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June 15, 2015

Mr. Paul Cho, P.G.
Water Resources Control Engineer
California Regional Water Quality Control Board, Site Cleanup Unit IV
Los Angeles Region
320 West 4th Street, Suite 200
Los Angeles, CA 90013

Subject: Revised Field Sampling and Analysis Plan and Strategy Plan;
Defense Fuel Support Point Norwalk
15306 Norwalk Boulevard, Norwalk, California
(SCP NO. 0286A, Site ID NO. 16638)

Dear Mr. Cho:

The Source Group, Inc. (SGI) on behalf of the Defense Logistics Agency – Energy (DLA – Energy) previously submitted the technical document titled *Field Sampling and Analysis Plan* (Plan), dated February 11, 2015 to the Regional Water Quality Control Board (RWQCB). The RWQCB reviewed the Plan and provided comments on March 4, 2015 comments. SGI subsequently revised the Plan to reflect the comments provided by RWQCB. Revisions were made to the Plan and an updated version of the Plan was submitted to the RWQCB on April 10, 2015.

On June 3, 2015, the RWQCB coordinated a conference call to discuss the April 10, 2015, version of the Plan. During that call, it became apparent that the document submitted on April 10, 2015, via upload to Geotracker was incomplete (missing sections: 7.0, 8.0, and 9.0). During that conference call, the LARWQB requested that the April 10, 2015, Plan be resubmitted. This letter transmits that revised and updated Plan and serves to highlight changes made based both on RWQCB comments and discussions held during the April 10, 2015, conference call. The Plan was also revised to reflect recommended changes provided by our soils remediation vendor (F4 Remediation) with respect to the monitoring of soil moisture.

To facilitate the RWQCB's review of the Plan we have synopsised the comments provided in the March 4, 2015, RWQCB letter and provide a description as to where modifications have been made in the Plan (indicated in ***bold italics***) along with a summary of the modifications or updates that we have made based on those comments:

1. Staff of the LARWQB recommended measuring soil moisture at depths deeper than three feet and a pattern/frequency of 50 cubic yards. ***Section 9.0*** of the revised Plan, reflects a modified the approach for moisture measurement and management based on field performance and technical recommendations provided by F4 Remediation.
 - a. In summary, the original parameter of maintaining soil moisture between 40 and 85% was deemed too high based on the soil type present at the Site potentially

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detrimental to the propagation of the bacteria and subsequently to the performance of the bacteria. Contributing to the decision to adopt an alternate to "Field Capacity" as the primary metric of concern proposed in the initial (April) Plan is the very high native field capacity of the majority of soil on site. In general, the field capacity as a moisture content percentage by volume is 40 to 45%. Based on guidance provided by F4 Remediation, moisture (as a percentage) sufficient for maintenance of a suitable environment for bacterial growth is on the order of 10%. Therefore the proposed moisture level and associated monitoring presented in the Plan is proposed as a tensiometer reading of <20 centibars (See Plan **Section 9**).

- b. Moisture augmentation once soil is in a treatment row, though feasible, has not proven to be necessary. Quality Control metrics of moisture content post F4 bacteria/surfactant amendment has been provided in the updated Plan. In general, a combination of under-performing soil in conjunction with dry soil (high tensiometer values) will serve as the decision basis for retreating and if necessary reconditioning the soil. (**See Plan sections 9**)
2. Within item 2 the LARWQCB staff inquired about the means of evenly distributing moisture to increase soil moisture above the lower field capacity range of 40%. As stated in item 1 (above). The protocol for soil conditioning, monitoring and augmentation of under performing soil remediation has been modified in **Section 9.0** of the revised Plan. The soil on site retains moisture reasonably well and the cycle time to remediate the soil will typically be less than 90 days. The introduction of water, if necessary will be done via low-pressure injection at locations exhibiting accelerated drying. Stockpile-wide distribution of moisture is not desirable, as it would indiscriminately introduce additional moisture where it is unnecessary/undesirable. (**See Plan sections 9**)
3. Item 3 inquired about the calculation of moisture enhancement and the use of soaker hose and vapor control piping. Soaker hoses and vapor control piping will no longer be used to enhance moisture. In general, any soil deemed dry, and that does not appear to be decreasing in hydrocarbon concentrations, will simply be reprocessed and placed in a treatment row for an additional remediation cycle. (**Plan Section 9**). Moisture will continue to be field monitored and monthly lab measurements will be taken as required by the Waste Discharge Requirement (WDR); FILE NO. 90-60-145, ORDER NO. 90-148, CI-10118.
4. Item 4 inquired about variable size stockpiles of assumed clean soil, whereas the original Soil Management Plan specified a strategy for confirmation sampling of 2,000 cubic yards. In general the Plan in **Section 3.3** and Table 2 adopts the SW-846 variable frequency protocol and follows the April 10, 2012 RWQCB letter to DLA commenting on the March 2012 Parsons Soil Management Plan. Field conditions have demonstrated that stockpile size can be highly variable. Assumed clean stockpiles will be surveyed for volume and the number and locations of samples will be determined accordingly. A grid, specific to the stockpile, will be generated and the locations of the samples field sketched and logged.
5. In item 5 it was noted that floor (bottom) post excavation sampling was being done at a frequency/spacing of 50 square feet (1 sample per 2,500 square feet), but it was further noted that the Plan did not specify the number of samples to be taken. **Section 3.6** was

modified to specify the frequency and the spacial distribution of confirmation samples. Due to the variable nature of the size of each excavation the number of samples to be taken cannot be specified in the Plan, but will be identified for each excavation depending on its floor size.

6. The Regional Board questioned the use of brass or butyrate tube wide mouth glass jar containers for the collection and shipment of soil samples intended for for volatile organic compound (VOC) analysis. Within this item, the RWQCB prescribed collecting and analyzing VOC sample as specified by EPA method 5035. The April 10, 2015 Plan listed Method 5035 for soil sample collection, but the RWQCB requested a specific 5035 methodology. In an email dated June 4, 2015, LARWQCB cited that for sites that require the highest quality analytical results, the soil subcores should be field preserved with methanol or sodium bisulfate solution. Accordingly, Section 9.0 of the revised Plan presents the methanol/bisulfate vials and Terra Core™ sampling as the default 5035 sampling and sample preservation method for all future samples specifically described as an attachment to the Revised Plan.

Related to this topic, SGI already collected and analyzed samples from various completed excavations and “assumed” clean stockpiles. It is understood at the time of this correspondence that the LARWQCB desires the use of methanol/bisulfate vials. To date, site samples have been taken via Terra Core™ samplers and preserved using the empty VOA approved method 5035 option. To validate this large body of previously collected VOC sampling and analytical results, SGI is also submitting with this revised Plan, a VOC Data Validation Workplan to resample selected locations for VOCs using the proposed methanol/bisulfate 5035 method.

Thank you for your comments and suggestions. If there are any questions regarding the information provided please call me at (562) 597-1055.

Sincerely,

Neil F. Irish, P.G.
Project Manager
The Source Group, Inc

Ec: Mr. Nick Carros, DLA Energy
Mr. Kenneth Wall, SGI
File: DFSP Norwalk – 04-NDLA-007

Attachment

Field Sampling and Analysis Plan

VOC Data Validation Work Plan



June 15, 2015

Paul Cho, P.G.

Water Resources Control Engineer
California Regional Water Quality Control Board, Site Cleanup Unit IV
Los Angeles Region
320 West 4th Street, Suite 200
Los Angeles, CA 90013

Subject: Workplan for Soil VOC Analyses Results Validation
Defense Fuel Support Point Norwalk
15306 Norwalk Boulevard, Norwalk, California
(SCP NO. 0286A, Site ID NO. 16638)

Dear Mr. Cho:

The Source Group, Inc. (SGI) on behalf of the Defense Logistics Agency – Energy (DLA – Energy) submitted the technical document titled *Field Sampling and Analysis Plan* (Plan), dated February 11, 2015. On March 4, 2015, the Regional Water Quality Control Board (RWQCB) provided comments to the Plan. On April 10 2015, SGI responded to the comments and revised the Plan. Subsequently the LARWQB requested the April 10, 2015 Plan be resubmitted and requested additional detailed description of the methodology proposed for the sampling preservation for Volatile Organic Compounds (VOCs) analyses.

As the excavation started in March 2015, soil samples of excavations and stockpiles have already been collected, and this sampling was conducted using one of the three approved EPA Methods 5035 field preservation options (the empty vials method – which entails field sub-sampling via a Terra Core™ device and placing the cores into two empty 40-milliliter VOA bottles and into one 40-milliliter VOA bottle containing a methanol preservative). Based on on-going discussions with RWQCB, that method is not the currently preferred method for final sampling at the site, with RWQCB recommending the preservative with both methanol (one VOA bottle) sodium bisulfate (two VOA bottles) for samples aimed at demonstrating closure of areas. Therefore SGI/DLA proposes the following program to validate the results of the samples previously collected to date at the site. The proposed verification sampling program is aimed at providing RWQCB technical data to demonstrate that previously collected VOC data is valid.

As of June 9, 2015, 517 soil samples have been collected for VOC analyses using the empty vial EPA 5035 method (a summary of the method is attached, with relevant sections highlighted), and the laboratory reports have submitted to SGI/DLA. These samples represent the following:

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Excavation Confirmation Samples: 342 (samples collected from in-situ locations along final sidewall and bottom of excavations)

Clean soil pile samples: 110 (samples collected from the soil segregated in the field as assumed to be clean during the excavation and/or assumed be clean overburden soil)

Baseline native surface soil samples collected on initial surface prior to constructing a soil treatment stockpile: 29 samples

Intermediate soil treatment progress samples: 36

This work plan proposes to take verification samples to validate the VOC results for samples from excavations and from clean stockpiles.

To validate the previous VOC results from excavation and clean soil piles, SGI is proposing the following resampling using the methanol/bisulfate 5035 method.

Excavation Confirmation Samples

It should be noted that the results of the 342 VOC analyses for excavation samples collected using the empty vials 5035 method indicate no sample with significant VOCs. To verify these findings, two sets of verification samples will be collected at previously sampled locations: sample locations with the highest residual concentrations of gasoline range hydrocarbons and randomly selected samples:

- The results of previous excavation confirmation samples reported Total Petroleum Hydrocarbon (TPH) values in the <C12 range (gasoline range organics – GRO). As VOCs can be expected to be present in samples that would also contain these lighter hydrocarbon-chain TPH concentrations, SGI is proposing to re-sample all excavation confirmation sampling locations where TPH <C12 concentrations were reported at a concentration of >5 mg/kg, conservatively representing 5% of the strictest cleanup goal for that TPH range (100 mg/kg). These locations will be resampled.
- In addition, 5% of the previous locations in each excavation will be resampled, using a random number generator, representing a 1 in 20 ratio similar to duplicate sampling standards, as recommended for example in the *DTSC 2004 Guidance Document* on EPA Method 5035. A minimum of two random locations per excavation will be collected.

Clean Soil Pile Samples

The results of the previous 110 samples collected by the empty vials method for soil piles segregated as clean soil piles indicate the overall absence of detectable VOCs in these soils. Similarly to the excavation confirmation samples sampling program described above, SGI proposes to resample all stockpile sample locations with reported TPH <C12 concentrations of >5 mg/kg.

SGI also proposes to collect duplicate samples at randomly selected locations among all the clean soil sample locations at the ratio of 1 in 20.

The detailed procedures for the sampling and sample preservations are described as an appendix to the *Revised Field and Analysis Plan* submitted concurrently with this validation work plan.

Upon approval of this proposed re-sampling work plan using the methanol and sodium bisulfate Method 5035 option, SGI will present to RWQCB the proposed resampling locations and rationale, and then conduct the proposed re-sampling of locations with TPH<C12 reported at over 5 mg/kg, and at selected random locations. After receipt of laboratory analytical results, SGI will prepare a summary report for RWQCB with data interpretation and justification, if applicable, to demonstrate that the previous set of data collected using the Method 5035 empty vials method provided valid and reliable results.

Closing

SGI/DLA believe that this technical approach to data validation will provide a defensible dataset to allay RWQCB's concerns on the empty vials method, and that this additional data collection, will allow RWQCB to accept the results of VOC analyses generated to date.

If there are any questions regarding the information provided please call me at (562) 597-1055.

Sincerely,

Neil F. Irish
Project Manager
The Source Group, Inc

Ec: Mr. Nick Carros, DLA Energy
Mr. Kenneth Wall, SGI
File: DFSP Norwalk – 04-NDLA-007

Attachment – Summary of Empty Vial EPA Method 5035 Preservation Method

**REVISED FIELD SAMPLING AND ANALYSIS PLAN
AND SAMPLING STRATEGY**

**Defense Fuel Support Point, Norwalk
Norwalk, California**

04-NDLA-007

Prepared For:

Defense Logistic Agency - Energy
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Prepared By:



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June 15, 2015



Prepared By:

Ken Wall
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1.0 INTRODUCTION

On behalf of our client, Defense Logistics Agency - Energy (DLA Energy), The Source Group, Inc. (SGI) is submitting this Revised Field Sampling and Analysis Plan and Sampling Strategy (FSAP). This document was requested by Los Angeles Regional Water Quality Control Board (LARWQCB), via correspondence dated January 7, 2015, which provided conditions and related information requests associated with the pending Waste Discharge Requirement (WDR) for excavation and soil treatment at the former Defense Fuel Support Point (DFSP) Norwalk facility (site). On February 6, 2015, LARWQCB approved coverage for the bioremediation project under General Waste Discharge Requirements (Order No. 90-148) which also included Monitoring and Reporting Program (MRP) No. CI-10118 (MRP CI-10118). On March 4, 2015 the LARWQCB commented on FSAP and requested modifications. This updated plan addresses those comments and additional comments by RWQCB on June 2, 2015 to an earlier FSAP submitted April 10, 2015 and includes modifications to the April 2015 FSAP including moisture conditioning methods, adapted based on site-specific field conditions, recognized during the initial stages of the project.

Petroleum contaminated soil is present at numerous locations throughout the 50 acre DFSP Norwalk facility. The objectives of the planned remedial activities are to reduce the concentrations of petroleum hydrocarbons and related compounds present in vadose zone soil in order to facilitate site redevelopment and to accelerate the remediation of the underlying groundwater. To achieve these objectives, contaminated soil will be excavated, primarily treated on site using biologic methods, and reused as fill once cleanup targets have been met. Accordingly large volumes of clean, contaminated, and treated soil will be generated, handled, treated, and/or re-used on the site. Therefore, the purpose of this FSAP is to provide field sampling procedures and data gathering methods that will be used to support the removal actions at the site. This document also provides the sampling strategy for confirmation sampling of untreated and treated soil intended for reuse for backfill of the excavations.

This FSAP will be used by field personnel as a reference during sampling and analysis of soil. This includes soil segregated as assumed clean, soil treated within a treatment stockpile, in-situ soil samples from the sidewalls and bottoms of completed excavations, and baseline characterization of surface soil under soil treatment rows prior to and post treatment.

The primary guidance and planning document for these activities at the Site, *Soil Management Plan: Treatment Cell Operation and Site Excavation (SGI, 2014)*, provides the basic information associated with the excavation, soil treatment and treatment cell construction and maintenance. Soil cleanup goals for TPH and VOCs are provided in the summary letter provided as Appendix A to this Plan.

For each excavation from which contaminated soil is removed for treatment, a letter report will be submitted to the LARWQCB. A diagram (Figure 1) will be prepared for each completed excavation. Included with this diagram will be all the supporting analytical data associated with excavation sidewalls and bottom and the source material for backfill. Similarly, reports on clean

soil piles and treated soil piles will be prepared and submitted along with a request for re-use of that soil. A comprehensive summary report will then be prepared to document the excavated soil treatment confirmation sampling and areas of backfilling.

2.0 SAMPLING OBJECTIVES

Soil sampling will generally be associated with seven activities:

- Baseline surface soil characterization and post-closure sampling of treatment cell areas;
- Pretreatment soil characterization and stockpile performance monitoring;
- Assumed clean soil confirmation sampling;
- Treated soil confirmation sampling;
- Exploratory trenching;
- Post-excavation confirmation soil sampling; and
- Waste profiling for off-site disposal.

The objectives of each of these sampling activities are described below, and detailed sampling and analysis procedures are described in later sections of this FSAP.

2.1 Native Soil Characterization and Post-Closure Sampling

Native baseline surface soil samples will be collected from areas (historical petroleum storage basin areas) that are assumed to be non-contaminated based on existing assessment data and proposed to be used for soil treatment stockpiles. The objective of that testing is to generate a baseline characterization of native surface soil conditions. This data will serve as a benchmark and will be compared against post-closure sampling of these same areas. The objective of the pre- and post-use soil sampling and analysis is to confirm and demonstrate that operation of the soil treatment cells has not adversely impacted surface soils. In the event there has been some impact the soil in those areas will either be treated onsite or disposed of offsite. The primary objective is to leave those areas used for treatment in good condition suitable to obtain a clean closure status.

2.2 Pretreatment Soil Characterization and Stockpile Performance Monitoring

Petroleum contaminated soil will be treated with a mixture of bacteria and surfactant to facilitate the bioremediation of the soil. F4 Remediation (F4) will be performing the soil treatment while SGI will provide project oversight and performance monitoring. Pretreatment soil sampling will be conducted to determine contaminant concentrations prior to amendment of soil with the F4 bacteria and surfactant mixture. The subsequent performance monitoring sampling will be compared to these initial concentrations and progress samples to evaluate the performance of the treatment remedy specific to a treatment row within a treatment cell.

Subsequent performance monitoring results will be compared against previous results and will serve to project the timing of collecting confirmation samples.

2.3 Assumed Clean Confirmation Soil Sampling

A substantial volume of clean over burden will be removed to reach impacted soils. Soil will also be field-screened via field observation and photo-ionization detector (PID) readings. If soil does not appear to be contaminated and PID readings are less than 50 parts per million (ppm), the soil will be staged in a “clean soil” staging area as identified on Figures 2 and 3. Confirmation samples will be collected to confirm the soil is suitable for reuse. The discrete sample results for each discrete soil pile will be compiled and the 95% upper confidence level for each chemical of concern (COC) will be calculated and compared to the pre-approved cleanup levels. Soil sampling frequency and evaluation are discussed in Section 3.3.

2.4 Treated Soil Confirmation Sampling

Identical to the confirmation sampling of assumed clean soil, the treated soil must be sampled to ensure the soil meets the cleanup goal criteria. Initially more samples will be required of treated soil versus untreated, assumed clean soil. The objective is to ensure the treatment process has sufficiently treated all the soil within a given stockpile.

2.5 Exploratory Trenching and Step Out Sampling

Exploratory Trenching will be conducted in selected areas throughout the site as a form of potholing. See Figure 2 for the location and identity of the trenching. The objective of sampling the soil from these trenches is to verify the soil from 0 to 10 feet below ground surface (ft bgs), within those areas has not been impacted. In the event contamination is encountered it will be removed and treated onsite or disposed of offsite.

When lateral or vertical excavation limits exceed the planned excavation footprints, step out or deeper sampling may be conducted, and soil samples associated with these investigations will be collected by potholing with excavation equipment or with push-probe equipment.

2.6 Post-Excavation Confirmation Soil Sampling

The objective of the post-excavation sidewall and bottom sampling is to confirm the extent of contamination has been removed within the upper 10 feet from current grade, and, for excavations greater than 10 feet, to document the condition of the sidewalls and bottoms of the excavations prior to backfill.

2.7 Waste Profiling for Off-site Disposal

Some waste will be segregated for off-site disposal. Sampling will be required to generate a waste profile.

3.0 SAMPLE LOCATIONS AND FREQUENCY

This section discusses the locations and frequency of soil samples that will be collected for analytical testing. Table 1 provides data definition and identification structure for excavation identification, treatment row identification, stockpile identification and sample identification.

3.1 Baseline Surface Soil Characterization and Post-Closure Sampling

Prior to placing liners down for treatment rows within a treatment cell, baseline surface, native soil samples will be collected. After termination of use of the treatment cells/rows, post-closure sampling will be conducted.

A grid of the cells and basins will be prepared to record the location of the baseline and post closure samples, and two samples will be collected for each proposed treatment cell. The soil samples will be collected from within the upper 6 inches of existing grade.

3.2 Pretreatment Soil Characterization and Stockpile Performance Monitoring

Concentrations of hydrocarbons in areas to be excavated are known from previous investigations. To supplement that information, soil samples will be collected as needed from the areas excavated and tested to further establish pre-treatment hydrocarbon concentrations. A typical staging stockpile of soil designated for treatment will contain 400 cubic yards. After the soil is placed in a treatment row, typically 800-900 cubic yards, 4 composite performance samples (4 locations for each composite sample, with 4 progress samples representing 16 locations in each stockpile) will be collected. The time intervals will be dependent on various factors including results of baseline and previous sampling and as progress is monitored, the frequency may be adjusted depending on rate of progress. Typically, performance monitoring samples will be collected every 30 days.

3.3 Stockpiled Soil Assumed Clean for On-Site Reuse

Discrete soil samples of assumed clean soil stockpiles will be collected for characterization in general conformance with the United States Environmental Protection Agency (USEPA) SW-846, "*Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*". The minimum number of discrete samples initially required follows the April 12, 2012 RWQCB letter to DLA commenting on the March 8, 2012 Soil Management Plan prepared by Parson, and is provided below:

Random sample points will be selected from locations on a three-dimensional grid. Stockpiles will be surveyed and contoured. The number of samples to be collected for each stockpile will be determined by the total volume of the stockpile. The locations will be randomly selected and the relative location will identified on the survey figure of the specific stockpile. Each stockpile considered for reuse will be sampled separately. Additional sample analyses may be required to meet the confidence levels specified in SW-846; therefore, archiving samples may be appropriate. Archived samples will be appropriately preserved and analyzed within the maximum holding time specified in SW-846.

The samples from assumed clean soil piles will be evaluated using a 95% upper confidence limit (UCL) value.

The stockpiled soil documentation will include the following information:

- An estimate of the volume of the stockpile;
- Stockpile type (i.e., impacted soil, or non-impacted soil);
- A plot plan (Figure 3) detailing the stockpile and sample locations;
- A copy of all sample results, chain-of-custody documents, and Quality Assurance/Quality Control (QA/QC) supporting data; and
- A summary of the laboratory results for the stockpile sampling.

The stockpile's 95% UCL hydrocarbon concentrations will be compared to the cleanup goals for soil targeted for backfilling in the 5-10 or 0-5 ft depth interval.

If a stockpile's 95% UCL hydrocarbon concentration does not meet cleanup goals, all or a portion of the stockpile will be routed for treatment or considered for off-site disposal. If a portion of a stockpile appears to be suitable for reuse that remaining volume will be resampled. The number of samples will be in accordance with the frequency specified in Table 2.

3.4 Treated Soil Confirmation Sampling

The typical stockpile row within a treatment cell will contain approximately 800-900 cubic yards. In accordance with current RWQCB requirements, 35 discrete samples will be collected for each treatment stockpile. The results will be compiled to calculate the 95% (UCL) for comparison to the cleanup goals. Figure 4 represents the confirmation sampling grid of a typical stockpile. The soil samples will be collected from a depth of 1-3 feet towards the center of the stockpile.

For the stockpile to be approved for reuse, the stockpiled treated soil documentation must include the following information:

- An estimate of the volume of the stockpile;
- Stockpile type (i.e., impacted soil, or non-impacted soil);
- The excavation ID
- A plot plan (Figure 3) detailing the stockpile and sample locations;
- A copy of all sample results, chain-of-custody documents, and QA/QC supporting data; and
- A summary of the laboratory results for the stockpile sampling.

3.5 Exploratory Trenching Sampling and Step Out Sampling

Exploratory trenches (test pits) will be advanced to a depth of 10 feet bgs, and will be sampled in areas where PID readings are greater than 50 parts per million (ppm) and in areas that are visually impacted. SGI estimates that up to 10 samples may be collected per basin from test pits as indicated on Figure 3.

Additional step-out samples may be collected to verify the lateral and vertical extent of impacted soil. The number and location of any additional samples for stepout and vertical delineation samples will be based on the results of the preliminary excavation soil sampling conducted as described in the following section.

3.6 Post-Excavation Confirmation Soil Sampling

Figure 3 identifies the location, identity and target depth interval of all individual excavations. Post-excavation confirmation soil sampling will be conducted after removal of impacted soil. Post-excavation sampling will be performed at the excavation floor and sidewalls to verify that sufficient soil has been removed to meet cleanup goals. Soil samples will be collected and submitted to the laboratory for analytical testing in accordance with Section 7 of this FSAP. If post-excavation soil sample results indicate the presence of COCs with concentrations greater than cleanup levels, then additional excavation may be performed with another round of soil confirmation sampling.

Once COC-impacted soil is removed, a sampling grid will be established for the excavation floor and sidewalls. For excavation floor sampling, the excavated areas will be divided into 50 by 50 foot sampling grids. One discrete soil sample will be collected randomly within each grid cell from the excavation floor, resulting in one floor sample for each 50 by 50 feet of square area of excavation bottom. For excavation sidewall sampling, a discrete soil sample will be obtained for every 25 linear feet of horizontal sidewall, or portion thereof, and every 3.0 feet of vertical sidewall, or portion thereof. Soil samples will be taken at a depth of approximately 6 inches to 1 foot into the exposed surface. Each soil sample will be analyzed for the constituents discussed in Section 7 of this FSAP.

