

CONTRACTOR QUALITY ASSURANCE PLAN

Riaz Chaudhary, PE



TABLE OF CONTENTS

Section	Page
1.0 INTRODUCTION.....	1
1.1 ORGANIZATION	1
1.1.1 QA DIRECTOR	2
1.1.2 QA MANAGER	2
1.1.3 CQC OFFICER.....	2
1.1.4 PROJECT MANAGERS.....	3
1.1.5 CQC SPECIALIST	3
1.1.6 TECHNICAL EDITOR.....	4
1.2 ORGANIZATIONAL CHANGES	4
1.3 KEY CQC STAFF QUALIFICATIONS.....	4
1.3.1 QC DIRECTOR – RIAZ CHAUDHARY, PE	4
1.3.2 QA/QC MANAGER – ROD REEVE, PG, REA	5
2.0 QA/QC PROGRAM DESCRIPTION.....	5
2.1 COORDINATION AND PLANNING PHASE	6
2.2 PRE-FIELD ACTIVITY PHASE.....	6
2.3 FIELD ACTIVITY PHASE.....	7
3.0 QUALITY CONTROL PROCEDURES.....	7
3.1 MANAGERIAL QUALITY CONTROL.....	7
3.2 SUPERVISION OF SUBCONTRACTOR’S OPERATIONS.....	8
3.3 INSPECTION ACCEPTANCE PROCEDURES	8
3.4 INSPECTION DISCREPANCY PROCEDURES	8
4.0 REPORTING QUALITY CONTROL.....	9
4.1 FIELDWORK	9
4.2 LABORATORY ANALYSES.....	9
4.3 REPORT REVIEW.....	9
5.0 SUBMITTAL CONTROL.....	10
5.1 PROJECT SUBMITTALS.....	10
5.2 SCHEDULING	11

5.3	SUBMITTAL PROCEDURES	11
6.0	DOCUMENTATION	11
6.1	RECORDS	11
6.2	STORAGE, PRESERVATION, AND SAFEKEEPING	11
6.3	RETRIEVAL	12
6.4	SPECIFIC DOCUMENTATION REQUIREMENTS	12
6.5	DOCUMENT CHANGES	12
6.6	SUBMITTALS	12
7.0	DATA ACQUISITION	13

APPENDICES

- A LETTER OF ASSIGNMENT
- B CHEMICAL DATA QUALITY MANAGEMENT

1.0 INTRODUCTION

This quality assurance/quality control (QA/QC) plan is intended to provide MARRS Service, Inc. (MARRS) and our clients with a well-founded quality baseline from which to execute the project work. The QA/QC Is prepared to meet the contract requirements and other regulatory requirements.

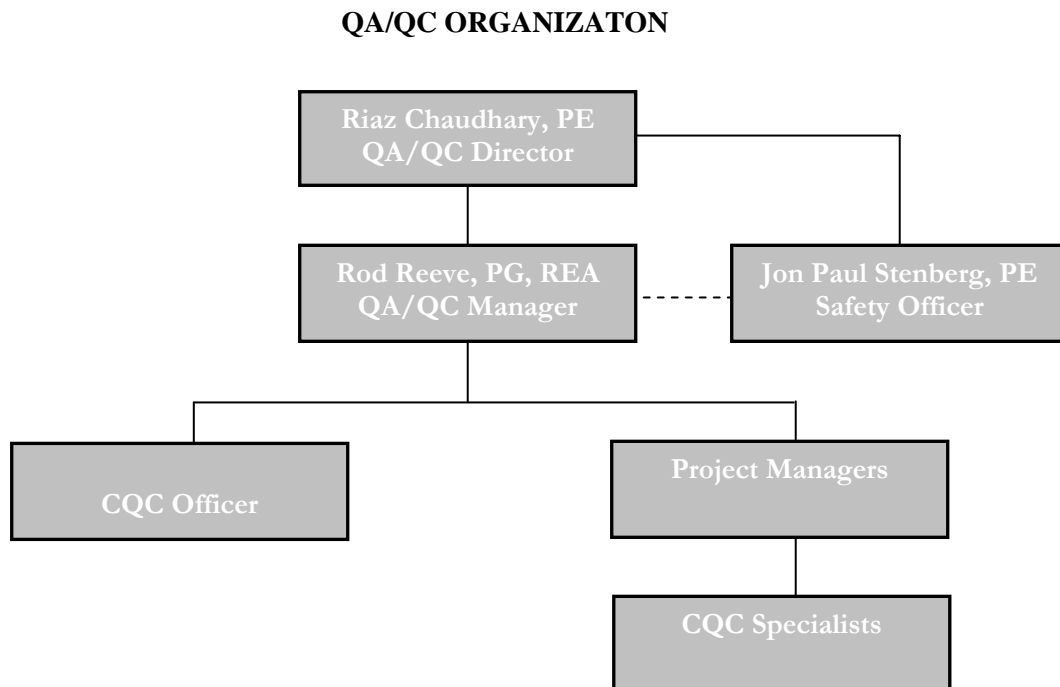
Quality Assurance (QA) and Quality Control (QC) are not synonymous. As a matter of definition:

Quality Assurance – is a program-wide approach that establishes and oversees project-related policies, procedures, standards, guidelines, and training aimed at producing an acceptable level of quality.

Quality Control – is the project specific activities that apply the QA policies, procedures, standards, guidelines, and training to maintain an acceptable level of quality through the application of sound project management, guidance, and review.

1.1 ORGANIZATION

The QA/QC organization is illustrated below. The functional organization and direct-line responsibility, accountability, and communication links among the various functional entities are outlined. Responsibilities of key project QC personnel are also described in the following subsections.



1.1.1 QA DIRECTOR

Mr. Riaz Chaudhary is the designated QC Director. His responsibilities include the following:

- Oversight of QA/QC management staff.
- Adjudication of conflicts, including conflicts regarding implementation of quality program or resolution of quality problems.
- Distribution of all plans, including this QA/QC plan, to all MARRS managers.

1.1.2 QA MANAGER

Mr. Rod Reeve, is the QA/QC Manager. His responsibilities will include the following:

- Ensure that the project specific plans are consistent with this QA/QC plan.
- Provide communication between the clients and MARRS personnel regarding QA/QC comments or decisions.
- Assist the QA Director in maintaining this QA/QC plan to maintain conformance with the best management practices and regulatory requirements.
- Assist the QA Director with distributing all approved plans, including this QA/QC plan.
- Oversee implementation of the project specific QC plans.

1.1.3 CQC OFFICER

MARRS assigns Contract Quality Control (CQC) Officer for each individual contract. The CQC is responsible for overall management of QC for each specific contract. The CQC or his/her designated representative is onsite during the fieldwork from the beginning to the end of the work shifts as appropriate per the project specific QA/QC plan. The CQC personally oversees the QC functions of work performed by MARRS. The CQC Officer's responsibilities include the following:

- Ensuring that a project/task specific QA/QC is prepared and implemented;
- Conducting regular reviews and reporting to the QA/QC Manager regarding the status and adequacy of the QC program.
- Appointing a CQC Officer designee when the CQC is not onsite during the fieldwork.
- Reviewing daily reports, test reports, photographs, and relevant documents written or submitted by field personnel.
- Providing technical peer review for documents prepared by MARRS.

The CQC Officer has direct access to the appropriate level of personnel to ensure resolution of any issues that may need to be addressed.

The CQC Officer is the primary spokesperson on matters relating to QA/QC issues relating to the assigned contact, and is also responsible for verifying that activities affecting quality have been correctly performed. To perform this responsibility, the CQC Officer is given authority, access to work areas, and organizational freedom to:

- Identify QA/QC problems and recommend solutions through designated channels;
- Identify the need for corrective actions and verify implementation and effectiveness of solutions and corrective actions;
- Ensure that further processing, delivery, installation, and/or use of items or services are controlled until a nonconformance, deficiency, or unsatisfactory condition has been corrected;
- Stop work when unsatisfactory conditions or further processing could impact project quality;
- Certify that all submittals are in compliance with contract requirements; and
- Assure that all certifications provided by others (e.g., equipment and material vendors or suppliers) are accurate and in compliance with contract requirements.

Whenever the complexity of the work warrants specialized personnel to provide assistance for QC functions, such specialized personnel are responsible for ensuring that the activity complies with the contract requirements for their area of specialization and report to the CQC Officer. These specialized personnel shall be physically present at the site during work in their areas of responsibility and have the necessary education and/or expertise to ensure contract and regulatory compliance.

The need for such specialized personnel will be determined on a contract by contract basis.

1.1.4 PROJECT MANAGERS

MARRS' project managers and the designated lead technical staff shall act as team leaders and shall oversee the work on an on-going basis. A team leader shall be designated for each task prior to the start of work. Each team leader shall report directly to the Project Manager. Any quality issues shall be reported to the CQC Officer. In addition, the fieldwork shall be supervised and reviewed by the team leader in conjunction with the Project Manager, CQC Officer, or CQC Specialist.

1.1.5 CQC SPECIALIST

MARRS will assign a CQC Specialist on a project by project basis. The designated CQC Specialist will have the written delegated authority sufficient to stop work not in compliance with the project QA/QC plan, is responsible for overall management of CQC, and has the authority to act in all CQC matters for the project. CQC Specialist shall be onsite at all times during field activities as appropriate per project QA/QC. The CQC Specialist's duties shall include the following:

- Reporting to the CQC Officer and ensuring that the project QA/QC plan is being implemented;
- Scheduling, reviewing, certifying, and managing submittals, including those of subcontractors, offsite fabricators, suppliers, and purchasing agents;

- Providing control, verification, and acceptance of testing procedures for tests that may be required;
- Following CQC reporting procedures, including reporting formats;
- Tracking construction deficiencies from identification through acceptable corrective action, including verification that identified deficiencies have been corrected; and
- Managing a scheduling system that shall include, as specific and separate activities, all coordination and planning phase meetings (inspections); all operation and maintenance (O&M) manuals; and all test plans of electrical and mechanical equipment or systems that require validation testing or instructions to Government representatives.

MARRS' commitment to the CQC system and the authority given to the CQC Specialist will be summarized in a Letter of Assignment for the project. A sample letter is included in Appendix A.

1.1.6 TECHNICAL EDITOR

MARRS normally assigns a technical editor as appropriate on a project by project basis. The technical editor reviews the submitted reports for language, writing style, consistency (terminology and format), mechanics, punctuation, and grammar.

1.2 ORGANIZATIONAL CHANGES

Changes to the project specific CQC staff will require the clients acceptance prior to replacement of any member. Requests for organizational changes shall include the name, qualifications, duties, and responsibilities of each proposed replacement. The Project Manager shall facilitate changes to CQC staff and notification of acceptance by the client.

1.3 KEY CQC STAFF QUALIFICATIONS

Their professional summaries of the QA/QC Director and QA/QC Manager are presented below.

1.3.1 QC DIRECTOR – RIAZ CHAUDHARY, PE

Mr. Chaudhary has master degrees in both water resources and sanitary engineering. Mr. Chaudhary is a licensed civil engineer in the State of California, and is certified as a “Qualified Environmental Professional”, a certification requiring expertise in multimedia pollution prevention issues and technologies.

Mr. Chaudhary has a broad educational background and experience in project management, water and wastewater systems compliance. For eight (8) years, he provided program management services for the City of Los Angeles Wastewater Program consisting of 100 projects including extensive source control and pre-treatment program to bring the treatment plants in compliance with the NPDES permit requirements.

Mr. Chaudhary has more than 32 years experience in developing and implementing major civil infrastructure and environmental projects. More than 18 years of this experience has been in responsible project development and implementation in southern California involving design management, constructability reviews, bid documents preparation, construction contract administration, analysis of

claims related to project schedule delays and related cost impacts, excessive change orders and their cost impact and cost impact due to disruption. Mr. Chaudhary's assignments have included projects for several major public agencies in Southern California including California Department of Transportation.

More recently, Mr. Chaudhary has served as program manager for MARRS' four competitively won IDIQ contract with NAVFAC SW involving water, wastewater and environmental compliance work. Under these contracts, he has provided oversight for completion of several water and wastewater projects at MCBCP.

1.3.2 QA/QC MANAGER – ROD REEVE, PG, REA

Mr. Reeve has over 23 years of operational and management experience involving hazardous materials characterization, mitigation and removal actions on behalf of the US Navy, US Marine Corps, municipalities, and private enterprises. He has worked extensively with regulatory personnel in Washington, California, Arizona, and Nevada regarding site assessment, characterization, and delineation and permitting activities. He has gained regulatory closure on many sites and has been able to reduce on-going monitoring requirements through negotiations with the lead regulators.

Recently, Mr. Reeve had been closely involved ensuring QA/QC protocols comply with NAVFAC Southwest's QA/QC Manager, Mr. Nars Ancog. He is a Registered Geologist in both California and Idaho and for the past 11 years provided QA/QC review of various NAVFAC documents. He has been the QA/QC Manager for the NAVFAC SW IDIQ Stormwater, Groundwater, Potable Water and Wastewater contract for the past five years. He also served as a QA/QC Manager for NAVFAC SW for CERCLA, RCRA and NPDES projects and has been involved with various FEAD/ROICC representatives at five different installations under the NAVFAC SW area of responsibility.

2.0 QA/QC PROGRAM DESCRIPTION

This section describes the baseline requirements of MARRS QA/QC program that should be incorporated in all project QA/QC plans. Detailed specifics for implementation are described throughout this plan.

The requirements contained in this plan are applicable to work conducted by MARRS, and its subcontractors to control both onsite and offsite work for this project. MARRS will be responsible for all activities necessary to manage, control, and document work to ensure compliance with contract plans and specifications.

This project QA/QC plan shall be used in conjunction with the work plan, the health and safety plan, and/or the sampling and analysis plans developed for field activities.

A three-phase control system has been developed to meet the CQC objectives:

- Coordination and planning phase,
- Pre-field activity phase, and
- Field activity phase.

These phases are described in the following subsections.

2.1 COORDINATION AND PLANNING PHASE

A coordination, planning, and mutual understanding meeting with the client shall be held to discuss the CQC system. The appropriate key MARRS CQC personnel (QA/QC Manager, Project Manager, CQC Officer, and/or CQC Specialist) shall attend. The meeting shall be held prior to the start of the fieldwork. Typically, the following topics are discussed as part of the coordination and planning phase meeting:

- Establishment of contract CQC requirements and mutual understanding of QC system details.
- Assignment of authority and responsibility for tasks.
- Confirmation of the respective points of contact for the client and MARRS who have the authority to correct any problems or make decisions on the project.
- Verification of data quality objectives (DQO) as presented in the work plans or sampling plans.
- Identification of testing procedures, samples required, analytical methods, and minimum number of QA tests.
- Establishment of corrective action procedures.
- Establishment of reporting standards, formats, and details including forms for recording CQC operations and control activities.
- Establishment of review and approval levels for submittals.
- Ensuring that MARRS personnel (including MARRS subcontractors) have a clear understanding of the projects QA/QC requirements.

2.2 PRE-FIELD ACTIVITY PHASE

Types of pre-field actions may include preparation of various plans, a site visit to observe materials and equipment for contract compliance, and/or approval of submittals regarding shop drawings, test reports, and plans. MARRS and the client representatives should conduct advance surveillance to make sure that pre-field contract requirements have been satisfied before beginning the fieldwork.

During this phase, the following activities, while not all inclusive, generally should be considered:

- Reviewing with assigned staff the contract plans and specifications for the particular fieldwork.
- Ensuring that all staff members have been provided with the most up-to-date plans and specifications, and that they fully understand the requirements.
- Ensuring that submittal requirements for the fieldwork have been fully understood.
- Addressing specific safety matters that relate to the work to be performed.
- Determining that staff members are adequately prepared for required fieldwork.

- Ascertaining whether an independent testing laboratory, subcontractor, and/or consultant will be used for the task.

2.3 FIELD ACTIVITY PHASE

All field activities shall be documented daily on MARRS CQC Report (Appendix B). Typically, the following topics are discussed as part of the preparatory phase of the field activity meeting:

- Ensuring that all staff and subcontractors understand the objective(s) of the work, their roles, and their responsibilities.
- Performance of a quality check on preliminary/previous work, including a double check of work done in earlier phases to ensure that MARRS is in compliance with the contract.
- Prompt identification of any contract errors or construction deficiencies to the Project Director and the client.
- Verification of corrections to any construction deficiencies identified by previous QC inspections.
- Prompt identification of any delay(s) or site condition(s) that may require time extensions from the Project Manager and the the client.
- Conduct of a daily tailgate meeting on health and safety issues. Safety matters relative to the specific fieldwork to be performed shall be addressed in the site-specific health and safety plan (HSP). All MARRS personnel and subcontractors are required to review, understand, and abide by the HSP.
- Continuous checking required throughout the entire fieldwork to ensure contract compliance.

3.0 QUALITY CONTROL PROCEDURES

3.1 MANAGERIAL QUALITY CONTROL

Prior to beginning work on a project, MARRS will conduct an internal managerial quality control meeting with all team members assigned to the project, including subcontractors as appropriate. The purpose of this meeting is to inform all team members of the project objective(s), cost, personnel and time constraints, and potential jurisdictional, social, and environmental impacts.

Internal managerial quality control consists of the following QC procedures:

- Review contract with client, team members, and subcontractors, as appropriate, to ensure that the full nature and scope of work for the project are clearly defined and understood by all parties.
- Analyze individual members' responsibilities and assign tasks to personnel as deemed appropriate.
- Develop preliminary design criteria based on the work to be performed and clarify other issues with the client and team members as appropriate.

- Confirm design criteria and standards to be used.
- Establish project schedule to complete project on time and under budget.

3.2 SUPERVISION OF SUBCONTRACTOR'S OPERATIONS

Supervision of subcontractors' operations is the responsibility of the project manager and CQC Specialist. Major discrepancies that come to their attention shall be recorded and transmitted to the appropriate subcontractor. The CQC Specialist has authority to act directly with subcontractor representatives on routine QC activities. If the discrepancy is related to concrete placement or will be covered by a succeeding operation, a resolution shall be made prior to the item being covered. Major discrepancies shall be followed up on a daily basis. Upon correction of the major discrepancy, the date corrected shall be recorded.

3.3 INSPECTION ACCEPTANCE PROCEDURES

All construction work shall be performed in accordance with the contract drawings and specifications. All rework or changes that revise existing engineering drawings or specifications must be authorized. All construction work shall be recorded on the CQC Specialist's daily report. Work found to be in compliance with the drawings and specifications shall be so noted. If discrepancies are found, they shall be handled in accordance with inspection discrepancy procedures.

3.4 INSPECTION DISCREPANCY PROCEDURES

The following procedures are designed to be an inspection system whereby all discrepancies in quality, workmanship, materials, equipment, supplies, and/or unauthorized deviations can be called to the attention of responsible supervision personnel:

- Discrepancies shall be recorded on the QC Daily Report form. Each discrepancy shall be assigned a number by the CQC Specialist. The CQC Specialist shall prepare a concise statement identifying the discrepancy and its location.
- When material, equipment, supplies, or workmanship that does not conform to the contract drawing or specifications is rejected, the CQC Specialist shall initiate a discrepancy report and immediately furnish copies to the Project Manager and the subcontractor's representative.
- Upon reviewing the discrepancy report, the Project Manager and the subcontractor's representative, if required, shall examine the rejected items or work. If, in their opinion, any of the rejected items or work can be reworked to a usable condition, the discrepancy report shall be so noted. However, if in their opinion, the item cannot be reworked from either a practical and/or economic standpoint, the item shall be scrapped and an entry made on the discrepancy reporting noting the decision to scrap that item.
- Upon completion of rework on items specified for rework, the CQC Specialist shall be notified, and he shall re-inspect the item(s) to the original requirement plus the rework information on the discrepancy report. If the rework is found acceptable, the discrepancy report shall be so noted. From that time on, the item(s) shall be handled in a normal manner. If, however, the item(s) is still not acceptable due to poor workmanship or other problems arising from the rework, the

item(s) shall be treated as a first-time rejection and shall be resubmitted for inspection only after further rework.

