

ATTACHMENT D.1

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Project Organization Chart

ATTACHMENT D.2

A T T A C H M E N T D.2

DRILLING AND BACKFILLING OF SOIL BORINGS PROTOCOL

1.0 INTRODUCTION

This protocol describes the procedures to be followed during drilling and backfilling of SVE wells and permanent soil vapor probes. SVE wells and permanent soil vapor probes will be installed in accordance with the protocols described in INSTALLATION OF SVE WELLS (Attachment D.3) and INSTALLATION OF PERMANENT SOIL VAPOR PROBES (Attachment D.4)

The procedures presented herein are intended to be of general use and may be supplemented by a work plan and/or health and safety plan. As the work progresses and if warranted, appropriate revisions may be made by the project manager. Detailed procedures in this protocol may be superseded by applicable regulatory requirements.

A DAILY FIELD RECORD will be completed for each day of fieldwork, and the original will be kept in the project files. Where required, soil boring permits will be acquired from the appropriate agency or agencies before drilling is initiated. At each boring location an underground utility check will be conducted before drilling begins. Underground utility checks will, at a minimum, consist of contacting Underground Service Alert.

2.0 DRILLING

Soil borings will be drilled using hollow stem augers technique. Hollow stem auger drilling technology generally does not require the use of drilling fluid. It employs flighted tubing and rotation to advance through the formation and remove drill cuttings. The hollow tubing maintains the integrity of the bore hole and facilitates soil sampling and well installation. In general, a helical or spiral tool form is used to move material from the subsurface to the surface, a bit at the bottom cuts into the subsurface material and spiral augers on outside convey the material to the surface while spinning. The center of auger is hollow like a straw when the inner drive rods and plug are removed. The hollow augers hold the borehole open for ground water sampling and well installation. During drilling with the hollow stem auger, the drill cuttings will be discharged through the open hole; the sediment will be shoveled and transferred into appropriate soil waste bins for transport and disposal.

The planned depth of each soil boring will be determined by the project manager before drilling. The field geologist/engineer will specify to the drill rig operator the desired total depth of the boring, the depth of soil sample collection, method of sample retrieval, and other matters pertaining to the satisfactory completion of the borings. Drill cuttings and drilling fluids generated during drilling of soil borings will be stored properly for future disposal by the client, unless other arrangements have been made.

The drill rods, drill pipe, hoses, bits, and other components that fluids and cuttings contact will be steam-cleaned before drilling at each boring location. Only potable water from a municipal

supply will be used for decontamination of drilling equipment. Decontamination rinseate will be collected and stored properly for future disposal by the client, unless other arrangements have been made.

3.0 BACKFILLING OF SOIL BORINGS

Soil borings that are not completed as SVE wells or vapor probes will be backfilled by grouting the borings with a neat cement grout, cement/sand grout, or cement/bentonite grout, or bentonite grout. ENVIRON field staff will calculate the borehole volume and compare it to the volume of grout used to evaluate whether bridging has occurred. These calculations and the actual volume emplaced will be noted on the BORING LOG. The grout will be placed in continuous lifts from the bottom of the boring to the ground surface. Additional grout will be added to the soil boring if significant settlement has occurred after the grout has set.

Forms Used: Field Investigation Daily Field Log
Field Soil Boring Log

P:\W\Wyle Labs\Norco\Presumptive RAW\Appendices\Appendix D\Attachments\Attachment D.2 Drilling and backfill\Drilling and Backfilling of Soil Borings Protocol.doc

FIELD SOIL BORING LOG

PROJECT NAME: _____ FIELD PERSON: _____
 PROJECT NUMBER: _____ PROJECT MANAGER: _____
 PROJECT LOCATION: _____ DATE: _____

BORING LOCATION MAP 	SOIL BORING NUMBER:
	DRILLING CONTRACTOR:
	DRILLER:
	RIG TYPE:
	OTHER EQUIPMENT:
	SAMPLING METHODS:
	HAMMER WEIGHT: DROP:
	TOTAL DEPTH: BOREHOLE DIAMETER:
	START TIME: STOP TIME: (DATE IF NEC.)
	BACKFILL TIME: DATE: BY:

SAMPLE DEPTH	SAMPLER TYPE	BLOWS IN 6 INCHES	FEET DRIVEN	FEET RECOVERED	PID/FID (TOVs)	SAMPLE NUMBER	TIME	DEPTH IN FEET	USGS CODE/ CONTACT	DEPTH IN FEET	COMMENTS
											SAMPLE DESCRIPTION

ATTACHMENT D.3

A T T A C M E N T D.3

SOIL VAPOR EXTRACTION WELL INSTALLATION PROTOCOL

1.0 INTRODUCTION

This protocol describes the procedures to be followed during installation of the soil vapor extraction (SVE) wells that are proposed to be installed at the Site. Drilling of the soil borings for the well installations will be performed in accordance with the protocol described in the DRILLING AND BACKFILLING OF SOIL BORINGS (Attachment D.2) attachment to this document. The procedures presented herein are intended to be of general use and may be supplemented by a work plan and/or health and safety plan. As the work progresses and if warranted, appropriate revisions may be made by the project manager. Detailed procedures in this protocol may be superseded by applicable regulatory requirements.

2.0 SVE WELL INSTALLATION

A DAILY FIELD RECORD will be completed for each day of fieldwork, and the original will be kept in the project files. At each intended well location an underground utility check will be conducted before drilling begins. Underground utility checks will, at a minimum, consist of contacting Underground Service Alert.

SVE wells will be installed to 10 feet below ground surface (bgs) at the Site. Screen intervals for each well will be from 5 to 10 feet bgs, and may be slightly modified based on field conditions by the field geologist/engineer after consultation with an ENVIRON California Registered Geologist (RG) or State-licensed professional engineer (PE). Construction of all monitoring wells will be in conformance with the following provisions. A TYPICAL SVE WELL CONSTRUCTION DIAGRAM is attached (Figure D.4).

2.1 Well Screen And Casing

Well casings will consist of clean, factory new, 2-inch diameter, flush-threaded schedule 40 polyvinyl chloride (PVC) casing. Well screens will consist of 2-inch diameter, flush-threaded schedule 40 PVC casing with factory-milled 0.020-inch slots, and will provide flow between the formation target zone and the well. The screened interval of each well will not exceed 5 feet (See Figure D.4). The base of each well screen will be plugged with a flush-threaded bottom cap. Blank PVC casing will be installed from ground surface to the top of the screen interval in each well.

2.2 Filter Material

Filter material will be well-graded, clean sand (generally less than 2 percent by weight passing a No. 200 sieve and less than 5 percent by weight of calcareous material). In this remedial action, clean No. 3 or No. 2/12 Monterey sand will be used for the filter pack.

2.3 Setting Screens and Riser Casing

Well casing materials will be measured to the nearest 0.1 foot and steam-cleaned before being lowered into the borehole. The well assembly will be designed so that the well screen is placed opposite the formation target zone. No PVC cement or other solvents will be used to fasten the well casing joints, well screen joints, or end caps.

The well casing will be placed in the center of the borehole and filter sand will be emplaced in a calculated quantity sufficient to fill the annular space from the bottom of the boring to a level of approximately 2 feet above the top of the well screen. The depth to the top of the filter pack will be verified by measuring, using a tremie pipe or a weighted tape. Sand will be added as required to bring the top of the sand surface to approximately 2 feet above the top of the well screen.

Once the depth to the top of the filter material has been verified, bentonite chips will be placed in the annular space above the sand filter pack as a transition seal between the filter material and the grout. A sufficient quantity of bentonite chips will be placed to fill the annular space to a level of about 2 feet above the top of the filter pack. Bentonite chips will be placed in approximately 6-inch lifts. Unless prohibited by well conditions, each lift should be hydrated using approximately 1 gallon of potable water per lift. The completed bentonite transition seal will be allowed to hydrate for at least 30 minutes prior to placing the grout. The depth to the top of the transition seal will be verified by measuring, using the tremie pipe or a weighted tape.

A neat cement/bentonite grout or bentonite grout will be installed from the top of the transition seal to the ground surface. Grout/additive/water mixtures will be determined on a site-specific basis. The typical neat cement/grout mixture consists of a mixture of one sack (94 pounds) of Portland Type I/II cement, approximately 2 to 5 percent by weight (of cement) powdered bentonite, and approximately 6 to 8 gallons of water. Only potable water will be used to prepare the grout. No work will be done on the monitoring well until after the grout has set at least 48 hours.

2.4 Surface Completion

Wells will be completed below grade and enclosed in a steel protective well cover (e.g., stovepipe) or a vault with a traffic-rated cover (if wells are on asphalt). All wells will be locked for security and will be designed to limit surface water infiltration.

2.5 Documentation

A well construction diagram for each well will be completed in the field on the WELL LOG by the field geologist/engineer and submitted to the reviewing geologist or engineer upon completion of each well. Well installation and construction data will be summarized on the DAILY FIELD RECORD or on a specialized form produced for this purpose. Following review by the project manager, the original records will be kept in the project file.

3.0 CLEANING OF DRILLING EQUIPMENT

Cleaning of the drill rig and associated drilling equipment will follow the procedures discussed in Section 2.0 of the protocol DRILLING AND BACKFILLING OF SOIL BORINGS (Attachment D.2).

All well casing materials will be cleaned before they are installed. Well development equipment will be cleaned before use. The following cleaning procedure has been found to be effective and will be used or adapted as appropriate for general conditions of materials or equipment to be cleaned.

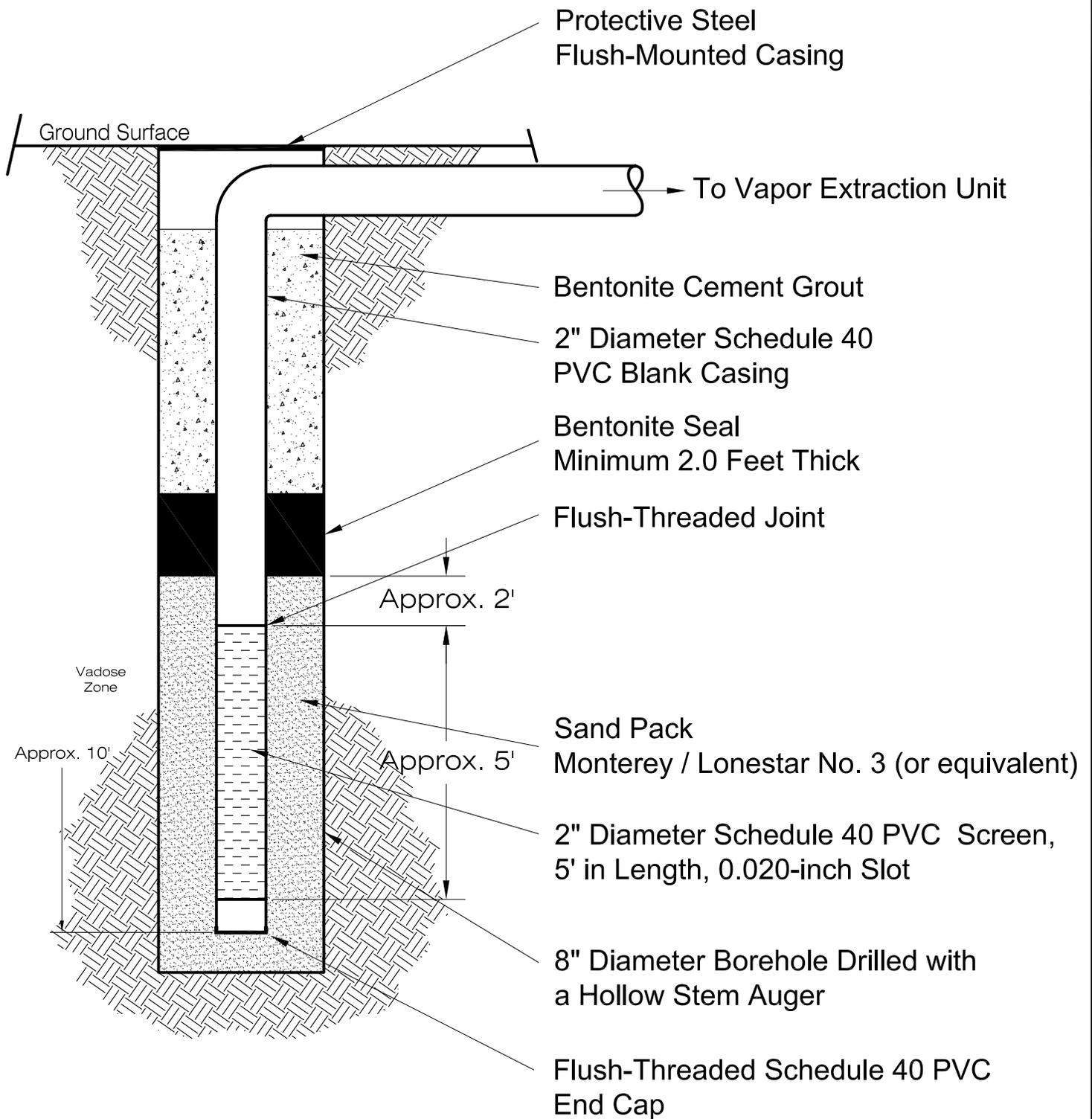
1. Steam-rinse with potable water or rinse in deionized or organic-free water.
2. Cover with clean plastic to protect materials and equipment from contact with chemical products, dust, or other contaminants.