3.7 Stockpiled Soil Designated for Off-Site Disposal

Soil stockpiled for off-site disposal is soil, based on visual inspection and field screening, impacted with COCs at excessive concentrations (e.g. tarry or sludge material). If encountered, the soil may be segregated for off-site disposal. Soil samples will be collected for the purpose of waste profiling and the waste will be disposed of offsite. Assuming the waste characteristics indicate non-hazardous soil, soil waste will be transported to Waste Management's Azusa's Land Reclamation Facility in Azusa, Simi Valley Landfill, or McKittrick facility, or to the Soil Safe's Thermal Desorption facility in Adelanto, CA. All off-site disposal or treatment will be directed to a state-approved facility.

3.8 Disposition of Debris Encountered During Excavation

Debris may be encountered during removal of the impacted soil. Excavated inert debris will have loose soil removed prior to placement on to stockpiles. Debris will be segregated, stockpiled, and disposed off-site at a Class III landfill or recycled at a DLA Energy-approved facility.

4.0 SAMPLE DESIGNATION

Samples sent to an analytical testing laboratory will be assigned a unique sample identification number according to the conventions described below. Sample numbers will be recorded in a dedicated field logbook, the excavation site plan, and on the chain-of-custody at the time of sample collection. A complete description of the sample, sample circumstances/conditions, date and time of sampling, and the location of the sample will be recorded in the dedicated field logbook.

4.1 Treatment Cell/Row Baseline and Post-Closure Sampling

Prior to placing liners down for treatment rows within a treatment cell, baseline surface samples will be collected. After termination of use of the treatment cells/rows, post closure sampling will be conducted. Each series of sampling will be identified as follows.

1. Surface, native soil, baseline samples (from treatment cell/row area before liner installation) – SB Series; and
2. Post-Closure (from treatment cell/row after removal of liner) – PC Series.

Sample numbering will be the same as other series with identification numbers ranging from 0001 to 9999. A grid of the cells and basins will be prepared to record the location of the native, baseline samples and post closure samples.

4.2 Stockpile Soil Samples

Stockpile soil samples will be assigned a series sample type and a unique sample number. The five series sample types are:

1. Baseline Sample (contaminated soil going to treatment) - B series;
2. Performance Sample - P series;
3. Treated Soil Confirmation Sample – T series;
4. “Assumed “ Clean Confirmation Sample - C series; and
5. Waste Profile Sample – W series.

The series sample type will be followed by 4 digits. As an example a Treated Soil Confirmation Sample would be identified as “T0003”. The sample number will be logged with the information details of the origin of that sample. For each series of samples there will never be a duplicate number. Each series can have up to 9,999 unique sample numbers.

The chain-of-custody will identify the origin of the sample. See Table 1 for identification structure for the various soil segregation categories. For confirmation sampling based on a grid, a figure will be generated recording the sample number for the associated grid location.

4.3 Post-Excavation Confirmation Soil Samples

Post-excavation verification soil samples will be assigned a unique number that will indicate the Excavation Number, followed by "N," "S," "E," "W," or "F" (indicating the sample was collected from the north [N], south [S], east [E], or west [W] sidewall, or from the excavation floor [F]), and then a sequential number (if more than one sample is collected from a sidewall or from the excavation floor). For example, sample E2-F2 would identify the second sample collected from the floor of Excavation Number 2. The sample location, sample number and description will be documented in the dedicated field logbook.

Figure 1 is a sample post excavation figure; figure 3 provides the excavation numbers for all planned excavations. The figure will show the location of the sidewall and bottom samples.

5.0 SAMPLING EQUIPMENT AND PROCEDURES

This section describes sampling equipment and procedures associated with post-excavation confirmation sampling and stockpile soil sampling. This section also includes a discussion of equipment blank sampling and decontamination procedures for sampling equipment.

All samples will be analyzed for VOCs by EPA Method 8260B, for TPH as gasoline range organics (GRO) by EPA Method 8260, and for TPH with carbon value >C12 by EPA Method 8015, as described in Section 7. Accordingly, soil samples will be collected in jars for 8015 TPH analyses, and using an EPA Method 5035 containers and preservatives for analysis by EPA Method 8260B. The samples collected for VOC analyses and TPH GRO will be taken using one of the EPA Method 5035 options. Based on discussions with RWQCB in June 2015, the preferred 5035 sampling method consists of the use of methanol- and sodium bisulfate-preserved sample vials, and therefore that sample preservation method is proposed as the default methodology. Appendix A presents in detail the proposed procedures for sampling and sample preservation.

5.1 Post-Excavation Confirmation Sampling

Confirmation soil samples associated with the remedial excavation(s) will be sampled using a glass 4-ounce jar and EPA method 5035 methanol- and sodium bisulfate- preserved vials.

5.2 Stockpile Soil Sampling for Soils Designated to Remain Onsite

Soil samples collected from stockpiles initially designated to remain onsite will be collected using a hand auger or trowel from predetermined sampling locations and depths. The hand auger or trowel will be decontaminated following procedures outlined in Section 5.4 at the start of sampling and between sampling locations. The soil will be retrieved from the stockpile, EPA Method 5035 subcore samples will be collected and field preserved, and an additional stockpile soil sample will be carefully placed in a 4-ounce glass jar. The samples will be placed in a cooler maintained at 4 degrees Celsius. Sample labeling, delivery, and chain-of-custody documentation will be completed per Sections 6.1 through 6.3.

5.3 Stockpile Soil Sampling for Soils Designated to be Disposed of Offsite

Soil samples collected from stockpiles initially designated for off-site disposal will be collected as described in Section 5.2.

5.4 Decontamination Procedures

Whenever possible, disposable sampling equipment will be used for this project. However, if non-disposable sampling equipment is used, it will be decontaminated to prevent cross contamination between samples. Sampling equipment will be decontaminated by washing with a non-phosphate detergent such as Liquinox™. Decontamination water will be collected and placed in a 55-gallon

drum or wastewater holding tank. The following steps will be followed for decontamination of non-disposable sample equipment:

- Wash with a non-phosphate detergent and water solution. This step will remove visible contamination from the equipment. Fill a 5-gallon bucket approximately 3/4 full and dilute with a non-phosphate detergent as directed by the manufacturer. Use a dedicated long-handled brush to assist with cleaning.
- Rinse with potable water. This step will decrease the gross contamination and reduce the frequency of changing of the non-phosphate detergent and water solution. Fill a 5-gallon bucket, 3/4 full with water. Use a dedicated long-handled brush to assist with cleaning of equipment. Frequent changing of this water will increase its effectiveness.
- Rinse with de-ionized water. Fill a 5-gallon bucket approximately 3/4 full of water and use a dedicated long-handled brush to assist with cleaning. Periodic changing of this water is required.

6.0 SAMPLE LABELING, DELIVERY, AND CHAIN-OF-CUSTODY

This section describes how samples will be labeled, picked up, delivered, and tracked.

6.1 Sample Labeling

Sample labels will be completed using preprinted labels with indelible, black ink, and affixed to each sample container. No sample number within any sample series will be reused. If a sample number label is destroyed, the sample number will be logged and recorded as destroyed. Sample containers will be placed into resealable plastic bags to protect the sample from moisture during transportation to the laboratory. Each sample container will be labeled at a minimum with the following:

- Unique sample identification number;
- Sample collection date (month/day/year);
- Time of collection (24-hour clock);
- Project number (04-NDLA-007);
- Sampler initials;
- Analyses to be performed; and
- Preservation, if any.

6.2 Sample Delivery

This section applies to samples that will be picked up by the analytical testing laboratory or samples delivered to the off-site analytical laboratory. Samples may be picked up in the field or at the Field Geologist/Engineer's office by the analytical testing laboratory. The samples will be maintained at 4° Celsius. The chain-of-custody documentation will be completed and signed by the laboratory-assigned courier. The samples may then be relinquished to the courier for transportation to the laboratory. The laboratory will record the temperature of the cooler immediately upon receipt of the samples.

6.3 Chain-of-Custody

A chain-of-custody is a vital tool for tracking samples and is a written record of sample possession from the time the sample is collected until it is analyzed. The following will be recorded on the chain-of-custody forms:

- Project name;
- Project location;
- Project number;
- Project contact;

- Client;
- Project Manager;
- Sample identification;
- Soil source identification
- Date sample was collected;
- Sample type (soil, wastewater etc.);
- Number of sample containers;
- Required analytical test methods;
- Remarks/observations specific to the sample;
- Number of samples to be relinquished to the analytical laboratory;
- Transfer signatures associated with relinquishing samples (the sampler will initiate the chain-of-custody procedure);
- Courier/laboratory representative signature (for commercial carrier, record air bill number);
- Date/time of custody transfers;
- Comments regarding the condition of the samples, (e.g. cooled with ice, etc.);
- Additional comments;
- Written request for electronic file for all samples analyzed;
- Information regarding sample storage/disposal;
- Turn-around-time requirement;
- Sampler signature; and
- Courier signature.

7.0 ANALYTICAL TESTING METHODS

This section describes analytical test methods, sample container, preservation, and holding time requirements for soil samples. Soil samples will be collected and analyzed to serve different purposes:

- Baseline surface soil characterization and post-closure sampling of treatment cell areas;
- Pretreatment soil characterization and stockpile performance monitoring;
- Assumed clean soil confirmation sampling;
- Treated soil confirmation sampling;
- Exploratory trenching;
- Post-excavation confirmation soil sampling; and
- Waste profiling for off-site disposal.

The principal chemicals of concern at the site are petroleum hydrocarbons and volatile organic compounds. Petroleum hydrocarbons will be analyzed using EPA Method 8015 for carbon chains of C13 and higher. Shorter chain hydrocarbons (C4 to C12) will be reported as gasoline range organics and analyzed by EPA Method 8260. Volatile organic compounds will be analyzed by EPA Method 8260.

Table 3 summarizes the sample preservation methods and analytical test methods for the types of samples to be collected based on regulatory requirements and site cleanup goals.

8.0 FIELD QUALITY ASSURANCE/QUALITY CONTROL

Field Quality Assurance/Quality Control (QA/QC) samples will be collected and analyzed during the post-excavation confirmation and stockpile soil sampling to assess the consistency and performance of the sampling program. Field QC samples for this project will include field duplicates and equipment rinsate samples.

8.1 Field Duplicates

Field duplicates consist of a sample of the same matrix as the primary sample collected. Duplicate samples will be collected, if available, at the same time and location as the primary sample, using the same sampling techniques. The purpose of field duplicate samples is to evaluate the precision of the overall sample collection and analysis process, particularly for stockpiled soil. The dense grid of proposed in-situ excavation sampling locations indicates that additional, duplicate sampling may not be required. Field duplicates will be collected from stockpiles at a frequency of one per every 20 samples and will be analyzed using the same method as the primary sample. Field duplicate sample numbers will be labeled with a DUP nomenclature. Locations of duplicate samples and their identifications will be recorded in the dedicated field logbook and on the appropriate stockpile map.

8.2 Equipment Rinsate Samples

Equipment rinsate samples will only be collected when using hand auger equipment. Rinsate samples consist of distilled water collected from the final rinse of the decontamination process. Subsequent to equipment decontamination, distilled water will be decanted over the sampling equipment in the appropriate containers. Rinsate samples will be collected, placed in appropriate pre-cleaned containers supplied by the analytical laboratory, and analyzed for the same constituents as the field samples. Equipment rinsate samples evaluate the effectiveness of the decontamination procedure and possible cross-contamination during sampling events.

8.3 Sample Containers, Preservatives, and Holding Times

Sample container requirements, preservatives, and holding time requirements for the soil analytical test methods to be used in this removal action project are summarized in Table 3.

9.0 SOIL MOISTURE

Moisture is an integral part of the bioremediation process, and will be monitored during the treatment process although ultimately the final hydrocarbon concentrations below cleanup goals are the final soil reuse criteria. F4 Remediation, the subcontractor that treats the contaminated soil using a mixture of bacteria and surfactant, has provided guidance regarding the soil moisture conditioning (Appendix C).

This section is divided into two subsections. The first section describes soil conditioning and evaluation of moisture preceding placement into a treatment row. The second section addresses monitoring of moisture after soil has been placed in a treatment row.

Soil Moisture Conditioning and Measurement Prior to Placement in Treatment Row

During the course of excavation, soil moisture is added liberally primarily as a function of dust and odor suppression.

Once the soil is excavated it is transported to, and staged, in the processing area. The soil will be visually inspected and a tensiometer measurement (QuickDraw SoilMoisture Probe, 36-inch length, field-portable tensiometer) will be taken. Provided the soil is visibly moist and the tensiometer reading is less than 20 centibars (as recommended by F4; Appendix C) the soil will be considered adequately moist. Once the soil is deemed adequately moist, the soil will be processed via F4 adding the water/bacteria/surfactant mixture at a rate of approximately 4 gallons per ton. Soil moisture will be logged approximately three times during each batch of 300 tons of soil processed.

In the event the field measurement indicates the soil is out of range or if the soil is visibly, noticeably dry, the soil will be conditioned within the staging area until satisfactory tensiometer measurements and visual inspection indicate the soil moisture is acceptable.

Once soil is placed in the treatment row and the entire row is ready for connection to the soil vapor extraction system and ready for final full coverage with plastic, tensiometer measurements will be taken. These readings will serve as the baseline soil moisture measurements.

Commensurate with performance sampling as prescribed in section 3.2, bacteria plate counts as required by the WDR and moisture measurements will be taken and evaluated. Based on the evaluation of such progress sampling, the soil will remain in the treatment row until acceptable hydrocarbon and VOC levels are achieved, or the soil will be sampled for final confirmation of treatment.

In the event the combination of bacteria counts, moisture measurements and static contamination concentration levels indicate the treatment row or a section therein is only marginally active, the decision to augment moisture through low-pressure injection of water at dry locations or to retreat (with moisture, and/or bacteria / surfactant) will be made.

Moisture Monitoring

Treatment rows will be inspected weekly to inspect the condition of plastic covering and to look for noticeable signs of drying. As necessary field measurements will be taken with a tensiometer.

If it is determined that locations within a treatment row require additional moisture, water will be injected with a root soaker irrigation tool with a starting rate of approximately 5 gallons per minute.

Moisture Observations on Treatment to Date

The treatment of a few thousand cubic yards conducted as the early stages of soil treatment operations at the site have included valuable observations and measurements of the bioremediation process. The initial stockpiles were equipped with a triple line of water irrigation soaker hoses located on the top and on each flank of the stockpiles. Visual observations of the treated soil stockpiles have allowed observations of condensation moisture on the inside face of the plastic sheeting covering the stockpiles. The vapor extraction pipes connected to the stockpiles also have been observed to contain entrained water. These observations indicate that the initial moisture of the stockpile was significant and that no water addition was needed to the stockpiles during the treatment of the initial soil piles.

Testing by F4 and SGI of the Site soil capacity to absorb moisture also indicated that the appropriate soil moisture could be measured with the field tensiometer at a value of <20 centibars, providing a Site-specific field target value for moisture monitoring.

10.0 SITE MANAGEMENT AND RECORD KEEPING

Sampling information will be recorded on chain-of-custody forms and on the specific excavation or stockpile map/plan and tables. These documents will be completed in the field at the time of sample collection. Entries will be legible and recorded in indelible black ink. A dedicated bound field logbook with consecutively numbered pages will be assigned to this project. If it is necessary to transfer the logbook to another person, the person relinquishing the logbook will sign and date the last page used and the person receiving the logbook will sign and date the next page to be used. At a minimum, the logbook will contain the following information:

- Project name and location;
- Date and time of entries;
- Personnel in attendance, including any visitors to the site;
- General weather conditions;
- Work performed on a daily basis;
- Field observations;
- Sampling information (including sample identification, sample location, sample description/type, and analytical testing);
- Field measurements data (including air monitoring results, instrument calibration records, and problems, if encountered);
- Descriptions of deviations from the FSAP, if applicable;
- Problems encountered and corrective action taken;
- QC-related activities and identification of field QC samples;
- Detailed record of oral and/or written requests by the regulatory agencies, client, subcontractor, and
- Any other events that may affect the sampling and analyses.

In addition to internal records keeping, the soil excavation, treatment, sampling and reuse will be required to be documented and reported to RWQCB's SLIC department and as part of the WDR department; detailed records will also need to be provided to the AQMD as part of the Rule 1166 monitoring and documentation, and to the city of Norwalk as part of the documentation prior to land conveyance.

11.0 REFERENCES

Parsons. 2013a. *Conceptual Site Model and Remedial Action Evaluation for Soil, Groundwater and LNAPL*.

Regional Water Quality Control Board, 2012. *Comments on Soil Management Plan*. April 10

SGI. 2014. *Soil Remedial Action Plan – Defense Fuel Support Point Norwalk*, November 30.

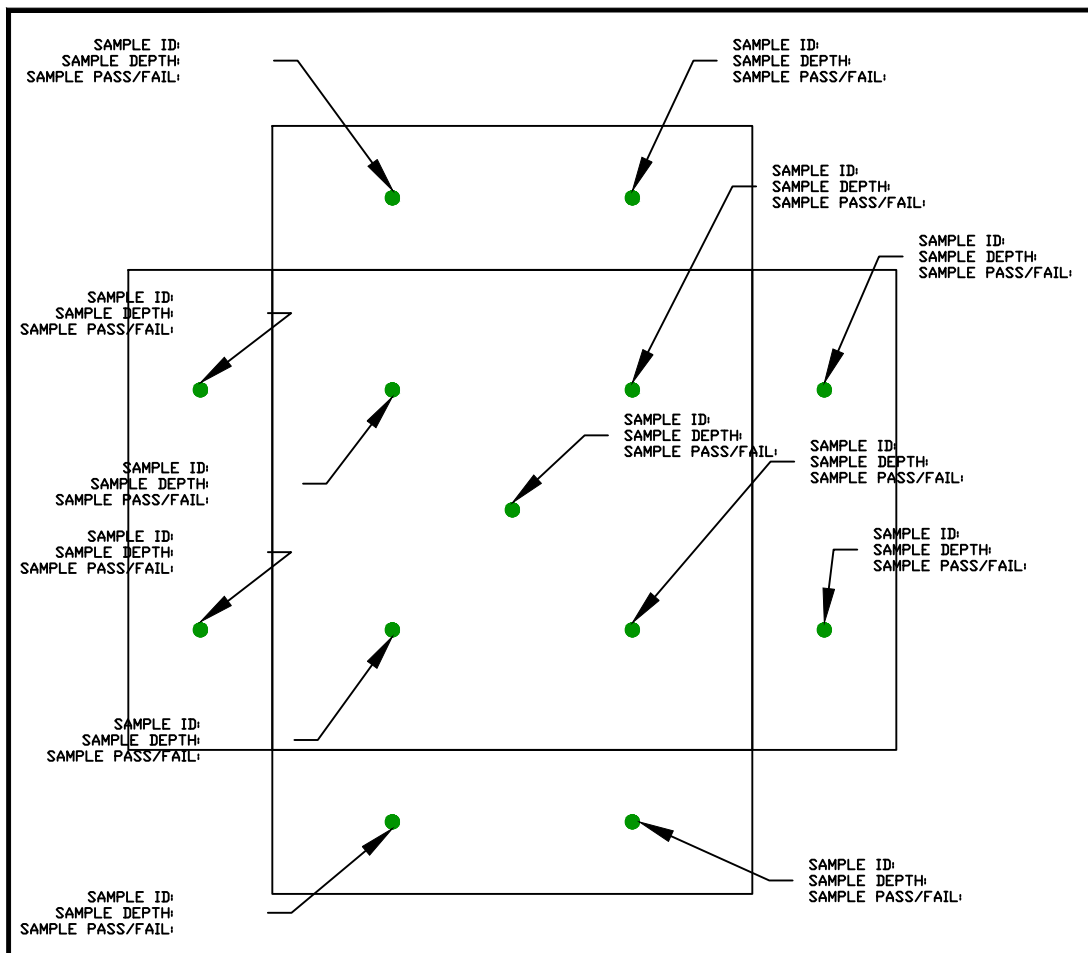
SGI. 2014. *Addendum to Soil Remedial Action Plan – Defense Fuel Support Point Norwalk, (F4 Bioremediation Description)*, November 30.