- The discrepancy report log shall be periodically reviewed by the Project Manager and the CQC Specialist to formulate the disposition for each uncorrected discrepancy. The Project Manager and the CQC Specialist shall establish timetables for final resolution of all discrepancies.

4.0 REPORTING QUALITY CONTROL

The reporting QC procedures consist of three categories: fieldwork, laboratory analyses, and report preparation.

4.1 FIELDWORK

MARRS uses standardized forms for typical survey services to minimize field input errors. Fieldwork shall be routinely checked by the CQC Specialist or his alternate for potential error(s) in sampling procedures. Samples collected shall be placed into containers appropriate for the respective analyses to be performed. The samples shall be labeled and stored using appropriate preservation methods pending delivery to the laboratory for analysis. Chain-of-custody procedures, including use of chain-of-custody forms, shall be used to document sample handling and transport from the time of sample collection to delivery to the laboratory.

Detailed field procedures will be addressed in the work plan for each task.

4.2 LABORATORY ANALYSES

If the contract stipulates that MARRS shall have any collected samples analyzed, then those samples shall be analyzed by laboratories certified by the United States Environmental Protection Agency (USEPA), the American Industrial Hygiene Association (AIHA), and/or the the contract requirements, as appropriate.

Actual laboratory analytical procedures should be addressed in the sampling and analysis plan.

4.3 REPORT REVIEW

MARRS has designed a procedure to minimize input errors. Once the data are input, the person entering the data shall double-check the results with laboratory reports. Then, another staff member shall recheck the results and locations for accuracy.

Reports receive several reviews prior to finalization. The review/submittal sequence is as follows:

- Author reviews report.
- CQC Specialist reviews report.
- Project Manager reviews report.
- Outside, independent technical editor reviews report.

- Report is prepared and submitted to the client.
- Client provides comments.
- Client's comments are incorporated or addressed.
- Final report is prepared and submitted.

Based on the above procedures, MARRS is confident that the finished product will adhere to the client's requirements.

5.0 SUBMITTAL CONTROL

This section describes the control of submittals developed by MARRS. All submittals referred to in this section may not apply to all projects. This section outlines the procedures for scheduling, reviewing, certifying, and managing all submittals. The CQC Specialist shall be responsible for maintaining and updating the submittal logs.

5.1 PROJECT SUBMITTALS

Documents and other items shall be submitted to the client as required and include, but are not limited to, the following:

- Data – Submittals that provide calculations, descriptions, or documentation regarding the work.
- Schedules – Tabular lists showing timetables, locations, features, or other pertinent information regarding products, materials, equipment, or components to be used in the work.
- Instructions – Preprinted material describing installation of a product, system or material, including special notices and material safety data sheets (MSDS), if any, concerning impedances, hazards, and safety precautions.
- Drawings – Submittals that graphically show the relationship of various components of the work, schematic diagrams of systems, details of fabrications, layouts of particular elements, connections, and other relational aspects of the work.
- Statements – Documents—required of the Contractor, or through the Contractor, from a supplier, installer, manufacturer, or other lower-tier Contractor—the purpose of which is to confirm the quality or orderly progression of a portion of the work by documenting procedures, acceptability of methods or personnel, qualifications, or other verifications of quality.
- Certificates – Statements signed by an official authorized to certify on behalf of the manufacturer of a product, system or material, attesting that the product, system or material meets specified requirements.
- Reports – Reports of inspections or tests, including analysis and interpretation of test results. Each report shall be properly identified. Test methods used shall be identified, and test results shall be recorded.

- Samples – Samples, including both fabricated and infabricated physical examples of materials, products, and units of work as complete units or as portions of units of work.
- Records – Documentation to record compliance with technical or administrative requirements.
- Operation and Maintenance (O&M) Manuals – Excerpts from pertinent O&M manuals.

5.2 SCHEDULING

Submittals covering component items forming a system or items that are interrelated shall be scheduled to be coordinated and submitted concurrently. Certifications to be submitted with the pertinent drawings shall also be scheduled. Adequate time (per contract requirements exclusive of mailing time) shall be allowed and shown on the submittal register for review and approval.

5.3 SUBMITTAL PROCEDURES

All contract deliverables shall be provided on a CD-ROM along with paper copies. This includes report drafts and incorporated review comments. At the conclusion of the project, the contractor shall record all of the documents, organized by submittal name, on a CD-ROM. All files shall be IBM PC compatible, submitted in MS Word 2000, MS Excel 2000, MS Project 98, or Adobe 5.0. CD-ROMs shall be labeled with a project tracking number, the site name, and project name if different from the site name.

6.0 DOCUMENTATION

This section describes the requirements for the control, identification, preparation, and maintenance of documentation that furnishes documentary evidence of quality. Detailed specifics for implementation and appropriate forms shall be documented in implementing procedures, instructions, or plans. The CQC Specialist shall be responsible for maintaining and submitting records for this project.

6.1 RECORDS

Records shall be legible, identifiable, retrievable, and protected against damage, deterioration, or loss. Records shall be indexed in accordance with applicable client requirements for the project. Records and/or indexing system(s) shall provide sufficient information to permit identification between the record and the item(s) or activity(ies) to which it applies.

6.2 STORAGE, PRESERVATION, AND SAFEKEEPING

MARRS' original file records shall be stored for no less than 3 years or per contract requirement after completion of this project. The records will be available at MARRS' office. In order to preclude deterioration of the records, the following procedures shall be used:

- Records shall be kept in a dry, secured storage area to prevent damage from moisture, heat, and pressure.
- Records shall be firmly attached in binders or placed in folders or envelopes for storage in steel file cabinets or on shelving in containers.

- Records that can be damaged from excessive light, electromagnetic fields, and temperature shall be stored away from these environmental elements. Also, records shall not be stacked if doing so will damage them in any way.

Security shall be established to preclude the entry of unauthorized personnel into the storage area. This security will guard against larceny and vandalism. Measures will be taken to provide for prompt replacement, restoration, or substitution of lost or damaged records.

The client shall be responsible for the storage and preservation and safekeeping of its own records upon acceptance of final reports and plans.

6.3 RETRIEVAL

After the project conclusion and within a 5-year period, the records can be retrieved from MARRS' office upon written request from the client. A nominal fee may be charged for record retrieval and/or duplication. Records will be made accessible to the client at MARRS' office.

6.4 SPECIFIC DOCUMENTATION REQUIREMENTS

Specific documentation requirements will be different for each project. The following is a list of the documents typically required.

- Site-Specific Sampling and Analysis Plan
- Site-specific Health and Safety Plan
- Site-specific Spill and Discharge Plan
- Site-specific Materials Handling Plan
- Site-specific Contractor Quality Control Plan
- Completion Report

6.5 DOCUMENT CHANGES

Changes to documents shall be reviewed and approved by the same organizations that perform the original review and approval unless other organizations are specifically designated for such review and approval. The reviewing organizations shall have access to pertinent background data or information upon which to base their approval.

Approved document changes shall be promptly incorporated where applicable. Obsolete or superseded documents shall be controlled to prevent their inadvertent use. The current revision status of documents shall be identified and maintained.

6.6 SUBMITTALS

Submittals shall be made in accordance with Section 5.0 of this plan.

7.0 DATA ACQUISITION

The acquisition of reliable data is of critical importance throughout the project, including the assessment, design, remedial action, O&M, and project closeout stages. Data include those acquired both in the field and in the laboratory. QC activities are tailored to specific activities and recognize the relationship between activities.

The project-specific work plan (and/or sampling and analysis plan) should identify the specific procedures to be used in the data acquisition process. Appendix B includes MARRS Chemical Data Quality Management Plan.

APPENDIX A

LETTER OF ASSIGNMENT

LETTER OF ASSIGNMENT

Subject: Appointment of Contract Quality Control (CQC) Officer

Our contract requires us to deliver a finished product that conforms to the contract specifications and good engineering practices. We have established this CQC plan to assure conformance to the contract specifications various workplans prepared by MARRS. Accordingly, you have been appointed as MARRS' CQC Officer with the authority and responsibility to accept or reject work or direct removal or replacement of any defective work performed under this contract in accordance with the approved quality standards, the contract specifications, workplans and good engineering practices. You are directed to comply with the CQC plan and various workplans prepared under this contract and authorized to stop work which is not in compliance with the contract. You are further directed to take actions and perform such duties as necessary to ensure that all work complies with the document and contract specifications, and to resolve quality matters as required.

QA/QC Manager

Date

APPENDIX B

CHEMICAL DATA QUALITY MANAGEMENT PLAN

Chemical Data Quality Management Plan

Prepared by:

MARRS Services, Inc.
101 State Place, Suite O
Escondido, California 92029



Chemical Data Quality Management Plan

Prepared For:



Prepared by:

MARRS Services, Inc.
101 State Place, Suite O
Escondido, California 92029



TABLE OF CONTENTS

1.0	PROGRAM MANAGEMENT	1
1.1	PROGRAM AND PROJECT ORGANIZATION	1
1.1.1	Program Manager	1
1.1.2	Quality Assurance Manager	1
1.1.3	Project Manager.....	2
1.1.4	Project Chemist.....	2
1.1.5	Health and Safety Officer	2
1.1.6	Sampling Team Leader.....	3
1.1.7	Field Personnel	3
1.2	PROJECT DESCRIPTION.....	3
1.2.1	Location and History	3
1.2.2	Geology	4
1.2.3	Hydrogeology	4
1.2.4	Project Specific Information.....	4
1.2.5	Project Description	4
1.2.5.1	Site and Project Background.....	4
1.3	DATA QUALITY OBJECTIVES	4
1.3.1	Data Categories.....	6
1.3.1.1	Screening Data	6
1.3.1.2	Screening Data with Definitive Confirmation	7
1.3.1.3	Definitive Data.....	7
1.4	DOCUMENTATION AND RECORDS.....	8
1.4.1	Field Documentation	8
1.4.1.1	Field Logbooks	8
1.4.1.2	Photographs.....	9
1.4.1.3	Chain-of-Custody Records.....	9
1.4.1.4	Sample Identification	10
1.4.2	Laboratory Documentation and Records	10
1.4.2.1	Sample Receipt and Laboratory Custody.....	11
1.4.2.2	Data Reporting/Comprehensive Certificate of Analysis.....	11
1.4.2.3	Raw Data Packages.....	13
1.4.2.4	Electronic Data Deliverables	17
1.4.3	Calculations	18
1.4.4	Data Integrity and Outliers	18
1.4.5	Data Management.....	18
1.4.6	Data Archive.....	19
2.0	MEASUREMENTS AND DATA ACQUISITION.....	21
2.1	SAMPLING PROCESS DESIGN	21
2.2	SAMPLING METHODS REQUIREMENTS	21
2.3	SAMPLING HANDLING PROCEDURES	21
2.3.1	Packing	22
2.3.2	Shipping.....	22
2.3.3	Sample Preservation and Holding Times.....	22
2.3.4	Laboratory Receipt and Entry of Samples.....	23
2.3.5	Pre-Analysis Storage	24
2.3.6	Post Analysis Storage	24
2.4	ANALYTICAL METHODS REQUIREMENTS.....	24
2.4.1	Overview of Analytical Methods.....	24
2.4.1.1	Organic Analysis.....	24

MARRS Chemical Data Quality Management Plan

2.4.1.2	Metals Analysis.....	26
2.4.2	Method Descriptions.....	27
2.4.2.1	Organics.....	28
2.4.2.2	Inorganic	34
2.4.3	Preventive Maintenance Program.....	39
2.4.4	Laboratory Data Reduction and Review.....	40
2.5	QUALITY CONTROL REQUIREMENTS	43
2.5.1	Analytical Quality Control Requirements	43
2.5.2	Definitions of Terms.....	43
2.5.3	Laboratory Batch Quality Control Logic.....	47
2.5.4	Laboratory Data Completeness.....	50
2.6	INSTRUMENT CALIBRATION AND FREQUENCY	51
2.6.1	Calibration Standards.....	51
2.6.2	Calibration	52
2.6.2.1	Organic Methods Calibration.....	52
2.6.2.2	Metals Methods Calibration.....	55
2.6.2.3	Wet Chemistry and Other Methods Calibrations	56
2.6.2.4	Analytical Calibrations and Results Calculations	56
2.7	DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS) ..	60
3.0	ASSESSMENT OVERSIGHT	63
3.1	QUALITY CONTROL	63
3.1.1	Definable Features of Work.....	63
3.1.2	Preparatory Phase	63
3.1.3	Initial Phase	64
3.1.4	Follow-up Phase	64
3.1.5	Completion Inspection.....	64
3.2	ASSESSMENT AND RESPONSE ACTION	64
3.2.1	Readiness Review.....	65
3.2.2	System Audit	65
3.2.3	Surveillance	66
3.2.4	Performance Evaluation Samples/Data Tracking Audits	66
3.2.5	The Nonconformance/Corrective Actions	66
3.3	REPORTS TO MANAGEMENT	66
3.3.1	Field Activities	67
3.3.2	Drilling Subcontractors.....	67
3.3.3	Subcontract Laboratory	67
4.0	DATA VALIDATION AND USABILITY	69
4.1	DATA REVIEW, VALIDATION AND VERIFICATION.....	69
4.1.1	Field Sampling/Non-analytical Data	69
4.1.2	Screening/Non-definitive Data	69
4.1.3	Definitive/Confirmatory Data.....	69
4.2	VALIDATION AND VERIFICATION METHODS	70
4.2.1	Data Verification	70
4.2.2	Data Validation.....	72
4.2.3	Data Validation Summary Report	72
4.2.4	Data Usability	73
4.3	RECONCILIATION WITH DATA QUALITY OBJECTIVES.....	73
4.3.1	Analytical/Statistical Control Parameters	73

APPENDIX A – Sampling and Analysis Plan Outline

APPENDIX B – Standard Operating Procedures

List of Acronyms

<i>AES</i>	<i>Atomic Emission Spectroscopy</i>
<i>BS/BD</i>	<i>Blank Spike/Blank Spike Duplicate</i>
<i>BTEX</i>	<i>Benzene, Toluene, Ethylbenzene, and Xylene</i>
<i>CAR</i>	<i>Corrective Action Requests</i>
<i>CCAL</i>	<i>Continuing Calibration</i>
<i>CCV</i>	<i>Continuing Calibration Verification</i>
<i>CCC</i>	<i>Continuing Calibration Check</i>
<i>CDQAR</i>	<i>Chemical Data quality Assessment Report</i>
<i>CDQMP</i>	<i>Chemical Data Quality Management Plan</i>
<i>COC</i>	<i>Chain of Custody</i>
<i>CRQL</i>	<i>Contract Required Quantitation Limit</i>
<i>DL</i>	<i>Detection Limit</i>
<i>DQO</i>	<i>Data Quality Objective</i>
<i>DRO</i>	<i>Diesel Range Organics</i>
<i>EB</i>	<i>Equipment Blank</i>
<i>ECD</i>	<i>Electron Capture Detector</i>
<i>EDL</i>	<i>Estimated Detection Limit</i>
<i>EPA</i>	<i>Environmental Protection Agency</i>
<i>FAAS</i>	<i>Flame Atomic Absorption Spectroscopy</i>
<i>GC</i>	<i>Gas Chromatograph</i>
<i>GFAAS</i>	<i>Graphite Furnace Atomic Absorption Spectroscopy</i>
<i>GRO</i>	<i>Gasoline Range Organics</i>
<i>H&S</i>	<i>Health and Safety</i>
<i>H&SO</i>	<i>Health & Safety Officer</i>
<i>HECD</i>	<i>Hall Electrolytic Conductivity Detector</i>
<i>HPLC</i>	<i>High Pressure Liquid Chromatograph</i>
<i>HRGC</i>	<i>High Resolution Chromatography</i>
<i>HRMS</i>	<i>High Resolution Mass Spectrometry</i>
<i>ICAL</i>	<i>Initial Calibration</i>
<i>ICO</i>	<i>Inductively Coupled Plasma</i>
<i>ICV</i>	<i>Initial Calibration Verification</i>
<i>ID</i>	<i>Identification</i>
<i>IDL</i>	<i>Instrument Detection Limit</i>
<i>L</i>	<i>Liter(s)</i>
<i>LCS/LCSD</i>	<i>Laboratory Control Sample/Laboratory Control Sample Duplicate</i>
<i>LCL</i>	<i>Lower Calibration Limit</i>
<i>LIMS</i>	<i>Laboratory Information Management System</i>
<i>LPM</i>	<i>Laboratory Project Manager</i>
<i>MB</i>	<i>Method Blank</i>
<i>MDL</i>	<i>Method Detection Limit</i>
<i>µg</i>	<i>Microgram(s)</i>
<i>µl</i>	<i>Microliter(s)</i>
<i>ml</i>	<i>Milliliter(s)</i>
<i>MS</i>	<i>Mass Spectrometer</i>
<i>MSA</i>	<i>Method of Standard Addition</i>
<i>SQL</i>	<i>Method Quantitation Limit</i>
<i>MS/MSD</i>	<i>Matrix Spike/Matrix Spike Duplicate</i>
<i>NCR</i>	<i>Nonconformance Report</i>

MARRS Chemical Data Quality Management Plan

<i>NIST</i>	<i>National Institute of Standard and Technology</i>
<i>OSHA</i>	<i>Occupational Safety and Health Administration</i>
<i>PA</i>	<i>Preliminary Assessment</i>
<i>PARCC</i>	<i>Precision, Accuracy, Representativeness, Completeness, and Comparability</i>
<i>PCBs</i>	<i>Polychlorinated Biphenyls</i>
<i>PCDD</i>	<i>Polychlorinated Dibenzodioxin</i>
<i>PCDF</i>	<i>Polychlorinated Dibenzofuran</i>
<i>PE</i>	<i>Performance Evaluation</i>
<i>PID</i>	<i>Photoionization Detector</i>
<i>PQL</i>	<i>Practical Quantitation Limit</i>
<i>PM</i>	<i>Project Manager</i>
<i>ppb</i>	<i>Parts per Billion</i>
<i>ppm</i>	<i>Parts per Million</i>
<i>PRP</i>	<i>Principle Responsible Party</i>
<i>QA</i>	<i>Quality Assurance</i>
<i>QA/QCO</i>	<i>QA/QC Officer</i>
<i>QAP</i>	<i>Quality Assurance Plan</i>
<i>QAPP</i>	<i>Quality Assurance Project Plan</i>
<i>QC</i>	<i>Quality Control</i>
<i>RC</i>	<i>Reporting Limit</i>
<i>RF</i>	<i>Response Factor</i>
<i>RRF</i>	<i>Relative Response Factor</i>
<i>RPD</i>	<i>Relative Percent Difference</i>
<i>RT</i>	<i>Retention Time</i>
<i>RRT</i>	<i>Relative Retention Time</i>
<i>SAP</i>	<i>Sampling and Analysis Plan</i>
<i>SDG</i>	<i>Sample Delivery Group</i>
<i>SOP</i>	<i>Standard Operating Procedure</i>
<i>SVOC</i>	<i>Semivolatile Organic Compounds</i>
<i>TDS</i>	<i>Total Dissolved Solids</i>
<i>TPH</i>	<i>Total Petroleum Hydrocarbon</i>
<i>TRPH</i>	<i>Total Recoverable Petroleum Hydrocarbon</i>
<i>TSS</i>	<i>Total Suspended Solids</i>
<i>UCL</i>	<i>Upper Calibration Limit</i>
<i>VOA</i>	<i>Volatile Organic Analysis</i>
<i>VOC</i>	<i>Volatile Organic Compound</i>
<i>WP</i>	<i>Work Plan</i>
<i>°C</i>	<i>Degrees Celsius</i>
<i>%D</i>	<i>Percent Difference</i>

MARRS Chemical Data Quality Management Plan

EXECUTIVE SUMMARY

This Chemical Data Quality Management Plan (CDQMP) sets the procedures that will be used to achieve the Data Quality Objective (DQO) based on quality control parameters that will assure accurate, precise, representative, complete, legally defensible and comparable data. The CDQMP presents functions, procedures and Quality Assurance (QA) and Quality Control (QC) activities to achieve quality goals set in the DQO.

