Appendix C
Site 12 Site Specific Long-term Monitoring Plan

SAP Worksheet #C1—Title Page

Appendix C

Final

Long-term Monitoring Sampling and Analysis Plan for Site 12

Joint Expeditionary Base (JEB) Little Creek-Fort Story
JEB Little Creek
Virginia Beach, Virginia

Contract Task Order 125

January 2011

Prepared for

Department of the Navy
Naval Facilities Engineering Command
Mid-Atlantic

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Prepared by

CH2MHILL

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SAP Worksheet #C1—Title and Approval Page (continued)

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Introduction

Appendix C contains the site-specific Long-term Monitoring (LTM) Sampling and Analysis Plan (SAP) information pertaining to Site 12 Exchange Laundry Waste Disposal Area. Site-specific details provided in this Appendix C do not apply to the sites discussed in the other appendices of this SAP. These site-specific details provided in Appendix C also do not apply to projects of this nature in general, and are therefore not included in the Main section of this SAP.

The worksheets included in this appendix correspond to the 37 worksheets in the United States Navy (Navy) SAP format. The LTM SAP worksheet numbers corresponds to the worksheet numbers provided in this appendix; however, worksheets included in this appendix include the letter "C" to designate inclusion in Appendix C. Information included in the main section worksheets are not repeated in the "C" designated site-specific worksheets, only information specifically regarding Site 12 is included in a "C" designated worksheet.

All tables include the "C" designation in the table number and are embedded within the worksheets. Figures include the C designation and are included at the end of the worksheets. The site-specific laboratory SOPs are included as **Attachment C1**, calculations for surface water screening levels using groundwater at MW-36D are included in **Attachment C2**, and calculations for vapor screening levels using groundwater at MW-36D are included in **Attachment C3**. Field standard operating procedures (SOPs) and the data management documentation are included respectively in Appendix A and B of the LTM SAP.

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Acronyms and Abbreviations

AM	Activity Manager
bgs	below ground surface
CA	corrective action
CAS	Chemical Abstracts Service
CCB	continuing calibration blank
CCC	calibration check compounds
CCV	continuing calibration verification
COC	contaminant of concern
CSM	conceptual site model
DCE	dichloroethene
DL	detection limit
DoD	Department of Defense
DQI	data quality indicator
EIS	Environmental Information Specialist
ELAP	Environmental Laboratory Accreditation Program
ERD	enhanced reductive dechlorination
FID	flame ionization detector
GC	gas chromatograph
GC/MS	gas chromatograph/mass spectrometer
gpm	gallons per minute
GW	groundwater
IC	ion chromatograph
ICAL	initial calibration
ICB	initial calibration blank
ICP	inductively coupled plasma
ICPAES	inductively coupled plasma atomic emission spectrometry
ICS	interference check solution
ICV	initial calibration verification
ISCO	in situ chemical oxidation
JEB	Joint Expeditionary Base
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management Systems
LOD	limit of detection
LOQ	limit of quantitation
LTM	long-term monitoring
LUC	land use control

µg/L	micrograms per liter
MCL	maximum contaminant level
MDL	method detection limit
MEE	methane, ethane, ethene
mg/L	milligrams per liter
MPC	measurement performance criteria
MS/MSD	matrix spike/matrix spike duplicate
Navy	United States Navy
NIRIS	Navy Installation Restoration Information System
PAL	project action limit
PC	Project Chemist
PCE	tetrachloroethene
PIL	performance indication level
PM	Project Manager
PQL	project quantitation limit
PQOs	project quality objectives
QA	quality assurance
QC	quality control
QL	quantitation limit
qPCR	quantitative polymerase chain reaction
QSM	Quality Systems Manual
RAO	remedial action objectives
RBC_{gw}	risk-based concentration-groundwater
RD	Remedial Design
RF	response factor
ROI	radius of influence
RPD	relative percent difference
RPM	Remedial Project Manager
RRT	relative retention time
RSD	relative standard deviation
SAP	Sampling and Analysis Plan
SDWA	Safe Drinking Water Act
SMP	Site Management Plan
SOP	standard operating procedure
SPCC	system performance check compound
SRI	Supplemental Remedial Investigation
SW	surface water
TAL	Test America Laboratories
TBD	to be determined
TCE	trichloroethene
TOC	total organic carbon

UFP	Uniform Federal Policy
UU/UE	unlimited use and unrestricted exposure
VC	vinyl chloride
VC-R	vinyl chloride - reductase
VFA	volatile fatty acids
VOA	volatile organic analyte
VOC	volatile organic compound

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SAP Worksheet #C2—SAP Identifying Information

The SAP identifying information is provided in **Worksheet #2** of the LTM SAP. Information on **Worksheet #C2** specific to Site 12 includes the dates of the scoping sessions and the dates and titles of any documents written for previous site work that is relevant to this current investigation.

List dates of scoping sessions that were held:

Scoping Session	Date
UFP-SAP LTM Sampling Strategy	September 22, 2009
UFP-SAP LTM Sampling Strategy	November 19, 2009
UFP-SAP LTM Sampling Strategy	January 27, 2010

List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date
Master Project Plans for Naval Amphibious Base Little Creek, Virginia Beach, Virginia	April 2007
Final Supplemental Remedial Investigation, Site 12	December 2000
Final Feasibility Study, Site 12	September 2004
Final Proposed Remedial Action Plan, Site 12	June 2005
Final Record of Decision, Site 12	September 2005
Explanation of Significant Difference, Site 12	October 2006
Final Remedial Action Work Plan, Site 12	February 2007
Final Remedial Action Work Plan Addendum, Site 12	July 2007
Final Remedial Action Construction Completion Report, Site 12	July 2008
Final Remedial Design for Land Use Controls, Site 12	February 2009

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SAP Worksheet #C3—Distribution List

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address
Adrienne Jones	PM	CH2M HILL	757-671-6238	Adrienne.jones@ch2m.com
TBD	FTL	CH2M HILL	TBD	TBD
Sonya Gordon	Project Manager (PM)	Empirical Laboratories	615-345-1115	sgordon@empirlabs.com
Anita Biernacki	PM	Microbial Insights	865-573-8188	abiernacki@microbe.com
Abbie Yant	PM	Test America Laboratories (TAL) Tallahassee (subcontracted by Empirical for Volatile Fatty Acids (VFA) analysis)	912-354-7858	Abbie.Yant@testamericainc.com
Laura Maschhoff	Data Validator	DataQual Environmental Services	314-330-1327	dataqual@charter.net

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SAP Worksheet #C4—Project Personnel Sign-Off Sheet

Name	Title/Role	Telephone Number	Signature/E-mail Receipt	Date SAP Read
Adrienne Jones	PM / CH2M HILL	757-671-6238		
TBD	FTL / CH2M HILL	TBD		
Sonya Gordon	PM/ Empirical	615-345-1115		
Anita Biernacki	PM/ Microbial Insights	865-573-8188		
Abbie Yant	PM/ TAL- Tallahassee	912-354-7858		
Laura Maschhoff	Data Validator/ DataQual	314-330-1327		

Once the UFP-SAP is finalized, CH2M HILL will send copies to all parties on the distribution list in Worksheet #4 and #C4and request signatures. If signatures are returned, copies will be kept on internal CH2M HILL project folders.

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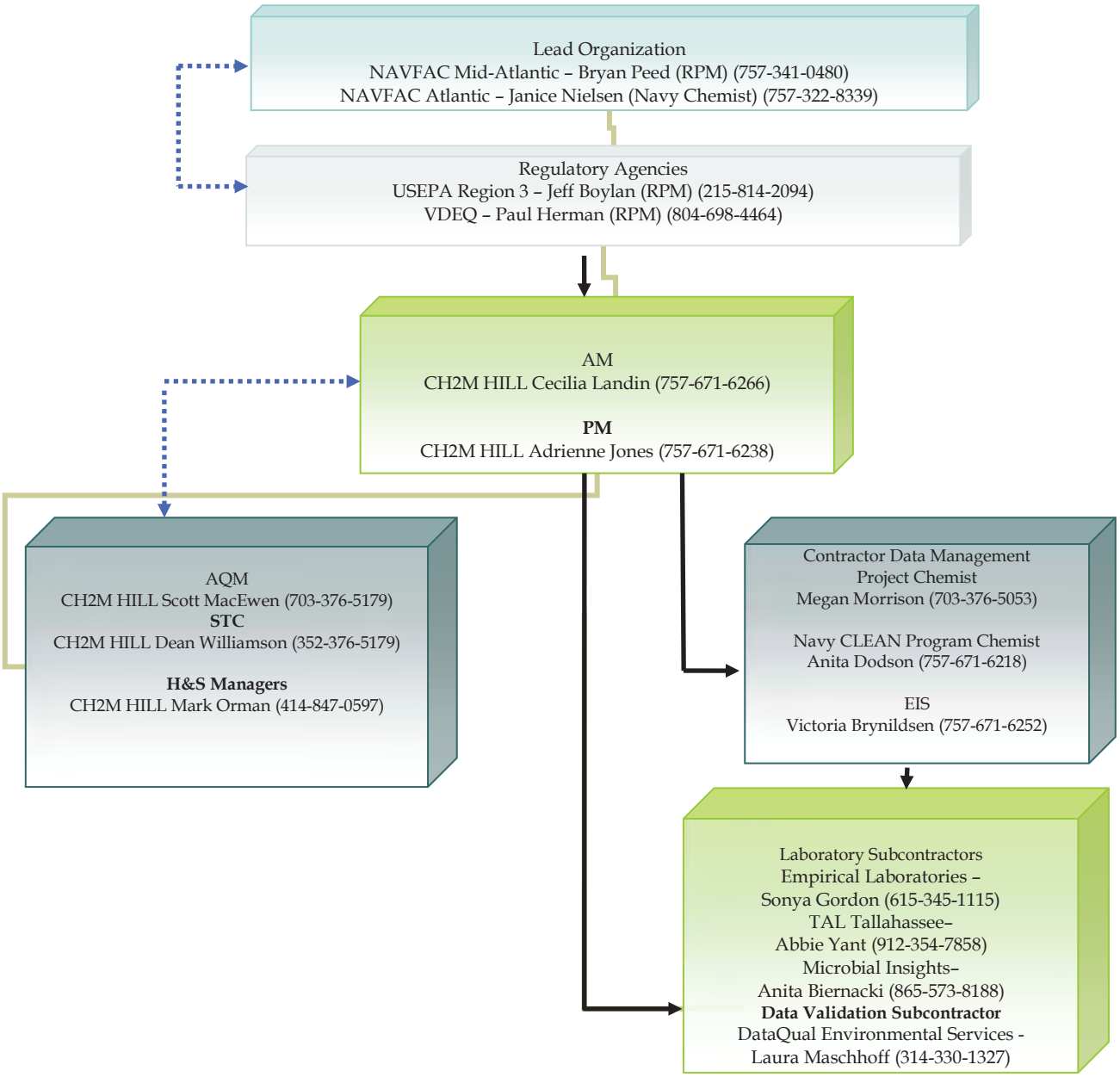
SAP Worksheet #C5—Project Organizational Chart



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SAP Worksheet #C6—Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Reporting Lab Data Quality Issues	Laboratory PM	Sonya Gordon/ Empirical Anita Biernacki/ Microbial Insights Abbie Yant/ TAL-Tallahassee	615-345-1115 865-573-8188 912-354-7858	All quality assurance/quality control (QA/QC) issues with project field samples will be reported as soon as possible to the Project Chemist (PC) and/or Environmental Information Specialist (EIS) by the laboratory. The second-tier laboratory (TAL-Tallahassee) will report all QA/QC issues as soon as possible to the primary laboratory (Empirical). The primary laboratory will report to the CH2M HILL VC and/or EIS.
Reporting Lab Data Quality Issues	Data validator PM	Laura Maschhoff/ DataQual Environmental Services	314-330-1327	All quality assurance/quality control (QA/QC) issues with project field sample results and reporting will be reported as soon as possible to the Project Chemist (PC) and/or Environmental Information Specialist (EIS) by the laboratory.

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SAP Worksheet #C7—Personnel Responsibilities Table

Name	Title/Role	Organizational Affiliation	Responsibilities
Sonya Gordon Abbie Yent Anita Biernacki	Laboratory Subcontractor PM	Empirical Laboratories TAL-Tallahassee Microbial Insights	Manages samples tracking and maintains good communication with VC and EIS. Second tier subcontractors (TAL-Tallahassee) will maintain communication with primary laboratory.
Laura Maschhoff	Data Validator Subcontractor	DataQual Environmental Services	Validate data received from laboratory prior to data use

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SAP Worksheet #C8—Special Personnel Training Requirements Table

This information is not site-specific; therefore, it is described in the main UFP-SAP.

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SAP Worksheet #C9-1—Project Scoping Session Participants Sheet

Project Name: Site 12 Long-term Monitoring (LTM) **Projected Date(s) of Sampling:** Baseline sampling May/June 2005, performance monitoring at 1,3,6,9,12 months post-injection, and annual monitoring 12 months post-injection **Current Activity Manager (AM):** Cecilia Landin	**Site Name:** Site 12, Exchange Laundry Waste Disposal Area **Site Location:** JEB Little Creek, Virginia Beach, VA
Date of Session: September 22, 2009 **Scoping Session Purpose:** Discuss approach to LTM	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
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Jeff Boylan	RPM	USEPA Region 3	215-814-2094	boylan.jeffrey@epamail.epa.gov	Regulator
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Adina Carver	Deputy AM (at time of meeting)	CH2M HILL	757-671-6213	adina.carver@ch2m.com	Deputy AM
David Livingston	Project Scientist	CH2M HILL	757-671-6239	david.livingston@ch2m.com	Project Scientist

Overview

Presentation material and flow charts were provided and CH2M HILL led the discussion. Before beginning the LTM discussion, CH2M HILL presented the data from the sampling event conducted 3 months following the second substrate injection and noted select contaminant of concern (COC) concentrations exceeded their respective MCLs at the point of compliance wells MW36S and MW36D (screened in the shallow and deep portions of the Columbia aquifer, respectively). The analytical results were sent for data validation. CH2M HILL recommended a conference call to discuss a path forward following the receipt of the validated data. The Team agreed to consult their respective technical experts concerning possible courses of action and provide feedback for the conference call. VDEQ questioned whether the deep aquifer contamination would discharge to the creek or go beneath the creek. CH2M HILL noted this question was previously raised to a hydrogeologist who indicated contamination in both shallow and deep parts of Columbia aquifer was expected to preferentially discharge to the creek and was not expected to travel beneath the creek. CH2M HILL will consult technical experts to confirm. VDEQ suggested the need for a surface water sample in the creek to confirm the groundwater plume is not migrating offsite via the creek. USEPA agreed with the collection of a surface water sample

SAP Worksheet #C9-1—Project Scoping Session Participants Sheet (continued)

and questioned how MCL exceedances at MW36S/D will affect groundwater migration status (offsite). The Team agreed to include this discussion during the Site 12 conference call.

The Team discussed the decision Flow Chart 1 (handout provided during the meeting). The rationale was to sample the source zone (center) and the edge of the plume (periphery) without sampling all of the annual monitoring wells. In the proposed decision tree, some of the monitoring wells designated as annual wells for the first year of post-injection sampling were included, but the annual wells MW01S/D and MW26T were excluded because the wells are up-gradient of the source zone and the analytical results from two rounds of data demonstrate the COC concentrations are below their respective MCLs. MW06T, MW08T, MW27S/D were not included because they were not necessary to characterize the source area or the periphery of the plume. The flow charts include the ability to change the sampling frequency based on analytical results.

In regards to the decision Flow Chart 1 (handout provided during the meeting), the Team agreed to change the phrasing in box 4 to "Is dechlorination occurring?" and box 7 to "Does the data indicate a need for a change in monitoring frequency?" to increase flexibility and allow technical experts to give their opinion based on a number of criteria. To retain flexibility and assess the possibility of an anomaly in relation to a trend analysis the term "consider" was incorporated. The Team agreed to change the phrasing in box 8 to "Continue sampling at predetermined frequency" to address the scenario for when the sampling frequency had already been changed from semiannual to another duration. The change made to box 8 in Flow Chart 1 (meeting handout) was also applied to box 8 in Flow Chart 3 (meeting handout). The Team agreed to USEPA's suggestion to add a note for boxes 3, 5 and 7 in Flow Chart 3 (meeting handout), which allows for the decision to add substrate and/or adjust pH to be completed on a site-wide basis, not well by well. The Team also agreed to change the phrasing in box 9 to "Continue sampling at predetermined frequency" and shift it above box 6 to avoid sampling for dehalococcoides if competing electron acceptors are present. A decision box above box 6 was added to evaluate the remedy.

In regards to Flow Chart 2 (handout provided during the meeting), VDEQ and USEPA questioned if there were sufficient down-gradient wells to adequately characterize plume. The Team agreed to change box 5 to include a decision box. If data is sufficient, box 5 would link to box 8 or box 10. If data is not sufficient, further sampling is needed. CH2M HILL suggested changing box 6 to specify why the up-gradient well is being sampled by changing the box to state, "Has next up-gradient well been sampled to replace down-gradient POC well?" The Team agreed with logic but recommended rewording to clarify.

The Team discussed the decision logic to sequentially eliminate the periphery wells, and replace with the next interior well, when concentrations in the periphery wells were below the MCL. The Navy suggested sampling the outer boundary wells until there are two sampling events below MCLs in a row. On the third sampling event, in addition to the outer boundary well, sample the next up-gradient well. If the outer boundary well is below MCLs and the next up-gradient well is also below MCL, the outer boundary well can be eliminated. VDEQ suggested the concentrations in the down-gradient and next up-gradient

SAP Worksheet #C9-1—Project Scoping Session Participants Sheet (continued)

well should be below MCLs for three sampling events in the down-gradient well independently and then concurrently in the down-gradient and next up-gradient well. The Team questioned whether the proposed sampling frequency was excessive to demonstrate the presence of a point of compliance well. VDEQ suggested if there are no wells directly up-gradient, sampling frequency could be reduced if concentrations are below the MCL. The Team categorized wells MW01S/D, MW03S/D, MW04S, MW09T, MW23D, and MW26T as Plume Boundary Wells to be sampled semiannually. The Team categorized MW 36S and MW36D as a Plume Wells to be sampled quarterly. The Team was uncertain whether Source Well data could solely indicate remedy effectiveness and agreed to reach out for technical support. The Team requested revisions be made to the flow charts provided during the meeting to incorporate the discussion and agreed a conference call is necessary to discuss the MCL exceedances at the POC wells.

Path Forward

The Team will discuss the MCL exceedances at Site 12 monitoring wells MW 36S and MW36D with technical experts to strategize a path forward. A conference call will be set up to discuss these strategies. Additionally, CH2M HILL will revise the flow charts based on Team discussion and the Team will reconvene to discuss the revised decision logic.

Action Items

CH2M HILL: Consult technical experts to determine the potential for the Site 12 plume to migrate beneath the creek.

CH2M HILL: Revise Site 12 groundwater monitoring SAP decision charts based on Team discussion. Consult technical support to determine if sampling the “Source Wells” will provide sufficient information to omit the initial proposed sampling of the “Plume Wells”. Set up a conference call to discuss the next revision of the Site 12 groundwater monitoring SAP.

Navy/USEPA/VDEQ: Consult technical experts to discuss options to address MCL exceedance of COCs at Site 12 monitoring wells MW36S and MW 36D and report these discussions back to the Team to establish the need for a conference call.

USEPA/VDEQ: Consult technical experts to discuss the regulatory ramifications of the MCL exceedance of COCs at Site 12 monitoring wells MW36S and MW36D and the possible migration of contaminants offsite.

USEPA: Determine how the groundwater classification for Site 12 “migration is under control” might change due to the MCL exceedances of COCs at Site 12 monitoring wells MW36S and MW36D.

SAP Worksheet #C9-2—Project Scoping Session Participants Sheet

Project Name: Site 12 LTM **Projected Date(s) of Sampling:** Baseline sampling May/June 2005, performance monitoring at 1, 3, 6, 9, 12 months post-injection, and annual monitoring 12 months post-injection **Current AM:** Cecilia Landin	**Site Name:** Site 12, Exchange Laundry Waste Disposal Area **Site Location:** JEB Little Creek, Virginia Beach, VA
Date of Session: November 19, 2009 **Scoping Session Purpose:** Discuss approach to LTM	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
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Paul Herman	RPM	VDEQ	804-698-4464	peherman@deq.virginia.gov	Regulator
Jamie Butler	AM (at time of meeting)	CH2M HILL	757-671-6212	jamie.butler@ch2m.com	AM
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David Livingston	Project Scientist	CH2M HILL	757-671-6239	david.livingston@ch2m.com	Project Scientist

Overview

Remedial action for the VOC groundwater plume at Site 12, including EOS® injection and performance monitoring, was conducted in 2007 and again in 2009. The LUC RD for Site 12 was completed in 2009 and requires LTM that not only continues performance monitoring of the selected remedy, but also evaluates plume stability and ensures LUC boundary compliance. CH2M HILL led the Site 12 LTM scoping session. Brett Fishwild/CH2M HILL offered technical support via conference call. The Team was presented with a "cheat sheet" outlining the LTM goals and a suggested sampling strategy. Based on the September 2009 scoping session, the overall sampling strategy was changed and new flow charts were created. As a result, some of the language changes proposed in the September 2009 scoping session no longer pertained to the current flow charts. USEPA noted the ROD did not state the explicit purpose of ensuring the groundwater plume is within the LUC boundary (maintaining boundary compliance), but it is implied by the other goals of the ROD. USEPA suggested the LTM plan and sampling results may require the Team to alter the LUC boundary even though the survey is complete and the survey plat will be filed with the City of Virginia Beach.

SAP Worksheet #C9-2—Project Scoping Session Participants Sheet (continued)

During the September 2009 Team meeting, it was noted point of compliance monitoring wells, MW36S/D showed concentrations of COCs exceeding screening criteria (Maximum Contaminant Levels [MCLs]). No monitoring wells lie between MW36S/D and the creek on the western boundary of the site. To evaluate the potential risk associated with groundwater discharging to the creek, the Navy proposed comparing the groundwater analytical results collected from monitoring well MW36D against surface water quality standards (undiluted). The Navy requested to sample the creek for VOCs in surface water only if undiluted groundwater analytical results exceed surface water quality standards. CH2M HILL noted both monitoring well MW36S (shallow) and monitoring well MW36D (deep) are in the same aquifer (Columbia Aquifer) and collecting groundwater results from monitoring well MW36D alone should sufficiently indicate if migration is taking place. USEPA noted if groundwater at monitoring well MW36S is discharging to the creek, then sampling at MW36S may be necessary to assess the creek. CH2M HILL technical experts suggested monitoring well MW36D would serve as the conservative screening location since the higher concentrations and the chemical properties of the COCs would result in the greater likelihood of their presence in the deeper part of the aquifer. The Team agreed with the rationale to only sample monitoring well MW36D as the more conservative sampling point. The Team also agreed if the concentrations of the COCs detected in groundwater collected during the quarterly sampling event(s) exceed the undiluted surface water quality standards, the Team will reconvene to discuss future sampling strategy to ensure protectiveness of human health and the environment.

CH2M HILL confirmed the data from each semiannual sampling will be evaluated, but a revised plume map will not be generated until after annual sampling events produce a more comprehensive picture. The Navy noted the survey plat filed with the City of Virginia Beach would not be updated annually; however, the survey plat states the plume boundary will be updated annually in the Site Management Plan (SMP). The Team agreed with the sufficiency of an annual assessment of the LUC boundary.

Historic data was used to determine migration rates and used to develop the proposed sampling approach. CH2M HILL noted hydrogeologic conditions are relatively stable at the site; however, water levels will be measured during each monitoring event. If changes to groundwater flow rate or direction are observed, the sampling strategy will be adjusted. CH2M HILL suggested monitoring wells MW01S/D and MW26T not be sampled on an annual basis unless there is an indication hydrogeologic conditions have changed or an injection has taken place. These wells may also be sampled during the Five-Year Review event if deemed applicable.

SAP Worksheet #C9-2—Project Scoping Session Participants Sheet (continued)

VDEQ suggested sampling monitoring well MW23D to ensure a clean western boundary. CH2M HILL confirmed monitoring well MW23D had been sampled in 2001 and had no detects of Site 12 COCs; however, no samples had been collected from that well since the sewer line was repaired. VDEQ suggested there is no way to know if upwelling is occurring into the creek and therefore monitoring well MW23D should be included to ensure no VOCs are migrating below the creek and outside the LUC boundary. Based on hydrogeologist evaluation of the site, CH2M HILL concluded all groundwater in the Columbia Aquifer intersects the creek because there is no confining unit restricting upwelling. VDEQ will confer with technical experts to confirm CH2M HILL conclusions.

USEPA suggested the Team acknowledge COCs migrating to the creek will volatilize. Volatilization of COCs is not considered a facet of the remedy, but rather an artifact of site conditions. USEPA questioned if there is a level above which VOC volatilization of COCs would be considered unacceptable. VDEQ suggested the use of ecological risk screening values for surface water to evaluate potential risk to ecological receptors since those screening values would be lower than a value that would lead to unacceptable levels of volatilization; however, the Team agreed CH2M HILL should consult with technical experts to determine the COC concentrations in groundwater that would present a potential risk to ecological receptors due to volatilization.

USEPA suggested explicitly naming monitoring well MW36D as a special Perimeter Well in order to clarify the exit strategy for that well.