SGI. 2014. *Soil Management Plan: Treatment Cell Operation and Site Excavation*

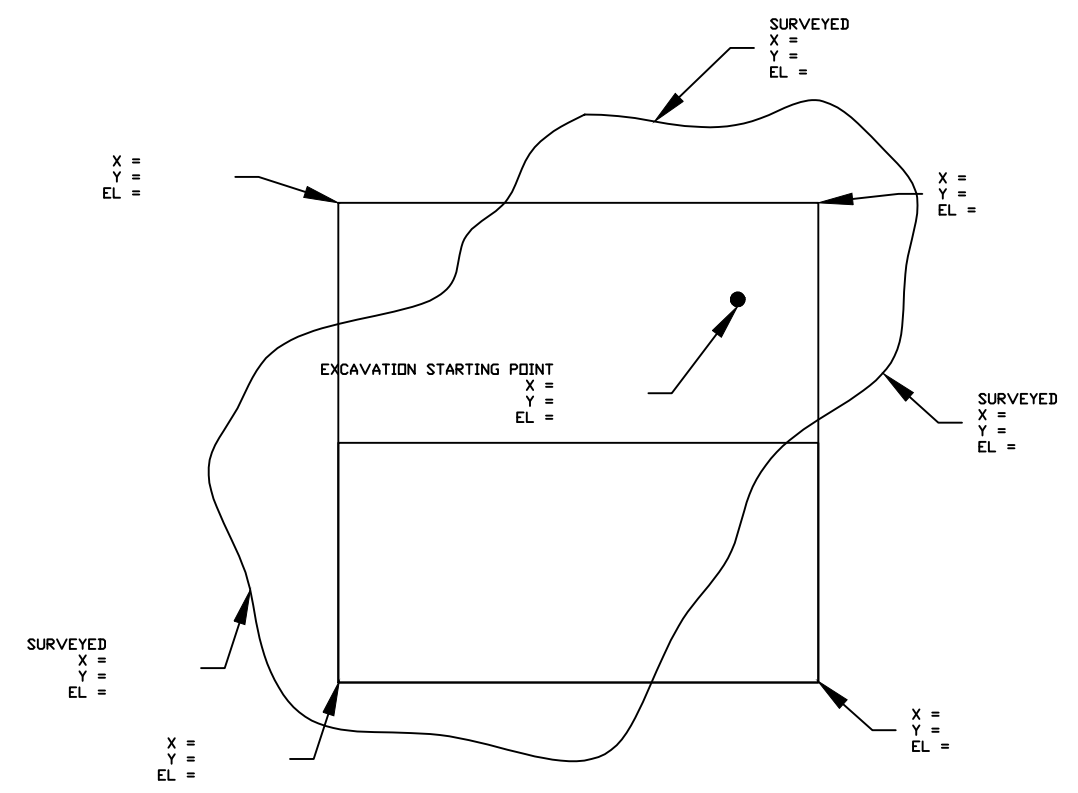
12.0 LIMITATIONS

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FIGURES

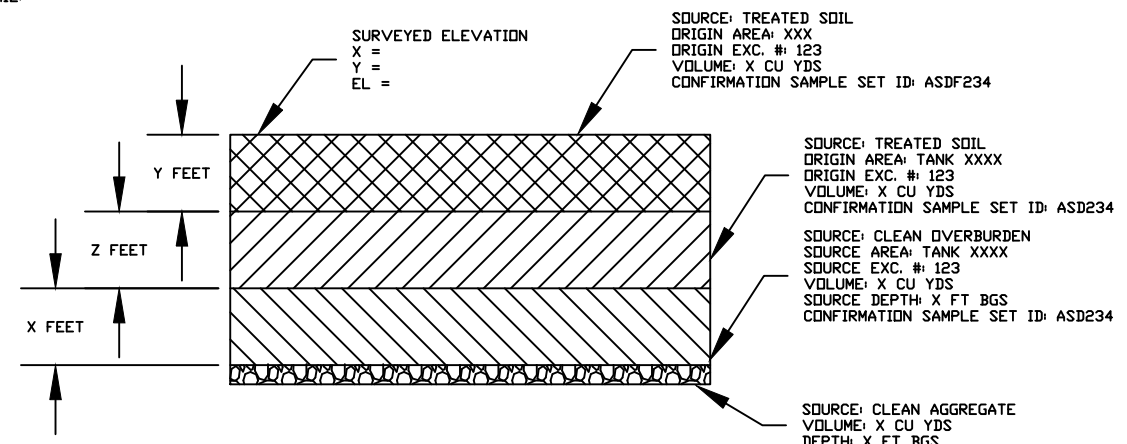


COMPLETED EXCAVATION DIAGRAM
EXPLODED PLAN VIEW



COMPLETED EXCAVATION DIAGRAM
PLAN VIEW

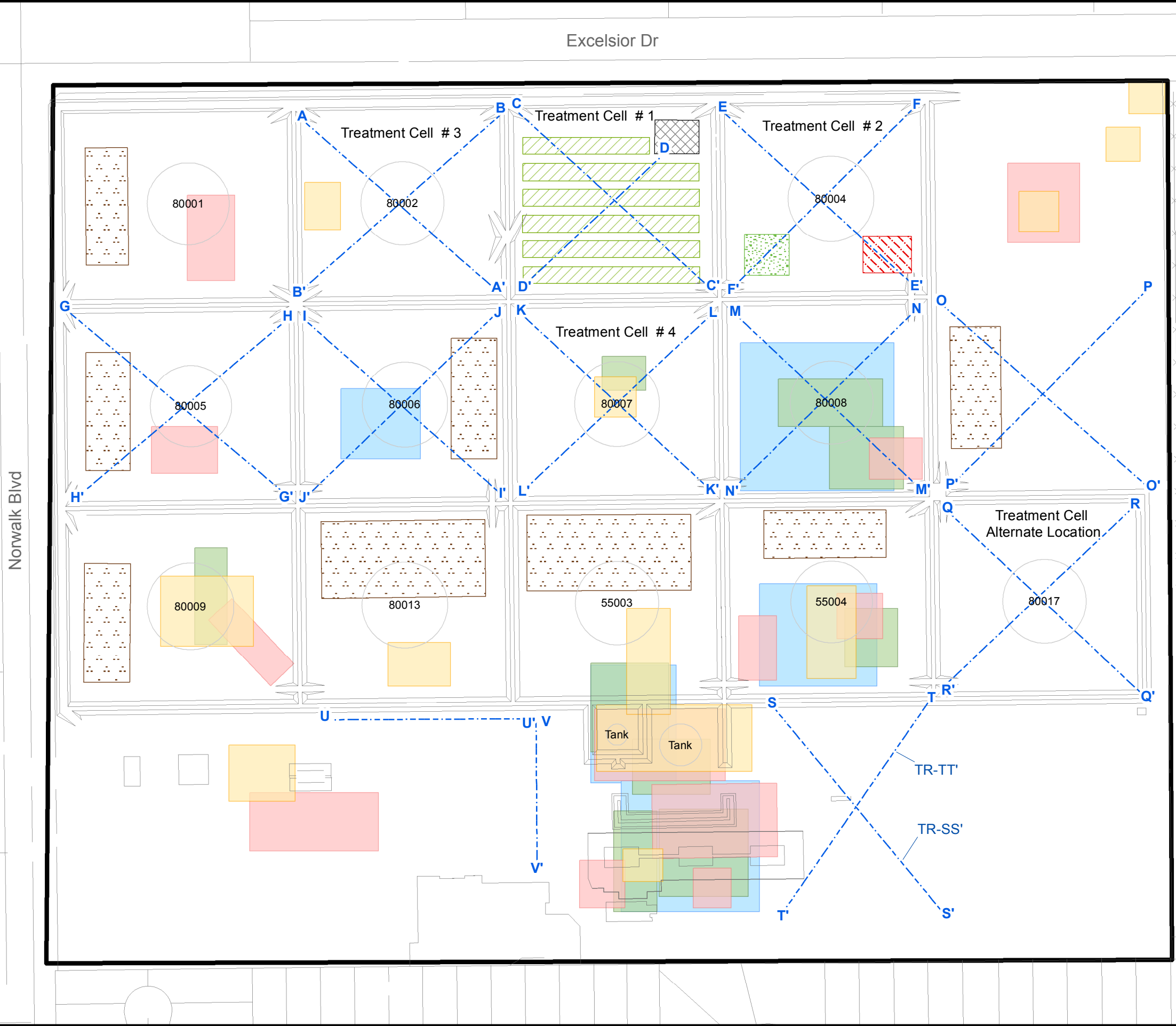
EXCAVATION SUMMARY
 LOCATION: TANK XXX
 ID: ASD234
 START DATE: XX/XX/XX
 COMPLETION DATE: XX/XX/XX
 PLANNED DEPTH: XX FT BGS
 FINAL DEPTH: XX BGS
 ESTIMATED VOLUME: XX CU YDS
 ACTUAL VOLUME: XX CU YDS
 PERIMETER LENGTH: XX FT
 BOTTOM AREA: XX SQ FT
 REQUIRED NUMBER OF BOTTOM SAMPLES: XX
 REQUIRED NUMBER OF SIDEWALL SAMPLES: XX



FINAL BACKFILL DIAGRAM
PLAN VIEW

- LEGEND**
- SOIL SAMPLE LOCATION
 - ▨ TREATED SOIL
 - ▧ TREATED SOIL
 - ▬ CLEAN OVERBURDEN SOIL
 - ◉ CLEAN AGGREGATE

DEFENSE FUEL SUPPORT POINT NORWALK 15306 NORWALK BOULEVARD NORWALK, CA 90650				EXCAVATION DETAILS EXCAVATION ID: XXX	
PROJECT NO.	DATE	DRAWN BY:	APP. BY:	 1962 FREEMAN AVENUE SIGNAL HILL, CA 90755	
04-NDLA-007	01/05/15	AD	KW		
NOT TO SCALE				FIGURE 1	



Legend

- Former Above Ground Storage Tanks
- DFSP Norwalk Border
- Excavations 0-5ft bgs
- Excavation 5-10ft bgs
- Excavation 10-15ft bgs
- Excavation 15-25ft bgs
- Clean Soil Stockpile Staging Areas
- Treatment Cells
- Existing Soil Vapor Extraction System
- Treated Soil Staging Area for Placement in Treatment Cell (Excavation Event # 1)
- Contaminated Soil Staging Area Prior to Treatment (Excavation Event # 1)
- Exploratory Trenching

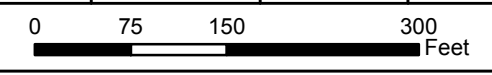
Notes:

1. Up to four treatment cells will be established.
2. Staging areas of contaminated soil and treated soil prior to placement in treatment cell will move throughout site as field conditions dictate.
3. Production rate ranges between 1,000 cubic yards and 2,000 cubic yards per day.
4. Bio-piles for only 1 of 4 cells shown.



DFSP Norwalk
15306 Norwalk Boulevard
Norwalk, California

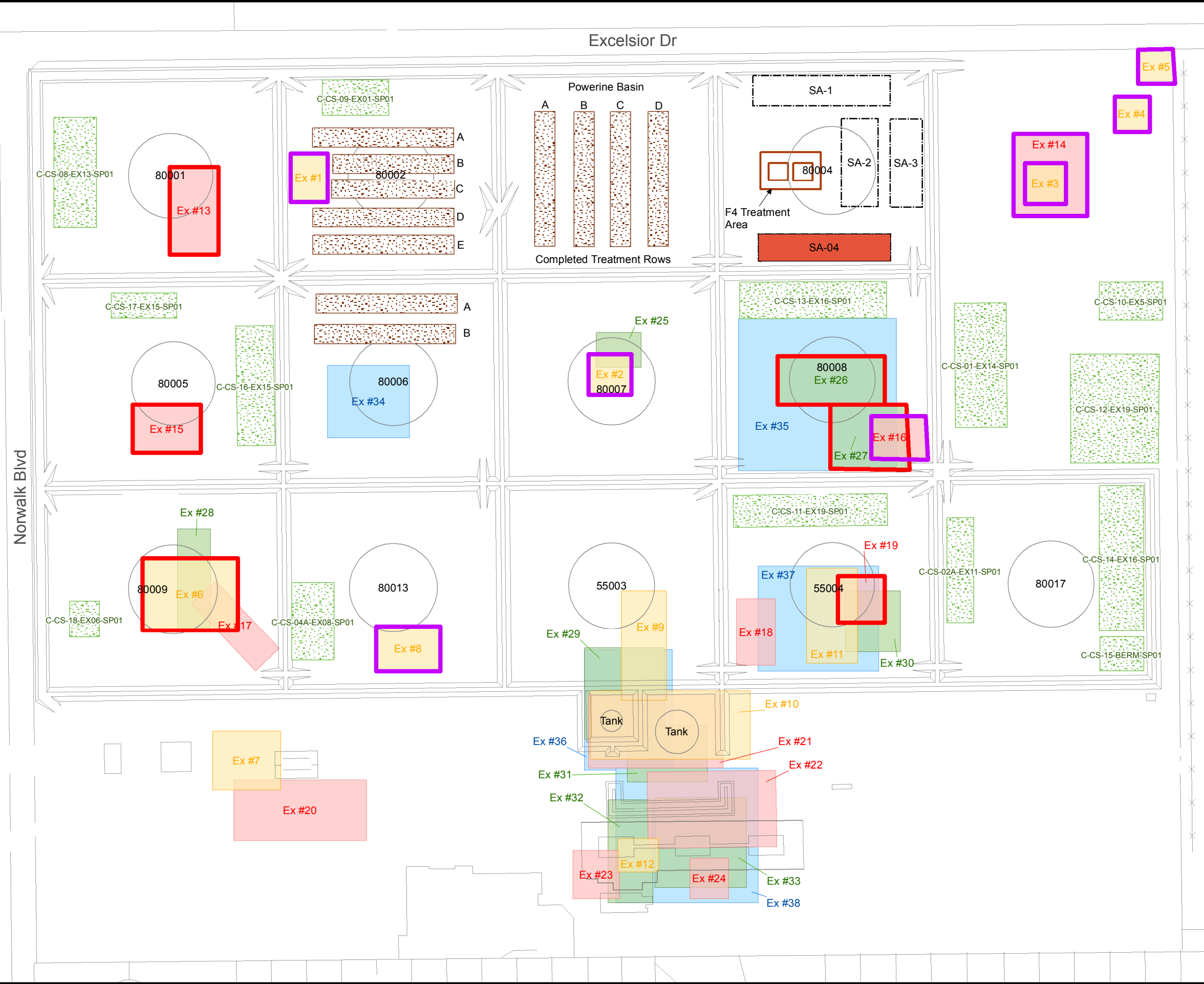
Project Number:	Date:	Drawn By:	Approved By:
04-NDLA-007	1/26/2015	A. Czuba	K. Wall



Operation Plan

SGI environmental
THE SOURCE GROUP, INC.
1962 Freeman Avenue
Signal Hill, CA 90755
(562) 597-1055

Figure
2



Legend

- Former Above Ground Storage Tanks
- DFSP Norwalk Border
- EX # 11 Proposed Excavation 0-5ft
- EX # 14 Proposed Excavation 5-10ft
- EX # 27 Proposed Excavation 10-15ft
- EX # 38 Proposed Excavation 15-25ft
- Completed Excavations
- Excavations in Progress
- SA-4 Staging Area with Soil
- Clean Soil Pile
- SA-3 Staging Area - Empty
- Completed Treatment Row

Notes:

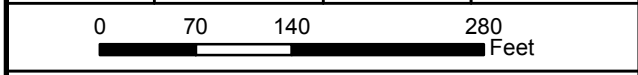
Completed Excavations as of June 8, 2015:
1, 2, 3, 4, 5, 8, 14, 16

Excavations in Progress as of June 8, 2015:
6, 13, 15, 19, 26, 27



DFSP Norwalk
15306 Norwalk Boulevard
Norwalk, California

Project Number:	Date:	Drawn By:	Approved By:
04-NDLA-007	06/11/2015	A. Czuba	K. Wall



**Excavation IDs and Clean Soil Areas
(From March 16 - June 8, 2015)**

THE SOURCE GROUP, INC.	Figure
1962 Freeman Avenue Signal Hill, CA 90755 (562) 597-1055	3

Figure -4
Confirmation Sampling Grid for Assumed Clean Stockpiles

Stockpile ID: 05056002
 Source: Excavation 05
 Interval: 0 to 5
 Ordinal Date: 60
 Lot: 2

Volume of a Rectangular Trapezoidal Trough
 $V = (H/3) [WL + \sqrt{Wlab} + ab]$

Hieght	h	8	Confirmation Samples Req	14
Width of Bottom	W	20		
Length of bottom	L	100		
Width of top	a	4		
Length of top	b	84		
V		9003 sq ft		
Cu yards		333		
Tons		533		

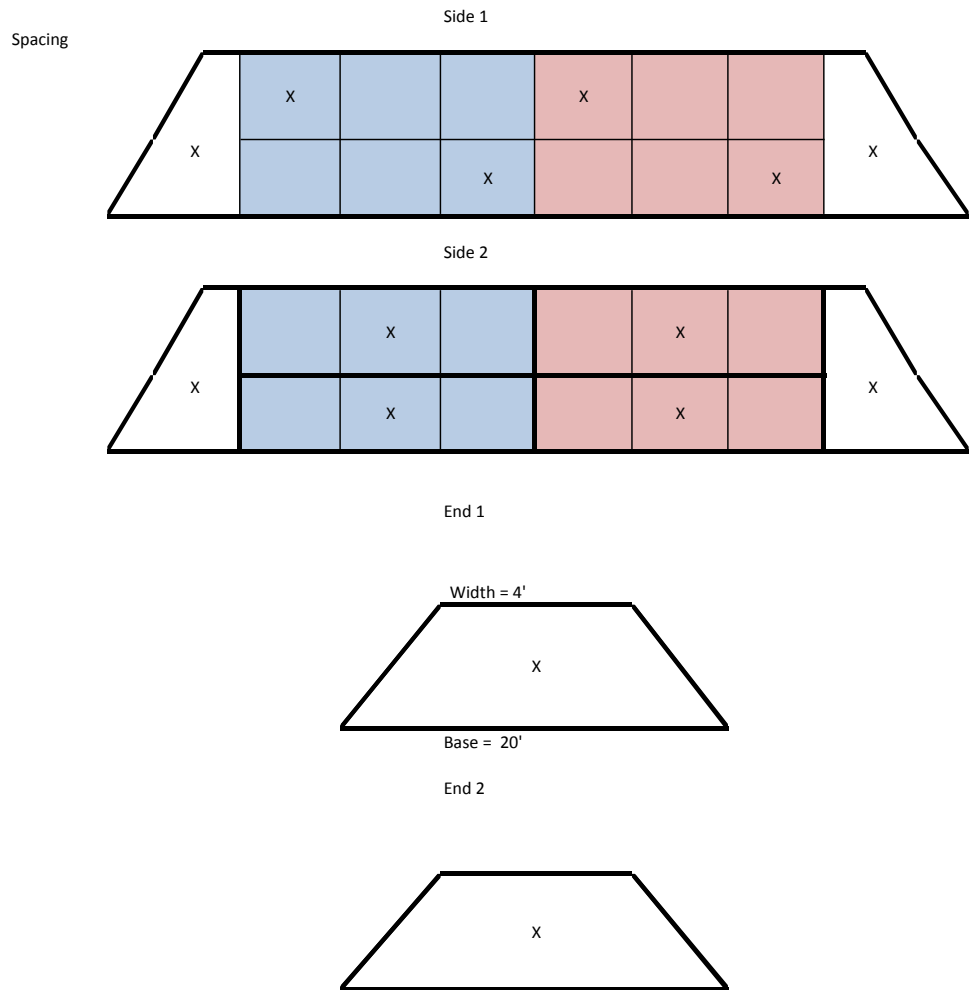


Figure 5
Confirmation Sampling Grid for Treated Stockpiles

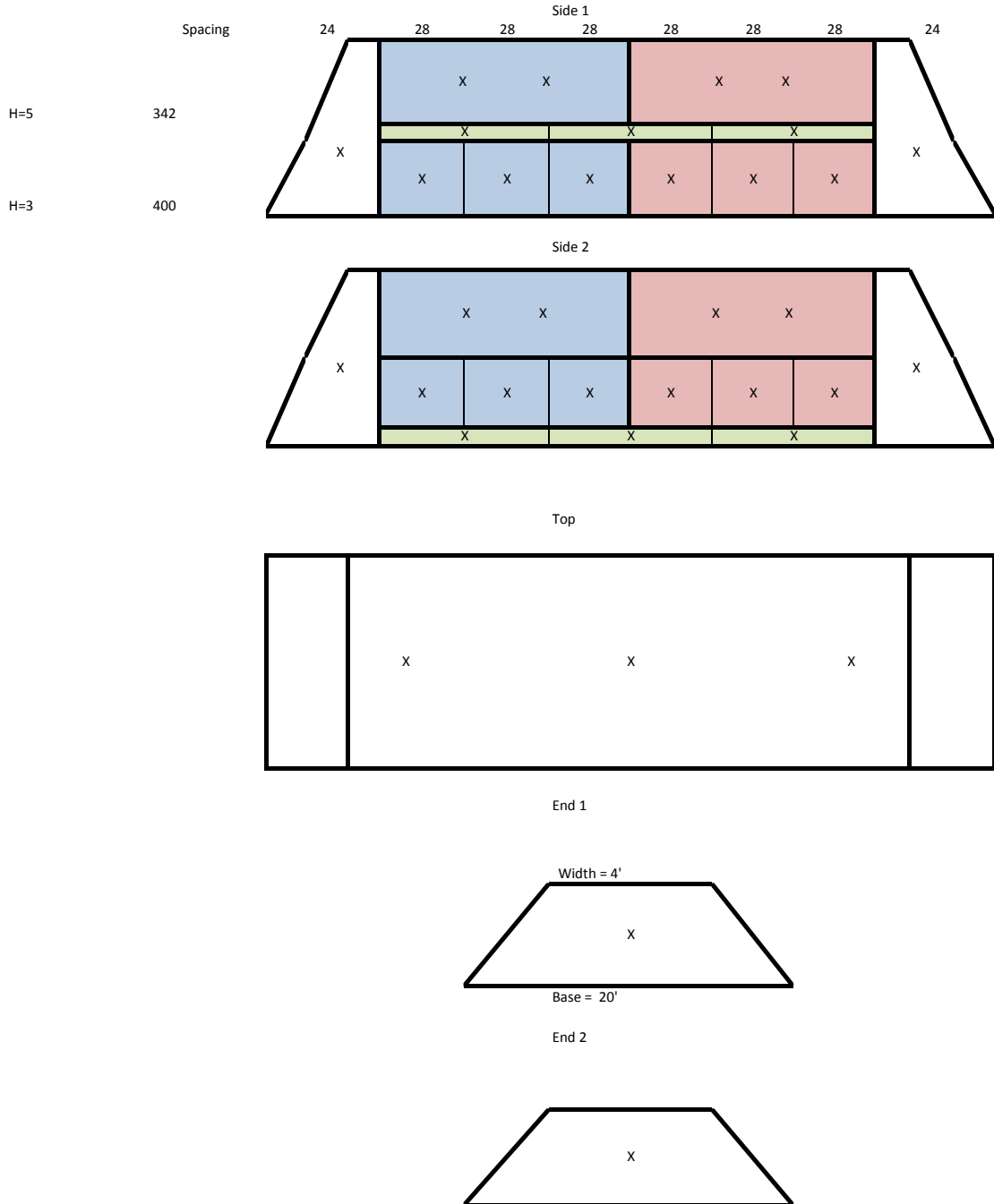
Treatment Row ID:
Date:

Volume of a Rectangular Trapezoidal Trough
 $V = (H/3) [WL + \text{sqrt}(Wlab) + ab]$

Hieght	h	8
Width of Bottom	W	20
Length of bottom	L	215
Width of top	a	4
Length of top	b	199
V		20042.6667 sq ft
Cu yards		742.320988
Tons		1187.71358

Confirmation Samples Reqt 35

Note: Samples in green will be obtained from middle of pile via access through side or top



TABLES

Table 1
Data Definition

Excavation ID	Valid Value	Notes
Series	EX	
Excavation number	01 through 38	See Figure 2 for Excavation ID location
Trench ID	Valid Value	Notes
Series	TR	
Trench designation	AA, BB, CC, DD, EE, FF, GG, HH, JJ, KK, LL, MM	See Figure 3 for Trenching ID location
Example	TR-AA	Trench AA
Assumed Clean Stock Pile ID	Valid Value	Notes
Series	C	
Clean Soil Storage Area	See Clean Soil Storage Area	
Excavation Number	See excavation ID table	
Stockpile Number	SP01, SP02 ...	
Example	C-CS-01-EX01-SP02	Assumed Clean stockpile from Clean Storage Area 01, from excavation 01, second stock pile removed
Soil Stockpile going to Treatment ID	Valid Value	Notes
Series	T	
Excavation Number	See excavation ID table	See Figure 2 for Excavation ID location
Stockpile Number	SP01, SP02 ...	
Example	T-EX15-SP03	Stockpile going to treatment from Excavation 15, 3rd stockpile removed
Treatment Pile ID	Valid Value	Notes
Treatment Cell Area	See Area Names List below	
Row	A-F	Rows oriented North-South in a treatment cell will be identified "A" through "F" going from west to east (left to right). Rows oriented East-West will be identified "A" through "F" going from north to south (top to bottom).
Sequence	01, 02 etc.	Each time a row is used the sequence increments
Sample Ports	SP-1, SP-2 etc.	Rows oriented North-South, label down ascending. Rows oriented East-West, label left to right.
Example	80008-B-02-SP-1	Treatment cell area 80008, Row B, 2nd pile treated in this row
Clean Soil Storage Areas ID	Valid Value	Notes
Series	CS	
Area ID	01 through XX	
Example	CS-01	Clean storage area 01
Sample Types	Valid Value	Notes
Baseline Sample of Contaminated Soil	B00001 through B99999	These numbers do NOT start over between excavations or treatment rows. Once a number is used it is NEVER repeated.
Performance Sample	P00001 through P99999	
Confirmation Sample of Treated Soil	T00001 through T99999	
Confirmation Sample of Assumed Clean Soil	C00001 through C99999	
Surface baseline samples of treatment cells/rows	SB0001 through SB99999/80002-ROW1-S-SB100	
Waste Profile Sample	W00001 through W99999	
Excavation Sidewall Sample	Valid Value	Notes
Series	EX	
Excavation ID	01 through 37	
Orientation	N, S, E, W, F	North, South, East, West, Floor
Number of Sample taken from the orientation	1,2,3,4 etc.	
Depth	1,3,5 etc. ft	F (floor) samples will have depth of excavation
Example	EX-01-N2-3	Second sidewall sample from north side of excavation 01 t a depth of 3 ft bgs.

Table 1
Data Definition

Trench Exploratory Samples	Valid Value	Notes
Trench ID	TR-XX	See Trench ID
Sample sequence number	000 - 999	Location and depth of sample to be identified on grid/figure.
Example (Sample Number)	TR-AA-005	
Area Designations	Valid Value	Notes
Powerine -Treatment Cell #1	Powerine	Powerine Basin (SVE and GWTS)
80002 - Treatment Cell #3	80002	Historical tank basin 80002
80004 - Treatment Cell #2	80004	Historical tank basin 80004
80007 - Treatment Cell #4	80007	Historical tank basin 80007
80017 - Treatment Cell #5	80017	Historical tank basin 80017
80001	80001	Historical tank basin 80001
80005	80005	Historical tank basin 80005
80006	80006	Historical tank basin 80006
80008	80008	Historical tank basin 80008
80009	80009	Historical tank basin 80009
80013	80013	Historical tank basin 80013
5503	5503	Historical tank basin 55003
5504	5504	Historical tank basin 55004
South-West	South-West	Excavation cluster in southwest area
South-Central	South-Central	Excavation cluster in south central area
North-East	North-East	Excavation cluster in north east area (next to Hollifield park)

Table 2
Protocol to Estimate the Minimum Number of Samples

Stockpile Size Unit = cubic yards (cy)	Sampling Frequency
<500	1 sample for every 25 cy (e.g., 20 samples for a 500 cy stockpile)
500 to < 1,000	20 samples plus 1 sample for every 100 cy in excess of the initial 500 cy (e.g., 25 samples for 1,000 cy stockpile)
1,000 to 10,000	25 samples plus 1 sample for every 500 cy in excess of the initial 1,000 (e.g., 43 samples for a 10,000 cy stockpile)
>10,000	43 samples plus 1 for every 5,000 cy in excess of the initial 10,000 cy (e.g., 61 samples for a 100,000 cy stockpile)

Note:

Reference: April 10, 2012, RWQCB Correspondence on Soil Management Plan.

