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Sampling and Analysis Plan For Additional Site Investigation To Define VOC Contamination in Portions of AOC 1, Area 82 MCB Camp LeJeune, North Carolina

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> > Submitted to:

Commanding Officer Atlantic Division Naval Facilities Engineering Services Center (NFESC) Norfolk, VA 23511-2699

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Project No. 16032

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This Sampling and Analysis Plan is a site-specific document that will describe the sampling and analysis tasks for AOC-1 Additional Site Investigation. Information regarding the site is detailed in the Site Work Plan.

This plan must be read, understood, and implemented by any or all of the following personnel prior to any field activities:

- Project Manager
- Site Supervisor/Scientist
- Project QC Manager
- Site QA/QC Officer
- Project Chemist
- Field Chemists
- Sampling Technicians

## 1.1 PROJECT TASK

The primary objective of this project is to determine the horizontal and vertical extent of soil VOC contamination for the purpose of completing the remedial design and constructing an effective soil vapor extraction system which will achieve the remedial goals. This will be accomplished by laying out a grid of the areas, collecting soil samples for headspace using direct push sampling methods, and sending samples to a laboratory for volatiles analysis.

## 1.2 DATA QUALITY OBJECTIVES

The task for this project is investigative. Therefore, the decisions made in the field will be critical in assessing whether the objectives for the project are met. Soil samples will be collected for headspace and confirmation analyses using direct push sampling. Although it has been shown that there is little, if any, correlation between headspace results and the actual concentration of volatiles in soil, the data from headspace screening will be used to qualitatively delineate the contaminated areas from the "clean" areas. For the purpose of this project,

any headspace screening results over 10 ppm will be considered as a contaminated sample.

Patterns should arise from this information and will be used in determining which samples will be sent to a laboratory for confirmation analysis. The results from the confirmation samples will be able to provide information regarding the nature and the actual concentration of contaminants in the soil. All confirmation samples will be sent to a Navy-approved and North Carolinaapproved laboratory. Data will include all information required in NFESC Level C packages. Field quality control will also include NFESC Level C requirements.

Ultimate remediation goals for VOCs, from which remedial design for the soil will be based, are as follows:

٠	Benzene	5.4 ug/kg
٠	Trichloroethene	32.2 ug/kg
٠	Tetrachloroethene	10.5 ug/kg

# 2.1 PROJECT MANAGEMENT ORGANIZATION

The project management organization is based on specific project requirements. The project team and their responsibilities are described in this section and on Figure 1. The project manager is the primary focal point for control of the project activities. The project manager will be supported by the QA Management team which will provide reviews, guidance, and technical advice on project execution issues. Members of this staff will be on an "as-needed" basis to assist in smooth project execution. The project manager will be supported by a supervisory, health and safety, and QA/QC staff to ensure that the project is safely executed in compliance with applicable laws, regulations, statutes, and industry codes.

Each specific project will be assigned to a project manager. Reporting to the project manager may be several individuals fulfilling as required the roles of deputy project manager, project chemist, health and safety officer, project engineer, QA/QC officers, and supervisors. These individuals are responsible for fulfilling appropriate portions of the project QA program, in accordance with assignments made by the project manager. The project manager is responsible for satisfactory completion of the project QA program. Specific responsibilities may be assigned by the project manager to the site supervisor and other members of the project staff.

The responsibilities of the key members in the project organization are:

• Project Manager

The project manager is responsible for the overall direction of this project executed under his/her supervision. The project manager provides the managerial administrative skills to ensure that resource allocations, planning, execution, and reporting meet contract requirements. The project manager is ultimately accountable for all work activities undertaken on this project. The global quality-related responsibilities of the project manager can include, but are not limited to the following:







# **QC** Organization Chart

- Organization of the project staff and assignment of responsibilities
- Understanding of contract and scope of work for a specific project
- Communication to the project staff regarding client requirements and QA practices
- Day-to-day execution of the project
- Identification, documentation, and notification to the client and project staff of changes in the scope of work
- Notification to the general manager or office manager if the project cannot be completed be completed with regards to quality, schedule, or cost
- Procurement of subcontractor services
- Supervision of preparation and approval of project-specific procedures, work plans, and QA project plans
- Approval of project design bases, design parameters, drawings, and reports
- Approval of project remedial action/construction methodologies
- Dissemination of project-related information from the client such as design bases, input parameters, and drawings
- Liaison for communications with the client and subcontractors Liaison between the project staff and other internal groups
- Decision of whether or not drawings require independent review

- Serving as the "collection point" for project staff reporting of nonconformances and changes in project documents and activities
- Determination of the effect of nonconformances on the project and the appropriateness for reporting such items to the client, and providing appropriate documentation for reporting
- Notification of project and QA personnel of nonconformances and changes
- Notification of the project staff and, as appropriate, QA personnel of void project-related documents and information
- Determination that changes, revisions, and rework are subject to the same QC requirements as the original work
- Serve as final reviewer prior to release of project information
- Approve and sign outgoing correspondence

Some of these responsibilities may be assigned by the project manager to the Delivery Order Manager and/or the Site Supervisor, who will remain on site throughout the project.

## Site QA/QC Officer

The site QA/QC Officer is responsible for implementing the project plans and ensuring that the quality assurance and data quality objectives are being met for the project. He/she is also responsible for informing the Regional QA/QC Director of any site-specific problems and for coordination QA efforts with the contracted laboratory. His/her specific responsibilities include, but are not limited to:

- Tracking validation data and ensuring adherence to published guidelines
- Determining if the levels of QA are being met for the project

- Certifying the level of QA that has been achieved during the generation of analytical data
- Implementing QA/QC procedures
- Assuring the continuity of chain-of-custody evidence
- Initiating and overseeing all audit functions
- Compiling and submitting all QA Reports (QARs)
- Compiling, revising, updating, and submitting QAPPs or CDAPS
- Reviewing subcontractor's QA Manuals and/or Laboratory Quality Management Plans (LQMPs)
- Ongoing QA/QC training of new and current personnel
- Stopping work if quality objectives are not being met

For this site, the project hydrogeologist will also serve as the site QA/QC officer.