CH2M HILL confirmed PCE degradation products (tetracholoroethene [PCE], trichloroethene [TCE], cis- and trans- dicholorethene [DCE] and vinyl chloride [VC]) are all considered Site 12 COCs. The Team collectively revised the decision trees and agreed to the path forward for completion of the Site 12 LTM UFP-SAP.

Path Forward

Writing of the Site 12 LTM UFP-SAP will continue and will incorporate relevant findings from the action items as they are made available.

Action Items

VDEQ: Consult technical experts to determine if groundwater in the Columbia Aquifer can migrate below the creek at Site 12, and therefore, sampling is necessary at monitoring well MW23D.

CH2M HILL: Consult technical experts to determine the concentrations of COCs in groundwater that would present unacceptable risk due to volatilization.

SAP Worksheet #C9-3—Project Scoping Session Participants Sheet

Project Name: Site 12 LTM **Projected Date(s) of Sampling:** Baseline sampling May/June 2005, performance monitoring at 1, 3, 6, 9, 12 months post-injection, and annual monitoring 12 months post-injection **Current AM:** Cecilia Landin	**Site Name:** Site 12, Exchange Laundry Waste Disposal Area **Site Location:** JEB Little Creek, Virginia Beach, VA
Date of Session: January 27, 2010 **Scoping Session Purpose:** Discuss approach to LTM	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
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Overview

Remedial action for the VOC groundwater plume at Site 12, including EOS® injection and performance monitoring, was conducted in 2007 and again in 2009. The LUC RD for Site 12 was completed in 2009 and requires LTM that not only continues performance monitoring of the selected remedy, but also evaluates plume stability and ensures LUC boundary compliance. Previous scoping sessions were conducted in September 2009 and November 2009. CH2M HILL led the discussion and presented the Site 12 UFP-SAP scoping materials; handouts were provided.

USEPA questioned why arsenic sampling was proposed for only one location (MW36D). CH2M HILL noted the well was selected as a downgradient well to ensure arsenic levels demonstrate a decreasing trend as the site is remediated and returns to pre-injection conditions (a more aerobic environment). USEPA requested arsenic be sampled at more than one data point. The Team agreed to analyze samples from three monitoring wells (located down/cross gradient of the plume) for arsenic annually, during the Five-year Review and during post-injection remedy performance monitoring. USEPA suggested box #6 on Decision Tree #2 (handout provided during meeting) should be revised to remove the phrase "and on Five-year Review event" since removal of the well from the monitoring network is only dependent on three consecutive sampling events. The Team agreed with USEPA's suggestion and confirmed the pre-Draft UFP-SAP preparation may move forward.

SAP Worksheet #C9-3—Project Scoping Session Participants Sheet (continued)

Path Forward

The pre-Draft UFP-SAP for LTM at Site 12 will be completed and provided to the Navy Chemist for review.

Action Items

None

SAP Worksheet #C10—Problem Definition

Site Background

JEB Little Creek encompasses 2,215 acres in the northwest corner of Virginia Beach, Virginia, adjacent to the Chesapeake Bay (**Figure C1**). The Site 12 Former Exchange Laundry/Dry Cleaning Facility, was located in the former Building 3323 along Third Street (**Figure C2**) in the eastern portion of the base. Use of the Former Exchange Laundry/Dry Cleaning Facility has resulted in a chlorinated solvent groundwater plume directly south of the commissary (Building 3445).

The laundry/dry cleaning facility operated from 1973 until it was demolished in 1987 for the construction of the existing commissary. During operation, approximately 1,320 gallons of waste including PCE, soap, sizing, and dyes from the laundry/dry cleaning facility were reportedly dumped into storm water lines on site. Of this total, 200 gallons were estimated to be PCE sludge. Site 12 is currently covered by buildings, a self-serve car wash, and asphalt or concrete; a drainage canal runs along the western border of the site (**Figure C2**). A human health risk assessment identified only a potential risk from shallow groundwater contamination, with no potential risk from soil, sediment, or surface water. COCs at the site comprise PCE, TCE, 1,1-dichloroethene (1,1-DCE), cis-1,2-DCE, trans-1,1- DCE, and VC. Unacceptable ecological risks were not identified at Site 12.

When the new commissary was constructed, a passive subsurface venting system was installed under the commissary floor to prevent the possibility of vapors from the VOCs in shallow groundwater from entering into the new building. A ROD was signed for Site 12 in August 2005 documenting in situ chemical oxidation (ISCO)/ERD, groundwater monitoring, and land use controls (LUCs) as the Selected Remedy. However, due to the potential adverse effects of ISCO based on the Site 12 Conceptual Site Model (CSM), an explanation of significant differences (ESD) was signed October 2006 implementing ERD, groundwater monitoring, and LUCs as the Selected Remedy. A LUC Remedial Design (RD) was finalized in 2009. In accordance with the ROD, the following LUC objectives were documented by the LUC RD: prohibit the withdrawal of groundwater except for environmental monitoring and testing; prohibit the use of the site for residential, child care, elementary or secondary school, or playground facilities; and maintain the integrity of any current or future remedial or monitoring system. Remedial action began at the site in February 2007 with the installation of monitoring wells, injection wells, and baseline groundwater sampling. Injection of emulsified vegetable oil product was completed May 2007 and performance monitoring was subsequently conducted. A second injection was performed in 2009.

SAP Worksheet #C10—Problem Definition (continued)

Conceptual Site Model

Hydrogeologic units relevant to this project underlying Site 12 include the Columbia Aquifer and Yorktown Confining Unit. A three dimensional representation of the site and the underlying geology is provided on **Figure C3**. Hydrogeologic characteristics consist of the following:

Columbia Aquifer

Silty sand and sand with an estimated effective porosity of 25 percent. Groundwater is first encountered 6–8 feet below ground surface (bgs) and generally flows to the west-northwest. Horizontal hydraulic gradients calculated from the 2006 Pre-Remedial Action Investigation and the 2007 Remedial Action Work Plan stated a gradient of approximately 0.0017 feet per foot (ft/ft) (**Figure C4**). The saturated thickness at Site 12 is approximately 16–18 ft, and pump test data at Site 12 determined an average hydraulic conductivity of 109 feet/day. Using these values, the estimated seepage groundwater velocity at Site 12 is approximately 222 feet/yr.

Yorktown Confining Unit

A 30 to 40-footthick sandy silt and silty clay layer found at approximately 24 feet bgs. The calculated vertical hydraulic conductivity is low at 3×10-8 centimeters per second. The Yorktown Aquifer is found below this confining unit.

Fate and Transport

- Chlorinated VOCs were released to the surface or near surface in two source areas and percolated downward to the Columbia Aquifer.
- These compounds are currently being transported predominantly laterally through the Columbia Aquifer through groundwater advection.
- The chlorinated VOCs are attenuating through a variety of processes including abiotic degradation, physical processes (dilution, dispersion, volatilization sorption) as well as microbiological degradation via reductive dechlorination.
- The dissolved chlorinated VOCs and daughter products do not appear to have impacted the surface water or sediment in the downgradient drainage canal. Groundwater sampling conducted on the western side of the canal, opposite the site source area, following the Supplemental Remedial Investigation (SRI) did not detect dissolved concentrations of Site 12 COCs.

The Site 12 LUC boundary completely encompasses the lateral extent of the current dissolved COC plumes. Investigations have only identified impacts in the Columbia Aquifer, and have not been found in the Yorktown Aquifer. The groundwater plume exhibits an oblong shape with its longitudinal axis in a general east-west orientation, consistent with groundwater flow patterns (**Figure C3**).

SAP Worksheet #C10—Problem Definition (continued)

Groundwater at Site 12 is not currently, and is not expected to be, used as a potable water supply at or near JEB Little Creek, because of its general poor quality (iron and manganese above secondary MCLs and low yield (generally less than 3 to 5 gallons per minute [gpm]). Potable water is currently, and is expected to continue to be, supplied to the Base by the City of Virginia Beach. However, while groundwater at Site 12 is not currently, nor is it expected to be, used as a potable water supply, the cleanup criteria for Site 12 groundwater are the Safe Drinking Water Act (SDWA) MCLs.

The Remedial Action Objectives (RAOs) for Site 12 groundwater are to:

- Prevent unacceptable risk to human health and the environment from exposure to chlorinated volatile organic compounds (VOCs) in groundwater.
- Reduce concentrations of VOCs in groundwater, to applicable MCLs, using best available technologies.

Project Description

This SAP provides the details for groundwater sampling and analysis to evaluate the size, location, and concentrations of the dissolved COC plumes along with the conditions of the aquifer. The data will be used to determine if the COCs are exceeding screening levels past the point of compliance, if there is a potential for an unreasonable risk to human or ecological receptors, and the extent to which the COCs are decreasing in concentration over time with a goal of achieving the RAOs and LTM exit criteria.

The LTM proposed in this SAP consists of collecting groundwater samples from:

- up to 24 monitoring wells initially (baseline)
- 1 monitoring well quarterly under certain conditions,
- up to 18 monitoring wells semiannually,
- up to 21 monitoring wells annually (inclusive of semiannual wells), and
- up to 24 monitoring wells at each Five-Year Review event and after any additional ERD injections

Groundwater samples will be analyzed for select VOCs, select inorganics, and wet chemistry. The plume is expected to decrease in size overtime, thereby reducing the number of monitoring wells sampled for LTM. The rationale for evaluating the data and revising the list of monitoring wells included in the LTM program is defined in this SAP.

Groundwater remediation of chlorinated VOCs will be achieved through implementation of an ERD remediation system. Land-use controls (LUCs) will be maintained at the site until groundwater concentrations allow for unlimited use and unrestricted exposure (UU/UE). Statutory five-year reviews will be conducted until the RAOs are achieved.

The portion of the Site 12 groundwater plume with total chlorinated VOCs greater than 100 micrograms per liter (µg/L) is limited to the parking lot and car wash area (**Figures C5** (shallow aquifer) and **C6** (deep aquifer)). Maximum concentrations of COCs since 2005 and their respective cleanup levels are provided in **Table C1**.

SAP Worksheet #C10—Problem Definition (continued)

TABLE C1
Site 12 Maximum Concentration Levels for COCs since 2005

COC	Maximum Concentration (µg/L)	Cleanup Level (µg/L)
Tetrachloroethene (PCE)	19,000	5
1,1-DCE	34(J)	7
TCE	2,200	5
VC	5,300	2
cis-1,2-DCE	15,000	70
trans-1,2-DCE	120	100

Note: a J-flag qualifier means the analyte is present, but the reported value may or may not be accurate or precise.

To ensure the downgradient migration of the groundwater plume was adequately characterized, monitoring wells MW36S and MW36D were installed as part of implementation of the remedial action to serve as downgradient point of compliance wells. COCs were not detected at concentrations exceeding their respective MCLs in groundwater samples collected from wells MW36S and MW36D at the time of monitoring well installation and for 12 months following the implementation of the remedial action (March through April 2007). However, 12 months following implementation of the remedial action, select COCs were detected at concentrations slightly exceeding respective MCLs in samples collected from monitoring wells MW36S and MW36D. Additionally, the COC detections at MW-36S and MW36D in 2008 occurred two years after a leaky sanitary sewer manhole and pipe at the car wash was repaired in the spring of 2006. This leaky sanitary sewer was thought to create a localized sink for groundwater flow which affected the groundwater gradient. As MW-36S and MW-36D is downgradient of the sanitary sewer, the repair may have contributed to changes in COC concentrations at the wells. Consequently, this LTM SAP includes the decision criteria for determining whether the Team needs to reconvene to establish a path forward for supplemental monitoring of the downgradient extent of the groundwater plume.

Project Decision Conditions

A remedy has been initiated that includes periodic injections of an organic substrate to stimulate biodegradation of the chlorinated VOCs. LTM is required to ensure the chlorinated VOCs do not migrate in a manner that causes unacceptable risk to human health or the environment and that RAOs achieved. Evaluation of data collecting during sampling will be used to determine whether additional remedy injections are needed, if the monitoring well network should be modified, or if additional actions are needed to change the remedy. The monitoring well network for Site 12 is illustrated on **Figure C7**, The flow diagrams illustrating these decisions processes are captured in **Figures C8 through C10**.

SAP Worksheet #C10—Problem Definition (continued)

Analytical data collected from LTM wells, shown on **Figure C7**, will be used to monitor successful delivery of the substrate and evaluate the performance of the ERD process. The ERD remedy will reduce onsite contaminant mass and prevent plume migration offsite. The usable life of the emulsified vegetable oil (EVO) is estimated at 2–3 years; periodic re-injections of EVO are expected to be required during the course of the remediation.

Environmental questions to be answered by this project

What is the direction of groundwater flow?

Groundwater flow direction will be determined through the use of synoptic water level measurements collected during each sampling event. The shallow and deep monitoring wells are contained in one continuous aquifer (the Columbia Aquifer) with approximately 20 feet of vertical saturated thickness. Therefore, water level measurements collected from all site monitoring wells installed in the Columbia Aquifer, regardless of depth designation, show a similar flow pattern. The Partnering Team may use changes in groundwater flow direction as reason to modify the monitoring well network or the implementation of the remedy.

Have the RAOs been achieved?

Concentrations of site COCs will be compared against MCLs to determine if RAOs have been met. Additionally, after each sampling event, concentrations of site COCs will be assessed to determine if the remedy effectiveness supports achieving RAOs. Concentrations of COCs will be evaluated for general trends, and the aquifer chemistry evaluated to determine if conditions are favorable for reductive dechlorination.

Using the decision processes in **Figures C8 through C10**, one of four possibilities will occur: no change/continue with current monitoring, revise monitoring well network, inject organic substrate for continued remedy effectiveness, or evaluate and establish as necessary changes to the system. Any recommended changes will be discussed by the Partnering Team for implementation during the next sampling event and will be subsequently documented in the next Five-Year Review.

Should the monitoring well network be revised?

During each sampling event, data will be collected on groundwater flow direction and COC concentrations. This data will be evaluated after each event to determine if the monitoring well network can effectively monitor the current and projected groundwater plume. Changes in the network may result from the plume flowing in a different direction or from the groundwater plume changing in size.

Using the decision processes illustrated in **Figures C8 and C9**, one of four possibilities will occur: no change to the network is recommended, the monitoring network is reduced, the monitoring network is changed to different wells, or the network is expanded. Any recommended changes will be presented to the Partnering Team for implementation during the next event and will be subsequently documented in the next Five-Year Review.

SAP Worksheet #C10—Problem Definition (continued)

Is ERD working?

Data from each sampling event will be semi-quantitatively evaluated to determine the effectiveness of the ERD remedy in promoting reductive dechlorination. Data utilized for this evaluation will include COC concentrations, transformation processes of source chemicals to daughter products, and geochemical data representative of aquifer conditions. In general, reductive dechlorination will be considered to be occurring effectively if the parent compound (PCE) exhibits a consistent decreasing trend in conjunction with a consistent decline in daughter products. Daughter product concentrations may temporarily increase as ERD treatment begins but will subsequently decrease with proper ERD treatment. The ERD remedy will be considered complete when concentrations of all COCs, including daughter products, have declined to below screening levels. **Worksheet #C10** illustrates the decision process for determining if reductive dechlorination is occurring and if ERD is effectively reducing COC concentrations at the site.

Is additional sampling warranted to ensure unacceptable concentrations of COCs are not reaching the surface water in the canal?

Since groundwater flow direction is currently toward the west-northwest, there is the potential for COCs in groundwater to enter the surface water at the canal west of the carwash. To assess whether monitoring of surface water is warranted, groundwater samples will be collected from monitoring well MW36D on a quarterly basis until concentrations of site COCs fall below MCLs at this well location. If COC concentrations in samples collected from monitoring well MW36D exceed surface water screening criteria (Table C2), then the Partnering Team will reconvene to discuss if a change in the monitoring approach is appropriate. These screening values reflect a trespasser and maintenance worker scenario for incidental ingestion and dermal contact exposure, Virginia Water Quality Standards (VWQS) for surface waters not designated as public water supplies, and freshwater ecological screening criteria. The complete calculations for trespasser and maintenance worker exposure are provided in Attachment C2.

TABLE C2
Site 12 Surface Water Screening Levels

COC	Human Health		Ecological	
	Trespasser/Maintenance Worker (µg/L)	VWQS (µg/L)	Ecological Screening Value	BTAG Screening Value
PCE	120	33	98	111
1,1-DCE	38,000	7,100	25	25
TCE	36,000	300	47	21
VC	750	24	930	930
cis-1,2-DCE	17,000	Not established	590	590
trans-1,2-DCE	34,000	10,000	590	970

SAP Worksheet #C10—Problem Definition (continued)

Is additional sampling warranted to ensure unacceptable concentrations of COCs are not volatilizing to the ambient air?

One potential pathway for transfer of COCs across media includes volatilization of COCs from groundwater to air. Since the commissary was constructed with a passive vapor barrier, an engineering control is in place to mitigate potential vapor intrusion into the building. There is also the potential for COCs in groundwater to enter the surface water at the canal west of the carwash and for the COCs to subsequently volatilize.

To assess the potential need for further monitoring, an extremely conservative screening level was calculated based on indoor air standards and direct inhalation. An exceedance of the screening level serves only as an indication the Partnering Team needs to reconvene to discuss a more appropriate monitoring approach; it is not intended to be used to represent potential risk, nor is it an accurate representation of potential risk. The screening value will be compared against groundwater data collected from monitoring well MW-36D. This well location was chosen because it is immediately upgradient of the surface water canal and along the longitudinal axis of the dissolved plume. The screening levels for site COCs, based on volatilization from surface water, are provided in Table C3 below. The complete calculations and narrative summary are provided in Attachment C3.

TABLE C3
Site 12 Screening Levels for Volatilization of COCs from Surface Water

Chemical	Risk-based Groundwater Concentration (RBC_{gw}) (micrograms per liter [µg/L])
PCE	200
1,1-DCE	79,000
TCE	600
VC	200
cis-1,2-DCE	23,000
trans-1,2-DCE	23,000

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SAP Worksheet #C11—Project Quality Objectives/Systematic Planning Process Statements

Who will use the data?

Participants from the Tier I Partnering Team (VDEQ, USEPA, Navy, and CH2M HILL) will use the data to evaluate overall remedy effectiveness with respect to plume containment and reduction of VOCs in the source area. Other technically focused disciplines within each organization may use the data as well. Chemists will use the data to evaluate overall data quality with respect to subcontracted laboratories and or changing chemical conditions in the aquifer. Engineers will use the data to evaluate the effectiveness of the remedial action and to make adjustments to the remedial action and the LTM plan as appropriate. Engineers may use the data in designing remedial systems in the future, and geologists may use the data to gain better understanding of aquifer conditions contributing to contaminant fate and transport mechanisms.

What are the Project Action Limits (PALs):

The action levels specified in the ROD are the MCLs unless concentrations over time indicate stabilized conditions and all parties (Navy, USEPA, and VDEQ) agree that chlorinated VOCs have been removed to the extent practicable. The action limits for the COCs are listed in **Worksheet #C10** (**Table C1**) and provided in **Worksheet #C15-1**.

The performance indication levels (PILs) for other chemicals/analytes are provided on **Worksheet #C15-2**. PILs for microbial sampling are provided on **Worksheet #C15-3**. Arsenic and ferrous iron will be analyzed because dissolution of these metals in the aquifer can occur when reducing conditions are induced by the remedy. Monitoring concentrations of these metals will help determine the efficacy of anaerobic biological activity, as well as the state of the aquifer. Other remedy performance indicators (e.g., MEE and TOC) will be monitored to evaluate geochemical conditions and the efficacy of the ERD remedy. **Worksheets #C15-2** and **#C15-3** include the rationale for sampling these performance indicators. Concentrations of remedy performance indicators will not be compared to risk-based screening criteria, but will be judge qualitatively and relative to site-specific conditions.

What will the data be used for?

The overall goals of LTM are to determine if the monitoring scheme for the site is appropriate, if the remedy being employed is effective, and if RAOs have been achieved. Should observations be made such that the data are unable to answer these questions, the monitoring will be modified accordingly. If results from the modified monitoring indicate that one or more of the goals are not being met or that the results against goals still cannot be assessed, the Partnering Team will discuss the remedy and monitoring plan.

SAP Worksheet #C11—Project Quality Objectives/Systematic Planning Process Statements (continued)

What types of data are needed (matrix, target analytes, analytical groups, field screening, onsite analytical or offsite laboratory techniques, sampling techniques)?

Worksheet #C17 contains detailed information on the types of data needed for this project, except for groundwater stabilization parameters which are discussed in the paragraph below. Monitoring well groundwater samples will be collected using a peristaltic pump and low-flow sampling protocol (SOP #004). Samples will be analyzed at an offsite laboratory for COCs, TOC, VFAs, MEE, and total and dissolved arsenic. Microbiological tests (specifically dehalococcoides and the VC-R functional gene), sulfate, sulfide, nitrate, and nitrite will be sampled and analyzed only on an as needed basis.

The gene on the dehalococcoides bacteria that controls its ability to metabolize vinyl chloride is the VC-R (vinyl chloride – reductase) functional gene. The presence of dehalococcoides bacteria is not adequate for confirming that the bacteria required for reductive dechlorination of DCE and VC are present, because not all dehalococcoides are capable of this. However, the confirmation of the presence of the functional gene VC-R (vinyl chloride reductase) confirms that the appropriate bacteria are present and capable of transforming VC to ethane. **Figure C10** provides the rationale for when sampling of the "as needed" parameters is appropriate.

Water quality parameters (pH, ORP, turbidity, specific conductivity, and temperature) will be recorded in the field using a water quality meter (e.g., Horiba U-22 with flow-through cell) using SOP #003. Dissolved oxygen and ferrous iron will be field measured using Chemets and Hach field test kits, respectively. Carbon dioxide and alkalinity will also be measured in the field, on an as-needed basis, using a YSI probe (or analogous) and a Hach test kit, respectively. With the exception of the quarterly sampling events where groundwater samples are collected only from monitoring well MW36D, each sampling event, will include a synoptic static water level survey (SOP #005).

How "good" do the data need to be in order to support the environmental decision?

Laboratory data specified for validation on **Worksheet #C36** will be forwarded to a third party validator for analytical method validation against measurement performance criteria (MPC) contained in this SAP. Level IV package and QC sampling are required for all validated data. QC data requirements are detailed in **Worksheet #C20** and **Worksheets #C28-1 through #C28-8**.

SAP Worksheet #C11—Project Quality Objectives/Systematic Planning Process Statements (continued)

How much data should be collected (number of samples for each analytical group, matrix, and concentration)?

This LTM SAP was prepared to be a dynamic sampling plan, designed to provide the maximum number of initial sampling events and monitoring wells, as well as the decision criteria and flexibility to change the frequency of sampling and the number and/or location of the monitoring wells based on the analytical data from the most recent sampling event. Detailed information on the initial sampling frequency scenarios and the maximum number of monitoring wells included in the sampling scheme is provided on **Worksheet #C17**. The sampling frequency will change based on sampling data as LTM progresses and the list of monitoring wells is expected to diminish over time as the plume decreases. The rationale for the appropriate sampling frequency and changes to the monitoring well scheme over time are provided on this worksheet and the decision trees provided as **Figures C8 through C10**.

Where, when, and how should the data be collected/generated?

Worksheet #C17 and #C18 describes which wells will be sampled under what frequency, and for what parameters. After approval of this SAP, the Initial sampling event will be conducted to collect a baseline of information for the site. This event will include well locations classified as Source, Plume, Perimeter, and Contingency.

Initially, the monitoring well location MW36D is classified as a Contingency well, and will be sampled quarterly (concurrent with any semiannual, annual, post-injection, or Five-Year event) until concentrations of COCs fall below MCLs. Monitoring well locations classified as Source and Plume wells will be sampled semiannually. Every other semiannual event is the annual event and therefore will include the monitoring well locations sampled semiannually as well as the monitoring wells classified as Perimeter Wells. Monitoring wells included in the post-injection monitoring wells scheme include monitoring wells located within the Radius of Influence (ROI) of the injection wells and monitoring wells selected to assess the overall extent of the groundwater plume. The monitoring well network for Site 12 is illustrated on **Figure C7**.

The decision logic to adjust the sampling scheme is presented in **Figures C8 through C10** and, for post-injection monitoring, is discussed in **Worksheet #C17**.

Who will collect and generate the data? How will the data be reported?

CH2M HILL will collect the long term groundwater samples. Data will be generated by Empirical Laboratories, TAL-Tallahassee, and Microbial Insights and validated by DataQual Environmental Services. Following third party validation, the Partnering Team will use the data to evaluate substrate performance, overall remedy effectiveness, and protection of human health and the environment. Monitoring will continue until RAOs are achieved. Following the first 2 years of groundwater monitoring a report will be prepared to document the groundwater monitoring results. The frequency for subsequent reports will be determined by the Partnering Team based on the path forward identified during the initial post-ROD LTM groundwater report.

SAP Worksheet #C11—Project Quality Objectives/Systematic Planning Process Statements (continued)

Data will also be summarized and reported, as appropriate, in the Five-Year Review report. Recommendations for path forward in regards to well network modification will also be included in the Five-Year Review Report.

How will the data be archived?

The data will be downloaded and archived in the Navy Installation Restoration Information System (NIRIS) data base. Hardcopy data will be released to the Navy following completion of the project.

List the Project Quality Objectives (PQOs) in the form of if/then qualitative and quantitative statements.

Quantifiable analytical results will be the primary basis for project decisions. The quantitation limit (QL), which is defined as the minimum level, concentration, or quantity of a target analyte that can be reported with a specified degree of confidence, will be the metric to define whether an analytical result is quantifiable. The Partnering Team agreed upon key decision conditions that will help answer the environmental questions being asked.

