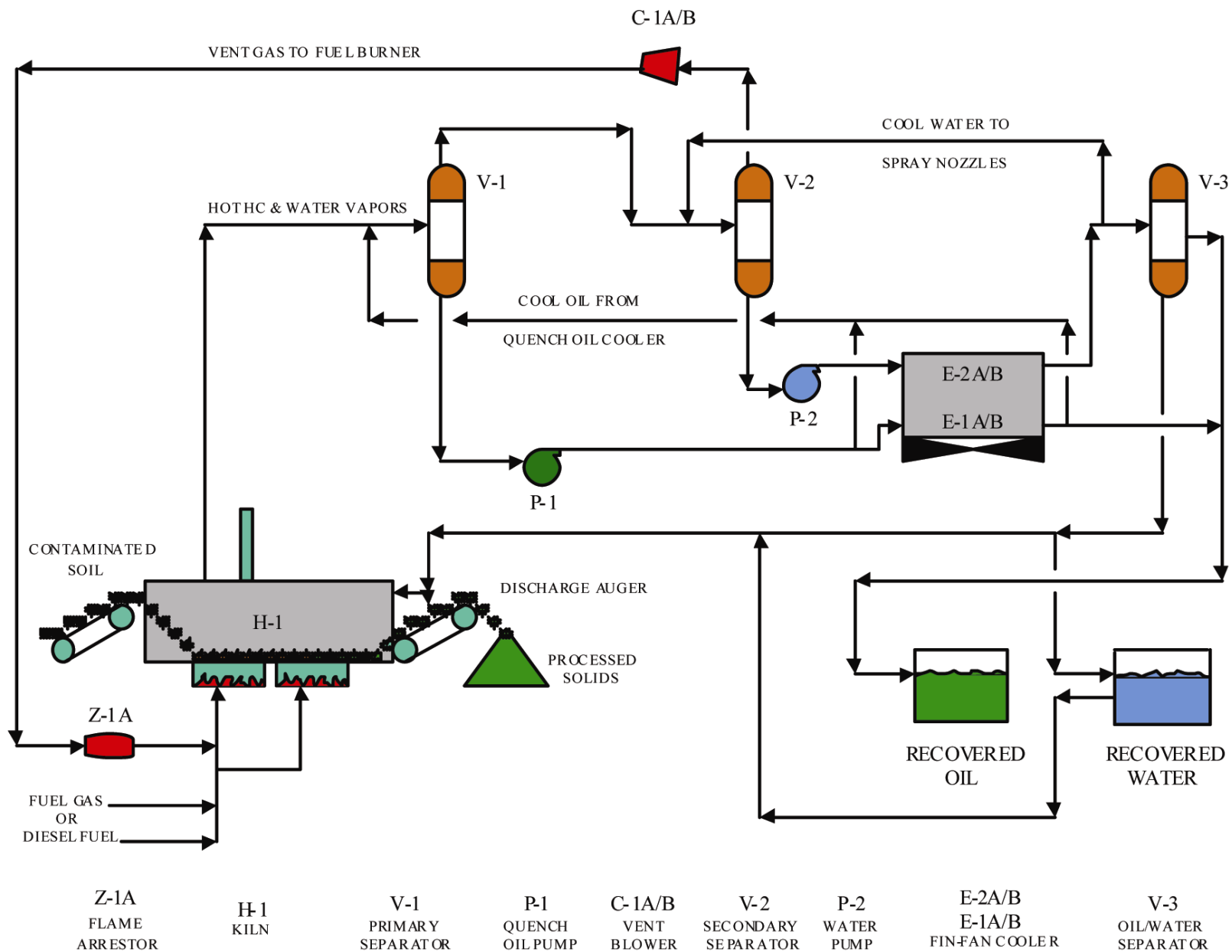
TRPH - total recoverable petroleum hydrocarbons