Alternatively, well casing materials that have been steam-cleaned and sealed in individual airtight plastic bags by the factory can be used.

Decontamination rinsate will be collected and stored properly for future disposal by the client, unless other arrangements have been made.

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Attachments: Daily Field Record
Well Completion Form
Typical SVE Well Construction Diagram



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ENVIRON

Typical SVE Well Construction Diagram

Drafter: JJC

Date: 7/07/05

Contract Number: 04-8099V

Revised:

ATTACHMENT D.4

ATTACHMENT D.4

PERMANENT VAPOR PROBE INSTALLATION PROTOCOL

1.0 INTRODUCTION

This protocol describes the procedures to be followed during the installation of permanent vapor probes that will be installed as part of the Presumptive RAW at the Northwest Area. Drilling of the soil borings for the vapor probe installation will be performed in accordance with the protocol described in the DRILLING AND DESTRUCTION OF SOIL BORINGS, Attachment D.2.

The procedures presented herein are intended to be of general use and may be supplemented by a work plan and/or health and safety plan. As the work progresses and if warranted, appropriate revisions may be made by the project manager. Detailed procedures in this protocol may be superseded by applicable regulatory requirements.

2.0 VAPOR PROBE INSTALLATION

A DAILY FIELD RECORD will be completed for each day of fieldwork, and the original will be kept in the project files. At each boring location an underground utility check will be conducted before drilling begins. Underground utility checks will, at a minimum, consist of contacting Underground Service Alert.

Vapor Probe: The shallower probe tips will be installed at 5 feet and the deeper probe tip will be installed 3 feet above the depth at which ground water is encountered (provided ground water is encountered at 13 or more feet below ground surface). If the depth to ground water exceeds 18 feet, probes will be installed at 5 and 15 feet bgs. Each tip will be attached to an 1/8-inch Nylaflo tubing. The deeper probe will be packed with approximately one foot of number 2/16 Lapis Lustre sand within the probe depth, and approximately one foot of hydrated granular bentonite will be placed above the sand pack. The soil boring will be backfilled with medium hydrated bentonite chips to approximately 6 1/2 feet bgs and overlain by one foot of hydrated granular bentonite. Sand pack, number 2/16 Lapis Lustre, will be placed up to 5 feet bgs where the tip of the shallower probe connected to an 1/8 inch Nylaflo tubing will be positioned. After placing the tip of the probe, the boring will be filled with additional sand pack up to 4 feet bgs where an additional foot of hydrated granular bentonite will be added from 4 to 3 feet bgs. The remainder of the boring will be filled with hydrated bentonite chips. The vapor probes will be completed below grade in water tight, traffic rated boxes. A TYPICAL DUAL-NESTED VAPOR PROBE CONSTRUCTION DIAGRAM is attached.

2.1 Surface Completion

Vapor probes will be completed either below or above grade depending on whether the vapor probe is located in asphalt or in the open field. The vapor probes will be enclosed in a steel protective well cover (e.g., stovepipe) or a vault with a traffic-rated cover (if vapor probes are on asphalt). All vapor probes will be locked for security and will be designed to limit surface water infiltration.

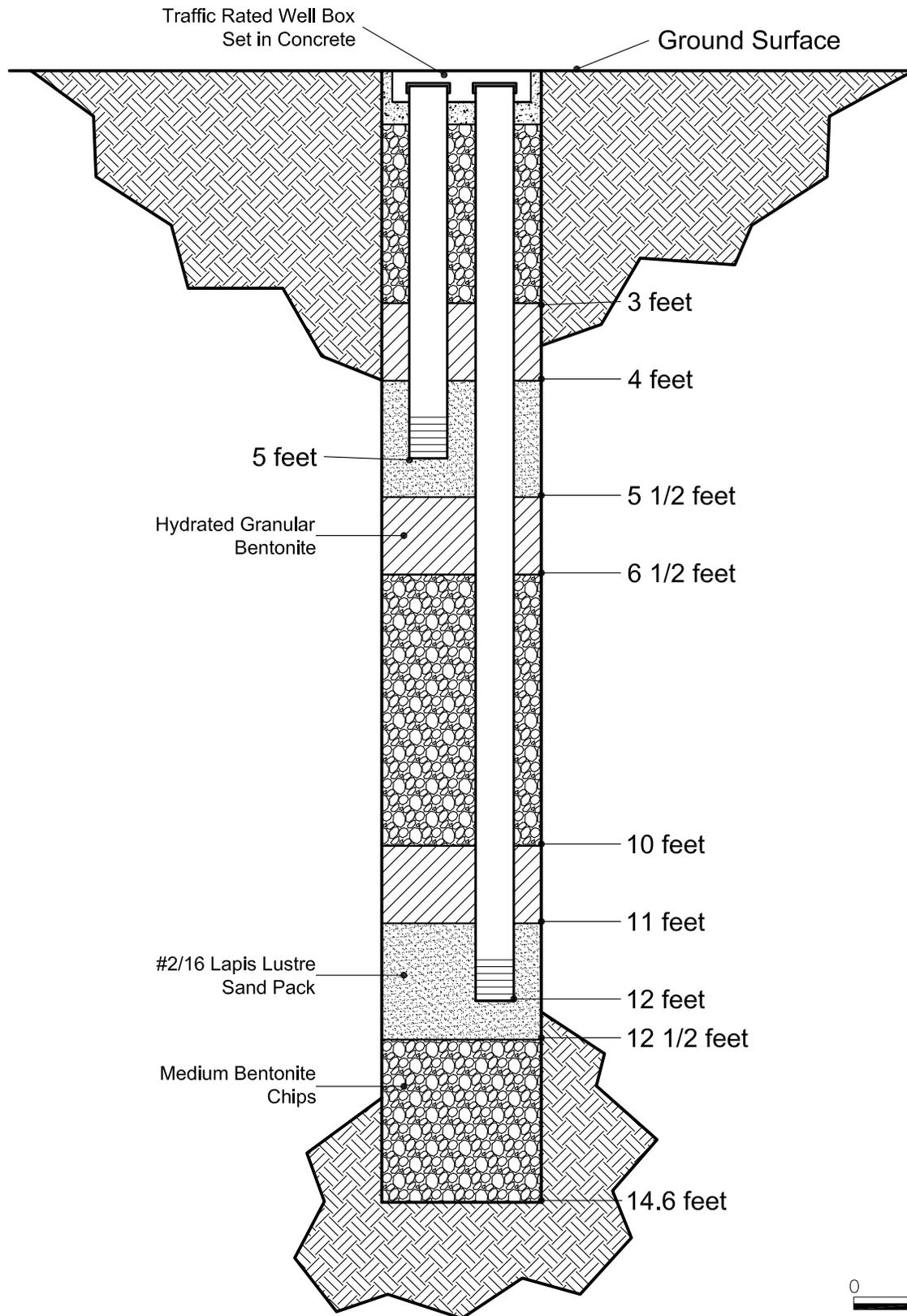
2.2 Documentation

Vapor probe installation will be summarized on the DAILY FIELD RECORD or on a specialized form produced for this purpose. Following review by the project manager, the original records will be kept in the project file.

3.0 CLEANING OF DRILLING EQUIPMENT

Cleaning of the drill rig and associated drilling equipment will follow the procedures discussed in Section 2.0 of the protocol DRILLING AND DESTRUCTION OF SOIL BORINGS.

Forms Used: Daily Field Record
Typical Nested Vapor Probe Diagram



FILE: P:\M\Wyle Labs\Norco\Figures\Remediation\8099SF\NestedVaporProbe

ENVIRON

Typical Nested Vapor Probe Construction Diagram
 Wyle Laboratories, Norco, California

Drafter: JJC

Date: 12/22/04

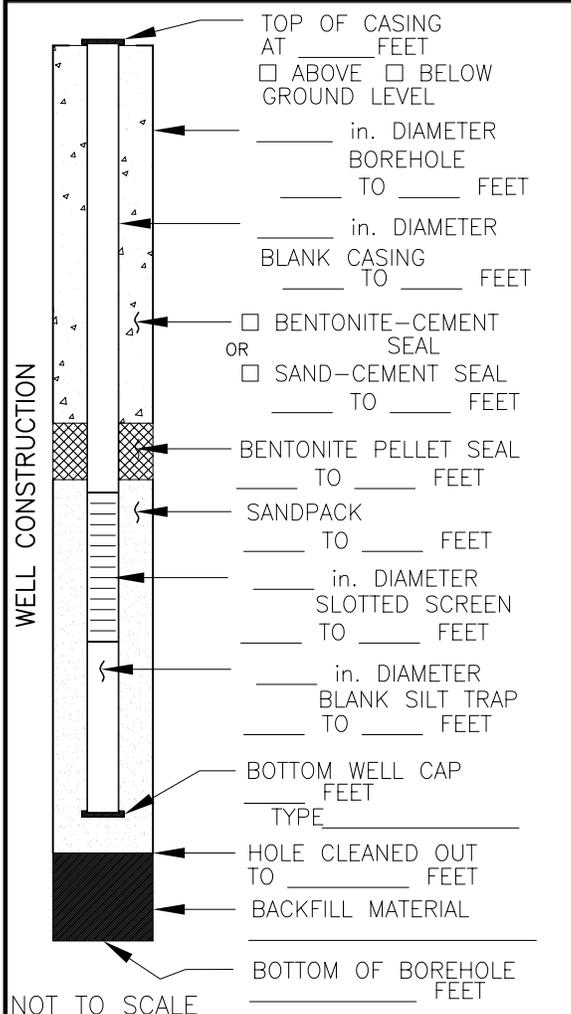
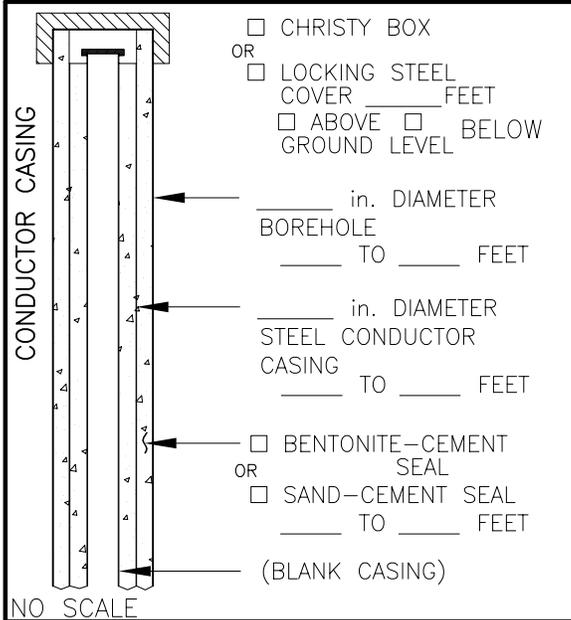
Contract Number: 04-8099S