A Quality Assurance Project Plan (QAPP), and Sampling and Analysis Plan (SAP) and Data Quality Objective (DQO) are the three components of CDQMP. This CDQMP is prepared in accordance with the following guidelines and publications:

Environmental Protection Agency (EPA):

- | | |
|--|--|
| EPA 540R-93-071 | Data Quality Objectives for Superfund, Interim Final Guidance (September 1993) |
| EPA SW-846 | Test Methods for Evaluating Solid Wastes, Third Edition (Update III, 1997) |
| EPA QA/R-5 | EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, Interim Draft Final, August 1994 |
| EPA-505-B-04-900C / DoD DTIC ADA 427486: | Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) |

U.S. Army Corps of Engineers (USACE)

- | | |
|-------------------|---|
| <i>EM 200-1-3</i> | <i>Requirements for the Preparation of Sampling and Analysis Plans (September 1994)</i> |
| <i>EM 200-1-1</i> | <i>Requirements for Contract Laboratory Validation (July 1994)</i> |

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1.0 PROGRAM MANAGEMENT

1.1 PROGRAM AND PROJECT ORGANIZATION

This section of the CDQMP details the program and project organization. Program personnel and their respective responsibilities will be clearly defined in the project Work Plan (WP) and associated project documents. Sampling and Analysis Plan (SAP) will clearly identify the project personnel and the specific personnel that will manage or perform specific tasks on each project. The line of authority and communication, and responsibilities of key personnel will be clearly defined on a project specific organizational chart.

1.1.1 Program Manager

The Program Manager will be identified. The Program Manager will be fully responsible and accountable for all program and contractual activities. He will serve as the focal point and main channel of communication between all parties. He will have authority to delegate various program management tasks to various Program staff. Using the program team, he will coordinate all program activities which will include, but not limited to, establishment and interpretation of program policies, prepare long-range program plans, monitor schedules and budget costs, coordinate all reporting and data base activities, ensure that necessary resources are made available to the project team, provide for safe environment for the Project Team to perform, and to ensure that the Data Quality Objectives DQO for the project are met.

The Program Managers additional duties, as appropriate, will include:

- Procurement, in coordination with procurement personnel, and supervision of Subcontractors
- Selection of personnel from Program Staff for key positions (e.g. Project Manager, Health & Safety officer, Project Chemist etc) for specific projects
- Approve, and implement, all program planning policies set out in various program documents (e.g. Quality Assurance Project Plan, Sampling and Analysis Plan etc)
- Ensure compliance with local, state, and federal laws and regulations
- Approve any changes to the project plan
- Provide technical guidance to ensure performance quality and consistency
- Track the program and expenditure to ensure it is on schedule
- Attend regular meetings with clients
- Review program audit reports and approve/monitor corrective actions
- Conduct regular meetings with sub-contractors for resolution of project related issues

1.1.2 Quality Assurance Manager

The PM will identify the Quality Assurance Officer (QA/QCM). QA/QCM will be responsible for monitoring the Quality Control (QC) operations of the program and report directly to the PM. He will ensure that all field and contract laboratory activities are in compliance with QC protocols as set in the project Quality Assurance Project Plan (QAPP).

MARRS Chemical Data Quality Management Plan

The (QA/QCM) will designate the day to day monitoring of the QC functions to the appropriate program staff member (Sampling Team Leader for all field QC Operations, Project Chemist for all analytical laboratory and data QC).

The (QA/QCM) will perform field and laboratory audits to verify compliance with work plans. As and when deemed necessary, he will review laboratory QC data to monitor laboratory performance. The QA/QCM will review any corrective actions performed, for compliance, to rectify any sampling or analytical problems.

QA/QCM will submit periodic QC reports to Program Manager.

1.1.3 Project Manager

The Project Manager (PM) will be assigned. The PM will be responsible for Quality Assurance (QA) of all field activities. The Project Manager will be present at the project site to monitor initial field activities and to verify that QA protocols are followed. He will hold a preparatory meeting with the field team to discuss the field activities to be performed, and answer any questions from the field team members arising from CDQMP protocols. The Project Manager will ensure that all documents related to QA, QC, Sampling Analysis Plan (SAP), and Health & Safety (H&S) are accessible and available to the field team. Checklists will be used to record the field activities. The Project Manager will review all completed field activity documents for completion. He will ensure that any issues arising from the review are addressed on the same day.

1.1.4 Project Chemist

The Project Chemist will be the primary point of contact, and will have an active role in the management of the project tasks associated with sampling, analysis, laboratory procurement and liaison, and coordination of sampling and analytical activities. The Project Chemist will also be responsible for providing instructions and guidance to field team in sampling and sample preservation requirements, sample container requirements, and provide general oversight of field sampling activities, including on-site analytical/field screening activities.

The Project Chemist will assist the Project Team in selecting an appropriate laboratory for the project, and to develop Project specific QAPP and SAP. He will coordinate sampling activities and laboratory service to ensure the laboratory's preparedness to:

- Implement project specific requirements
- Review analytical data immediately following analysis to ensure compliance to project required QC requirements
- Implementation of corrective actions defined in CDQMP and SAP to non-compliant data uncovered during in-house data review
- To meet the specific holding and turn-around times

The Project Chemist will ensure that a copy of SAP and QAPP are made available to the laboratory to help it implement required protocols to meet the goals set in these documents.

1.1.5 Health and Safety Officer

The Health and Safety Officer (H&SO) will be designated by the Program Manager. The H&SO

MARRS Chemical Data Quality Management Plan

will be a certified Industrial Hygienist. The H&SO will be responsible for the generic H&S plan development, health and safety training of the field personnel, keeping track of each field team member's Occupational Health and Safety Office (OSHA) training and current certification, and to ensure enforcement of health and safety procedures by all field team members. He will report any safety violations to the proper authority (e.g. Program Manager, PM, Sampling Team Leader) and correct such violations. H&SO will ensure that current Health and Safety Plan is available and accessible to the field team during field activities. He will also ensure that an emergency procedure is in place to deal with unexpected incidences of accidents and personnel injuries.

1.1.6 Sampling Team Leader

The Program Manager will delegate the Sampling Team Leader the responsibilities of implementing and overseeing all field activities. The field activities will include review and implementation of project field QA/QC program, data compilation, preparation of field activity documents, and review of field sampling documents prepared by the sampling team members. He will maintain and keep custody of all field activity documents such as log-books, field notes, field photographs, and sign-in books of field personnel and authorized visitors.

The Sampling Team Leader will also be responsible for reviewing and implementing the project field QA/QC program. He will report any deviations/changes that need to be made to the QA/QC program on site to the Program Manager for his approval. The Sampling Team Leader will record such changes in the log book with explanation as why the change in QA/QC program was implemented.

He will be fully conversant with the program H&S policies and implement those to create safe working environment in the field. He will resolve problems encountered during sampling activities. Sampling Team Leader will report to Program Manager, and apprise the Program Manager of any situation that will affect the sampling activities and/or project schedule.

1.1.7 Field Personnel

Field personnel will be identified in the SAP. The field personnel will be responsible for project mobilization, demobilization, sample collection and shipping, and oversight.

Each individual will thoroughly review the QAPP, SAP, field QA/QC program, procedures for completing technical field documentation such as chain of custody forms, field log books, sample identifying system, and sample packing and shipping procedures. Field personnel will review the generic and site specific Health and Safety Plan before the commencement of the field activities.

Field personnel will report to the Sampling Team Leader.

1.2 PROJECT DESCRIPTION

This section will discuss site and project background, problem definition, site history, geology and hydrogeology. The detailed site specific information will be discussed in site and project specific Work Plan, QAPP and SAP.

1.2.1 Location and History

Project and site specific location and history will be discussed under this section.

1.2.2 Geology

Project and site specific geology will be discussed under this section.

1.2.3 Hydrogeology

Project and site specific hydrogeology will be discussed under this section.

1.2.4 Project Specific Information

A detailed definition of the problem, based on the DQO process and the associated background information, will be included in the project-specific SAP, as described below.

This section of the project specific SAP will include a narrative that will describe the specific problems to be solved, and/or decisions to be made.

The narrative will describe the project and specific problems to be solved or decisions to be made. The goal of the project activity will be clearly stated. A description of the project site and relevant documents, such as area and site location maps, specific site map, site history as it relates to the project and any photographs will be included as applicable. The narrative will also include diagrams detailing areas to be sampled as relevant to achieving the project goals. The project site geology and hydrogeology, based on the previous site investigations, will be summarized under narrative. The section will include information on past history of the site, information from previous investigations, regulatory or legal impact to present a clear understanding of the project objectives and goals.

1.2.5 Project Description

The full project description will be included in the project specific SAP. The description will include, at a minimum, the information described below.

1.2.5.1 Site and Project Background

This section will provide site specific project description in response to project problem defined previously. The project sampling strategy, including the project schedule will be discussed in the SAP. In addition, the section will discuss:

- Projected measurements and approaches to achieve the DQO goals
- Applicable regulatory requirements, specifications, or standards to meet technical, regulatory or quality objectives of the Program
- Specific requirements for successful completion of the Program
- Methods to evaluate Program compliance
- Program tracking and monitoring

1.3 Data Quality Objectives

Data Quality Objectives (DQOs) specific to a project will be set out explicitly. The type of project specific data needed to meet project specific objectives will be discussed under DQO. The text will discuss how the data acquired for the project will be used in decision making, and the QA/QC

measures that will be implemented to obtain data of known quality to meet or exceed the project objectives. The development of DQOs will follow the EPA Guidance Document EPA-QA/G-4: Guidance for the Data Quality Objectives Process, Final September 1994. To ensure that DQOs are satisfied, the on-site laboratory, if utilized, will meet the same performance standard as an off-site fixed laboratory. Precision, accuracy, sensitivity, and completeness parameters will be specified to meet the DQOs. The SAP will define these parameters in quantitative terms. The SAP will define different levels of data that will be collected (definitive, quantitative, screening, or qualitative). The DQOs for each project will address the following topics in the specific order in accordance with the EPA Guidance Document.

- **Problem Statement**

Summarize the problem that requires the collection of the environmental data. Identify the resources that are available to resolve the problem. This step of DQO will answer the following:

- Type(s) of contaminant(s) suspected to be present at the project site
- Type(s) of pathways and receptors present at the site
- Disposal sites present
- Type(s) of contaminated media

- **Identification of Decisions**

Identify and define the decision that requires the collection of the environmental data from the project site to resolve the stated problem. Specify the expected uses of the data projected to be acquired. This step of the DQO process should address

- Expected decisions based on the type of data that will be collected' and
- Actions that will be taken based on these decisions

- **Identify Inputs to Support Decisions**

Identify the information that will be needed to support the decisions made. Specify and discuss the inputs that will require environmental measurements.

- List all types of data that will be needed to achieve the objectives. Include already existing data and data that must be collected.
- Identify methods to establish clean-up action level (regulatory, risk based, technological limits, etc).

- **Definition of Study Boundaries**

Define the study boundaries of the media that the data must represent to support the decisions made. Specify the temporal and spatial aspects of the environmental media. The DQO steps will include, but not limited to:

- Defining project site study boundaries
- Defining discreet boundary for specific suspected contamination source
- Sampling logistics
- On-site sampling and investigation constraints

- Actions that will be taken to overcome such constraints
- **Development of Decision Rules**

Define the decision making logical rules that will support the decision making process based on the final, validated analytical data.
- **Defining Limits of Errors**

Define the limits of decision making errors acceptable to the decision makers. These limits will establish performance goals for limiting uncertainty in environmental data.
- **Optimization of Investigation Design for Obtaining Data that will Satisfy DQOs**

Develop a sampling and analysis plan that will generate level and quality of data expected to satisfy project specific DQOs.

Project specific DQOs will be defined as applicable. The DQOs will identify the decisions to be made, and will define how the data will be used, in text supported by tables that will describe:

- Data needed - Measurement parameters, compounds of concern, and sample matrices.
- The action level or regulatory standard upon which the decisions will be based, and the method detection limits and practical quantitation limits for applicable parameters
- The acceptable level of confidence or the level of uncertainty needed for the intended use of the data.

The text will quantitatively describe sensitivity, precision, accuracy, and completeness goals for each measurement parameter, and matrix. Representativeness and comparability will be discussed qualitatively.

1.3.1 Data Categories

One of the following three data categories will be used to meet specific goals defined under site specific DQO:

- Screening data
- Screening data with definitive confirmation
- Definitive data

Specific QA/QC parameters are associated with each data category. In hierarchy, screening data are of the least known quality, while the definitive data is of known and legally defensible quality.

1.3.1.1 Screening Data

Screening data will be generated on site using quick, qualitative or semi-quantitative analytical methods. Screening data generated on site will enable the field investigators to perform rapid investigations to define, for example, hot spots of contamination and quick qualitative identification and semi-quantitative concentrations of the contaminants. Screening data is neither definitive nor legally defensible and are associated with minimal QA/QC elements described below:

- Sample documentation (location, date, time, matrix, batch, project etc)

- Chain-of-Custody (if applicable)
- Sampling design approach (random, systematic, judgmental etc.)
- Calibrations
- Instrument detection limits and supporting documents
- Analytical summary (quantitation and identification)
- Method Detection Limit studies (compare to method specific performance requirements to determine analytical errors)

1.3.1.2 Screening Data with Definitive Confirmation

Screening data with definitive confirmation will be generated by collecting split samples in the field. One split will be analyzed in the field using a rapid screening analytical method. The second split will be sent to a certified fixed laboratory for definitive confirmation of the results obtained in the field screening analysis. Definitive confirmation of the screening data will provide data of known quality, and reduce the level of uncertainty of the screening data. Minimum of 10% of the screening data will be confirmed by using EPA approved analytical method and the associated QA/QC elements as described below:

- At a minimum, three samples with concentration reported above the action level (if any), and three samples reported non-detect below the action level will randomly be selected for the conformational analysis, supported by appropriate elements of definitive QA/QC.

1.3.1.3 Definitive Data

Definitive data will be acquired using an approved analytical method, such as EPA SW-846 Methods. The generated data will be analyte specific, with definite qualitative identification and quantitation. Definitive data will be supported by rigorous QA/QC elements as specified in the method used. The data will be generated either by an on-site laboratory or an off-site laboratory. QA/QC elements to support definitive data are extensive as described below:

- Project information
- Sample location
- Chain of Custody
- Sampling design approach
- Initial and supporting calibrations
- Determination of MDLs and IDLs and supporting documentation
- Determination of project required detection or reporting limits
- Field QC samples
- Laboratory QC samples
- Laboratory QC recoveries
- Analyte identification and quantitation
- Performance Evaluation (PE) sample if applicable

MARRS Chemical Data Quality Management Plan

- Analytical error determination based on duplicate or replicate analysis of QC specified in the QAPP. The QC parameters such as variance, mean, coefficient of variation, precision accuracy will be compared to the method specific performance requirements.
- Total measurement error determination – from sampling through analysis. An appropriate number of co-located samples, as determined by SAP, will be independently collected from the same location and analyzed following standard operating procedures and methods. Based on these results, parameters such as variance mean coefficient of variation and mean will be calculated and compared to established measurement goals.

1.4 Documentation and Records

Information and records for each Project under this CDQMP, discussed below, will be maintained.

1.4.1 Field Documentation

1.4.1.1 Field Logbooks

MARRS SOP No. 03 presents procedures for maintaining Logbooks.

Information on sample identification numbers, chain of custody, and any relevant information on field sampling activities will be recorded in a bound, with serially page numbered field log book. The field log-book will have the following information listed in ink:

- Project name
- Project number
- Site location
- Sampling event
- Name of the Project Manager
- Program Manager's telephone number
- Address of the contractor's office

The field note book will be main source of information on all field activities related to the specific project. As such, it will have sufficient information, including significant observations or problems encountered and corrective action implemented, to allow a field personnel or a Program Manager to reconstruct all events that transpire the sampling activity. The field log-book will be in the custody of the Project Leader. The Project Leader will sign and date the field book prior to the commencement of the field activities, and at the conclusion of the day's activity.

The Project Leader or his designee will maintain the custody of the field log-book. In the event that the book is relinquished to an alternative person, the person relinquishing the book will sign and date the log-book at the time of the transfer. Similarly, the person receiving the book will also sign and date the book therefore accepting the custody of the log-book. Corrections to any errors will be made by crossing out the erroneous data with a straight line and entering the correct information. All corrections will be initialed and dated by the person making the correction. Any unused portions of field log-book pages will be crossed out, signed, and dated at the end of each day. No pages will be removed from the book. All entries in the book will be in ink. Deleting any

MARRS Chemical Data Quality Management Plan

information using such correcting material as white out will not be acceptable.

The date, time, location, sample identification number and personnel who conducted sampling will be recorded in the book, sample container tag and the chain-of-custody (COC). The book will also be used to record the day to day meteorological and other data that may impact the sampling activities, apparent representativeness of the sample, or sample analysis.

Names of the authorized visitors who are not members of the field team will be recorded either in the field book, or separate visitor's book specifically assigned for this purpose. Any visitor on the site will report to the Project Leader.

1.4.1.2 Photographs

If required, the written description of sampling activities will be supported by photographs. All photographs will be fully documented to include project name, project number, date the photograph was taken, reference to the location the photograph was taken, the photographer, and a brief description of the reason the photograph was taken. The photographs will be identified by unique numbering system and traceable to the negatives.

1.4.1.3 Chain-of-Custody Records

MARRS SOP No.02 presents procedures for Sample Handling.

All relevant sampling information for a sample will be transferred from the field log-book to the Chain-Of-Custody (COC) record. This includes the sample matrix and sample identification number. The COC will also record the laboratory analysis to be conducted for each sample.

Custody of the samples will be maintained from the time the sample is collected to the completion of the sample analysis. In the field, each sample will be considered to be in the sampler's custody. The sampler will be personally responsible for the custody of the sample until such time that they are packaged and shipped for delivery to the laboratory.

A sample will be considered to be under a person's custody when:

- The sample is in the person's physical possession
- The sample is in the view of the person after the person has taken the possession of the sample
- The sample is secured in tamper proof environment
- The sample is secured in an area with restricted access by authorized personnel only

COC will accompany all samples delivered to the laboratory. The COC will have the following information recorded:

- Project name
- Sample identification number
- Collection point
- Sampling date
- Time of sample collection (must match with time on the sample label)

- Sample matrix
- Analysis requested for each sample
- Sample preservation method
- Number and type of sample containers used
- Special sample handling or analysis requirements
- Signature of the person who collected the sample
- Signatures of all persons involved in the chain of possession

1.4.1.4 Sample Identification

Each sample will be identified by a unique identification number. The site specific SAP will outline the strategy for specific sample identification procedure for field samples and QC samples. The sample identification number will be recorded in the field book, sample container, and the COC. The recorded identification numbers will be reviewed for accuracy. The sampling personnel will be responsible for recording the sample identification numbers, and will do so immediately after collecting the sample using waterproof ink. Sample containers will not be pre-labeled with identification number. The sample label will be secured on the container using a wide tape so that the label is completely covered by the tape.

The label on each sample container will include the following information:

- Unique sample identification number as described in the SAP
- Project name and sample location
- Date and time of collection
- Analysis requested
- Preservative if any used
- Initials of the person collecting the sample
- Any other information deemed to be pertinent to the sample

1.4.2 Laboratory Documentation and Records

The laboratory will have all SOP formalized in writing and available for all laboratory staff. The contracted laboratory will provide copies of the approved SOP to the primary contractor and the regulating lead agency when requested. The SOPs will be available for:

- Sample receiving, tracking and management
- Approved procedures for sample preparation and extraction
- Approved procedures for sample analysis
- Instrument maintenance
- Result calculations and data reduction
- Database management

MARRS Chemical Data Quality Management Plan

- Health and safety
- QA/QC

The QA/QC officer will be responsible to ensure that all SOPs are current. Any additions, omissions or modifications to the SOPs will be approved and signed by the QA/QC officer.