Table 3
Analytical Test Methods, Sample Container, Preservation, and Holding Time Requirements
Defense Fuel Support Point
 Norwalk, California

Water				
Parameter	Preservative	Holding Time	EPA Method #	Container
VOCs	4°C; HCL; no HS	14 days	Former 8010 List by 8260B or 8260B+gasoline	3 x 40ml glass vials
TPH-gasoline	4°C; HCL; no HS	14 days	8260 GRO	2 x 40ml glass vials
TPH-Diesel	4°C	7 days (extraction) 40 days (analysis)	EPA 8015B diesel range	1L amber glass
Soil				
Parameter	Preservative	Holding Time	EPA Method #	Container
VOCs	4°C	14 days	Epa 5035 Sodium Bisulfate/Methanol Method; Former 8010 List by 8260B or 8260B	3 VOAS with Methanol/Sodium Bisulfate
TPH-Diesel	4°C	14 days (extraction) 40 days (analysis)	8015B diesel extractable	brass or butyrate tube/4 oz. wide mouth glass jar
TPH-gasoline	4°C	14 days	8260B + gasoline range organics	3 VOAS with Methanol/Sodium Bisulfate
Hazardous Waste Characterization for Toxicity - Samples for STL/C/TCLP Extraction				
Parameter	Preservative	Holding Time (from field collection to extraction)	EPA Method #	Container
Metals	None	6 months	6010B	250 ml jar
VOCs	None	7 days	8010 List by 8260B	3 x 40ml glass vials
Mercury	HNO ₃ , pH<2	28 days	7470A / 7471A	16 oz plastic
Metals except Mercury	HNO ₃ , pH<2	180 days	6010B	16 oz plastic

Legend:

VOCs = Volatile organic compounds
 TPH = Total petroleum hydrocarbons
 HS = Headspace
 HCL = Hydrochloric Acid

APPENDIX A
SITE CLEANUP GOALS



June 15, 2015

Paul Cho, P.G.
Water Resources Control Engineer
California Regional Water Quality Control Board, Site Cleanup Unit IV
Los Angeles Region
320 West 4th Street, Suite 200
Los Angeles, CA 90013

Subject: Proposed Addendum to the Soil Cleanup Goals
Defense Fuel Support Point Norwalk
15306 Norwalk Boulevard, Norwalk, California
(SCP NO. 0286A, Site ID NO. 16638)

Dear Mr. Cho:

On July 12, 2012, the LARWQCB approved soil cleanup goals for the former Defense Fuel Support Point (DFSP) Norwalk facility, located at 15306 Norwalk Boulevard, Norwalk, California. The approved cleanup goals included three ranges of total petroleum hydrocarbons (TPH), specifically C4-C12, C8-C17, and C5-C25 (where C represents carbon and the following number represents the number of carbons present in the hydrocarbon molecule).

However, longer chain hydrocarbons (C25 and greater) were not included in the list of approved site cleanup goals. To address the full range of hydrocarbons present in site soils, the Department of Logistics Agency - Energy (DLA Energy) and The Source Group, Inc. (SGI) reviewed the protocol used to develop the cleanup goals for soil.

The cleanup goals were based on the application of the LARWQCB's 1996 Interim Site Assessment and Cleanup guidebook (Guidebook). The Guidebook specifies that the soil cleanup goals should be calculated by the same general formula used by the United States Environmental Protection Agency (EPA) to calculate Soil Screening Levels (SSLs), as follows:

Soil cleanup goal = total attenuation factor x water quality standard

Table 4-1 of the Guidebook (Attachment A) includes maximum soil screening levels (SSL) for hydrocarbon compounds based on carbon range and depth to the underlying groundwater. As an example, at sites where the depth of the contamination is between 20 to 150 feet, the C13-C22 hydrocarbons is 1,000 milligrams per kilogram (mg/kg), whereas the longer chain hydrocarbons in the C23-C32 range, a SLS of 10,000 mg/kg is derived based on the greater attenuation rate for longer chain hydrocarbons. As the length of the hydrocarbon chains increases, the corresponding SSL also increases. Conversely, for a given hydrocarbon range,

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as the depth to groundwater decreases so does the SSL (e.g., for the C13-C22 carbon range, the SSL for depth to groundwater between 20 and 150 feet is 1,000 mg/kg whereas the SLS decreases to 100 mg/kg when the depth to groundwater is less than 20 feet.

You will recall that the cleanup level approved for the Norwalk site with the longest-chain TPH values (C5-C25) is comparable to the SSL values provided for carbon range C13-C22 in the Guidebook Table 4-1. However, no cleanup goals were provided for longer chain hydrocarbon ranges in the July 12, 2012 correspondence. Below is a summary of the cleanup goals provided in the July 12, 2012, correspondence:

July 12, 2012 Approved Soil Cleanup Goals	(feet below ground surface)					
	0.5	5	10	15	20	25
Depth Below Ground Surface						
Depth to Groundwater	25.5	21	16	11	6	1
Constituent	Proposed Soil Cleanup TPH Goal (mg/kg)					
TPH as Gasoline (C4-C12)	500	500	100	100	100	100
TPH as JP-5 (C8-C17)	500	500	100	100	100	100
TPH as Diesel (C5-C25)	1,000	1,000	100	100	100	100

Using the methodology used to develop SSL in the Guidebook, the cleanup goal for the longest chain hydrocarbons (C26-C44) would be up to an order of magnitude higher than the cleanup goal provided for the C23-C32 range in Table 4-1. The July 12, 2012, LARWQCB Soil Cleanup goal table for TPH concentrations is proposed to be reflect those levels provided in Table 4-1 of the RWQCB guidance as presented below.

Proposed Revised Soil Cleanup Goals	(feet below ground surface)					
	0.5	5	10	15	20	25
Depth Below Ground Surface						
Depth to Groundwater	25.5	21	16	11	6	1
Constituent	Proposed Soil Cleanup TPH Goal (mg/kg)					
Carbon Range (C4-12)	500	500	100	100	100	100
Carbon Range (C13-C22)	1,000	1,000	100	100	100	100
Carbon Range (C23-C32)	10,000	10,000	1,000	1,000	1,000	1,000
Carbon Range (C33-C44)	50,000	50,000	10,000	10,000	10,000	10,000

Carbon Ranges C4 to C12 concentrations will be determined with EPA Method 8260 analysis; Carbon Ranges C13 to C44 will be determined with EPA Method analysis.

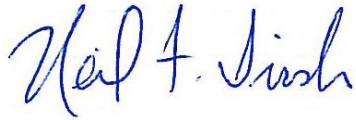
We also recommend that the proposed cleanup goals for volatile components be modified to recognize common laboratory detection limits. We have prepared the attached table (Appendix B) that summarizes the cleanup goals for VOCs as provided in the July 12, 2012, and included state of California-certified laboratory American Analytics' typical detection limits for these compounds in soil. For those cleanup goals that are less than the detection limit, we have highlighted the values with red, bold font. It is proposed that cleanup goals be revised to reflect either the analytical laboratory detection limit or the current proposed cleanup goal, whichever value is higher.

DLA and SGI believe that this proposed cleanup goal addendum is consistent with site cleanup directives and the Guidebook, and we appreciate RWQCB's concurrence with this proposed

addendum to allow for implementation of the soil remediation program underway at the site.

We appreciate the LARWQCB considering this request. If you have any questions, please call me at (562) 597-1055.

Sincerely,



Neil F. Irish, P.G. 5484
Principal Geologist
The Source Group, Inc

Cc:

Mr. Nicolas Carros, DLA Energy

File: DFSP Norwalk – 04-NDLA-007

Attachments: Attachment A – LARWQCB Interim Site Assessment and Cleanup
 Guidebook Table 4-1
 Attachment B – Table 1 – Comparison of Laboratory Detection Limits to
 Soil Cleanup Goals – DFSP Norwalk

Attachment A

Table 4-1: Maximum Soil Screening Levels (mg/kg) for TPH, BTEX and MTBE above Drinking Water Aquifers

T P H	Distance Above Groundwater	Carbon Range			
		C4-C12	C13-C22	C23-C32	
	>150 feet	1,000	10,000	50,000	
	20-150 feet	500	1,000	10,000	
<20 feet	100	100	1,000		

B T E X & M T B E	Distance Above Groundwater	Lithology			
		Gravel	Sand	Silt	Clay
	150 feet	B=0.044 T=2 E=8 X=23 MTBE = 0.039	B=0.077 T=4 E=17 X=48 MTBE = 0.078	B=0.165 T=9 E=34 X=93 MTBE = 0.156	B=0.8 T=43 E=170 X=465 MTBE = 0.78
	120 feet	B=0.035 T=1.57 E=6.3 X=17.9 MTBE = 0.028	B=0.058 T=3.1 E=12.7 X=36 MTBE = 0.061	B=0.123 T=7 E=25.9 X=70.3 MTBE = 0.117	B=0.603 T=32 E=128 X=351 MTBE = 0.591
	100 feet	B=0.028 T=1.3 E=5.1 X=14.4 MTBE = 0.020	B=0.046 T=2.57 E=9.86 X=28 MTBE = 0.05	B=0.094 T=5.4 E=20.4 X=55.1 MTBE = 0.091	B=0.471 T=25 E=101 X=276 MTBE = 0.464
	80 feet	B=0.022 T=1 E=4 X=11 MTBE = 0.013	B=0.033 T=2 E=7 X=20 MTBE = 0.039	B=0.066 T=4 E=15 X=40 MTBE = 0.065	B=0.34 T=18 E=73 X=200 MTBE = 0.338
	60 feet	B=0.018 T=0.72 E=2.9 X=7.9 MTBE = 0.013	B=0.026 T=1.4 E=4.9 X=13.9 MTBE = 0.03	B=0.048 T=2.8 E=10.7 X=28.4 MTBE = 0.048	B=0.241 T=13 E=52 X=141.5 MTBE = 0.247
	40 feet	B=0.015 T=0.43 E=1.8 X=4.8 MTBE = 0.013	B=0.018 T=0.87 E=2.8 X=7.8 MTBE = 0.022	B=0.029 T=1.6 E=6.3 X=16.9 MTBE = 0.03	B=0.143 T=7.5 E=30 X=83 MTBE = 0.156
20 feet	B=0.011 T=0.15 E=0.7 X=1.75 MTBE = 0.013	B=0.011 T=0.3 E=0.7 X=1.75 MTBE = 0.013	B=0.011 T=0.45 E=2 X=5.3 MTBE = 0.013	B=0.044 T=2.3 E=9 X=24.5 MTBE = 0.065	

- TPH = Total petroleum hydrocarbons.
- BTEX = benzene, toluene, ethylbenzene, and xylenes, respectively. MTBE = methyl tertiary butyl ether.
- Respective MCLs (ppm): B=0.001, T=0.15, E=0.7, X=1.75, MTBE=0.013.
- BTEX screening concentrations determined per the attenuation factor method as described in RWQCB Guidance for VOC Impacted Sites (March 1996), with a natural degradation factor of 11 for BTEX and of 3 for MTBE. Table

- values can be linearly interpolated between distance above groundwater and are proportional to fraction of each lithological thickness.
- Values in Table 4-1 are for soils above drinking water aquifers. All groundwaters are considered as drinking water resources unless exempted by one of the criteria as defined under SWRCB Resolution 88-63 (TDS>3000 mg/L, or deliverability <200 gal/day, or existing contamination that cannot be reasonably treated). Regional Board staff will make a determination of potential water use at a particular site considering water quality objectives and beneficial uses. For non-drinking water aquifers, regardless of depth, TPH for ">150 feet" category in the table should be used.
 - Distance above groundwater must be measured from the highest anticipated water level. Lithology is based on the USCS scale.
 - In areas of naturally-occurring hydrocarbons, Regional Board staff will make determinations on TPH levels.

(revised 1/7/05)

ATTACHMENT B

TABLE 1
COMPARISON OF LABORATORY DETECTION LIMITS TO SOIL CLEANUP GOALS
DFSP Norwalk
15306 Norwalk Boulevard, Norwalk, California

	Acetone (mg/kg)	tert-Amyl Methyl Ether (TAME) (mg/kg)	Benzene (mg/kg)	Bromobenzene (mg/kg)	Bromochloromethane (mg/kg)	Bromodichloromethane (mg/kg)	Bromoform (mg/kg)	Bromomethane (mg/kg)	2-Butanone (MEK) (mg/kg)	tert-Butyl alcohol (TBA) (mg/kg)	sec-Butylbenzene (mg/kg)	tert-Butylbenzene (mg/kg)	n-Butylbenzene (mg/kg)	Carbon Disulfide (mg/kg)	Carbon Tetrachloride (mg/kg)	Chlorobenzene (mg/kg)	Chloroethane (mg/kg)	Chloroform (mg/kg)	Chloromethane (mg/kg)	2-Chlorotoluene (mg/kg)	4-Chlorotoluene (mg/kg)	1,2-Dibromo-3-chloropropane (mg/kg)	Dibromochloromethane (mg/kg)	1,2-Dibromoethane (EDB) (mg/kg)
STD Lab D/L	<0.050	<0.0050	<0.0020	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.050	<0.020	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.010	<0.0050	<0.0050
July 12, 2012 CGs (DTW = 25.5 ft bgs)	0.994	NE	0.015	NE	NE	NE	NE	0.0015	0.557	0.001	2.59	2.07	2.18	0.049	NE	0.119	2.23	0.0000738	NE	0.558	0.547	0.00025	NE	0.0000305
July 12, 2012 CGs (DTW = 1.0 ft bgs)	1.60	NE	0.012	NE	NE	NE	NE	0.0010	0.661	0.0016	0.129	0.110	0.114	0.023	NE	0.013	2.83	0.0000	NE	0.039	0.038	0.0000352	NE	0.0000096

	Dibromomethane (mg/kg)	1,2-Dichlorobenzene (mg/kg)	1,3-Dichlorobenzene (mg/kg)	1,4-Dichlorobenzene (mg/kg)	Dichlorodifluoromethane (R12) (mg/kg)	1,1-Dichloroethane (mg/kg)	1,2-Dichloroethane (EDC) (mg/kg)	trans-1,2-Dichloroethylene (mg/kg)	cis-1,2-Dichloroethylene (mg/kg)	1,1-Dichloroethylene (mg/kg)	1,2-Dichloropropane (mg/kg)	1,3-Dichloropropane (mg/kg)	2,2-Dichloropropane (mg/kg)	1,1-Dichloropropylene (mg/kg)	trans-1,3-Dichloropropylene (mg/kg)	cis-1,3-Dichloropropylene (mg/kg)	Diisopropyl ether (DIPE) (mg/kg)	Ethylbenzene (mg/kg)	Ethyl-tert-Butyl Ether (ETBE) (mg/kg)	Hexachlorobutadiene (mg/kg)	2-Hexanone (MBK) (mg/kg)	Isopropylbenzene (mg/kg)	4-Isopropyltoluene (mg/kg)
STD Lab D/L	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0020	<0.0050	<0.010	<0.050	<0.0050	<0.0050
July 12, 2012 CGs (DTW = 25.5 ft bgs)	NE	NE	NE	NE	0.984	NE	0.000106	NE	NE	NE	NE	NE	NE	NE	NE	NE	0.449	2.07	NE	NE	0.0073	5.56	2.82
July 12, 2012 CGs (DTW = 1.0 ft bgs)	NE	NE	NE	NE	0.167	NE	0.0000692	NE	NE	NE	NE	NE	NE	NE	NE	NE	0.212	1.10	NE	NE	0.0047	0.303	0.154

	4-Methyl-2-pentanone (MIBK) (mg/kg)	Methylene Chloride (mg/kg)	Methyl-tert-Butyl Ether (MTBE) (mg/kg)	Naphthalene (mg/kg)	n-Propylbenzene (mg/kg)	Styrene (mg/kg)	1,1,1,2-Tetrachloroethane (mg/kg)	1,1,2,2-Tetrachloroethane (mg/kg)	Tetrachloroethylene (PCE) (mg/kg)	Toluene (mg/kg)	1,1,2-Trichloro-1,2,2-trifluoroethane (R113) (mg/kg)	1,2,3-Trichlorobenzene (mg/kg)	1,2,4-Trichlorobenzene (mg/kg)	1,1,1-Trichloroethane (mg/kg)	1,1,2-Trichloroethane (mg/kg)	Trichloroethylene (TCE) (mg/kg)	Trichlorofluoromethane (R11) (mg/kg)	1,2,3-Trichloropropane (mg/kg)	1,2,4-Trimethylbenzene (mg/kg)	1,3,5-Trimethylbenzene (mg/kg)	Vinyl chloride (mg/kg)	o-Xylene (mg/kg)	m,p-Xylenes (mg/kg)
STD Lab D/L	<0.050	<0.050	<0.0050	<0.010	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0020	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0020	<0.0020
July 12, 2012 CGs (DTW = 25.5 ft bgs)	NE	0.000778	0.000907	0.270	2.18	0.463	NE	0.0023	NE	0.614	NE	0.0740	NE	NE	0.0032	0.0070	NE	0.000000874	2.10	2.06	NE	5.55	5.55
July 12, 2012 CGs (DTW = 1.0 ft bgs)	NE	0.000682	0.000686	0.012	0.114	0.030	NE	0.0002	NE	0.367	NE	0.0034	NE	NE	0.0008	0.0009	NE	0.000000123	0.120	0.118	NE	2.84	2.84

Notes: STD Lab D/L = standard laboratory detection limit.
July 12, 2012 CGs (DTW = 25.5 ft bgs) = cleanup goals approved by RWQCB; DTW = 25.2 ft bgs.
DTW = depth to water.
ft bgs = feet below ground surface.
mg/kg = milligrams per kilogram.
RWQCB = Regional Water Quality Control Board.
NE = cleanup goal not established.
Red Font = STD Lab D/L is greater than the approved RWQCB Cleanup Goal.

APPENDIX B

EPA METHOD 5035

SUMMARY OF FIELD SAMPLING AND LABORATORY ANALYTICAL PROTOCOL

**SUMMARY OF FIELD METHOD
PROPOSED SOIL SAMPLING AND SAMPLE PRESERVATION,
NORWALK DFSP
Terra Core™ Sampler with Methanol and Sodium Bisulfate Preservative
June 2015**

Obtain sets of 3 pre-weighed vials prepared by the laboratory. Vials should contain sodium bisulfate (2 vials) and methanol (1 vial) in the measured quantities in vials weighed by the laboratory. The sodium bisulfate vials may contain a magnetic stir bar.

Using a disposable Terra Core™ sampler, collect a 5 gram sample by pushing the barrel of the coring tool into a freshly exposed surface and then remove the corer once filled. Quickly wipe the exterior of the barrel with a clean disposable towel. Immediately extrude the sample into a vial by gently pushing the plunger. Hold the vial at an angle when extruding the sample into the container to minimize splashing. Prior to capping the vial, inspect the lip and threads of the sample vessel and remove any foreign debris with a clean towel, allowing an airtight seal to form. Observe the soil for effervescence, an indication of carbonate reaction with the acid. If effervescence is noted, consider an alternate sample preservation method.

Collect a total of 3 core samples from each location, adding soil to the 3 vials containing sodium bisulfate and methanol.

After labeling the vials, place the 3 vials in a Ziploc bag and in a chilled cooler along with the appropriate chain of custody. The vials must arrive at the laboratory within 48 hours of sample collection, and analysis must be completed by the laboratory within 14 days of sample collection.

Recommended Use Of The Terra Core®



NOTE: The Terra Core® Sampler is a single use device. It cannot be cleaned and/or reused.



Step 1

Have ready a 40ml glass VOA vial containing the appropriate preservative. With the plunger seated in the handle, push the Terra Core® into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 or 10 grams of soil.



Step 2

Wipe all soil or debris from the outside of the Terra Core® sampler. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.



Step 3

Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the 40ml VOA vial containing the appropriate preservative and extrude the sample by pushing the plunger down. Quickly place the lid back on the 40ml VOA vial. **Note:** When capping the 40ml VOA vial, be sure to remove any soil or debris from the top and/or threads of the vial.

**GUIDANCE DOCUMENT FOR THE IMPLEMENTATION OF
UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
METHOD 5035: METHODOLOGIES FOR COLLECTION,
PRESERVATION, STORAGE, AND PREPARATION OF SOILS
TO BE ANALYZED FOR VOLATILE ORGANIC COMPOUNDS**

**Department of Toxic Substances Control
California Environmental Protection Agency**

November 2004



ACKNOWLEDGEMENTS

Preparation of this guidance document was achieved through the efforts of the following individuals at the Department of Toxic Substances Control:

Craig Christmann	Senior Engineering Geologist
Dan Gallagher	Senior Engineering Geologist
Theo Johnson	Senior Engineering Geologist
Greg Sweel	Senior Engineering Geologist
Fred Zanoria	Senior Engineering Geologist

Additional assistance was provided by Ruth Chang, Russ Chin and William Lum of the Hazardous Materials Laboratory at the Department of Toxic Substances Control. We thank them for their substantial contribution towards the completion of this guidance document.

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1.0 INTRODUCTION

The United States Environmental Protection Agency (USEPA) Office of Solid Waste promulgated Method 5035, *Closed-System Purge-and-Trap Extraction for Volatile Organics in Soil and Waste Samples* in June 1997 in SW-846, *Test Methods for Evaluating Solid Waste, Physical / Chemical Methods, Update III* (Method 5035). More recently, in July 2002, USEPA updated the Method within SW-846 as Method 5035A¹. Method 5035 describes procedures and protocols for the collection of three types of solid samples contaminated with volatile organic compounds (VOCs)²: low-concentration solids (i.e., soil, sludge and sediment), high-concentration solids, and solid samples with oily waste. For low-concentration samples, Method 5035 describes a “closed-system purge-and-trap” process to minimize the loss of VOCs due to sample collection and handling. For high-concentration samples, Method 5035 describes procedures for the collection and preservation of samples, but references Method 5030 (Revision 2, December 1996) for the actual analysis of the prepared sample extracts. Method 5030 remains as a part of Method 5035 and is applicable to the analysis of high-concentration soil and solid waste extracts prepared with Method 5035, as well as aqueous samples. As such, when USEPA promulgated Method 5035, there was no intention to make Method 5030 obsolete.

The procedures in Method 5035 should be used for the collection of soil samples at all sites in California contaminated with VOCs in order to comply with USEPA Region IX’s Interim Policy and the California Code of Regulations. USEPA Region IX issued their Regional Interim Policy concerning Method 5035 on June 23, 1999. The Region IX Interim Policy requires the use of Method 5035, or an equally or more effective method, for the collection of VOC data for soil in California. The Region IX Interim Policy is included in this Guidance Document as Appendix D. The objective of the Interim Policy is to minimize VOC loss from volatilization and biodegradation during sample collection and handling. By minimizing soil sample transfer steps from sampling to analysis, VOC loss due to atmospheric volatilization is reduced. The use of chemical preservatives further minimizes microbial action, yielding soil samples that are more representative of site conditions. Likewise, the protocols of SW-846 are referenced within the California Code of Regulations as a mechanism to achieve representative samples of waste materials (Title 22, Chapter 11, Article 5, Appendix I).

2.0 PURPOSE

The Department of Toxic Substances Control (DTSC) has compiled this Guidance Document in order to provide assistance in the implementation of Method 5035 at sites regulated by the DTSC where VOCs are chemicals-of-concern. This Guidance Document is meant to supplement USEPA Method 5035 by summarizing the sampling options for the collection of soil samples for VOC analysis. It is DTSC’s intent to provide the minimum requirements and minimum standards to prevent loss of VOCs during sample collection and handling. Thus, DTSC encourages all parties involved in site cleanup to read and understand SW-846 in conjunction with the use of this Guidance Document. This Guidance Document, along with SW-846 and the Region IX Regional Interim Policy, will provide technically defensible and consistent approaches for sampling VOCs in soils. However, while Method 5035 should always be implemented at sites in California

¹ Method 5035 and the associated 2002 update in SW-846 are collectively referred to as “5035” in this Guidance Document.

² The term “volatile organic compounds” refers to low molecular weight compounds which possess boiling points below 200°C, are insoluble or slightly soluble in water, and have been traditionally analyzed by purge-and-trap methods.

contaminated with VOCs, the procedures within this Guidance Document are recommendations only. Other technically equivalent procedures may exist that minimize VOC loss during soil sample collection, storage, preservation, and preparation, and the intent of this Guidance Document is not to exclude alternative sampling approaches as long as the alternative procedures are functionally equivalent to Method 5035. This Guidance Document addresses the collection and handling of soil samples. Sludge samples and sediment samples are not specifically addressed, although some of the procedures herein may apply. Likewise, this Guidance Document does not address the collection of solid samples contaminated with oily waste.

This Guidance Document does not address all aspects of VOC soil sampling and analysis. The focus of this Guidance Document is the field procedures associated with soil sample collection, storage, preservation, and preparation for VOC analysis, since most VOC loss during soil sampling occurs before the samples arrive at the laboratory. It is not the intent of this Guidance Document to provide specific instructions to stationary and mobile laboratories on how to perform the analysis of VOCs in soil samples, but rather to provide guidance on the collection of soil samples in the field.

3.0 SCOPE

The implementation of Method 5035 impacts multiple technical disciplines. Therefore, successful implementation of Method 5035 in the field will require increased communication, planning, and coordination among the project team responsible for site characterization. Method 5035 is more complex than previous soil sample preparation methods because it involves multiple soil preservation options for the project team, each suited for specific project objectives depending upon the action levels for the chemicals-of-concern and prior knowledge of the soil VOC concentrations. The final selection of sampling procedures will require input from all data users, such as project managers, geologists, chemists, risk assessors, and engineers. Items to consider before selecting the VOC sampling and analysis methods at a site are as follows:

- Compounds of interest
- Concentration range of the VOCs
- Potential compound interferences
- Data quality objectives
- Physical character of the soil
- Reactive character of the soil
- Chemical preservation techniques
- Laboratory equipment specifications

Accordingly, a high degree of coordination and planning is required between field and laboratory personnel before the start of field activities.