Revised: 7/07/05

2010 Main St., Suite 900
Irvine, California 92614
(949) 261-5151
(949) 261-6202 (FAX)

FIELD WELL COMPLETION LOG

PROJECT NAME: _____ FIELD PERSON: _____
PROJECT NUMBER: _____ PROJECT MANAGER.: _____
PROJECT LOCATION: _____ DATE: _____



WELL NUMBER: _____
WELL LOCATION: _____
DRILLING COMPANY: _____
DRILLER: _____
DRILLING METHOD: _____
GALLONS OF WATER USED DURING DRILLING: _____
METHOD OF DECONTAMINATION: _____

WATER CONTAINMENT:

GROUND SURFACE TANK TRUCK
 STORM SEWER STORAGE TANK
 DRUM OTHER: _____

BEFORE DEVELOPMENT:

DEPTH TO WATER: _____ FEET BGS
DATE: _____ TIME: _____

MATERIALS USED:

LENGTH OF _____ IN CONDUCTOR CASING: _____ FEET
LENGTH OF _____ IN SLOTTED SCREEN: _____ FEET
MATERIAL: _____
SLOT SIZE: _____

LENGTH OF _____ IN BLANK CASING: _____ FEET
MATERIAL: _____

NUMBER OF (_____ LB) SACKS OF SAND: _____
SAND NAME/NUMBER: _____

POUNDS OF BENTONITE PELLETS: _____
PELLET SIZE: _____

NUMBER OF (_____ LB) SACKS OF CEMENT: _____
NUMBER OF (_____ LB) SACKS OF POWDERED BENTONITE: _____
AMOUNT OF GROUT: _____ (LBS)
AMOUNT OF REDIMIX CEMENT-SAND ORDERED: _____ (YD³)
AMOUNT OF REDIMIX CEMENT-SAND USED: _____ (YD³)
NUMBER OF (_____ LB) SACKS OF REDIMIX USED: _____
CONCRETE PUMP USED: _____

NOTES:

ATTACHMENT D.5

ATTACHMENT D.5 SOIL VAPOR SAMPLING PROTOCOL

1.0 INTRODUCTION

This protocol describes the procedures to be followed during sampling of soil vapor for laboratory chemical analysis. The laboratory must be California State-Certified by the appropriate regulating agency for the analyses to be performed.

The procedures presented herein are intended to be of general use and may be supplemented by a work plan and/or health and safety plan. As the work progresses, and if warranted, appropriate revisions may be made by the Project Manager or Project Engineer. Detailed procedures in this protocol may be superseded by applicable regulatory requirements.

2.0 ACTIVE SOIL VAPOR SAMPLING

2.1 Sample Collection

Purging Monitoring Well/Probe

The well or probe to be sampled will be purged before sampling in order to remove stagnant or ambient air, and therefore to obtain vapor that is representative of general subsurface conditions. The well or probe should be purged and sampled as follows:

- Connect the well or probe to be sampled to the extraction device and purge the tube. A site-specific purge volume versus contaminant concentration test will be conducted as the first soil gas sampling activity. This test will be performed at the test point where the contaminant concentrations are suspected to be the highest. The purge volume will be estimated based on summation of the volume of the sample container, internal volume of tubing used, and annular space around the probe tip. The step purge test of one, three, and seven purge volumes will be conducted as a means to determine the purge volume to be used at all sampling points. If no contaminants are detected during the step purge test, three purge volumes will be extracted prior to sampling at each location.

Leak Test

- A leak test will be conducted at every soil gas probe in order to prevent sample dilution with ambient air. A leak check compound will be placed where ambient air could enter the sampling system (sample system connection, surface bentonite seal, top of the temporary soil gas probe).

Purge/Sample Flow Rate

- Sampling and purging flow rate will be selected not to enhance compound partitioning during soil gas sampling. A vacuum device (gas tight syringe) will be used between the soil gas sample tubing and the soil gas extraction device (vacuum pump, Summa™ canister) to qualitatively determine if a high vacuum (no-flow or low-flow) soil condition is present.
- Purging/sampling will be conducted at low flow rates between 100 to 200 milliliters per minute (ml/min). The purge/sample rate may be modified based on conditions encountered in individual soil gas probes.

2.2 Sample Containers

- Soil gas samples will be collected in gas-tight, opaque/dark containers (e.g., syringes, glass bulbs wrapped in aluminum foil, Summa™ canisters). Tedlar™ bags will not be used to collect volatile organic compound (VOC) samples.
- If a syringe is used, it will be leak-checked before each use by closing the exit valve and attempting to force ambient air through the needle. If syringe samples are analyzed within five minutes of collection, aluminum foil wrapping will not be applied.
- If Summa™ canister is used, a flow regulator will be placed between the probe and the canister to ensure that the canister is filled at the low flow rate as specified above.

2.3 Sample Container Cleanliness and Decontamination

Prior to its use at a site, each sample container will be assured clean by the analytical laboratory as follows:

- New containers will be determined to be free of contaminants by the supplier and
- Reused/recycled containers: method blank(s), as specified below, should be used to verify sample container cleanliness.

After each use, reusable sample containers will be properly decontaminated, as follows:

- Glass syringes or bulbs will be disassembled and baked at 240° C for a minimum of 15 minutes.
- Summa™ canisters will be properly decontaminated as specified by appropriate EPA analytical method.
- Plastic syringes should be used only once.

A SOIL GAS SAMPLING LOG will be used to record the following information:

- Sample I.D.
- Duplicate I.D., if applicable
- Date and time sampled
- Name of sample collector
- Probe number
- Depth at which soil gas sample is collected
- Purge volume and purge rate
- Extraordinary circumstances (if any)
- Number and type of sample container(s)

3.0 SAMPLE LABELING

Sample containers will be labeled before or immediately after sampling with self-adhesive tags with the information written in waterproof ink:

- Company name
- Project name
- Project number
- Sample I.D. number
- Date and time sample was collected
- Initials of sample collector

4.0 FIELD QUALITY CONTROL SAMPLES

In order to evaluate the precision and accuracy of analytical data, quality control samples will be prepared as described below. These samples will be collected, or prepared and analyzed by the laboratory, as specified below.

A. Trip Blanks for Off-Site Shipments

If VOC samples are shipped offsite for analysis, a minimum of one trip blank per day will be collected and analyzed for the target compound. Trip blanks, consisting of laboratory grade ultra pure air, will be prepared to evaluate sample cross-contamination during shipment.

B. Duplicates

At least one duplicate sample per laboratory per day will be collected from areas of concern.

C. Method Blank

During sampling activities using reused/recycled sampling containers (e.g., glass syringes), at a minimum one decontaminated sample container per 20 samples or per every 12 hours, whichever is more often, should be used as a method blank to verify and evaluate the effectiveness of decontamination procedures and to detect any possible interference from ambient air.

5.0 HANDLING, STORAGE, AND TRANSPORTATION

Exposure to light, changes in temperature and pressure will accelerate sample degradation. To protect sample integrity the following steps will be undertaken:

- Soil gas samples will not be chilled;
- If condensation is observed in the sample container, the sample will be discarded and a new sample will be collected
- Soil gas samples will be analyzed within 30 minutes by an on-site mobile laboratory
- Soil gas samples collected in Summa™ canisters will be analyzed within 72 hours after collection

6.0 DOCUMENTATION

6.1 Field Data Sheets

A FIELD INVESTIGATION DAILY LOG will be completed for each day of fieldwork. Information recorded on the FIELD INVESTIGATION DAILY LOG will include a description of any deviations from the SAP that were necessitated by field conditions, such as equipment failure, wells that could not be sampled, etc. Sample numbers may also be recorded on the FIELD INVESTIGATION DAILY LOG as a means of identifying and tracking the samples. Following review by the project manager, the original records will be kept in the project file. Photographs may also be included in the project file, as appropriate.

6.2 Chain-of-Custody Procedures

After samples have been collected and labeled they will be maintained under chain-of-custody procedures. These procedures document the transfer of custody of samples from the field to the laboratory. Each sample sent to the laboratory for analysis will be recorded on a CHAIN-OF-CUSTODY, which will include instructions to the laboratory for analytical services and special turnaround times.

Information contained on the triplicate CHAIN-OF-CUSTODY RECORD will include:

- Project name
- Project number
- Signature of sampler(s)
- Date and time sampled
- Sample I.D.
- Number of sample containers
- Sample matrix (water)
- Analyses required
- Remarks, including preservatives, special conditions, or specific quality control measures
- Turnaround time and person to receive laboratory report
- Release signature of sampler(s), and signatures of all people assuming custody
- Condition of samples, including temperature, when received by laboratory

Blank spaces on the CHAIN-OF-CUSTODY will be crossed out and initialed by the sampler between the last sample listed and the signatures at the bottom of the sheet.

The field sampler will sign the CHAIN-OF-CUSTODY and will record the time and date at the time of transfer to the laboratory or to an intermediate person. A set of signatures is required for each relinquished/reserved transfer, including internal transfer. The original imprint of the chain-of-custody record will accompany the sample containers. A duplicate copy will be placed in the project file.

If the samples are to be shipped to the laboratory, the original CHAIN-OF-CUSTODY will be sealed inside a plastic bag within the ice chest, and the chest will be sealed with custody tape that has been signed and dated by the last person listed on the chain-of-custody. U.S. Department of Transportation shipping requirements will be followed and the sample shipping receipt will be retained in the project files as part of the permanent chain-of-custody document. The shipping company (e.g., Federal Express, UPS) will not sign the chain-of-custody forms as a receiver; instead the laboratory will sign as a receiver when the samples are received.

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Forms Used: Field Investigation Daily Log
Chain-of-Custody

2010 Main St., Suite 900 Irvine, Calif. 92614 (949) 261-5151 (949) 261-6202 (fax)

707 Wilshire Blvd., Suite 4950 Los Angeles, Calif. 90017 (213) 943-6300 (213) 943-6301 (fax)

MSA#: _____ WO#: _____

PROJECT NAME / FACILITY ID: _____

FIELD PERSON: _____

PROJECT NUMBER: _____ DATE: _____

PROJECT MANAGER: _____

PROJECT LOCATION: _____

LABORATORY: _____

IS THIS A UST PROJECT OR IS EDF REQUIRED? Y N IF YES, GLOBAL ID #: _____

SAMPLER: SIGNATURE:	YEAR	SAMPLE DATE	SAMPLE TIME	SAMPLE DEPTH	AIR SAMPLE VOLUME (L)	MATRIX (A) AIR (S) SOIL (G) GAS (W) WATER	NUMBER OF CONTAINERS	FILTERED/UNFILTERED (F/U)	PRESERVATION (SEE KEY)	ANALYSIS REQUIRED										COMMENTS							
SAMPLE I.D. NUMBER																											
TOTAL		X	X	X																							

RELINQUISHED BY: _____ TIME/DATE: _____	RECEIVED BY: _____ TIME/DATE: _____	TURNAROUND TIME (CIRCLE ONE)	SAMEDAY	72 HOURS
RELINQUISHED BY: _____ TIME/DATE: _____	RECEIVED BY: _____ TIME/DATE: _____		24 HOURS	5 DAYS
RELINQUISHED BY: _____ TIME/DATE: _____	RECEIVED BY: _____ TIME/DATE: _____		48 HOURS	NORMAL
		SAMPLE INTEGRITY	IF SEALED, SEAL INTEGRITY	
		INTACT: Y N Temp _____	INTACT: Y N	

H = HCL; N = HNO3; S = H2SO4; U = UNKNOWN; NO = NONE; O = OTHER

ATTACHMENT D.6

ATTACHMENT D.6

Data Quality Objective Planning Process

The data quality objective (DQO) process is a systematic planning process used to plan data collection activities to support decision-making. Through the DQO process, acceptance or performance criteria for the collection, evaluation, and use of environmental data are established. The DQOs for this project were developed according to USEPA's *Guidance for the Data Quality Objectives Process* (USEPA, 2000). The seven-step DQO decision-making process is presented below.

Step 1: State the Problem

Identify the planning team members – The members of the planning team are the DTSC Project Manager, and the ENVIRON project team, including the Project Manager, Project Engineer, Project Quality Assurance Officer, Task Leaders, and the Data Manager.