The monitoring well network is illustrated on **Figure C7**. The if/then statements are detailed in the form of decision diagrams and are presented in **Figures C8, C9, and C10**.

Analytical COC data collected from the three most recent sampling events will be used for a qualitative approach to trend analysis, as opposed to a statistical approach. Reduction of COC concentrations will be assessed through showing a decreasing (negative slope) trend line. If concentrations of COCs have decreased below all MCLs for 3 consecutive events, the well location is considered eligible for removal or reassignment in the monitoring program. **Figures C8 and C9** present when Source, Plume, and Perimeter Wells can be removed from the monitoring network, or reassigned as a Plume or Perimeter Well. For the Source and Plume wells, the first decision is whether all site COCs have fallen below their respective MCLs. If they have, the monitoring well may be further sampled or removed from the monitoring plan depending on the trend analysis. If the trend analysis indicates an increasing trend or no change, the Partnering Team will reconvene to assess the site.

For Perimeter Wells, the primary criterion is the concentrations for all COCs are below their respective MCLs. If the COC concentrations do not meet this criterion, the Partnering Team will reconvene to discuss a modification to the monitoring plan. If the COC concentrations are below their respective MCLs, depending on the trend analysis the well may be temporarily removed from the monitoring plan and the overall network modified.

Select COCs in groundwater samples collected from monitoring well MW36S and MW36D were detected at concentrations slightly exceeding their respective MCLs at the time this LTM SAP was prepared. Since monitoring wells MW36S and MW36D were initially installed to serve as downgradient point of compliance wells, **Figure C9** includes criteria to designate if the Partnering Team needs to reconvene to further discuss the monitoring approach used to assess the protection of human health and the environment for the downgradient extent of the groundwater plume.

SAP Worksheet #C11—Project Quality Objectives/Systematic Planning Process Statements (continued)

Figure C10 presents a decision process for assessing the effectiveness of the ERD remedy. The decision processes on **Figure C10** will be implemented when sample results, as evaluated on **Figures C8 and C9**, indicate COC concentrations are not reducing over time and the remedy may not be proceeding as planned. The process detailed on **Figure C10** provides the logic to determine if aquifer enhancements are recommended. If the additional enhancements are recommended and implemented and are subsequently determined to be ineffective or if ERD is not determined to be operating as planned, the Partnering Team will reconvene to discuss an appropriate path forward.

The decision processes detailed on **Figures C8 through C10** should be followed after the receipt of each events data, though decisions may not be formalized until the annual groundwater reports or until the next Five-Year Review. However, if at any time changes occur that are deemed time sensitive, the Partnering Team will be notified before the next sampling event.

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SAP Worksheet #C12-1—Measurement Performance Criteria Table – Field QC Samples

Matrix: Groundwater

Analytical Group: Select VOCs

Concentration Level: Low

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	MPC	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
Field Duplicates	Select VOCs	One per 10 field samples	Precision	Relative Percent Difference (RPD) < 25%	S&A
Equipment Rinsate Blank		One per week of sampling	Contamination/bias	All target compounds <1/2 LOQ	S&A
Trip Blank		One per cooler to the laboratory containing VOCs	Contamination/bias		S&A
Ambient Field Blank		One per week of sampling	Contamination/bias		S&A
Cooler Temperature Blank		One per cooler to the laboratory	Accuracy / Representativeness	4 ± 2 ° C	S

SAP Worksheet #C12-2—Measurement Performance Criteria Table – Field QC Samples

Matrix: Groundwater

Analytical Group: Total and Dissolved Arsenic

Concentration Level: Low

QC Sample	Analytical Group	Frequency	DQIs	MPC	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
Field Duplicates	Total and Dissolved Arsenic	One per 10 field samples	Precision	RPD < 25%	S & A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	4 ± 2 ° C	S

SAP Worksheet #C13—Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Supplemental Remedial Investigation	*Final Screening Remedial Investigation - Site 12 Naval Amphibious Base Little Creek.* *CH2M HILL, October 2000*.	CH2M HILL, in agreement with the Navy, USEPA, and the VDEQ completed a SRI in October 2000. The purpose of the SRI was to fill information gaps including the horizontal and vertical extent of the chlorinated VOC plume, VOC source area, aquifer (shallow and deep) characteristics, and conduct a HHRA.	The groundwater analytical data from this investigation may be used in conjunction pre-construction/ baseline data to evaluate temporal concentrations of COCs and performance indicators, and to assist in determining locations for additional monitoring wells (if necessary).	Groundwater data were collected prior to the implementation of the remedial action and may not represent the current extent of contamination present on-site.
Supplemental Remedial Investigation	*Enhanced Reductive Dechlorination Annual Groundwater Monitoring Summary. Naval Amphibious Base Little Creek.* CH2M HILL, August 2009.	CH2M HILL in agreement with the Navy, USEPA, and VDEQ. Collected groundwater samples were collected prior to and 1, 3, 6, 9, and 12 months following the implementation of ERD in May 2007-2008. The groundwater data was analyzed for TCL VOCs, select inorganics, and wet chemistry.	The groundwater analytical data from this investigation will be used to judge the effectiveness of the remedial strategy and determine the optimal array of sampling wells for LTM.	Groundwater data were collected to evaluate the effectiveness of the remedial alternative. The samples were collected over a year ago and may not represent the current concentrations present on-site.

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SAP Worksheet #C14—Summary of Project Tasks

The collection of field data is consistent for all sites and is detailed on **Worksheet #14** in the main SAP. Site-wide synoptic water level gauging will occur before each groundwater sampling event such that all wells in the current monitoring well network will be gauged and recorded within a 24-hour period. Static water levels and total depths will be gauged and recorded immediately prior to purging and sampling of a well, and static water levels gauged and recorded during purging to document low flow procedures. Stabilization parameters will be collected and recorded during purging to document low flow sampling procedures.

The parameters collected for laboratory analysis varies on a site-specific basis. Details regarding sample collection for the analytical parameters for Site 12 are listed on **Worksheet #C17, #C18, and #C19**.

The parameters listed on **Worksheets #C15-2 and C15-3** are to be considered screening data and compared to PILs. They will support intermediate decisions but no final decisions will be made based on them. Therefore, Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) accreditation is not a requirement for these parameters. The COCs will provide definitive data for Site 12.

The SOPs for all laboratory analytical tasks, as well as a detailed list of analyses to be performed, are included in **Worksheet #C23** and the laboratory Standard Operating Procedures (**Attachment C1**).

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SAP Worksheet #C15-1—Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: Select VOCs

Analyte	CAS Number	MCLs[1] (µg/L)	Screening Levels for Incidental Ingestion and Dermal Contact of COCs from Surface Water[2] (µg/L)	Screening Levels for Volatilization of COCs from Surface Water[2] (µg/L)	Project Quantitation Limit (PQL) Goal[3] (µg/L)	Laboratory-specific			MS/MSD and LCS Recovery Limits		
						LOQ (µg/L)	LOD (µg/L)	DL (µg/L)	Lower Limit (%)	Upper Limit (%)	RPD (%)
TCE	79-01-6	5	36,000	600	1.67	1	0.5	0.25	70	125	30
PCE	127-18-4	5	120	200	1.67	1	0.5	0.25	45	150	30
cis-1,2-DCE	156-59-2	70	17,000	23,000	23.33	1	0.5	0.25	70	125	30
trans-1,2-DCE	156-60-5	100	34,000	23,000	33.33	1	0.5	0.25	60	140	30
VC	75-01-4	2	750	200	0.67	1	0.5	0.25	50	145	30
1,1-DCE	540-59-0	70	38,000	79,000	23.33	1	0.5	0.25	70	125	30

[1] Refer to **Worksheets #C10 and #C11** for a detailed discussion on development of PALs.

[2] "Incidental Ingestion and Dermal Contact of COCs from Surface Water" and "Volatilization of COCs from Surface Water" screening values are only for comparison to groundwater collected from location MW-36D.

[3] PQL Goals are 3 times lower than the minimum PAL (which are the MCLs).

SAP Worksheet #C15-2—Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: Various

Analyte	CAS Number	PILs[1] (mg/L)	Effect	Laboratory-specific			MS/MSD and LCS Recovery Limits		
				LOQ (mg/L)	LOD (mg/L)	DL (mg/L)	Lower Limit (%)	Upper Limit (%)	LCS/LCSD and/or MS/MSD RPD (%)
Total Organic Carbon	TOC[2]	> 20	Carbon and energy source; drives dechlorination; can be natural or anthropogenic	1.5	1.0	0.5	80	120	20
Arsenic (total and dissolved)	7440-38-2	> Baseline concentration (from initial event before LTM)**	Concentrations of arsenic elevated above baseline concentrations indicate mobilization due to reducing conditions. Because mobilization of arsenic is undesirable, the PIL is set to ensure that arsenic levels cease to increase following the achievement of the RAOs.	0.003	0.0015	0.00075	80	120	20
Lactic Acid	79-33-4	> 0.1	Intermediates resulting from biodegradation of complex organics; carbon and energy source	10	*	2.2	32	152	43
Propionic Acid	79-09-4	> 0.1		0.5	*	0.093	77	107	20
Butyric Acid	107-92-6	> 0.1		0.5	*	0.16	74	105	20
Pyruvic Acid	127-17-3	> 0.1		2	*	0.21	61	114	26
Acetic Acid	64-19-7	> 0.1		0.5	*	0.24	63	114	20
Methane	74-82-8	> 5.0	These indicate the presence of reductive dechlorination processes.	0.004	0.002	0.001	80	120	30
Ethane	74-84-0	>0.1		0.004	0.002	0.001	80	120	30
Ethene	74-85-1	>0.1		0.004	0.002	0.001	80	120	30
Nitrate	14797-55-8	< 1.0	Reductive pathway possible	0.2	0.1	0.033	90	110	20
Nitrite	14797-65-0			0.2	0.1	0.033	90	110	20
Sulfate	14808-79-8	< 20	At higher concentrations may compete with reductive pathway	2.0	1.0	0.33	90	110	20
Sulfide	18496-25-8	> 1	Reductive pathway possible	3.1	1.5	0.8	80	120	20

[1] PQL Goals are equivalent to the PIL. If no PIL is available, the PQL Goal is equivalent to the LOD.
[2] Contractor-generated CAS number
Compounds in **Worksheet #C15-2** will not be compared to risk-based screening criteria. Instead, they will be compared to the above PILs. These compounds will be analyzed to provide more information about the state of the aquifer. See **Worksheet #C11** for more information.
* = not provided. Subcontracted laboratory is not yet reporting by the DoD QSM version 4.1. For VFA's, LOQ = QL and DL = MDL
** = average value of three well locations specified for arsenic monitoring

SAP Worksheet #C15-3—Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: Dehalococcoides and functional genes

Concentration Level: Medium

Analyte	CAS Number	Laboratory-specific	
		QLs (cells/mL)	MDLs (cells/mL)
Dehalococcoides spp.	DHC	0.5	0.1
Dehalococcoides functional gene (vcrA)	VC-R	0.5	0.1

Dehalococcoides is a dechlorinating microbe noted for its potential use in the bioremediation of PCE and TCE contaminated groundwater sites. Other dechlorinating microbes are also responsible for the PCE → TCE → 1,2-DCE sequence of ethene dechlorination, but these organisms often fail to continue the degradation sequence from 1,2-DCE to VC, and finally, ethene (1,2-DCE → VC → ethene), which is environmentally benign. Dehalococcoides effectively converts 1,2-DCE to VC, and its presence in groundwater may be necessary for proper bioremediation of COCs at Site 12.

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SAP Worksheet #C16—Project Schedule / Timeline Table

Activities	Organization	Dates		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Baseline	CH2M HILL	3 months after SAP approval, once only	Within 1 week	n/a	n/a
Quarterly	CH2M HILL	6 months after SAP approval, then every 3 months*	Within 1 week	n/a	n/a
Semiannual	CH2M HILL	9 months after SAP approval, then every 6 months*	Within 1 week	***	n/a
Annual	CH2M HILL	15 months after SAP approval, then every 12 months*	Within 1 week	***	6 months after sampling event
Five-Year Review	CH2M HILL	1 year before Five-Year Review due date, until Five-year reviews are no longer required.	Within 1 week	Five-Year Review Report	Included in the facility wide Five-Year Review Report
Post-injection Monitoring**	CH2M HILL	1, 3, 6, 9, and/or 12 months after an ERD injection*	Within 1 week	Groundwater monitoring technical memorandum	6 months after 12-month sampling event

* See **Worksheet #C17** for specifics

** Completed only after an injection is performed. See **Worksheet #C17** and **Figure #C10** for scenario resulting in an injection.

*** A groundwater monitoring report will be completed after the first four sampling events defined as the Initial, first Semiannual, the first Annual, and the second Semiannual events. The frequency for additional reports will be dictated by the Partnering Team following receipt and review of the most recent sampling data and/or if additional remedy action is taken (e.g. additional ERD injections). Timeframe indicates submittal of draft reports.

Five-Year Review Reports for JEB Little Creek are completed on a facility wide basis; all sites at JEB Little Creek with a signed ROD requiring action are included in the Five-Year Review Report. The schedule for the JEB Little Creek Five-Year Review reports was initiated by the signature of RODs for JEB Little Creek Site 9 and Site 10 in December 2003.The first Five-Year Review Report was due March 2009. Site 12 will be included in all subsequent five-year reviews, which are to be completed every 5 years thereafter, until RAOs are achieved and No Further Action is warranted at the site.

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SAP Worksheet #C17-1—Site 12 – Post-Remediation Long-Term Monitoring

		Number of wells to be sampled for each standard monitoring parameter			Number of wells to be sampled for each additional "as needed" parameter		
		COCs	**TOC, VFA, MEE**	**Total and dissolved arsenic**	**bacterial**	**sulfate/sulfide, nitrate/nitrite**	
		VOCs			Dehalococcoides		**Wells to be Sampled**
LTM Baseline	**Baseline** (with MW23D)	24	24	3*	--	--	- All wells initially included in LTM program - MW23D located on opposite side of creek - If MW23D has concentrations above MCL, will reconvene with Partnering Team
Until MW36D below MCL	**Quarterly** (between semiannual and annual events)	1	--	--	--	--	- MW36D last downgradient well before stream - Will sample quarterly until concentrations fall below MCLs for three consecutive events
Normal plume monitoring	**Semiannually** (with MW36D)	18	18	--	As needed	As needed	- All Source Wells and Plume Wells - Will collect additional aquifer parameters if dechlorination in question
	Annually (with MW36D)	21	18**	3*	As needed	As needed	- All Source Wells and Plume Wells, select Perimeters Wells - Will only do cross and down-gradient Perimeter Wells unless plume conditions change
	Five-Year Review (with MW23D and MW36D)	24	24	3*	As needed	24	-All wells in LTM program at that time - Initially all designated Source Wells, Plume Wells, and Perimeters Wells - If a monitoring well is below MCLs for 3 consecutive events, can recommend removal from program during Five-Year Review
Post-Injection Remedy Performance Monitoring	**1 month following injection at a minimum**	TBD	TBD	TBD	As needed	As needed	- Frequency of sampling and well network determined by Partnering Team based upon scoped injections

*Arsenic samples will be collected from down/cross gradient monitoring wells

** Perimeter wells are not sampled for TOC, VFA, and MEE in annual sampling event

Note – the wells to be sampled in each event are identified on **Worksheet #C18**

If groundwater concentrations in MW36D rise above surface water screening levels for either volatilization or incidental ingestion/dermal contact, surface water sampling will be discussed with Partnering Team. See **Worksheet #C10** for additional details.

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SAP Worksheet #C17-2—Site 12 – Post-Remediation Long-Term Monitoring

Starting Rationale for Sampling Design

Frequency

The wells that initially fall under the definitions of Source, Plume, Perimeter, and Contingent wells are listed in Worksheet #18. The frequency in which each of these types of wells are sampled is primarily based on the groundwater flow velocity.

Groundwater flows with a linear hydraulic velocity of approximately 18 feet/month across the site. This parameter was used to establish a minimum sampling frequency of a quarterly interval for the Contingent well MW-36D, equating to the approximate time for groundwater to travel between MW-36D and the canal in the downgradient direction. As long as COC concentrations detected in samples collected from monitoring well MW-36D continues to exhibit concentrations above their respective MCLs, monitoring well MW36D will be sampled quarterly.

Well locations classified as Source and Plume Wells will be sampled semiannually, that is a frequency respective of the longer time of travel for groundwater to the downgradient point of compliance and the rate of chemical changes likely to occur (e.g. dilution, degradation, oxidation, etc.).

Alternating semiannual events, which are the annual events, will include the well locations sampled semiannually as well as wells classified as Perimeter Wells. A well can only be classified as a Perimeter Well if COC concentrations in groundwater do not currently exhibit COC concentrations above their respective MCLs. Therefore, it is unlikely COC concentrations would be detected in a Perimeter Well at concentrations exceeding their respective MCLs before an increasing trend is observed in an upgradient well (Plume or Source Well) during a semiannual event.

The monitoring frequency will be increased following an injection to ensure the substrate was adequately distributed in the aquifer, the ERD treatment is progressing appropriately, and the injection did not result in changes to distribution in the groundwater plume that would require changes to the monitoring well network.

Groundwater samples will be collected before each Five-Year Review report to allow the data to be evaluated and discussed by the Partnering Team for inclusion of comments and recommendations for a path forward in the Five-Year Review report.

Locations

The monitoring wells adequately describe the vertical and horizontal extent of the known dissolved COC plume. Therefore, the LTM network was chosen as a subset of the existing well network. Well locations were chosen based on three criteria: locations in close proximity to source areas and having relatively high concentrations (Source Wells), locations around the periphery of source areas which exhibit lower concentrations (Plume Wells), and locations beyond the extent of the current dissolved plume that do not exhibit concentrations above MCL (Perimeter Wells). Perimeter wells were chosen in down, cross, and upgradient directions.

SAP Worksheet #C17-2—Site 12 – Post-Remediation Long-Term Monitoring (continued)

Similarly, the monitoring well scheme established for post-injection monitoring was selected based on the CSM at the time this LTM SAP was prepared. The post-injection monitoring well scheme includes monitoring wells located within the ROI of the injection wells and monitoring wells selected to assess the overall extent of the groundwater plume. If the injections are completed at all injection well locations, 18 monitoring wells are designated for sampling during the 1 month, 3 month, 6 month, and 9 month post-injection sampling events. Seventeen of the monitoring wells are located within the ROI: MW-2S/D, MW-24S/D, MW-27S/D, MW-28S/D, MW-30S/D, MW-31D, MW-32S/D, MW-33S/D, and MW-34S/D. Monitoring well MW-36D is also included in the initial sampling scheme to monitor the downgradient extent of the plume. Twelve months post-injection, six additional monitoring wells (Perimeter Wells: MW-01S/D, MW-04S, MW-07T, MW-23D, and MW-26T) will be included in the sampling scheme. If the extent of the injections is reduced, then the monitoring well scheme will be reduced accordingly. Changes to the post-injection monitoring well network will be discussed by the Partnering Team.

The number of well locations, the well network density, was chosen by a subjective and not a mathematical or statistic method. To maximize cost effectiveness, duplications in well locations in close proximity and with similar historical analytical data were combined. The monitoring and injection well network is illustrated on **Figure C7**. The sampling scheme is presented on **Worksheets #C17 and #C18**. The rationale for changing the sampling scheme is presented in **Figures C8 through C10**.

Analytical Methods

All well locations, at each sampling frequency, are at a minimum sampled for the VOC constituents stipulated in the Record of Decision. The additional analytical parameters of Total Organic Carbon (TOC), VFA, Methane/Ethane/Ethene (MEE), bacterial counts dehalococcoides, nitrate/nitrite, and sulfate/sulfide will be sampled to monitor the effectiveness of the approved remedy. Concentrations of these parameters are less likely to change significantly over short distances and are indications of the general dissolved plume conditions, therefore a smaller set of monitoring wells is sufficient to answer the environmental questions being asked by this project. The decision process for when to sample these parameters is described in **Figure C10**. Arsenic can be mobilized under anaerobic conditions and therefore will be sampled to ensure arsenic concentrations have stabilized or are decreasing as RAOs are achieved.

Exit Criteria:

- Source and Plume Wells: if three consecutive semiannual events document COC concentrations are detected below their respective MCLs, sampling will stop until the next Five-Year Review after which the well will be assessed for re-designation, removal from the monitoring plan, or abandonment.

SAP Worksheet #C17-2—Site 12 – Post-Remediation Long-Term Monitoring (continued)

- Perimeter wells: as Plume Wells are removed from the monitoring well network, those Plume Wells will be assessed for re-designation to Perimeter Well classification. The appropriate downgradient Perimeter Well will then be assessed for removal from the monitoring plan or abandonment.
- Contingency: if COC concentrations detected in groundwater samples at a given location repeatedly exceed their respective MCLs and asymptotic conditions are identified, the partnering Team will reconvene to discuss a modified monitoring frequency. At a minimum, Five-Year Review sampling events will occur until COC are not detected at concentrations exceeding their respective MCLs or a new target level is identified.

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SAP Worksheet #C18—Sampling Locations and Methods/SOP Requirements Table

Site	Matrix	Sample Location	Screening Interval (feet bgs)	Sample ID[1,2]	Sampling Frequency						Sampling SOP Reference	Analytical Group and Corresponding Sampling Frequency[3]				
					LTM Baseline (B)	Quarterly (Q)	Semiannually (SA)	Annually (A)	Five-year Review (FY)	Post-Injection (PI)		Select VOCs	Total and Dissolved Arsenic	Sulfate/ Sulfide Nitrate/Nitrite	TOC/ VFA/ MEE	Bacterial[4]
Source Wells																
Site 12	GW	MW02S	2.8 - 12.8	LS12-MW02S-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12	GW	MW02D	18 - 20	LS12-MW02D-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12	GW	MW32S	7.5 - 15.5	LS12-MW32S-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12	GW	MW32D	17.5 - 25.5	LS12-MW32D-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12	GW	MW33S	6.5 - 14.5	LS12-MW33S-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12 QA/QC- MS/MSD	*GW*	*MW33S*	*6.5 - 14.5*	*LS12-MW33S-MMYY*	*X*		*X*	*X*	*X*	*X*	Worksheet #21, Appendix A	*B,SA, A, FY, PI*		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	
Site 12 QA/QC- MS/MSD	*GW*	*MW33S*	*6.5 - 14.5*	*LS12-MW33S-MMYY*	*X*		*X*	*X*	*X*	*X*	Worksheet #21, Appendix A	*B,SA, A, FY, PI*		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	
Site 12	GW	MW33D	16.5 - 24.5	LS12-MW33D-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12 QA/QC	*GW*	*MW33D*	*16.5 - 24.5*	*LS12-MW33DP-MMYY*	*X*		*X*	*X*	*X*	*X*	Worksheet #21, Appendix A	*B,SA, A, FY, PI*				
Site 12	GW	MW34S	6.5 - 14.5	LS12-MW34S-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12	GW	MW34D	16.5 - 24.5	LS12-MW34D-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Plume Wells																
Site 12	GW	MW24S	6 - 13	LS12-MW24S-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12	GW	MW24D	17 - 25	LS12-MW24D-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12	GW	MW27S	6 - 13	LS12-MW27S-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12	GW	MW27D	17 - 23	LS12-MW27D-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12	GW	MW28S	6.5 - 14.5	LS12-MW28S-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12	GW	MW28D	17.5 - 25.5	LS12-MW28D-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12 QA/QC- MS/MSD	*GW*	*MW28D*	*17.5 - 25.5*	*LS12-MW28D-MMYY*	*X*		*X*	*X*	*X*	*X*	Worksheet #21, Appendix A	*B,SA, A, FY, PI*		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	
Site 12 QA/QC- MS/MSD	*GW*	*MW28D*	*17.5 - 25.5*	*LS12-MW28D-MMYY*	*X*		*X*	*X*	*X*	*X*	Worksheet #21, Appendix A	*B,SA, A, FY, PI*		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	

SAP Worksheet #C18—Sampling Locations and Methods/SOP Requirements Table (continued)

					Sampling Frequency							Analytical Group and Corresponding Sampling Frequency[3]				
Site	Matrix	Sample Location	Screening Interval (feet bgs)	Sample ID[1,2]	LTM Baseline (B)	Quarterly (Q)	Semiannually (SA)	Annually (A)	Five-year Review (FY)	Post-Injection (PI)	Sampling SOP Reference	Select VOCs	Total and Dissolved Arsenic	Sulfate/ Sulfide Nitrate/Nitrite	TOC/ VFA/ MEE	Bacterial[4]
Site 12	GW	MW30S	7.5 - 15.5	LS12-MW30S-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12 QA/QC	*GW*	*MW30S*	*7.5 - 15.5*	*LS12-MW30SP-MMYY*	*X*		*X*	*X*	*X*	*X*	Worksheet #21, Appendix A	B,SA, A, FY, PI				
Site 12	GW	MW30D	17.5 - 25.5	LS12-MW30D-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Site 12	GW	MW31D	16 - 24	LS12-MW31D-MMYY	X		X	X	X	X	Worksheet #21, Appendix A	B,SA, A, FY, PI		FY; then SA, A, PI As Needed	B,SA, A, FY, PI	SA, A, FY, PI (As Needed)
Perimeter Wells																
Site 12	GW	MW01S	2.8 - 12.8	LS12-MW01S-MMYY	X				X	X	Worksheet #21, Appendix A	B, FY, PI		FY; then PI As Needed	B, FY, PI	FY, PI (As Needed)
Site 12	GW	MW01D	18.5 - 26.5	LS12-MW01D-MMYY	X			X	X	X	Worksheet #21, Appendix A	B, A, FY, PI		FY; then PI As Needed	B, FY, PI	FY, PI (As Needed)
Site 12	GW	MW04S	2.2 - 12.2	LS12-MW04S-MMYY	X			X	X	X	Worksheet #21, Appendix A	B, A, FY, PI	B, A, FY, PI	FY; then A and PI As Needed	B, FY, PI	A, FY, PI (As Needed)
Site 12	GW	MW07T	4 - 24	LS12-MW07T-MMYY	X			X	X	X	Worksheet #21, Appendix A	B, A, FY, PI	B, A, FY, PI	FY; then A and PI As Needed	B, FY, PI	A, FY, PI (As Needed)
Site 12 QA/QC	*GW*	*MW07T*	*4 - 24*	*LS12-MW07TP-MMYY*	*X*			*X*	*X*	*X*	Worksheet #21, Appendix A	B, A, FY, PI	*B, A, FY, PI*			
Site 12	GW	MW23D	Unknown	LS12-MW23D-MMYY	X			X	X	X	Worksheet #21, Appendix A	B, A, FY, PI	B, A, FY, PI	FY; then A and PI As Needed	B, FY, PI	A, FY, PI (As Needed)
Site 12	GW	MW26T	6 - 21	LS12-MW26T-MMYY	X				X	X	Worksheet #21, Appendix A	B, FY, PI		FY; then A As Needed	B, FY, PI	FY, PI (As Needed)
Contingent Wells																
Site 12	GW	MW36D	7 - 15	LS12-MW36D-MMYY	X	X	X	X	X	X	Worksheet #21, Appendix A	All Events		FY; then SA, A, PI As Needed	B, SA, A, FY, PI	SA, A, FY, PI (As Needed)
Non-Well Location QA/QC Samples[5]																
QA/QC	*GW*	*N/A*	*N/A*	*LS12-EB01-MMDDYY*	*X*	*X*	*X*	*X*	*X*	*X*	Worksheet #21, Appendix A	*All Events*				
QA/QC	*GW*			*LS12-FB01-MMDDYY*	*X*	*X*	*X*	*X*	*X*	*X*	Worksheet #21, Appendix A	*All Events*				
QA/QC	*GW*			*LS12-TB01-MMDDYY*	*X*	*X*	*X*	*X*	*X*	*X*	Worksheet #21, Appendix A	*All Events*				

QA/QC are dependent on the total number of samples collected and therefore will be adjusted as the number of wells included in the monitoring well network is adjusted. QA/QC sample IDs include additional identifiers: Spike; P = Duplicate; EB = Equipment Blank; FB = Field Blank; TB = Trip Blank.