USEPA Method 5035 arranges the soil sampling options into three groups: low-concentration soil, high-concentration soil, and soil with oily waste. Sample preservation is then given within each group as a sub-option, followed by the appropriate type of sample container to use. This arrangement by USEPA emphasizes the systematic steps that are needed to determine the proper choice of VOC sampling methods, as follows: 1) determine target compounds and their concentration to select low concentration or high concentration methods; 2) select the preservation options that are best suited for the VOC target compounds and data quality objectives; and 3) determine the appropriate container or sampler for the sample collection.

In contrast, this Guidance Document does not emphasize a systematic approach for selecting a soil sampling technique within Method 5035. Rather, the Guidance Document groups the sampling options according to the sampling devices, followed with sub-options for low-concentration and high-concentration methods. The intent of this Guidance Document is to summarize the options available for the sampling of soils contaminated with VOCs and provide detailed field procedures for the use of each sampling approach. Nonetheless, the selection of a field sampling technique must be technically justifiable pursuant to the systematic steps within Method 5035. The technical justification for the selection of a particular sampling technique must be provided to DTSC for our approval within appropriate workplans. A sampling technique should not be selected based upon the availability of sample containers and convenience of use. Instead, the sampling and preservation options must meet the scientific requirements of the data quality objectives.

4.0 APPLICATION

This Guidance Document describes the field sampling and preservation procedures for soil samples subject to VOC analysis. The applicable analytical methods described in SW-846 to be used in conjunction with Method 5035 are as follows:

Method 8015A:	Non-halogenated Organics
Method 8021B:	Aromatic and Halogenated Volatiles
Method 8260B:	Volatile Organic Compounds

Accordingly, all soil samples collected at sites regulated by DTSC that are analyzed using the above methods should also be handled pursuant to the Method 5035 procedures described in this Guidance Document.

5.0 SUMMARY OF METHOD

Method 5035 soil sample collection and preparation procedures are dependent on the desired detection limits needed for the project. For the low VOC concentration method, the available options are summarized in Table 1. For the high VOC concentration method, the available options are summarized in Table 2. The selection of a preservation option must be a function of the data quality objectives as outlined above in Section 3.0. Soil samples with VOC concentrations below 200 micrograms per kilogram ($\mu\text{g}/\text{kg}$) are generally considered as "Low Level Analysis" and have a method detection limit of approximately $0.5 \mu\text{g}/\text{kg}$. Soil samples with VOC concentrations above $200 \mu\text{g}/\text{kg}$ are generally considered as "High Level Analysis" and have a method detection limit of approximately $200 \mu\text{g}/\text{kg}$.

The procedures for a Low Level Analysis utilize a hermetically-sealed sampling container and analysis of the sample by a closed-system purge-and-trap process. The Low Level Analysis method uses a direct purging of the VOCs from an aqueous medium. The aqueous medium can be either sodium bisulfate solution or reagent water. The sodium bisulfate solution acts both as a preservative and extractant medium whereas reagent water is strictly an extractant medium with minimal preservation benefit. The aqueous medium is introduced into the sampling container either in the field or at the laboratory. No sample dilution is involved, yielding detection limits of approximately $0.5 \mu\text{g}/\text{kg}$.

The procedures for a High Level Analysis utilize a hermetically-sealed sampling container and analysis of the sample at the laboratory by Method 5030. The High Level Analysis method uses a

methanol solvent extraction technique. The methanol is introduced into the sampling container either in the field or at the laboratory. Detection limits of greater than 200 $\mu\text{g}/\text{kg}$ occur due to dilution of the sample with methanol.

When designing and implementing a sampling program for VOC contaminated soil, the project team must consider the appropriate analytical detection limits needed for the site characterization. Ultimately, the detection limits should be a function of the end-use of the data. For example, if the objective of the sampling is to quantify the human health risk to exposure to VOCs where the action levels are very low, then nothing less than Low Level Analysis is acceptable for the project. Conversely, if the objective is waste classification where the regulatory concentration thresholds are relatively high, then High Level Analysis is warranted. Another case where High Level Analysis is appropriate is the delineation of non-aqueous phase liquid (NAPL) in soil for remedial system design.

5.1 Number of Samples Needed for Analysis

In contrast with past soil sampling practices, Method 5035 now requires, if necessary, that multiple soil samples be collected from each sampling location. If needed, both Low Level Analysis and High Level Analysis sample sets are collected with proper preservation at each sampling point. The need for multiple samples is pertinent to sites with unknown VOC concentrations and for the need to have the lowest possible detection limits.

If detection limits of approximately 0.5 $\mu\text{g}/\text{kg}$ are needed for the soil at a site, three samples are collected pursuant to Method 5035. One sample is collected for High Level Analysis and two samples are collected for Low Level Analysis. First, the High Level Analysis sample is analyzed by the laboratory to determine if VOCs exist at the site in high concentrations. If this first sample yields VOC concentrations below the detection limit (<200 $\mu\text{g}/\text{kg}$), then a Low Level Analysis sample is analyzed. The second Low Level Analysis sample is available as a backup if the first Low Level Analysis run is unacceptable or re-analysis is warranted.

If detection limits of greater than 200 $\mu\text{g}/\text{kg}$ are acceptable at a site, then only one sample is collected for High Level Analysis pursuant to Method 5035. As necessary, the laboratory can perform multiple dilutions on the methanol extract to meet the instrument's calibration range. However, under this scenario, Low Level Analysis cannot be performed after the High Level Analysis due to the lack of available soil. To assist in the determination of the number of samples needed for Method 5035, a soil sampling decision flowchart is provided in Figure 1.

A general overview of the sampling options with Method 5035 is summarized below. A more detailed description of the options is provided within the Appendices³.

5.2 Option 1: Preserved VOA Vials (Field Chemical Preservation)

Tared and labeled VOA vials with polytetrafluoroethylene (PTFE)-lined septum caps are provided by the laboratory or a vendor with appropriate chemical preservatives. Typically, the VOA vials are

³ The Appendices were written as stand-alone documents which could be detached from this Guidance Document and taken into the field as a resource for Method 5035 sampling; hence, the Appendices reiterates numerous procedures presented within the text of this Guidance Document. Likewise, the individual Appendices are repetitious due to the numerous commonalities of the sampling procedures.

40 milliliters in size. The preservation fluid is either methanol or another water-miscible solvent such as polyethylene glycol⁴ (High Level Analysis), or sodium bisulfate solution⁵ (Low Level Analysis). Also, for Low Level Analysis, the VOA vials can contain reagent-grade extractant water⁶. Magnetic stir bars should be added to the VOA vials for Low Level Analysis pursuant to the laboratory's requirements. The selection of the preservation fluid is based on the chemistry of the target compounds and the type of soil, along with the desired method detection limits. In the field, the pre-preserved VOA vials for High Level Analysis are re-weighed before use to verify no evaporative loss of methanol since last tared. Re-weighing of the VOA vials before use for Low Level Analysis is not necessary because sodium bisulfate solution and reagent water have no affect on the dilution calculation. The soil subcores are obtained from appropriate sample locations using a field coring device. Then the soil subcores of appropriate mass are placed into the VOA vials in the field and are capped, forming an airtight seal.

To avoid VOC loss, Low Level Analysis samples preserved with sodium bisulfate solution or placed into reagent-grade extractant water are never again opened throughout the entire storage, preparation, and analysis process. Thus, the physical dimensions of the VOA vials must be compatible with the laboratory's autosampler instrumentation since sample re-handling is not possible. At the laboratory, the capped VOA vials are re-weighed to obtain the weight of the soil samples. For Low Level Analysis samples preserved with sodium bisulfate solution or placed into reagent-grade extractant water, the samples are prepared and analyzed with the caps in-place. All surrogates, internal standards, and matrix spikes are introduced through the PTFE-lined septum caps either manually or mechanically. For samples preserved with methanol, the VOA vials may be opened pursuant to the procedures of Method 5030 but only after the soil subcore is completely immersed in methanol and shaken gently to completely capture the VOCs in the headspace.

There are five options available for sample collection, preservation, and analysis for preserved VOA vials, as follows.

Option 1A: Field Preservation with Methanol. After collecting the soil samples in tared VOA vials preserved with methanol, the vials are re-weighed in the field, and then are chilled at $4 \pm 2^\circ\text{C}$ in a cooler and shipped with adequate ice to ensure that $4 \pm 2^\circ\text{C}$ is maintained during transport to the laboratory. The samples must arrive at the laboratory within 48 hours of the sample collection time. The VOA vials are weighed again at the stationary laboratory to verify no methanol loss during transport. The laboratory must prepare and analyze the samples by Method 5030 within 14 days of the sample collection date. This technique applies only to High Level Analysis so it should be used if detection limits of greater than $200 \mu\text{g}/\text{kg}$ are warranted.

Option 1B: Field Preservation with Sodium Bisulfate Solution. After collecting the soil samples in tared VOA vials preserved with sodium bisulfate solution, the samples are kept chilled at $4 \pm 2^\circ\text{C}$ in a cooler and shipped with adequate ice to ensure that $4 \pm 2^\circ\text{C}$ is maintained during transport to the laboratory. The samples must arrive at the laboratory within 48 hours of the sample collection

⁴ Ten milliliters of methanol or another water-miscible solvent is added to each VOA vial.

⁵ A twenty percent sodium bisulfate solution is generally used for preservation. The solution is usually produced by adding one gram of sodium bisulfate to five grams of reagent water, thus producing a solution with a pH of less than two.

⁶ Five milliliters of reagent-grade extractant water is added to each VOA vial.

time. The laboratory must prepare and analyze the samples within 14 days of the sample collection date. This preservation technique provides detection limits to approximately 0.5 µg/kg (Low Level Analysis). However, sample preservation with sodium bisulfate solution presents four potential problems. One, acid preservation may cause the chemical breakdown of certain reactive VOC compounds in the soil sample, specifically styrene, acrylonitrile, vinyl chloride, and 2-chloroethylvinyl ether. Two, in soil samples with a high proportion of organic material, acid preservation may generate acetone as a byproduct. Three, calcareous soil samples may effervesce upon contact with sodium bisulfate solution and cause VOC loss. Four, calcareous soil samples may increase the pH of the preservation fluid above 2.0, producing a sample in an unpreserved state. Accordingly, the soils at the site should be evaluated for potential problems prior to sampling activities. In cases where preservation by acid is a potential problem, an alternate sample collection method should be utilized.

Option 1C: Field Extraction into Reagent Water (Laboratory Freezing). After collecting the soil samples in tared VOA vials containing reagent-grade extractant water, the samples are kept chilled at $4 \pm 2^\circ\text{C}$ in a cooler and shipped with adequate ice to ensure that $4 \pm 2^\circ\text{C}$ is maintained during transport to the laboratory. The laboratory must receive and immediately freeze the sample vials to $<-7^\circ\text{C}$ within 48 hours of the sample collection time. During the freezing process, the VOA vials should be stored in a 45° angle to prevent water expansion from shattering the vials. The samples may be held at $<-7^\circ\text{C}$ for up to seven days prior to analysis from the sample collection date. The sample vials should not be frozen below -20°C due to potential problems with the vial seals. This technique applies to samples for Low and High Level Analysis.

Option 1D: Field Extraction into Reagent Water (Field Freezing). After collecting the soil samples in tared VOA vials containing reagent-grade extractant water, the samples are frozen to $<-7^\circ\text{C}$ in a cooler in the field and shipped with adequate dry ice⁷ to ensure that $<-7^\circ\text{C}$ is maintained during transport to the laboratory. The sample vials should not be frozen below -20°C due to potential problems with the vial seals. A temperature blank should be included with the samples so that the laboratory can verify the temperature upon receipt and the arrival temperature of the samples should be annotated on the chain-of-custody form. During the freezing process, the VOA vials should be stored in a 45° angle to prevent water expansion from shattering the vials. To avoid potential rupture of the PTFE-lined septum caps, the dry ice should not directly contact the top of the VOA vials. The laboratory must immediately freeze the sample vials to $<-7^\circ\text{C}$ upon receipt. The samples may be held at $<-7^\circ\text{C}$ for up to seven days prior to analysis from the sample collection date. This technique applies to samples for Low and High Level Analysis. This option is used in the situations where it is difficult or impossible to deliver the samples to the laboratory within 48 hours of the sample collection time.

Option 1E: Field Extraction into Reagent Water; Analysis within 48 Hours. After collecting the soil samples in tared VOA vials containing reagent-grade extractant water, the samples are kept chilled at $4 \pm 2^\circ\text{C}$ in a cooler and shipped with adequate ice to ensure that $4 \pm 2^\circ\text{C}$ is maintained during transport to the laboratory. Upon receipt of the samples, the laboratory chills the tared VOA vials to $4 \pm 2^\circ\text{C}$ and analyzes the samples within 48 hours of the sample collection time. This technique applies to samples for Low and High Level Analysis.

⁷ There two potential difficulties in using dry ice to achieve $<-7^\circ\text{C}$ in the field; 1) dry ice will only last about eight hours within a field cooler, and 2) dry ice may contain low concentrations of VOCs, such as acetone. Hence, care must be taken in overnight shipment of field coolers to insure proper freezing and trip blanks should always accompany coolers containing dry ice.

It should be noted that extruding soil samples into vials containing reagent-grade extractant water may have an adverse effect on sample results in that water may actually promote bacterial degradation of certain VOCs (See Section 6.3.1). Likewise, some VOCs may be unstable in reagent water, such as 1,1,2,2-tetrachloroethane. Accordingly, reagent water-filled VOA vials should only be used for chemicals that do not readily biodegrade or breakdown.

The field procedures for Options 1A, 1B, 1C, 1D and 1E are furthered discussed in Appendix A.

5.3 Option 2: Multi-Functional Sampling Devices (No Field Chemical Preservation)

Multi-functional sampling devices (MFSDs) act as both a coring tool and airtight storage container. Examples of MFSDs are the EnCore™ Sampler and the Core N' One™ Sampler⁸. In MFSDs, a small subcore of soil is collected directly into the volumetric storage chamber of the MFSD from a soil core or soil surface, filling it completely with zero headspace. The storage chamber is then capped to form an airtight seal. The intact MFSDs are placed into a plastic bag for transport to the laboratory at $4 \pm 2^\circ\text{C}$. At the stationary laboratory, the soil content of the MFSD is extruded into a prepared VOA vial for analysis. The opening of the VOA vial must be sufficiently large to accept the soil content from the MFSD without obstruction. Since the VOA vial may be used directly for analysis, it must be compatible with the stationary laboratory's purge and trap apparatus to avoid further sample handling which might promote VOC loss. Field personnel should contact the laboratory for the required dimensions.

There are three options available for sample collection, preservation, and analysis for MFSDs, as follows.

Option 2A: The Subcore is Extruded into a VOA Vial Containing Chemical Preservative at the Laboratory. The field cooler is kept chilled at $4 \pm 2^\circ\text{C}$ and shipped with adequate ice to ensure that $4 \pm 2^\circ\text{C}$ is maintained during transport to the laboratory. The laboratory must receive the MFSDs and extrude the samples into VOA vials that contain appropriate extraction fluid within 48 hours of the sample collection time. For MFSDs for Low Level Analysis, the soil can be extruded, weighed, and preserved with sodium bisulfate solution. Also, for Low Level Analysis, the soil can be extruded into reagent-grade extractant water. For MFSDs for High Level Analysis, the soil must be extruded, weighed, and preserved with methanol. After extrusion of the soil into an appropriate extraction fluid, the sample may be held up to 14 days prior to analysis from the sample collection date.

Option 2B: The Subcore is Extruded into an Empty VOA Vial at the Laboratory. The field cooler is kept chilled at $4 \pm 2^\circ\text{C}$ and shipped with adequate ice to ensure that $4 \pm 2^\circ\text{C}$ is maintained during transport to the laboratory. The laboratory must receive the MFSDs and extrude the samples within 48 hours of the sample collection time. Upon receipt of the samples, the laboratory extrudes the subcores into empty VOA vials and then freezes the unpreserved VOA vials at $<-7^\circ\text{C}$. The samples may be held at $<-7^\circ\text{C}$ for up to seven days⁹ prior to analysis from the sample collection

⁸ The mention of trade names or commercial products in this Guidance Document is for illustrative purposes only, and does not constitute an endorsement or exclusive recommendation for use at DTSC sites. Equipment other than that listed may be used provided that the resulting performance meets the project data quality objectives.

⁹ The holding time of seven days from the sample collection date is consistent with guidance from the Los Angeles Regional Water Quality Control Board (General Laboratory Testing Requirements for Petroleum Hydrocarbon Impacted Sites, June 5, 2000)

date. For Low Level Analysis, the samples are prepared and analyzed with the VOA vial caps in place. For High Level Analysis, the samples are handled pursuant to Method 5030.

Option 2C: The MFSD is Analyzed within 48 Hours. The field cooler is kept chilled at $4 \pm 2^{\circ}\text{C}$ and shipped with adequate ice to ensure that $4 \pm 2^{\circ}\text{C}$ is maintained during transport to the laboratory. Upon receipt of the samples, the laboratory chills the MFSDs to $4 \pm 2^{\circ}\text{C}$ until analysis. The laboratory must extrude and analyze the samples within 48 hours of the sample collection time. The samples may be subject to either High or Low Level Analysis.

The field procedures for Options 2A, 2B, and 2C are furthered discussed in Appendix B.

5.4 Option 3: Non-Preserved VOA Vials (Empty Vial Technique)

Empty, tared and labeled VOA vials with a PTFE-lined septum caps are taken into the field as provided by the laboratory. Likewise, these vials may be purchased as specially prepared vials from scientific suppliers or can be prepared by the field staff using empty VOA vials, certified clean to USEPA specifications. The VOA vials do not contain chemical preservatives, water-miscible solvents, or reagent water but may contain small magnetic stir bars as required by the laboratory. Soil cores of appropriate mass are placed into the VOA vials in the field and are capped, forming an airtight seal. For Low Level Analysis, the VOA vials are never again opened throughout the entire storage, preparation, and analysis process. Thus, the physical dimensions of the VOA vials must be compatible with the laboratory's autosampler instrumentation since sample re-handling is not possible. At the laboratory, the capped VOA vials are re-weighed to obtain the weight of the soil samples. For Low Level Analysis, the samples are prepared and analyzed with the caps in place. All preservatives, surrogates, internal standards, and matrix spikes are introduced through the PTFE-lined septum caps either manually or mechanically and analyzed with a closed-system purge-and-trap process. For High Level Analysis, methanol is introduced through the septum and the resulting extract is analyzed with Method 5030.

There are three options available for sample collection, preservation, and analysis for non-preserved VOA vials, as follows.

Option 3A: Laboratory Freezing. After collecting the soil samples in tared VOA vials, the samples are kept chilled at $4 \pm 2^{\circ}\text{C}$ in a cooler and shipped with adequate ice to ensure that $4 \pm 2^{\circ}\text{C}$ is maintained during transport to the laboratory. The laboratory must receive the samples within 48 hours of the sample collection time and immediately freeze the sample vials to $<-7^{\circ}\text{C}$ upon receipt. The samples may be held at $<-7^{\circ}\text{C}$ for up to seven days prior to analysis from the sample collection date. The sample vials should not be frozen below -20°C due to potential problems with the vial seals and the samples may be subject to either High or Low Level Analysis.

Option 3B: Field Freezing. After collecting the soil samples in tared VOA vials, the samples are frozen to $<-7^{\circ}\text{C}$ in a cooler in the field and shipped with adequate dry ice¹⁰ to ensure that $<-7^{\circ}\text{C}$ is maintained during transport to the laboratory. The sample vials should not be frozen below -20°C due to potential problems with the vial seals. A temperature blank should be included with the samples so that the laboratory can verify the temperature upon receipt and the arrival temperature

¹⁰ There two potential difficulties in using dry ice to achieve $<-7^{\circ}\text{C}$ in the field; 1) dry ice will only last about eight hours within a field cooler, and 2) dry ice may contain low concentrations of VOCs, such as acetone. Hence, care must be taken in overnight shipment of field coolers to insure proper freezing and trip blanks should always accompany coolers containing dry ice.

of the samples should be annotated on the chain-of-custody form. During the freezing process, the VOA vials should be stored in a 45° angle to prevent sample expansion from shattering the vials. To avoid potential rupture of the PTFE-lined septum caps, the dry ice should not directly contact the top of the VOA vials. Upon receipt, the laboratory must commence with analysis. Otherwise, the laboratory must immediately freeze the sample vials to <-7°C upon receipt. The samples may be held at <-7°C for up to seven days prior to analysis from the sample collection date. The samples may be subject to either High or Low Level Analysis. This option is used in the situations where it is difficult or impossible to deliver the samples to the laboratory within 48 hours of the sample collection time.

Option 3C: Analysis within 48 Hours. After collecting the soil samples in tared VOA vials, the samples are kept chilled at $4 \pm 2^{\circ}\text{C}$ in a cooler and shipped with adequate ice to ensure that $4 \pm 2^{\circ}\text{C}$ is maintained during transport to the laboratory. Upon receipt of the samples, the laboratory chills the tared VOA vials to $4 \pm 2^{\circ}\text{C}$ and analyzes the samples within 48 hours of the sample collection time. The samples may be subject to either High or Low Level Analysis.

The field procedures for Options 3A, 3B, and 3C are furthered discussed in Appendix C.

6.0 CONDITIONS OF THE METHOD

6.1 Limitation of Methanol Preservation

The preservation and extraction of samples with methanol is not appropriate for soils with low VOC concentrations. The use of methanol as a preservative introduces a significant dilution factor that will raise the method detection limit beyond the operating range of the Low Level Analysis procedure. For gas chromatography, depending on the analytical method, methanol may also mask the elution of some VOCs. Accordingly, the potential for coelution should be discussed with the laboratory prior to sample collection. Potentially, these limitations could render the soil analytical results useless in evaluating sites for risk assessment purposes.

6.2 Subcoring Devices

With most standard drilling techniques, soil cores are retrieved from the subsurface during site characterization with a core barrel. When analyzing soil samples pursuant to Method 5035, the soil from the core barrels must be subcored and then these subcore samples must be placed into airtight containers. With Option 2, the MFSD acts as both a subcoring tool and airtight storage container. The MFSD is designed to collect, transport, and deliver intact soil sample subcores to the stationary laboratory. The coring body of the MFSD is pushed into a freshly exposed soil surface, obtaining a headspace-free subcore. The sample chamber is then sealed with the cap, becoming airtight. Once back at the laboratory, the sample subcore is extruded into a tared empty or preserved VOA vial, as appropriate. To aid in the extrusion of the subcore from the MFSD into the VOA vial, the opening of the VOA vial must be larger than the diameter of the subcore. Accordingly, the project planning team must contact the stationary laboratory to ensure that the MFSDs are compatible with the VOA vials used in the laboratory's autosampler instrumentation to avoid further sample handling which might promote VOC loss.

For Options 1 and 3, a subcoring device must be used to obtain the subcore soil samples. The subcoring device must have a diameter smaller than the opening of the VOA vial into which the subcore is extruded. Additionally, the project planning team must contact the stationary laboratory to ensure that the VOA vials used in the field are compatible with the laboratory's autosampler

instrumentation. The subcoring devices are used to obtain either five or ten grams of soil, as appropriate. Five grams is the preferred weight to minimize sample handling by the laboratory. Numerous subcoring devices are available for the collection of the soil subcores. The following list contains a description of three subcoring devices. However, any equivalent device may also be used.

- Disposable Plastic Syringe. A disposable plastic syringe can be easily converted to an inexpensive subcoring device. The "needle end" of the syringe barrel can be cut off, thus creating a blunt, even coring end. The end of the syringe should be cut with a knife or scissors rather than a saw so that the blunt end is smooth to prevent soil disaggregation upon collection. Prior to use, the plunger of the syringe must be in the "down position" so that its end directly contacts the soil, not allowing for any trapped air. The soil subcore is collected by pushing the cut end of the syringe into the freshly exposed soil surface until the soil column fills the inside of the syringe with five grams of soil, or as needed. The soil subcore is then removed from the syringe and extruded into the VOA vial using the syringe's plunger.
- EasyDraw Syringe™ and PowerStop Handle™. The soil subcore is obtained with the sampling device and transferred into a VOA vial in the field. The PowerStop Handle™ is reusable but a new syringe must be used for each sampling location. There are three 5 gram positions and three 10 gram positions on the PowerStop Handle™. The three positions are labeled light, medium, and heavy to correspond to low, medium and high soil densities. There is also one 13 gram position. In general, one of the 5 gram positions will be used to collect the soil subcores. The soil subcore is collected by pushing the EasyDraw Syringe™ into the freshly exposed soil surface until the soil column inside the syringe has forced the plunger to the stopping point.
- Lock N' Load™ Soil Sampling Tool. The soil subcore is obtained with the sampling device and transferred into a VOA vial in the field. There are two settings, a 5 gram position and a 10 gram position, on the Lock N' Load™ Soil Sampling Tool. In general, the 5 gram position will be used to collect the soil subcores. The Lock N' Load™ Soil Sampling Tool is reusable but a new syringe must be used for each sampling location. The Lock N' Load syringe fits securely into the neck of a 40 milliliter glass vial and by turning the Lock N' Load handle, one can dispense the soil into a VOA vial without removing the syringe.