Identify the primary decision maker – The decisions will be made by consensus of the planning team members.

Describe the problem; develop a conceptual model of the environmental hazard to be investigated – In order to address mitigate VOC in soil gas at the southern terminus of Golden West Lane in the Northwest Area, Soil Vapor Extraction (SVE) will be used. Monitoring will include quarterly soil gas sampling to evaluate the effectiveness of the SVE system and to evaluate whether modifications are needed.

Specify available resources and relevant deadlines for the project – SVE is anticipated to be active for 12 months. Quarterly monitoring of soil gas will continue after this time period, in accordance with present sampling requirements.

Step 2: Identify the Decision

Identify the principal study question(s) – Will SVE provide adequate mitigation of VOC concentrations in soil gas during its 12 months of operation at the Northwest Area?

Define alternative actions that could result from resolution of the principal study question – Discontinue SVE and continue the soil gas monitoring programs.

Combine the principal study question and the alternative actions into a decision statement – Following 12 months of operation, the SVE system at the Northwest Area will be discontinued and quarterly monitoring soil gas monitoring will continue.

Organize multiple decisions – Based on the answer to the principal study question, decisions about further potential remedial actions will be made by the planning team. These decisions may be:

Is there a need to conduct additional remedial actions at the Northwest Area?

Step 3: Identify the Input to the Decision

Identify the information needed to resolve the decision statement – To resolve the decision statement, the planning team will evaluate soil gas generated during monthly sampling activities. The data also will be compared to the established performance criteria.

Determine sources for this information – Soil gas samples will be collected on a quarterly basis and analyzed for VOCs,.

Determine the basis for determining the Action Level – SVE is intended to provide immediate mitigation of TCE migration to residences through soil vapor at the southern terminus of Golden West Lane in the Northwest Area (see Presumptive RAW).

Identify sampling and analysis methods than can meet the data requirements – The analytical methods that will be used to analyze ground water samples are presented in the body of the QAPP.

Step 4: Define the Boundaries of the Study

Define the target population of interest – Existing soil gas data have been obtained during quarterly sampling events conducted previously at the Northwest Area. The target population of interest comprises the soil gas that will be collected and analyzed at the Northwest Area on a quarterly basis during the SVE period.

Specify the spatial boundaries that clarify what the data must represent -

Define the geographic area to which the decision statement applies – The Area Considered in the RAW is located at the southern terminus of Golden West Lane in a residential area of the City of Norco, California.

Divide the population into strata that have relatively homogeneous characteristics – The target population can be divided into three subpopulations based on the locations of the vapor probes in relation to the SVE well locations - upgradient, central, and downgradient wells and probes.

Determine the time frame for collecting data and making the decision – It is anticipated that soil gas will be conducted on a quarterly basis for approximately 12 months, during the Presumptive RAW implementation, consistent with the current sampling plan.

Determine the practical constraints on collecting data – None.

Define the scale of decision-making – Soil gas data collected during the sampling events will be compiled and assessed quarterly during the SVE period.

Step 5: Develop a Decision Rule

Specify the statistical parameter that characterizes the population of interest – The goal is to obtain soil gas data to assist in the evaluation of the effectiveness in mitigation of VOCs in soil gas.

Confirm the Action Level exceeds measurement detection limit – Analytical methods to be used specify detection limits that are lower than MCLs for these compounds

Develop a decision rule (if...then... statement) – If soil gas concentrations suggest mitigation of TCE in soil gas is not effective, then modify or enhance SVE system.

Step 6: Specify Tolerable Limits on Decision Error

Data generated during performance of the Presumptive RAW will be subjected to data validation procedures as specified in this QAPP. Data are determined to be valid if the specified limits on precision, accuracy, representativeness, comparability, and completeness are achieved.

Step 7: Optimize the Design for Obtaining Data

This QAPP has been designed to ensure that the type and quantity of data needed to satisfy each of the aforementioned objectives is achieved.

ATTACHMENT D.7

5. METHOD SUMMARY

- 5.1 EPA Methods TO-14A and 15 describe chromatographic procedures that will allow for the separation of volatile organic compounds and their qualitative and quantitative analysis by mass spectrometry. Detection is achieved using a mass selective detector. Method TO-15 is significant in that it extends Method TO-14A capabilities in the following ways.
 - 5.1.1 Method TO-15 requires the use of a GC/MS which results in more scientifically defensible data.
 - 5.1.2 Method TO-15 establishes method performance and quality control criteria for acceptance of data.
- 5.2 A known volume of sample is directed from the container (Summa® canister or Tedlar™ bag) through a solid multi-module (glass beads, tenex, cryofocuser) concentrator. A very minimal portion of the water vapor in the sample may break through the concentrator during sampling. Dry purging the concentrator with helium, while retaining target analytes can further reduce the water content of the sample. Post concentration, the VOCs are thermally desorbed onto a gas chromatographic column for separation. Detection is by mass selective detector.

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SPECTROMETRY (GC/MS)
EPA METHOD 8260B**

SOP Number: 8260B.SOP
Revision: Rev. 7

Effective Date: 3/13/01
Supersedes: Rev 6 (09/14/99)

1. SCOPE AND APPLICATION

- 1.1. Method 8260 is used to determine volatile organic compounds in a variety of matrices, including but not exclusively: ground and surface water, aqueous sludge, waste solvents, oily wastes, various air sampling trapping media, tars, fibrous wastes, filter cakes, spent carbons, soils, and sediments.
- 1.2. Water samples (Method 5030B) and soils samples (Method 5030A or 5035) with low levels of target analytes are loaded onto a purge and trap autosampler. Samples containing high levels of target analytes are diluted prior to being loaded onto an autosampler. High level soil samples are extracted (Method 5035) and diluted with Methanol (MeOH) and water samples are diluted with ultrapure water. The samples are purged with Helium, which extracts the target compounds into a vapor phase. The vapor concentrates onto the trap which is then heated to release the target compounds into the gas chromatograph column.
- 1.3. The capillary gas chromatography column is interfaced to a mass spectrometer. Analytes are identified by comparing their measured mass spectra and retention times to the calibration standards' reference spectra and retention times. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using at least a five-point calibration curve.

2. SUMMARY

- 2.1. The volatile compounds are introduced into the gas chromatograph by the purge and trap method. The analytes are introduced to a capillary column which is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer.

3. STANDARDS AND REAGENTS

- 3.1. The vendors indicated below currently supply the following standards and reagents. In the instance where a different vendor might be used, an equivalent product will be purchased. As long as the item is documented clearly in a logbook or on the raw data, this SOP will remain valid though the vendor may have changed.
- 3.2. Methanol (MeOH) – purge and trap grade (Burdick and Jackson or equiv).
- 3.3. Absolute Standard Gases Mix #1 (2000 µg/ml)
- 3.4. Absolute Standard Volatile Compound Mix (2000µg/ml)
- 3.5. Ultrascientific DBVM-580 VOC mixture (200 µg/ml) with Restek MTBE (2000 µg/ml) added to make the LCS and MS/MSD spiking solution
- 3.6. Absolute Internal Standard (2000 µg/ml)
- 3.7. Absolute Surrogate Standard (2000 µg/ml)
- 3.8. Absolute MTBE (1000 µg/ml)

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- 3.9. Absolute Standard Ketone Mix (2000 µg/ml) (two separate lot#s)
- 3.10. Absolute 4-Bromofluorobenzene (2500 µg/ml)
- 3.11. Absolute 2-CEVE (1000 µg/ml and 2000 µg/ml)
- 3.12. Absolute Vinyl Acetate (2000 µg/ml)
- 3.13. Absolute Carbon Disulfide (1000 µg/ml)
- 3.14. Absolute Oxygenates Initial Calibration Standard, custom mix (2500 µg/ml)
- 3.15. Absolute gasoline additive mix (2000 µg/ml)
- 3.16. Absolute Tert-butyl alcohol (1000 µg/ml and 20,000 µg/ml)
- 3.17. Absolute TBA-d9 Standard (1000ug/L and 4000 µg/ml)
- 3.18. Accustandard Oxygenates Mix for the LCS (1000 µg/ml)
- 3.19. Restek Tert-butyl alcohol for the LCS (2000 µg/ml)
- 3.20. Ultrapure water
- 3.21. Absolute Ethanol (20,000 µg/ml)

4. APPARATUS AND MATERIALS

- 4.1. 1-ml vials with polytetrafluoroethylene (PTFE)-lined screw caps
- 4.2. Teflon tape
- 4.3. Fume hood
- 4.4. 10 µl, 100 µl, and 250 µl micro syringes
- 4.5. 1-ml and 10-ml syringes
- 4.6. 1-ml vials with mininert screw caps
- 4.7. Beakers
- 4.8. Glass test tubes, disposable and reusable
- 4.9. Sparge tube wrench
- 4.10. pH Test Strips
- 4.11. Encore™ sample extrusion tool

5. DEFINITIONS

- 5.1. No specific definitions are associated with this SOP. See QAPM and EPA 8000B, and EPA 8260B for general definitions.

6. SAFETY

- 6.1. Since all of the hazards of samples and chemicals used in this procedure are not entirely known, strict adherence to safety rules and use of prescribed personal protection equipment is mandatory. The health hazards of the standards, reagents and samples are not entirely known so caution must be exercised in all cases.
- 6.2. Employees performing this procedure must be familiar with the Chemical Hygiene Plan (CHP), and the precautions stated on the appropriate Material Safety Data Sheets (MSDS).

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6.3. Personal Protective Equipment Required: Safety Glasses, Gloves

7. INTERFERENCES

7.1. Contamination may occur when a low concentration sample is analyzed immediately after a high concentration sample.

7.1.1. If a port is contaminated by a sample, rinse the purge needles and purge valves two to three times with methanol. After rinsing with methanol, analyze a blank. If the target compounds are not present in the blank then analysis may continue.

7.1.2. If a low concentration sample is analyzed immediately after a high concentration sample, reanalysis of the low concentration sample is necessary if the concentrations are less than 10x the reporting limit. Reanalysis is not necessary if the low concentration sample is N.D. for all target analytes at their reporting limit.

7.1.3. Rinse the suspected contaminated port by analyzing a DI water blank in it. Identify the port with a tag. Continue the analysis of DI blanks in the port until all target compounds are shown to be Non Detect.

7.2. Contamination may also occur when a sample contains surfactants. Signs of surfactant are foaming and/or bubbling when the sample is purged. After a sample, which contains surfactants, is analyzed, rinse the system carefully (7.1). Look carefully for signs of carry over in the samples that are analyzed immediately after the surfactant sample and in the same port the next time it is used.

7.3. The sample storage area must be free of organic solvent vapors. This is verified weekly with the use of refrigerator blanks analyzed by GCMS.

8. SAMPLE HANDLING AND PRESERVATION

8.1. The holding time for water samples is fourteen (14) days from the date of collection. All samples must be preserved with HCl to a pH < 2.

8.1.1. Using pH test strips, measure the pH of the sample after the sample aliquot is removed for analysis to ensure it was properly preserved.

8.1.2. Document the pH on the run log. If the pH >2, document on the run log that the sample was analyzed though improperly preserved. Flag the results in ELMNT with a 'P' qualifier and notify the Project Manager.

8.2. For EPA Methods 5030A, the holding time for solid samples is fourteen (14) days from the date of collection except under the Well Investigation Program (7 days).

8.3. EPA Method 5035, when using Encore sampler, states that soil samples must be either analyzed or extracted within 48 hrs. The holding time may be extended to 7 days by storing the samples in a freezer until the time of analysis.

8.4. For preservation, all samples must be stored at 4°C ± 2°C prior to analysis.

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- 8.5. Water samples are submitted in 40 ml glass VOA vials with Teflon lined silicon septa screw caps in duplicate or triplicate. They should not have any air bubbles present in the vials when they are inverted. Flag the results with an 'HS' qualifier if any headspace is present and notify the project manager. Solid samples can be submitted in brass or stainless steel boring tubes, or zero-headspace sampling devices such as Encore™ sampling devices.
- 8.6. The holding time for air samples is three days (72 hours) from the time of sample collection.
 - 8.6.1. The sample must be loaded on the autosampler within 72 hours of sampling. (The sample does not necessarily have to be analyzed within 72 hours of sample collection.)
 - 8.6.2. There is no preservation of air samples.
- 8.7. Notify the project manager immediately if the method holding time has been exceeded.