#### Project Chemist

The project chemist will:

- Implement designated QA/QC procedures
- Implement sample acquisition numbering system(s)
- Analyze and interpret all subcontracted technical and laboratory results
- Report all QC data to the Project QA/QC Officer for review and
- Implement corrective actions as required and ordered by the Project QA/QC Officer

# Field Technician

The field technician will:

- Carry out approved sampling and QC methods
- Fill out sample tracking forms and related analytical and QC forms and logbooks
- Report QC data to the project chemist

## Sample Technicians

The sample technicians will be responsible for:

- Carrying out all sampling in accordance to approved procedures and methodologies as defined in the QAPP
- Generating trip blanks, field blanks, equipment blanks, and acquiring field duplicate samples as required by the QAPP
- Completing sampling logbooks, sampling forms, and chain-of-custody forms.

# 3.1 SAMPLING GRID

An accurate sampling grid will be established for each of the two sampling areas prior to any sampling activities. Figure 3.1 shows these areas along with the proposed sampling grids. The grid (40'-50' spacing) will be established by the Site Supervisor and expanded or reduced as necessary. Sampling will begin near previous sample points of each area and extend outward along the established grid.

# 3.2 SOIL SAMPLING

Soil samples will be collected using the direct push sampling method. These will be collected for headspace and confirmation analyses beginning near Baker's sample points for each area and will extend outward along the grid. Approximate groundwater levels will also be recorded at several locations relative to the site topography using four to eight 1-inch diameter piezometers which will be installed using direct-push methods to establish a pilot hole, then an enlarged hole (to approximately 1-7/8 inches). The 1-inch slotted PVC screen and riser can then be set and sand-packed as a temporary or permanent well point.

On-site equipment which will be used for direct push sampling and well point installation will include a truck-mounted or ATV-mounted "cone penetrometer" rig with attachments specifically designed for environmental sampling. This rig utilizes a hydraulic hammer unit that advances a small diameter hollow steel probe to the desired depth. Samples from discrete depths will be collected using the following criteria:

- If groundwater is determined to be greater than 12 feet below ground surface, then three samples at varying depths will be collected.
- If groundwater is determined to be 10 feet or less below ground surface, then a minimum of two samples at varying depths will be collected.



An aliquot of each sample will be transferred from the probe tube into a ziploc bag for headspace screening. The remainder of the sample in the tube will be transferred into a specialty-cleaned 4 oz glass container with a teflon-lined lid. This sample jar will be labeled, sealed in a ziploc bag, and placed on ice in a cooler until a determination is made as to whether or not it will be sent to the laboratory for confirmation analysis. The sample aliquot will then be screened per the method described in section 3.2.1. The result will then be documented in the field log book and later plotted on the grid map, if necessary.

When all sample points and depths of concern have been screened and plotted, the map will then be studied by the Site Supervisor/Scientist. Samples will be sent to the laboratory using the scheme shown on Table 3.1.

Sample Collection	Sample Collection I Quantity or I Analytical I Turn sound I Right				
Location	Frequency	Parameter	Time Required		
All sample points and depths	100% of all samples points and depths	Headspace Screening (field procedure)	15-45 minutes	10% FD	
Area A: Clean areas	5-10 samples at varying depths and locations	Volatiles (8240) (laboratory procedure)	5 days	1 TB per cooler, 1 FB per sampling event, 1 RB per sampling event, 10% FD	
Area A: Contaminated areas	15-20 samples at varying depths and locations (Note: More samples may be collected if the proposed grid area is exceeded.)	Volatiles (8240) (laboratory procedure)	5 days	1 TB per cooler, 1 FB per sampling event, 1 RB per sampling event, 10% FD	
Area B: Clean areas	5-10 samples at varying depths and locations	Volatiles (8240) (laboratory procedure)	5 days	1 TB per cooler, 1 FB per sampling event, 1 RB per sampling event, 10% FD	
Area B: Contaminated areas	30-40 samples at varying depths and locations (Note: More samples may be collected if the proposed grid area is exceeded.)	Volatiles (8240) (laboratory procedure)	5 days	1 TB per cooler, 1 FB per sampling event, 1 RB per sampling event, 10% FD	

## Table 3.1 **Confirmation Sampling Scheme**

Notes:

\* Contaminated areas entail headspace screening results at greater than 10 ppm. TB = Trip Blank, FB = Field Blank, RB = Rinsates Blank, FD = Field Duplicates

### 3.2.1 Headspace Screening

Headspace screening will be performed using an Organic Vapor Analyzer (OVA) on sample aliquots at varying locations and depths. This method will provide a qualitative picture of the horizontal and vertical extent of contamination. The result represents a total concentration of volatile organics being released from the soil sample. The screen will be performed using the following method:

- 1) Calibrate the OVA per the manufacturer's instructions using Methane as the gas standard.
- 2) Zero the OVA by taking a reading in an analyte-free area.
- 3) Place an aliquot of the soil sample in a ziploc bag and seal the bag.
- 4) Allow the sample to come to an equilibrium in the bag, i.e. let the bag sit for approximately 5-10 minutes.
- 5) Unzip the bag, allowing only the tip of the OVA probe to enter the bag. Do not let the probe come in contact with the soil.
- 6) Take a reading and record it in the field log book.
- 7) For a designated number of samples (10 percent), prepare a field duplicate for headspace screening by transferring 2 aliquots of the same sample into 2 different bags and analyze as specified above.

## 3.2.2 Confirmation Samples

At the end of each day, samples chosen to be sent to the laboratory will be shipped via Federal Express. These will be documented, packaged, and shipped as specified in this plan.

#### 3.3 SAMPLE IDENTIFICATION

All samples collected on-site will be provided with a unique sample designation.