Section 6.10 provides guidance on the collection of samples from consolidated soil, such as cemented soil, dense sand, stiff clay, or bedrock, where subcores cannot be obtained with a MFSD, disposable plastic syringe, EasyDraw Syringe™, Lock N' Load™ Soil Sampling Tool, or other appropriate subcoring device.

6.3 Procedural Incompatibilities

6.3.1 Aromatic Hydrocarbons

Chemicals, such as aromatic hydrocarbons (AH), are subject to VOC loss by biodegradation under certain Method 5035 sampling procedures. Accordingly, to obtain AH soil concentrations that are representative of site conditions, only a subset of the available Method 5035 options are available for use. To reduce the biological activity in soil contaminated with AH, soil samples should be preserved with methanol or sodium bisulfate solution in the field, collected with MFSDs, or frozen in the field at $<-7^{\circ}\text{C}$ in non-preserved VOA vials. Under no circumstances should soil samples contaminated with AH be collected in the field with VOA vials containing reagent-grade extractant

water. The introduction of unpreserved water to the soil sample may enhance the biodegradation of the AH.

6.3.2 Chemical Reactions

Acid preservation of soil by sodium bisulfate solution, whether done in the field or in the stationary laboratory, may cause the chemical breakdown of certain compounds. Some olefins, ketones, esters, ethers, and sulfides may react under low pH conditions, yielding analytical results that are not representative of soil conditions. Hence, precaution should be taken when preserving soil samples with sodium bisulfate solution when these compounds are present. If the degree of potential chemical reaction is unknown, an alternative Method 5035 procedure should be used.

6.3.3 Calcareous Soil

Calcareous soil samples may react upon contact with sodium bisulfate solution, causing VOC loss through effervescence and potentially cause failure of the VOA vial septum through pressure build-up. Additionally, when soil samples are highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the aqueous solution to below 2.0, potentially rendering the preservative useless. If carbon dioxide is generated due to carbonate reaction with the acid, the carbon dioxide in the VOA vial may interfere with the detector of the analytical equipment. Hence, precaution should be taken when preserving soil samples with sodium bisulfate solution when carbonates are present.

6.4 Selection of Appropriate Sampling Procedures

The selection of a Method 5035 sampling technique for a site should not be based upon on the availability of sample containers and convenience of use. Instead, the sampling and preservation options should be selected based upon the requirements of the data quality objectives for the project. Accordingly, this hierarchy of techniques is offered as a guide for users when evaluating the data quality needs for a project.

- 1) Option 1: Field Chemical Preservation. Chemical preservation of VOA vials in the field with sodium bisulfate solution (Low Level Analysis) or methanol (High Level Analysis) yields the best possible data quality for VOC analysis of soil. The introduction of these chemical preservatives in the field inhibits VOC loss by biodegradation. Also, VOC loss due to sample handling is minimized.
- 2) Option 2: Multi-Functional Sampling Devices. When the MFSDs are received by the stationary laboratory, the soil subcores within the MFSDs are extruded into VOA vials for analysis. As the soil subcores pass from the MFSDs to the VOA vials during the extrusion process, the soil subcores are open to ambient air and VOC loss could occur. This VOC loss could yield analytical results that are potentially biased low. Users of MFSDs must recognize this limitation when evaluating the data quality objectives for their project.
- 3) Option 3: Empty Vial Technique. The extractant fluid, whether methanol, sodium bisulfate solution, or reagent water, must be added by the stationary laboratory to the VOA vials after the soil has been sealed into the vials in the field. To do this, the PTFE-lined septum caps must be pierced for the introduction of the extraction fluid into the VOA vials. After the introduction of the extraction fluid, the vials must be stirred or sonicated to promote the partitioning of the VOCs into the extraction fluid. Upon completion of the stirring or sonication, the sample is then

analyzed for VOC concentration. During the stirring or sonication, VOCs can escape from the VOA vial through the pierced septum. Hence, the Empty Vial Technique may potentially yield analytical results that are biased low. Users of the Empty Vial Technique must recognize this limitation when evaluating the data quality objectives for their project.

Thus, the sampling options within Method 5035 do not potentially yield similar data quality results. Accordingly, for sites that require the highest quality analytical results, the soil subcores should be field preserved with methanol or sodium bisulfate solution.

For sites where the contaminants may react with the sodium bisulfate solution or where the soils may react with the sodium bisulfate solution due to high carbonate content, water may be substituted for the sodium bisulfate solution in the field in cases where chemical biodegradation is not a concern.

6.5 Field Screening

Not all soil samples collected from a borehole during site characterization are submitted to a stationary laboratory for analysis. Usually, the soil samples exhibiting the highest concentrations of VOCs through field screening techniques are submitted for analysis. Accordingly, to comply with the intent of Method 5035, care must be taken in the field to minimize VOC loss during the field screening process. Typically, during split-spoon sampling, cores are obtained within brass sleeves and one brass sleeve is field screened for VOC concentration. Meanwhile, another brass sleeve from the same depth interval is placed on ice in a sample cooler for possible laboratory analysis. Upon completion of the drilling, the soil samples for laboratory analysis are then selected. To comply with Method 5035, DTSC allows this style of procedure to continue but with minor modification. Brass sleeves can be held in a cooler filled with ice onsite awaiting subcoring for Method 5035 upon completion of borehole drilling if the following conditions are met:

- 1) The ends of the brass sleeve are covered with teflon sheeting, capped with tight-fitting plastic end-caps, and then placed into a resealable plastic (Ziploc™ type) bag.
- 2) No headspace exists within the brass sleeve.
- 3) The resealable plastic bag containing the brass sleeve is placed directly on ice within the shipping cooler.
- 4) No more than two hours transpire between core retrieval from the subsurface and the collection of the Method 5035 subcores from the brass sleeve.
- 5) The field log or boring log must reflect the time of core retrieval and the time of subcoring.

If the conditions listed-above are met, the brass sleeves can be held and subcored upon completion of the drilling of the borehole or within two hours of core retrieval, whichever is less. For Method 5035 sampling upon completion of borehole drilling, the brass sleeve is uncapped and the first inch of soil is removed from the brass sleeve with an appropriate instrument. The subcoring then takes place on the newly exposed surface as quickly as possible and as deep as possible within the brass sleeve. However, if the above conditions are not achieved in the field, all brass sleeves that might be subject to VOC analysis must be subcored immediately pursuant to Method 5035.

In some situations, acetate-lined core barrels are used rather than brass sleeves during the collection of subsurface soil samples. The above-mentioned approach applies to acetate-lined core samples where the cores would be manually sliced with a knife for field screening and subcoring.

Under no circumstances should brass sleeves or acetate-lined cores be submitted to a stationary laboratory for Method 5035 analysis. However, brass sleeves and acetate-lined cores can be hand-carried to an onsite mobile laboratory pursuant to the conditions referenced in Section 6.9 in this Guidance Document.

6.6 Dry Weight Determination

If the soil analytical results for a project must be reported on a dry weight basis, an additional soil sample must be collected from the sampling location in order to determine the dry weight of the soil. The soil sample submitted to the stationary laboratory specifically for dry weight determination does not need chemical preservation in the field and may be collected by conventional methods, such as in glass jars, brass sleeves, or acetate liners. However, the collection method should minimize sample handling, sample disaggregation, and moisture loss. As such, the sample containers used for the collection of these samples should have appropriate seals to prevent moisture loss and be clearly labeled to avoid confusion at the laboratory. Also, this sample for dry weight determination can be used by the laboratory to evaluate soil reactivity to sodium bisulfate solution.

6.7 Quality Assurance / Quality Control Samples

6.7.1 Trip Blanks

Soil samples can be contaminated by diffusion of VOCs through the septum on VOA vials or through the seal on MFSDs during shipment and storage. A trip blank prepared with laboratory-grade methanol, sodium bisulfate solution, or reagent water, dependent on the field methods, can be carried through sampling and handling protocols as a check on such contamination. DTSC recommends that, ideally, one trip blank should be used for each field sample cooler, but, at a minimum, one trip blank should be used per day.

6.7.2 Temperature Blanks

Temperature blanks should be used so that the laboratory can verify the temperature upon receipt of the samples. In the case of field freezing, the temperature blanks should be frozen upon arrival at the laboratory. The temperature of the samples upon arrival should be annotated on the chain-of-custody form and also mentioned in the laboratory narrative that accompanies the analytical results.

6.7.3 Matrix Spike and Matrix Spike Duplicate Samples

An important measure of the performance of an analytical method relative to the specific sample matrix of interest is the matrix spike and matrix spike duplicate (MS/MSD). The MS/MSD is an important aspect of an overall quality assurance program for a project. When soil sampling, a MS/MSD sample should be collected for each analytical method at a frequency of five percent of the field samples. The MS/MSD sample should be prepared in a fashion similar to the other samples pursuant to Method 5035. Samples taken for MS/MSD should be labeled as such and

specified on the chain-of-custody form. The primary purpose of MS/MSD analyses is to establish the applicability of the overall analytical approach to the specific sample matrix from the site.

6.7.4 Other Field Quality Control Samples

Field quality control samples to demonstrate the integrity of the field samples should also be collected. Field duplicates, field blanks, and equipment rinsate blanks should be collected at a frequency of five percent of the samples, or, at a minimum, one should be collected each day.

6.8 Bulk Soil Sampling

The collection of soil samples in bulk containers that would require laboratory subcoring is *not* an option pursuant to Method 5035. Large bottles, wide-mouthed jars, acetate liners, or brass sleeves from split-spoon samplers are not appropriate sample containers under Method 5035 for VOC analysis. However, with DTSC prior approval, these traditional, non-Method 5035 approaches can be used at sites for characterization purposes only, but VOC concentrations from these soil samples should never be used in fate and transport modeling or to quantify the risk associated with human and ecological exposure.

6.9 Mobile Laboratories

VOC analysis may be performed by a certified mobile field laboratory as long as their procedures and analytical equipment meet the performance standards of Method 5035. Tables 1 and 2 summarize the options available for sample preservation for both Low Level Analysis and High Level Analysis, respectively. Obviously, sample preservation for long holding times is not warranted with use of an onsite mobile laboratory. Accordingly, DTSC anticipates that soil samples for analysis by a mobile laboratory will be collected in a non-preserved manner. There are two options available for mobile laboratories for the analysis of soil samples pursuant to Method 5035, as follows:

- 1) Laboratory Subcoring. After the acquisition of soil cores from the subsurface, which are usually obtained within brass sleeves, the field geologist or technician covers the ends of one of the sleeves with teflon sheeting, caps the ends with tight-fitting plastic end-caps, and then places the sleeve into a resealable plastic bag. No headspace should exist within the brass sleeve. The brass sleeve is quickly brought to the mobile laboratory where the chemist can either carefully perform the subcoring of the brass sleeve and then immediately analyze the sample or the brass sleeve is placed into the mobile laboratory's freezer for later subcoring. A brass sleeve placed into the laboratory's freezer should only be held for two hours prior to analysis. Otherwise, the sample should be preserved pursuant to Method 5035 and then analyzed later as appropriate.

At the mobile laboratory, the chemist performing the subcoring of the soil from the brass sleeve has two options for sample preparation and analysis. The soil subcore can be placed into either a VOA vial or a test tube for preparation and analysis. Prior to sample collection, the mobile laboratory chemist prepares pre-tared test tubes or VOA vials, with magnetic stir bars as needed. A subcoring device, such as a disposable plastic syringe, is used to remove a five gram sample plug from a newly exposed surface of the soil core. The barrel diameter of the disposable plastic syringe should be smaller than that of the test tube or VOA vial. The sample plug is immediately transferred to the test tube or VOA vial, which is then hermetically sealed. The test tube or VOA vial is then weighed to obtain the actual sample weight and the

test tube or VOA vial is loaded immediately on the closed system purge-and-trap for analysis. The time between removal of the sample plug from the soil core and the sealing of the test tube or VOA vial should be no more than two minutes. All surrogates, internal standards, and matrix spikes are introduced either through the PTFE-lined septum cap of the VOA vial or through the sampling valve on the test tube cap.

- 2) **Field Subcoring.** Pre-tared, labeled VOA vials with PTFE-lined septum caps are taken into the field. The analytical instrumentation of the mobile laboratory should be capable of mechanically accepting the VOA vials. Magnetic stir bars are added to the VOA vials as necessary. Once a soil core is available from the drilling or sampling activities, a subcoring device, such as plastic syringe, is used to remove a five gram sample plug from a fresh surface of the soil core. The plastic syringe should be disposable with a barrel diameter that is smaller than the diameter of the VOA vial. Each sample subcore is immediately transferred to the VOA vial, which is then hermetically sealed. The time between removal of the soil core from the subsurface and hermetically sealing the VOA vial should be no more than two minutes. The sample is quickly brought to the mobile laboratory where the chemist immediately analyzes the sample. If the sample cannot be analyzed immediately, the sample is placed into the mobile laboratory's freezer for later analysis. The sample in the laboratory's freezer should only be held for two hours prior to analysis, otherwise the sample should be preserved pursuant to Method 5035 and then analyzed later as appropriate, either at the mobile laboratory or at a stationary laboratory.

The onsite mobile laboratory should process the samples immediately upon receipt. The chain-of-custody form should be checked and signed, the samples logged-in, and the samples should then be weighed as appropriate. For Low Level Analysis samples, the samples are prepared and analyzed with the caps in-place. All surrogates, internal standards, and matrix spikes are introduced through the septum, either manually or mechanically. For High Level Analysis, the VOA vials may be opened but only after the soil subcore is completely immersed in methanol, as introduced through the septum, and shaken gently to completely capture the VOCs in the headspace. All samples, while in the custody of either the field investigator or the mobile laboratory, should be chilled to $4 \pm 2^{\circ}\text{C}$.

6.10 Sampling of Consolidated Soil

Some materials that require sampling may be too cohesive for subcoring tools to penetrate. Examples of such materials include cemented soil, dense sand, stiff clay, or bedrock. Samples of these materials can be collected by exposing a fresh surface and using an appropriate tool such as a clean chisel or spatula to generate aggregates of a size that can be placed into a VOA vial. When transferring the aggregates, care must be taken to prevent compromise of the sealing surfaces and threads of the VOA vials. The VOA vial should be handled and preserved pursuant to the data quality objectives for the site. When sampling under these conditions, field personnel should note the occurrence in their field logs. Although the inevitable disaggregation of the sample increases the possibility of VOC losses, there may be no alternative under these conditions. Therefore, caution should be used in the interpretation of the data obtained from this type of material.

**TABLE 1: METHOD 5035 LOW LEVEL ANALYSIS
Low Concentrations of VOCs Are Anticipated in the Soil Samples
Sample Detection Limits are Approximately 0.5 µg/kg**

Option	Sample Container	Field Preservation	Laboratory Activity	Holding Time ²
1B	VOA Vial ^{1,3}	Sodium bisulfate solution and cool to 4 ± 2°C	Cool to 4 ± 2°C until analysis	14 days
1C	VOA Vial ^{1,5}	Water and cool to 4 ± 2°C	Freeze to <-7°C within 48 hours from sample collection	7 days
1D	VOA Vial ^{1,5}	Water and freeze to <-7°C ⁴	Freeze to <-7°C	7 days
1E	VOA Vial ^{1,5}	Water and cool to 4 ± 2°C	Cool to 4 ± 2°C until analysis	48 hours
2A	Multi-Functional Sampling Device ³	Cool to 4 ± 2°C	Extrude into sodium bisulfate solution within 48 hours of sample collection and cool to 4± 2°C	14 days
2B	Multi-Functional Sampling Device	Cool to 4 ± 2°C	Extrude into VOA vial within 48 hours of sample collection and freeze to <-7°C	7 days
2C	Multi-Functional Sampling Device ³	Cool to 4 ± 2°C	Cool to 4 ± 2°C until analysis	48 hours
3A	VOA Vial ¹	Cool to 4 ± 2°C	Freeze to <-7°C within 48 hours from sample collection	7 days
3B	VOA Vial ¹	Freeze to <-7°C ⁴	Freeze to <-7°C	7 days
3C	VOA Vial ^{1,3}	Cool to 4 ± 2°C	Cool to 4 ± 2°C until analysis	48 hours

¹ VOA vials are never opened after being sealed in the field.

² Holding time is measured from the time of sample collection.

³ Preferred method for aromatic hydrocarbons due to potential biodegradation.

⁴ Field freezing is needed when the samples cannot be transported to the stationary laboratory within 48 hours of the sampling time.

⁵ Water should be used as a replacement for sodium bisulfate solution when soils and contaminants are incompatible with low pH conditions.

TABLE 2: METHOD 5035 HIGH LEVEL ANALYSIS
High Concentrations of VOCs are Anticipated in the Soil Samples
Sample Detection Limits are Approximately 200 µg/kg

Option	Sample Container	Field Preservation	Laboratory Activity	Holding Time ²
1A	VOA Vial ^{1,3}	Methanol and cool to 4 ± 2°C	Cool to 4°C until analysis	14 days
1C	VOA Vial ¹	Water and cool to 4 ± 2°C	Freeze to <-7°C within 48 hours from sample collection	7 days
1D	VOA Vial ¹	Water and freeze to <-7°C ⁴	Freeze to <-7°C	7 days
1E	VOA Vial ¹	Water and cool to 4 ± 2°C	Cool to 4 ± 2°C until analysis	48 hours
2A	Multi-Functional Sampling Device ³	Cool to 4 ± 2°C	Extrude into methanol within 48 hours of sample collection and cool to 4 ± 2°C	14 days
2B	Multi-Functional Sampling Device	Cool to 4 ± 2°C	Extrude into VOA vial within 48 hours of sample collection and freeze to <-7°C	7 days
2C	Multi-Functional Sampling Device ³	Cool to 4 ± 2°C	Cool to 4 ± 2°C until analysis	48 hours
3A	VOA Vial ¹	Cool to 4 ± 2°C	Freeze to <-7°C within 48 hours of sample collection	7 days
3B	VOA Vial ¹	Freeze to <-7°C ⁴	Freeze to <-7°C	7 days
3C	VOA Vial ^{1,3}	Cool to 4 ± 2°C	Cool to 4 ± 2°C until analysis	48 hours

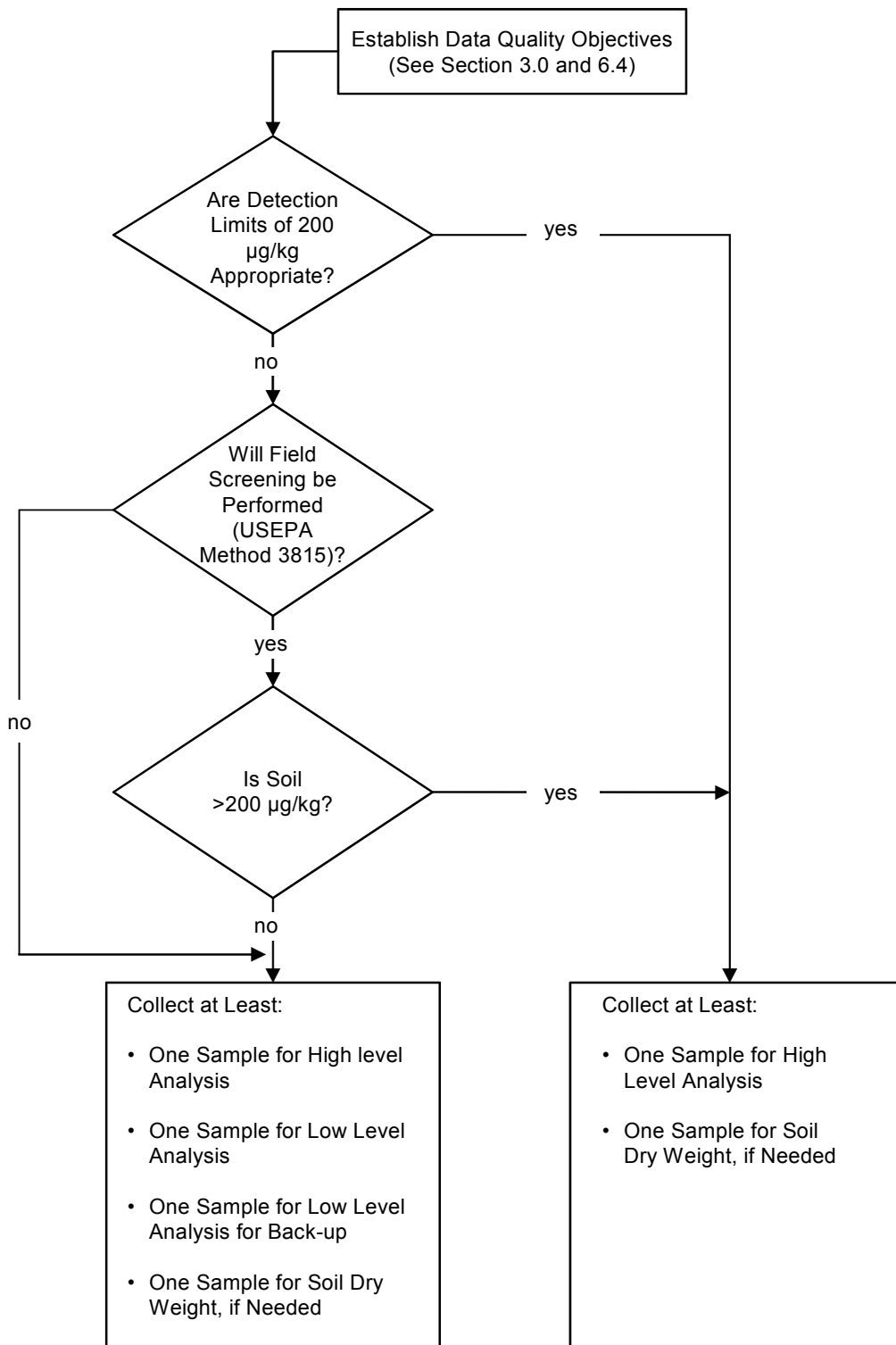
¹ VOA vials are never opened after being sealed in the field.

² Holding time is measured from the time of sample collection.

³ Preferred method for aromatic hydrocarbons due to potential biodegradation.

⁴ Field freezing is needed when the samples cannot be transported to the stationary laboratory within 48 hours of the sampling time.

FIGURE 3: SOIL SAMPLING DECISION MATRIX
Selection of the Number of Soil Samples at a Sample Location Point



Note: The options for Low Level Analysis and High Level Analysis are shown in Tables 1 and 2.

Appendix A: Sampling Option 1

Sampling Options 1A, 1B, 1C, 1D, and 1E Preserved VOA Vials

The stationary laboratory that will perform the soil analysis will provide preserved, tared, and labeled VOA vials that have PTFE-lined septum caps. Alternatively, VOA vials can be purchased from scientific suppliers that are certified clean to USEPA specifications. Typically, the VOA vials are 40 milliliters in size. The preservation fluid is either methanol or sodium bisulfate solution. Also, the VOA vials may contain reagent-grade extraction water. The methanol, sodium bisulfate solution, and reagent water must be laboratory-grade fluids. The fluids must be purge-and-trap grade, certified to be free of VOCs. The selection of the preservation fluid is based upon the desired method detection limits (Low Level Analysis or High Level Analysis) and the data quality objectives. In the field, the methanol-preserved VOA vials are re-weighed to verify no preservative loss due to volatilization. Re-weighing the VOA vials containing reagent water or sodium bisulfate solution is not necessary because these fluids have no affect on the dilution calculation. However, magnetic stir bars may be needed in VOA vials containing reagent water or sodium bisulfate solution pursuant to the laboratory's requirements. Soil subcores of appropriate mass are placed into the VOA vials in the field and are capped, forming an airtight seal. The soil samples preserved with methanol are re-weighed in the field to determine the soil sample weight. The soil samples in reagent water or sodium bisulfate solution are not reweighed in the field because the stationary laboratory determines the sample weight.

Usually, three co-located samples are taken and placed into their individual vials so that the laboratory has an appropriate sample volume. At the laboratory, the capped vials containing reagent water or sodium bisulfate solution are weighed to obtain the weight of the soil. The methanol-preserved samples are re-weighed to verify any preservative loss due to volatilization. For Low Level Analysis, the samples containing reagent water or sodium bisulfate solution are prepared and analyzed with the caps in-place. The vial caps are not removed throughout the entire storage, preparation, and analysis procedure. Hence, the VOA vials must be compatible with the laboratory's autosampler instrumentation to avoid further sample handling which might promote VOC loss. All surrogates, internal standards, extract aliquots and matrix spikes are introduced and removed through the septums, either manually or mechanically. For High Level Analysis, the methanol-preserved samples are analyzed by Method 5030.

Several coring devices are available for the collection of soil subcores, which can readily transfer the soil subcores into the relatively narrow opening of a VOA vial. These devices include the EasyDraw Syringe™ and PowerStop Handle™, the Purge-and-Trap Soil Sampler™, the Lock N' Load™ Soil Sampling Tool, and a cut plastic syringe. Any equivalent device may also be used after consultation with DTSC prior to sampling. The coring devices are usually disposable and should not be re-used. To expedite the soil sampling, numerous coring devices should be taken into the field.