9. PROCEDURE

- 9.1. Standard Preparation
 - 9.1.1. Initial calibrations are performed as needed. All daughter solutions of the non-gases expire with the parent or at one month. Whichever comes first. Standards for the permanent gases are monitored frequently by comparison to the initial calibration curve. Fresh standards are prepared if this check exceeds 20% drift.
 - 9.1.2. Prepare the working standards, LCS standard, and the calibration standards before initial calibration. Initial calibrations are performed as needed. Prepare the internal standard/surrogate solution every two weeks, or as needed. Also refer to the SOP "Reagent and Standard Control and Documentation".
 - 9.1.3. Demonstrate that every new lot of MeOH (new lot number), received from the supplier, is free of analytes by analyzing a blank of MeOH.
 - 9.1.4. Transfer the Absolute standards (2000 µg/ml) from their 1-ml ampule to 1-ml vials with PTFE-lined screw caps.
 - 9.1.4.1. Seal the vials with Teflon tape.
 - 9.1.4.2. Store the stock standards in the freezer for no longer than six months.
 - 9.1.4.3. Replace the standards sooner if a change in response is observed.
 - 9.1.4.4. Prepare the LCS standards in the same manner.
 - 9.1.5. Work under a hood when handling stock standards containing high concentrations of toxic analytes. Note that the following compounds are classified as known or suspected carcinogens and should be treated as potential health hazards. Keep exposure to these chemicals to a minimum:

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- 9.1.5.1. Benzene
- 9.1.5.2. Carbon tetrachloride
- 9.1.5.3. 1,4-Dichlorobenzene
- 9.1.5.4. 1,2-Dichloroethane
- 9.1.5.5. Hexachlorobutadiene
- 9.1.5.6. 1,1,2,2-Tetrachloroethane
- 9.1.5.7. 1,1,2-Trichloroethene
- 9.1.5.8. Chloroform
- 9.1.5.9. 1,2-Dibromoethane
- 9.1.5.10. Tetrachloroethene,
- 9.1.5.11. Trichloroethene
- 9.1.5.12. Vinyl chloride.
- 9.1.6. Treat all compounds, not just carcinogens, as potential health hazards.
- 9.1.7. Prepare the internal standard/surrogate solution
 - 9.1.7.1. Add 850 μ l of MeOH with a 1-ml syringe into a 1-ml mininert vial.
 - 9.1.7.2. Add 25 μ l of the Absolute Internal Standard (2000 μ g/ml) and 25 μ l of the Absolute Surrogate Standard (2000 μ g/ml) to the 1-ml mininert vial to attain a final concentration of 50 μ g/ml for the internal standard/surrogate solution. Also, add 0.1 mL of the absolute standard TBA-d9 (4000 ug/mL) to the 1 ml mininert vial for a final concentration of 400 ug/mL TBA-d9.
 - 9.1.7.3. Cap the 1-ml vial with a mininert screw cap and seal it with Teflon tape. Store the vial in the freezer with minimal headspace.
 - 9.1.7.4. Enter the standard information into ELMNT and print out a hard copy. ELMNT will create a unique ID. Have the information reviewed and signed. Put in the standards notebook and store the solution in the freezer.
- 9.1.8. Prepare the internal standard (without surrogate)
 - 9.1.8.1. Add 875 μ l of MeOH with a 1-ml syringe into a 1-ml mininert vial.
 - 9.1.8.2. Add 25 μ l of the Absolute Internal Standard (2000 μ g/ml) to the 1-ml mininert vial to attain a final concentration of 50 μ g/ml for the internal standard solution. Also, add 0.1 mL of the absolute standard TBA-d9 (4000 ug/mL) to the 1 mL mininert vial for a final concentration of 400 ug/mL TBA-d9.

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- 9.1.8.3. Cap the 1-ml vial with a mininert screw cap and seal it with Teflon tape. Store the vial in the freezer with minimal headspace.
- 9.1.8.4. Enter the standard information into ELMNT. See 9.1.7.4.
- 9.1.8.5. The Absolute Internal Standard (2000 µg/ml) contains the internal standards, Chlorobenzene-D₅, 1,4-Difluorobenzene, 1,4-Dichlorobenzene-D₄, and Pentafluorobenzene. The Absolute Surrogate Standard (2000 µg/ml) contains the surrogates, 4-Bromofluorobenzene, Dibromofluoromethane and Toluene-D₈.
- 9.1.9. Prepare the surrogate standard at 50 µg/ml
- 9.1.9.1. Add 975µl of MeOH with a 1-ml syringe into a 1-ml mininert vial.
- 9.1.9.2. Add 25 µl of the Absolute Surrogate Standard (2000 µg/ml) to the 1-ml mininert vial, using a 100 µl micro syringe to attain a final concentration of 50 µg/ml for the surrogate standard solution.
- 9.1.9.3. Cap the 1-ml vial with a mininert screw cap and seal it with Teflon tape. Store the vial in the freezer with minimal headspace.
- 9.1.9.4. Enter the standard information into ELMNT. See 9.1.7.4.
- 9.1.10. Prepare the 4-BFB Tuning Solution at 25 µg/ml
- 9.1.10.1. Add 990µl of MeOH with a 1-ml syringe into a 1-ml mininert vial.
- 9.1.10.2. Add 10 µl of the Absolute 4-Bromofluorobenzene Standard (2500 µg/ml) to the 1-ml mininert vial, using a 10 µl micro syringe to attain a final concentration of 25 µg/ml for the tuning standard solution.
- 9.1.10.3. Cap the 1-ml vial with a mininert screw cap and seal it with Teflon tape. Store the vial in the freezer with minimal headspace.
- 9.1.10.4. Enter the standard information into ELMNT. See 9.1.7.4.
- 9.1.11. Prepare the surrogate standard at 200 µg/ml
- 9.1.11.1. Add 900µl of MeOH with a 1-ml syringe into a 1-ml mininert vial.
- 9.1.11.2. Add 100 µl of the Absolute Surrogate Standard (2000 µg/ml) to the 1-ml mininert vial, using a 100 µl micro syringe to attain a final concentration of 200 µg/ml for the surrogate standard solution.

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- 9.1.11.3. Cap the 1-ml vial with a mininert screw cap and seal it with Teflon tape. Store the vial in the freezer with minimal headspace.
- 9.1.11.4. Enter the standard information into ELMNT. See 9.1.7.4.
- 9.1.12. Prepare the 50 µg/ml working standard
 - 9.1.12.1. Using a 1ml syringe, add 780 µl of MeOH into a 1 ml mininert vial.
 - 9.1.12.2. Add 25 µl of the Absolute Standard Gases Mix #1 (2000 µg/ml) standard, 25 µl of the Absolute Standard Volatile Compounds Mix (2000 µg/ml), 25 µl of the Absolute Ketone mix (2000 µg/ml), 25µl of the 2-CEVE standard (2000 µg/ml), 20 uL of the absolute standards oxygenated gas mix (2500 ug/mL), 25 uL of the absolute standards gasoline additive mix (2000 ug/mL), 25 µl of the Vinyl Acetate standard (2000 µg/ml), and 50 µl of the Carbon Disulfide standard (1000µg/ml) to the 1-ml mininert vial to attain a final concentration of 50 µg/ml.
 - 9.1.12.3. Use this working standard for the calibration solutions.
 - 9.1.12.4. Cap the 1-ml vial with a mininert screw cap and seal it with Teflon tape. Store the vial in the freezer with minimal headspace.
 - 9.1.12.5. Enter the standard information into ELMNT. See 9.1.7.4.
- 9.1.13. Prepare the TBA/Ethanol working standard (low level).
 - 9.1.13.1. Using a 1mL syringe, add 725uL of MeOH into a 1mL mininert vial.
 - 9.1.13.2. Add 250uL TBA(1000ug/mL) and 25uL Ethanol (20,000 ug/mL) to the 1mL mininert vial to attain a final concentration of 250 ug/mL TBA and 500 ug/mL EtOH.
 - 9.1.13.3. Use this working standard for the calibration solutions.
 - 9.1.13.4. Cap the 1mL vial with a mininert screw cap and seal it with Teflon tape. Store the vial in the freezer with minimal headspace.
 - 9.1.13.5. Enter the standard information into ELMNT. See 9.1.7.4.
- 9.1.14. Prepare the TBA/ethanol high level working standard.
 - 9.1.14.1. Using a 1 mL syringe, add 850 uL of MeOH into a 1mL mininert vial.
 - 9.1.14.2. Add 100 uL of absolute standard ethanol (20,000 ug/mL) and 50 uL of absolute standard TBA (20,000 ug/mL) to attain a final concentration of 2000 ug/mL ethanol and 1000 ug/mL TBA.
 - 9.1.14.3. Use this working standard for the calibration solution.

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- 9.1.14.4 Cap the 1 mL vial with a mininert screw cap and seal it with teflon tape. Store the vial in the freezer with minimal headspace.
- 9.1.14.5 Enter the standard information into ELMNT. See 9.1.7.4.
- 9.1.15. Prepare the 5 µg/ml working standard
- 9.1.15.1. Add 950 µl of MeOH with a 1-ml syringe into a 1-ml mininert vial.
- 9.1.15.2. Add 25 µl of the 200 µg/ml working standard and 25 uL of the high level TBA/EtOH solution to the 1-ml mininert vial to attain a final concentration of 5 µg/ml for the 8260 and oxy compounds, 25 ug/ml for TBA, and 50 ug/ml for ethanol.
- 9.1.15.3. Use this working standard for the calibration solutions.
- 9.1.15.4. Cap the 1-ml vial with a mininert screw cap and seal it with Teflon tape. Store the vial in the freezer with minimal headspace.
- 9.1.15.5. Enter the standard information into ELMNT. See 9.1.7.4.
- 9.1.16. Prepare the 200 µg/ml working standard
- 9.1.16.1. Add 120 µl of MeOH with a syringe into a 1-ml mininert vial.
- 9.1.16.2. Add 100 µl of the Absolute Standard Gases Mix #1 (2000 µg/ml) standard, 100 µl of the Absolute Standard Volatile Compounds Mix (2000 µg/ml), 80 uL of the absolute standards oxygenates gas mix (2500 ug/ml), 100 ul gasoline additives mix (2000 ug/ml), 100 µl of the Absolute Ketone mix (2000 µg/ml), 100µl of the 2-CEVE standard (2000 µg/ml), 100 µl of the Vinyl Acetate standard (2000 µg/ml), and 200 µl of the Carbon Disulfide standard (1000µg/ml) to the 1-ml mininert vial to attain a final concentration of 200 µg/ml.
- 9.1.16.3. Use this working standard for the calibration solutions.
- 9.1.16.4. Cap the 1-ml vial with a mininert screw cap and seal it with Teflon tape. Store the vial in the freezer with minimal headspace.
- 9.1.16.5. Enter the standard information into ELMNT. See 9.1.7.4.
- 9.1.17. Prepare the Laboratory Control Sample (LCS) standard
- 9.1.17.1. Add 700 µl of MeOH with a 1-ml syringe into a 1-ml mininert vial.
- 9.1.17.2. Add 250 µl of the Ultrascientific DBVM-580 VOC mixture (200 µg/ml) standard, 25 µl of the Restek MTBE (2000 µg/ml) standard, and 25 µl of the Absolute Ketone Mix (2000 µg/ml) to the 1-ml mininert vial to attain a final concentration of 50 µg/ml.