1.4.2.1 Sample Receipt and Laboratory Custody

All samples will be deemed to be under the custody of the laboratory once the samples are received and COCs are signed off by the laboratory receiving department. The laboratory will be responsible to carefully check that:

- Sample containers are free of leakage or breakage
- Sample identification numbers on sample containers match with COCs
- COCs are accurately completed, and make note of any missing information

The ambient temperature in the cooler and the temperature blank will be measured immediately after the cooler is opened. The temperature will be noted in the cooler receipt form. Any significant out-of-control conditions, such as damaged sample cooler, broken glass sample containers, leaked samples etc will be noted and communicated to the Program Manager immediately. Photographs are recommended to document such incidences.

The pH of the samples for metal analysis will be measured at the time of the sample receipt and recorded in the cooler receipt form. The pH of the VOC samples will be measured at the time of the analysis and recorded in the injection log book.

The laboratory will, within one working day of sample receipt, fax an acknowledgement and cooler receipt form to the Project Chemist at the fax number provided in the site specific SAP.

The laboratory will assign a unique laboratory identification number for the internal tracking of the samples through Laboratory Information Management System (LIMS). The LIMS system will track the sample from the point of receipt to the storage and through each step of laboratory procedure until the sample analysis is complete and the sample is returned to the custody of Sample Control for disposal. The laboratory will have in place measures to restrict the access to the sample/extract/document storage area to authorized personnel only.

The laboratory will have an approved SOP made available to the responsible laboratory personnel. A copy of the SOP will be made available to the primary contractor and the lead regulatory agency when requested.

1.4.2.2 Data Reporting/Comprehensive Certificate of Analysis

The laboratory will be equipped to provide data in various hard copy and electronic formats to meet the needs of different clients. The laboratory will deliver preliminary certificates of analysis within 10 business days from sample receipt. The preliminary report will include summary of sample results, MS/MSD, LCS, method blank and calibration results, COCs, cooler receipts and brief summary of any QC outliers. The laboratory will submit final comprehensive certificates of analysis within 21 days after the receipt of the last sample for the delivery order. Turn around times for certain projects may be different from the times discussed. These times will be project specific and will replace routine turn around times for that project only.

MARRS Chemical Data Quality Management Plan

The comprehensive certificate of analysis will include:

- Original copies of COCs/requests for analysis, cooler receipts, and forms documenting the sample conditions upon arrival at the laboratory.
- Analytical results for each sample and method used. The result will be submitted as detected concentrations or non-detect below PQL, RL, or DL as applicable. For soils, the results will be reported on dry weight basis and percent moisture reported for each sample. All information pertinent to the sample will be recorded and reported, including dilutions and rationale for dilutions, date of extraction and analysis, analytical method used etc. All samples with out-of-control recoveries of spikes will be discussed in the narrative section of the data submitted. Any procedural problems encountered during extraction or analytical steps will be discussed in the narrative, including any corrective actions performed. Samples with out-of-control spike recoveries being attributed to matrix interference will be reported supported by rationale for such conclusion.
- Method blank (MB) results for all matrices and each analytical method. Each field sample and field QC will be associated with a specific MB. Any analyte detected in the MB at concentration above one-half the PQL/RL/DL will be reported for each analyte.
- Surrogate and internal standard spike recoveries, where applicable, with control limits. Out of control recoveries will be flagged and discussed in the narrative. The narrative will discuss the corrective actions implemented to rectify the problem and the outcome of the corrective action applied. If the sample is re-injected or re-extracted as corrective action, both sets of results will be submitted with the data package.
- Matrix Spike/Matrix Spike Duplicate (MS/MSD) results for all analysis, with recoveries, Relative Percent Difference (RPD), and control limits. Sample results will be associated with a specific MS/MSD set. Any outliers will be recorded and discussed in the narrative. If a MS/MSD set is reanalyzed to confirm first set of results or as a corrective action for outliers, and if the results are still out of control, both sets of results will be reported and the data flagged appropriately.
- Laboratory duplicate results with RPD and control limits. The narrative will include discussion on any out of control recoveries and any corrective actions implemented. The out of control results will be flagged.
- Laboratory control samples (LCS) results with control limits. The narrative will include discussion on any out of control recoveries and any corrective actions implemented. The out of control results will be flagged. Samples will be associated with specific LCS analysis.
- Initial and continuing calibration summaries. The reports will flag any deviations from the acceptable QC limits. The calibration summary reports will include the date of the analysis, time of the injection of the standards, the method associated with the calibration, the instrument identification associated with the calibrations, the response factors for initial calibration, and for continuing calibration percent difference of the response factors compared to the initial calibration
- Summary of the mass spectrometer (MS) tuning results for applicable organic methods. The tuning results will be reported as auto calculated by the MS computer system. Manually calibrated tuning performance will not be accepted.
- Injection logs for calibrations and the field and QC samples
- Logs of instrument performance and maintenance

MARRS Chemical Data Quality Management Plan

- Summary results and raw data of all samples with concentrations detected for target compounds indexed by method and sample identification numbers.
- Surrogate and internal standard recoveries for all samples identified for organic analysis, with recovery ranges clearly defined.
- Summary of laboratory and field duplicates with QC acceptance range and RPD. Summary of field duplicates will be prepared by the contractor.
- A table identifying all QA samples and the associated field samples prepared by the contractor.
- A case narrative for each sample delivery group (SDG) that will include discussions of all QC outliers, corrective actions implemented, and samples impacted by the outliers. A detailed discussion will be included for out of control recoveries attributed to matrix effects.
- The comprehensive data package will include the data for analysis covered by the comprehensive certificate of analysis as an appendix to the comprehensive report. The data package will be paginated, with an index or table of contents describing the contents of each appendix. Raw data including all chromatograms, mass spectra, and other measurements will be submitted with the comprehensive data package.

1.4.2.3 Raw Data Packages

The laboratory will deliver raw data package for the project within 21 days after the last sample for the project has been submitted. The package will include a case narrative, COCs, summary of results of environmental samples, summary of QA/QC results, and raw data complete with chromatograms and mass spectra where applicable..

Following sections discuss the requirements for each element of raw data package that will be submitted by the laboratory.

1.4.2.3.1 Case Narrative

The case narrative will be submitted on the laboratory letterhead. The release of certificates of analysis and accompanying data will be authorized by the appropriate laboratory manager or the designee. The case narrative will include:

- List of field sample ID associated with the case narrative, with their corresponding laboratory ID
- Analytical parameters for each sample
- Applicable analytical method for each parameter
- A statement whether holding times were met or exceeded
- Any analytical challenges encountered and corrective actions implemented
- List of out of control QA/QC parameters and supporting discussion of possible reason and factors for noncompliance
- Any observations that may impact the integrity of the sample data quality (such as homogeneity, matrix, presence of excessive vegetative material, pebbles/stones in soil samples etc).

The case narrative will be signed and dated by the laboratory manager or the designee.

1.4.2.3.2 Chain-of-Custody Documentation

Each data package will include a legible copy of field chain-of-custody (COC), laboratory internal COC, and cooler log-in forms for each SDG. All COCs will be reviewed for completion before submission with the data package. Any missing information will be retrieved by the responsible personnel.

1.4.2.3.3 Summary of Analytical Results

Summarized environmental results for each sample will be included in the data package. The summary will include:

- Field sample ID and corresponding laboratory ID
- Sample matrix
- Date the sample was prepared if applicable
- Date and time of analysis
- Identification of the instrument used for analyzing the sample
- Instrument specifications
- Chromatographic column and detector used if applicable
- Weight/volume used for the analysis
- Dilution factor if required
- Percent moisture for dry weight reporting
- Sample PQL/RL/DL as applicable
- Method Detection Limit (MDL)
- Surrogate/internal standard recoveries for organic analysis
- Data qualifiers if used
- Analytical results, with concentrations above quantitation limit highlighted in bold, and in appropriate measurement units

1.4.2.3.4 Summary QA/QC Results

The results for the QA/QC parameters, discussed in the following sections, will be presented in summary form. The mathematical expressions for calculating these parameters will be included in project specific QAPP and SAP.

1.4.2.3.5 Instrument Calibration

The laboratory will report the calibrations for each analyte in the order in which the standards were analyzed. The instrument calibration reports will also include tuning performed for MS.

1.4.2.3.6 Initial Calibration

The laboratory will perform initial calibration of an instrument whenever deemed necessary. Routinely, the initial calibrations are performed when new sets of calibration standards are made, when the chromatographic column is replaced, or when subsequent analysis of continuing calibration or calibration verification analysis fails.

The initial calibration report forms will include all pertinent information required. This will include the date and times individual standards were analyzed, the instrument ID, analytical method used, instrument method used, correlation coefficient (r), calibration factor, relative response factor, percent relative standard deviation (%RSD), retention times for each analyte and the analyst's name.

The records of the calibration standards will be maintained in such a way that it will be possible for anyone to track the source of working calibration standard to its origin.

1.4.2.3.7 Continuing Calibration

The laboratory will analyze continuing calibration with each SDG samples to verify the initial calibration and to monitor daily instrument performance. This calibration primarily applies to GC and GCMS analysis. Continuing calibration will be analyzed at mid-level concentration of the initial calibration and should meet the percent difference (%D) criteria for response factor as defined in the QAPP. The continuing calibration report will include response factor, %D, and retention time for each analyte. The daily continuing calibration will be linked to sample analysis by daily summary and analysis logs.

1.4.2.3.8 Method Blank Analysis

A matrix specific method blank will be analyzed for each SDG. Method blank monitors any laboratory based contamination introduced in the samples during laboratory procedures. All samples from an SDG will be associated to the MB analyzed with the SDG. Any contaminants found in the MB will be discussed in the narrative.

1.4.2.3.9 Interference Check Sample

Interference check sample is analyzed for metals analysis using Methods 6010B and 6020B. This standard is used to measure the interference level introduced by some non-target analytes to the measurement of the target analytes. The laboratory report will include the source of this standard, as well as the percent recovery for each element analyzed, the instrument ID, and the time and date of the analysis.

1.4.2.3.10 Surrogate Standard Recovery

Surrogate standards are spiked into all field and QC samples for GC and GCMS analysis prior to sample extraction to monitor the efficiency of laboratory extraction and analytical procedures. The laboratory will report each surrogate and the concentration used for spiking. Percent recovery of each surrogate for each sample with sample ID will be summarized, together with the QC range.

1.4.2.3.11 Precision and Accuracy

The laboratory will analyze MS/MSD and LCS/LCSD to measure the precision and accuracy. The laboratory will report the sample results, spiked sample results, percent recovery, and RPD with the associated control limits for MS/MSD and LCS/LCSD. These QCs will be associated with corresponding samples identified by their sample IDs.

Post digestion QC will be analyzed for metals analysis. Post digestion spikes, sample results, concentration of spiking solution added, percent recoveries and control limits will be reported. Date and time of analysis for all QCs analyzed will be reported. Summary QC form, listing samples with field ID, corresponding laboratory ID and matrix type will be submitted with the data package.

1.4.2.3.12 Retention Time Windows (GC, GCMS, HPLC)

Chromatographic retention time (RT) windows for each organic analyte will be determined by analyzing Retention Time Window Standard. RT windows for each analyte will be updated daily as per EPA SW-846 requirements. Sample analysis will be stopped if significant shift in the RT window is observed. Analysis will be continued only after appropriate corrective actions are implemented to resolve the problem successfully.

1.4.2.3.13 Compound identification (GC, GCMS, HPLC)

Identification of organic analytes will be based on RT value for GC and HPLC, and mass spectrometric confirmation when applicable. The laboratory will report both absolute RT and Relative Retention Times (RRT) when required.

1.4.2.3.14 Method Detection Limits

The laboratory will establish MDL for each analyte on each instrument annually at a minimum. The most recent MDL for each analyte will be reported for each raw data package.

1.4.2.3.15 Injection Log

The laboratory will maintain injection log for each sample in a project SDG. The injection log will include the time, date, instrument ID, and the analyst's name, and the associated QA/QC samples. The injection log will identify initial calibrations, continuing calibrations and laboratory and field QC samples associated with the project sample and dilutions performed when applicable. Injection log for GCMS analysis will include the time and date of injection of mass spectrometric calibration (MS tuning) compound as applicable. Injection log will also record any analytical problems encountered and corrective action implemented.

1.4.2.3.16 Method of Standard Addition

A summary of Method of Standards Addition (MSA) for metals analysis will be provided. The summary will include the absorbance values, corresponding concentration values, and the final analyte concentrations. Correlations of coefficients will be reported for all analysis. The time and date of MSA analysis will be included in the summary report.

1.4.2.3.17 Inductively Coupled Plasma (ICP) Serial Dilutions

For metals analysis, serial dilution test will be performed per batch to monitor the effects of dilution on final concentrations measured. An analysis of 5 fold dilution will be performed for an analyte with original concentration within the linear range of the instrument and sufficiently high concentration (minimally, a factor if at least 100 times greater than the concentration in the reagent blank). The concentration in the diluted analysis must agree within 10 % of the original concentration.

1.4.2.3.18 ICP Linear Ranges

The linear range for each instrument for each metal will be established. The raw data package will include the date the linear range was established, the integration time, the upper limit of concentration (ULC), and the instrument detection limit (IDL). The report will identify the instrument for which the linear range was established.

1.4.2.3.19 ICP Interelement Correction Factors

The laboratory will report, for each instrument applicable, the wave length used and the date on which correction factors were established. Specific correction factors for Al, Ca, Fe, Mg, and any

other elements and analytes to which they are applied will be detailed.

1.4.2.3.20 Analytical Records

All analysis logs for all instruments used for the project will be included in the data package. The logs will provide the date and time of all calibrations, QA/QC samples, project samples, any corrective actions implemented, and any sample specific problems encountered (such as matrix effect, insufficient sample, bubbles in VOC sample container etc).

1.4.2.3.21 Raw Data

The laboratory will be responsible to organize the raw data systematically on numbered pages. The raw data package will include a table of contents, legible copies of the raw data for the project samples arranged in ascending order of the field ID, corresponding laboratory ID, all calibrations, QA/QC samples, sample preparation logs, instrument logs for each instrument for all days the instruments were used for the project, and the maintenance logs for each instrument. Copies of all measurement printouts will be submitted. The format of the raw data will be client and project specific and will be stated in the QAPP and SAP.

1.4.2.3.22 GC/HPLC Analysis

All GC and HPLC data will be arranged under the appropriate section corresponding to the table of contents. This section will include legible copies of raw data for the project sample analysis, arranged in the ascending order of the field ID, corresponding laboratory ID, all calibrations and QA/QC analysis, sample preparation logs, injection logs. Any mass spectrometric confirmation to support the chromatographic data will be included in this section with the sample's chromatographic data. The chromatographic peaks will be identified as target compound, internal standards and surrogate with their percent recoveries. The quantitative data will have the concentrations reported in project required units and format. The data will also have the per cent recoveries for surrogates and internal standards listed with applicable recovery limits.

1.4.2.3.23 GCMS Analysis

All GCMS data will be arranged under the appropriate section corresponding to the table of contents. This section will include legible copies of raw data for the project sample analysis with instrument ID, arranged in the ascending order of the field ID, corresponding laboratory ID, all calibrations and QA/QC analysis, sample preparation logs, injection logs. The total ion chromatogram peaks will be identified as target compound, internal standards and surrogates with their percent recoveries. The quantitative data will have the concentrations reported in project required units and format. The data will also have the per cent recoveries for surrogates and internal standards listed with applicable recovery limits. All mass spectrometric data will include full total ion chromatogram, single ion monitoring profiles for target compounds, and full spectrum for tentatively identified compounds with library search results.

1.4.2.4 Electronic Data Deliverables

The contracted laboratory will submit quality control and all sample data in electronic format as described in QAPP and SAP.

The electronic data submitted by the laboratory shall be error free and complete agreement with the hard copy data submitted. The electronic data will be submitted by an e-mail, and a compact data disk (CD). The CD will be accompanied by a transmittal letter on company header that the data e-mailed and submitted by CD is in complete agreement with each other and with the hard

copies submitted with the data package. Any errors will be corrected by the laboratory at their own cost.

- Preferably, the analytical data should be transmitted electronically from the instrument software system to the laboratory's information management system (LIMS), which will convert data into electronically deliverable format. Manually entered data, though strongly not recommended, may be required for certain wet chemistry tests where results may not be captured by LIMS. The laboratory will ensure that hand entered wet chemistry data are accurate and agree with hard copy data.

1.4.3 Calculations

Formulae for calculating various analytical parameters such as QC recoveries and concentrations are included in SOPs or standard reporting forms developed by the laboratories. The reporting forms will not include formulae for those parameters that are calculated by computer-based data reduction programs. The laboratory will be responsible to demonstrate the validity of such programs. Such validations will be documented by the laboratory. These programs shall be based on the calculation procedures specified in respective SOPs for GC, GCMS, HPLC, ICP, ICP-MS and other applicable laboratory SOPs and EPA SW-846 Methods.

Some instruments are not interfaced with computers. The results, therefore, can not be downloaded directly to a computer for processing and are recorded in a bound laboratory notebook. In such cases the analyst will manually reduce the results to reportable format and hand enter the data in the computer for final report.

Some wet chemistry analytical results, such as titrations, pH, electrical conductivity and specific gravity do not require calculations and are directly recorded in the laboratory notebook. These data do not need to be transformed in any way and are manually entered in the data base directly from the laboratory notebook.

Hand entering of such data in the computer for the final report and data package assembly is unavoidable. The laboratory will ensure that such data transfers are error free.

1.4.4 Data Integrity and Outliers

The laboratory will ensure that the data integrity is maintained for all analysis. The analyst responsible for sample analysis will check the QC parameters for non-compliance against the established limits of acceptance. The analyst will immediately bring to the attention of the group leader whenever the QC check shows that the analysis is out of control. The group leader will make the decision, with consultation with the laboratory QC manager and the laboratory project manager, whether the analysis can proceed, whether the impacted samples should be reanalyzed or specific corrective action needs to be implemented before the analysis can continue. The analyst will document the out-of control QC analysis and the corrective action taken. The group leader will review the documented entry and file a Nonconformance Report with the Laboratory QA manager.

Potential interferences due to the sample matrix will be identified in the SAP based on the history or the previous analytical records related to the site if available.

1.4.5 Data Management

Data management is a multi-level task, requiring data management at various stages of the

project. Specific details of the personnel responsible for the data management and related data management activities will be set in the program procedures, work plans, and project specific QAPP.

1.4.6 Data Archive

The laboratory will maintain all analytical data records in hard copies and magnetic media for a minimum period of five years following the submission of Certificates of Analysis. The data will be archived in secured storage and will be accessible within 24 hours of making a retrieval request.

The laboratory will have an approved SOP for each laboratory task and activity. Copies of such SOP will be made available to the responsible laboratory personnel, and to the primary contractor and the lead regulatory agency when requested.

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2.0 MEASUREMENTS AND DATA ACQUISITION

This section describes field sampling process and method requirements, analytical methods and quality control requirements, instrument calibrations and data acquisition.

2.1 SAMPLING PROCESS DESIGN

This CDQMP and applicable SOPs set pre-defined guidance in sampling process designs and other related activities. Project specific SAP will define project specific design requirements for sampling process expanded or based on the guidance set in the CDQMP and related SOPs. Additionally, the SAP will provide the project specific sampling design to include the following:

- Sampling network design
- Sample matrices
- Types of QC samples
- Types of field samples
- Sampling frequencies
- Field measurement parameters of interest

The SAP will define the rationale for the sampling design at the project site. Maps, figures or other mode of documents will be used to clearly identify the sampling locations. The SAP, text and tables will describe the field sample matrices and QC samples to be collected. Field measurement parameters of interest will include, but not limited to, hydrogeological, chemical, geological, and geophysical parameters.

2.2 Sampling Methods Requirements

Approved work plans and applicable SOPs will specify qualitative and quantitative measures for specific sample collection method. These procedures will discuss collection errors which may impact the Representativeness of the samples and the goals set in the DQOs. Various methods are available for collecting soil and water samples. Soil sampling methods will include split spoon sampling, hand auger for shallow sampling, EnCore™ sampling, grab sampling and stock pile sampling. Water sampling will include ground water sampling using appropriate water pumps, surface water sampling using e.g. bailers, and drum (waste) sampling using drum sampling pumps. These procedures will be discussed in detail under project specific SAP.