A.1 Field Procedures at a Sample Location Point

- 1) On the day of the field activities, weigh the pre-tared methanol-preserved VOA vials to verify that no preservative has evaporated. All weights must be recorded to within 0.05 grams¹¹. To the extent possible, field personnel should weigh the VOA vials in a protected environment to permit accurate weighing. The VOA vials containing reagent water or sodium bisulfate solution do not require weighing prior to sample collection.
- 2) Discard all methanol-preserved VOA vials with unacceptable preservative loss of greater than 0.05 grams.
- 3) Record the weight of the methanol-preserved VOA vials in the field log book.
- 4) Construct or assemble the subcoring device pursuant to the manufacturer's instructions.
- 5) Push the coring device into a freshly exposed soil surface. Continue pushing until the soil column inside the coring device has forced the device's plunger to the stopping point or until the appropriate amount of soil has been collected, usually five grams (two to three cubic centimeters).
- 6) Use a paper towel to quickly wipe the exterior of the coring device to remove excess soil.
- 7) Insert the end of the coring device into the pre-tared VOA vial and eject the soil sample into the vial by pushing on the plunger of the coring device. Avoid splashing the preservative out of the VOA vial by holding the VOA vial at an angle. The mouth of the coring device should not contact the preservative.
- 8) Use a paper towel to quickly wipe the VOA vial threads to remove excess soil and cap, hermetically sealing the vial. Note: Steps 5 – 8 should be done as quickly as possible, usually within two minutes, to prevent VOC loss.
- 9) Gently swirl the soil sample in the VOA vial to mix and break up the soil aggregate until the soil is covered with the preservative. The swirling of the VOA vial should not allow the soil to contact the PTFE septum. The PTFE septum must remain free of soil to allow for the analysis of the sample through the septum. Hence, do not vigorously shake the vial.
- 10) Re-weigh the methanol-preserved VOA vials to determine the weight of the soil sample. The VOA vials containing reagent water or sodium bisulfate solution do not require weighing after sample collection.
- 11) Using the pre-adhered label on the VOA vial, complete the label information as needed. The VOA vials as supplied from the laboratory or a certified vendor will be pre-labeled. Hence, no additional labeling of the VOA vials in the field should be done that might alter the weight of the sample container. If it is necessary to include another label, a label can be applied to the exterior of the plastic bag containing the vial.

¹¹ USEPA Method 5035 specifies that all weights should be recorded within 0.01 grams but most commercially available electronic balances only have the capacity to accurately measure within 0.05 grams; hence, 0.05 is used within this Guidance Document as the accuracy threshold.

- 12) Place the VOA vial into a resealable plastic bag and place the package into a cooler chilled to $4 \pm 2^{\circ}\text{C}$ or $<-7^{\circ}\text{C}$, as needed. The VOA vials should be transported to the laboratory in an upright position whenever possible. However, VOA vials subject to freezing at $<-7^{\circ}\text{C}$ should be transported to the laboratory at a 45° angle to prevent vial breakage due to preservative expansion.
- 13) Repeat the procedure as necessary to obtain the required number of Method 5035 soil samples.
- 14) As needed, collect a soil sample for the measurement of the dry weight of the soil. The sample does not need chemical preservation and can be collected in either a sealable glass jar or empty VOA vial.

A.2 Field Considerations

- a) Disposable plastic syringes can be easily converted into an inexpensive coring device. The "needle end" of the syringe barrel is cut-off with a sharp knife or scissors, creating a blunt, even coring end. The barrel diameter of the plastic syringe must be narrower than the diameter of the VOA vial for soil extrusion. Prior to field activities, the approximate volume associated with five grams of soil must be determined. Hence, it may be necessary to calibrate the syringe by collecting and weighing trial soil quantities with the plastic syringe to determine the length of soil in the syringe barrel that corresponds to 5.0 ± 0.5 grams.
- b) Do not use or submit samples for analysis if the preservative has spilled or splashed from the VOA vial. Extra tared and preserved VOA vials should be taken into the field anticipating potential preservative loss due to evaporation or spillage. Methanol-preserved VOA vials should be weighed in the field prior to soil sample collection. A significant change in weight of the VOA vial indicates preservative loss and the VOA vial should not be used. Unacceptable preservative loss is 0.05 grams. After sample collection and before transport to the laboratory, the samples are reweighed to determine the weight of the samples.
- c) Rough trimming of a sampling location's surface layer should be considered if the soil has been exposed to ambient air for more than two minutes. Removal of the surface layer can be accomplished by scraping the soil surface with a clean spatula, scoop, trowel, or knife.
- d) The collection of numerous co-located subcores from a core sample may be difficult due to the small diameter of the core. Accordingly, care should be taken in obtaining all the necessary subcores. All subcores should be taken from a fresh surface. If a fresh surface is not available after the first or second co-located sample, the core sample should be slowly extruded from the core barrel and cut to expose additional subcore sampling areas.
- e) Many core barrels are not entirely full upon retrieval from the subsurface. Hence, upon subcoring, these partially full core barrels will require bracing or support so that subcoring can occur without pushing the soil core into the interior of the barrel.
- f) The collection of subsequent co-located subcores should not begin until the previous subcore is sealed in its vial.
- g) Field personnel should wear powderless gloves during sample collection to avoid VOC exposure.

- h) The threads of the VOA vials must be free of soil; otherwise the cap will not seal properly, compromising the integrity of the sample.
- i) To maintain sample integrity, only two minutes should ideally transpire between core retrieval from the subsurface and subcoring by the coring device.
- j) Soil samples with known high concentrations of VOCs should be separated from soil samples with low concentrations to prevent cross contamination. Ideally, the soil samples of differing VOC concentrations should be placed into different shipping bags and, if possible, placed into separate field coolers.
- k) Calcareous soil samples may effervesce upon contact with sodium bisulfate solution and compromise the integrity of the sample. Off-gassing could result in VOC loss as the soil contacts the effervescing acid. Pressure build-up after sealing the VOA vial could cause the vial to shatter or the carbonates in the soil could buffer the acid, rendering sodium bisulfate solution ineffective as a preservative. Accordingly, the soils at the site should be evaluated for potential effervescence prior to sampling activities and the occurrence of effervescence should be reported to the laboratory. In cases where effervescence is a potential problem, an alternative sample collection method should be utilized.
- l) Methanol is a toxic and flammable liquid, and must be handled with appropriate safety precautions. Inhalation of methanol vapors should be avoided. It should be handled in well ventilated areas and stored away from open flames and other ignition sources as well as extremely hot areas. Sodium bisulfate solution is a mineral acid and must be handled with appropriate safety precautions. Contact with skin and eyes should be avoided. Protective gloves and eye protection should be worn when handling vials containing sodium bisulfate solution.
- m) Depending on the quantity and method of packaging, methanol and sodium bisulfate solution may be considered Department of Transportation (DOT) Hazardous Materials and subject to DOT hazardous materials regulations.
- n) Consult the laboratory to determine if magnetic stir bars should be added to the VOA vials prior to hermetic sealing in the field. Soil samples subject to Low Level Analysis must be agitated during analysis to assist the VOC purge process. Agitation can be accomplished by either sonication or stirring with magnetic bars. Hence, if the stationary laboratory does not have the ability to sonicate the soil sample with their instrumentation, magnetic stir bars must be added to the VOA vials subject to Low Level Analysis.

A.3 Potential Field Equipment

- VOA Vials – Extra preserved VOA vials should be taken into the field due to potential breakage or expansion of the sampling program due to unanticipated field conditions. Typically, the VOA vials are 40 milliliters in size.
- Digital Field Scale – Used to weigh VOA vials to verify no methanol loss prior to sampling; if the field scale is a balance-type, calibrated weights must also be taken into the field. All

scales must have an accuracy of 0.05 grams. The field scale is also used to determine the volumetric amount of soil needed for five grams of sample.

- Coring Devices – Used to obtain the soil subcores; must have a diameter that is slightly smaller than the VOA vials.
- Gloves – Used for health and safety protection; powderless preferable.
- Paper Towels – Used to clean VOA vial threads for proper cap attachment.
- pH Test Strips – Used to verify that soil subcores are preserved to a pH of less than 2.0 in the VOA vials.
- Acid Preservative – Used as needed to reduce the pH in the VOA vials to less than 2 before adding the soil subcore to the vial; the addition of acid should only be done if the VOA vials were incorrectly preserved by the laboratory or vendor.
- Field Cooler – Insulated ice chest for sample storage and shipment; must be capable of cooling and maintaining the soil samples to $4 \pm 2^{\circ}\text{C}$ or $<-7^{\circ}\text{C}$, as needed.
- Ice – Used to chill field cooler and samples to $4 \pm 2^{\circ}\text{C}$. Wet ice preferred over blue ice for chilling to $4 \pm 2^{\circ}\text{C}$. The wet ice should be double bagged to prevent filling the field coolers with water which may damage the labels on the samples. Dry ice, as needed, for chilling the field cooler to $<-7^{\circ}\text{C}$.
- Field Blades – Clean spatula, scoop, trowel or knife used for exposing fresh soil surfaces before subcoring.
- Indelible Ink Pens – Labeling of the soil sample containers; pens that use VOCs, such as markers, are not recommended due to potential cross-contamination.
- Resealable Plastic Bags – Used as a secondary container to prevent moisture infiltration during sample transport.
- Glass Containers – Used to collect soil samples for dry weight determination. Samples for dry weight determination can also be collected in brass sleeves or acetate liners. These samples do not need preservation. However, the collection method should minimize sample handling, sample disaggregation, and moisture loss.
- Decontamination Equipment – Used to decontaminate the field blades and coring devices, for repeated use, as needed.

Appendix B: Sampling Option 2

Sampling Options 2A, 2B, and 2C Multi-Functional Sampling Devices

Multi-functional sampling devices (MFSDs) are used which act as both a coring tool and airtight storage container. Examples of MFSDs are the EnCore™ Sampler and the Core N' One™ Sampler. The MFSDs collect a small sample subcore directly into a volumetric storage chamber, filling it completely with zero headspace. The soil sample size can be either five grams or 25 grams, dependent on the MFSD size¹². MFSDs are to be used on cohesive but uncemented soils that will form a cohesive plug when sampled. The storage containers are then capped, forming an airtight seal. The intact samples are transported to the laboratory in the sealed device at $4 \pm 2^\circ\text{C}$. Usually, three co-located samples are taken with the MFSDs so that the stationary laboratory has an appropriate sample volume; however, fewer number of samples may be taken pursuant to Figure 1 if the VOC concentrations can be quantified with high detection limits ($200 \mu\text{g}/\text{kg}$). At the stationary laboratory, the soil sample within the MFSD is transferred to a VOA vial. The diameter of the VOA vial must be sufficiently large to accept the soil sample from the MFSD without alteration and the VOA vial must be compatible with the laboratory's autosampler instrumentation.

B.1 Field Procedures at a Sample Location Point

- 1) Enter the preliminary sample identification information on the label of the MFSD package. Usually, each sampler is individually packaged in a resealable plastic bag with usage instructions attached. No pre-sampling container preparation is required.
- 2) Remove the sampler and cap from the package and assemble the MFSD pursuant to its instructions.
- 3) Push the coring body of the MFSD into a freshly exposed soil surface, filling the sampling chamber. The MFSD should be visually checked to verify that a headspace-free subcore has filled the chamber. Any excess soil extruding from the sample chamber should be carefully removed by trimming away the excess with a clean field blade.
- 4) Use a paper towel to quickly wipe the sampler head to remove excess soil from the exterior so that the cap can be tightly attached.
- 5) For an Encore™ Sampler, carefully push the cap on with a gentle twisting motion to firmly attach the cap to the chamber, taking care not to damage the o-ring seal on the sampler. For a Core N' One™ Sampler, the cap is gently treaded onto place on the sampling chamber, taking care to properly seat the sealing gasket on the chamber. Note: Steps 3 – 5 should be done as quickly as possible, usually within two minutes, to prevent VOC loss.
- 6) Complete the label information and attach label as needed or required. The label should be placed only on the exterior bag containing the MFSD and not on the MFSD itself.

¹² Generally, the 5 gram MFSDs are used for the determination of VOC concentrations in soil and the 25 gram MFSDs are used for Toxicity Characteristic Leaching Procedure (TCLP) testing. When conducting TCLP testing, usually two 25 gram samples are taken and submitted to the laboratory.

- 7) Place the MFSD back into its original package and place the package into a cooler chilled to $4 \pm 2^{\circ}\text{C}$.
- 8) Repeat the procedure as necessary to obtain the required number of Method 5035 soil samples.
- 9) As needed, collect a soil sample for the measurement of the dry weight of the soil. The sample does not need to be collected within a MFSD and can be collected in either a sealable glass jar or empty VOA vial.

B.2 Field Considerations

- a) The exterior surface of the MFSD must be free of soil; otherwise the cap will not seal properly, compromising the integrity of the sample.
- b) Rough trimming of a sampling location's surface layer should be considered if the soil has been exposed to ambient air for more than two minutes. Removal of the surface layer can be accomplished by scraping the soil surface with a clean spatula, scoop, trowel, or knife.
- c) The collection of numerous co-located subcores from a core sample may be difficult due to the small diameter of the core. Accordingly, care should be taken in obtaining all the necessary subcores. All subcores should be taken from a fresh surface. If a fresh surface is not available after the first or second co-located sample, the core sample should be slowly extruded from the core barrel and cut to expose additional subcore sampling areas.
- d) Many core barrels are not entirely full upon retrieval from the subsurface. Hence, upon subcoring, these partially full core barrels will require bracing or support so that subcoring can occur without pushing the soil core into the interior of the barrel.
- e) To maintain sample integrity, only two minutes should ideally transpire between core retrieval from the subsurface and subcoring by the MFSD.
- f) The collection of subsequent co-located subcores should not begin until the previous subcore is sealed in its MFSD.
- g) Soil samples with known high concentrations of VOCs should be separated from soil samples with low concentrations to prevent cross contamination. Ideally, the soil samples of differing VOC concentrations should be placed into different shipping bags and, if possible, placed into separate field coolers.
- h) Field personnel must communicate the required detection limits of the soil samples to the stationary laboratory so that the proper extraction procedures can be followed.
- i) A 25 gram MFSD is used to collect, store, and transfer soils for Toxicity Characteristic Leaching Procedure (TCLP) testing, and must not be subsampled by the laboratory into five gram aliquots for VOC analysis per Method 5035.
- j) Field personnel should wear powderless gloves during sample collection to avoid VOC exposure.

- k) The soil sample collected for measurement of dry weight will also be used by the laboratory to evaluate the soil for reactivity with sodium bisulfate solution prior to Low Level Analysis.

B.3 Potential Field Equipment

- Multi-Functional Sampling Devices – Extra MFSDs should be taken into the field due to potential MFSD breakage or expansion of the sampling program due to unanticipated field conditions.
- Gloves – Used for health and safety protection; powderless preferable.
- Paper Towels – Used to clean sampler head of the MFSD for proper cap attachment.
- Field Cooler – Insulated ice chest for sample storage and shipment; must be capable of cooling and maintaining the soil samples to $4 \pm 2^{\circ}\text{C}$.
- Ice – Used to chill field cooler and samples to $4 \pm 2^{\circ}\text{C}$. Wet ice preferred over blue ice for chilling to $4 \pm 2^{\circ}\text{C}$. The wet ice should be double bagged to prevent filling the field coolers with water which may damage the labels on the samples.
- Field Blades – Clean spatula, scoop, trowel or knife used for exposing fresh soil surfaces before subcoring.
- Indelible Ink Pens – Labeling of the soil sample containers; pens that use VOCs, such as markers, are not recommended due to potential cross-contamination.
- Resealable Plastic Bags – Used as a secondary container to prevent moisture infiltration during sample transport.
- Glass Containers – Used to collect soil samples for dry weight determination. Samples for dry weight determination can also be collected in brass sleeves or acetate liners. These samples do not need preservation. However, the collection method should minimize sample handling, sample disaggregation, and moisture loss.
- Decontamination Equipment – Used to decontaminate the field blades, for repeated use, as needed.

Appendix C: Sampling Option 3

Sampling Options 3A, 3B, and 3C Non-Preserved VOA Vials

Tared and labeled VOA vials with a PTFE-lined septum caps are taken into the field as supplied by the laboratory or certified vendor, cleaned to USEPA specifications. Typically, the VOA vials are 40 milliliters in size. The VOA vials do not contain chemical preservatives, water-miscible solvents, or reagent water. Soil cores of appropriate mass are placed into the VOA vials in the field and are capped, forming an airtight seal. However, magnetic stir bars may be needed in the VOA vials pursuant to the laboratory's requirements.

Usually, three co-located samples are taken and placed into their individual vials so that the laboratory has an appropriate sample volume. At the laboratory, the capped vials are weighed to obtain the weight of the soil. For Low Level Analysis, the samples are prepared and analyzed with the caps in-place. The vial caps are not removed throughout the entire storage, preparation, and analysis procedure. Hence, the VOA vials must be compatible with the laboratory's autosampler instrumentation to avoid further sample handling which might promote VOC loss. All surrogates, internal standards, extract aliquots and matrix spikes are introduced and removed through the septums, either manually or mechanically. For High Level Analysis, the samples are analyzed by Method 5030.

Several coring devices are available for the collection of soil subcores, which can readily transfer the soil subcores into the relatively narrow opening of a VOA vial. These devices include the EasyDraw Syringe™ and PowerStop Handle™, the Purge-and-Trap Soil Sampler™, the Lock N' Load™ Soil Sampling Tool, and a cut plastic syringe. Any equivalent device may also be used after consultation with DTSC prior to sampling. The coring devices are usually disposable and should not be re-used. To expedite the soil sampling, numerous coring devices should be taken into the field.

C.1 Field Procedures at a Sample Location Point

- 1) Obtain tared and labeled VOA vials from the laboratory or certified vendor.
- 2) Construct or assemble the subcoring device pursuant to the manufacturer's instructions.
- 3) Push the coring device into a freshly exposed soil surface. Continue pushing until the soil column inside the coring device has forced the device's plunger to the stopping point or until the appropriate amount of soil has been collected, usually five grams (two to three cubic centimeters).
- 4) Use a paper towel to quickly wipe the exterior of the coring device to remove excess soil.
- 5) Insert the end of the coring device into the pre-tared VOA vial and eject the soil sample into the vial by pushing on the plunger of the coring device.
- 6) Use a paper towel to quickly wipe the VOA vial threads to remove excess soil and cap, hermetically sealing the vial. Note: Steps 2 - 5 should be done as quickly as possible, usually within two minutes, to prevent VOC loss. Also, care should be taken so that the PTFE

septum remains free of soil to allow for the analysis of the sample through the septum. Hence, do not shake the vial.

- 7) Using the pre-adhered label on the VOA vial, complete the label information as needed. The VOA vials as supplied from the laboratory or a certified vendor will be pre-labeled. Hence, no additional labeling of the VOA vials in the field should be done that might alter the weight of the sample container. If it is necessary to include another label, a label can be applied to the exterior of the plastic bag containing the vial.
- 8) Place the VOA vial into a resealable plastic bag and place the package into a cooler chilled to $4 \pm 2^{\circ}\text{C}$ or $<-7^{\circ}\text{C}$, as needed. The VOA vials should be transported to the laboratory in an upright position whenever possible. However, VOA vials subject to freezing at $<-7^{\circ}\text{C}$ should be transported to the laboratory at a 45° angle to prevent vial breakage due to sample expansion.
- 9) Repeat the procedure as necessary to obtain the required number of Method 5035 soil samples.
- 10) As needed, collect a soil sample for the measurement of the dry weight of the soil. The sample can be collected in a sealable glass jar or empty VOA vial.

C.2 Field Considerations

- a) Disposable plastic syringes can be easily converted into an inexpensive coring device. The "needle end" of the syringe barrel is cut-off with a sharp knife or scissors, creating a blunt, even coring end. The barrel diameter of the plastic syringe must be narrower than the diameter of the VOA vial for soil extrusion. Prior to field activities, the approximate volume associated with five grams of soil must be determined. Hence, it may be necessary to calibrate the syringe by collecting and weighing trial soil quantities with the plastic syringe to determine the length of soil in the syringe barrel that corresponds to 5.0 ± 0.5 grams.
- b) Rough trimming of a sampling location's surface layer should be considered if the soil has been exposed to ambient air for more than two minutes. Removal of the surface layer can be accomplished by scraping the soil surface with a clean spatula, scoop, trowel, or knife.
- c) The collection of numerous co-located subcores from a core sample may be difficult due to the small diameter of the core. Accordingly, care should be taken in obtaining all the necessary subcores. All subcores should be taken from a fresh surface. If a fresh surface is not available after the first or second co-located sample, the core sample should be slowly extruded from the core barrel and cut to expose additional subcore sampling areas.
- d) Many core barrels are not entirely full upon retrieval from the subsurface. Hence, upon subcoring, these partially full core barrels will require bracing or support so that subcoring can occur without pushing the soil core into the interior of the barrel.
- e) The collection of subsequent co-located subcores should not begin until the previous subcore is sealed in its vial.
- f) Field personnel should wear powderless gloves during sample collection to avoid VOC exposure.

- g) The threads of the VOA vials must be free of soil; otherwise the cap will not seal properly, compromising the integrity of the sample.
- h) To maintain sample integrity, only two minutes should ideally transpire between core retrieval from the subsurface and subcoring by the coring device.
- i) Soil samples with known high concentrations of VOCs should be separated from soil samples with low concentrations to prevent cross contamination. Ideally, the soil samples of differing VOC concentrations should be placed into different shipping bags and, if possible, placed into separate field coolers.
- j) Field personnel must communicate the required detection limits of the soil samples to the stationary laboratory so that the proper extraction procedures can be followed.
- k) Consult the laboratory to determine if magnetic stir bars should be added to the VOA vials prior to hermetic sealing in the field. Soil samples subject to Low Level Analysis must be agitated during analysis to assist the VOC purge process. Agitation can be accomplished by either sonication or stirring with magnetic bars. Hence, if the stationary laboratory does not have the ability to sonicate the soil sample with their instrumentation, magnetic stir bars must be added to the VOA vials subject to Low Level Analysis.
- l) The soil sample collected for the measurement of dry weight can also be used by the laboratory to evaluate the soil for reactivity with sodium bisulfate solution prior to Low Level Analysis.

C.3 Potential Field Equipment

- VOA Vials – Extra VOA vials should be taken into the field due to potential breakage or expansion of the sampling program due to unanticipated field conditions. Typically, the VOA vials are 40 milliliters in size.
- Coring Devices – Used to obtain the soil subcores; must have a diameter that is slightly smaller than the VOA vials.
- Gloves – Used for health and safety protection; powderless preferable.
- Paper Towels – Used to clean VOA vial threads for proper cap attachment.
- Digital Field Scale – Used to determine the volumetric amount of soil needed for five grams of sample.
- Field Cooler – Insulated ice chest for sample storage and shipment; must be capable of cooling and maintaining the soil samples to $4 \pm 2^{\circ}\text{C}$ or $<-7^{\circ}\text{C}$, as needed.
- Ice – Used to chill field cooler and samples to $4 \pm 2^{\circ}\text{C}$. Wet ice preferred over blue ice for chilling to $4 \pm 2^{\circ}\text{C}$. The wet ice should be double bagged to prevent filling the field coolers with water which may damage the labels on the samples. Dry ice, as needed, for chilling to $<-7^{\circ}\text{C}$.

- Field Blades – Clean spatula, scoop, trowel or knife used for exposing fresh soil surfaces before subcoring.
- Indelible Ink Pens – Labeling of the soil sample containers; pens that use VOCs, such as markers, are not recommended due to potential cross-contamination.
- Resealable Plastic Bags – Used as a secondary container to prevent moisture infiltration during sample transport.
- Glass Containers – Used to collect soil samples for dry weight determination. Samples for dry weight determination can also be collected in brass sleeves or acetate liners. These samples do not need preservation. However, the collection method should minimize sample handling, sample disaggregation, and moisture loss.
- Decontamination Equipment – Used to decontaminate the field blades and coring devices, for repeated use, as needed.

Appendix D: USEPA Interim Policy

**United States Environmental Protection Agency
Region IX**

**Regional Interim Policy for Determination of Volatile Organic Compound (VOC)
Concentrations in Soil and Solid Matrices**

June 23, 1999



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION IX
75 Hawthorne Street
San Francisco, CA 94105-3901

June 23, 1999

MEMORANDUM

SUBJECT: Regional Interim Policy for Determination of Volatile Organic Compound (VOC) Concentrations in Soil and Solid Matrices.

FROM: Nora McGee, Assistant Regional Administrator
USEPA Region 9

TO: USEPA Region 9 Personnel and Parties Collecting Environmental Measurements Under Regional Programs.