VOCs- volatile organic compounds

BTU - British Thermal Unit

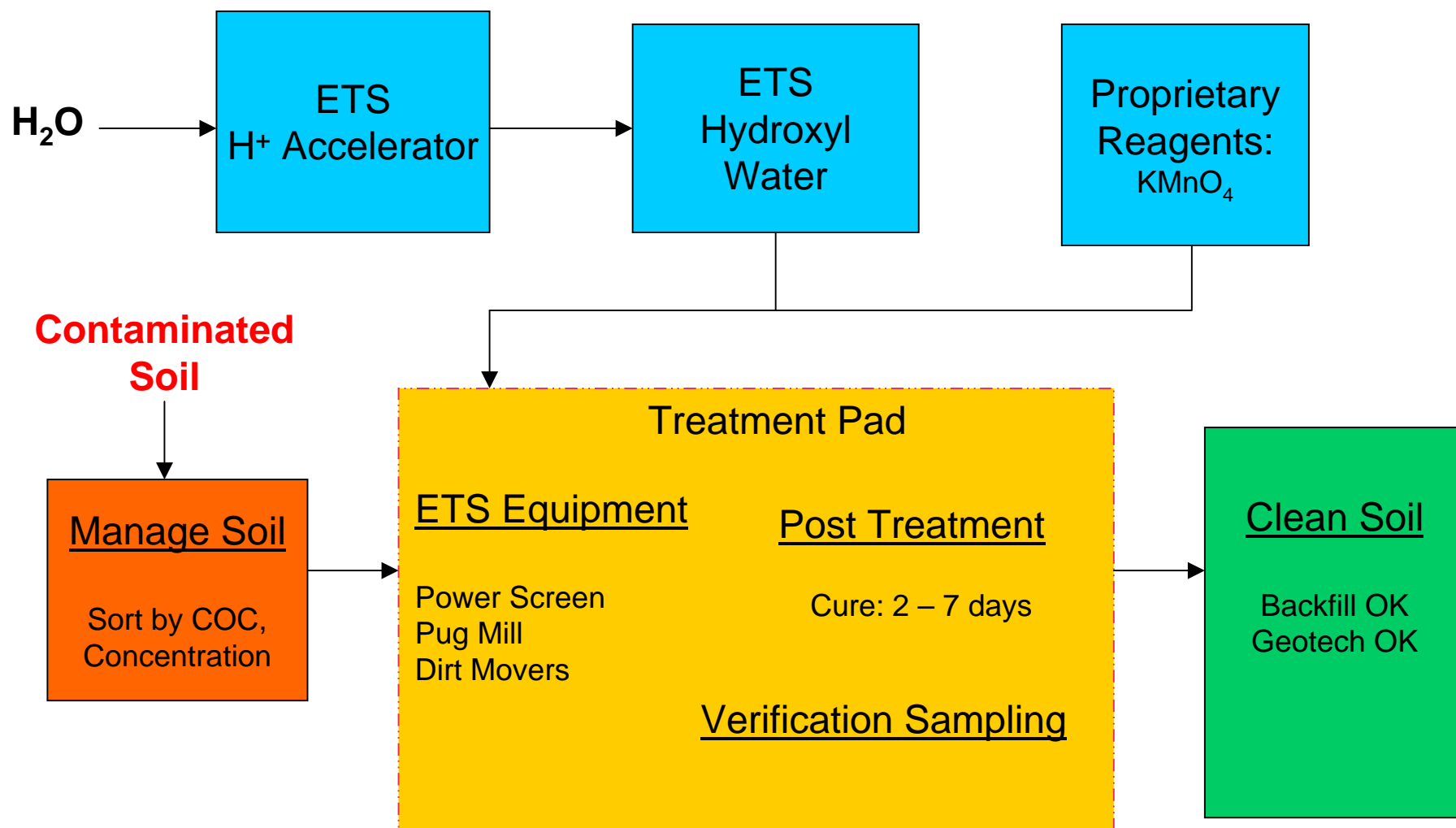
ASTM - American Society of Testing Materials