MARRS SOP No. 14 details the type of sample containers, preservatives, and handling requirements for different sample matrices.

Each sample will be uniquely labeled and tracked according to the procedures described in the QAPP. A second label, corresponding to the label on the sample container, will be pasted in field log-book with identical information. The field personnel will designate field QC samples for field duplicates and MS/MSD. The information will be clearly included in appropriate COC. The laboratory and its designated analyst will ultimately be responsible to ensure that field QC samples are analyzed with corresponding SDG.

2.3 Sampling Handling Procedures

Approved work plans and applicable SOPs will specify qualitative and quantitative measures for

MARRS Chemical Data Quality Management Plan

specific sample collection method. These procedures will discuss collection errors which may impact the representativeness of the samples and the goals set in the DQOs.

Samples will be collected in appropriate sample containers, labeled with all pertinent information. Each sample container will be packed securely and placed in coolers filled with ice. Each cooler will be prepared for shipping or transportation by laboratory courier. A copy of COC will be placed in clear waterproof plastic, bag and placed inside the cooler. The laboratory receiving department will be responsible to ensure that the sample IDs on the containers agree with the sample ID numbers in the COC. Any discrepancies will be communicated with the laboratory project manager and the field team leader.

2.3.1 Packing

Packed samples, stored in coolers filled with bagged ice, will be transported as soon as possible after the collection of the last sample of the day or SDG.

2.3.2 Shipping

The sample coolers will be shipped by overnight express carrier for the next day delivery to the laboratory. Alternatively, the sample coolers will be transported by a designated laboratory courier if available. A copy of the COC and a bill of landing will accompany each sample cooler. A copy will be retained by the field team leader. The laboratory project manager will be informed in advance if the sample delivery to the laboratory falls on a weekend. In such instances, the laboratory will ensure that appropriate laboratory personnel are available to receive and process the samples upon delivery.

2.3.3 Sample Preservation and Holding Times

Sample preservation and holding time requirements are presented in MARRS SOP No. 14. All samples will be placed in coolers with ice for shipment at approximately 4 degrees Celsius ($^{\circ}\text{C}$). The laboratory, upon receipt, will store the samples in controlled refrigerators at 4°C ($\pm 2^{\circ}\text{C}$). The refrigerators will be securely locked and entry limited to authorized personnel only.

The receiving personnel of the laboratory will identify the samples with chemical preservatives. The pH of these samples will be measured and recorded. Temperature blank will be checked immediately upon receipt of the cooler at the laboratory and recorded in COC. The pH of samples for VOC will be recorded at the time of the analysis, if required. The laboratory will visually inspect the sample containers for leakage or breakage. Any discrepancies will be recorded in the COC.

Observed nonconformance for pH, temperature, leakage and breakage will be communicated to the Project Chemist within 8 hours. The Project Chemist, in consultation with the Program Manager and Field Team Leader will decide, on project specific basis, whether the analysis should proceed or should the samples be recollected and resubmitted to the laboratory. The laboratory, regardless of pending decision, will take measures to adjust the nonconforming pH to the required value. The temperature requirement will be waived for the samples delivered to the laboratory within 4 hours of collection if the samples were handled in accordance with the procedures specified.

2.3.4 Laboratory Receipt and Entry of Samples

The process of maintaining the integrity and documentation of sample custody starts from the moment the empty sample containers for collecting samples are shipped to the field team by the laboratory. The samples will be under the custody of a designated field team member up to the point of shipping. The custody of the samples will transfer to the laboratory once the sample custodian receives the shipped consignment of the samples. Upon receipt of the sample consignment, the laboratory sample custodian will:

- Verify that the sample security seals are intact
- Measure the cooler temperature by recording the temperature of the temperature blank.
Record the temperature in the COC
- Verify that the sample containers are intact and that there is no leakage
- Verify that the sample ID on the container labels agree with the sample ID in the accompanying COC.
- Review the COCs for accuracy and completeness

The samples, after being processed by the sample custodian, will be assigned laboratory ID numbers from the Laboratory Information Management System (LIMS). The sample ID will be entered in the LIMS data base together with associated information regarding the weight or volume of the sample received, matrix and the analysis required. The sample custodian will immediately inform the Laboratory Project Manager (LPM) if any discrepancies regarding the status of the sample containers, documentation, or the coolers are observed. The Program Manager will communicate the information to the Project Chemist and the Field Team Leader verbally followed by a complete written Non-Conformance Report (NCR) report. LIMS hard copy record will be generated upon the resolution of the discrepancy. The hard copy of the LIMS record will include:

- Date and time the sample was received
- Sample ID number generated by LIMS and the corresponding field ID number
- Source and the matrix of the sample.

LIMS sample management functions will be used to track the samples while in the laboratory's custody. The labels generated by LIMS system for each sample will display the date and time of sampling, sample matrix, sample analysis holding time and due dates and the analytical tests to be performed. The samples will be transferred to a secured laboratory refrigerator in the sample storage area. The laboratory will take required steps and precautions to minimize the potential cross contamination of the samples, especially for samples for VOC analysis. For example, the laboratory will isolate samples for trace or low level analysis from those for medium level and high level samples. The samples will be stored in locked and secured refrigerators maintained at 4°C +/- 2 °C. The laboratory sample custodian, or a designated staff member, will monitor the refrigerator temperature using National Institute of Standards and Technology (NIST) certified thermometers. Samples for VOC analysis will be stored in a segregated refrigerator to minimize cross contamination. Potential cross contamination in VOC storage refrigerator will be monitored by analyzing storage VOC blank on weekly basis. The red and entry limited to only authorized personnel.

Internal sample tracking system will be implemented by the laboratory. The samples will be internally tracked using internal COC. An internal COC will be initiated for each batch of 20 or less samples. Access to the samples will be limited to the personnel directly involved in the

MARRS Chemical Data Quality Management Plan

analytical process of the samples. The laboratory will ensure that the custody of the samples is maintained during the period they are out of the secured storage area. Each person who removes the sample from the secured storage area, either as a batch or individually, will assume the custody or will sign the appropriate laboratory COC. The COC, in conjunction with the LIMS, will also be used for tracking the samples movement within the laboratory.

Unused portions of the samples will be retained by the laboratory and disposed off as per client's instructions, or returned to the client for disposal.

2.3.5 Pre-Analysis Storage

Laboratory personnel will retrieve the samples from the storage area, and sign and date the custody tracking form. The samples will be stored in temporary storage refrigerators inside the laboratory until analyzed. The storage procedure will be specified in the laboratory QA manual. Storing the samples following a prescribed procedure is essential because these procedures are intended to retard biological action and hydrolysis of chemical constituents, and reduce the loss of volatile constituents from VOC and other samples. Methods of preservations available are pH control, chemical addition, and refrigeration.

The samples for VOC analysis will be accessed by the VOC analyst, who will be responsible for the custody of the samples until analyzed, after which they will be returned to the secured storage refrigerators. The samples that require pre-analysis extraction and clean-up will be accessed by the personnel from sample preparation laboratory, who will be responsible for the sample custody until extracted, after which the samples will be returned to the secured refrigerator storage. Appropriate custody forms will be signed by the person in the custody of the samples at any given time.

2.3.6 Post Analysis Storage

Water samples will be archived for a minimum period of two months commencing from the date of data package delivery, stored in refrigerator or cold storage walk-in facility with temperature maintained at 4°C +/- 2 °C. Soil samples will similarly archived for a minimum of 6 months. Sample extracts will be archived for a minimum of 6 months and stored at 4°C +/- 2 °C. The final extracts may be stored in a refrigerator located in the analytical laboratory as long as the security measures are maintained. Samples and extracts will be disposed off in accordance with the State and Federal regulations. All QA elements applicable to sample storage discussed in preceding sections will apply.

2.4 Analytical Methods Requirements

2.4.1 Overview of Analytical Methods

This section outlines analytical procedures, including instrumental procedures and QA/QC elements for sample analysis. The section will provide an outline of the procedures that will be applied to various analyses. The selection of the analytical procedures and required QA/QC elements will be project specific and will be included in project specific SAP and QAPP. Project specific analytical SOPs will be provided with project specific SAP.

2.4.1.1 Organic Analysis

Organic Extractions

MARRS Chemical Data Quality Management Plan

The analysis of Semi-Volatile Organic Compound (SVOC) requires that the target analytes of interest be isolated from the matrix by serially extracting a known volume or weight of a sample with a solvent, concentrate the extract, subject the concentrated extracts to clean-up procedures to remove potentially interfering chemical compounds, and re-concentrate the final extract to specified volume with appropriate solvent prior to instrumental analysis.

Organic extracts will be subjected to either chromatographic or mass spectrometric analytical procedures as required.

Gas Chromatography

The complex mixture of analytes present in the sample extracts are first separated into individual entities using the Gas Chromatograph (GC) or High Pressure Liquid Chromatograph (HPLC). The nature of the chemical structure of the analytes will decide whether GC or HPLC will be used for the separation and identification of individual analytes. Typically, GC analysis is performed under high temperature conditions. A capillary column, inside coated with a stationary phase, is mounted inside a GC oven between an injection port and a detector. An inert gas, called the mobile phase, is connected to flow through the column. The injection port and the detector are maintained at high temperature. The GC oven is temperature programmable so that the column temperature can be raised in programmed steps. One to two micro liters of the sample extract in an organic solvent is injected into the heated injection port. The solvent and the solutes are flash vaporized in the injection port and swept onto the capillary column by the inert gas. The solutes, at ambient temperature, will have higher affinity to the solid stationary phase and will be adsorbed. As the temperature of the oven and the column is slowly raised, the solute with low boiling points will be desorbed from the stationary phase and carried down the column by the carrier gas. The action of adsorption and desorption will continue throughout the migration through the column. The solutes least retained by the stationary phase adsorption process will elute first, normally those with low boiling points, followed by those retained the most (higher boiling points). Each analyte is detected by a GC detector as they elute through the column.

Compound identification is based on the time it takes from the point of injection to the point of elution into the detector. This time is called the Retention Time (RT) of an analyte, which is determined by analyzing calibration standards.

Not all analytes will have a unique retention time. Such analytes will be confirmed either by mass spectrometry, or by analyzing the extract using a second, dissimilar GC column.

HPLC analysis is based on the same principle of adsorption and desorption. The process takes at ambient temperature. HPLC uses solvents as mobile phase, in place of an inert gas, which transports the injected extract through an HPLC column under high solvent pressure. The separation of individual compounds is based on their partition coefficient process between the stationary phase of the column and the mobile phase. Compound identification is by RT, similar to GC procedure.

The chromatographs will be calibrated following the procedures described in EPA SW-846 Organic Methods, 3rd Edition. The project specific SAP will specify the order of calibration that should be used for the project.

General Chromatographic Detectors

Target analytes separated by the chromatographic column are detected by a detector. The detector generates a small electrical signal as an analyte is introduced into the detector system. The signal

is amplified and sent to a dedicated computer system for processing.

Various kinds of chromatographic detectors are available for use. Universal detectors, such as flame ionization detectors, are non-selective and for general purpose analysis. Some detectors are specific and will respond to certain groups of compounds. Electron Capture Detectors (ECD) will respond to chlorinated compounds, while Nitrogen-Phosphorous Detectors (NPD) will respond to compounds with nitrogen and phosphorous moiety. Selective detectors will generally provide lower reporting limits. Their selectivity provides additional level of confidence in the target compound identity. Mass spectrometers (MS), when used as GC detectors provide highest level of confidence for the identification of a target analyte. MS will provide a definitive identification of a target analyte irrespective of its chemical structure.

The detectors are calibrated, prior to the start of a project, using a set of calibration standards. Once calibrated successfully, the detectors will provide qualitative and quantitative results for an analyte.

Gas Chromatography-Mass Spectrometry

Gas chromatography, when interfaced with a mass spectrometer (GC-MS) offers a definitive identification and concentration of a target analyte. The separated analyte from a GC is introduced into the ionization chamber of mass spectrometer. The molecules of the analyte are bombarded with an electron beam strong enough to produce a molecular ion and corresponding daughter ions. The ions formed are channeled to an electron multiplier where they produce an electrical signal. The signals are amplified and captured by a dedicated computer system. Using a complex algorithm, the computer generates a bar chart called mass spectrum. The mass spectrum is compared against a library of reference mass spectra. The match of the unknown target analyte spectrum against the reference mass spectrum, and the RT established by analyzing standards are used to identify the analyte with very high confidence level. The quantitation is based on the measurement of the intensity of the primary (base peak) ions and comparing the response against an internal standard with multipoint calibration curve.

2.4.1.2 Metals Analysis

Metals analysis in environmental samples will be performed using one of the three techniques described briefly. The technique selected will be project specific and appropriate to meet the goals of the project DQO.

Most of the methods require sample digestion for all matrices prior to analysis for all techniques. Detailed analytical process will be included in the project specific SAP and QAPP, or referenced in appendix by inclusion of the corresponding SOP.

Inductively Coupled Plasma-Atomic Emission Spectroscopy

Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) requires that the sample to be analyzed be in acidic solution. To meet this condition, all matrices irrespective of the nature of the matrix require acidic digestion prior to analysis. EPA SW-846 Method 3010A and 3020A describe the procedures for aqueous and lechate samples. EPA SW-846 Method 3050B describes the procedures for the digestion of the solid samples.

Metal analysis by ICP-AES is described fully in EPA SW-846 Method 6010B. The method provides simultaneous or sequential multi-element determination of elements. The digested sample is nebulized to form an aerosol. The aerosol is directed to a plasma torch. The elements in

the sample emit a element specific emission light which is measured by photomultipliers. Element-specific atomic emission spectra are produced by radio-frequency inductively plasma. Accuracy of trace element measurements are enhanced by back-ground emission corrections.

Inductively Coupled Plasma-Mass Spectrometry

Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) requires that the sample to be analyzed be in acidic solution. To meet this condition, all matrices irrespective of the nature of the matrix require acidic digestion prior to analysis. EPA SW-846 Method 3020A describe the procedures for aqueous and lechate samples. EPA SW-846 Method 3050B (soil-modified for ICP-MS analysis) describes the procedures for the digestion of the solid samples. ICP-MS EPA Method SW-846 6020B Modified provides simultaneous multi-element determinations. Analytes from the sample are nebulized and introduced by argon gas into the plasma torch where the analytes are ionized. The ions formed are further transported to a quadrupole mass spectrometer. The ions are differentiated according to their mass to charge ratios and sequentially detected by channel electron multiplier. Interference as background ions from plasma gas, reagents and sample matrix may get introduced in the analysis impacting the final results. Interference correction for ICP-MS will include compensation for background ions contributed such sources.

Atomic Absorption Spectroscopy

Arsenic, lead and selenium are analyzed by Atomic Absorption Spectrometry (AAS) to achieve the required detection limits or when interferences are encountered.

AAS uses Graphite Furnace (GFAAS) and Flame (FAAS) to determine the elements in the final digested solutions.

GFAAS

The digested solution (1-2 micro liters) is introduced into a specially designed hollow graphite tube. The tube is placed inside a furnace. High electro thermal heat is applied to the furnace which dries and chars the digested solution, and the elements atomized. A light beam at a wavelength specific to the analyte of interest is passed across the atomized sample vapor. The resulting absorption of the light is measured which is proportional to the concentration of the element being analyzed. Background correction is applied to all analysis. Highly concentrated samples are diluted until the concentration falls within the linear range of the calibration. Interferences from other elements can be reduced by the use of modifiers as described in the corresponding EPA SW-846 Methods.

FAAS

FAAS provides lower detection limits than ICP and is used for the analysis of organic lead. The sample for organic lead is prepared using the procedures described in EPA SW-846 Method 3010A and FAAS can be used for other metal analysis as well if required for a project. The prepared sample extract is nebulized and aspirated in an air/acetylene flame. A beam of light at a specific wavelength corresponding to the target analyte is passed across the axis of the flame. The resulting absorption by the sample is proportional to the metal concentration. The interferences due to the flame, reagents, sample matrix etc will be compensated by background correction.

2.4.2 Method Descriptions

This section presents a brief summary of EPA SW-846 organic and inorganic analytical methods. Project specific QAPP and SAP will describe these methods in full.

2.4.2.1 Organics

2.4.2.1.1 Sample Preparation

EPA SW-846 Methods manual describes sample preparation methods for the analysis of organic compounds in detail. This section presents a brief synopsis of each method.

SW-3510C Separatory Funnel Liquid-Liquid Extraction

This method is used to extract target analytes from aqueous environmental samples. A measured volume of the sample is placed in the separatory funnel. The pH is adjusted as required. The sample in the funnel is extracted with methylene chloride by either manually shaking the funnel or using an automatic shaker. The organic phase is then separated from the aqueous phase, dried with anhydrous sulfate, concentrated, solvent exchanged with toluene or hexane to make the extract compatible to GC analysis and concentrated to 100 micro liters. A small volume (1-2 micro-liters) is used for analysis.

SW-3520C Continuous Liquid-Liquid Extraction

SW-3520C is an automatic, continuous extraction method to extract the target analytes from aqueous samples. A measured volume of the sample is placed in the continuous extractor, pH adjusted to the required value and extracted with methylene chloride for 18-24 hours. The extract is dried in a rotary evaporator, dissolved in organic solvent compatible to GC and concentrated for analysis.

SW-3540C Soxhlet Extraction

Soxhlet extraction procedure is specific for the extraction of semi volatile (SVOC) target analytes from solid sample matrices such as soil, sludge, and biological samples. A weighed amount of the sample is mixed with enough anhydrous sodium sulfate to make a free flowing mix. The mixed sample is placed in thimble which is placed inside the flask of the Soxhlet extractor. Organic solvent is added to the flask and sample extracted for a method specified time. The extract is dried in a rotary evaporator, dissolved in organic solvent compatible to GC and concentrated for analysis.

SW-3550B Sonication Extraction

Sonication extraction of solid samples such as soil, sludge and biological samples is a quick, efficient method for the extraction of SVOC target analytes. A weighed sample is mixed with enough anhydrous sodium sulfate so that the mixture becomes free flowing. The mixture is placed in a flask, solvent such as methylene chloride or other specified solvent is added, and sonicated for method specified time. The contents of the flask are filtered to remove the solids. The solvent extract is dried in rotary evaporator. The dried extract is dissolved in GC compatible solvent. The final extract is concentrated and prepared for the analysis.

SW-5030B Purge and Trap

Method SW-5030B is specific to the determination of Volatile Organic Compound (VOC) in liquid matrices. A 5 milliliter aliquot of the sample is placed in a purge vessel. An inert gas is bubbled through the sample at a constant rate. The volatile compounds are purged for a specific time from the sample and transported onto a sorption media. The sorption media will retain and concentrate the volatile compounds. At the completion of the purge cycle, the sorption media is flash heated and at the same time back flushed by an inert gas. The inert gas will transport the volatile compounds to the injection port of the gas chromatograph where they are condensed and

concentrated under low temperature. The volatile compounds condensed on the column are flash-vaporized at the end of the purge and trap cycle. The column is heated to elute the compounds, which are detected by the appropriate detector. For low-level aqueous analysis, 25 milliliter of the sample is used.

This method is also used for the analysis of non-aqueous matrices and soil samples with very high concentrations of the analyte present. This is a dilution method whereby a known volume of methanol is added to a known amount of the sample. An aliquot of methanol added to certified reagent water and purged with an inert gas. This method is also applicable to the low level soil samples. A known weight of the soil sample is placed in the soil purge tube, 5 milliliters of water added and the purge tube is heated to 40°C. The inert gas is bubbled through the tube to purge the volatile organic compounds. The purged volatile compounds are transported to the sorption tube where they are adsorbed and concentrated. The compounds are flash vaporized at the end of the purge cycle, back flushed with an inert gas and transported to the GC capillary column for analysis.

2.4.2.1.2 Gas Chromatography and Gas Chromatography-Mass Spectrometry

This section will briefly describe the applications of gas chromatographic analysis for various organic analytical parameters. All methods are referenced by EPA-SW 846 Methods.