Purpose

Appropriate methodologies to minimize volatilization and biodegradation losses in solid matrices have not been consistently implemented throughout Region 9. This memorandum articulates the Region's policy on the adoption of sampling and laboratory methodologies for the collection of volatile organic compound (VOC) data from soil or solid matrices. USEPA SW-846, Update III, Method 5035, "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," incorporating procedures to minimize VOC losses was finalized by USEPA in June 1997. This Region 9 policy requires the use of Method 5035, or an equally or more effective method, for the collection of representative and precise data for VOCs in soil and solid matrices. Additionally, this policy was developed to be consistent with the Agency's Data Quality Objectives (DQO) Process (outlined in "Guidance for the Data Quality Objectives Process," USEPA QA/G-4, September 1994) by allowing for a graded approach through the collection of representative data that meets project data quality needs.

Policy

Scope and Applicability

Environmental data collection activities performed under USEPA Region 9 programs for the determination of VOC concentrations in soil and solid matrices.

This policy is applicable to data collection activities conducted by USEPA staff and contractors, USEPA grantees, Federal Facilities, entities complying with USEPA regulatory requirements and/or other entities producing data for USEPA decision making. This includes data being collected under ongoing quality assurance plans and sampling plans.

INTERIM POLICY

Time Frame for Implementation

This policy should be adopted quickly and to the maximum practicable extent. Cases where it is not practicable to implement this policy should be brought to the attention of the USEPA Region 9 QA Office. This is being put forth as an interim policy, as USEPA is still evaluating technical information to further refine procedures for minimization of VOC losses. Please note, an amendment to this policy may be required.

Statement of Policy

Methods for the collection and analysis of VOCs in soil or other solid matrices must minimize volatile losses. Because USEPA SW-846 Method 5035 does not rigorously dictate specifics of field sample collection¹ and laboratory sample handling protocols, project specific procedures to minimize volatile losses must be developed and be included in the site/program quality assurance project plan (QAPP) or sampling and analysis plan (SAP). USEPA SW-846 Method 5021 “Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis,” also incorporates procedures to minimize volatile losses. However, Method 5021 should be used with caution, as it can be reasonably interpreted and performed in a way which does not prevent loss of VOCs. USEPA Region 9 considers the following practices as minimum requirements to reduce volatile losses in soil samples:

1. Samples are handled as intact² soil cores in the field and laboratory.
2. Samples are stored in containers which can be reliably sealed to prevent volatilization losses³ over the project specified analytical holding time.
3. Samples are analyzed or chemically, acid or methanol, preserved within 48 hours of collection, if any contaminant may undergo biodegradation.
4. Exposure of the sample core to the atmosphere in the field and laboratory should be minimized⁴.

¹ ASTM Method D4547-98 “Standard Guide for Sampling Waste and Soils for VOCs,” is a good reference for VOC sampling protocols.

² Soils should always be collected and transferred using a coring device, such as a metal sleeve or cut off syringe. Use of transfer devices, such as spatulas, is not acceptable either in the field or laboratory.

³ Volatilization losses from sampling/storage containers must be less than what would be expected from a volatile organic analysis vial with a Teflon/silicon septa stored for 14 days, unless project DQOs require more stringent requirements.

⁴ Field sub-cores should be taken immediately upon exposing the soil core to ambient conditions. Sub samples should be directly extruded into the analysis containers. Total exposure of samples to ambient conditions should not be more than 15 seconds.

USEPA Region 9 will consider exceptions to this policy on a case-by-case basis. All deviations from procedures outlined in Method 5035 should be documented in a QAPP or a SAP which must be submitted to, and approved by, the Region 9 QA Office. Additionally, the party responsible for data collection must demonstrate that the methodologies proposed will result in data that meet project/program data quality objectives (DQOs).

Additional Considerations

Field Laboratories: The use of field laboratories, that analyze samples within several hours of collection, is an excellent choice to prevent loss of volatiles in transit and storage. However, the sample collection and analysis procedures used must prevent volatilization losses and comply with requirements 1 and 4 articulated in the Statement of Policy. Additionally, the quality control criteria and quality assurance system used by a field laboratory must be adequate for generation of data which will meet project DQOs.

Addition of Surrogates and Matrix Spiking Compounds in the Field: The most appropriate time for addition of analytical surrogate and matrix spiking compounds into soils is prior to sample extraction, by water or a solvent. Method 5035 does not incorporate the addition of the compounds prior to extraction in the field. Because this is an important control check on the analytical process, which begins at extraction, for some project/program DQOs it may be appropriate to incorporate a procedure which adds surrogate and/or matrix spiking compounds prior to extraction.

Holding Times: The holding time for preserved soil samples should be interpreted as 14 days from the time of sample collection (stored at $4\pm 2^{\circ}\text{C}$). Due to potential biodegradation losses, samples stored in sealed containers, but not chemically preserved, should not be stored for more than 48 hours. On a project/program specific basis, USEPA Region 9 will consider other alternatives to extend the holding time of soils that have not been chemically preserved (see Attachment A). Holding time will be considered as cumulative (see Attachment B for holding time examples). Exceptions should be documented in a QAPP or a SAP submitted to and approved by the Region 9 QA Office.

Unconsolidated Solid Matrices: Solid Matrices that are not amenable to the use of a coring technique should be collected in such a way as to preserve the integrity of the sample matrix. Transferring of these soils with spatulas or similar devices into sampling containers is discouraged as this disrupts the sample pore spaces and greatly increases the sample surface area available for volatilization. For soil piles, fresh soil at an adequate depth should be sampled.

Calcareous Soils: Method 5035 notes that, “Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial.” Calcareous soils that effervesce on contact with the low-level preservative solution should be collected using an alternative preservation technique (see Attachment A).

Soil Gas: This policy is not intended to address the role of soil gas in the environmental decision making process. The Region recognizes that soil gas data is used extensively, in USEPA Region 9, for site decision making and in some cases soil gas is the preferred tool for gathering data on subsurface conditions. However, there are also scenarios where soil gas data are unacceptable for agency decision making (e.g., in excavated soils and when determining disposal options).

Drilling Techniques: This policy does not address the impact of drilling techniques on the collection of a representative VOC sample. Site/program QAPPs and SAPs should address the impact of all collection techniques on sample integrity and select those appropriate for the DQOs. Potential VOC losses due to drilling techniques include, but are not limited to: sample compression and loss of pore space; air introduction into the sample matrix; heat introduced in the drilling process; and volatilization from prolonged periods in a non-hermetically sealed sampling apparatus.

Background

Traditional practices for the sampling and analysis of volatile organic compounds (VOCs) in soil have been shown to have a significantly low bias of inconsistent magnitude (Grant, 1996) from volatilization (Hewitt, 1996) and biodegradation (Hewitt, 1994). Based on this and other research, the USEPA modified the methodology in SW846 for collection and analysis of volatiles in soil. Soil was deleted as an option from Method 5030 and Method 5035 and Method 5021 were added. These methods provide for handling of samples as intact soil cores, chemical preservation techniques, storage of samples in hermetically sealed containers and minimization of analyte losses due to direct volatilization (both in the field and the laboratory) and biodegradation.

“Traditional” collection techniques, such as transferring soils to a glass jar with minimal head space and collecting samples directly into a brass sleeve (e.g., CA Split Spoon) do not yield accurate or consistent results. It has been specifically demonstrated that capped brass sleeves show significant losses. Hewitt and Lukash (Hewitt, 1996) demonstrated capped sleeves can show substantial losses in less than one day. Hewitt and Lukash also demonstrated volatile losses in uncapped core liners of up to 90% in less than 40 minutes for trichloroethene (TCE). Because other analytes and matrix types can have higher mobility than those tested, substantial losses may occur in an even shorter period of time. Grant, Jenkins and Mudambi (Grant, 1996) examined split sampling results from a cross section of laboratories. For VOCs in soil they noted that, “The magnitude of this scatter [for a typical data comparison] is so large that it is

impossible to recommend effective limits of acceptability. Instead, we believe that steps are urgently needed to improve data quality.” Hewitt noted (Hewitt, 1994) that biodegradation of Benzene and Toluene in soil samples stored in sealed glass ampules at 4 C for 14 days could be substantial, demonstrating a need for chemical preservatives. Turriff and Reitmeyer (Turriff, 1998) demonstrated that a variety of soil matrices could be held for 48 hours at 4 C, in sealed zero headspace containers, without substantial VOC losses. Additionally, Turriff and Reitmeyer demonstrated that freezing was an option to extend holding times of En Core™ sampling devices. Because volatile losses have been linked to disturbance of the soil matrix and exposure to the atmosphere, samples should be handled in intact soil cores and stored in hermetically sealed vessels in both the field and the laboratory.

This USEPA Region 9 policy is based on the best scientific information available at this time and is subject to further clarifications and additions as other research becomes available. If you have any questions please call Vance Fong at 415 744-1492 or Mathew Plate at 415 744-1493.

References

Hewitt, A.D. (1994) Concentration Stability of Four Volatile Organic Compounds in Soil Subsamples. US Army Cold Regions Research and Engineering Laboratory, Special Report 94-6.

Grant, C.L., T.F. Jenkins and A.R. Mudambi (1996) Comparison Criteria for Environmental Chemical Analyses of Split Samples Sent to Different Laboratories, Corps of Engineers Archived Data. US Army Cold Regions Research and Engineering Laboratory, Special Report 96-9.

Hewitt, A.D. and J.E. Lukash (1996) Obtaining and Transferring Soils for In-Vial Analysis of Volatile Organic Compounds. US Army Cold Regions Research and Engineering Laboratory, Special Report 96-5.

Turriff, D. Ph.D. and C. Reitmeyer (1998) Validation of Holding Times for the EnCore™ Sampler. En Novative Technologies, Inc.

Attachment A

Preservation Alternatives: The following are preservation alternatives that may be appropriate for some projects/programs and are subject to project/program specific approval by the USEPA Region 9 QA Office.

Freezing of unpreserved samples: It has been shown in several studies that freezing of unpreserved soils is an effective means of slowing the biodegradation process. At this time, USEPA Region 9 will accept freezing of unpreserved soils as a method to extend holding times up to seven days on a project specific basis. While there is some evidence that freezing for longer periods may also be acceptable for some data needs, USEPA Region 9 does not believe that the current scientific evidence supports a longer holding time for frozen samples in most cases. Samples should be frozen in containers that have an air tight seal and can maintain this seal while frozen. Because water expands in the freezing process, VOA vials with water or samples with extremely high moisture contents may rupture the storage container.

Preservatives: Acids other than sodium bisulfate may be used to preserve low level samples. The choice of an alternative acid should be made in consultation with the USEPA Region 9 QA Office. In all cases the preserved sample pH should be 2.

Sampling Containers: Currently the Region recognizes three sample collection/storage alternatives which can be used (other than acid/water or methanol, as specified in Method 5035).

1. A VOA vial with 5 mL of water without preservative and approximately 5 g of sample. Which must be analyzed within 48 hours of collection by closed system purge and trap.
2. A VOA vial with approximately 5 g of sample. Water must be introduced through the septa at time of analysis by closed system purge and trap. Sample must be analyzed within 48 hours of collection if stored at $4\pm 2^{\circ}\text{C}$ or 7 days if frozen. (This alternative must be approved on a project specific basis.)
3. An En Core™ sampler which is analyzed or preserved within 48 hours of collection if stored at $4\pm 2^{\circ}\text{C}$ or analyzed within 7 days if frozen. (Freezing of En Core™ samplers must be approved on a project specific basis.)

If requested, USEPA Region 9 QA Office will consider the applicability of other sampling containers/devices that have been demonstrated, with appropriate supporting documentation, to be adequate for collection and storage of VOCs.

INTERIM POLICY

Attachment B Examples of Holding Time Policy

Example 1 Sample is placed into a vial without chemical preservative in the field (due to effervescence) and stored at $4\pm 2^{\circ}\text{C}$.

Sample must be analyzed within 48 hours of collection.

Example 2 Sample is collected into a hermetically sealed sub-coring and storage device in the field, stored at $4\pm 2^{\circ}\text{C}$ and transferred into a vial without chemical preservative in the laboratory.

Sample must be analyzed within 48 hours of collection.

Example 3 Sample is collected into a hermetically sealed sub-coring and storage device, transported/stored at $4\pm 2^{\circ}\text{C}$, frozen at the laboratory 28 hours after collection, defrosted after 2 days and transferred into a vial without chemical preservative in the laboratory.

Sample must be analyzed within 20 hours from the time the sample is defrosted to $4\pm 2^{\circ}\text{C}$.

48 (hours allowed) - 28 (hours before freezing) = 20 (hours allowed from defrosting to analysis)

COMMENT SHEET
Method 5035 Guidance Document

As a user of this guidance document, your comments are important to the Department of Toxic Substances Control. Please use this sheet to inform us of any errors, deficiencies, or suggestions that may improve this document. If you identify errors or technical deficiencies, please provide suggestions for their rectification.

Please send comments to:

Department of Toxic Substances Control
8800 Cal Center Drive
Sacramento, California 95826-3200
Attention: Geological Services Unit

Your name and address are optional, but if included, a written response will be provided.

APPENDIX C

F4 REMEDIATION

SOIL MOISTURE GUIDANCE MEMORANDUM

June 10, 2015

Mr. Paul Parmentier, P.G., C.HG.
The Source Group, Inc.
1962 Freeman Avenue
Signal Hill, CA 90755

Subject: Soil Moisture – DFSP Norwalk Project

Dear Mr. Parmentier:

F4 Remediation, Inc. (F4) has reviewed the on-site processing of soil, soil characteristics, and the results of progress sampling conducted to date at the DFSP Norwalk Site. Attached, is a table of summary data of moisture measurements, a figure summarizing contaminant levels of treatment rows in the Powerine Basin (Performance Map) and a figure with surveyed volumes for each row within the Powerine Basin. Based on this data and site-specific field experience, F4 is providing this correspondence to modify the soil moisture conditioning and maintenance requirements. F4 requests that SGI implement these revised requirements for future treatment rows.

Conditioning of Soil for Placement in Treatment Rows

F4's original guidance recommended maintaining soil moisture between 40 and 85% of field capacity. However, the soil at DFSP Norwalk, in general, has a very high field capacity; on the order of 40% to 45% water by volume. This level of moisture is potentially detrimental to the effectiveness of the F4 bacteria. Excessive soil moisture restricts the movement of air through the soil thereby reducing the availability of oxygen which is necessary for aerobic bacterial growth. In general the soil should be moist but not overly saturated. The quantification of soil moisture (post conditioning) is important after the soil is deemed adequately hydrated, as it facilitates future recognition of unexpected drying.

Pre-treated soil should be visually inspected and tensiometer measurements taken. The target tensiometer measurement for pre-treated soil **should not exceed a threshold of 20 centibars or fall below 4 centibars** (a higher pressure reading indicates dryer soil). This tensiometer range represents a soil moisture percentage range of approximately 5 to 10% by volume, which is sufficient moisture to establish and maintain the bacteria. If surfactant is applied during excavation for odor suppression, avoid taking tensiometer readings in soil that may have surfactant mixed in the soil. The surfactant will cause the tensiometer to overstate the actual moisture pressure measurements. Provided the soil moisture is visually acceptable and the tensiometer

measurement validates the moisture is adequate, the soil can be processed and placed in the treatment rows.

Tensiometer measurements should then be taken at the completed treatment rows and laboratory analysis conducted to correlate the field tensiometer data with laboratory soil moisture (expressed as percent water by volume). This reference data, in conjunction with contaminant concentration levels, can then be used to evaluate the performance of the treatment pile over the period of remediation.

F4 has also observed that once the treated soil is placed into rows and covered, it retains moisture well. As can be seen in the summary table of moisture data, soil excavated and placed in treatment rows between mid-March through mid-April still had adequate moisture by mid-May. No moisture was introduced beyond water applied during excavation. The ventilation system, which is plumbed to the treatment rows (at four locations along a typical row) and operates at a flow rate of 35 cubic feet per minute (cfm), appears to have had little “drying” effect during the first approximately 60-days of treatment.

F4 originally prescribed installation of irrigation with soaker hoses to mitigate potential overall drying of the treatment piles. With soil remediation cycle times expected to be less than 90 days, augmentation of moisture in the treatment rows by the use of the soaker hose installations is unnecessary.

In addition, the soaker hose configuration would have the effect of applying moisture indiscriminately and potentially over-hydrating areas that have adequate moisture inadvertently disrupting bacterial growth and distribution. If hydration is required, injection of water at specific locations will better serve moisture augmentation.

F4 requests that SGI terminate installing the surface irrigation systems. In the unlikely event moisture is needed in select locations, F4 will direct SGI to apply moisture using a root soaking tool connected to a standard water hose at a rate of approximately 5 gallons per minute (gpm).

Post Augmentation (Re-Treatment) of Soil During Remediation Cycle in Treatment Row

The combination of performance data (i.e., contaminant concentration trends, bacterial plate counts, and moisture measurements) are adequate to make the determination of the performance of a given treatment row. The performance data provide a more deterministic means of ensuring soil is effectively remediated to the desired clean up goals. In the event the performance data of a treatment row (or section within a row) indicates the processed material is not being effectively remediated, the soil can be reprocessed.

As can be seen on the Performance Map figure, the majority of the soil in the Powerine Basin has been remediated to levels below the cleanup goals. After future confirmation sampling is conducted, the contaminant concentration and moisture data will be evaluated.

Paul Parmentier
June 10, 2015
Page 3 of 3

Please do not hesitate to contact me directly with any questions or comments at our Pleasant Hill office at (925) 951-6453.

Sincerely,

F4 Remediation, Inc.

A handwritten signature in black ink, appearing to read "Kent R. Reynolds". The signature is fluid and cursive, with the first name "Kent" being the most prominent.

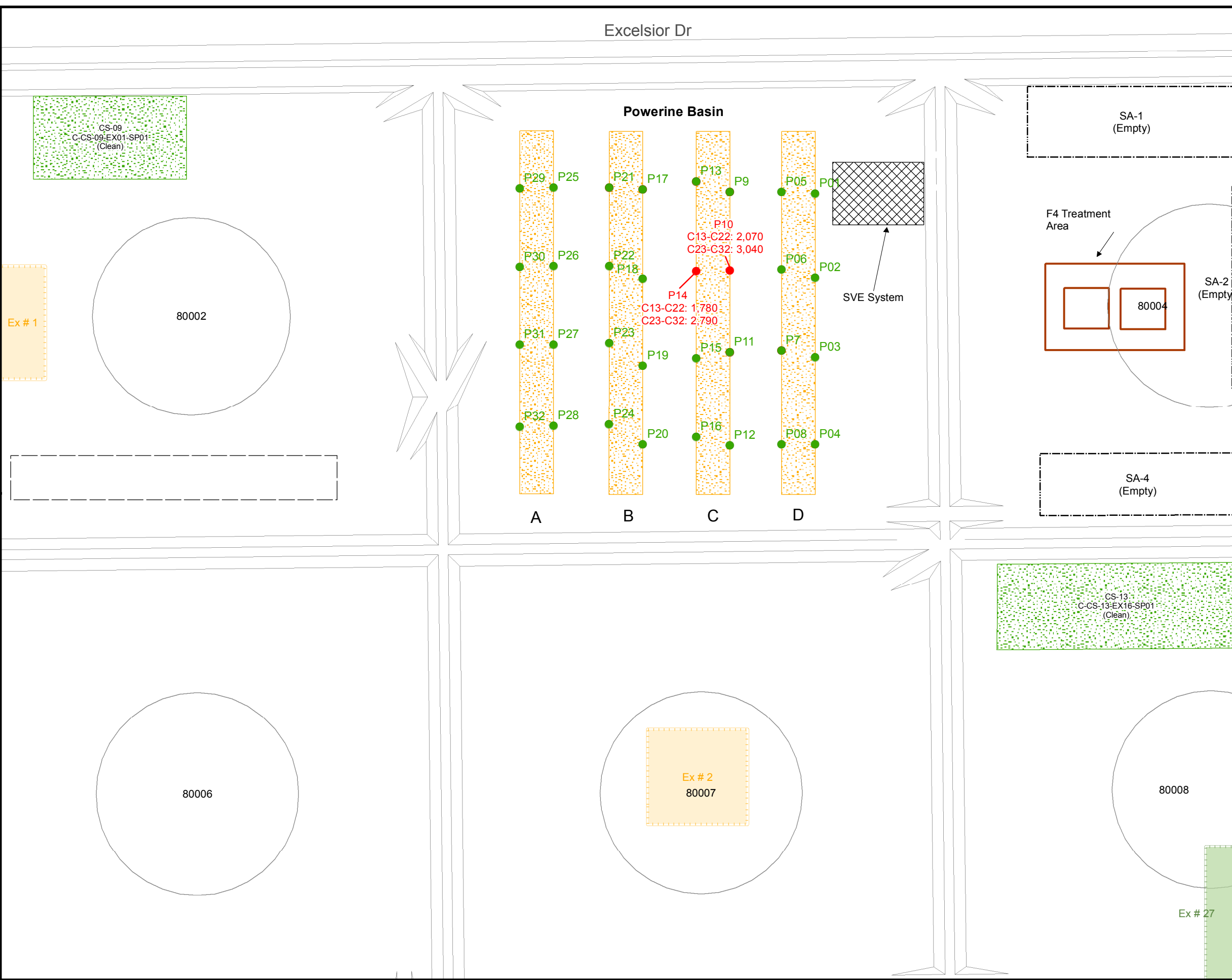
Kent R. Reynolds
Vice President/Operations Manager

Attachments: Moisture Summary
Performance Map
Volume of Existing Stockpiles – Powerine Basin

Description	Date
Excavation Started	3/16/15
Soil Treatment Started	3/31/15
Treatment Row D Completion	4/3/15
Treatment Row C Completion	4/8/15
Treatment Row B Completion	4/14/15
Treatment Row A Completion	4/17/15

Description	Date	Powerine-A- SP01	Powerine-B- SP01	Powerine-C- SP01	Powerine-D- SP01	Total
Volume of Soil (cubic yards)		984	918	855	815	3572
Average Tensiometer Reading (centibars)	4/24/15	7.5	7.5	12.9	14.3	
Average Moisture Measurement (Lab)	5/12/15	4.90%	6.70%	5.50%	6.39%	
Percentage of Soil Meeting Cleanup Goals	4/30/15	100%	100%	75%	100%	

Excelsior Dr



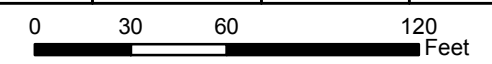
Legend

- Performance Sampling Concentration for TPH ≥ Cleanup Goal (mg/kg)
- Performance Sampling Concentration for TPH < Cleanup Goal (mg/kg)
- Former Above Ground Storage Tanks
- EX # 2 Completed Excavations 0-5ft bgs (as of 5/15/2015)
- SVE System
- Existing Treatment Pile
- CS-13 Clean Soil Pile
- SA-1 Staging Area - Empty
- E Treatment Row Under Construction



DFSP Norwalk
15306 Norwalk Boulevard
Norwalk, California

Project Number:	Date:	Drawn By:	Approved By:
04-NDLA-007	5/26/2015	P. WU	K. Wall



Performance Map

SGI environmental
THE SOURCE GROUP, INC.
1962 Freeman Avenue
Signal Hill, CA 90755
(562) 597-1055

Figure
2

Volume of Existing Stockpiles

date of field topo: June 04, 2015

DATUM

HORIZONTAL :

North American Datum of 1983 , (NAD'83)
CCS'83, ZONE V, (0405)

VERTICAL :

North American Vertical Datum of 1988 , (NAVD'88)
Los Angeles County Dept. of Public Works

Site Volume Table: Unadjusted

Site	Stratum	Surf1	Surf2	Cut yards	Fill yards	Net yards	Method
Powerine-A-01							
	powerine-a-01	powerine-a-01_ex-surface	powerine-a-01_stockpile	0	983	983 (F)	Grid
				0	984	984 (F)	Composite
Powerine-B-01							
	powerine-b-01	powerine-b-01_ex-surface	powerine-b-01_stockpile	0	917	917 (F)	Grid
				0	918	918 (F)	Composite
Powerine-C-01							
	powerine-c-01	powerine-c-01_ex-surface	powerine-c-01_stockpile	0	855	855 (F)	Grid
				0	855	855 (F)	Composite
Powerine-D-01							
	powerine-d-01	powerine-d-01_ex-surface	powerine-d-01_stockpile	0	814	814 (F)	Grid
				0	815	815 (F)	Composite



GRAPHIC SCALE



(IN FEET)
1 inch = 30 ft.



STEPHEN E. EVANS, PLS 7017

THIS DRAWING WAS PREPARED UNDER MY SUPERVISION
USING DATA FROM FIELD SURVEYS PERFORMED BY
ELS&M AND DATA PROVIDED BY THE SOURCE GROUP, INC.

DRAWN BY: S.E. APPROVED BY: S.EVANS DATE: 6/05/2015 DWG. NO.: DFSP-NORWALK.DWG	REVISIONS: INITIAL SITE VISIT, 6/04/2015	PREPARED FOR: The Source Group, Inc. 1962 Freeman Avenue Signal Hill, CA 90755	PROJECT: DFSP - Norwalk 15306 Norwalk Blvd. Norwalk, CA 90650	PREPARED BY: EVANS LAND SURVEYING and MAPPING 3436 Paloma Avenue La Verne, CA 91750 ph. (909) 592-5501	SHT. NO. 1 OF 1
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