[1] MMYY will be replaced by the 2-digit month and 2-digit year that the sample is collected; MMDDYY will be replaced by the 2-digit month, 2-digit day, and 2-digit year that the sample is collected.

[2] For EB, FB, and TB non-well location QA/QC samples, the actual number of equipment, field, and trip blanks to be collected will be based upon the number of days it takes to perform sampling.

[3] B, Q, SA, A, FY, and PI correspond to the sampling events listed under Sampling Frequency.

[4] Well locations and frequency of bacterial samples for dehalococcoides to be determined per **Figure C11**.

[5] QA/QC samples consisting of Duplicates and MS/MSD are identified with sample locations for nomenclature purposes only. The actual number and location of these samples may vary between sampling events.

SAP Worksheet #C19—Analytical SOP Requirements Table

Matrix		Analytical and Preparation Method/ SOP Reference[1]	Containers (Number, Size, and Type)	Sample Volume	Preservation Requirements (Chemical, Temperature, Light Protected)	Maximum Holding Time (Preparation/ Analysis)[2]
Groundwater	Select VOCs	SW-846 8260B/ SOP-202	3 x 40mL VOA vials	25 mL	HCl to pH < 2, Cool to 4 ± 2°C	14 days
	Methane, Ethane, Ethene (MEE)	USEPA RSK-175 / SOP-236	2 x 40 mL VOA vials	40 mL		14 days
	Total and Dissolved Arsenic	SW-846 6010B/ SOP-100, SOP-105	250mL poly	50 mL	HNO3 to a pH<2; Cool to 4°C	180 days
	Nitrate, Nitrite, Sulfate	USEPA 300.0/ SOP-145	250mL poly	50 mL	Cool to 4 ± 2° C	48 hours - nitrate, nitrite/ 28 days - sulfate
	Sulfide	SM 4500 S-2F/ SOP-153	250mL poly	250 mL	2N Zn Acetate, 6 N NaOH, Cool to 4 ± 2° C	7 days
	VFAs	SOP TL-SG-043	1 x 40mL amber VOA vial	40 mL	Cool to 4 ± 2° C	28 days
	TOC	SW-846 9060/ SOP-221	250mL poly	5 mL	H_2SO_4 or HCl to pH 2	28 days
	Dehalococcoides and functional genes	qPCR/ DNA-qPCR	1 L	1 L	Cool to 4 ± 2° C	24-48 hours to extraction, 1 year to analysis (frozen at -20°C)

[1] See **Worksheet #C23**

[2] Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted. (Not VTSR)

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SAP Worksheet #C20—Field Quality Control Sample Summary Table

The frequency of sampling is based on all initial sampling frequency scenarios. The sampling frequency will change based on sampling data as LTM progresses. This Worksheet contains information for all initial sampling frequencies identified. This Worksheet also contains sample counts for the initial maximum numbers of wells included in the sampling scheme; the list of monitoring wells is expected to diminish over time as the plume decreases. The rationale for the appropriate s sampling frequency and changes to the monitoring well scheme over time are provided in **Worksheet #C11** and the decision trees in **Figures C8 through C10**.

Matrix	Analytical Group	Maximum No. of Sampling Locations	No. of Field Duplicates (one per 10 samples)[1]	No. of MS/MSDs[2] (one per 20 samples)[1]	No. of Field Blanks (one per week)[1]	No. of Trip Blanks (one per cooler that contains VOC samples)[1]	No. of Equip. Blanks (one per week)[1]	Maximum Total No. of Samples to Lab
Site 12 Site-wide LTM Baseline Sampling								
Groundwater	Select VOCs	24	3	2/2	2	6	2	41
	MEE	24		2/2				28
	Total and Dissolved Arsenic	3	1	1/1				6
	VFAs	24		2/2				28
	TOC	24		2/2				28
Site 12 Quarterly Sampling (between semiannual and annual events)								
Groundwater	Select VOCs	1	1	1/1	1	1	1	7
Site 12 Semiannual Plume Monitoring								
Groundwater	Select VOCs	18	2	2/2	1	5	1	31
	MEE	18		1/1				20
	VFAs	18		1/1				20
	TOC	18		1/1				20
	Sulfate	TBD		TBD[2]				TBD[3]
	Sulfide	TBD		TBD[2]				TBD[3]
	Nitrate	TBD		TBD[2]				TBD[3]
	Nitrite	TBD		TBD[2]				TBD[3]
	DHC	TBD						TBD[3]
	VC-R	TBD						TBD[3]

SAP Worksheet #C20—Field Quality Control Sample Summary Table (continued)

Matrix	Analytical Group	Maximum No. of Sampling Locations	No. of Field Duplicates (one per 10 samples)[1]	No. of MS/MSDs[2] (one per 20 samples)[1]	No. of Field Blanks (one per week)[1]	No. of Trip Blanks (one per cooler that contains VOC samples)[1]	No. of Equip. Blanks (one per week)[1]	Maximum Total No. of Samples to Lab
Site 12 Annual Plume Monitoring								
Groundwater	Select VOCs	21	3	2/2	1	5	1	37
	MEE	18		2/2				22
	VFAs	18		2/2				22
	TOC	18		2/2				22
	Total and Dissolved Arsenic	3	1	1/1				6
	Sulfate	TBD		TBD[2]				TBD[3]
	Sulfide	TBD		TBD[2]				TBD[3]
	Nitrate	TBD		TBD[2]				TBD[3]
	Nitrite	TBD		TBD[2]				TBD[3]
	DHC	TBD						TBD[3]
	VC-R	TBD						TBD[3]
Site 12 Five-Year Review Plume Monitoring								
Groundwater	Select VOCs	24	3	2/2	2	6	2	41
	MEE	24		2/2				28
	VFAs	24		2/2				28
	TOC	24		2/2				28
	Total and Dissolved Arsenic	3	1	1/1				6
	Sulfate	24		2/2				28
	Sulfide	24		2/2				28
	Nitrate	24		2/2				28
	Nitrite	24		2/2				28
	DHC	TBD						TBD[3]
	VC-R	TBD						TBD[3]

SAP Worksheet #C20—Field Quality Control Sample Summary Table (continued)

Matrix	Analytical Group	Maximum No. of Sampling Locations	No. of Field Duplicates (one per 10 samples)[1]	No. of MS/MSDs[2] (one per 20 samples)[1]	No. of Field Blanks (one per week)[1]	No. of Trip Blanks (one per cooler that contains VOC samples)[1]	No. of Equip. Blanks (one per week)[1]	Maximum Total No. of Samples to Lab
Site 12 One, Three, Six, Nine-Month Post-Injection Remedy Performance Monitoring Sample Counts are for one event								
Groundwater	Select VOCs	24	3	2/2	2	6	2	41
	MEE	24		2/2				28
	VFAs	24		2/2				28
	TOC	24		2/2				28
	Sulfate	TBD		TBD[2]				TBD[3]
	Sulfide	TBD		TBD[2]				TBD[3]
	Nitrate	TBD		TBD[2]				TBD[3]
	Nitrite	TBD		TBD[2]				TBD[3]
	DHC	TBD						TBD[3]
	VC-R	TBD						TBD[3]
Site 12 Twelve-Month Post-Injection Remedy Performance Monitoring								
Groundwater	Select VOCs	24	3	2/2	2	6	2	41
	MEE	24		2/2				28
	VFAs	24		2/2				28
	TOC	24		2/2				28
	Total and Dissolved Arsenic	3	1	1/1				6
	Sulfate	TBD		TBD[2]				TBD[3]
	Sulfide	TBD		TBD[2]				TBD[3]
	Nitrate	TBD		TBD[2]				TBD[3]
	Nitrite	TBD		TBD[2]				TBD[3]
	DHC	TBD						TBD[3]
	VC-R	TBD						TBD[3]

[1]QA/QC samples are being collected only for VOCs. The number of QA/QC samples required is based on the number of samples collected; therefore, as the number of monitoring wells sampled for LTM decreases over time, the number of QA/QC will be adjusted accordingly

[2] MS/MSDs are collected as triple volume from sampling locations but are not considered to be separate samples. They are included in the total number of samples in order to count the total amount of sample volume sent to the laboratory.

[3] To be determined. The project Team will evaluate the need for these parameters as the sampling event approaches. For decision criteria, see **Worksheet #C11** and the decision trees in **Figures C8 though C10** for more information.

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SAP Worksheet #C21—Project Sampling SOP References Table

This information is not site-specific; therefore, it is described in the main UFP-SAP.

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SAP Worksheet #C22—Field Equipment Calibration, Maintenance, Testing, and Inspection Table

This information is not site-specific; therefore, it is described in the main text of the UFP-SAP.

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SAP Worksheet #C23—Analytical SOP References Table

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work (Y/N)
SOP-202	*GC/MS Volatiles by USEPA Method 624 and SW846 Method 8260B,* Rev 18 09/30/09	Definitive	Select VOCs	Gas Chromatograph (GC)/ Mass Spectrometer (MS)	Empirical	N
SOP-236	*Methane, Ethane, Ethene in Aqueous Samples by Modified RSK-175* (Automated Headspace) Rev 1 04/28/09	Screening	MEE	GC/ Flame Ionization Detector (FID)		
SOP-100	*Metals Digestion/Preparation Methods USEPA SW846 3005A,3010A,3030C,3031,3050B USEPA CLPILM 04.1 Aqueous and Soil/Sediment (NJDEP does not accept CLPILM 04.1 after June 2003) Addendum for USEPA CLPILM 05,2 Aqueous & Soil/Sediment USEPA Methods for Chemical Analysis of Water and Wastes 200.7, Standard Methods 3030C Rev 19* 04/20/09	Screening	Total and Dissolved Arsenic	None		
SOP-105	*Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICPAES) Technique Rev. 16*, 04/11/2010	Screening	Total and Dissolved Arsenic	Inductively Coupled Plasma (ICP)-Atomic Emission Spectrometer (AES)		
SOP-145	*Determination of Inorganic Anions in water by ION Chromatography using Dionex DX-500 Ion Chromatograph with Hydroxide Eluent And Dionex Column AS18, Method 300.0 Guidance Rev 7* 3/25/10	Screening	Nitrate, Nitrite, Sulfate	Ion Chromatograph (IC)		
SOP-153	*Sulfide Method 376.1 and Standard Methods SM4500S-2 F(19th ED) (Titrimetric, Iodine) with Sample Pretreatment to Remove Interfering Substances or to Concentrate the Sulfide Rev* 3 5/27/09	Screening	Sulfide	Spectrophotometer		

SAP Worksheet #C23—Analytical SOP References Table (continued)

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work (Y/N)
SOP-221	*Total Organic Carbon SM5310C, USEPA Method 415.1 and SW846 Method 9060/9060A and Lloyd Kahn Method* Rev 8 4/28/09	Screening	TOC	TOC Analyzer	Empirical	N
SOP-404	*Sample Receipt, Handling and Processing* Rev. 13 06/29/09	n/a	Login and Storage	n/a		
SOP-405	*Analytical Laboratory Waste Disposal* Rev. 5 06/23/09		Waste Disposal	n/a		
SOP-410	*Standard Operating Procedures for Laboratory Sample Storage, Secure Areas and Sample Custody* Rev. 7 06/23/09		Login and Storage	n/a		
TL-SG-043	*The Analysis of Volatile Fatty Acide by DAI-GC/FID, Rev. 5,* Effective Date 4/28/2008	Screening	VFAs	GC/FID	TAL Tallahassee	
MI-SAMREC	*Sample Receiving,* 11/14/08, rev 1.1	n/a	n/a	n/a	Microbial Insights	
DNA-EXT	*Extraction of DNA from Environmental Samples,* 1/5/06, rev 1	Screening	Groundwater/ DHC and Functional genes	n/a		
DNA-qPCR	*Quantitative Polymerase Chain Reaction (qPCR),* 1/10/06, rev1			ABI 7000		
SOP-ABI 7000	*Exporting Results from the ABI 7000 Instrument,* 6/2/06, rev1			ABI 7000		
SOP-Calculating qPCR	*Calculating qPCR Results,* 8/14/06, rev1			n/a		

SAP Worksheet #C24—Analytical Instrument Calibration Table

The MPC presented in **Worksheet #C24** is based on those presented in the DoD QSM version 4.1. If MPCs are not provided in the DoD QSM, then laboratory internal criteria were applied.

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/FID (VFAs)	Initial Calibration (ICAL)	Five point calibration. Prior to analysis.	RSD<20% or R^2 >0.990	Correct problem and repeat ICAL.	Analyst/Laboratory Manager	TL-SG-043
	Initial Calibration Verification (ICV)	Once after each ICAL, prior to analytical sequence	All target analytes within ± 30 % of the true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.		
	Continuing Calibration Verification (CCV)	Perform after every 12 hours or 20 samples, whichever is more frequent and at the end of the analysis sequence.	All target analytes within ± 20% of the true value. For internal standard analyses, the capping standard does not need to pass; for external standard analyses, the capping standard must pass.	Correct problem. Then rerun calibration verification. If that fails, repeat ICAL and reanalyze all samples run since last acceptable CCV		
GC/MS (VOCs)	Tuning	Prior to ICAL and at the beginning of 12-hour analytical sequence	Must meet the ion abundance criteria required by the method. See SOP 202 section 13.4.	Retune instrument and verify; rerun affected samples.	Analyst/Laboratory Manager	SOP-202
	ICAL	After major instrument maintenance; prior to sample analysis (five points required for linear calibration and six points required for quadratic calibration)	1. Average response factor (RF) for VOC system performance check compounds (SPCCs), if target analytes, ≥0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. Relative standard deviation (RSD) for calibration check compounds (CCCs), if target analytes, for VOCs ≤ 30%; For all other analytes, RSD for each analyte ≤ 15%, linear least squares regression r ≥ 0.995 or squared correlation coefficient r^2 ≥ 0.990	Correct problem then repeat ICAL		
	ICV	After each ICAL (second source)	All target analytes within ± 20% of the true value	Correct problem and verify second source standard; prepare new standard(s) if that fails and repeat ICAL.		
	CCV	Daily before sample analyses and once per 12-hour analysis period	1. Average RF for VOC SPCCs, if target analytes, ≥0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane 2. % difference/drift for all target analytes ≤20%.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.		
	Retention time window position establishment	For each target analyte and surrogate; once per ICAL	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	N/A		
	Evaluation of relative retention time (RRT)	With each sample	RRT of each target analyte within +/- 0.05 RRT units	Correct problem then rerun CCV and/or ICAL		
GC/FID (MEE)	ICAL	After major instrument maintenance; upon 2nd consecutive failure of CCV standard (five-point calibration)	RSD between the calibration factors must be ≤ 25%	Recalibrate	Analyst/Laboratory Manager	SOP-236
	ICV	After each ICAL (second source)	Run at midpoint of the curve. All target analytes within ± 20% of the true value.	Correct problem and verify second source standard; prepare new standard(s) if that fails and repeat ICAL.		
	CCV	At the beginning of the analytical sequence; after each 10 field samples; at the end of the analytical sequence	%D between the calibration factors in the CCV and the average calibration factors must be ≤25%	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.		

SAP Worksheet #C24—Analytical Instrument Calibration Table (continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
ICP-AES (Total and Dissolved Arsenic)	ICAL	Daily before sample analyses (three-point calibration)	R ≥ 0.995	Correct problem, then repeat ICAL	Analyst/Laboratory Manager	SOP-100, SOP-105
	ICV (second source)	Perform each analytical sequence, after ICAL.	The percent recovery of all analytes must be 90-110%.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.		
	Initial Calibration Blank (ICB)	Once after each ICAL, prior to analytical sequence	All analyte concentration in the ICB must be ≤ LOD.	Correct problem and repeat ICAL.		
	Interference Check Solution (ICS)	Once after each ICAL, prior to analytical sequence	The results of the ICS must agree within ± 20% of the accepted values. The absolute value of the concentration of all the non-spiked analytes in the ICS-A must be < LOD (unless the analyte is a verified trace impurity from one of the spiked analytes).	Recalibrate the instrument. If re-calibration does not fix the problem, shut down the instrument and perform instrument maintenance and recalibrate.		
	Continuing Calibration Blank (CCB)	Every 10 samples and at the end of the analytical sequence	All analyte concentration in the ICB must be ≤ LOD.	Correct problem. Then rerun calibration verification. If that fails, repeat ICAL and reanalyze all samples run since last acceptable CCV		
	Low level check standard	Daily, after ICAL	80-120% recovery	Correct problem, reanalyze		
	CCV	Perform after each 10 samples and at the end of the analysis sequence.	The percent recovery of all analytes must be 90-110%.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable continuing calibration		
IC (Nitrate, Nitrite, Sulfate)	ICAL	Daily before analysis	R ≥ 0.995	Correct problem and repeat ICAL.	Analyst/Laboratory Manager	SOP-145
	ICB	Once after each ICAL, prior to analytical sequence	Target analytes less than the QL.	Correct problem and repeat ICAL.		
	ICV	Once after each ICAL, prior to analytical sequence	The percent recovery of all analytes must be 90-110%.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.		
	CCB	Every 10 samples and at the end of the analytical sequence	Target analytes ≤ 1/2 QL.	Correct problem. Then rerun calibration verification. If that fails, repeat ICAL and reanalyze all samples run since last acceptable CCV		
	CCV	Perform after each 10 samples and at the end of the analysis sequence.	The percent recovery of all analytes must be 90-110%.	Correct problem. Then rerun calibration verification. If that fails, repeat ICAL and reanalyze all samples run since last acceptable CCV		
Spectrophotometer (Sulfide)	ICAL	Quarterly	R ≥ 0.995	Correct problem and repeat ICAL.	Analyst/Laboratory Manager	SOP-153
	ICV	Once after each ICAL, prior to analytical sequence	The percent recovery of all analytes must be 90-110%.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.		
	CCV	Perform after each 10 samples and at the end of the analysis sequence.	The percent recovery of all analytes must be 90-110%.	Correct problem. Then rerun calibration verification. If that fails, repeat ICAL and reanalyze all samples run since last acceptable CCV		

SAP Worksheet #C24—Analytical Instrument Calibration Table (continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
TOC analyzer (TOC)	ICAL	Quarterly	R ≥ 0.995	Correct problem and repeat ICAL.	Analyst/Laboratory Manager	SOP-221
	ICV/CCV	Beginning and end of analysis and every 10 samples	All target analytes within ± 15% of the true value.	Reprepare and reanalyze. If it fails again, repeat ICAL.		
ABI 7000 (DHC and Functional genes)	Dye Calibration	Semi Annual	Meets manufacturer's specifications for each dye	Contact ABI service Rep	Analyst/Laboratory Manager	DNA-qPCR
	Initial Assay Calibration (standard curve)	Once per assay	Standard curve R2 >0.95	Rerun and/or optimize assay		
	CCV	Primary- every 4 months	Standard curve R2 >0.95	Rerun assay/ check reagents		
	CCV	Secondary- every plate (assay)	CT value within 2 units of same point on standard curve	Rerun assay/ check reagents		

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SAP Worksheet #C25—Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference
GC/MS (VOCs)	Clean the source and replace the filaments. Replace the seal, liner and septum. Change the column.	VOCs	Check the gas supply. Check the seal, liner, and septum.	Source cleaning is performed when the instrument response deteriorates. Other instrument maintenance is done as needed to keep the instrument performing at peak performance.	Must meet initial and/or continuing calibration criteria. See **Worksheet #24**.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Laboratory Manager	SOP-202
GC/FID (MEE)	Check pressure and gas supply daily. Bake out column, change septa, liner, seal as needed, cut column as needed.	MEE	Liner, seal, septum, column	Prior to initial calibration or as necessary				SOP-236
ICP-AES (Total and Dissolved Arsenic)	Clean the torch assembly and the spray chamber when they become discolored or when degradation in data quality is observed. Clean the nebulizer, and check the argon supply. Replace the peristaltic pump tubing as needed	Metals	Inspect the torch, nebulizer chamber, pump, and tubing	Maintenance is performed prior to ICAL or as necessary.				SOP-105
IC (Nitrate, Nitrite, Sulfate)	Replace column	Anions	Check gas supply, check for leaks, check pistons	Daily or as needed				SOP-145
Spectrophotometer (Sulfide)	Clean reagent tubes. Change lamp.	Sulfide	Check wavelength	At the beginning of every run.				SOP-153
TOC analyzer (TOC)	Replace sample tubing, clean sample boat, replace syringe	TOC	Tubing, sample boat, syringe	As needed		Repeat maintenance activity or remove from service.		SOP-221
GC/FID (VFAs)	1. Clean autosampler syringe and tubing		Inspect daily	1. As needed		Repeat maintenance activity or remove from service.		TL-SG-043
	2. Change septa			2. Daily				
	3. Inspect/change gas tank			3. As needed, when pressure is <500 psi				

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SAP Worksheet #C26—Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FTL and Field Team Members/ CH2M HILL
Sample Packaging (Personnel/Organization): FTL and Field Team Members/ CH2M HILL
Coordination of Shipment (Personnel/Organization): FTL and Field Team Members/ CH2M HILL
Type of Shipment/Carrier: Overnight/ FedEx
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Empirical, Microbial Insights, and TAL-Tallahassee personnel
Sample Custody and Storage (Personnel/Organization): Empirical, Microbial Insights, and TAL-Tallahassee personnel
Sample Preparation (Personnel/Organization): Empirical, Microbial Insights, and TAL-Tallahassee personnel
Sample Determinative Analysis (Personnel/Organization): Empirical, Microbial Insights, and TAL-Tallahassee personnel
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 90 days from receipt
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 1 year
Biological Sample Storage (No. of days from sample collection): n/a
SAMPLE DISPOSAL
Personnel/Organization: Empirical, Microbial Insights, and TAL-Tallahassee personnel
Number of Days from Analysis: After submission, the laboratory will keep samples 90 days and the sample extracts for a minimum of 60 days.

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SAP Worksheet #C27—Sample Custody Requirements Table

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Samples will be collected by field Team members under the supervision of the FTL. As samples are collected, they will be placed into containers and labeled. Labels will be taped to the jar to ensure they do not separate. Samples will be cushioned with packaging material and placed into coolers containing enough ice to keep the samples 4 ± 2 degrees Celsius until they are received by the laboratory. The chain-of-custody will be placed into the cooler in a Ziploc bag. Coolers will be taped up and shipped to the laboratories via Fed Ex overnight, with the air bill number indicated on the COC (to relinquish custody). Upon delivery, the laboratory will log in each cooler and report the status of the samples to CH2M HILL. See **Worksheet #21** for SOPs containing sample custody guidance. The CH2M HILL field Team will ship environmental samples to Empirical. Empirical will ship VFA samples to TAL-Tallahassee. All DHC/ functional genes samples will be shipped directly to Microbial Insights.
Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): Laboratory custody procedures can be found in the following SOP, which is referenced in **Worksheet #C23** and can be found in Attachment C1 of this SAP: SOP-404
Sample Identification Procedures: Sample labels will include, at a minimum, client name, site, sample ID, date/time collected, analysis group or method, preservation, and sampler's initials. The field logbook will identify the sample ID with the location and time collected and the parameters requested. The laboratory will assign each field sample a laboratory sample ID based on information in the chain-of-custody. The laboratory will send sample log-in forms to the EIS to check that sample IDs and parameters are correct.
Chain-of-custody Procedures: Chain of custodies will include, at minimum, laboratory contact information, client contact information, sample information, and relinquished by/received by information. Sample information will include sample ID. Date/time collected, number and type of containers, preservative information, analysis method, and comments. The chain-of-custody will link location of the sample from the field logbook to the laboratory receipt of the sample. The laboratory will use the sample information to populate the Laboratory Information Management Systems (LIMS) database for each sample.