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- 9.1.18. Prepare the Laboratory Control Sample (LCS) standard-TBA only
- 9.1.18.1 Add 875 uL of MeOH with a 1mL syringe into a 1-mL mininert vial.
- 9.1.18.2 Add 125 uL of 2000ug/mL TBA solution to the 1-mL miniert vial to attain a final concentration of 250ppm.
- 9.1.19. Prepare the Laboratory Control Sample (LCS) standard for methanol extractions with each extraction batch.
- 9.1.19.1 Add 775 ml of MeOH into a 40 ml VOA vial. And add the following.
- 9.1.19.2 Add 125 µl of the Ultrascientific DBVM-580 (200 µg/ml)mixture (See 3.5) - Final Concentration 2.5 µg/ml
- 9.1.19.3 Add 12.5 µl of the Restek MTBE standard (2000 µg/ml), (See 3.5) - Final Concentration 2.5 µg/ml
- 9.1.19.4 Add 12.5 µl of the Absolute Ketone Mix (2000 µg/ml) (See 3.9) - Final Concentration 2.5 µg/ml
- 9.1.19.5 Add 12.5 µl of the Accustandard Oxygenate Mix (1000 mg/ml) (See 3.17) Final Concentration 25 µg/ml
- 9.1.19.6 Add 62.5 µl of the Restek TBA standard (2000µg/ml) (See 3.18) - Final Concentration - 12.5 µg/ml.
- 9.1.19.7 Add 12.5 uL Absolute sturrogates mix (2000ug/mL).
- 9.1.19.8 Vortex as a sample.
- 9.1.20. Prepare the calibration standards as specified in the following table. Check the working standard frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

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9.1.21. 8260 Calibration with a 10-ml purge

Calib. Points	Internal Std./Surrogate Mix (50 µg/ml)		5 ppm Standard	
0.5 ppb	5 µl		1 µl	
1 ppb	5 µl		2 µl	
2 ppb	5 µl		4 µl	
	Internal Std (50 µg/ml)(400 ug/ml TBA-d9)	Surrogate (50 µg/ml)	50 ppm Standard	250/500ppm TBA/EtOH standard
5 ppb	5 µl	1 µl	1 µl	1 µl
10 ppb	5 µl	2 µl	2 µl	2 µl
25 ppb	5 µl	5 µl	5 µl	5 µl
	Internal Std (50 µg/ml)(400 ug/ml TBA-d9)	Surrogate (200 µg/ml)	200 ppm Standard	1000/2000 ppm TBA/EtOH standard
50 ppb	5 µl	2.5 µl	2.5 µl	2.5 µl
100 ppb	5 µl	5 µl	5 µl	5 µl
200 ppb	5 µl	10 µl	10 µl	10 µl

9.2. Initial Calibration

9.2.1. Perform a BFB tune on the mass spectrometer prior to performing the initial calibration.

9.2.1.1. Tune the mass spectrometer by directly injecting 50 ng (2 µl of the 25 µg/ml tuning solution) of BFB or evaluating Bromofluorobenzene from the daily check standard, or calibration standard.

9.2.1.1.1 Generally the direct inject is utilized unless the instrument is being recalibrated, then the 4-BFB from the first calibration standard is used for tuning.

9.2.1.2. The mass spectrometer must produce a mass spectrum that meets all the relative ion abundance criteria for BFB in Table 4 of the method. The mass spectrum may be acquired as follows (pre-set in the software):

9.2.1.2.1 Three scans are acquired and averaged (the peak apex scan and the scans immediately preceding and following the apex).

9.2.1.2.2 Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB.

9.2.1.2.3 Do not background subtract part of the BFB peak.

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- 9.2.1.3. If the BFB mass spectrum does not meet all the criteria, then replace the septa, bake the column and try the BFB tune again.
- 9.2.1.4. If the criteria are still not met, then perform a FC-43 (perfluorotributylamine) tune followed by a BFB tune. Tuning with FC-43 is a last resort since this compound prematurely contaminates the ion source.
- 9.2.2. Perform initial calibrations on an as needed basis, after a FC-43 tune, and after major instrument maintenance. Analyze a minimum of five concentrations to establish the initial calibration curve.
 - 9.2.2.1. The surrogates are calibrated at a minimum of 5 levels in conjunction with the calibration standards.
- 9.2.3. Analyze the initial calibration curve in the same manner as samples.
 - 9.2.3.1. Attach a heater jacket to each standard to increase the purge efficiency for the ketones and other water soluble and late eluting compounds.
 - 9.2.3.2. Heat the standards to $40^{\circ}\text{C} \pm 1^{\circ}\text{C}$ during the purge.
- 9.2.4. The System Performance Check Compounds (SPCCs) are analyzed with the initial calibration curve, and must meet acceptance criteria before the calibration curve is used.
 - 9.2.4.1. The SPCCs are checked for a minimum average relative response factor.
 - 9.2.4.2. These compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene.
 - 9.2.4.3. The minimum acceptable average response factor (RF) for these compounds should be 0.30 for Chlorobenzene and 1,1,2,2-Tetrachloroethane; 0.10 for Bromoform, Chloromethane and 1,1-Dichloroethane.
- 9.2.5. The primary calibration criteria involve the use of average Response factors (RFs). The RFs from the initial calibration curve should have an $\text{RSD} \leq 15\%$ for all compounds, except for the Calibration Check Compounds (CCCs).
 - 9.2.5.1. The %RSD for the CCCs must be $\leq 30\%$.
 - 9.2.5.2. Due to poor purge efficiency, the % RSD for Alcohols and Ketones must be $\leq 30\%$. (also see section 17.2).
 - 9.2.5.2.1. The Average % RSD must meet the method required $\pm 15\%$ criteria.
 - 9.2.5.2.2. The analytical reports must have a disclaimer in the case narrative to outline the 30% criteria for the alcohols and ketones.

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- 9.2.5.3. The CCCs are 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride.
- 9.2.6. If one or more of the compounds, other than the CCCs and ketones, has a %RSD >15%, generate a calibration curve and follow the steps outlined below (9.2.6.1 - 9.2.6.4.).
- 9.2.6.1. A first order (linear) or second order (quadratic) regression may be used for quantitation (not forced through the origin).
- 9.2.6.1.1 Print out a linear curve (5pts minimum) and a quadratic curve (6 pts minimum).
- 9.2.6.1.2 The Coefficient of Determination (r^2) must be ≥ 0.99 (Correlation Coefficient (r) ≥ 0.995) for the curve to be acceptable. If r^2 is < 0.99 then the instrument must be recalibrated for that compound.
- 9.2.6.1.3 If the software does not calculate r^2 quadratic curves, use the linear curve r^2 for validation.
- 9.2.6.1.4 If r^2 for the linear curve is at least 0.99 (rounded to 2 sig. figs.) then use the curve that gives the y intercept closest to zero (where applicable).
- 9.2.6.2. Print out any calibration curves to evaluate linearity and obtain the correlation coefficients.
- 9.2.6.3. Since the curve is not forced through the origin, inaccuracies may be present near the low end of the curve or negative values may be obtained at the reporting limit.
- 9.2.6.3.1 Negative values occur when the regression calculation of "best fit" does not pass through the lowest calibration standard and the y- intercept falls above the y coordinate (area count) of the low standard of the curve.
- 9.2.6.3.2 The cause is sometimes related to the slight bending in the curve at higher analyte concentrations and a 1st order curve is used. If this is the case the a 2nd order fit will take care of the problem.
- 9.2.6.3.3 Because of the high potential for inaccuracies or negative numbers at the low end of the curve, positive results below the low calibration standard may not be quantitated and may not be reported. **(No "J" flags below the low calibration standard when a calibration curve is used.)**
- 9.2.6.3.4 If the RF >15% but <50% and the total average RF <15%, then use the RF and requantitate "J" flags.

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- 9.2.6.4. When evaluating results near the RL review them carefully to ensure they make sense (i.e. no negative values or positive values with areas below the lowest standard). If a result is questionable, the sample should be re-analyzed on another instrument or the result reported as estimated.
- 9.2.7 As an alternative to generating a calibration curve, if the compounds > 15% RSD are known historically to be not detected (ND) or the calibration curve does not meet the acceptance criteria, calculate the average RSD for all compounds and if < 15% quantitate using the average RF. Report data with a form identifying which compounds did not meet the 15% RSD criteria.
- 9.2.7.1 If the % RSD is > 50% for any compound re-evaluate the standard concentrations and perform any necessary maintenance and recalibrate the instrument.
- 9.3. Continuing Calibration
- 9.3.1 Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift before any samples are analyzed:
- 9.3.1.1. The BFB tuning criteria in Table 4 of method 8260B must be achieved. Refer to the initial calibration section 9.2.1.
- 9.3.1.2. Analyze a midpoint calibration standard (CCV). SPCC and CCC criteria must be met.
- 9.3.1.3. Analyze a method blank to ensure that the total system is free of contaminants.
- 9.3.2. Prepare the CCV standard.
- 9.3.2.1 Pour approximately 12 ml of ultrapure water into the 10ml syringe.
- 9.3.2.2 Adjust the water in the syringe to 10-ml.
- 9.3.2.3 Purge the empty glass test tube with nitrogen gas to minimize laboratory contamination.
- 9.3.2.4 Inject the syringe contents into the nitrogen purged glass test tube.
- 9.3.2.5 Add 5 μ l of the 50 μ g/ml concentration internal standard/surrogate solution to the ultrapure water in the test tube.
- 9.3.2.6 Add 5 μ l of the 50 μ g/ml concentration working standard to the ultrapure water in the test tube .
- 9.3.2.7 Attach the test tube to the port and tighten it with a sparge tube wrench. Attach a heater jacket.

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- 9.3.2.8 Enter the port numbers corresponding to the samples to be analyzed into the concentrator. Press the start button.
- 9.3.3. Check that the performance criteria of the SPCCs and CCCs are met prior to sample analysis.
- 9.3.3.1 The SPCCs must meet the same acceptance criteria as defined in the initial calibration.
- 9.3.3.2 The CCCs must have a percent difference $\leq 20\%$ from the initial calibration.
- 9.3.3.3 Calculate acceptance limits for the non-CCC analytes by performing a statistical evaluation of the %Difference of 20 Midpoint (CCV) standards. Use ± 3 SD or $\pm 20\%$, whichever is greater, for the acceptance limits.
- 9.3.3.4 The non-CCC analytes should have an average percent difference within the acceptance limits previously established. This would include the SPCCs.
- 9.3.3.4.1 If the Midpoint (CCV) non-CCC result is greater than the high acceptance limit of the expected value and all samples are ND for the compound then report the results with a CAR. Qualify sample results with a "C" for each analyte that meets this criteria.
- 9.3.3.4.2 If the Midpoint non-CCC result is below the acceptance limit, flag the result and report with a CAR and a GCMS calibration Check Criteria form.
- 9.3.3.4.3 If any analyte exceeds the criteria, an average percent recovery $< 20\%$ may be used to accept the analytical run. A GCMS calibration Check Criteria form must be utilized if this condition exists. (if samples are ND and CCV is biased high, the Check Criteria form is not required.
- 9.3.3.5 If the SPCCs and/or CCCs fail, reanalyze the standard. If they pass, continue with analysis. If they fail again, take corrective action and recalibrate the instrument.
- 9.3.4. Determine that the retention times for any internal standard have not changed by more than 30 seconds from the midpoint standard measured during the most recent initial calibration.
- 9.3.5. Determine that the absolute areas of the quantitation ions of the internal standards in the CCV check standard have not changed by a factor of two (-50% to +100%) from the IS area from the midpoint standard measured during the most recent initial calibration (5 pt). If the retention times or areas have changed by more than these amounts, perform necessary maintenance and recalibrate.

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9.3.5.1 For TBA-d9 internal standard is not monitored as (-50%-100%). TBA-d9 is used for the quantitation of TBA and ethanol due to the stability in water. TBA-d9 is temperature dependent and also monitors the behavior of TBA and ethanol in the system.

9.4. Analysis

9.4.1. Perform a BFB tune and analyze a CCV check standard and a method blank before analyzing any samples.

9.4.2. Bake the purge apparatus daily, in the morning, to clean it prior to analysis of samples. Syringes are rinsed and baked at 120° C between uses to prevent cross contamination.

9.4.3. All sample and standard solutions must be allowed to warm to ambient temperature before analysis.

9.4.4. Water Samples

9.4.4.1 Analyze a water sample by pouring approximately 12 ml of the sample water into the 10-ml syringe.

9.4.4.2 Adjust the water in the syringe to 10-ml.

9.4.4.3 Inject the syringe contents into a nitrogen purged glass test tube.

9.4.4.4 Add 5 µl of the 50 µg/ml concentration internal standard/surrogate solution to the sample water in the test tube.