The number will serve to identify the site, location, grid and specific sample number. The sample designation format will appear as follows:

## A(x,y)D

Where:

A or B = Particular test area within wooded part of area 82, north of lot 203, west of Piney Green Road, and south of Wallace Creek (Figure 1)

(x,y) = Grid Coordinates of sample

D = Depth of sample

Dup = Field Duplicate designation, when applicable

Examples:

#### B(2,5)6

Denotes the sample removed from sample area B at grid coordinates x = 2, Y = 5, at 6 feet below land surface.

## 3.4 SAMPLE PRESERVATION

All environmental samples, as well as QA/QC samples, will be preserved by cooling with ice or refrigeration to a temperature of  $4^{\circ}$ C prior to shipment to the analytical laboratory. This temperature should be maintained during shipment by placing ice in plastic zip-loc bags, and placing the bags above and below the sample containers. All other shipping guidelines listed in following sections will be followed.

## 3.5 QA/QC SAMPLES

The appropriate number of field QC samples will be collected during this project. These samples will include trip blanks, field blanks, rinsate blanks and duplicate samples. These samples will be collected at the following frequencies:

• Field Blanks – Field blanks consist of the source water used in decontamination and steam cleaning. At a minimum, one field blank

from each sampling event and each source of water must be collected and analyzed for the same parameters as the related samples.

- Rinsate Blank Equipment rinsate blanks are the final analyte-free water rinse from equipment cleaning collected daily during a sampling event. One equipment rinsate blank must be collected daily for NFESC Level C reporting.
- Field Duplicate Duplicates for soil samples are collected, homogenized, and split. All samples except Volatiles are homogenized and split. Volatiles are not mixed, but select segments of soil are taken from the length of the core and placed in 4 oz glass jars. Field duplicates must be collected at a frequency of 10% per sample matrix for Level C reporting. All the duplicates should be sent to the primary laboratory responsible for analysis, along with the samples. The field duplicates should be used by the laboratory to prepare the laboratory duplicate or matrix spikes.
- Trip Blank -- Trip blanks are defined as samples which originate from analyte-free water taken from the laboratory to the sampling site and returned to the laboratory with the volatile samples. One trip blank should accompany each cooler containing volatiles, should be stored at the laboratory with the samples, and analyzed by the laboratory. Trip blanks are only analyzed for volatile organic compounds.

#### 3.6 PERSONAL PROTECTION

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It has been anticipated that activities in AOC-1 (area 82) will be conducted in Level C. Level C will include tyvek overgarments, steel toe boots with tingley overboots, nitrile gloves, respiratory protection and hard hats. Refer to the Health and Safety Plan for this project for more details.

#### 3.7 EQUIPMENT DECONTAMINATION

All sampling equipment (hand augers, spoons, steel probes, etc.) will be decontaminated before sampling commences, between each sample location,

and prior to leaving the site. The procedures for decontamination of equipment are described below.

- 1) Remove gross contamination by scraping or brushing
- 2) Clean with tap water and liquinox, using a stiff brush to remove all surface contaminants
- 3) Rinse thoroughly with tap water
- 4) Rinse thoroughly with deionized water
- 5) Rinse twice with isopropanol
- 6) Rinse thoroughly with organic-free water and allow to air dry. The final rinse will be collected in a 40 ml VOA vial and labeled as the rinsate blank for the day.
- 7) Wrap equipment with aluminum foil prior to storage or transportation to sample locations

Decontamination fluids will be collected in properly labelled 55-gallon drums, and staged in a secure area until final disposal.

#### 3.8 SAMPLE LOG BOOK

It is necessary for the sampling crew to maintain daily field notes. Items that must be included are sampling protocol, any changes to the procedures, meetings, instructions, safety precautions, personnel protection, and activities pertaining to the samples. The person taking notes must be knowledgeable enough about these activities to know which details are important.

Repetition of information recorded in other permanent logs should be avoided, but enough should be recorded to present a clear and accurate picture of technical activities. At a later date, should a question arise concerning a specific

event or a procedure used, it will be answered from these notes. Some items that would be considered noteworthy are as follows:

- Termination of a sample point or parameter and reasons
- Unusual appearance or odor of a sample
- Measurements, volume of flow, temperature, and weather conditions
- Additional samples and reasons for obtaining them
- Levels of protection used (with justification)
- Meetings and telephone conversations held with clients, regulatory agencies, citizens, OSC, project manager, or supervisor.
- Details concerning any samples split with another party
- Details of QC samples obtained

These notes must be dated and signed (each page) for validity in a court of law. All log book entries will be made with indelible ink and legibly written. The language will be factual and objective. No erasures will be permitted. If an incorrect entry is made, the error will be crossed out with a single strike mark, initialed, and dated. When audits are performed, the auditor's remarks and decisions must also appear in these notes. These audits should be followed up by written report submitted by the auditor, including opinions and conclusions. A copy of this report should be placed in the project file and one copy kept in the sampling file for easy reference.

All samples should be logged in the logbooks. The following columns are standard for all projects:

1) DATE -- Date sample was obtained

- 2) SAMPLE NUMBER -- Consecutive series of numbers which are assigned to every sample.
- 3) LOCATION -- Description of area sampled
- 4) TIME -- Military time sampled
- 5) SAMPLERS -- Initials of persons obtaining sample (usually two, at least witnessing if not involved in actual sampling task)
- 6) DESCRIPTION OF SAMPLE -- Physical description of sample (e.g., clear, cloudy, odor)
- 7) WEIGHT OR VOLUME -- Size of sample (500ml, 1L, etc)
- 8) DATE RESULTS ARE DUE -- Date analytical results should be reviewed
- 9) LABORATORY -- Laboratory who performed analytical work
- 10) RESULTS -- Will vary according to project requirements; should be in consistent units (ppm, ppb, etc.,) when possible
- 11) CHAIN-OF-CUSTODY NUMBER-- For samples sent to laboratory or given to client
- 12) ADDITIONAL COMMENTS -- Space reserved for any other information concerning particular sample or special procedure or analysis.