FIGURES



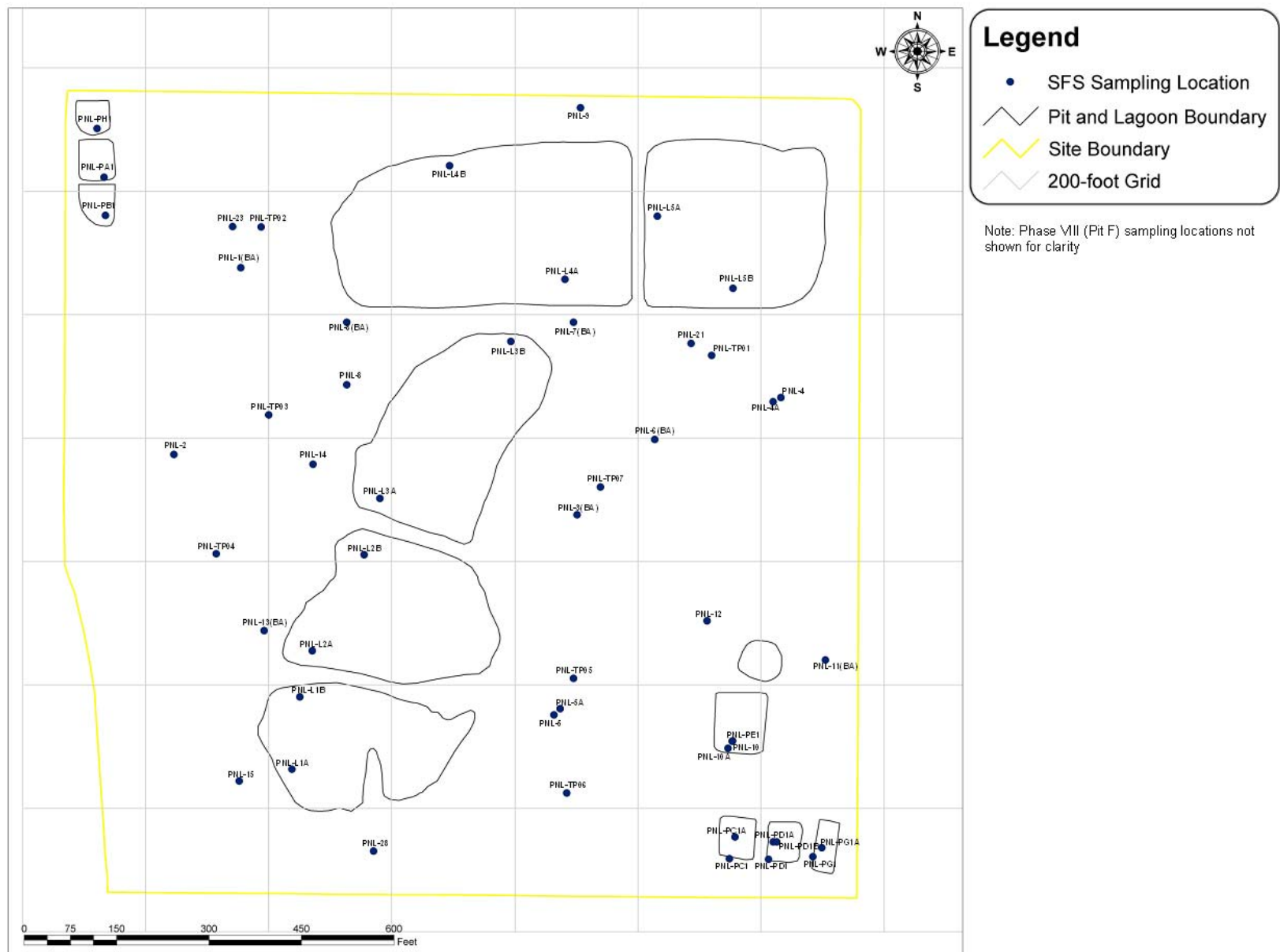
Onsite Technology Indirect *Ex Situ* Thermal Desorption -- Process Flow Diagram

Figure 3-1



ETS *Ex Situ* Chemical Oxidation -- Process Flow Diagram

Figure 3-2



Technology Bench Scale Testing Sampling Locations [Includes additional Pilot Study No. 3 (SFS) Locations]

Figure 4-1

APPENDIX A

CASE STUDIES: *EX SITU* THERMAL DESORPTION TREATMENT OF DRILLING MUDS

Appendix A
Case Studies: *Ex Situ* Thermal Desorption Treatment of Drilling Muds

Summary of Drill Cuttings Treated

Client	Location	Tons Processed	Mud Weight (ppg)	Oil Content before Treatment % by Volume	Oil Content after Treatment	Process Temperature	Process Rate (TPH)	Water Quality	Base Oil Type
Exxon	Chalk Creek, UT	5,000.00	14 - 18	19.50%	<500ppm	550 F	4.2	<250 ppm	Escad
Exxon	Fogarty Creek, WY	3,350.00	14 - 18	21%	<300ppm	550 F	4.6	<250 ppm	Escad
Texaco	Table Rock, WY	2,835.00	14 - 18	32%	<300ppm	550 F	4.5	<250 ppm	Escad
LL&E	Lysite, WY	5,650.00	14 - 20	22%	<300ppm	600 F	5.5	<250 ppm	Diesel
British Petroleum	Yopaul, Colombia	347,782.00	*Unknown	28%	<500ppm	600 F	4.7	<250 ppm	Diesel
ELF Aquataine	Granare, Venezuela	2,133.00	14 - 18	27%	<1000ppm	600 F	5.2	<250 ppm	Diesel
PDVSA	Maracibo, Venezuela	4,642.00	*Unknown	20%	<1000ppm	600 F	4.5	<250 ppm	Diesel
Shell	Maracibo, Venezuela	13,308.00	*Unknown	15%	<1000ppm	600 F	4.9	<250 ppm	Diesel
OSCA	Villahermosa, Mexico	18,086.50	*Unknown	15%	<1000ppm	600 F	5.1	<250 ppm	Diesel
Pemex	Villahermosa, Mexico	50,006.00	*Unknown	20%	<1000ppm	600 F	4.8	<250 ppm	Diesel
PASA	Villahermosa, Mexico		*Unknown	10%	<1000ppm	600 F	5.2	<250 ppm	Diesel

*Unknown Mud Weights on these projects due to System being located at a central site, Processing cuttings from multiple locations

Process Rate is Based on 1 Unit. Some projects listed above utilized a multiple Unit Setup. Therefore the Process Rate listed above could be multiplied by the number of Units needed for your project.