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SAP Worksheet #C28-1—Laboratory QC Samples Table

The MPC presented in Worksheet #C28 is based on those presented in the DoD QSM version 4.1. If MPCs are not provided in the DoD QSM, then laboratory internal criteria were applied.

Matrix	Groundwater
Analytical Group	Select VOCs
Analytical Method/ SOP Reference	SW846 8260B/ SOP-202

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One is performed for each batch of up to 20 samples.	Target analytes must be < ½ LOQ or <1/10 the concentration found in the sample	Re-prepare and analyze all associated samples. Discuss with client/qualify if re-extraction/re-analysis not feasible.	Analyst/ Laboratory Area Supervisor	Contamination/ Bias	Target analytes must be < ½ LOQ or <1/10 the concentration found in the sample
Laboratory Control Sample (LCS)		Contains all target analytes. Percent recoveries must meet the control limits listed in **Worksheet #15-1**. No marginal exceedances are allowed.			Accuracy/Bias	Contains all target analytes. Percent recoveries must meet the control limits listed in **Worksheet #15-1**. No marginal exceedances are allowed.
MS/MSD		Percent recoveries must meet the control limits and RPD listed in **Worksheet #15-1**	Examine results of LCS. Report and narrate.		Precision / Accuracy / Bias	Percent recoveries must meet the control limits and RPD listed in **Worksheet #15-1**

SAP Worksheet #C28-1—Laboratory QC Samples Table (continued)

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Internal Standards (IS)	Every field sample, standard, and QC sample	Retention times for internal standards must be + 30 seconds from retention time of the midpoint standard in the ICAL and the responses within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Analyst/ Laboratory Area Supervisor	Precision / Accuracy / Bias	Retention times for internal standards must be + 30 seconds from retention time of the midpoint standard in the ICAL and the responses within -50% to +100% of ICAL midpoint standard.
Surrogates	All field and QC samples.	Percent recoveries: Dibromofluoromethane 85-115%; 1,2-dichloroethane-d4 70-120%; Toluene-d8 85-120%; 4-Bromofluorobenzene 75-120%	Re-prepare and reanalyze all failed samples in the associated preparatory batch for confirmation of matrix interference. Discuss with client/qualify if re-extraction/re-analysis not feasible.		Accuracy / Bias	Percent recoveries: Dibromofluoromethane 85-115%; 1,2-dichloroethane-d4 70-120%; Toluene-d8 85-120%; 4-Bromofluorobenzene 75-120%

SAP Worksheet #C28-2—Laboratory QC Samples Table

Matrix	Groundwater
Analytical Group	MEE
Analytical Method/ SOP Reference	RSK-175/ SOP-236

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One is performed for each batch of up to 20 samples.	Target analytes must be < ½ LOQ	Re-prepare and analyze all associated samples. Discuss with client/qualify if re-extraction/re-analysis not feasible.	Analyst/Laboratory Manager	Contamination/ Bias	Target analytes must be < ½ LOQ
LCS		Contains all target analytes. See **Worksheet #15-2**			Accuracy/Bias	Contains all target analytes. See **Worksheet #15-2**
MS/MSD		Percent recoveries must meet the control limits and RPD listed in Worksheet #15-2	Examine results of LCS. Report and narrate.		Precision / Accuracy / Bias	Percent recoveries must meet the control limits and RPD listed in **Worksheet #15-2**

SAP Worksheet #C28-3—Laboratory QC Samples Table

Matrix	Groundwater
Analytical Group	Total and Dissolved Arsenic
Analytical Method/ SOP Reference	SW846 6010B/ SOP-100, SOP-105

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One is performed for each batch of up to 20 samples.	Target analytes must be < ½ LOQ or <1/10 the concentration found in the sample	Re-prepare and analyze all associated samples. Discuss with client/qualify if re-extraction/re-analysis not feasible.	Analyst/ Laboratory Area Supervisor	Contamination/ Bias	Target analytes must be < ½ LOQ or <1/10 the concentration found in the sample
LCS		Percent recoveries must meet the control limits listed in Worksheet #15-2			Accuracy/Bias	Percent recoveries must meet the control limits listed in Worksheet #15-2.
MS/MSD		Percent recoveries must meet the control limits and RPD listed in **Worksheet #15-2**	Examine results of LCS. Report and narrate.		Precision / Accuracy / Bias	Percent recoveries must meet the control limits and RPD listed in Worksheet #15-2
Serial Dilution	One is performed for each preparation batch with sample concentration(s) > 50x MDL	The five-fold dilution result must agree within ±10% of the original sample result.	Perform Post Digestion Spike		Precision / Accuracy	The five-fold dilution result must agree within ± 10% of the original sample result.
Post Digestion Spike	One is performed when serial dilution fails or analyte concentration(s) in all samples < 50x MDL.	The result must agree within ± 25% of expected result.	Run all associate sample in the preparatory batch by method of standard additions or qualify results.		Precision / Accuracy	The result must agree within ± 25% of expected result.

SAP Worksheet #C28-4—Laboratory QC Samples Table

Matrix	Groundwater
Analytical Group	Nitrate, Nitrite, Sulfate
Analytical Method/ SOP Reference	USEPA 300.0/ SOP-145

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One is performed for each batch of up to 20 samples.	Target analytes must be < ½ LOQ or <1/10 the concentration found in the sample	Reprepare and reanalyze any associated samples	Analyst/Laboratory Manager	Contamination/ Bias	Target analytes must be < ½ LOQ or <1/10 the concentration found in the sample
LCS		Percent recoveries must meet the control limits listed in **Worksheet #15-2**.			Accuracy/Bias	Percent recoveries must meet the control limits listed in **Worksheet #15-2.**

SAP Worksheet #C28-5—Laboratory QC Samples Table

Matrix	Groundwater
Analytical Group	Sulfide
Analytical Method/ SOP Reference	SM 4500-S2F/ SOP-153

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One is performed for each batch of up to 20 samples.	Target analytes must be < ½ LOQ or <1/10 the concentration found in the sample	Re-prepare and analyze all associated samples. Discuss with client/qualify if re-extraction/re-analysis not feasible.	Analyst/Laboratory Manager	Contamination/ Bias	Target analytes must be < ½ LOQ or <1/10 the concentration found in the sample
LCS/ LCSD		Percent recoveries and RPDs must meet the control limits listed in Worksheet #15-2.			Accuracy/ Bias/ Precision	Percent recoveries and RPDs must meet the control limits listed in Worksheet #15-2.

SAP Worksheet #C28-6—Laboratory QC Samples Table

Matrix	Groundwater
Analytical Group	VFAs
Analytical Method/ SOP Reference	TL-SG-043

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One is performed for each batch of up to 20 samples.	Target analytes must be < ½ LOQ or <1/10 the concentration found in the sample	Re-prepare and analyze all associated samples. Discuss with client/qualify if re-extraction/re-analysis not feasible.	Analyst/Laboratory Manager	Contamination/ Bias	Target analytes must be < ½ LOQ or <1/10 the concentration found in the sample
LCS/LCSD		Contains all target analytes. See **Worksheet #15-2**			Precision / Accuracy / Bias	Contains all target analytes. See **Worksheet #15-2**
MS/MSD		Percent recoveries must meet the control limits and RPD listed in **Worksheet #15-2**	Examine results of LCS. Report and narrate.		Precision / Accuracy / Bias	Percent recoveries must meet the control limits and RPD listed in **Worksheet #15-2**

SAP Worksheet #C28-7—Laboratory QC Samples Table

Matrix Groundwater

Analytical Group TOC

Analytical Method/ SOP Reference SW846 9060/ SOP-221

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One is performed for each batch of up to 20 samples.	Target analytes must be < ½ LOQ or <1/10 the concentration found in the sample	Re-prepare and analyze all associated samples. Discuss with client/qualify if re-extraction/re-analysis not feasible.	Analyst/Laboratory Manager	Contamination/ Bias	Target analytes must be < ½ LOQ or <1/10 the concentration found in the sample
Lab duplicate		RPD < 20%			Precision/ Accuracy	RPD < 20%
MS/ MSD		See **Worksheet #15-2**	Examine results of LCS. Report and narrate.		Accuracy/ Bias/ Precision	See **Worksheet #15-2**
LCS		See **Worksheet #15-2**	Re-prepare and analyze all associated samples. Discuss with client/qualify if re-extraction/re-analysis not feasible.		Accuracy/Bias	See **Worksheet #15-2**

SAP Worksheet #C28-8—Laboratory QC Samples Table

Matrix Groundwater

Analytical Group Dehalococcoides plus functional genes

Analytical Method/ SOP Reference Polymerase Chain Reaction/ DNA-qPCR

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Laboratory (sample) Duplicate	All field samples	CT value within 2 units of other duplicate	Rerun assay; if still not within 2 CT units, flag J (estimated)	Analyst/Laboratory Manager	Precision/ Accuracy	CT value within 2 units of other duplicate
Assay negative control (Blank)	1 per analytical assay plate	values for positive samples are set above any fluorescence for the negative control	Rerun assay; may have to reoptimize assay		Contamination/ Bias	values for positive samples are set above any fluorescence for the negative control
DNA extraction negative control		CT ≤ Assay negative control	Rerun assay or reextract samples if problem persists		Contamination/ Bias	CT ≤ Assay negative control
Positive control		CT value within 2 units of same point on standard curve	Rerun assay/ check reagents		Contamination/ Bias	CT value within 2 units of same point on standard curve

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SAP Worksheet #C29—Project Documents and Records Table

This information is not site-specific; therefore, it is described in the main UFP-SAP.

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SAP Worksheet #C30—Analytical Services Table

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization	Backup Laboratory / Organization[1]
Groundwater	Select VOCs	See **Worksheet #18**	SW-846 8260B	28 calendar days	Empirical Laboratories Sonya Gordon 621 Mainstream Drive, Suite 270, Nashville, TN 37228 615-345-1115	TBD
	Total and Dissolved Arsenic		SW-846 6010B			
	MEE		RSK-175			
	Nitrate, Nitrite, Sulfate		USEPA 300.0			
	Sulfide		SM 4500-S-2F			
	TOC		SW-846 9060			
	VFAs		Lab SOP		Test America Laboratories-Tallahassee Abbie Yant 2846 Industrial Plaza Dr. Tallahassee, FL 32301 912-354-7858	
	DHC, VC-R		qPCR		Microbial Insights Anita Biernacki 2340 Stock Creek Blvd Rockford, TN 37853 865-573-8188	

[1] A backup laboratory has not been determined. If circumstances render the subcontracted laboratory unable to perform analytical services, another laboratory will be determined at that time.

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SAP Worksheet #C31—Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing CA	Person(s) Responsible for Monitoring Effectiveness of CA
Field Performance Audit	Once	Internal	CH2M HILL	Program Field Auditor	Field Team Leader	Adrienne Jones, PM, CH2M HILL	Brett Doerr, CH2M HILL
Laboratory Technical Systems Audit[1]	Laboratories must have current DoD ELAP accreditation letter which will identify the period of performance. The laboratories must be re-evaluated prior to expiration of period of performance	External	Third party accrediting bodies	TBD, Third party accrediting bodies	Empirical Laboratory Marcia McGinnity, Quality Assurance Officer (QAO)	Empirical Laboratory Marcia McGinnity, QAO	Anita Dodson, Program Chemist, CH2M HILL
Safe Work Observation	One during sampling activities	Internal	CH2M HILL	Site Safety Coordinator CH2M HILL	Field Team Members observed CH2M HILL	Mark Orman H&S Manager CH2M HILL	Site Safety Coordinator CH2M HILL

[1] This applies to laboratories performing analysis of definitive data only. Microbial Insights and TAL-Tallahassee will be generating screening data for this project; therefore, DoD ELAP accreditation is not a requirement.

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SAP Worksheet #C32—Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings	Timeframe of Notification	Nature of CA Response Documentation	Individual(s) Receiving CA Response	Timeframe for Response
Field Performance Audit	Checklist and Written Audit Report	Adrienne Jones, CH2M HILL	Within 1 week of audit	Memorandum	TBD	Within 1 week of receipt of CA Form
Laboratory Performance and Systems Audits	Written Audit Report	Empirical Laboratory Marcia McGinnity, QAO	Within 2 months of audit	Memorandum	Third Party Auditor, TBD	Within 2 months of receipt of initial notification.

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SAP Worksheet #C33—QA Management Reports Table

This information is not site-specific; therefore, it is described in the main text of the UFP-SAP.

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SAP Worksheet #C34—Verification (Step I) Process Table

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Planning Documents	Evidence of approval and completeness of UFP-SAP.	Internal	Adrienne Jones (CH2M HILL)
Chain-of-custody and shipping forms	Chain-of-custody forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the chain-of-custody will be initialed by the reviewer, a copy of the chain-of-custody retained in the site file, and the original and remaining copies taped inside the cooler for shipment. See **Attachment 1, SOP-009** for further details.	Internal	FTL, Victoria Brynildsen (CH2M HILL)
Field Log Notebooks	Field notes will be reviewed to ensure completeness of field data parameters, shipping information,sample collection times, etc. The logbook will also be used to document, explain, and justify all deviations from the approved UFP-SAP.	Internal	FTL, Cecilia Landin (CH2M HILL)
Laboratory Receipt	Upon their arrival at the laboratory, the samples will be cross-referenced against the chain-of-custody records. All sample labels will be checked against the chain-of-custody, and any mislabeling will be identified, investigated, and corrected. The samples will be logged in at every storage area and work station required by the designated analyses. Individual analysts will verify the completeness and accuracy of the data recorded on the forms.	External	Sonya Gordon (Empirical Laboratory) Abbie Yant (TAL-Tallahassee) Anita Biernacki (Microbial Insights)
QC Summary Report	A summary of all QC sample results will be verified for completeness once the data is received from the laboratory.	Internal	Victoria Brynildsen (CH2M HILL)
Laboratory Data Package	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. All received data packages will be examined by an EIS and a Chemist according to the procedures specified in the Navy CLEAN Data Management Plan (Appendix B).	Internal	Sonya Gordon (Empirical Laboratory) Abbie Yant (TAL-Tallahassee) Anita Biernacki (Microbial Insights) Victoria Brynildsen (CH2M HILL) Megan Morrison (CH2M HILL)

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SAP Worksheet #C35—Validation (Steps IIa and IIb) Process Table

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation
IIb	Onsite Screening **Worksheet #22** **Appendix C Worksheet # C11, C17, C18, and C20** Fieldbook	Ensure that all field data meet UFP-SAP requirements for completeness and accuracy based on the field calibration records.	FTL (CH2M HILL)
IIa	SOPs **Worksheet #21** **Appendix C Worksheet #C19** Fieldbook, and Laboratory Report	Ensure that all sampling and analytical SOPs were followed.	FTL (CH2M HILL) Empirical, TAL-Tallahassee, Microbial Insights employees
IIa	Method QC Results **Appendix C Worksheet #C24, and C28** and Laboratory Report	Ensure that all required QC samples were run and meet method and/or project required limits.	Laura Maschhoff (DataQual)
IIb	QC Sample Results **Appendix C Worksheet #C12 and C20** Laboratory Report	Ensure that all required QC samples were run and meet required limits.	Megan Morrison (CH2M HILL) Laura Maschhoff (DataQual)
IIb	QLs **Appendix C Worksheet #C15** Laboratory Report	Ensure all sample results met the project quantification limit specified in the UFP-SAP.	Cecilia Landin (CH2M HILL) Megan Morrison (CH2M HILL)
IIa	Raw Data Laboratory Report	Ten percent review of raw data to confirm laboratory calculations	Laura Maschhoff (DataQual)

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SAP Worksheet #C36—Analytical Data Validation (Steps IIa and IIb) Summary Table

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator
IIa	Groundwater	Select VOCs	Analytical methods and laboratory SOPs as presented in this SAP will be used to evaluate compliance against QA/QC criteria. Should adherence to QA/QC criteria yield deficiencies, data may be qualified. The data qualifiers that may be used are those presented in *Region III Modification to National Functional Guidelines for Organic Data Review, Multi-Media, Multi Concentration (*September 1994). National Functional Guidelines will not be used for data validation; however, the specific qualifiers listed therein may be applied to data should non-conformances against the QA/QC criteria as presented in this SAP be identified.	Laura Maschhoff (DataQual)
IIa		DHC and VC-R, MEE, Total and Dissolved Arsenic, Nitrate, Nitrite, Sulfate, Sulfide, VFAs, TOC	Data will be reviewed against the analytical methods for outstanding QA/QC issues and anomalies by the laboratory. Issues will be summarized in the case narrative. CH2M HILL chemist and PM will review the analytical results and case narrative before the data is loaded to ensure no major problems exist.	Empirical, TAL-Tallahassee, Microbial Insights employees Megan Morrison (CH2M HILL) Cecilia Landin (CH2M HILL)
IIb	Groundwater	Select VOCs, DHC and VC-R, MEE, Total and Dissolved Arsenic, Nitrate, Nitrite, Sulfate, Sulfide, VFAs, TOC	See PALs in **Worksheet #C15**; See Method calibration and QC criteria in **Worksheets #C24 and #C28**.	Megan Morrison (CH2M HILL) Cecilia Landin (CH2M HILL)

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SAP Worksheet #C37— Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:

- Non-detected site contaminants will be evaluated to ensure that project required QLs in **Worksheet #C15** were achieved. If PQLs were achieved and the verification and validation steps yielded acceptable data, then the data is considered usable.
- During verification and validation steps, data may be qualified as estimated with the following qualifiers: J, UJ, K, L, or UL. These qualifiers represent minor QC deficiencies which will not affect the usability of the data. When major QC deficiencies are encountered, data will be qualified with an R and in most cases is not considered usable for project decisions.
  - J- Analyte present. Reported value may or may not be accurate or precise
  - UJ- Analyte not detected. QL may be inaccurate or imprecise
  - K- Analyte present. Reported value may be biased high. Actual value is expected to be lower
  - L- Analyte present. Reported value may be biased low. Actual value is expected to be higher
  - UL- Analyte not detected. QL is probably higher.
  - R- Rejected result. Result not reliable.
- Additional qualifiers that may be given by the validator are:
  - B- Not detected substantially above the level reported in laboratory or field blanks (less than 5 times the concentration in the blanks)
  - I- Interferences present which may cause the results to be biased high
  - N- Tentative Identification. Consider Present. Special methods may be needed to confirm its presence or absence in future sampling efforts
  - NJ- Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity
  - U- Not Detected
- For statistical comparisons non-detect values will be represented by a concentration equal to one-half the sample reporting limit. For duplicate sample results, the most conservative value will be used for project decisions.
- Analytical data will be checked to ensure the values and any qualifiers are appropriately transferred to the electronic database. These checks include comparison of hardcopy data and qualifiers to the electronic data deliverable. Once the data has been uploaded into the electronic database, another check will be performed to ensure all results were loaded accurately.

SAP Worksheet #C37—Usability Assessment (continued)

- Field and laboratory precision will be compared as RPD between the two results.
- Deviations from the SAP will be reviewed to assess whether CA is warranted and to assess impacts to achievement of project objectives.

Describe the evaluative procedures used to assess overall measurement error associated with the project.

- To assess whether a sufficient quantity of acceptable data are available for decision making, the data will be reconciled with MPC following validation and review of DQI.
- If significant biases are detected with laboratory QA/QC samples it will be evaluated to assess impact on decision making. Low biases will be described in greater detail as they represent a possible inability to detect compounds that may be present at the site.
- If significant deviations are noted between lab and field precision the cause will be further evaluated to assess impact on decision making.

Describe the documentation that will be generated during the usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

The following will be prepared by CH2M HILL and presented to and submitted to the Tier I Partnering Team (VDEQ, USEPA, Navy, and CH2M HILL) for review and decisions on the path forward for the site.

- Data tables will be produced to reflect detected and non-detected constituents. Data qualifiers will be reflected in the tables and discussed in the data quality evaluation.
- Plume maps will be produced to reflect areas of groundwater contamination if present.
- A data quality evaluation considering all of the above will be provided as part of presentations to the Tier I Partnering Team (VDEQ, USEPA, Navy, and CH2M HILL), followed by the Groundwater monitoring technical memorandum to assess nature and extent of contamination and make recommendation for further investigation/action if warranted. The Groundwater monitoring technical memorandum will identify any data usability limitations and make recommendations for CA if necessary.

Identify the personnel responsible for performing the usability assessment.

The CH2M HILL Team, including the PMs and VC, will review the data and compile a presentation for the Partnering Team. The Tier I Partnering Team (VDEQ, USEPA, Navy, and CH2M HILL) as a whole will assess the usability of the data.

Figures

\\NORTHEND\PROJ\USNAVFACENGCOM\LITTLECREEK\MAPFILES\345721_SITE12_SAP\FIGURE C1 - SITE 12 LOCATION MAP.MXD CBOWMAN 12/28/2010 09:28:51

Legend

- Installation Boundary
- Groundwater Plume Treatment Area

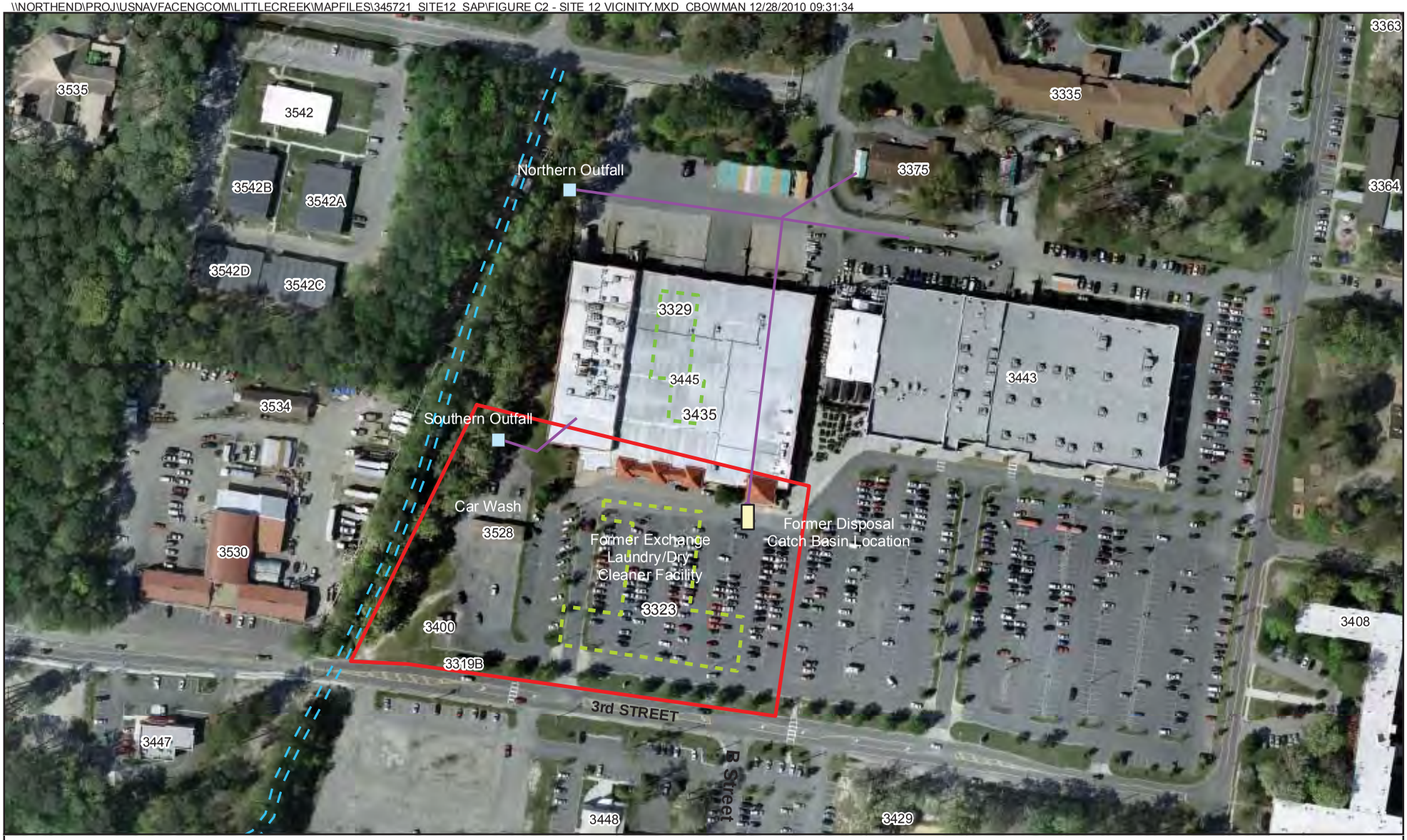
N
W E
S

0 1,100 2,200 4,400 Feet

Figure C1
Site 12 Location
Post-ROD LTM SAP for Sites with Groundwater Impacted by Chlorinated Organic Compounds
JEB Little Creek
Virginia Beach, Virginia

CH2MHILL

\\NORTHEND\PROJ\USNAVFACENGCOM\LITTLECREEK\MAPFILES\345721_SITE12_SAP\FIGURE C2 - SITE 12 VICINITY.MXD CBOWMAN 12/28/2010 09:31:34



Legend

- Outfall Location
- Canal
- Former Storm Water Line
- Land Use Control Boundary
- Former Buildings
- Former Disposal Catch Basin Location

N W E S

0 100 200 Feet

Note: Storm Sewer was removed during construction of the commissary (1987 - 1992).