13) PRESERVATIVES -- Preservatives used or included by the lab

14) DATE SAMPLES SENT -- Date samples were sent to the lab

15) AIRBILL NUMBER

The following guidelines will be implemented for all log books:

- Each page will be signed, dated, and numbered;
- Blank pages will be identified as such;
- The time of each entry will be noted (24 hour clock);
- Logbook extensions (field sheets, purge records, etc.) will be recorded in the logbook; and
- Logbooks will be returned to the on-site coordinator upon completion, during periods of absence, and at the end of the investigation.

#### 3.9 SAMPLE LABELS

Samples other than in situ measurements are identified by a sample label attached to the sample. Included on the label are the following information:

1) JOB NUMBER

. .

- 2) DATE -- Month, day, year
- 3) TIME -- Military time
- 4) SAMPLE NUMBER -- see Table 1 for designations
- 5) SAMPLE DESCRIPTION
- 6) TAKEN BY -- Sampler name
- 7) WITNESS

The information described above should be printed neatly using an indelible marker. After the sample is taken and the label is securely attached, the sample is logged into the sample log book.

#### 3.10 CUSTODY SEALS

Custody seals are narrow strips of adhesive tape of glass fiber used to demonstrate that no tampering has occurred. They may be used on sampling

equipment, sample transport containers, and individual sample jars. They should be signed and dated by the sampler and placed from one side, across the top, and to the other side of the sample bottle or across the opening of the sample transport containers.

#### 3.11 CHAIN-OF-CUSTODY (COC) PROCEDURES

Because of the evidentiary nature of samples collected throughout the project, the possession of samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. To maintain and document sample possession, chain-of-custody procedures are followed as described below:

A sample is under your custody if:

- 1) It is in your actual possession, or
- 2) It is in your view, after being in your physical possession, or
- 3) It was in your physical possession and then you locked it up to prevent tampering, or
- 4) It is in a designated secure area.

COCs specific to each sampling event have been provided in the Appendix of this plan. The following information is required on the COC:

- 1) PROJECT NAME
- 2) PROJECT LOCATION -- City and State in which the project is located
- 3) JOB NUMBER
- PROJECT CONTACT -- OHM employee responsible for overseeing the sampling operation. This person should be the individual to whom questions are to be directed or verbal results given (Project Manager, Site Supervisor, or Project Chemist)

- 5) PROJECT TELEPHONE NUMBER -- Telephone number of on-site office trailer or number where person responsible for samples can be contacted.
- 6) DATE -- Month, Day, Year
- 7) TIME -- Military time

8) SAMPLE IDENTIFICATION -- Sample number/location

9) BOTTLE SIZE -- 12 ounces, 8 ounces, 1 liter, etc.

10) ANALYSES REQUESTED

11) LABEL, TAG NO./ REMARKS

12) AIRBILL NO

13) LABORATORY -- Laboratory where samples are to be sent

14) PHONE -- Telephone number of laboratory

15) ATTN -- Contact for laboratory

16) RELINQUISHED BY -- Signature of sender (OHM)

17) DATE -- Date samples are sent

18) TURNAROUND TIME -- Turnaround times requested or date the results are required from the lab.

The COC needs to be sealed in a ziploc bag and taped in place on the underside of the top of the sample transport container (cooler).

#### 3.12 SHIPMENT OF SAMPLES

Samples will be shipped via Federal Express to the appropriate laboratory. Also, COCs have been prepared accordingly and are organized according to sampling events.

The following instructions are for shipping samples with unknown or limited hazards. NO CHANGES OR SUBSTITUTIONS TO THESE INSTRUCTIONS ARE ALLOWED – NO MATTER HOW INSIGNIFICANT THEY MAY SEEM.

- 1) Samples must be shipped in "strong outer packaging". A plastic cooler is acceptable.
- 2) Both the shipper's and receiver's addresses must be on the container.
- 3) The following shipping name must be printed on the container:

OTHER REGULATED SUBSTANCES, ID # 8027

- 4) A Class 9 hazardous material shipping label must appear on the top of the box.
- 5) Inner packages cannot exceed 1 gallon each, and the entire shipment (cooler, samples, and absorbent) cannot exceed 66 lbs.
- 6) Coolers must be packed with absorbent such as vermiculite or kitty litter.
- 7) Inner containers should have their lids secured with tape or wire.
- 8) The materials must be shipped using a Federal Express Hazardous Materials Airbill
- 9) Refer to Figure 6.1 (next page) for details on how to fill out the Federal Express Hazardous Materials Airbill.

10) Any questions regarding shipment of samples should be referred to Tom Mears in the Norcross OHM office.

MCB Camp Lejeune SAP/Additional Site Information 3-14

OHM/16032

Data management is the system by which data is reduced, reviewed, validated, reported, distributed, and finally archived. The criteria in this system are designed to meet the project objectives.

## 4.1 DATA REDUCTION

Data reduction includes the identifications and calculations necessary to convert the raw instrument readings to the final reported compounds and their respective concentrations.

## 4.1.1 Laboratory Data Reduction

The following paragraphs outline the data reduction plan for the collected, data criteria used to validate the date and the decision flow from raw data to the validated concentrations.

These criteria will be used by the contracted analytical laboratory. More detailed procedures should be included in the laboratory's Quality Assurance Plan (QAP).

## Responsibilities of Analyst

**\*\*\*** 

Each analyst is responsible for converting raw data into reportable values. These specific duties include:

- Proper identification of the analyte
- Generation of calculations
- Checking all calibrations to ensure support of data
- All QA/QC checks are supportive of data
- All documentation is complete and accurate in respective log books

• All chromatograms and strip chart recordings are labeled with data, instrument number, run parameters and analyst

#### Gas Chromatograph/Mass Spectrometry Results

Qualitative identification of an analyte is determined by obtaining the extracted ion current profiles (EICPs) for the three identifying mass ions and following the criteria listed below:

- The intensity of the three characteristic masses of each analyte must maximize within one scan of one another.
- The relative peak height ratios of the three characteristic masses must be within ±25 percent when compared to the mass spectrum of the reference standard analyte.
- The relative retention time of the suspect peak must be with ±0.06 of the standard reference peak.