APPENDIX B

PHASE IX TREATABILITY TEST PROTOCOL SUMMARIES

CHEMICAL OXIDATION TREATABILITY TEST (TPH REDUCTION AND EMISSIONS CONTROL)

**By Environmental Technology Solutions
Oakland, California**

Overview

Environmental Technology Solutions' (ETS) patented *ex situ* chemical oxidation process is part of a process option for remediation of total petroleum hydrocarbon contamination in soils and drilling muds from the Ascon Landfill Site. The objective of this treatability study is to determine if this technology is effective in reducing VOC emissions via reduction in low-end hydrocarbon concentrations to facilitate replacement of waste materials at the Ascon Landfill Site. Through performing the bench scale test described herein, ETS will determine the optimal mixture, sequence, and schedule of reagents, and ultimately the unit cost required for treatment. The treatability tests will be performed by a third party environmental laboratory, PRIMA Environmental (Sacramento, California), with guidance from ETS.

Approximately 10 pounds of sample will be collected for each of the 3 waste types requiring testing from 55-gallon drums at the site: impacted soils, and drilling muds from lagoon and non-lagoon areas. Specific sample collection locations for each waste type are listed in Table 4-1. Prior to and following testing, split samples will be analyzed for chemicals of concern listed in Table 4-1/4-2. Pre-test samples will also be analyzed for physical parameters (grain size and moisture content) that will influence reagent selection and treatment residence time. Emissions will be measured with a calibrated photoionization detector (PID) prior to, during and following testing.

ETS will separately determine the effectiveness of a patented emission control treatment (OdorPro™) on the same samples of impacted soil and drilling mud. The vendor will provide a qualitative evaluation of emission control, based upon PID measurements performed in the laboratory.

Laboratory Equipment and Materials Needed

- 8 oz. glass sample jars
- ETS Hydrogen Accelerator
- Mixing vessel
- Electrical/mechanical mixer
- ETS reagents
- Trowels
- Rubber gloves
- Photoionization detector (PID)
- Miscellaneous laboratory equipment

Steps for Bench Emissions Testing

1. Use a hand-held PID to determine the level of VOC emissions from the bench sample bucket. First allow headspace vapors to dissipate before making the PID measurement. Disturb the surface of the soil and re-measure, recording all measurements. Repeat the process until confidence is achieved regarding levels of emissions. Record all readings.
2. Using a hand-held sprayer, apply the ETS Odor Pro™ treatment to the surface of the soil in the bench sample bucket, and re-measure using the PID. Repeat the process three times to verify initial observations. Record all readings.

Steps for Bench Treatability Testing

1. Collect pre-test samples using laboratory-provided glass sample jars from the 10 pounds of material collected per waste type. To preserve levels of volatile organic contaminants (VOCs) in the pre-test sample, collect samples from the “interior” of the bench sample, and follow appropriate protocols for sample labeling, preservation, etc. Submit samples to Del Mar Analytical, Inc. for analysis for total recoverable petroleum hydrocarbons (TRPH), extractable fuel hydrocarbons and gasoline range organics (EFH/GRO), and VOCs, as defined in the Work Plan. Submit remaining sample to Prima ENVIRONMENTAL. Physical analyses (grain size and moisture content) will be performed by PRIMA Environmental.
2. Examine bench samples for the presence of any foreign objects, such as plant parts, pieces of metal, trash, and any other visible properties or characteristics that could affect the outcome of the bench-scale testing. Remove any materials (by hand or with screen) that is greater than about 1-inch diameter. Document significant observations.
3. If necessary, dewater drilling mud samples (e.g., by adding lime) prior to reagent application. Document significant observations.
4. Apply and mix reagents with the soil/mud samples using a mechanical mixer for a few minutes until homogenous. Reagents will be injected using a small metering pump. At this time, 3 different recipes are planned plus one control (see below table of test conditions). Ensure that amounts, sequences, and timing of reagents are quantified and documented. All procedures and results will be documented.
5. After an approximate 48-hour cure time¹, collect post bench test samples for analysis using the same procedures described above. Note material consistency and any special handling requirements.

¹ Based on vendor experience with TPH reduction in soils at other sites, average cure time is about 48 hours. Actual cure time will be documented as a significant test parameter.

Test Conditions – ETS Treatability Test

Test	Reagent Dose	Water Type
Control ²	None	Tap water
Low	1.5 g/kg soil	ETS Accelerator Water
Medium	2.5 g/kg soil	ETS Accelerator Water
High	5 g/kg soil	ETS Accelerator Water

² The control is designed to show that performance of the ETS process is not due to simply aerating (and in turn, oxidizing) the samples.