Figure C2
Site 12 Boundary and Vicinity
Post-ROD LTM SAP for Sites with Groundwater Impacted by Chlorinated Organic Compounds
JEB Little Creek
Virginia Beach, Virginia

CH2MHILL

FIGURE C3
Site 12 Conceptual Site Model
ost M or ites it Groun ater mpa te b
lorinate rgani ompoun s
E ittle ree
irginia ea , irginia

ES082009019MKE LittleCreekSite12_3-D_CSM_v6.ai 01-18-09 mlb

CH2MHILL

\\NORTHEND\PROJ\USNAVFACENGCOM\LITTLECREEK\MAPFILES\345721_SITE12_SAP\FIGURE C4 - SITE 12 POTMAP.MXD CBOWMAN 12/28/2010 09:34:26

Legend

- Monitoring Well Locaiton
- Groundwater Contour (March 2006)
- Inferred Groundwater Contour
- Canal
- Land Use Control Boundary
- Groundwater Flow

All elevations in feet mean sea level.

N
W E
S

0 50 100 Feet

Figure C4
Site 12 Potentiometric Surface (March 2006)
Post-ROD LTM SAP for Sites with Groundwater Impacted by Chlorinated Organic Compounds
JEB Little Creek
Virginia Beach, Virginia

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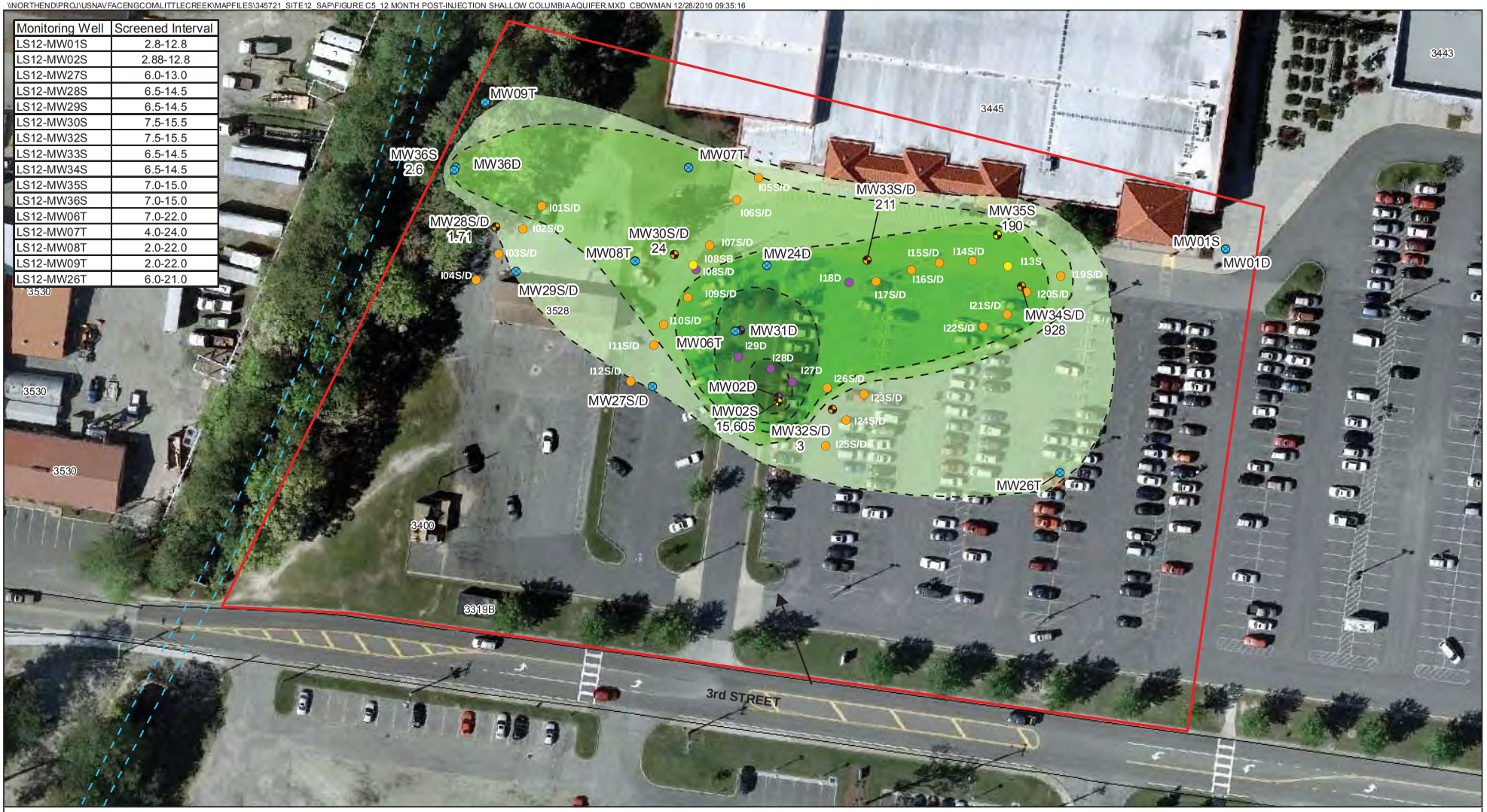
\\NORTHEND\PROJ\USNAVFACENGCOM\LITTLECREEK\MAPFILES\345721_SITE12_SAP\FIGURE C5_12 MONTH POST-INJECTION SHALLOW COLUMBIA AQUIFER.MXD CBOWMAN 12/28/2010 09:35:16

Monitoring Well	Screened Interval
LS12-MW01S	2.8-12.8
LS12-MW02S	2.88-12.8
LS12-MW27S	6.0-13.0
LS12-MW28S	6.5-14.5
LS12-MW29S	6.5-14.5
LS12-MW30S	7.5-15.5
LS12-MW32S	7.5-15.5
LS12-MW33S	6.5-14.5
LS12-MW34S	6.5-14.5
LS12-MW35S	7.0-15.0
LS12-MW36S	7.0-15.0
LS12-MW06T	7.0-22.0
LS12-MW07T	4.0-24.0
LS12-MW08T	2.0-22.0
LS12-MW09T	2.0-22.0
LS12-MW26T	6.0-21.0



Legend

- Land Use Control Boundary
- Canal
- Groundwater Flow

Annual Monitoring Wells
(includes Performance Monitoring Wells)

- Monitoring Wells

Shallow Isoconcentrations (May 2008)

- 1-10
- 10-100
- 100-1000
- 1000-10000
- >10,000
- Shallow Isoconcentration
- Shallow Isoconcentration (Inferred)

Performance Monitoring Wells

- Co-located
- Deep
- Shallow

Injection Wells

- Co-located
- Deep
- Shallow

N W E S

0 30 60 Feet

Concentrations are in µg/L

Figure C5
Shallow Aquifer Total COC Isoconcentration Map
Post-ROD LTM SAP for Sites with Groundwater Impacted by Chlorinated Organic Compounds
JEB Little Creek
Virginia Beach, Virginia

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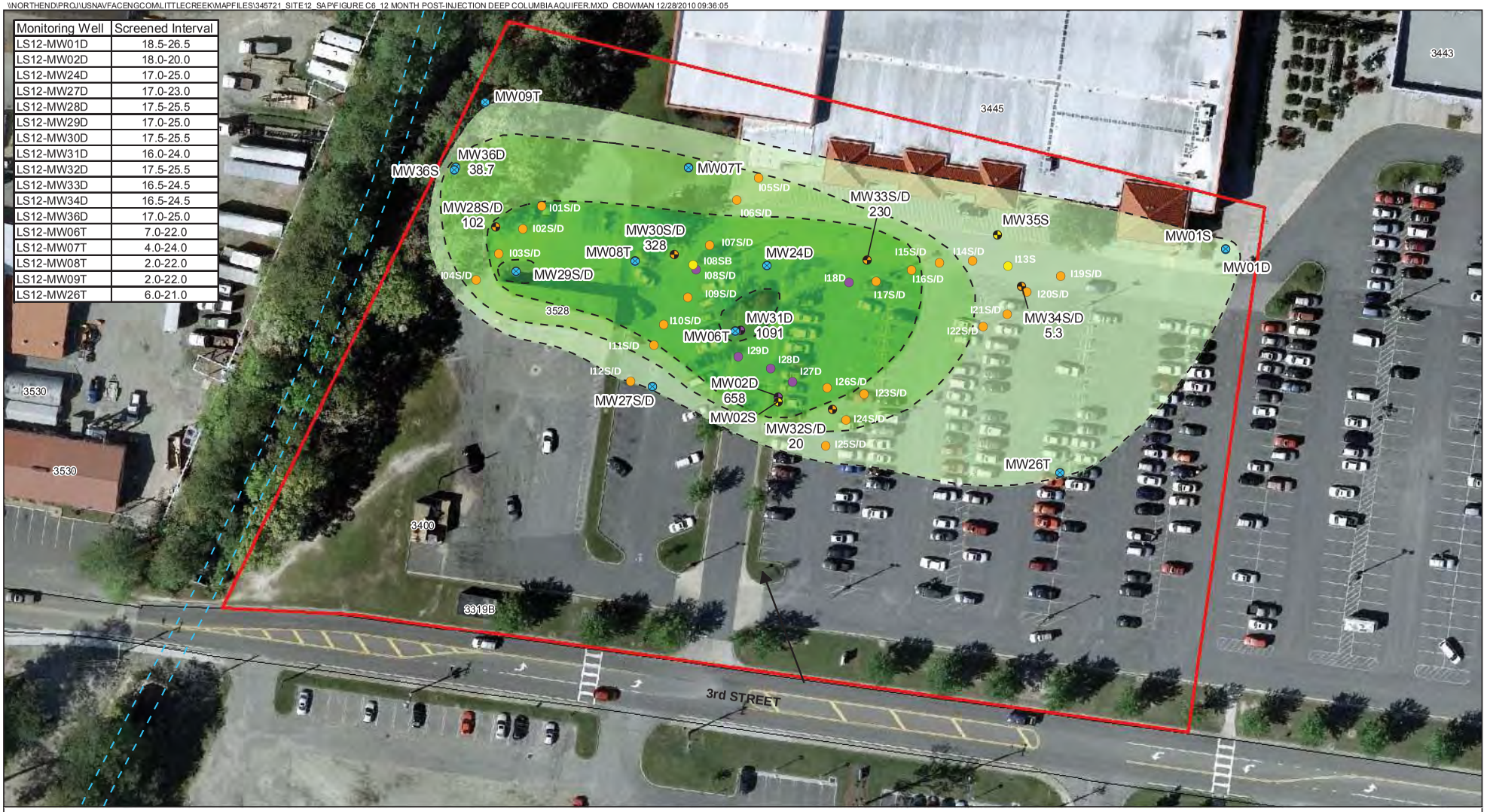
\\NORTHEND\PROJ\USNAVFACENGCOM\LITTLECREEK\MAPFILES\345721_SITE12_SAP\FIGURE C6_12 MONTH POST-INJECTION DEEP COLUMBIA AQUIFER.MXD CBOWMAN 12/28/2010 09:36:05

Monitoring Well	Screened Interval
LS12-MW01D	18.5-26.5
LS12-MW02D	18.0-20.0
LS12-MW24D	17.0-25.0
LS12-MW27D	17.0-23.0
LS12-MW28D	17.5-25.5
LS12-MW29D	17.0-25.0
LS12-MW30D	17.5-25.5
LS12-MW31D	16.0-24.0
LS12-MW32D	17.5-25.5
LS12-MW33D	16.5-24.5
LS12-MW34D	16.5-24.5
LS12-MW36D	17.0-25.0
LS12-MW06T	7.0-22.0
LS12-MW07T	4.0-24.0
LS12-MW08T	2.0-22.0
LS12-MW09T	2.0-22.0
LS12-MW26T	6.0-21.0



Legend

- Canal
- Land Use Control Boundary
- Groundwater Flow

Annual Monitoring Wells
(includes Performance Monitoring Wells)

- Monitoring Wells

- Deep Isoconcentration
- Deep Isoconcentration (Inferred)

Deep Isoconcentrations (May 2008)

- 1-10
- 10-100
- 100-1000
- >1000

Performance Monitoring Wells

- Co-located
- Deep
- Shallow

Injection Wells

- Co-located
- Deep
- Shallow

Concentrations are in µg/L

N W E S

0 30 60 Feet

Figure C6
Deep Aquifer Total COC Isoconcentration Map
Post-ROD LTM SAP for Sites with Groundwater Impacted by Chlorinated Organic Compounds
JEB Little Creek
Virginia Beach, Virginia

CH2MHILL

\\NORTHEND\PROJ\USNAVFACENGCOM\LITTLECREEK\MAPFILES\345721_SITE12_SAP\FIGURE C7 - POST-ROD GROUNDWATER MONITORING SAMPLING.MXD CBOWMAN 12/28/2010 09:37:45

Legend

- Injection Well
- Monitoring Well
- Source Well
- Contingent Sample Location
- Plume Wells
- Perimeter Well
- Canal
- Former Dry Cleaner/Laundry
- Land Use Control Boundary

N
W E
S

0 80 160 Feet

Figure C7
Site 12 Long-term Monitoring Well Network
Post-ROD LTM SAP for Sites with Groundwater Impacted by Chlorinated Organic Compounds
JEB Little Creek
Virginia Beach, Virginia

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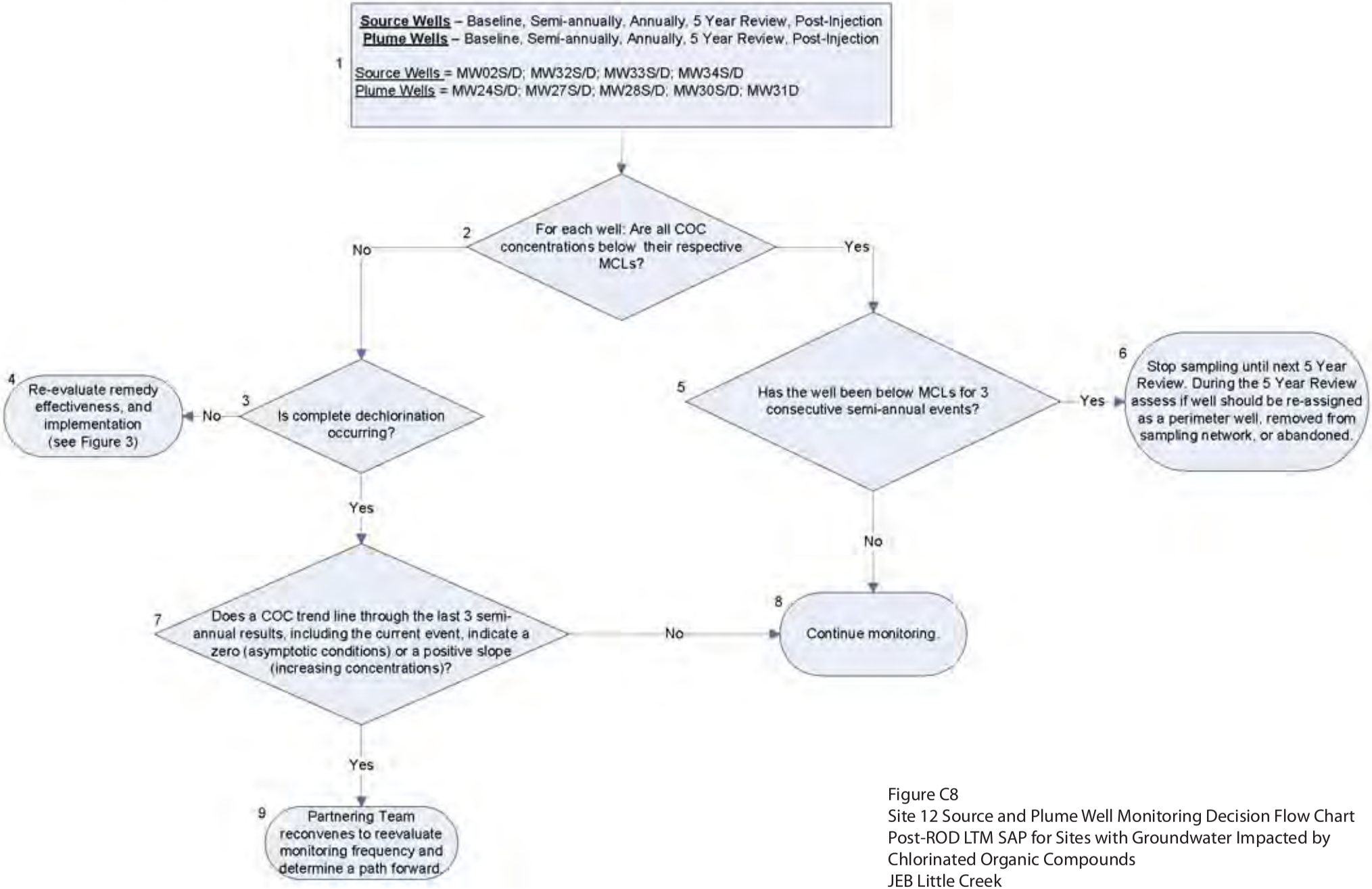


Figure C8
Site 12 Source and Plume Well Monitoring Decision Flow Chart
Post-ROD LTM SAP for Sites with Groundwater Impacted by Chlorinated Organic Compounds
JEB Little Creek
Virginia Beach, Virginia

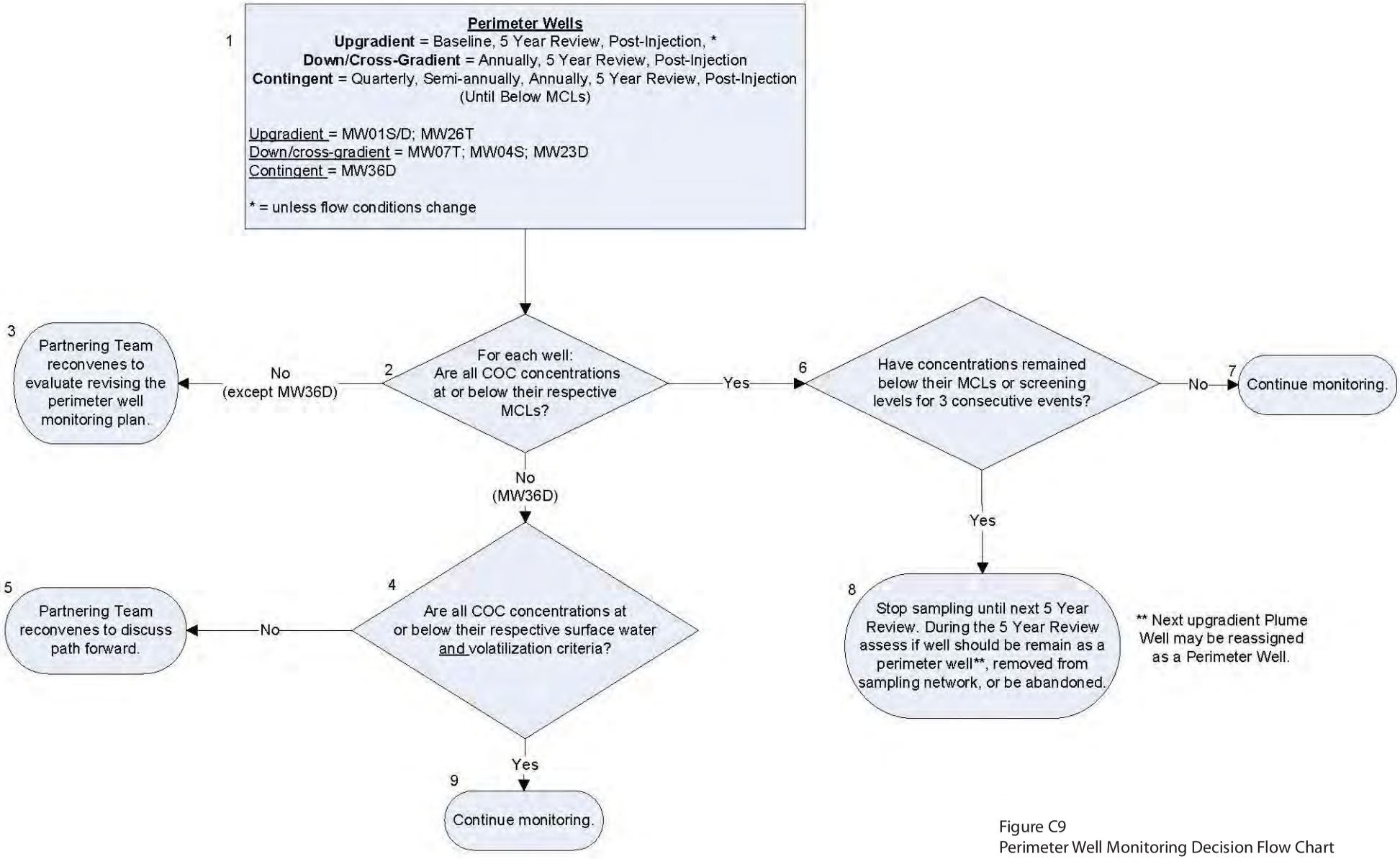
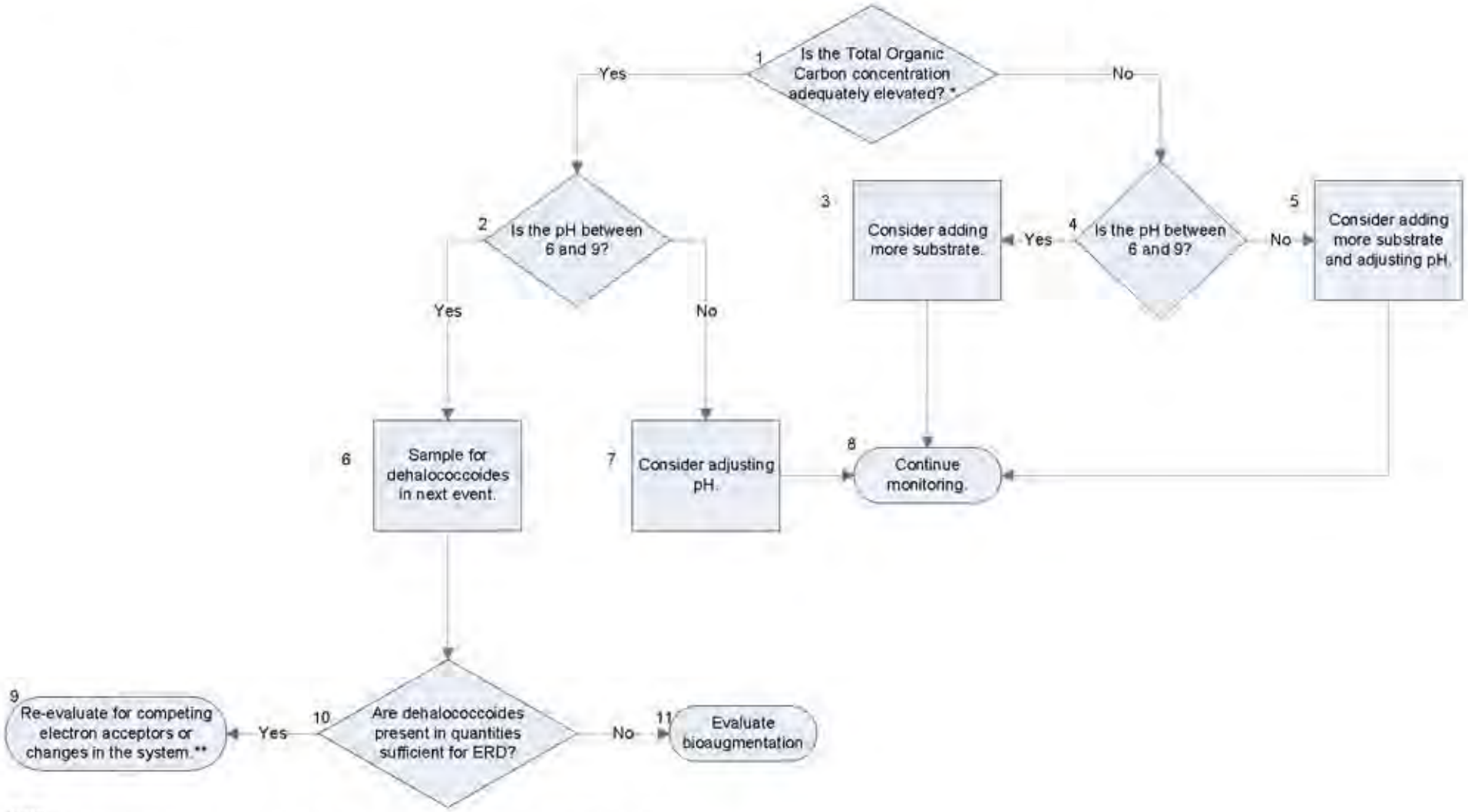


Figure C9
Perimeter Well Monitoring Decision Flow Chart
Post-ROD LTM SAP for Sites with Groundwater Impacted by Chlorinated Organic Compounds
JEB Little Creek
Virginia Beach, Virginia



Note:
Complete dechlorination is indicated by reduction in PCE concentrations, appearance and subsequent sequential transformation of its daughter products, and the continued transformation and reduction in concentration of daughter products.