In order to list structural isomers as separate analytes, they must have acceptable resolution. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25 percent of the sum of the two peaks. Otherwise, structural isomers must be identified as unresolved isomeric paris.

GC/MS Volatiles

$$ug/L(ug/kg) = \frac{As(AMT)(D)}{Ais(RF)(Vf)(P)}$$

Where:

As = Area response for targeted analyte

AMT = Amount of internal standard in ng

D = Dilution factor if necessary

Ais = Area of internal standard

Vf = Volume purged in ml or g

P = Percent solids in decimal (if results in dry weight are needed)

RF = Response factor from standard analysis calculated as

 $RF = \frac{As (AMTis)}{Ais (AMTs)}$ 

Where:

As = Area response for targeted analyte AMTis = Amount of internal standard in ng Ais = Area response of the internal standard AMTs = Amount of the targeted analyte in ng

#### 4.2 LABORATORY DATA VALIDATION

All data generated within the laboratory will be extensively checked for accuracy and completeness. The data validation process consists of data generation, reduction, and three levels of review.

The analyst who generates the raw data has the prime responsibility for the correctness and completeness of the data. All data generated and reduced follows protocols specified in the laboratory (SOP). Each analyst reviews the quality of his work based on an established set of guidelines. The guidelines are:

- Sample preparation information is correct and complete
- Analysis information is correct and complete
- The appropriate SOPs have been followed
- Analytical results are correct and complete
- QC samples are within established control limits
- Blanks are within appropriate QC limits
- Special sample preparation and analytical have been met
- Documentation is complete

The next level of review is performed by the section supervisor or data review specialist. The review is structured to ensure that:

• Calibration data are scientifically sound, appropriate to method, and completely documented.

- QC samples are within established limits.
- Reporting units are consistent with the method and the matrix.
- Quantitative results are correct.
- Data results are consistent with information on the COC.
- Documentation is complete.
- The data is ready for incorporation into a final report.
- The data package is complete and ready for data archive.

The second level of review is structured to ensure all calibration data and QC sample results are reviewed and all of the analytical results from 10 percent of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is complete. If problems exist, an additional 10 percent is reviewed, the process continues until no errors are found or the package has been reviewed in its entirety.

The final level of review by the laboratory comes from the program administrator or laboratory QA Officer. He/she reviews the report to ensure that the data meets the overall objectives of the project.

Once the data has been validated, it is ready for report production. The report will contain:

- Description of sample types
- Tests performed, problems encountered during testing
- Dates sampled
- Date received

- Date extracted
- Analytical results
- Reportable limit
- QC information: percent recovery, relative percent difference, control limits, blanks analyses, matrix spikes, and other additional special QC information
- Qualifiers for data falling outside of QC limits
- Methodology
- Name of the analyst
- Signature of laboratory representative
- Dual column confirmation results
- Calibrations (when requested)
- Instrument performance checks (when requested)

The report from the laboratory will also include a copy of the original COC for the samples analyzed.

## 4.3 PROJECT DATA REVIEW

#### 4.3.1 Field Chemist Data Review Responsibilities

The field chemist is responsible for initial review of the data from the laboratory. This review includes:

• Verifying that all requested data are reported

- Verifying that samples are analyzed according to the contract specified method
- Verifying that holding times are not exceeded
- Verifying that matrix spike, matrix spike duplicate, and surrogate recoveries fall within the laboratory's acceptable criteria
- Reviewing blank data for gross contamination
- Reviewing field quality control results for gross inconsistencies

The field chemist is then responsible for informing the Project Manager and Project QA/QC Officer of any laboratory and/or sampling deficiencies or issues. The field chemist alone should not make decisions on the acceptability of the data. These issues and subsequent decisions will be documented on a weekly report to the Regional QA/QC Director and Project Manager.

#### 4.3.2 **Project QC Officer Data Review Responsibilities**

The Project QC Officer is responsible for interfacing with the project chemist, project manager, and the laboratory's QA Officer to resolve any QA/QC issues affecting the data. He/she is also responsible for finalizing any QA/QC issues with the laboratory and/or the project chemist. This includes obtaining a corrective action from the parties involved.

#### 4.4 PROJECT DATA VALIDATION

Data validation is an extensive review of the data for technical and legal validity. This procedure will be performed only by qualified and experienced personnel either within OHM or by a consultant. This procedure is expensive and should be considered on projects with a high probability for litigation. In general, it is used for Superfund projects in which the highest level of data quality is necessary.

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The guidelines to be used for data validation will be the USEPA National Functional Guidelines for Data Validation of Organics and Inorganics. For this project, data from the confirmation samples will be validated by OHM personnel prior to submittal to the Navy.

### 4.5 DATA REPORTING

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Generally, preliminary data is faxed to the project manager. This data may or may not have undergone the full laboratory review process and may contain errors and discrepancies. Approximately two days later, the hard and final copy will be received on-site and will be reviewed by the project chemist or scientist, the project manager, and the site QC officer. Any discrepancies will be brought to the Regional QA/QC Director's attention, who will contact the laboratory regarding the issues.

When QA issues have been satisfactorily settled and data validation has been completed, the project manager may release the data to the client and/or regulating agencies.

#### 4.6 DATA STORAGE AND ARCHIVE

After OHM has completed its work for the project, all documents generated will be assembled in the project file. Individuals may retain clean (no handwritten comments) copies of documents for their personal files but only after personally verifying that the original or similar copy is in the project file. The project manager/supervisor is responsible for ensuring the collection, assembly, and inventory of all documents relative to the project at the time the objectives are met. The file then becomes accountable. Any records leaving the file must be signed out.