SW-8021B Halogenated Volatile Organic Compounds by GC/HECD and Purgeable Aromatic Compounds by GC/PID

Method SW-8021B is purge and trap method to determine halogenated volatile organic compounds and aromatic organic compounds simultaneously using two GC detectors. Sample preparation is based on methods SW-5030B and SW-5035. The purged compounds are separated by using GC capillary column installed in a temperature programmable GC oven. The temperature programmed GC oven will separate individual compounds from the mixture. The separated compounds are split two ways at the end of the elution from the GC column which is connected to the two detectors using a 'Y' connector. Hall Electrolytic Conductivity Detector (HECD) will respond to halogenated compounds, while the Photoionization Detector (PID) will respond to the Aromatic Volatile Compounds. While low-level volatile compounds in aqueous and soil samples can be analyzed directly by this method, medium and high level determination will require extraction and dilution using methanol.

SW-8015B Modified – Total Petroleum Hydrocarbons by GC/FID

Total Petroleum Hydrocarbon (TPH) includes gasoline, diesel fuel, jet fuel and motor oil. Each is differentiated by the length of carbon chains of the hydrocarbons:

- Gasoline C-7 to.... C-10
- JP-4 (Jet Fuel)..... C-8..... to.... C-13
- Diesel C-10..... to.... C-24
- Motor Oil C-24..... to.... C-36

Gasoline is classed as volatile petroleum hydrocarbon and can be determined by purge and trap methods (SW-5030B or SW05035).

Other TPH fractions are extractable hydrocarbons and are analyzed after extraction using methods SW-3510C for aqueous samples and 3550B for solids. A sample, either from purge and trap or the extract, is introduced into the temperature programmed gas chromatograph. The

compounds are separated and detected by the Flame Ionization Detector (FID).

The extractable hydrocarbons are extracted with methylene chloride, concentrated and analyzed by GC-FID.

A field sample may contain two different fractions of TPH that may overlap during GC analysis. Under such conditions it would not be possible to use two specific calibrations for the two fractions. The laboratory may, in such cases, decide to calibrate and quantify the TPH against one reference fuel. The laboratory will be required to be consistent in their use of reference fuels under such conditions.

SW-8081A and SW-8082A Organochlorine Pesticides and Polychlorinated Biphenyls (PCB)

Both these groups are halogenated compounds which respond well to Electron Capture Detectors (ECD). Methods SW-8081A and SW-8082 are GC methods equipped with ECD detectors.

The pesticides generate single peak chromatograms at RT unique to only one pesticide. PCBs, also known as arochlors, generate complex multiple peak chromatograms in unique and recognizable patterns. There are several sub-groups of arochlors and each generates its unique chromatographic pattern. Toxaphene also generates a chromatogram with unique pattern.

Methods SW-3510 or SW-3520C is used to extract the pesticides and PCBs from aqueous samples. The pH of the sample is adjusted to neutral prior to extraction. Soil samples are extracted using sonication Method SW-3550B, using methylene chloride and acetone. The extract solvent is exchanged to hexane prior to GC-ECD analysis.

Arochlors or Toxaphene, if present, will be detected during the pesticides analysis. These will be qualitatively identified from pattern recognition and keeping in mind that the pesticides will generate only single peak chromatograms. The presence of arochlors and toxaphenes should be confirmed, and quantified, using the GC-ECD system calibrated for these compounds with all applicable calibrations and QA/QC criteria as per the method.

SW-8141A Organophosphorous Pesticides

Method Sw-8141A uses capillary gas chromatograph equipped with either Nitrogen Phosphorous Detector (NPD) or Flame Photometric Detector (FPD). The GC is temperature programmed. These groups of pesticides will generate single peak chromatograms. The individual analyte peaks are identified and quantified by comparing the RT and intensities against the chromatograms from the calibration standards. The presence of the pesticides is confirmed using a second GC column analysis or mass spectrometry.

The pesticides from aqueous samples are extracted with methylene chloride using EPA SW-846 Methods 3510 or 3520C. Soil samples are extracted using EPA SW-846 Method 3550B, an ultrasonic extraction procedure. Extraction methods EPA SW-846 3540C or 3541 will be used for preparing samples for analysis by EPA SW-846 Method 8141A. For both the extracts, aqueous and soil, the final extract solvent is solvent exchanged with hexane for GC analysis.

SW-8151A Chlorinated Herbicides

Chlorinated herbicides, once extracted by using the procedures provided in EPA SW-846 Method 8151A is analyzed by capillary GC equipped with ECD. The aqueous samples are extracted using

MARRS Chemical Data Quality Management Plan

EPA Method SW-846 3510C with ethyl ether, and soil samples using EPA Method SW-846 3550B ethyl ether/acetone. The extracts, prior to GC analysis, are first hydrolyzed with potassium hydroxide, acidified and re-extracted in ethyl ether, and concentrated using rotary evaporator followed by a gentle nitrogen gas streaming onto the surface of the solvent. The final extract is methylated with diazomethane and solvent exchanged with hexane for GC analysis.

SW-8260B Volatile Organic Compounds by Purge and Trap and GCMS

EPA Method SW-846 8260B is based on extracting the Volatile Organic Compounds (VOC) from aqueous or soil samples by purging an inert gas through the sample. The extracted VOCs are adsorbed and concentrated using a sorption tube filled with sorptive media through a purge cycle. The VOCs adsorbed on the sorption tube are desorbed and transported to the capillary GC using MS as detector. The individual VOCs are separated by the capillary column and detected by the MS for identification and quantitation. Aqueous samples are purged at room temperature, while the soil samples are heated to 40°C to help releasing the VOCs from the matrix.

VOC analysis for water and soil samples is performed at two levels of concentrations: low, medium. Samples for low-level analysis are purged and analyzed directly by using purge and trap methods SW-846 5030B or SW-5035B as applicable. Low level water analysis uses 25 ml of water and 5 ml of water for medium level analysis. Soils for medium level analysis will require methanol extraction prior to the analysis.

Liquid wastes can be analyzed using the aqueous method, but with dilution. A small volume of the sample is diluted in 5 ml of reagent water. The diluted sample is purged and analyzed. It may be necessary to prepare a number of serially diluted samples if the concentrations of the VOCs expected are significantly higher than the upper limit of the calibration.

SW-8270C Semivolatile Organic Compounds by GCMS

EPA Method SW-846 8270C is used identify and quantify acidic, basic and neutral target analytes which make up semivolatile class of organic compounds (SVOC). This include a range of compounds such as pesticides, polynuclear aromatic hydrocarbons, polychlorinated biphenyls, phthalates, organophosphorous pesticides, chlorinated herbicides, nitrosamines, haloethers, aldehydes, anilines, ketones, pyridines, quinolines, aromatic nitrocompounds and phenols.

Samples for SVOC analysis are extracted prior to the GCMS analysis. Aqueous samples are extracted using EPA Method SW-846 3550B for soils and 3510C or 3520C for aqueous samples. The extracts are concentrated and solvent exchanged with hexane prior to GCMS analysis.

SW-8280A Polychlorinated Dibenzodioxins and Dibenzofurans by Low Resolution GCMS

Two EPA SW-846 methods are available for the analysis of Polychlorinated Dibenzodioxins (PCDD) and Polychlorinated Dibenzofuran (PCDF). EPA SW-846 Method 8280A is a high resolution GC and low resolution MS method, and EPA SW-846 Method 8290 which is a high resolution GC and high resolution mass spectrometry. Both methods involve additional quality control elements. Both methods are based on the use of isotopically labeled analogs of target analytes. This provides much lower quantitation limits than possible by routine GCMS and also to monitor method performance.

Method SW-846 8280A requires use of six isotopically labeled C13 analogs. These analogs behave just like unlabeled target analytes (also called congeners). They will elute from GC at the

same RT as their unlabeled analogs. The labeled analogs are differentiated from the unlabeled target analytes by the difference in their mass spectra. The labeled analogs, therefore, exhibit the same characteristics as the unlabeled analogs. The calibration curve for target analyte quantitation is relative to the isotopic analog. Since both the groups behave identically under extraction, and all samples are spiked with C13 analogs, the matrix effect on the recovery efficiency of the analytical procedures can be evaluated from the recovery of the C13 analogs. The analysis of MS and MSD therefore is not required under this method. The batch QC control will be provided by LCS and LCSD which will be analyzed with each batch.

Analysis of PCDD and PCDF is based on Single Ion Monitoring. The congeners are constituted of different chlorination levels. Environmentally important congeners are the ones with chlorination levels of four to eight. The method uses formulae to calculate detection limits of each individual congener and each chlorination level. The calculation is based on the noise level present within the elution RT window of each congener and the response (intensity) of the peak of the labeled analog also called internal standard. The complex calculation is performed by the computer which determines the detection limit of each congener.

The detection limits are reported at one of the three levels of detection limits:

- If the target analyte is not detected within a given RT window, than an “Estimated Detection Limit” (EDL) is determined based on the noise level within the RT window. The target congener is reported as “not detected” at the calculated EDL level.
- The congener detected within the Lower Calibration Limit (LCL) and Upper Calibration Limit (UCL) is reported as detected and without qualifiers. If a congener is present at a level below LCL, it is reported but flagged as J.
- If a peak is present within the RT window, but does not satisfy the other required criteria to be identified as PCDD/PCDF (ion ratios, absolute RT, absence of diphenyl ether interference, and elution of the congener and internal standard quantitation peaks within specified time), than an Estimated Maximum Possible Concentration (EMPC) would be reported as detection limit and the target congener as non-detect at the EMPC level.

SW-8290 PCDD/PCDF by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS)

Method SW- 846 8290 is based on operating a mass spectrometer in high resolution mode, at a resolution of about 10,000, and acquiring the MS data under Single Ion Monitoring (SIM) conditions. HRGC/HRMS at high resolution provides a technique for the detection of all PCDD and PCDF congeners (tetra through to octa chlorinated PCDD and PCDF) at a concentration of parts-per-trillion in solid matrices to parts-per-quadrillion in aqueous matrix.

This method uses isotopically labeled C13 analogs corresponding to each PCDD and PCDF congener, except for octa-PCDD/PCDF where only one analog is applied to both. More rigorous QC elements are required for this method.

The results are reported at three different levels. For a ‘non-detect’ congener, an EDL is determined based on the signal-to-noise ratio within the RT elution window of the congener. The target congener is reported as non-detect at the EDL.

The confirmed and detected congeners are reported down to the LCL without any qualifications. Qualitatively confirmed analytes at below the LCL, but above target detection limit (TDL), are

reported as 'estimated'. TDL is determined by the laboratory, and is the value at which there is a minimal probability of false positive result.

A peak detected below the TDL that does not meet the criteria for qualitative confirmation (RT, ion ratio, signal to noise ratio, absence of diphenyl ether) are reported as non-detect at the detection limit determined by the peak detected within the RT window of the congener.

MS/MSD to monitor the matrix effect is not required for this method. The matrix effect is determined by the recoveries of the labeled analogues. Similarly, LCS and LCSD to monitor the laboratory analytical procedure is also not required. The method blank, which also is spiked with the labeled analogues serves both as a method blank and LCS. Any problems with the laboratory procedures will be reflected in the unacceptable recoveries of the labeled analogs in the blank.

2.4.2.1.3 High Performance Liquid Chromatography

High Pressure Liquid Chromatography (HPLC) is used for the analysis of a group of compounds that are not amenable to GC analysis. HPLC is performed at an ambient temperature. The extract of a sample is injected in the injection loop of HPLC. The injected aliquot is transported to the HPLC column by solvent under high pressure. The analytes within the extract aliquot are separated based on their differing affinity between the solvent mobile phase and the stationary phase. The eluted analytes are detected by non-destructive detectors like Ultra Violet (UV) or Photo Diode Array (PDA) detectors. The signal is then further processed with dedicated computer systems and reported.

SW-8310 Polynuclear Aromatic Hydrocarbons

Method SW-8310 is an HPLC procedure for the detection of Polynuclear Aromatic Hydrocarbons (PAH) in aqueous and solid matrices using UV and fluorescent detectors. The chromatograms generated by HPLC are discrete peaks of analytes. The identification and quantitation of the peak is by comparison of the analyte RT against the RT determined by the analysis of calibration standards and the response factor calculated by the standards calibration curve.

Aqueous samples are extracted following the procedures provided in method SW-3510C or SW-3520C using methylene chloride. The solids are extracted by method SW-3550B using methylene chloride and acetone. The final extract is solvent exchanged for methanol for compatibility for analysis by HPLC.

SW-8330, SW-8321A Modified-Nitroaromatics and Nitroamines

The analysis of nitroaromatics and nitrosamines can be performed either using an HPLC method (SW-8330 or). Method 8321A is a HPLC-Thermo Spray Mass Spectrometry (HPLC/TS/MS) method used for the analysis of Solvent Extractable Nonvolatile Compounds. Both methods are used, based on project specific DQO and SAP, for the analysis of explosives residues in water, sediments and soils. The analytes are separated using Reversed Phase C-18 (RP C-18) HPLC columns. The analytes are detected by UV detector at the wavelength of 250nm. The positive results from the HPLC-UV analysis are confirmed by a cyano-column.

Aqueous samples destined for low level explosives analysis are first "salted out" using sodium chloride, then extracted with acetonitrile and the extract concentrated for analysis. Aqueous samples suspected of having high levels of explosives present can be analyzed directly. These samples are filtered prior to HPLC to remove suspended solids that would otherwise interfere with the analysis.

Soil and sediment samples are extracted with acetonitrile, treated with calcium chloride solution, and filtered followed by HPLC analysis.

2.4.2.2 Inorganic

2.4.2.2.1 Sample Preparation

SW-3020A Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA

This method is used to prepare aqueous samples for metals analysis by GFAA. An aliquot of the sample is mixed with 3 ml of nitric acid and refluxed in closed environment in Griffin beaker. The nitric acid is subsequently added to the digestate until the color is stabilized or goes colorless. The digestate is cooled and brought up to the volume specified in the method with dilute nitric acid. The final dilution will be in 3% nitric acid. The digested extract is ready for analysis. The digestate will require filtration if suspended solids are present.

The method is modified if arsenic and selenium are also target metal analytes. The modifications are the addition of hydrogen peroxide with nitric acid during digestion, and reducing the final volume make-up with nitric acid to 10-20 ml rather than 5 ml specified in the method.

SW-3020A Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP/MS

This method is used to prepare aqueous samples for metals analysis by ICP/MS. An aliquot of the sample is mixed with 3 ml of nitric acid and refluxed in closed environment in Griffin beaker. The hydrogen peroxide is subsequently added to the digestate until the color is stabilized or goes colorless. The digestate is cooled and brought up to the volume specified in the method (approximately 20 ml) and cooled. 1 ml of 1:1 hydrochloric acid: water is added to the digestate and brought up to the required volume with deionized water. Suspended solids, if present, are removed by filtering or centrifugation.

The method is modified for the digestion of certain elements (such as silver). The modifications are the addition of hydrogen peroxide and hydrochloric acid during digestion. The final volume of the digestate is reduced to 20 ml to 25 ml to avoid the loss of more volatile elements like antimony.

SW-3050B Acid Digestion of Sediments, Sledges and Solids

The acid digestion using Method SW-3050B will prepare soil and sediment samples for analysis by ICP, ICP/MS, GFAA and FAAS. An aliquot of the sample is digested in 1:1 nitric acid until the color is stable or the digestate becomes colorless. The digestion is further continued with the addition of hydrochloric acid and hydrogen peroxide for FAAL and ICP analysis for metals. The final volume is adjusted to 100 ml for analysis.

SW-3060A Alkaline Digestion for Hexavalent Chromium

This method provides the procedure for digesting soil and sediment for Hexavalent Chromium (HexChrom). A 2 gm aliquot of the sample is weighed in a beaker. 8 ml of NaCO₃/NaOH is added and the mixture digested for 45 minutes. The solution is cooled and quantitatively transferred to a hundred ml volumetric flask. The final extract is neutralized with nitric acid just prior to the analysis.

2.4.2.2.2 Atomic Emission Spectroscopy

SW-6010B Inductively Coupled Plasma Atomic Emission Spectroscopy

Inductively Coupled Plasma Emission Atomic Spectroscopy (ICP-AES) determines a range of trace elements, including metals in various matrices including unfiltered ground water, aqueous samples, sludge, solids, industrial wastes and sediments. The method can be used for the analysis of multiple elements using sequential or simultaneous optical systems. A radio frequency induced coupled plasma will generate element specific emission spectra from an aerosol of the sample digestate introduced into the plasma., which are further dispersed by a grating spectrometer. Photosensitive devices are employed to monitor the emission spectra. Trace level determinations require background correction at or near the emission spectral line of the target analyte.

The ICP-AES is calibrated daily using minimum of three standards, supported by calibration verification standard, interference check standard, and blanks. The project specific SAP and QAPP will define QC elements and the method in detail.

SW-6020 Inductively Coupled Plasma /Mass Spectrometry

ICP/MS is used for the determination of elements at sub-microgram concentrations. The method is applicable to the metals analysis in water samples and waste extracts or digests. Water samples are analyzed directly without digestion. Acid digestion prior to analysis is required for ground water, aqueous samples, industrial wastes, soils, sludge, and sediments.

The ICP/MS is tuned and calibrated daily. Other QC elements include internal standard monitoring, calibration verification standards, interference calculation checks, and blanks. The project specific SAP and QAPP will define QC elements and method in detail.

2.4.2.2.3 Atomic Absorption Spectroscopy

SW-7000A Total Metals by GFAA

Acid digestion of an aliquot of the sample is required for analysis by GFAA. A small aliquot of the sample digestate is place inside a small graphite tube. The sample tube is placed in a furnace. The furnace temperature is rapidly raised to the required temperature. The digestate extract is rapidly charred and the element species in the digestate are atomized. A light beam from a hollow cathode lamp is passed through the furnace open at both the ends. The light beam traverses the atomized metal species. The atomized metal species will absorb the light at a specific wave length. The light is than directed to a grating monochromator that measures the absorbance. The concentration is proportional to the absorbance.

The GFAA is calibrated daily. Instrument QC elements include calibration verification standards, and blanks. The project specific SAP and QAPP will define QC elements and the method in detail.

SW-7470A, SW-7471A Mercury by Cold Vapor AAS

Method SW-7470A describes the method to determine mercury in liquid waste using Manual Cold-Vapor Technique. Method SW-7471A is used for determining mercury in solid or semisolid waste using Manual Cold-Vapor Technique.

A quantitative aliquot of the sample is digested with sulfuric acid, followed by nitric acid, stirring the sample continuously. Potassium permanganate is than added to the mixture. The

permanganate is added to eliminate possible interference from sulfides. The permanganate is added to the standards and the blank as well. The permanganate is added until the purple color persists for 15 minutes. Potassium persulfate is added to each sample, standard and the blank, and then digested on water bath at 95°C for 2 hours. The mixture is allowed to cool; sodium chloride hydroxylamine sulfate is added to reduce the excess permanganate. Stannous sulfate is added just prior to the attachment of the aeration bottle to the aeration apparatus. The sample from the aeration bottle is introduced into the AAS for analysis.

The standards for the five point calibration are digested with the samples. Daily AAS QC checks are the calibration curve and the blanks. The project specific SAP and QAPP will define QC elements and the method in detail.

2.4.2.2.4 Spectrophotometric Methods

EPA-364.2 Phosphorous

Various forms of dissolved and insoluble phosphorous and orthophosphorous in drinking water, surface and saline water, and domestic and industrial waters can be determined using this method. The procedure is based on colorimetric reaction of ammonium molybdate and antimony potassium tartarate with dilute solutions of phosphorous to form a antimony-phospho-molybdate complex. The complex, when reduced by ascorbic acid, generates an intensely blue color. The color is proportional to the phosphorous concentration. The color is spectrophotometrically measured at a wavelength of 650 nm.

EPA 418.1/SW-9071A/SM-5520C.E.F Total Recoverable Petroleum Hydrocarbons

This methods use Freon-113 to extract hydrocarbons from various matrices such as water, waste water, and solids. Except for Method SW-9071, which uses n-hexane to extract the extractable material (HEM), water samples are extracted with Freon-113 under acidic conditions. TPH from soil samples are extracted using reflux extraction system. The concentration of TPH is determined using infrared spectrophotometer.