SLUDGE FLUIDIZATION TREATABILITY TEST

By Petromax Technology
Los Angeles, California

Overview

Petromax Technology's patented formulas are part of a process option for fluidizing the lagoon tars and drilling muds for removal from the Ascon Landfill Site. Petromax's product works by modifying the opposing surface charges between the inorganics and the crude oil materials that causes their attraction (see Appendix C). This modification is permanent – such that the hydrocarbons will not reattach to the inorganic solids, and allows the oils to flow freely in suspension. Petromax's formulas can be hydroblasted (at 3,000 to 5,000 psi or greater) into the waste material or applied via high shear mixing while simultaneously injecting the product either *in situ* or *ex situ*. These formulas are all water based, inorganic solutions with potentially some organic additives. It is anticipated that tests will be conducted at ambient temperatures. Increased temperatures, while not required, would likely enhance the fluidization process.

The objective of this treatability study is to determine if the fluid properties (e.g., viscosity) of the lagoon tars and drilling muds can be altered to the extent that these materials can be recovered by pumping. The possibility of recovering the oil as a separate phase following treatment will not be considered in this test. If oil recovery is desired, additional amendments, mixing and process steps (e.g., oil separation) would be required in the field. The feasibility of oil separation from the drilling muds is complicated by the presence of debris and other commingled wastes.

Jar samples of drilling mud from Lagoons 4 and 5 and the former lagoon areas (see Table 4-1 for specific locations), and tarry sludge from Lagoons 1 and 2 will be collected for testing from 55-gallon drums at the Site. It is anticipated that one specific Petromax formula will be developed for the lagoon tars, and another for the drilling muds (lagoon and non-lagoon). The formula will be added to the jars and blended with the waste material using a handheld electric shear mixer. Mixing will take place until sufficient time has elapsed for the formula to contact with the inorganic soil particles/hydrocarbons to modify the surface charges to create a permanent suspension. This mixing time is expected to vary from a few seconds to a few minutes depending upon application method and waste characteristics.

Tests will be performed onsite under PNL supervision. PNL will also perform photoionization detector (PID) emissions monitoring as required. Petromax representatives will conduct all other aspects of the treatability program.

Laboratory Equipment and Materials Required

- Bamix[®] handheld mixer
- 1 quart glass containers
- Petromax[®] Formulas
- Liquinox[®] Decon Solution
- Calibrated PID

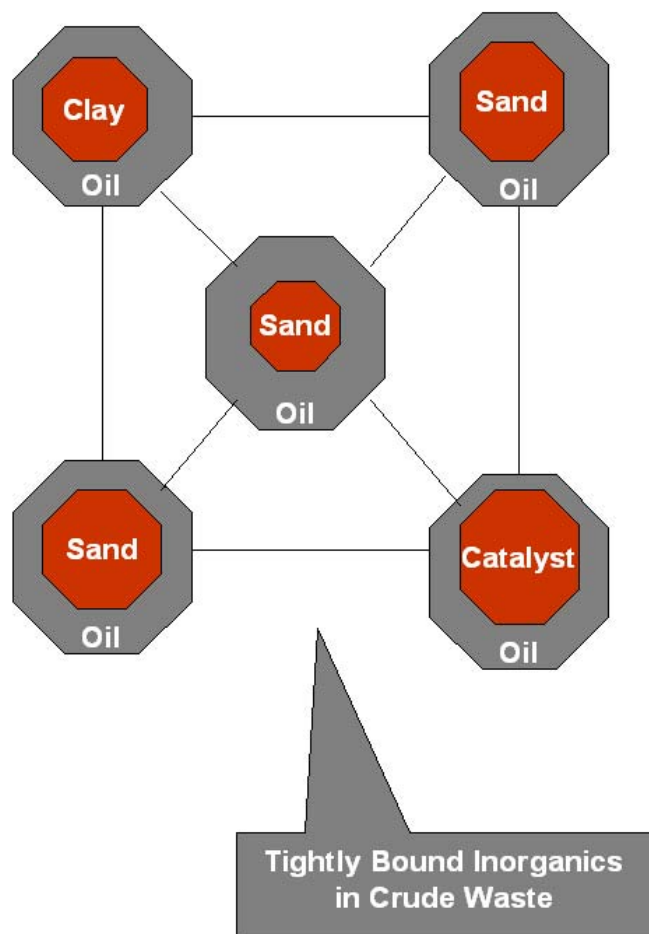
Steps to Perform Tests

- 1) Place representative sample of mud/tar into a glass jar, add Petromax solution to the sample (after blending, there will be an approximate 20 to 30% increase in overall sample volume).
- 2) Mix sample with solution using a handheld shear mixer to provide the appropriate contact time. Monitor the mixing process with a PID and document the measured VOC concentrations.
- 3) Perform a visual evaluation of the product mixture. Solid material should be suspended in Petromax solution. Evaluate fluid properties of mixture for comparison against a known standard using a viscometer or other appropriate testing device (e.g., Marsh funnel). Note any unusual odors, vapors, etc.
- 4) Submit treated liquid sample to Del Mar Analytical, Inc. and Conti Testing Laboratory for analysis for TRPH, VOCs, fish bioassay, viscosity, and BTU content as defined in the Work Plan.
- 5) Repeat steps 1 to 4 above for the other two waste types not tested first iteration. Document final results.