When a contractor has completed the project objectives, all file documents are reviewed and submitted to the general file. The project file contains the following document classes:

A. Project logbooks

- B. Drum logs and other forms
- C. Sample identification documents
- D. Chain-of-custody records
- E. Analytical logbooks, laboratory data, calculations, graphs, etc.
- F. Correspondence
  - Intra-office
  - Client
  - Regulating agencies
  - Record of confidential material
- G. Report notes, calculations, drafts
- H. References, literature
- I. Sample (on-hand) inventory
- J. Check-out logs
- K. Litigation documents
- L. Miscellaneous photographs, maps, drawings, etc.

Once deposited in the file, documents must be checked out.

The final report is usually generated by use of computer. A back-up copy of the report on diskette is filed along with the project file. The original report remains in the hard drive of the computer until such a time is required to download it on a diskette. This diskette is also archived.

All information under the corresponding project number is maintained in the archive system for eight years. All archives are accessed by the archives file master list which is maintained in a separate location from the archives.

Reliability in analytical determination is maintained through strict adherence to quality control procedures. Procedures are designed to control both the accuracy and precision of analytical results. Depending on the level of certification of the data, a known method spike is routinely analyzed to ensure the accuracy of results. The procedure is to run the standard QA/QC and sample analysis with each lot of samples sent to the laboratory. If more than ten individual analyses are made, additional standards will be analyzed at a rate of one standard per ten analyses. Some procedures call for the use of either a surrogate spike or the standard addition of a known quantity of the analyte to a split of the sample being analyzed.

Control charts will be prepared using an estimate of the spike recovery obtained from the literature or determined by repeated analyses run in the laboratory. Each time the analyst runs a method spike, the results is entered on the control table. If a standard addition technique is used, a plot of instrument response versus added analyte concentration is made in order to determine analyte concentration in the original sample. These are further explained in the laboratory's QAM.

Replicate analyses will be performed on at least 10 percent of the samples processed by the laboratory. A record of the precision of most analyses is kept by calculating and plotting the industrial statistic I (which is equivalent to the coefficient of variation). Blanks are also run with each batch of samples or individual sample analyzed regardless of the level of certification of the data.

The purpose of spikes, blanks, and replicates is to provide a sound scientific basis from which the degree of certification of the resultant data can be objectively concluded. These are not management decisions, but follow naturally from the results of the above QC procedures.

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#### 5.1 ACCURACY

Data accuracy is a reflection of the efficiency of the analytical procedure. It is determined by use of spiked samples and standard reference materials or laboratory control samples performed at the rate of one set every 20 samples. A control chart is generated using historical laboratory data where warning and control limits are established to assess data accuracy.

The accuracy (check standards) samples will have concentration values of the mid-standard. During analysis, a minimum of 10 percent of samples must be accuracy samples. The accuracy samples must be staggered through the analysis, not placed one after another. After a minimum of seven accuracy samples are analyzed, the percent recovery is calculated for each sample.

The accuracy criteria is determined by calculating the standard deviation of seven or more percent recovery values and setting the upper and lower control limits using the following equations:

Upper control limit = p + 3SDLower control limit = p - 3SD

Where:

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p = Average percent recovery
SD = Standard deviation

After the standard deviation, for the seven or more samples has been calculated, the accuracy control limits will be used to determine if the analysis is out of control. This is done by checking the results against the control limits. If any values are above the upper control limit or below the lower control limit, all sample results after the last qualifying accuracy sample must be repeated or discarded. If seven consecutive values fall below the lower control limit, new limits must be calculated using the new accuracy check values. If the values fall between the upper and lower limits, then conditions are reported as "within limits."

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## 5.1.1 Recovery Control

Recovery control is necessary to determine if the sample matrix is interfering with the constituent being analyzed. A minimum 5 percent of samples will be recovery check samples (matrix spikes). Samples involving different types of matrices must have at least one recovery check for each type.

Control limits will be determined for each matrix, determining the deviation for seven or more percent recovery values.

#### 5.2 PRECISION

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Duplicate and replicate samples analyzed by the laboratory assess the precision of the sampling effort. Control limits for duplicate/replicate RPDs is set at 0 to 20 percent to provide interim guidelines. Once a sufficient amount of replicate data becomes available, field precision control charts are constructed similar to the laboratory precision charts. For any given concentration, the mean and the standard deviation(s) of the replicates are calculated. The mean is the centerline of the control chart. Data from each sample set are pooled with the previous sample sets to generate control and warning limits for the next set. Warning and control limits for water samples are set at  $\pm 2s$  and  $\pm 3s$ , respectively. Control limits for solid samples are more liberally established due to matrix heterogeneity. Data outside any control limit are subject to QA review.

Precision is based upon the results of the relative percent differences as calculated from the percent recoveries of the matrix spike and duplicate samples. The control limits for precision is based on historical laboratory data.

Present practice is to include MS and MSD samples on a per batch basis or a minimum frequency of 5 percent. Duplicate results are compared and the relative percent difference (RPD) is then determined.

The RPD will be entered into the laboratory's data system and will be used to define the precision of the analysis. This value should be less than 20 percent.

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## 5.3 COMPLETENESS

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The field supervisor is responsible for ensuring that all field instrumentation and equipment are functioning properly and calibrated according to set procedures, and that all data are recorded accurately and legibly. In addition, the field supervisor must ensure all sites are sampled for all the specified analyses, that sufficient sample volume has been provided to complete those analyses, and that all of the QA samples have been included with each sample set. The goal for completeness for each sample set shipped to the laboratory is 100 percent. The minimum acceptable completeness limit is 95 percent.

Completeness is expressed as the percentage of the amount of valid data obtained to the amount of data expected. For a set of data to be considered complete, it must include all QC data verifying its accuracy and precision.