9.4.4.5 Attach the test tube to the port and tighten it with a sparge tube wrench.

9.4.4.6 Attach the heater jacket to the glass test tube. The use of a heated purge at 40°C ± 1°C increases the purge efficiency for the ketones and other water soluble and late eluting compounds.

9.4.4.7 Enter the port numbers corresponding to the samples to be analyzed into the concentrator. Press the start button.

9.4.4.8 Dip pH test paper into the sample remaining in the VOA vial. Record the pH on the sample track sheet as pH <2 or pH >2. If the sample has a pH >2 and the sample was analyzed after 7 days, flag the results with a 'P' qualifier and notify the project manager.

9.4.4.9 Dilute and re-analyze any sample that exceeds the highest calibration standard. Use a different sample VOA vial if one is available. See 9.4.5.

9.4.5. Dilution of a highly concentrated water sample

9.4.5.1 Withdraw 10-ml of ultrapure water into a 10-ml syringe.

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- 9.4.5.2 Use a micro syringe to withdraw and discard the appropriate volume of ultrapure water from the 10-ml syringe. The appropriate volume of ultrapure water is equal to the volume of sample used in the dilution. Add to test tube.
- 9.4.5.3 Add the appropriate amount of the sample to the water, using a micro syringe to attain the desired concentration.
- 9.4.5.4 Add 5 μ l of the 50 μ g/ml concentration internal standard/surrogate solution to the sample water in the test tube. Analyze the diluted sample.
- 9.4.6. Low Level Soil Samples – From a Boring Tube
 - 9.4.6.1 Prepare a low-level soil sample by discarding one-half inch to one inch of the top layer of the soil sample before removing a portion to analyze. This top layer may have lost any volatile analytes it may have contained and therefore should be discarded.
 - 9.4.6.2 Using a scoopula, weigh 0.5 gram to 5.0 grams of the sample into a test tube.
 - 9.4.6.3 Immediately, measure 10-ml of ultrapure water into a 10-ml syringe and add to test tube. Then add 5 μ L of 50 μ g/mL concentration internal standard/surrogate solution.
 - 9.4.6.4 Cover the tube with parafilm and mark the test tube with the sample amount and the sample number, using a permanent marker.
 - 9.4.6.5 Do not weigh more than 5-10 samples at a time before proceeding to the next step.
 - 9.4.6.6 Attach the tubes to the autosampler port, tightening the mount with a sparge tube wrench.
 - 9.4.6.7 Attach the heater jacket to the glass test tube. Enter the port numbers corresponding to the samples to be analyzed into the concentrator. Press the start button.
 - 9.4.6.8 Re-analyze at a dilution, any sample that exceeds the highest calibration standard. See 9.4.8.
- 9.4.7. Low level Soil samples – From an Encore™ sampling device
 - 9.4.7.1 Weigh each Encore™ sampling device containing samples to the nearest 0.1 g. and record the weight.
 - 9.4.7.2 At the instrument, extrude the sample directly into a test tube. Immediately, measure 10-ml of ultrapure water into a 10-ml syringe and add to test tube. Then add 5 μ L of 50 μ g/mL concentrated internal standard/surrogate solution.

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- 9.4.7.3 Cover the tube with parafilm and mark the test tube with the sample amount and the sample number, using a permanent marker.
- 9.4.7.4 Do not transfer more than 5-10 samples at a time before proceeding to the next step.
- 9.4.7.5 Attach the tubes to the autosampler port, tightening the mount with a sparge tube wrench.
- 9.4.7.6 Attach the heater jacket to the glass test tube. Enter the port numbers corresponding to the samples to be analyzed into the concentrator. Press the start button.
- 9.4.7.7 Reweigh the empty Encore™ sampling devices. The difference between the initial weight and the final weight is the actual sample weight used to calculate final results.
- 9.4.8. Dilution of High Level Soil Samples
- 9.4.8.1 Remove another one-half inch of soil from the boring and weigh out between 0.5 grams and 10 grams of the sample into a 40 ml VOA vial, using a scoopula.
- 9.4.8.2 Label a VOA vial with the sample number and sample amount.
- 9.4.8.3 Immediately add 10-ml of MeOH and add 12.5 µl of the Absolute 2000 µg/ml concentration surrogate solution to the Sample / MeOH mixture.
- 9.4.8.4 Mix with a vortex on the fast setting.
- 9.4.8.5 Next, centrifuge the sample/methanol mixture and cap and store in the refrigerator until analysis. Wipe the scoopula clean with a Kimwipe before weighing another sample.
- 9.4.9. Dilution of soil samples containing high levels of analytes -- From an Encore™ sampling device
- 9.4.9.1 Weigh each Encore™ sampling device containing samples to the nearest 0.1 g. and record the weight.
- 9.4.9.1 Extrude the sample directly into a VOA vial.
- 9.4.9.2 Reweigh the empty Encore™ sampling devices. The difference between the initial weight and the final weight is the actual sample weight used to calculate final results.
- 9.4.9.3 Immediately add 5 ml of MeOH.
- 9.4.9.4 Label the VOA vial with the sample amount, client code and sample number.
- 9.4.9.5 Using a 10 ul syringe, add 6.25 ul of the 2000 ug/mL surrogate standard (without internal standard) into the 40 ml VOA vial.

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- 9.4.9.6 Cap the VOA vial and mix with a vortex on the fast setting. Let the mixture settle for 10 minutes. If the sample and MeOH have not separated, then centrifuge.
- 9.4.9.7 To avoid cross contamination between samples, wipe the scoopula clean with a clean Kimwipe before weighing another sample, and do not touch the soil with gloves.
- 9.4.10. Prepare the diluted soil sample for analysis
- 9.4.10.1 Measure 10-ml of ultrapure water into a 10-ml syringe and add to test tube.
- 9.4.10.2 Depending on the dilution ratio, withdraw 1 μl to 100 μl of the MeOH layer of the diluted soil sample, using the appropriate size syringe. Add this aliquot of the MeOH layer to the ultrapure water in the test tube.
- 9.4.10.3 Using a 10 μl syringe, add 5 μl of the 50 $\mu\text{g}/\text{ml}$ concentration internal standard solution to the test tube.
- 9.4.10.4 Attach it to the autosampler port, tightening the mount with a sparge tube wrench.
- 9.4.10.5 Attach the heater jacket to the glass test tube. Enter the port numbers corresponding to the samples to be analyzed into the concentrator. Press the start button.
- 9.4.10.6 Record the amount of soil weighed, the volume of MeOH used and the volume of MeOH extract analyzed in the computer and on the sample track sheet. These weights and volumes are needed to calculate the final results.
- 9.4.11. Air samples
- 9.4.11.1 Analyze an air sample by pouring approximately 12 ml of nanopure water into a 10 ml syringe.
- 9.4.11.2 Adjust the water to 10 ml.
- 9.4.11.3 Inject the syringe contents into a nitrogen purged glass test tube.
- 9.4.11.4 Add 5 μL of the 50 $\mu\text{g}/\text{ml}$ internal standard/surrogate solution to the water in the test tube.
- 9.4.11.5 Attach the test tube to the port and tighten it with a sparge tube wrench.
- 9.4.11.6 Using a gas tight syringe measure 5cc of the air sample.
- 9.4.11.6.1 For dilutions, use a lower volume of the air sample.
- 9.4.11.7 Add the measured volume of air sample to the test tube through the luer lock valve.
- 9.4.11.8 Attach the heater jacket to the glass test tube.

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9.4.11.9 Enter the port number corresponding to the samples to be analyzed into the concentrator. Press the start button.

9.4.11.10 Dilute and re-analyze any sample that exceeds the highest calibration standard.

9.5. Compound Identification

9.5.1. For most analytes, at least two Mass Ions must be present on the mass spectra for qualitative ID and quantitation purposes. The exceptions to this are when only one Mass Ion can be detected in the low end calibration standard(s).

9.5.2. If the instrument can only detect one ion, qualitative and quantitative ID will be based on Retention time of the target peak and the presence of the highest mass (Primary) ion. Examples would include tert-Butyl Alcohol (TBA), Ethanol, etc. In these cases, flag the sample result with the qualifier "ID".

9.5.3. If the secondary ion is present but the primary is not, then the compound will not be identified.

9.6. Instrument Conditions

9.6.1. The following are general instrument conditions. These may vary slightly between instruments, or because of necessary instrument maintenance (e.g. column trimming) or because of column age.

9.6.2. Column: DBVRX 60mm x 250 μ m x 1.4 μ m

9.6.3. Carrier gas (he) flow rate: 1.0 ml/min

9.6.4. Purge and Trap Times

9.6.4.1 Pre-heat – 1 min

9.6.4.2 Purge – 11 min.

9.6.4.3 Desorb – 2 min.

9.6.4.4 Bake – 8 min.

9.6.5. Purge and Trap Temperatures

9.6.5.1 Pre-heat – 40 $^{\circ}$ C

9.6.5.2 Purge – 40 $^{\circ}$ C

9.6.5.3 Desorb – 190 $^{\circ}$ C

9.6.5.4 Bake – 210 $^{\circ}$ C

9.6.5.5 Valve – 120 $^{\circ}$ C

9.6.5.6 Transfer Line – 110 $^{\circ}$ C

9.6.5.7 Ext. Heater – 110 $^{\circ}$ C

9.6.6. Initial temperature: 40 $^{\circ}$ C, hold for 8 minutes

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- 9.6.7. Temperature program: 12 °C/min to 190 °C, hold for 1 minute
6 °C/min to 225 °C, hold for 1 minute
- 9.6.8. Final temperature: 225 °C
- 9.6.9. Column Bake out: overnight at 210 °C
- 9.6.10. Injector temperature: 200 °C
- 9.7. Preventative Maintenance
 - 9.7.1. Record all performed maintenance in the instrument maintenance logbook.
 - 9.7.2. Replace OI #10 traps and Tekmar trap K if necessary.
 - 9.7.3. Replace injector septa and deactivate liners when necessary.
 - 9.7.4. Clean the ion source and replace filaments when necessary.
 - 9.7.5. Back-up all data monthly.
 - 9.7.6. Fill a 20 ml plastic syringe with methanol and flush the purge needle and purge valve whenever needed (after high level or surfactant containing samples).
 - 9.7.7. Change the oil in the foreline pump twice per year and record on the maintenance schedule posted on the instrument.
 - 9.7.8. Check the diffusion pump oil at least annually and replace as necessary and record on the maintenance schedule posted on the instrument.
 - 9.7.9. Replace the carrier gas trap at least annually and record on the maintenance schedule posted on the instrument.
 - 9.7.10. If an instrument is unusable or has limitation to its use (bad port, bad heater jacket, not usable for ketones, not for low level samples, etc) , it must be tagged accordingly until such a time the problem has been corrected. Record the problem, solution and verification of proper operation into the instrument maintenance logbook.
- 9.8. Results Reporting
 - 9.8.1. Create a batch in ELMNT prior to loading the instrument. (it can always be edited later if something changes)
 - 9.8.2. Create a bench sheet in ELMNT. Enter the initial sample volumes and the final volumes. Enter the QC information: initial and final volumes, spike source, sample source for MS/MSD.
 - 9.8.3. Use data tool to transfer the data electronically into ELMNT.
 - 9.8.4. Verify all sample results and QC results transferred correctly and verify QC acceptance prior to updating the status in ELMNT to analyzed.