APPENDIX C

PETROMAX SLUDGE FLUIDIZATION MECHANISM OF ACTION

Petromax Sludge Conditioning - Mechanism of Action

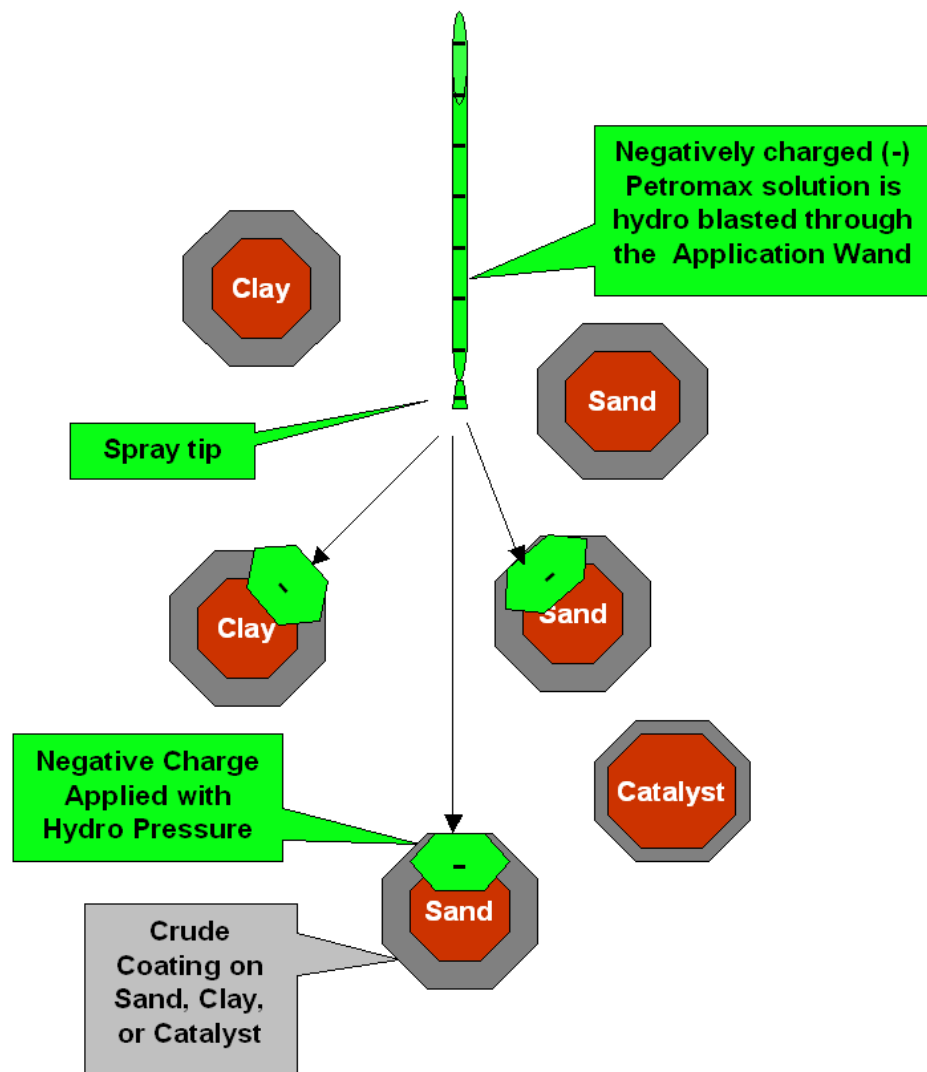


The Problem: Crude oil tank bottoms contain settled inorganic particles which create a very tacky sludge at the bottom of storage tanks. The inorganics and crude are tightly bound by the balance of negative and positive charges in this non-polar environment throughout the sludge.

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.