Sustained increases in vinyl chloride and/or cis-DCE concentrations may indicate incomplete dehalogenation.

* Assessment of dechlorination and remedy effectiveness is a site-wide, not a well specific, evaluation.

** Evaluation of competing electron acceptors or changes in the system may include, but is not limited to: the evaluation of ferrous iron, nitrate, sulfate, methane, ethane, and ethene; the evaluation of groundwater flow direction; and consideration of other site specific activities potentially impacting the system.

Figure C10
Site 12 Reductive Dechlorination Effectiveness Flow Chart
Post-ROD LTM SAP for Sites with Groundwater Impacted by Chlorinated Organic Compounds
JEB Little Creek
Virginia Beach, Virginia

Attachment C1
Laboratory Standard Operating Procedures (SOPs)

2340 Stock Creek Blvd
Rockford TN 37853

STANDARD OPERATING PROCEDURE

ABI 7000 Absolute Quantification

SOP Number: MI SOP-ABI 7000

Revision Number: 1.0 Final

Effective Date: 06/02/06

MI Controlled Document

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to define the steps necessary for processing samples on the Applied Biosystems 7000 Real Time PCR System.

2.0 SCOPE AND APPLICATION

The Applied Biosystems 7000 Real Time PCR system allows quantitative detection of nucleic acid sequences through fluorescent-based PCR chemistries. Absolute quantification is the method that provides the absolute quantity of a single nucleic acid target sequence within an unknown sample.

3.0 METHOD SUMMARY

Reaction plates or tubes in the 96-well format are prepared according to the quantitative PCR SOP. They are then ready to be loaded onto the Applied Biosystems 7000 Real Time PCR system. This SOP then demonstrates the steps taken for the instrument to analyze this plate.

4.0 APPARATUS, AND MATERIALS

- Applied Biosystems 7000 Real Time PCR system
- 96-well reaction plate (Applied Biosystems)

5.0 PROCEDURE

5.1 Creating an Absolute Quantification (AQ) Plate Document

5.1.1 Turn on the computer, then the 7000 instrument using the button on the front of the instrument.

5.1.2 Double click on the 7000 icon to start the ABI 7000 software on the computer. Select File> ew.

5.1.3 In the assay drop-down list of the New Document Wizard, select Absolute Quantification. Accept the default settings for Container and Template (96-Well Clear and Blank Document).

5.1.4 Enter a name in the Default Plate Name field using the analysis type and set name (i.e. DHC-048DC). Click ext.

5.1.5 Choose detectors to add to the plate document. Click Add. Click ext.

5.1.6 Check the selected detectors. Click Finish.

5.1.7 Enter the sample names and specify the detectors for each well.

5.1.8. Click on a well or group of wells and select View>Well Inspector.

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5.1.9 Enter the sample name. Select the proper detector for the sample. Select eubacterial-sybergreen for all assays run on this machine. Leave the task option at the default of **unknown**. Click **Close**.

5.1.10 Repeat until all reaction wells are set. Omit any wells not used by clicking on the "**omit well**" button.

6.2 Specifying Thermal Cycling Conditions and Starting the Run

6.2.1 Select the **Instrument** tab. The standard PCR conditions for the PCR step of the two-step RT-PCR method are displayed. Change conditions based on the specific test that is being run by changing cycles, steps, temperatures and times on this screen (see MI SOP-Quantitative PCR). Sample volume is 50uL and 9600 emulation should be selected.

6.2.2 Select **File>Save As**, name the document (see 6.1.4), then click **Save**.

6.2.3 Load the plate into the instrument. (The A1 position corresponds to the top-left side of the instrument tray.)

6.2.4 Click **Start**.

7.0 WASTE MANAGEMENT

Waste and laboratory-generated waste shall be handled in accordance with Microbial Insights' Standard Operating Procedure for Waste Disposal.

8.0 REFERENCES

2001. Applied Biosystems. ABI Prism 7000 Sequence Detection Systems User Guide.

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microbialinsights
2340 Stock Creek Blvd
Rockford TN 37853

STANDARD OPERATING PROCEDURE

Extraction of DNA from environmental samples (Matrix-Water, Soil, Biofilm, Bio-Sep Beads)

SOP Number:	MI SOP-DNA EXT
Revision Number:	1.0 FINAL
Effective Date:	1/05/06

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ATTACHME TS

Attachment 1: Laboratory Worksheet

PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the activities involved in the preparation, handling, documentation, and extraction of nucleic acids of samples collected from a variety of matrices (water, soil, bio-traps, etc).

1.0 SCOPE AND APPLICATION

The extraction process involves both chemical and mechanical lysis of cells to release chromosomal and plasmid nucleic acids. After the extraction, Microbial Insights, Inc. (MI) uses these products in a variety of analyses.

2.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

Samples should be collected in field following Microbial Insights, Inc. (MI) standard operating procedure for sample collection. Samples must be maintained at 4ºC and extracted within 24-48 hours of collection. DNA extractions will be held at -20ºC for at least 1 year in case there is a need for additional analysis.

3.0 EQUIPMENT AND SUPPLIES

[redacted]

- Micropipetters (Gilson P-10, P-100, P-1000) and appropriate sterile tips (ART). [See pipet calibration and maintenance in calibration log.]
- Sartorius AG Analytical scale, Model BP310. (Sartorius) [See scale calibration and maintenance in calibration log]
- Sterile spatulas and transfer paper.
- Microcentrifuge Eppendorf model #5415D.
- Vortex Mixer (MO BIO Adapter) .

4.0 REAGENTS

[redacted]

5.0 TESTING PROCEDURE

5.1 Sample Preparation:

5.1.1 Cell Cultures

If the sample consists of bacterial colonies on plated media, use a flame sterilized inoculating loop to pick a colony from the plate. If the sample is a liquid culture, use a sterile 1ml syringe to remove 500µl of sample. Place the sample into the 2ml bead solution tube.

5.1.2 Water samples

Filter the water through a 0.2 micron Supor filter. Filter the entire volume of water received until the filter becomes clogged. If less than 250mls of water has been filtered, a second filter should be used. Record the volume of water filtered, and discard the filtered water. Place the filter/filters into a sterile Petri dish and leave at 4ºC until the sample is processed. For processing, use a sterile razor blade to cut the filter into sections and place into the 2ml bead solution tube.

5.1.3 Soil, solid and Biofilm samples

Use a flame sterilized spatula to place 0.5 grams of the soil/solid/biofilm sample into the 2ml bead solution tube.

5.1.4 Bio-Sep Bead samples

Use flame sterilized tweezers to place 20 Bio-Sep beads into the 2ml bead solution tube.

5.2 Extraction process

5.2.1. Label 2ml bead tubes with sample name and number. For every extraction set a blank tube must be labeled with the set name/names and "x" to indicate this tube as the extraction blank for that set. Add 60µl of solution 1 and [redacted] to each tube. Add the sample as described in section 5.1 into the bead tube.

5.2.2 Place tubes onto [redacted] at maximum speed for 10 minutes.

5.2.3 While tubes are vortexing, label 1 spin filter tube for each sample as well as the blank and 4- 1.5ml tubes for each sample and blank with sample name and number. To the first 1.5ml tube add 250µl of [redacted]. To the second 1.5ml tube add 200µl of [redacted]. To the third 1.5ml tube add 1200µl of [redacted]. Leave the final 1.5ml tube empty and be sure that it is labeled legibly with the entire sample name as this is the final tube in the process and will be used for long term storage.

5.2.4 After the 2ml bead tubes have vortexed for 10 minutes, place them in the centrifuge at 10,000 g for 1 minute.

5.2.5 Transfer the supernatant to the 1.5ml tube containing solution C2. Invert 4-5 times and place at 4ºC for 5 minutes.

5.2.6 Place tubes in the centrifuge at 10,000 g at room temperature for 1 minute.

5.2.7 Transfer the supernatant to the 1.5ml tube containing [redacted] being sure not to disturb the pellet. Invert 4-5 times and place at 4ºC for 5 minutes.

5.2.8 Place tubes in the centrifuge at 10,000g at room temperature for 1 minute.

5.2.9 Transfer 750µls of this to the 1.5ml tube containing [redacted]. Invert 4-5 times to mix. Transfer 675µls of this to the spin filter tubes.

5.2.10 Spin at 10,000g for 1 minute. Discard flow through and repeat until all of this solution is used.

5.2.11 After all solution has passed through the spin filter, discard the final flow through. Add 500µls of [redacted] directly to the filter. Discard flow through and centrifuge at 10,000g for 1 minute.

5.2.12 Transfer the filter to the remaining clean, labeled 1.5ml tube. Add 100µls of [redacted] directly to the filter. Spin at 10,000g for 1 minute. Check tube to be sure that there are 100µls of eluted DNA then discard the filter. Store the extracted DNA at 4ºC for up to 6 months. Transfer to -20ºC for long term storage.

6.0 QUALITY CONTROL

6.1 For quality control of the extraction process, a recovery standard is added to each sample. An extraction blank is also processed with each extraction set to ensure no cross contamination of samples or solutions.

6.2 Ongoing calibration checks and maintenance of the instruments must be documented in the appropriate logbook.

6.3 The Laboratory Director has the responsibility to ensure that this procedure is performed by an employee who has been properly trained in its use and has the required experience to process the samples.

6.4 All deviations from this SOP must be documented in a Nonconformance Report (NCR) or an equivalent system (i.e., database).

7.0 LABORATORY SAMPLE FOLDERS

Extraction volumes for each sample will be logged in at the time of extraction in the worksheet attached in each sample folder. An example of this worksheet is presented in *tta ment* .

8.0 DOCUMENT CORRECTIONS

Changes or corrections on any laboratory documentation will be made by crossing out the erroneous item with a single line and writing the new information above the crossed-out item. The person correcting the information should initial and date the correction. The original entry, although erroneous, must remain legible beneath the cross-out line. All information will be recorded using black or blue indelible ink.

ATTACHME T 1

LABORATORY WORKSHEET

2340 Stock Creek Blvd
Rockford TN 37853

STANDARD OPERATING PROCEDURE

Quantitative Polymerase Chain Reaction (qPCR)

SOP Number:	DNA-qPCR
Revision Number:	1.0 FINAL
Revision Date:	01/10/06
Effective Date:	01/10/06

Reviewed By:	Dora Ogles DNA Lab Director	Date
Reviewed By:	Aaron Peacock QA Coordinator	Date
Reviewed By:	Greg Davis President/Lab Director	Date

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PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the activities involved in the preparation, handling, documentation, and analysis of samples collected from a variety of matrices (water, soil, bio-traps, etc) to enumerate target populations using quantitative Real-Time polymerase chain reaction or qPCR.

1.0 SCOPE AND APPLICATION

Quantitative Real-Time polymerase chain reaction (qPCR) is used to enumerate target populations of microorganisms. Microbial Insights, Inc. (MI) uses qPCR to target the following organisms associated with the remediation of a priority pollutants such as Tetrachloroethene (PCE) and Methyl *tert*-butyl ether (MTBE).

Q-Target	MI Code or CAS Number	Real-time assay
e alo o oi es spp	qDHC	Taqman
e aloba ter spp	qDHB	SYBR green
esul uromonas spp	qDSM	Taqman
esul itoba terium spp	qDSB	SYBR green
Methanogen (mcrA gene)	qMGN	SYBR green
Iron and Sulfate Reducing Bacteria	qIRBSRB	Taqman
Dissimilatory Sulfite Reductase	qDSR	SYBR green
Methane Oxidizing Bacteria	qMOB	SYBR green
PM1	qPM1	Taqman
Universal Baceteria	qEBAC	Taqman
Soluble Methane Monooxygenase	qsMMO	SYBR green
Ammonia Oxidizing Bacteria	qAOB	Taqman
Anaerobic Toluene	qbssA	Taqman
Geoba ter spp	qGEO	Taqman
Denitrifying Bacteria (nirS and nirK)	qDEN	SYBR green
Catechol Dioxygenase	qCAT	SYBR green
Toluene Monooxygenase	qRDEG	SYBR green
Toluene Monooxygenase	qRMO	SYBR green
Xylene Monooxygenase	qTOL	Taqman
Naphthalene Dioxygenase	qNAH	Taqman
Phenol Monooxygenase	qPHE	SYBR green
Acetogens(FTHFS gene)	qACE	SYBR green
Alkane Monooxygenase	qALKb	Taqman
Butane Monooxygenase	qBMO	Taqman
Toluene Dioxygenase	qTOD	Taqman

2.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

Samples are collected following the standard operating procedure developed by Microbial Insights, Inc (MI). DNA extraction and RNA extraction are performed following the extraction standard operating procedure developed by Microbial Insights, Inc. DNA is stored in 1/10 Tris-EDTA(TE) at 4ºC while RNA is stored in RNA Storage solution at -20ºC. RNA samples are converted to cDNA following (SOP-RNA ext) prior to the quantitative PCR analysis.

3.0 EQUIPMENT AND SUPPLIES

- 96-well plate (Applied Biosystems).
- Micropipettes (Gilson P-10, P-100, P-1000) and 10 ml pipettes, and appropriate sterile tips (ART). [see calibration listed in calibration log]
- Applied Biosystems SDS 7000 or 7300 instrument. [see calibration listed in calibration log]
- Sterile 0.5ml thin wall strip tubes and optical clear caps (Applied Biosystems)

4.0 REAGENTS AND POSITIVE CONTROLS

4.1 TaqMan DNA polymerase and master mix for Taqman assays (Applied Biosystems)

4.2 Sterile filtered (0.2mM) nanopure, organic-free, deionized water.

4.3 Forward and Reverse primers/Probe: these are gene/assay specific (IDT Technologies primers, Applied Biosystems FAM/TAMRA probes). See notes section for primer handling. Final concentration of probe and primers: [redacted] or adjusted as needed for each primer set. [redacted]

4.4 Positive and Negative Control DNA: DNA is purchased from ATCC or extracted and purified in-house from pure isolates. The amount of DNA used in all assays is 3µl. For negative controls, water is added in place of template DNA.

4.5 [redacted] for SYBR green assays ([redacted])

4.6 [redacted] for SYBR green assay

4.7 [redacted]

[redacted]

[redacted]

5.0 PROCEDURE

5.1 Preparation of Stock Primer

5.1.1 It is necessary to resuspend and dilute our primers (IDT Technologies) before we can use them for our PCR reaction. This procedure is done when new primers are received and both working stocks and solution stocks should be prepared at this time. The label on the tube will provide the oligo length and concentration.

5.1.2 To the stock tube, aseptically add 1ml of sterile PCR dH2O and vortex the tube for 1 minute to resuspend the primer. Centrifuge for 1 minute at 14,000 rpm to remove any solution from the lid then allow the tube to sit at room temperature for 5 minutes to ensure complete dissolution. This provides the solution stock.

5.1.3 To make the working stock to be used in each reaction, add the amount of nanomoles of the stock solution (listed on the stock tube) to a clean 1.5ml tube and bring this volume up to 1ml with sterile dH2O.

5.2 Mixing Reagents-Taqman Assays

5.2.1 Prepare a master mix including the appropriate amounts of each of the following: PCR water, Taqman Universal PCR mix, Primers (forward and reverse) and Probe. The appropriate amounts are listed in the MI primer list (primer.xls) prepared for each primer/probe combination.

5.2.2 Add [redacted] to each well of the 96 well plate or strip tube. Add [redacted] template DNA/cDNA to the appropriate wells. Add [redacted] positive control DNA to the appropriate wells. Add [redacted] PCR water to the negative control wells.

5.2.1 Spin the plate in the centrifuge at 3600 rpm for 2 minutes to be sure all mix is in the bottom of the tube and that no bubbles are present.

5.2.2 The plate of samples is then placed in either the ABI 7000 or 7300. The conditions of each assay will vary depending upon the primer/probe combination (see table1).

5.2.3 Upon completion of the run, export the data to an Excel worksheet to determine the number of target molecules, using an equation derived from standard curves of known numbers of target molecules.

5.2.4 Standard curves are developed from a serial dilution of a known concentration of the positive control organism. At least 4 dilutions will be used for each curve and the R^2 of each equation must be at least 0.95.

5.3 Mixing Reagents-SYBR green Assays

5.3.1 Prepare a master mix including the appropriate amounts of each of the following ingredients: PCR water, [redacted], PCR Primers (forward and reverse), [redacted] Reaction Buffer, [redacted] The appropriate amounts are listed in the excel sheets prepared for each primer combination.

5.3.2 Add [redacted] of master mix to each well of the 96 well plate or strip tube. Add [redacted] template DNA/cDNA to the appropriate wells. Add [redacted] positive control DNA to the appropriate wells. Add [redacted] PCR water to the negative control wells.

5.3.3 Spin the plate in the centrifuge at 3600 rpm for 2 minutes to be sure all mix is in the bottom of the tube and that no bubbles are present.

5.3.4 The plate of samples is then placed in either the ABI 7000 or 7300. The conditions of each assay will vary depending upon the primer/probe combination (see DNA primers xls data sheet)

5.3.5 Upon completion of the run, export the data to an Excel worksheet, then determine the number of target molecules, using an equation derived from standard curves of known numbers of target molecules. The standard curves are developed from a serial dilution of a known concentration of the positive control organism. At least 4 dilutions will be used for each curve and the R^2 of each equation must be at least 0.95.

6.0 QUALITY CONTROL

6.1 For quality control of the amplification process, positive and negative controls are always performed. The extraction blank from each set is also amplified to ensure no cross contamination of samples or solutions during the extraction process.

6.2 Ongoing calibration checks and maintenance of the instruments must be documented in the appropriate logbook.

6.3 The Laboratory Director has the responsibility to ensure that this procedure is performed by an employee who has been properly trained in its use and has the required experience to process the samples.

6.4 All deviations from this SOP must be documented in a Nonconformance Report (NCR) or using an equivalent system (i.e., database).

7.0 ANALYTICAL REPORT

7.1 All data regarding quantities are entered into a data report using the laboratory information management systems (LIMS).

7.2 All documentation pertinent to a project will be placed in the "project folder" identified with the unique LIMS Project number. Hardcopy records, data sheets, and soft copy reports will be stored for a period of three years.

8.0 DOCUMENT CORRECTIONS

Changes or corrections on any laboratory documentation will be made by crossing out the erroneous item with a single line and initialing (by the person performing the correction) and dating the correction.
The original item, although erroneous, must remain legible beneath the cross-out line. The new information should be written clearly above the crossed-out erroneous item. All information will be recorded using black or blue indelible ink.

9.0 TABLES, DIAGRAMS, AND FLOWCHARTS

See DNA primer xls sheet for primer conditions.

2340 Stock Creek Blvd
Rockford TN 37853

STANDARD OPERATING PROCEDURE

Calculating QPCR Results

SOP Number: SOP-qPCR Calc

Revision Number: FINAL

Effective Date: 08/14/2006

Controlled Copy No.

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Anita Biernacki

Anita Biernacki

Operations Manager

06/09/2010

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1.0 Purpose

1.1 The purpose of this method is to describe the analysis of absolute quantification. Absolute quantification determines the input copy number of the transcript of interest through the use of comparing the PCR signal to a standard curve.

2.0 Procedure

2.1 Open the exported excel sheet with the qPCR results. This will be saved as a .csv file. Resave as a Microsoft Excel worksheet file.

2.2 Copy and paste all samples with their corresponding results onto a blank part of the worksheet so that all samples are in order and grouped accordingly to the type of assay that was run.

2.3 The standard curve that corresponds to the type of assay tested is used to determine the copy number per reaction.

2.4 Next this result is divided by 3 to determine the copy number per µl (3µl are used in the qPCR reaction).

2.5 If DNA is being analyzed, the last step for quantification is to multiply by 100 to account for the dilution factor of the DNA extraction. Now the result is in gene copies per extraction.

2.6 If RNA is being analyzed, the next step is to multiply by 15 which accounts for the volume of the initial complementary DNA PCR reaction. Divide the number by 5 (5µl is used in the cDNA reaction). This result is now gene copies per µl of RNA.

2.7 The last step for RNA analysis is to multiply by 100 to account for the dilution factor of the RNA extraction. The final result is in gene copies per extraction.

2.8 These numbers are then entered into the data sheets for each sample set generated by the LIMS system.

2.9 Once these sheets are uploaded into the LIMS system, it will then process the remaining calculation. These calculations consist of dividing the number from 2.5 or 2.7 by the initial volume extracted to get the number of gene copies per volume of sample.

2.10 For DNA, that number is then converted to cells per volume of sample by dividing the average number of gene copies per cell for that target organism.

2.11 The final number for DNA will then be in cells per volume of sample. For RNA, it will be in gene copies per volume of sample.

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3.0 Waste Management

3.1 Waste and laboratory-generated waste shall be handled in accordance with Microbial Insights' Standard Operating Procedure for Waste Disposal.

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2340 Stock Creek Blvd
Rockford TN 37853

STANDARD OPERATING PROCEDURE

STANDARD OPERATING PROCDURE
for Sample Receiving

SOP Number: MI SOP- SAMREC

Revision Number: 1.1 FINAL

Effective Date: 11/14/2008

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1.0 PURPOSE

The purpose of this Standard Operating Procedure is to outline the steps involved in sample receipt and storage.

2.0 DEFINITIONS

Aliquot: A portion of a sample

Holding Time: The maximum time that a sample may be held prior to analysis

LIMS: Laboratory Information Management System

on-conformance: Sample documentation that is received with incorrect, incomplete, or inadequate information.

Preservation: Refrigeration and/or reagents added prior to sample collection to maintain the chemical, physical and/or biological integrity of a sample.

Short Holding Time: Samples must be analyzed within 48 hours or less

Temperature Blank: A sample bottle of water that accompanies the samples in each cooler. This blank is used to monitor cooler temperature upon receipt of samples.

Trip Blank: A set of 40mL VOA vials filled with de-ionized water that travels with the samples to be analyzed for Volatile Organic Compounds (VOC). These samples are analyzed to determine if cross contamination occurred during sampling.

3.0 RESPONSIBILITIES

3.1 Sample Custodian

The responsibility of the Sample Custodian is to sign and date all appropriate receiving documents including Chain of custodies (COCs) and shipping papers. Signing the COC indicates that the laboratory accepts the samples in their condition upon arrival. Conditions under which COCs will be signed or left unsigned are discussed in Section 4.0 of this SOP. The Sample Custodian is also responsible for notifying the client of all the discrepancies that occur during the sample receipt process. In each case, the client shall be consulted to resolve all discrepancies involving their samples. The Sample Custodian shall log samples in accurately and in a timely manner. The sample Custodian reports to the Director or Laboratory Supervisors.

3.2 Reporting Level Requirements and Responsibility

Microbial Insights' Director or Laboratory Manager(s) have the responsibility to alert the Sample Custodian, in writing or through the Laboratory Information Management System (LIMS), of any projects that require more than standard reporting requirements prior to delivery of those samples. Quality control reporting levels other than standard must be arranged with Microbial Insights Director or Laboratory Manager(s) prior to delivery of the samples. The

MI Controlled Document

Director or Laboratory Manager(s) will then serve as the Project Manager for that project. If notification is not made prior to sample receipt, the level of reporting requested by the client cannot be assured.

3.3 General Documentation Requirements and Responsibility

The responsibility for quality control and documentation during sample receiving requires a diligent effort to ensure that all documentation is present and complete. The Sample Custodian is responsible for the sample receipt documentation and for client contact and documenting client responses in the event of non-conformance.

All records must be written in ink or printed electronically. When any changes are made, a single line should be drawn through the errors and the corrections to be made and initialed and dated. All notations written on COCs shall be dated and initialed.

4.0 SAMPLE RECEIPT

The following policy and procedures are in place to ensure that all samples and COC forms that are accepted at Microbial Insights are thoroughly inspected and all discrepancies are fully documented. The Sample Custodian and/or Laboratory Manager(s) shall contact the client in the event that there is any discrepancy involved in the condition or the documentation of samples, upon receipt, that may affect the sample's integrity or the analytical process.

4.1 Sample Receipt Record

A permanent record of sample receipt shall be maintained electronically in the LIMS. At a minimum, that record will include (1) Client Name; (2) Project Name; (3) Date and time of sample receipt; (4) Unique laboratory identification; (5) Name or initials of the person making the entries; (6) Requested analyses.

4.2 Sample Acceptance Policy

- Samples that are shipped to Microbial Insights must be accompanied by proper full and complete documentation. This documentation shall be marked on a COC and shall include: sample identification, the location, date and time of collection, sampler's name, preservation type (if needed), sample matrix, specific parameters to be analyzed, and any other pertinent information to that sample set.
- Sample labels shall be supplied by Microbial Insights or the client. Those labels must be water resistant and completed using indelible ink. Each sample label must include a unique identification number that links it to the COC form.
- Samples must be received within the specific holding times. Clients are requested to notify Microbial Insights Sample Custodian if unsure of the holding times.

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- Samples must arrive at Microbial Insights with sufficient volume to conduct the requested analyses (All bottles should be filled completely, if possible).
- When problems with samples or documentation are found during the sample receiving process, the Sample Custodian will make every attempt to contact the client as soon as possible to make decisions concerning those discrepancies.
- In the event the client cannot be reached, a message will be left either on voice mail, a brief message with the receptionist to return the call, or by an email. Samples will be placed in a storage refrigerator and held until Microbial Insights gets a response from the client. (Exceptions will be made when samples are received that have short holding times and the samples are from a client with whom Microbial Insights has regular and frequent dealings. Also when the samples have short holding times and the samples are from a client with whom Microbial Insights has a signed contract, work order or purchase order).