10. QUALITY CONTROL

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- 10.1. Analyze a method blank after each CCV check standard every 12 hours, and prior to sample analysis, with every batch of 20 samples or less, and after a highly concentrated sample as a cross-contamination check. Prepare a method blank with 10-ml of ultrapure water. Add 5 µl of the 50 µg/ml concentration internal standard/surrogate solution to the ultrapure water.
 - 10.1.1. The method blank results must be below the reporting limit (RL). If detection above the RL is observed, and associated samples detect the same compound, flag results with "B". Samples containing the compound(s) of contamination must be reanalyzed if they are < 20x the level found in the blank.
 - 10.1.2 If data is being reported down to the MDL, if there are values in the blank above the MDL, the blank and data shall be flagged with a "B".
 - 10.1.3. A methanol blank is analyzed whenever methanol extracts are prepared and analyzed. The methanol must be free of contaminants below the reporting limit. See 10.1.1.
- 10.2. If there is a positive hit in a Trip Blank, verify if there are any positive hits for the compound(s) in the associated project samples. Notify the project manager when reporting the data if there are any affected samples.
- 10.3. Prevent cross contamination by rinsing, between samples, the purge apparatus and syringe with two portions of ultrapure water. Avoid carry over by cleaning a system contaminated by saturated samples.
- 10.4. Analyze a LCS with every batch of 20 samples or less. Prepare the LCS by injecting 5 µl of the internal standard/surrogate solution to 10-ml of ultrapure water. Add 5 µl of the 50 µg/ml concentration LCS standard.
 - 10.4.1 The acceptance limits for the LCS are determined semi-annually by in-house statistical analysis. If any reported LCS compound exceeds the pre-established limits:
 - 10.4.1.1 If the LCS is out above the acceptance limits and the sample results are ND, fill out a CAR and report the data with an 'L' qualifier.
 - 10.4.1.2 If the LCS is out of the acceptance limits and the sample results are positive, reanalyze the sample with acceptable QC.
 - 10.4.1.3 If the LCS is out below the acceptance limits and the sample results are ND, reanalyze once. If still out, reprepare a fresh LCS solution and reanalyze.
 - 10.4.1.4 A LCS is prepared in Methanol for MeOH soil extraction batches (See section 9.1.19). Add 100 µl to 10 ml of purged DI water, add internal standard and analyze as a sample. Follow the acceptance criteria and corrective actions listed above.

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- 10.5. Surrogates are added to each sample, Calibration check method blank, LCS, MS and MSD. The surrogate recoveries must fall within the acceptance limits established semi-annually by in-house statistical analysis. If any surrogates are outside of the acceptance limits, determine the cause of the problem and take corrective action. If necessary, fill out a corrective action form. If the surrogate is out due to sample matrix, note this information on the results when reporting by flagging the results with a 'Z' qualifier.
 - 10.5.1. If any surrogates are outside of the acceptance limits, determine the cause of the problem and take corrective action. If the cause is not due to obvious chromatographic matrix interference, the sample must be reanalyzed to confirm matrix effects.
- 10.6. Analyze a matrix spike/matrix spike duplicate with every batch of twenty samples, or less.
 - 10.6.1. Add 5 μ l of the 50 μ g/ml concentration LCS standard to two separate aliquots of a sample and process the same as the other samples.
 - 10.6.2. The acceptance limits for the MS/MSD are determined semi-annually by in-house statistical analysis.
 - 10.6.2.1 If the MS/MSD are outside of the acceptance limits due to matrix effects flag the MS/MSD with the appropriate 'M' qualifier.
 - 10.6.2.2 If the MS/MSD are outside of the acceptance limits due to instrument problems or due to analyst error, re-analyze the MS/MSD if possible, otherwise fill out a CAR with **detailed** explanation and corrective action.
- 10.7. For every sample, determine that the retention times for any internal standard have not changed by more than 30 seconds from the midpoint level standard of the most recent initial calibration.
 - 10.7.1. Determine that the absolute areas of the quantitation ions of the internal standards in each sample have not changed by a factor of two (-50% to +100%) from the IS area measured from the midpoint level standard of the most recent initial calibration.
 - 10.7.2. If the retention times or areas have changed by more than these amounts, reanalyze the sample. If it is still out, note it on the worksheet when reporting results.
- 10.8. The continuing calibration verification retention times and areas must be compared to the mid-point standard level of the most recent initial calibration.
- 10.9. Perform a method detection limit (MDL) study initially and after extensive instrument maintenance or a significant change in the method. An MDL study is accomplished by analyzing seven replicates of the lowest calibration standard and multiplying the SD by 3.143.
- 10.10. Perform retention time window studies at the same time as the MDL study.

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10.10.1. Use the CCV check standard from the three previous days to determine the retention time windows.

10.11. Perform an Initial Demonstration of Capability (IDOC) before performing analyses by analyzing 4 LCS samples with an average recovery which meets the in-house acceptance limits. If the average does not meet the requirement identify the problem and repeat the process.

11. CALCULATIONS

11.1. Calculation to determine Response Factors (RF), Calibration Factors (CF), and sample concentrations are found in SW 846 method 8000B, sections 7.4 and 7.5.

11.1.1. Response Factor: $RF = \frac{(R_A)(C_{IS})}{(C_A)(R_{IS})}$

R_A = Analyte response

C_A = analyte concentration

R_{IS} = internal standard response

C_{IS} = analyte concentration

RF = response factor

11.1.2. Waters

11.1.2.1 $C_f = C_i \times PF \times DF$

C_f = Final concentration in $\mu\text{g/L}$ or $\mu\text{g/Kg}$

C_i = Concentration in $\mu\text{g/L}$ from instrument

PF = Preparation Factor

DF = Any additional Dilution Factor

11.1.3. Low Level Soils

11.1.3.1 $C_f = C_i \times DF \times CF$

C_f = Final concentration in $\mu\text{g/L}$ or $\mu\text{g/Kg}$

C_i = Concentration in $\mu\text{g/L}$ from instrument

DF = Dilution Factor (5g / wt of soil purged, in g)

CF = Calibration Factor (2x soils)

11.1.4. High Level Soils (MeOH extracts)

11.1.4.1 $C_f = C_i \times CF \times PF \times DF$

C_f = Final concentration in $\mu\text{g/L}$ or $\mu\text{g/Kg}$

C_i = Concentration in $\mu\text{g/L}$ from instrument

PF = Prep Factor (Vol of MeOH in ml / Weight of soil, in g)

DF = Dilution Factor (5 ml / actual volume injected, in ml)

CF = Calibration Factor (2x soils)

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11.1.5. Surrogate Spike Results in High Level Soils (MeOH extracts)

$$11.1.5.1 C_f = C_i \times CF \times SF \times DF$$

C_f = Final concentration in $\mu\text{g/L}$ or $\mu\text{g/Kg}$

C_i = Concentration in $\mu\text{g/L}$ from instrument

SF = Surrogate Factor (Final Prep Vol of MeOH in ml / 10 ml)

DF = Dilution Factor (100 μl / actual volume injected, in μl)

CF = Calibration Factor (2x)

11.1.6. Determine the % Difference of the CCV using the following equation:

$$\% \text{ Difference} = \frac{[\text{Apparent conc. } (\mu\text{g/l}) - \text{True conc. } (\mu\text{g/L})] \times 100}{\text{True conc. } (\mu\text{g/l})}$$

11.1.7. Determine the % recovery for the LCS, Surrogates, and MS/MSD as follows:

$$\% \text{ Recovery} = \frac{(\text{Sp} - \text{S})}{\text{Sa}} \times 100 \qquad \text{RPD} = \frac{|\text{R1} - \text{R2}|}{(\text{R1} + \text{R2})/2} \times 100$$

Where: Sp = Spike result

S = Sample result (LCS and Surrogate = 0)

Sa = Spike amount

R1 = Conc. of MS

R2 = Conc. of MSD

12. PAPERWORK FLOW

12.1. Daily

12.1.1. Query ELMNT for the desired analysis codes.

12.1.2. Compare the worklist with the previous day's Sample Track Sheets to determine which sample numbers to cross off. Cross off the sample numbers of those analyses that have been completed or are currently being analyzed. Write the date analyzed and GCMS # beside the crossed out line.

12.1.3. Complete the Sample Track Sheet and instrument logbook as each sample is loaded onto the autosampler. Use the Comments section to note whether or not a sample is a confirmation, a re-analysis, or a rush. Also include a dilution amount or any other important information in the Comments section.

12.1.4. Record the appropriate analyst (load, run, spectra) and check the method name on the sequence files.

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- 12.1.5. All raw data must undergo second level review. All raw data must be initialled and dated.
- 12.1.6. Reports and their corresponding chromatograms automatically print out as each sample is analyzed. Perform a manual integration when a chromatogram has a co-elution or interferences. All manual integrations must undergo second level review. Follow SOP titled "Manual Integrations".
- 12.1.7. Print out the mass spectra that correspond to positive results on the quantitation report. Compare these spectra with the calibration spectra. If there is no spectral match (NSM), draw a line through the result on the report and write NSM beside it. Initial and date the first page of the quantitation report for each sample. Also, the code WRT, which stands for wrong retention time can be used if applicable.
- 12.1.8. Staple behind each chromatogram, its corresponding quantitation report and mass spectra. Match the chromatograms, quantitation reports, and spectra to their corresponding Sample Track Sheets and file them in the cabinet by ascending date and ascending gas chromatograph/mass spectrometer number.
- 12.2. Monthly
 - 12.2.1. Print initial calibrations and keep them on file for each instrument.
 - 12.2.2. Enter the standard information into ELMNT. See 9.1.7.4.
- 12.3. As Needed
 - 12.3.1. Document on the Daily Log Summary and sample results whenever the sample matrix interferes with the recovery of the surrogate, internal standard and/or MS/MSD.
 - 12.3.2. Document on a Corrective Action Report, any non-matrix related analytical problems or variances.
 - 12.3.2.1 Keep the pink copy with the data package,
 - 12.3.2.2 Submit the white and yellow copy to the QC department so the project manager has the information prior to the release of the data to the client.
 - 12.3.2.1 Complete an instrument maintenance log for each instrument and include the date that the instrument became inoperable, the steps taken to fix the instrument, replacement parts, the initials of the analyst/technical specialist (or the name of the service company that performed the maintenance), and the date that the instrument became operable again.
 - 12.3.3. Fill out a GCMS Initial Calibration Criteria form whenever any compound exceeds the ICAL criteria. Include a copy of the form with each data set affected.

13. WASTE MANAGEMENT

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13.1. See 'Waste Disposal' Standard Operating Procedure

14. POLLUTION PREVENTION

14.1. Hazardous samples should be segregated and disposed of as hazardous waste.

15. METHOD PERFORMANCE

15.1. See the attached ELMNT pages that contain the analyte list, Reporting Limits, and Performance Information (Control Limits and method detection limits).

16. METHOD REFERENCES

16.1. EPA Method 8260B, EPA SW-846 Update III, December 1996

16.2. EPA Method 8000B, EPA SW-846 Update III, December 1996

16.3. EPA Method 5030A, EPA SW-846 Update I, July 1992

16.4. EPA Method 5030B, EPA SW-846 Update III, December 1996

16.5. EPA Method 5035, EPA SW-846 Update III, December 1996

17. METHOD VARIANCES

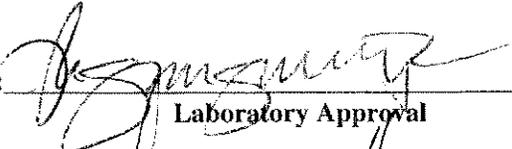
17.1. Due to poor response for the primary ions, the secondary ions are used for the quantitation of the following ketones. The primary ion must be present, however, for the compound to be positively identified. The following table lists the ketones with their primary and secondary ions used for quantitation.

Compound	Primary Ion	Secondary Ion
Acetone	58	43
2-Butanone	72	43
4-Methyl-2-pentanone	100	43

17.2. In order to add confidence to the non-CCC analytes' quantitation, though not specified in the method, control limits are generated for the non-CCC compounds based on ± 3 SD of at least 20 CCV. Acceptance limits are based on either 20% or the calculated limits whichever is greater.

17.3. Due to the need (client demand) of achieving lower detection limits, a 10 mL purge volume is used. Because of the low purge efficiency of Alcohols and Ketones a % RPD requirement of 30% (same as CCCs) is used for these compounds. The method criteria, of the average % RSD within $\pm 15\%$ **must** still be met and the clients **must be notified** of the criteria in the final report.

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Approved By:  Date: 3/26/01
Laboratory Approval

Approved By:  Date: 3/14/01
Quality Assurance Approval