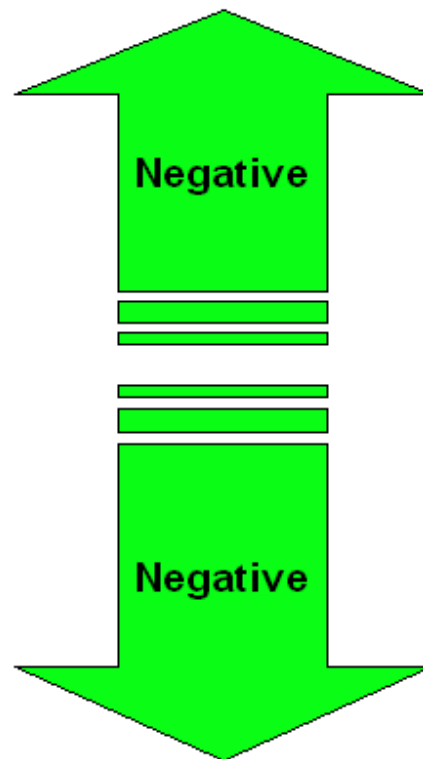
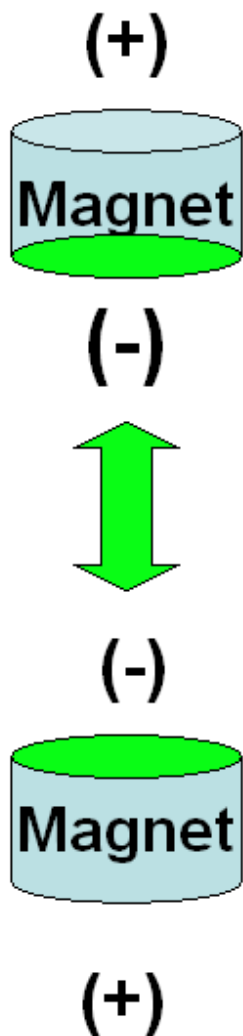
Petromax Technologies
 a subsidiary of **AQUATECH Ecologics, LLC**
 2705 Krim Drive, Los Angeles, CA 90064
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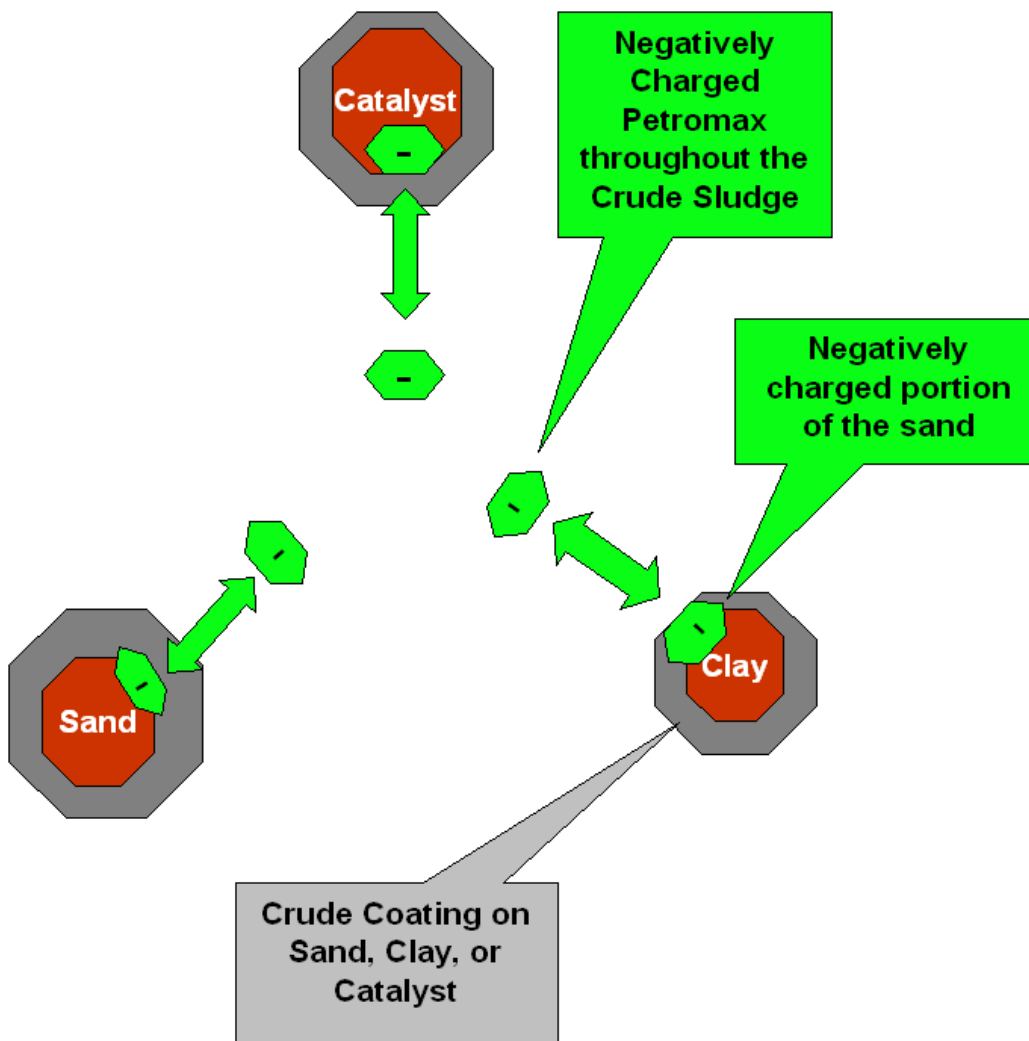
The Solution: The solution is to break the bond between the inorganic particle and the oil. Once the balance of “charges” have shifted to a negative state, the inorganic particles, the waste oil, and the Petromax solution instantly begin to repel from each other.