Soil samples for extractable TPH are prepared by method SW-9071A/SM-5520C. A known weight of sludge is acidified with hydrochloric acid, and mixed with enough magnesium sulfate so that the mixture with sludge forms a free flowing powder. The mixture is transferred to a Soxhlet thimble and refluxed with Freon-113, or n-hexane as applicable, for 4 to 6 hours. Silica gel is added to the final refluxed extract to remove polar hydrocarbons. The TPH concentration is determined using infrared spectrophotometer as for aqueous samples.

Quantitation is based on calibration curve based on standard concentration versus IR peak height. The concentration of the unknown is determined by comparing the peak height against the calibration curve. Method EPA-418.1 recommends chlorobenzene as a reference oil compounds while Method SM-5520C recommends benzene. The laboratory will ensure that their analytical procedures are stringent enough to ensure removal of non-target analytes for TPH analysis.

SW-7196A Hexavalent Chromium

This method is based on colorimetric reaction of hexavalent-chromium with diphenylcarbazide under acidic conditions. The method is applicable to water, lechate, and alkaline digestate from SW-3060A.

A 9.5 ml of the sample or digestate is transferred to a 10 ml volumetric flask and 0.2 ml of the

diphenylcarbazine is added. The pH of the solution is adjusted to pH 2 with enough sulfuric acid. The volume is then made up to 10 ml with ASTM Type II reagent water. The flask is let stand for 5 to 10 minutes for the color to develop. The absorbance of the colored solution is read using 1 centimeter cell at a wavelength of 540 nm.

SW-9010B/EPA-335.3 Cyanide

These methods are based on the generation of hydrocyanic acid from cyanide, converted to cyanogens hydrochloride by reacting with chloramine-T, which reacts with pyridine and barbituric acid to generate a red-colored complex. The hydrocyanic acid is generated by the UV digestion and distillation. These methods are applicable to drinking water, surface waters, and domestic and industrial wastes.

2.4.2.2.5 Ion Chromatography

Ion chromatography is applicable to the determination of inorganic anions in surface and saline waters, domestic water, and industrial wastes. The principle of ion chromatography is much like that of HPLC. The mobile solvent phase in ion chromatography is a solution of inorganic salts such as Na_2CO_3 and NaHCO_3 rather than organic solvents used in HPLC.

EPA-300.0/SW-9056 Anions by Ion Chromatography

Anions chloride, bromide, fluoride, nitrate, nitrite, orthophosphate, sulfate, and perchlorate are analyzed using these two methods.

Method 300.0 is primarily applicable to drinking waters. Method SW-9056 is adopted for the analysis of anions in soil matrix. The anions are separated in ion chromatograph equipped with an ion chromatography column. The anions are eluted by a mixed mobile phase composed of Na_2CO_3 and NaHCO_3 . The separation is effected by the differing partition coefficients of the anions between the stationary ion exchange resins in the analytical column and the mobile phase. Non-aqueous samples are leached in deionized water at sample to water ratio of 1:5 (w/v) for 60 minutes, followed by filtering the solids. The resulting leachate is analyzed in the same manner as an aqueous sample.

2.4.2.2.6 Gravimetric Methods

EPA 413.1/SW-9070 Extractable Oil and Grease

Method EPA 413.1 uses Freon-113 to extract fats, oils, waxes, soaps, greases etc. from water and waste waters. Sludge, soil and sediments are extracted using method SW-9070. For both the methods, the samples are acidified and extracted with Freon-113. The extracts are placed in pre-weighed evaporation dishes. The solvent is evaporated using a method recommended procedure and the resulting residue dried till constant weight is achieved. The weight of the residue is determined by subtracting the pre-weight of the empty dish from the total weight of the dish and the residue. The quantitative range is from 5 mg/L to 1000 mg/L.

Following EPA Method 413.1 for aqueous samples, one liter of sample is acidified with hydrochloric acid to $\text{pH} < 2$. The sample is extracted serially three times with Freon-113. The three extracts are combined, filtered through anhydrous sodium sulfate to remove moisture, and solvent evaporated using a water bath. The Freon is allowed to evaporate off. The residue is dried until constant weight is achieved. The dish with residue is weighed. The weight of the empty dish is subtracted from the total weight of the residue and the dish. The gravimetric weight of the residue is calculated in mg/L.

Method SW-9070 is used to determine oils and greases in sediment, soil, and sludge. An aliquot of the sample is acidified with hydrochloric acid to pH <2, and mixed with enough anhydrous magnesium sulfate to produce a free flowing powder. The powder is placed in a Soxhlet thimble. The sample is refluxed for a minimum of 4 hours with Freon-113. The extract is placed inside a pre-weighed evaporation dish and placed on a water bath. The Freon is allowed to evaporate off. The residue is dried until constant weight is achieved. The dish with residue is weighed. The weight of the empty dish is subtracted from the total weight of the residue and the dish. The gravimetric weight of the residue is calculated in mg/L.

EPA 160.1 Total Dissolved Solids

Using this method, the sample is well shaken and a known volume of the sample filtered through a glass-fiber filter paper of a method specified porosity. The filtrate is transferred to a pre-weighed evaporation dish and dried until constant weight is achieved. The weight of the residue is determined by subtracting the weight of the pre-weighed dish from the total weight of the dish and the residue. The weight of the dissolved solids is reported as mg/L.

EPA 160.2 Total Suspended Solids

A well shaken aliquot of the sample is measured accurately and filtered through a pre-weighed glass fiber filter. The filter is dried in an oven until constant weight is achieved. The resultant weight of the suspended solids is calculated and expressed as mg/L.

2.4.2.2.7 Miscellaneous Methods

EPA-120.1 Specific Conductance

This method uses a temperature compensated conductivity meter to measure the specific conductance of aqueous samples. The conductance is measured as a ratio of electric current to applied voltage. A conductance cell and a variable output Wheatstone bridge circuit is used to measure the conductance, which is reported as $\mu\text{mho/cm}$.

EPA-150.1/SW-9045C pH

Using this method, pH of a water sample is measured using a pH meter equipped with a calibrated and temperature compensated pH electrode. A clean beaker is filled with enough volume of sample so that the electrode is immersed to the appropriate level. The beaker is placed on a magnetic stirrer and the sample is stirred for a few seconds. The electrode is immersed in the sample taking care that it does not touch the stirrer. It is kept immersed in the sample until the pH reading is stabilized. The electrode is removed from the sample, rinsed with deionized water and stored as recommended in the method. The electrode is calibrated against certified buffer solutions in both, the acidic and alkaline range.

EPA-180.1 Turbidity

Turbidity of an aqueous sample is measured by nephelometric method. This method compares the intensity of light scattered by a sample with the intensity of light scattered by a reference standard. The instrument, Nephelometer, is calibrated against a reference standard solution made up of Formazine suspension. The procedure is performed under defined conditions. The higher the intensity of the scattered light higher the turbidity. The result is expressed as Nephelometric Turbidity Unit (NTU).

2.4.3 Preventive Maintenance Program

The laboratory will maintain an effective maintenance program for all analytical instruments. Effective instrument maintenance will ensure that the analytical instruments' performance will meet the project requirement specifications and the goals set out in the project specific DQOs. The preventive maintenance will focus on four areas to ensure the availability of operational instruments to meet a specific project's demands.

Maintenance Responsibilities

The laboratory will assign the maintenance responsibilities to the respective laboratory managers. The managers will establish appropriate maintenance schedules and SOPs for each instrument. The managers will delegate the responsibility to perform the maintenance of the instrument to instrument operators. The laboratory managers will be responsible to review the reports to ensure that the maintenance is performed as required by the established SOP and as per schedule established.

The managers will assign maintenance logbooks to each instrument under their individual responsibilities. The logbook will be kept next to the instruments at all times. The instrument operators will use the logbooks to record the maintenance performed, problems encountered, corrective actions implemented, parts ordered and any other activity regarding the instrument under their control. The logbooks will be dated and signed by each operator making an entry in the book. Respective laboratory managers will review the logbooks at regular intervals to ensure conformance to the maintenance schedule and procedures set out in appropriate SOPs.

Maintenance Schedules

The maintenance of analytical instruments is scheduled based on manufacturer's recommendations, sample through-put, as-needed, and experience. The maintenance program will be effective when the maintenance schedules are complied with. The schedules will be either in house maintenance programs for some for some instruments, such as the routine maintenance of a GC, or by service contract for more complex systems and maintenance procedures such as for MS, GFAA and ICP. The maintenance activities will be recorded in the instrument log-books.

Spare Parts

The laboratory will ensure that the spare part inventory carries essential parts for routine maintenance and repairs to minimize instrument down-time. The inventory will be stocked with spare parts required for daily maintenance (e.g. GC injection parts that are replaced on daily basis), subject to frequent failures (e.g. MS source filaments), and those parts which cannot be obtained in time. The laboratory will maintain an inventory of supplies such as GC capillary columns, gas line fittings including flow and pressure gauges, spare lamps for AAS, graphite tubes for GFAA, and any other supplies deemed necessary to minimize the down time of an analytical instrument.

Back-up Analytical Instruments

The laboratory will be equipped with back-up analytical systems in case the primary system experiences a catastrophic break-down. The back-up systems will be maintained in the same fashion as the primary systems. At the minimum, a set of calibration standards will be analyzed regularly on the back-up systems to ensure that the system satisfies the required QC criteria, and that the back-up system will be available immediately in case the primary system experiences a catastrophic breakdown.

The laboratory manager will communicate with the Program Manager immediately if an instrument experiences a catastrophic break down, and the possible impact it may have on the projects associated with the instrument.

2.4.4 Laboratory Data Reduction and Review

Data Reduction

Data reduction is the process of converting raw data to the final data using calculations as specified in the EPA analytical methods, QAPP, and SAP. Computer-based data reduction programs are developed using the data reduction calculations. The calculation formulae presented in the documents referred to above are used to manually review the computer-based calculations for accuracy.

The laboratory will be responsible for maintaining a list, and documentation of the validity of these data-reduction programs. The laboratory will also validate any spread-sheets and data base developed for computerized data reduction. All information required for data reduction will be documented by the laboratory and be available to reconstruct the final result during data validation process. The information required include raw data, calibration data, MS tuning records, weight/volume of the sample, final volume of the extracts, per cent moisture, blank results, internal standard concentrations, interference check results, background corrections and any other information directly connected with data reduction processes.

Manual data reduction by the analyst will be required for the raw data from instrument that are not interfaced with computers. The analyst will reduce the raw data by manual calculations, specified in the methods, and manually enter the final data in the assigned laboratory note book. The final data is then entered manually in the computer data base. The analyst will ensure that the data entry in the computer is error free and reflects the data in the laboratory note book.

Some analytical data do not require data reduction, such as pH and temperature. Such data is directly recorded in the laboratory note book and latter manually entered in the computer data base. The analyst will ensure that the data entry in the computer is error free and reflects the data in the laboratory note book.

The final computer based data are archived on magnetic tapes, compact discs, or other compatible media, and are archived for one year or as per client's decision. The hard copy data is archived in special storage areas and are archived for 5 years.

Laboratory Data Review Assessment

Data review by the laboratory for accuracy, precision, and completeness is a multi-level process. Review at each level performs a specific function to ensure that no erroneous data are released to the client. Any errors discovered are corrected, records made and appropriate corrective actions implemented.

The laboratory will review the data at three different levels described below:

Level 1 Data Review will be performed by the analyst, who generates the data, at bench level. The analyst will review the data for correctness and completeness following protocols set out in appropriate SOPs. The review will include:

- Sample tracking for sample preparation and extraction

MARRS Chemical Data Quality Management Plan

- Sample identification
- Accuracy and completeness of analytical information
- Confirm appropriate calibration standards have been analyzed
- Confirm the tuning parameters for the MS analysis are acceptable
- Confirm that the analytical results are correct
- QC samples are analyzed as required and are within acceptance range
- Method blanks are analyzed and are free of contamination
- The raw data identifies the instrument and corresponding analytical method and initial calibration
- Dilution factors are reported when applicable
- The project specific goals are satisfied
- All required documentation is available with the data package, including challenges faced during sample preparation and analysis, corrective actions implemented, holding times have been met, non-conformance records are available

Level-2 data review will be performed by a data review specialist or a group leader. The goal of this review will be to provide an independent assessment of the data package. Level-2 data review process will include:

- Review of the calibration data to ensure that the calibrations are scientifically sound, corresponds to the method used, the dates and times of calibrations agree with dates and times of the sample analysis. Manually check the calculations for response factors at random
- Field COCs and inter-laboratory COCs and sample tracking information is complete
- The required QC samples have been analyzed and are in compliance with the established limits
- Correct reporting limits/detection limits are used to meet the project requirements
- The analytes are correctly identified based on parameters such as RT and the mass spectra
- Correct formulae and values are used for data reduction and calculations are accurate
- Required documentation to support sample preparation and analysis are present
- The final data is acceptable and accurately entered in the data base and ready for reporting
- The hard copy data package is complete and ready for archiving.

Level 2 data review is more in-depth review than Level 1 review. Level 2 provides for the review of all QC parameters, QC recoveries of the samples, and random manual check of the calculations. At least 10 percent of the data are reviewed in depth from sample preparation documents to final data reporting. The data package is considered to be acceptable if no errors or procedural problems are noted in the 10 percent of the data reviewed in-depth. If any errors are discovered during this stage of the review, than the analyst is instructed to review the data again and implement corrective actions as required. In addition, 100 percent of the data package will be reviewed by the group leader or the data review specialist to ensure that the rest of the data

MARRS Chemical Data Quality Management Plan

package is error free. If errors are found than the data package will be resubmitted to the analyst to resolve the issues, corrective actions implemented, complete additional documentation as required for reporting the errors and corrective actions implemented. The revised data package will be resubmitted to the Level 2 reviewer. Data review at Level 2 will be documented, and the reviewer will sign and date the appropriate page declaring the acceptance of the data package ready for the release.

The Laboratory Project Manager (LProgram Manager) or QA/QC Manager will perform the Level 3 review of the data package. This will be the final review before the data package is released to the client. At Level 3, the LProgram Manager or QA/QV Manager will review the data package reports, COCs and other supporting documents to ensure that the data meets the overall objectives and goals set out in the project specific DQOs and QAPP.

The documentation reviewed at Level 3 includes:

- Laboratory name and address included in all applicable documents
- Pertinent sample information (Laboratory and field sample ID numbers, time and date of sample collection, sample matrix, date of receipt of the sample, compliance with holding times, dates and times of sample analysis and the final project report).
- Analytical results reported (correct units and significant numbers)
- Correct reporting limits (based on dry weight correction, dilution factor, and interferences).
- Batch associated QC results (traceability to associate QCs to the batch and batch samples)
- Appropriate data qualifiers and interpretation of the qualifiers
- Batch narrative with discussion of any QC outliers, problems encountered and corrective actions implemented, factors affecting sample reporting limits and list of samples associated with the QC and the data package.

The data review will be based on the results of the QC elements, and the reviewer's professional experience, especially in interpreting the chromatograms, mass spectra and ICP-MS data. The professional experience of the reviewers combined with adherence to the QC protocols will ensure the data to be of high, defensible quality to meet the goals set in project DQOs and the requirements of the QAPP.

Non-compliant Data

The laboratory analyst will be primarily responsible to ensure that the QC data generated are compliant with the project requirements. The analyst will immediately notify the group leader if any of the QC samples (blanks, spikes, verification standards, duplicates, calibrations, and sample's QC recoveries) are out of control. The group leader will review the data and the project requirements to decide on corrective action to be implemented. He will, if necessary, consult with the QA/QC officer and/or the LProgram Manager to decide the course of action to be taken. The decisions to be taken will include if the samples should be reanalyzed, if only a selected number of the samples should be reanalyzed, or if the complete batch needs to be re-extracted and reanalyzed after implementing the required corrective action. Analysts will generate a QC non-compliant report with supporting documents and file it with the QA/QC manager. The project chemist will be informed as soon as possible if any non-compliant QC will result in unacceptable sample data.

2.5 Quality Control Requirements

Quality control requirements are introduced during project planning to ensure that the data generated are of known quality and will satisfy the DQO goals and the project requirements. How rigorous the quality control requirements are will be based on the goals set out in DQOs, QAPP and other Project Planning Documents. The following section will describe the Analytical Control Requirements.

2.5.1 Analytical Quality Control Requirements

Analytical quality controls monitor the analytical method performance, and it's adherence to the established limits of precision and accuracy. The analytical precision and accuracy are determined by the laboratory QC elements and the sample matrix. The laboratory QC elements measure the performance of the laboratory analytical procedures. The sample matrix element measures the impact of the matrix on the laboratory analytical procedures.

The performance of the analytical procedures is measured by analyzing method blanks and laboratory spikes. The impact of the matrix on the analytical procedures is measured by analyzing matrix spike, matrix spike duplicates, surrogates spike recoveries, laboratory sample duplicates, and post-digestion spike recoveries.

Field sampling performance and the impact of the environmental conditions during sampling events is measured by analyzing field quality control samples. The field quality control samples include field blanks, field duplicates, equipment rinsate and trip blanks which are for the assessment of the field sampling precision and accuracy.

2.5.2 Definitions of Terms

This section defines the QC elements that determine the precision and accuracy of sampling and analytical procedures. The mathematical expressions for the measurement of these QC elements will be defined in project specific QAPP and SAP, and are included in EPA SW-846 Methods and the laboratory SOPs. These expressions will also be included in the project specific QAPP and SAP. The following QC elements are defined under this section:

- Laboratory Batch
- Detection and Quantitation Limits
- Method Blank
- Instrument Blank
- Laboratory Control Sample
- Laboratory Control Sample Duplicate
- Matrix Spike and Matrix Spike Duplicate
- Laboratory Sample Duplicate
- Field Sample Duplicate
- Surrogate Compounds
- Internal Standards

Laboratory Batch

The discussion on the laboratory batch is included in this section because the QC elements listed are associated with a laboratory batch. The laboratory generates a) preparation batch and b) an analytical batch. The preparation batch is made up of twenty or less samples, including the associated QC samples, which are prepared at the same time and under the same conditions and using the same lots of reagents. Once the sample preparation process (e.g. extraction, digestion, filtration etc.) is initiated for a batch, the samples must be prepared as continuous process. A preparation batch must include samples of the same type of matrices (e.g. solids, aqueous, sludge, biological etc).

The analytical batch is made up of twenty or less samples, including the QC samples, which are to be analyzed for the same group of compounds. The analytical batch is analyzed under the same analytical sequence, instrument conditions and calibrations. A new analytical sequence must be started for a new batch.

For volatile organic compound (VOC) analysis by GC or GCMS, there is no distinction between sample preparation and analytical batches. The sample preparation and analysis of samples for VOC is a continuous process since sample preparation for purge and trap is a part of the analytical process. A batch for VOC analysis can be defined as a batch of twenty or less samples, including QC samples, analyzed under a single GCMS calibration and MS tuning.

Initially, a preparation batch must be analyzed together at the same time within the same analytical batch. The following action is required if any samples from the batch need to be reanalyzed with a different analytical batch or instrument (e.g. because a sample requires dilution, QC non-compliance or because of matrix interference etc):

- All samples in a preparation batch must be associated with the batch QC. Corrective actions resulting from QC outliers must apply to all the associated batch samples. Any sample from a batch scheduled for reanalysis based on dilution, sample QC recoveries, matrix interference etc. must be associated with the original preparation batch QC. For example, the sample reanalysis sequence must include the reanalysis of the corresponding method blank prepared with the batch. The LCS/LCSD and MS/MSD need not be reanalyzed on another instrument if the initial analysis met the acceptance criteria as per the method, SOP or the QAPP.
- The instrument used for the reanalysis must satisfy the instrument QC criteria for the method.
- Instrument blank must be analyzed to demonstrate that the system is contamination free.
- Any single or multiple instruments used for a specific project must meet all the QC criteria that the primary instrument must meet.

Detection and Quantitation Limits

Concentration of an analyte in an environmental sample is expressed using four different terms, two related to detection limits and two related to reporting limits.