If samples analyzed do not meet all QC requirements in terms of accuracy and precision for any specific parameter, the sample preparation and analysis will be repeated pending adequate volume.

## 5.4 CRITERIA FOR REJECTION OF OUTLYING MEASUREMENTS

There are many statistical tests for rejection of outlying data points obtained from a set of measurements from a single population. A test recommended in "Statistical Manual of the Associate of Official Analytical Chemists," 2nd Edition, W. J. Youden and E. H. Steiner, 1975, pg. 86, is the Dixon Test. This test is not dependent on the distribution of the data and can be used for as few as three measurements. A more complete description for this broadly applicable test can be found in the referenced text.

Another reference is the USEPA National Functional Guidelines for Data Validation of Organics and Inorganics. Also, specific programs may have quality objectives with criteria for rejection of outlying measurements.

# 5.5 METHOD DETECTION LIMITS AND PRACTICAL QUANTITATION LIMITS

A number of terms have been used, by the EPA and other technical groups, to express the lowest concentration of an analyte which can be measured. Some of these terms, their definitions, and sources are listed in Table 5.1.

It is not always possible to use RLs because of the regulating body's or client's requirements. In this case, the MDLs required will be provided by the regulating body or by the client.

## 5.6 LABORATORY AND FIELD CONTAMINATION

It is not unusual to find the following analytes at trace levels in the samples:

- Methylene chloride
- Acetone
- Freon (1,1,2-trichlorotrifluorethane)
- Bis(2-ethylhexyl)phthalate
- Hexane
- Isopropanol
- 2-Butanone

These are common solvents used in the field and in the laboratory.

In order to fully evaluate data containing trace levels of these contaminants, one must have data from trip blanks, field blanks, equipment blanks, and all applicable laboratory blanks for that batch of samples.

The determination on the use of the data will be made during the Data Validation process.

#### TABLE 5.1 DEFINITION OF DETECTION LIMIT TERMS

	DEFINITION	DETERMINATION	CALCULATION	SOURCE
Detection Limit (DL)	The concentration which is distinctly detectable above, but close to a blank.	Analysis of replicate standards	Two times the standard deviation that is derived from procedures used to determine MDL.	Methods for Chemical Analysis of Water and Blank.
Limit of Detection (LOD)	The lowest concentration that can be determined to be statistically different from a blank.	Analysis of replicate samples	Three times the standard deviation	ACS Definition
Method Detection Limit (MDL)	The minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the ana- lyte concentration is greater than zero.	Analysis of a minimum of seven replicates spiked at 1 to 6 times the expected detection limit.	The standard deviation times the Student t- value of the desired confidence level. (For seven replicates, the value is 3.14).	40 CFR 136 Definition for PA Water Programs
Instrument Detection Limit (IDL)	The smallest signal above background noise that an instrument can detect reliably.	Analysis of three replicate standards at concentrations of 3-6 times the detection fimit.	Three times the standard deviation	Contract Laboratory Program
Method Quantitation Limits (MQL)	The minimum concentration of a substance that can be measured and reported	Analysis of replicate samples	Five times the standard deviation	SW-846
Limit of Quantitation (LOQ)	The level above which quantitative results may be obtained with speci- fied degree of confidence.	Analysis of replicate samples	Ten times the standard deviation	ACS Definition
Practical Quantitation	The lowest level that can	Interlaboratory	1) ten times the MDL	RCRA
cirini (FGC)	within specified limits of precision and accuracy during routine laboratory operating conditions.	anaysis of the k samples	2) Value where 86% of laboratories are within 20% of the true value	SDWA Programs
Contract Required Detection Limit (CRDL)	Reporting limit specified for laboratories under contract to the EPA for Superfund activities.	Unknown	Unknown	Contract Laboratory Program

OHM takes very seriously its responsibility to report technically defensible data. Therefore, we have established a Reporting limit (RL) for each analyte in each method. The RL represents the value above which were believe reliable data can be routinely obtained.

These Reporting Limits were established by collecting Method Detection Limit (MDL) data and Instrument Detection Limit (IDL) data for analyses from the laboratory. The MDL data were collected using the procedures described in 40 CFR 136 Appendix B. IDL data were calculated using the procedures outlined in the EPA Contract Laboratory Program (CLP) Statement of Work dated December 1987. The MDL/IDL data were then compared to various limits published in EPA methods and in the regulations. For example, for volatile organics, the MDL data generated in OHM field laboratories were compared to the Practical Quantitation Limits (PQLs) published in SW-846 Method 8240; the PQLs contained in the July 9, 1987 Federal Register Final Rule-making on Appendix IX; the Contract Required Detection Limits (CRDLs) in the CLP Method for volatile organics; and the MDLs in Method 624. Then a Reporting Limit for each analyte, considering all of this information, was established. The RL was set at a level above which we were confident that our laboratories could detect and quantify the analyte consistently. Using this procedure, the Reporting Limits established are generally between 2 and 5 times the laboratory MDL/IDL. This range is consistent with the American Chemical Society definition for the Limit of Quantitation (LOQ).

Audit is defined as systematic check to determine the quality of operation of field and laboratory activities. It is comprised of the following:

- Performance audit
- System audits

These include a detailed review of each operating component of the network. Auditing will ultimately assist in determining if each element within a system is functioning appropriately per the QA program requirements.

## 6.1 FIELD PERFORMANCE AUDITS

Field performance audits are performed on an ongoing basis during the project as field data is generated, reduced, and analyzed. All numerical analyses, including manual calculations are documented. All records of numerical analysis are legible, of reproduction quality, and supporting to complete permit logical reconstruction by a qualified individual other than the originator.

Other indicators of the level of field performance are the analytical results of the blank, duplicate, and replicate samples. Each blank analysis is an indirect audit of effectiveness of measures taken in the field to ensure sample integrity. The results of the field duplicate and replicate analysis is an indirect audit of the ability of each field team to collect representative sample portions of each matrix type.