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.



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.

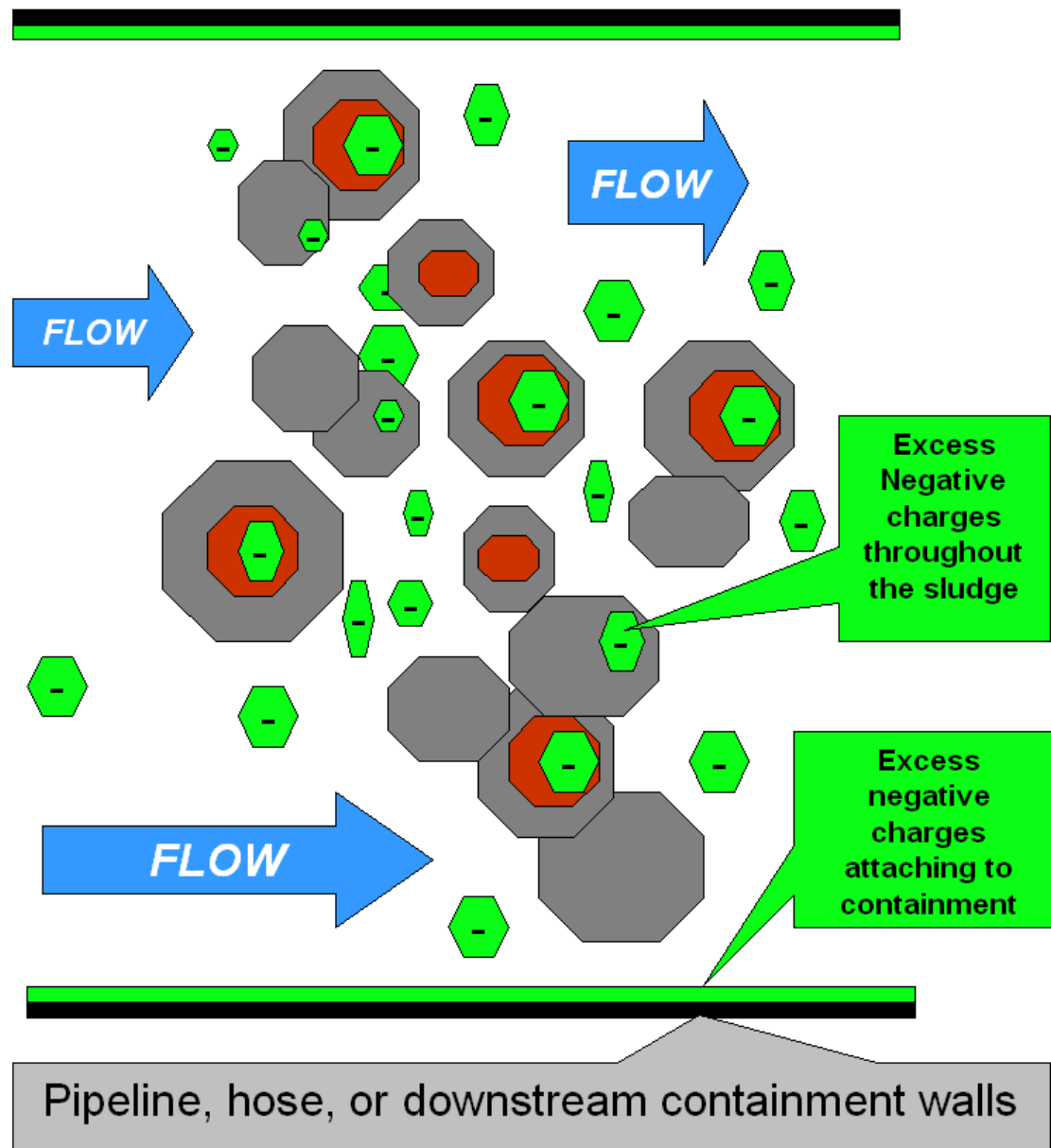


The Conditioning Process:

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.



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 Action



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