4.3 on-Conformance otification

The corrective action form should be filled out and the client should be notified in case of any of the following occurrences:

- Temperature discrepancy
- Hold time discrepancy
- Broken, leaked or missing samples
- Insufficient sample volume

Any analyses that are conducted on samples that do not meet these acceptance criteria will be noted within the client data folder, Microbial Insights LIMS, and noted on the front cover sheet of the client's final report.

4.4 Sample Rejection Criteria

The following situations dictate when samples will be rejected:

- Coolers and samples arrive at Microbial Insights' facility with no client information
- Coolers do not arrive on time at Microbial Insights' facility due to error by the carrier (ie. Fedex, UPS or DHL) in which samples have strict holding times.
- Samples arrive with no COC form and there is no means to obtain one.
- Coolers that arrive with hazard labels on them for which Microbial Insights is not equipped or certified.

4.5 Sample Receipt Procedure

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4.5.1 Prior to signing electronic shipping document from courier, ensure that the number of packages listed on the electronic document corresponds with the number of packages actually delivered.

4.5.2 Remove any air bills from the outside of the coolers, place in the designated data folder assigned to that project set.

4.5.3 Put on safety glasses and protective gloves before handling, opening, or unpacking packages and coolers that contain environmental samples.

4.5.4 Retrieve the COC form from the inside of cooler and set aside or place in designated data folder.

4.5.5 Place thermometer in cooler and record cooler temperature, note on designated form, initial and date entry. The temperature in the cooler should be 4°C ± 2 degrees. If the temperature is not within those parameters, note the discrepancies on the designated form and include it with the data folder. Also, bring to the attention of the Laboratory Managers the temperature for any further decisions regarding the validity of the samples.

4.5.6 Inspect each sample and label while removing it from the cooler. Sample containers or filter packages should be intact. At a minimum, sample labels should be completed with the following information:

a. Sample Name/Number
b. Date and Time of Collection
c. Location
d. Analyses to be done

4.5.7 If samples were received in grouped sets, keep the sets grouped together as they are unpacked. If samples were not received in sets, organize them into sets while unpacking.

4.5.8 Match the sample identifications to the COC form. Note any discrepancies on the COC and with the data set folder.

4.5.9 Make sure that field and trip blanks (if needed) are present and identified. Document all missing or potentially missing samples on the COC form and in the data set folder.

4.5.10 Check the COC form to ensure that all samples are entered and the specific analysis is checked appropriately for each bottle, filter, or Bio-Trap.

4.5.11 Ensure that all sample receipt documentation is complete, sign the COC form.

4.6 Weekend Sample Receipt Procedure

All coolers shipped for weekend delivery are signed for by a Microbial Insights employee and stored in the storage appropriately until normal business hours Monday morning. Water samples have to be stored in a refrigerator, all other samples need to be stored in a

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freezer. Occasionally, laboratory technicians will work on weekends and will conduct an abbreviated sample receipt process, and the complete process will occur during normal business hours Monday morning.

5.0 SAMPLE STORAGE

Samples shall be stored according to the conditions specified by preservation protocols. The storage conditions shall be maintained, monitored, and documented. Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources. Samples shall be stored in segregated areas to prevent cross contamination.

5.1 Sample Storage Temperature

5.1.1 Samples which require thermal preservation shall be stored under refrigeration. It is the Laboratory Technician(s) responsibility to ensure that the refrigerator temperatures within the sample reception area are monitored and recorded on the Temperature Log Sheet. The Temperature Log Sheet is available within each laboratory.

5.1.2 If the temperature of the refrigerator/ freezer is outside of the acceptable range limits, the Laboratory Technician(s) must immediately notify the Laboratory Manager(s) and the Company Manager. Maintenance will be arranged through the Laboratory Manager or his or her representative. If it is apparent that the proper sample temperature cannot be maintained in the area needing maintenance, then every effort will be made to move samples to another refrigerator that is functioning within the temperature control limits.

6.0 SAFETY

Personnel safety is a priority at Microbial Insights. All employees are required to wear appropriate personal protective equipment in accordance with Microbial Insights Chemical Hygiene Plan. The Sample Custodian has been provided with safety glasses, protective gloves and a laboratory coat.

It is required that the Sample Custodian wear gloves and safety glasses while handling all coolers and samples. Coolers shall be opened in a well-ventilated area. If odors are detected upon opening, the cooler shall be closed and moved to the fume hood in the Commercial Laboratory before proceeding with the sample receipt procedures.

7.0 COOLER STORAGE AND DISPOSAL

Coolers shall be emptied of any melted ice and left to air dry. Coolers are then stored in a designated storage area. For any cooler that has aqueous residue from a sample container, proper disposal of the aqueous waste is followed then the cooler is rinsed, air dried and stored.

Any coolers which are damaged are then stored in separate storage section to be later discarded.

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METALS DIGESTION/PREPARATION

METHODS

USEPA SW846

3005A, 3010A, 3030C, 3031, 3050B

USEPA CLPILM 04.1 Aqueous & Soil/Sediment (NJDEP does not accept CLPILM 04.1 after June, 2003)

Addendum for USEPA CLPILM 05.2 Aqueous & Soil/Sediment

USEPA Methods for Chemical Analysis of Water and Wastes

200.7, Standard Methods 3030C

SOP NUMBER: **SOP-100**

REVISION NUMBER: **19**

APPROVED BY:

Betty DeVille

SECTION MANAGER

Randy D. Ward

QUALITY ASSURANCE OFFICER

07/25/06

EFFECTIVE DATE

04/20/09

DATE OF LAST REVIEW

METALS DIGESTION/PREPARATION

References:
Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3031, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)

I. SCOPE AND APPLICATION

A. AQUEOUS

1. Method 3005A and USEPA CLP ILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy".

   a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).

2. Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry"

   a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).

3. Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".

   a. This method is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by ICP. The procedure is used to determine total metals.

4. Method 3030C (Standard methods), "Preliminary Treatment for Acid-Extractable Metals".

   a. This method is used to prepare ground water samples from North Carolina for analysis by ICP.

B. SOLIDS

1. Method 3050B, "Acid Digestion of Sediments, Sludges and Soils".

   a. This method is used to prepare sediments, sludges and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.

   b. It should be noted that some metals could be biased high with the soil digestion when dilution is necessary. Take necessary measures to ensure that dilutions are made as accurately as possible.

2. USEPA CLP ILM0 4.1, “Acid Digestion of Soil/Sediment”

   a. This method is used to prepare sediments and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.

C. OILS

1. Method 3031, "Digestion Procedure for Oils".

   a. This method is used to prepare samples containing oils, greases or waxes for analysis by inductively coupled argon plasma emission spectroscopy (ICP).

D. **NOTES:**

1. "Total Metals" includes all metals, inorganically and organically bound and both dissolved and particulate.

2. "Dissolved metals" includes all metals present in a sample after filtration through a 0.45 micron filter followed by digestion.

II. SUMMARY OF METHODS

A. A representative sample of water, soil or oil is put into an acid medium and exposed to heat for a certain amount of time. This allows for reduction of interferences by organic matter and converts metals bound to particulates to form the free metal that can be determined by ICP-Atomic Emission Spectrometry.

NOTE: When a reporting limit is required for a project lower then is customary, a four times concentration must be used in order to reach that lower level. Care

must be taken to matrix match this concentrated aliquot. A blank and laboratory control sample (at a reduced concentration) are required with this concentration. A matrix spike (not at reduced concentration) and duplicate or matrix spike and matrix spike duplicate is needed per 20 samples or per batch.

III. SAMPLE HANDLING AND PRESERVATION

A. AQUEOUS

1. Samples are taken in high density polyethylene, one liter bottles. Samples should be preserved with concentrated HNO_3 to a pH <2 immediately once sampled. If dissolved metals are to be analyzed the sample should be filtered before the HNO_3 is added. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

B. SOLIDS

1. Samples are taken in high density polyethylene(CLP only) or glass bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

C. OILS

1. Samples are taken in high density polyethylene bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

IV. INTERFERENCES

A. AQUEOUS

1. Method 3005A and USEPA CLPILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy", SW846, July, 1992.

   a. This digestion procedure may not be sufficiently vigorous to destroy some metal complexes.

2. Method 200.7

3. Method 3010A

   a. See method 6010B.

B. SOLIDS

1. Method 3050B

   a. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

2. USEPA CLP ILM0 4.1

   a. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

C. OILS

1. Method 3031

   a. These digestates can have very high dissolved solids, which may necessitate the use of internal standards, dilutions, or the method of standard addition.

V. SAFETY

A. Normal accepted laboratory safety practices should be followed while performing this analysis.

B. Be certain the exhaust hood is functioning before you begin the digestion procedure.

C. Hot acids can be extremely corrosive. Avoid inhalation or contact with skin.

VI. EQUIPMENT/APPARATUS

A. Fume hood, Labconco or equivalent.

B. Hot plate, Thermolyne cimarec-3 or equivalent source for use at 95°C. The temperature of the hot plate must be monitored via the use of a temperature blank.

C. Thermometer capable of reading 80 to 120 degrees C – ERTCO cat# 611-3-SC or equivalent.

D. Vacuum pump for filtering dissolved metals- Gast or equivalent.

E. Analytical balance capable of weighing to 0.01 gram. Mettler model BB300 or equivalent.

F. Beckman CS-6R centrifuge.

G. Various class A volumetric glassware and ribbed watchglasses, Pyrex or equivalent.

H. Whatman No. 41 filter paper or equivalent.

I. Whatman No. 42 filter paper or equivalent.

J. Whatman 0.45 micron filter paper or equivalent.

K. 250 mL beaker or other appropriate vessel such as polypropylene block digester tubes, watch glasses and caps.

L. Stirring device, e.g. magnetic stirrer, glass rod or equivalent.

M. Manual Sample Mill

N. Wiley Sample Mill

O. Clippers for cutting vegetation

NOTE: All glassware should be acid washed.

VII. REAGENTS AND STANDARD PREPARATION

A. REAGENTS

1. Metals grade Nitric acid (HNO_3). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

2. Metals grade Hydrochloric acid (HCl). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

3. 30% hydrogen peroxide reagent, ACS Grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

4. Metals grade Sulfuric acid (H_2SO_4). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

5. Reagent water (Deionized water).

6. Potassium Permanganate - Ultra pure grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

7. Ammonium hydroxide, concentrated, reagent grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

8. Ammonium phosphate, reagent grade- Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

9. Base oil, analyte-free. Oil should be analyzed to determine level of impurities. If method blank is < MDL, then the reagent can be used.

B. STANDARDS

1. Traceability

a. A bound logbook record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the logbook as well as on the container's label.

b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

c. The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

2. PREPARATION

A. Laboratory control sample

1. Aqueous

a. This solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO_3, 1 mL of CLP-CAL-1, Solution A, 1 mL of CLP-CAL-1 Solution B, 0.25 mL of CLP-CAL-2, and 0.25 mL of CLP-CAL-3 diluted to 1 L in a volumetric flask. Use 50 mL (100 mL for strict CLPIlM0 4.1) for digestion. This solution is given a unique identifier and recorded in sample digestion logbook.

b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO_3, 1mL CLPP-SPK-4 (Inorganic Ventures) (This solution contains 10 mg/L Selenium, 100 mg/L Antimony, 50 mg/L Cadmium and Thallium, 40 mg/L Arsenic and 20 mg/L Lead) to 1 L in a volumetric flask. This solution is given a unique identifier. Use 12.5 mLs to 50 mLs and prepare two aliquots. Heat at 90 to 95°C to reduce the volume in each vessel to ten mLs and then combine each 10 mL aliquot into one vessel and take to a final volume of 25 mLs. Take care to matrix match acids so that the final 25 mL portion will contain 2% HNO_3 and 5% HCl. Use 0.125 mLs HNO_3 and 0.3125 mLs HCl to each 50 mL vessel.

2. Solids

a. A 1.0 ±0.02 gram aliquot of teflon chips is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier according to the Lot# for the teflon chips used and when digested is given the descriptor. i.e. LCSS(date)A and then B etc. plus the unique identifier number assigned. Alternatively a solid matrix standard reference material is obtained from the manufacturer. This sample is given a unique identifier and recorded in the sample digestion logbook.

3. Oils

a. **An analyte free oil MUST be used or explosive reactions can occur.** An analyte free oil (wesson oil which has been analyzed previously to prove that it is < MDL.) is spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier. i.e. LCSO(date)A and then B etc.

B. Spiking solution

1. Sample is spiked using 0.1 mL of CLP-CAL-1, Solution A, 0.1 mL of CLP-CAL-1 Solution B, 0.025 mL of CLP-CAL-2 and 0.025 mL of CLP-CAL-3 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers. Record the amount spiked and the unique identifier of the standard.

2. CLP sample is spiked using 0.1 mL CLPP-SPK-1 and 0.1 mL CLPP-SPK-4 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers.

3. For samples that require four times concentration, the sample is spiked using 0.0125 mLs of CLPP-SPK-4 to each of two vessels with 50 mLs of sample in each. The volume of each of the vessels is lowered to less then 10 mLs and combined and the final volume of this concentrated sample is 25mLs.

VIII. CALIBRATION

A. The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. 85° for oil samples. Record in digestion logbook.

IX. PROCEDURE

A. Glassware preparation for oil digestion or when the hot-block can not be used:

1. Wash glassware with hot soapy water and rinse thoroughly. (Beakers must be washed as soon as possible after being used, dirty beakers must not be allowed to sit overnight.)

2. Rinse glassware with reagent water that contains 5% HNO_3 and 5% HCl followed by a rinse with reagent water.

3. Prior to use, all glassware must be confirmed clean via a glassware check. Otherwise, repeat step "2" until the glassware check passes.

B. Aqueous sample filtration (for dissolved metals):

1. Thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 HNO_3 followed by a thorough D.I. water rinsing. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.

2. Rinse a 0.45 micron filter with 1:5 HNO_3 thoroughly, followed by D.I. water.

3. Filter the unpreserved sample. If dissolved Hg analysis is requested for the sample, filter at least 200 mL.

4. Discard the first 50 to 100 mL.

5. A preparation blank must be taken through the filtration step and analyzed with the sample.

6. Preserve the sample with HNO_3 to pH<2.

7. Soluble samples that are clean and clear do not have to be digested. Use 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated HNO_3. **Samples must be digested unless approval for analysis without digestion is received from the project manager.**

C. Aqueous sample preparation

1. Method 3005A and USEPA CLP ILM0 4.1, "**Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP ".**

   a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.

   b. Add 0.50 mL (1 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO_3 to the sample. For samples which require concentration, add 0.125 mL (0.25 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO_3 to the sample.

   c. Add 2.5 mL (5 mL of 1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample. For samples which require concentration, add 0.3125 mL (0.625 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample.

   d. Cover the sample with a ribbed watch glass or equivalent source.

   e. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be recorded in the digestion log book. Take the volume down to between 5 to 10 mL, (12 to 25 mLs when strict CLP ILM0 4.1 is required) **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot plate and cool

   f. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

   g. Bring sample to its predigestion volume (or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the digestion log book.

   h. The sample is now ready for analysis.

   i. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.

2 Method 200.7, "**Acid digestion procedure for total recoverable metals**".

a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel. If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.

b. Add 1.0 mL concentrated HNO_3 to the sample.

c. Add 2.50 mL concentrated HCl to the sample.

d. Cover the sample with a ribbed watch glass or equivalent source.

e. Transfer the digestion vessel to a pre-heated hot plate or equivalent source at 85°C. Take the volume down to between 10 to 15 mL, **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes.**

f. Leave sample on hot plate and gently reflux for 30 minutes. Remove from hot plate and cool.

g. Bring sample to its predigestion volume with DI water in the digestion vessel.

h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

i. The sample is now ready for analysis.

j. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

3. Method 3010A, "**Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy**".

a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel.

b. Add 1.5 mL concentrated HNO_3 to the sample.

c. Cover the sample with a ribbed watch glass.

d. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank must be used, with the temperature

being recorded in the log book. Take the volume down to a low volume (~5 mL), **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of the bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries.** Remove the sample from the hot plate and cool.

e. Add another 1.5 mL portion of concentrated HNO_3 to the sample.

f. Cover the sample with a ribbed watch glass.

g. Transfer the vessel to the hotblock or equivalent source. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

h. Uncover the vessel and evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.

i. Add 2.5 ml of 1:1 HCl (10 mL/100 mL of final solution).

j. Cover the digestion vessel and reflux for an additional 15 minutes.

k. Bring sample to its predigestion volume in digestion vessel.

l. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

Note: When preparing USACE project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

m. The sample is now ready for analysis.

n. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

4 Method 3030C (Standard Methods), "**Preliminary treatment for Acid-Extractable Metals"**

a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a 50 mL digestion vessel.

b. Add 2.5 mL 1:1 HCl to the sample.

c. Heat 15 minutes in a hot bath.

d. Filter through a membrane filter.

e. Adjust filtrate volume to 50 mL with DI water.

f. Transfer to ICP analyst.

D. Solid sample preparation

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- *The material in the sample pan(inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

Grinding of Vegetation Samples

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill

or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. USEPA CLP ILM0 4.1, **"Acid digestion of Soil/Sediment"**

   a. Mix the sample thoroughly to achieve homogenity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a digestion vessel.

   b. Add 10 mL of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass or equivalent source. Heat the sample to 92 to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5.0 mL of concentrated HNO_3, replace with watch glass or equivalent source, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.

   c. After the second reflux step has been completed and the sample has cooled, add 2 mL of Type II water and 3.0 mL of 30% hydrogen peroxide (H_2O_2). Return the heating vessel to the hot plate or equivalent heating source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.

   d. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2.)

   e. If the sample is being prepared for ICP analysis of Al, As, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, and Zn, add 5 mL of 1:1 HCl and 10 mL of Type II water, return the covered heating vessel to the hot plate or equivelent heating source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 50 mL with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Dilute the digestate to 144 mL with DI water, add 5 mLs concentrated HCl and 1 mL of concentrated HNO_3, mix well and place into the appropriate container. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v) HNO_3. The sample is now ready for analysis.

f. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards and ID of matrix spikes and the amounts used for spiking.

2. Method 3050B, **"Acid digestion of Sediments, Sludges and Soils"**

a. Mix the sample thoroughly for 5 minutes using a plastic spatula or Teflon coated spatula in a glass or plastic weigh boat to achieve homogeneity.

b. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the digestion log.

NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.

c. Add 5 mL D.I. water and 5 mL concentrated HNO_3(1:1), mix the slurry and cover with a watch glass. Place the sample in a preheated hot block and reflux at 95°C for 10 to 15 minutes being certain that the sample does not boil. Record temperature in digestion log book

d. Allow the sample to cool. Add 5 mL concentrated HNO_3, replace the watch glass and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) over and over until <u>no</u> brown fumes are given off by the sample indicating the complete reaction with HNO_3. Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at 95°C $\pm$ 5°C for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.

e. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of H_2O_2 if it is still warm.) Cover the vessel with a watch glass and return the sample to the hot block or equivalent source and heat until the bubbling subsides. Care must be taken to

ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Add two more 3 mL portions of H_2O_2 to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2.)

f. Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate at 95°C $\pm$ 5°C without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.

g. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling

h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

i. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle. Invert the 50 ml sample digestion vessel several times to mix the sample and pour sample into the 150 ml of the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.

NOTE1: When preparing USACE project samples, if any sample in a digestion batch requires filtration , all samples (including QC samples) must be treated in the same manner.

NOTE2: To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.

j. The sample is now ready for analysis.

k. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

E. Oils

1. Method 3031, "**Digestion Procedure for Oils**"

NOTE: THIS METHOD IS VERY TIME CONSUMING--DISCUSS SUB-CONTRACTING SAMPLES WITH YOUR SUPERVISOR AS SOON AS THEY COME IN THE DOOR.

a. Homogenize sample and Weigh approximately (to the nearest 0.01 g) a 0.5 g representative portion of the sample into a 250 mL beaker. Separate and weigh proportional aliquots of the phases if more than one phase is present. Record the exact mass in the digestion log. Larger or smaller sample sizes can be used if needed.

g. Add 0.5 g of potassium permanganate powder. If larger sample sizes are used, increase the amount of potassium permanganate so that the ratio of oil to postassium permanganate is still 1:1. Mix the oil and permanganate thoroughly until homogenous. Thick oils and tars that cannot be mixed should be heated to achieve mixing (the oil may react mildly). It is important to record the amount of potassium permanganate used for each sample if analysis is by ICP-AES and correction is to be made for the amount of manganese. If more than 10% of the sample is aromatic material, such as xylene, then the reaction will be incomplete. If this is the case, increase the amount of potassium permanganate. If the sample is a mixture of oil and other non-organic materials, reduce the amount of potassium permanganate.

<u>NOTE</u>: All steps requiring the use of acids should be conducted under a fume hood by properly trained personnel using appropriate laboratory safety equipment. This should include face shields and latex gloves.

h. Cautiously add 1.0 mL concentrated H_2SO_4, and stir with an appropriate stirring device. If larger sample sizes are used, increase the volume of the sulfuric acid so that the ratio of oil to sulfuric acid is 1 g to 2 mL. The H_2SO_4 can be added dropwise or all at once, depending on analytical needs. (Generally, dropwise is preferred when low reporting limits are needed.)

<u>NOTE</u>: To prevent a strong exothermic reaction, H_2SO_4 should be added dropwise to all samples unfamiliar to the analyst and to all samples that are known to be highly reactive.

The reaction can take several seconds to begin, but when it occurs it will be very quick, vigorous, and exothermic. Generally larger sample sizes will react faster than smaller. Likewise, lower average molecular weight materials will react faster than heavier. Do not be mislead by an initial lack of reactivity. A grey-white vapor will be ejected from the beaker (SO_3) and splattering and bubbling can occur. The beaker will become very hot. This step is complete when no more gases are given off and the sample would be a thick black lumpy paste. Allow the beaker to cool as needed.

NOTE: Care must be taken when working with very light organic materials, such as diesel fuels, as they may flash. Generally, the lower the average molecular weight of the meterial correlates to a greater danger of flashing. The danger of flashing is reduced by adding the sulfuric acid dropwise.

NOTE: If more than 10% of the sample is aromatic material, such as xylene, only a little grey-white vapor will form. This will reduce accuracy and complicate nebulization. If there is a signigicant amount of non-hydrocarbon material, a sputtering recation will occur and black MnO_2 particulates will be given off. See section (b.) above under procedure.

i. Add 2 mL of concentrated HNO_3 and stir. This reaction will be slightly exothermic. If larger sample sizes are used, it is not always necessary to increase the volume of HNO_3 proportionately, depending on analytical needs. Some reddish-brown vapor (NO_2) may be given off. Allow the reaction to continue until complete, that is when the digestate no longer gives off fumes. Allow the beaker to cool as needed.

j. Add 10 mL of concentrated HCl and stir. If larger sample sizes are used, it is not always necessary to increase the volume of HCl proportionately, depending on analytical needs. This reaction will be slightly exothermic and gas formation and foaming will occur. Lighter oils will foam more than will heavier oils. If excess foaming occurs, add water to prevent sample loss. Allow the beaker to cool as needed.

k. Heat the beaker until there is no further gas evolution. (temperature should not exceed 150 °C to prevent volatilization). There may be additional foaming or other milder reactions which may result in overflow from the beaker. If excess foaming occurs, either remove the beaker from the heating source until foaming subsides or add

sufficient water to prevent overflow. The final digestate should be a clear yellow liquid with black or dark reddish-brown particulates.

l. Filter the digestate through Whatman 41 filter paper and collect filtrate in a volumetric flask or beaker.

m. Wash the digestion beaker and filter paper, while still in the funnel, with no more than 5 mL of hot HCl.

NOTE: The purpose of this next step is to recover antimony, barium, and silver that may not have been completely solubilized. If the sample is not being prepared for these analytes, the next step may be skipped.

n. (Optional) After having washed the filter paper, remove the filter and residue from the funnel and place it back in the beaker. Add 5 mL of conc. HCl and place the beaker back on the heating source until the filter paper dissolves (temperature should not exceed 150 °C ±5 °C to prevent volatilization). Remove the beaker from the heating source and wash the cover and sides with reagent grade water and then filter the residue and collect the filtrate in the same flask or beaker as in sections f. and g. above. Allow the filtrate to cool and quantitatively transfer to a volumetric flask. Bring to volume.

o. (Optional) If the filtrate is collected in a beaker, the filtrate can be heated again to drive off excess HC1. This can reduce matrix effects in sample introduction (temperature should not exceed 150 °C ± 5 °C to prevent volatilization). When sufficient HCl has been removed, remove the beaker from the heating source, allow to cool, and then transfer the contents to a volumetric flask and bring to volume. However, if too much HCl is removed, barium, silver and antimony can be lost.

p. Analyze the flitrate by ICP-AES. Depending on the final volume selected, the total solids in the digestate may be high enough to cause nebulization problems. Problems due to high dissolved solids may be corrected by 1) following optional Section i., 2) using internal standards, 3) using flow injection analysis, or 4) using other matrix correction procedures.

Manganese Removal Steps

NOTE: The purpose of these next steps is to remove the manganese in the digest by precipitating it as manganese ammonium phosphate