## 6.2 FIELD SYSTEM AUDITS

System audits of site activities are accomplished by an inspection of all field activities by the Project QC Officer. This audit is composed of comparisons between current field practices and standard procedures. The following is a list of criteria to be used in the evaluation of field activities:

• Overall level of organization and professionalism

- All activities conducted in accordance with work plan
- All procedures and analyses conducted according to procedures outlined in this document
- Sample collection techniques versus the site sampling and analysis plan or CDAP
- Level of activity and sample documentation
- Working order of instruments and equipment
- Level of QC conducted by each field team
- Contingency plans in case of equipment failure or other event preventing the planned activity from proceeding
- Decontamination procedures
- Level of efficiency which each team conducts planned activities at the site
- Sample packaging and shipment

After the audit, any deficiencies are discussed with the field staff, and corrections are identified. If any of these deficiencies might affect the integrity of the samples being collected, the QA Officer informs the field staff immediately, so corrections can be made. The field performance audit will be conducted at the start of the project, one before the end of the project, and as directed by the project manager.

OHM will also submit to all requests by regulatory agencies, or other clients for external field systems audits.

## 6.3 LABORATORY PERFORMANCE AUDIT

The laboratory performance audit verifies the ability of the laboratory to correctly identify and quantitate compounds in blind check samples submitted by an auditing agency. If the laboratory participates in Performance Evaluation (PE) programs such as USEPA WS/WP studies, AIHA, PAT studies, etc., results from these studies will be generally acceptable by OHM. However, during the course of the project, it may be necessary for the Project QA/QC Officer to send PE samples to the laboratory to evaluate specific parameters.

The contracted laboratories will undergo performance audits throughout the project consisting of field QC samples. Occasionally PE samples will be supplied by the client or external organizations which will be spiked with the same analytical parameters that are being investigated on site. External laboratory performance audits by auditing agencies such as the USEPA, USACE-MRD, DOD, NFESC, etc, are not routinely scheduled. However OHM and its subcontracted laboratories will submit to any external audit upon request by the USEPA or the client.

## 6.4 LABORATORY SYSTEM AUDITS

The laboratory system audit is a review of analytical laboratory operations to verify that the facility has the necessary equipment, staff, and procedures in place to generate acceptable data. It is also to determine that each element within an activity is functioning appropriately and within the guidelines of applicable methodology, approved procedures, and the site QAPP. An on-site inspection is routinely performed by the laboratory's QA Manager and may also be frequently performed by the OHM Project QC Officer. If the laboratory participates in certification programs, audits performed by the certifying agencies may satisfy the criteria of systems audits for the project.

If the laboratory is in question, a system audit can be directed by the client and performed by OHM or the client's representative. Any recommendations made will be considered for implementation and any corrective actions will be taken to correct any deficiencies found. Project-specific audit reports will be placed in the project files and laboratory audit reports will be kept by the laboratory for future reference.

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Corrective actions may be necessary as a result of the following QA activities:

- Field and laboratory performance audits
- Field and laboratory system audits
- Inter-laboratory comparison studies
- Calibration data fall out of specified limits
- Failure to adhere to the CQMP
- Failure to adhere to the site CDAP
- Failure to adhere to standard operating procedures and methods
- Data completeness below required limits
- Control limits are exceeded for QC samples

If, during system and performance audits, deficiencies or problems are discovered, corrective action will be initiated immediately. The appropriate field and laboratory personnel will be notified immediately an investigative process will be implemented immediately to find solutions to these issues. The investigative process will consist, but is not limited to, the following:

- Determining when the problem occurred
- Determining which systems were affected by the problem
- Determining the cause of the problem
- Determining a corrective action to eliminate the problem
- Assigning the responsibility for implementing the corrective action
- Implementing the corrective action
- Evaluating the effectiveness of the corrective action

- Investigating alternative corrective actions if the original action was not sufficient in eliminating the problem
- Documenting that the corrective action has eliminated the problem

The Project QC Officer has the authority to require that all site activities threatened by the problem be stopped or limited until the corrective action has been implemented and satisfactorily verified to eliminate the problem.

Corrective actions may include, but is not limited to:

- Modifications to procedures
- Recalibration of instruments
- Replacement of solvents, reagents, and/or standards
- Additional training of personnel
- Reassignment of personnel

# 7.1 CORRECTIVE ACTION REPORT

A Corrective Action Report (CAR) is necessary documentation of the investigative process. Depending on the issues, the CAR may be generated by the laboratory or the field personnel. Copies of the CAR will be given to the Project QC Officer and Project Manager, who will distribute it to the client. A copy of the CAR will be placed in the project files for future reference.

The CAR should include, but is not limited to:

- A description of the problem, deficiency, or issue
- Proposed resolutions
- Resulting actions
- Effectiveness of the resolutions
- Personnel responsible for implementation of the corrective actions
- Personnel responsible for monitoring the effectiveness of the actions.

## 7.2 QUALITY ASSURANCE REPORT

The Project Manager, Project QC Officer, and Project Chemist will converse on a regular basis to review possible and potential problem areas and to ensure that all QA/QC procedures are being carried out. It is important that all data abnormalities be investigated to ensure that they are not a result of operator or instrument deviation but are a true reflection of the methodology or task function. The project final report will contain a separate section that covers the data quality and validity. At a minimum, the following information will be included in the report:

- Assessment of measurement data precision, accuracy, and completeness
- System and performance audit results
- Significant QA problems and corrective actions implemented
- Copies of documentation such as memos, reports, etc.

The Project QC Officer will be responsible for preparing this report weekly or daily, as well as monthly written QA reports to OHM QA management. The Regional QA/QC Director will be responsible for reviewing and approving these monthly reports. Verbal reports will be made on a more frequent basis. All reports will be made available to the Project Manager, client, and regulating agencies. If no project audits were performed and no significant QA/QC problems occurred, a letter stating these facts will be submitted to the referenced parties in lieu of a QA Report.