Instrument Detection Limit (IDL)

Instrument detection limit applies to metals analysis. The IDL is determined by replicate analysis of certified standards over several days. The concentration data is statistically treated to derive the

standard deviation of the replicate analysis. This represents the instrument response to the analyte concentration. The IDL is set at a response level of 3X the standard deviation. IDL must be determined for all instruments used for metals analysis. IDL is independent of method detection limit, and is determined by analyzing pure standards directly without any preparation process.

Method Detection Limit

The Method Detection Limit (MDL) is defined as ‘the minimum concentration of an analyte in a matrix that can be measured and reported with 99% confidence that the analyte concentration is greater than zero’. The MDL for an analyte is dependent upon the analytical method, sample matrix and the instrument’s response to the analyte. The MDL for a specific matrix is determined by the analysis of replicate aliquots of the matrix, each aliquot spiked with the same concentration of the analyte of interest, and processing all replicates through the sample and extraction preparation procedures. Ideally, a minimum of seven replicates are prepared and analyzed. The results are statistically treated to determine standard deviation. The laboratory will set the MDL for a given analyte at 3 times the standard deviation. The MDL will be greater than the IDL.

Practical Quantitation Limit

The Practical Quantitation Limit (PQL) is normally set at 3 to 5 times the MDL based on reagent water as matrix. PQL is defined as the minimum concentration that can be reliably achieved within the confines of the accuracy and precision during the routine analytical operations. The PQL is analyte and matrix dependent, and can be less or more than the PQL set for the reagent water. EPA SW-846 Methods provide PQL for all the analytes for guidance only. They may not be achieved at all times. PQL will be greater than MDL.

Contract Required Quantitation Limit

The Contract Required Quantitation Limit (CRQL) is the contract approved PQL. The laboratory will demonstrate the PQL that are achieved for the target analytes which are then reviewed by the regulators. The approved PQLs are defined as CRQL following the approval and acceptance process by the regulators. Generally, the quantitation limits for MB are set at MDL, since the MB is based on reagent grade or deionized water which is free of matrix effect. The sample quantitation limits are based on PQL. The field sample target analytes with concentrations found between MDL and PQL ($> \text{MDL}$ but $< \text{PQL}$) that meet all criteria for qualitative identification are routinely reported as estimated because of the lower degree of accuracy and precision.

The MDL and PQL for project specific analytes will be tabulated in project specific SAP and QAPP.

Method Blank

Possibilities exist in the laboratory environment that contamination and interferences may get introduced into the field sample during the analytical procedures performed. The sources of contamination and interferences are improperly cleaned glassware, reagents, extraction procedures, contaminated analytical systems, and especially for VOC the general laboratory environment where solvents like methylene chloride and acetone are regularly used. Method blanks are used to monitor the contaminations and interferences. Method blank is analyte free matrix that is processed with reagents, extractions, concentration and analysis just like a field sample. Analytical protocols require that a method blank is included with each batch of field sample. Certain procedures in inorganic analysis do not have sample preparation steps. An instrument blank which contains all reagents used for the analysis is equivalent to method blank.

Instrument Blank

Instrument blank is used to confirm that the analytical system is contamination free. Instrument blank is a solvent or an acid solution used to prepare calibration standards. Instrument blank is not required if the analytical batch includes a MB. Instrument blank and MB both may be analyzed to isolate the source of a contamination or interference problem. Routinely, instrument blanks are required for inorganic analysis only. Instrument blanks are analyzed at a frequency of one for every 10 samples for metals analysis.

Surrogate Compounds

Surrogate compounds are used to monitor the total laboratory performance of chromatographic and mass spectrometric analytical process. Surrogates also help to monitor the impact of the sample matrix on the analysis. Surrogates should meet the following characteristics:

- Should be a non-target compound
- Should not interfere with the required analysis (e.g. should not coelute with any target compound)
- The properties should be similar to the target compounds so that they behave similar to the target compounds during the analytical procedures such as sample preparation and extraction
- Should exhibit similar instrument response to the target analytes
- Readily available

Surrogates are added to each QC and the field sample prior to the start of the sample preparation procedures. The percent recovery is used to monitor the matrix effect and the analytical procedures. Re-analysis of the sample extract or re-extraction of the sample may be required to reconfirm the non-compliant surrogate recoveries. The percent recoveries must be within the method specific laboratory and EPA Methods SW-846 SOPs. It is possible that the laboratory establishes its own range of the surrogate recoveries. The range is based on data collected over a period of a few months. The upper and lower recovery limits are than statistically determined for each surrogate.

Internal Standards

Internal standards are used to determine the concentrations of target analytes by chromatographic and mass spectrometric methods. The internal standards have properties similar to the surrogates, but are added to the sample extracts just prior to the instrumental analysis. The internal standards compensate for the variability of injection of the extracts into the analytical instruments. Mass spectrometric methods use isotopically labeled internal standards, and the recovery ranges are evaluated based on the chromatographic peak areas.

Laboratory Control Samples and Laboratory Control Duplicate Sample

Laboratory Control Sample (LCS) is used to monitor laboratory's total efficiency independent of any matrix interference. LCS will monitor the efficiency of the procedures established by the laboratory, and the analyst's proficiency in performing analytical operations he or she has been assigned. LCS is made up of matrices that are free of environmental target analytes. Reagent water is used for aqueous LCS sample, and inert material like sodium sulfate or other certified and approved media is used for solid LCS sample. The LCS is spiked with known concentrations of known target analytes. The spiked samples are taken through the entire analytical process from sample preparation to analysis, just like a field sample. The recovery of the spiked analytes will

measure the accuracy and the precision of the laboratory analytical procedures and the analyst's efficiency in performing the procedures. The laboratory is required to include LCS and Laboratory Control Sample Duplicate (LCSD) with each batch. Together with MS/MSD sample, the LCS/LCSD can help determine if there are any matrix effects in the analysis.

Matrix Spike and Matrix Spike Duplicate

MS and MSD analysis is used to determine the effect of the matrix on the analysis. Three separate samples are collected from a selected location at the site at the same time. Two of the aliquots are used for MS and MSD analysis, the third aliquot is analyzed unmodified in any way as a regular field sample. The MS and MSD samples are spiked with known concentrations of known analytes prior to sample preparation. The precision and accuracy of the analytical procedure is determined by the recovery of the spiked analytes. The recoveries of the spiked analytes in MS and MSD are determined by subtracting the concentration of the target analyte in the unspiked sample (if detected) from the concentration of the analyte from the MS and MSD. The concentration of the analyte in MS and MSD will represent the concentration detected in the unspiked sample plus the amount spiked. The accuracy and the precision of the analytical procedure is determined based on the recoveries of the target analytes.

Laboratory Duplicate Samples

Laboratory duplicate sample is used to determine matrix specific precision of the analytical procedures. For laboratory duplicate analysis, the laboratory will analyze two aliquots of the same sample from the same sample container and prepared at the same time. The precision is determined by determining by calculating the difference between the two results and dividing the difference by the average of the two measurements.

2.5.3 Laboratory Batch Quality Control Logic

Each sample preparation and analytical batch will be associated to a set of QC samples prepared and analyzed with the samples in the same batch and of the same matrix. This section discusses the functions of the batch QC and possible corrective actions for the non-compliant QC elements.

Required batch analytical QC elements will be included in the project specific SAP and QAPP for project specific methods. MB and LCS/LCSD will determine the efficiency of the batch preparation. Project specific MS and MSD will be used to monitor any matrix effects, systematic trends or errors representative of the batch. Samples used for MS/MSD analysis will be project specific samples, designated in the field at the time of the sampling. The MS/MSD samples will be representative of the batch and decisions based on MS/MSD will be applicable to the specific batch. Surrogate recoveries and individual analyte recoveries will be reviewed for matrix effect. Recoveries below the lower QC limits will indicate a negative bias in the recoveries due to the matrix effect. The laboratory introduced contamination during batch preparation is monitored by MB. One MB will be analyzed with each batch. Corrective action will be initiated when contamination is found in the MB. The corrective action may include re-analysis of the blank and the samples, re-preparation of the batch including the QC samples. The goal will be to have a MB free of any contamination but this may not always be achievable especially in MB for VOC analysis. Methylene chloride, acetone, and 2-butanone (methyl ethyl ketone) are the most common contaminants likely to be detected in the VOC MB. Phthalate esters may be present in MB analyzed for semi-volatile organic compounds.

Method Blank

MB should be free of any target analytes. EPA SW-846 Methods states that MB should not have

any target analytes detected above MDL.

For instances where contamination in MB is detected, the first step of corrective action will be to evaluate the impact on the samples. Presence of contamination in MB will not automatically lead to re-analysis or re-preparation of the batch. If the analyte detected in the MB is not detected in any of the QC or field sample than no batch corrective action will be required. The appropriate corrective action taken will be to investigate, identify and eliminate the source of the contamination in the MB and documenting the findings for future reference. If the analyte found in the MB is a target analyte and is also detected in the sample then the quantitative results will be reviewed and evaluated. If the concentration of the analyte in the sample is found to be X5 or greater than the concentration found in the blank, than the data will be accepted with 'B' qualification to indicate that the analyte was detected in the MB.

Blank subtractions will not be allowed. If the MB detects a contamination that does not satisfy the acceptance criteria, than the MB and all associated samples will be re-extracted and re-analyzed

The Project Chemist will be informed immediately if the MB fails to meet the required acceptance criteria.

Laboratory Control Sample/Laboratory Control Sample Duplicate

LCS and LCSD will be used to monitor the precision and accuracy of the total analytical operations, from sample preparation to extract analysis. For organic analysis the LCS/LCSD will be spiked with known concentrations of representative target analytes. For metals analysis, the LCS/LCSD will be spiked with all target analytes. The recoveries of the spikes analytes should be within the established limits. For the organic LCS/LCSD, if only representative spike list used than all spiked analyte recoveries will be required to be within the limits established for the batch to be acceptable. When a full list id used for spiking, the recoveries of spiked analytes that fall within the established limits will be accepted for the batch. Corrective action will be initiated for the analytes that are non-compliant.

The corrective action process will be first initiated by evaluating the impact of the non-compliant LCS/LCSD recoveries on the sample. For example, if the recoveries of the spiked analytes are above the upper acceptable limit, and if the samples do not have any detectable target analytes and other QC elements (e.g. surrogate recoveries) are in control, than no further action may be required. Otherwise re-extraction and reanalysis of the batch will be required. Additional corrective action will include identifying the cause if non-compliance, resolving the problem and documenting the evaluation and corrective action implemented. A common cause of non-compliant recoveries is double spiking. By an oversight, the analyst may have spiked the LCS/LCSD sample twice. This would be evident from the consistently high recoveries for all spiked analytes, introducing a systematic error. The batch, in this situation, will be rejected and complete batch re-extracted and reanalyzed. When the recoveries of the spiked analytes are below the lower limit, the complete batch with the associated QC samples will be re-extracted and reanalyzed following the corrective action and resolution of the problems.

Some analyses will not require MS/MSD, for example if the method uses isotopically labeled surrogates and internal standards. In such cases the batch precision and accuracy will be determined by LCS and LCSD.

Matrix Spike and Matrix Spike Duplicate

Impact of matrix on precision and accuracy will be determined by analyzing a set of MS/MSD

MARRS Chemical Data Quality Management Plan

with each batch for organic analysis. For inorganic analysis, one MS sample and a set of LCS/LCSD will be analyzed with each sample batch. Accuracy will be determined by calculating the percent recovery of the spiked analyte. Precision will be evaluated by calculating the relative percent difference between recoveries of duplicate samples. The calculated precision and accuracy must be within the acceptance limits given in the method or the laboratory established values stated in the laboratory copy of SOP.

Corrective action will be implemented when the MS/MSD results are out of control. Evaluation of MS/MSD and the impact on sample batch is more complex than the other QC, because in addition to monitoring the preparatory and analytical efficiency, they also evaluate the effect of the matrix such as homogeneity and possible presence of interfering compounds.

If the MS/MSD fails to meet the accuracy or precision criteria, the extracts will be re-analyzed once if there is no significant, non-target analyte interference is present. Only the results from re-analysis will be reported if acceptable results are obtained. If the re-analysis also fails to meet the precision and accuracy criteria, the MS/MSD will be re-extracted and re-analyzed once if enough sample is available and the holding time has not expired. If the repeat extraction and analysis fails to produce acceptable results, both the original analysis and the repeat results will be reported. This corrective action will also apply to laboratory duplicate samples.

The evaluation of the MS/MSD results will also be based on other QC elements e.g. surrogate recoveries, MB, LCS/LCSD, the chromatograms and mass spectra, and the physical examination of the sample and the extracts for abnormalities. If all the QC elements are in control except for MS/MSD, then it may be concluded that the abnormality of the MS/MSD is matrix based. The evaluation and the corrective actions implemented for non-compliant MS/MSD results will be documented.

Evaluation of matrix spikes for inorganic analysis is more complex. The analyst may use other procedures to ascertain the presence of interference in a sample. For example, the analyst may perform serial dilutions of the extract until the percent recoveries of the spikes are within acceptable limits. The analyst may also do a post-digestion spike of the original, unspiked sample followed by additional corrective actions such as serial dilutions.

The analyst will evaluate the post-digestion spike recoveries based on the following recovery limits:

1. The %R post-spike recovery is within 85% to 115%, the sample result is <PQL or \geq PQL: the results are reported without qualification.
2. The %R of the post-spike is between 115 % and 150%, the result <PQL: the result is reported without qualification
3. The %R of the post-spike is between 115% and 150%, the result is \geq PQL: Dilute the extract and re-analyze. Use the method of standard addition for quantitation
4. The %R of the post-spike is \geq 150%, the result is <PQL: verify that there is no error in spiking. Report the result.
5. The %R of the post-spike is \geq 150%, the result is >PQL: Dilute the extract and re-analyze. Use the method of standard addition for quantitation
6. The %R of the post-spike is between 40% and 85%, the result is <0.5 x PQL: the result is reported as 'Non-Detect' at the OQL

MARRS Chemical Data Quality Management Plan

7. The %R of the post-spike is between 40% and 85%, the result is $\geq 0.5 \times$ PQL: Dilute the extract and re-analyze. Use the method of standard addition for quantitation.
8. The %R of the post-spike is $<40\%$, the result is $<PQL$ or $\geq PQL$: Dilute and analyze. Raise the reporting limit accordingly.

The recoveries of the labeled isotopes and internal standards may also provide information on possible matrix interferences.

Laboratory Batch Quality Control for Trip, Field and Equipment Blanks

The trip blank, field blank and the equipment blank (also called rinsate) are the field sampling QC elements.

Trip blanks are used to monitor potential contaminations that may affect field samples during collection, storage, transportation and shipment to laboratory. Trip blanks are primarily used for VOC samples, and are shipped with VOC containers to the field and back to the laboratory. Field blanks are used to monitor potential background contamination in the field. Field blanks are created in the field, and shipped to the laboratory with the associated sample batch. Trip and field blanks are made up of analyte free reagent water, irrespective of the matrix of the samples. The blanks are associated with the samples in a batch and are analyzed in the same way as other field samples.

Equipment blanks, which consist of analyte free reagent water, are used to monitor the efficiency of the procedures used to decontaminate sampling equipment blanks. Equipment blank blanks are associated with soil and water non-disposable sampling equipment used in the field for sampling.

Field blanks will be associated with the samples collected at the same project site, and shipped to the laboratory with the samples.

The trip blank, field blank, and the equipment blank will be evaluated together with the laboratory method blank.

2.5.4 Laboratory Data Completeness

For a successful conclusion of a project, the measurement system should generate statistically acceptable quantity of data of valid and known quality. This requirement is expressed as Completeness of the project.

Completeness

Completeness is the ratio of amount valid data from a measurement system compared to the amount expected to be obtained under normal conditions.

Data completeness will measure the extent to which the data base resulting from measurement systems fulfill the project objectives set out in the project DQO and the QAPP. The target of completeness will be project specific, but will not be less than 90 percent for the analytical parameters, and will be 100 percent for sample holding times and preservation. Completeness of a project will be expressed as a percentage of number of valid data requested:

$$C\% = S/R (100\%)$$

Where:

MARRS Chemical Data Quality Management Plan

C% = Completeness

S = number of validated successful analysis

R = Number of requested Analysis

For successful analysis, the samples should arrive at the laboratory intact, on-time, properly preserved, with enough quantity to perform the analysis requested, and accompanied by completed, error free chain of custody. The samples must be analyzed with attention so that the QC elements and goals as set out in DQO and QAPP are met.

Factors that may hinder achieving targeted data completeness include, but not limited to:

- Loss of sample due to leaking or broken sample containers
- Insufficient or compromised information in a COC
- Insufficient quantity of sample to perform the initial analysis, perform repeat analysis or dilutions
- Preservation requirements compromised, such as temperature.
- Holding times compromised, especially for those parameters with short holding times
- Failure to implement proper corrective actions, such as dilutions, re-extraction etc.
- Catastrophic breakdown of an analytical instrument with no back up system available

It is inevitable that at some points errors will happen either in field or laboratory procedures despite following the sampling and analysis plans. The laboratory will strive to achieve 100 % completeness, or project specific completeness but the minimum acceptable level of completeness will be 90 % of all project specific methods with in-control, acceptable QC.

2.6 Instrument Calibration and Frequency

All analytical instruments will be calibrated at frequency defined in EPA SW-846 Methods and analytical SOPs, and as briefly discussed in this section.

2.6.1 Calibration Standards

Instruments are calibrated to accurately quantify an analytical parameter with high level of confidence. The accuracy of the quantitation of a target analyte is dependent on the accuracy of the instrument calibration, which in turn is controlled by the accuracy of the standards. For this purpose, the laboratory will ensure that the standards used for instrument calibration are of the highest quality reference standards, accompanied by Certificate of Analysis, and procured from a reputable vendor of environmental standards accredited by National Institute of Standards and Technology (NIST) for inorganic standards, and by American Association of Accreditation (A2LA) for the organic standards. An accurate record for the tracking of the standards will be maintained by the laboratory. The records will include the name of the standards, vendor's name and address, accreditation, the date the standard was received, the concentration of the standard, the standards' batch and lot numbers, Certificate of Analysis and MSDS documents, expiration date, and the initials of the person who received the standards at the laboratory. All the information regarding the standards will be recorded in the laboratory LIMS system for tracking purpose.

The primary standards obtained from the vendors are used to prepare working standards by the laboratory analysts. The laboratory may also purchase pre-prepared working standards directly

from the vendors. In either case, all pertinent information about the working standards will be recorded in working standards log-book. Following information will be included:

- Primary standard lot and batch number, vendor's name, analyte name and concentration, the date primary standard opened
- For the working standard: analyst's name, date, concentration of the diluted working standard, volume of primary standard diluted and volume of solvent added, date the working standard validated against 2nd source, and the expiry date.
- Method of storage

The primary and working standards will be labeled with an identification system for tracking purposes. The label will also show the date the working standard was prepared, analyst's initials, analytes and concentration, vendor's name and expiry date. All standards will be stored in a refrigerator away from the field samples and extracts. The working standards will be stored in their respective laboratory and the primary standards will be stored in a separate refrigerator. Both storage facilities will be accessible to authorized personnel only.

All standards and organic solvents used for preparing the working standards will be checked for purity by chromatographic analysis under the same conditions as the corresponding method or SOP. The standards will be periodically physically examined for the presence of precipitation, turbidity, color change etc. that would be indicative of deterioration. The affected standard will be discarded, entry made in the log-book giving the reason for discarding the standard, and the analyst will prepare a new working standard.

2.6.2 Calibration

Calibrations are method specific. Accurate calibrations of measuring systems are essential for generating accurate and reliable data.

The instrument calibration will establish the dynamic range of the instrument, establish linear range of response and response factors, and establish the instrument sensitivity. Calibrations and the resulting response factors are used for the quantitation of the analyte concentration in a given sample. The calibration will establish upper and lower limits of calibrations where the instruments' response is linear. If the analyte response falls outside these limits, the concentrations generated by the system will be inaccurate. Corrective actions than will be required to ensure that the response falls within the established calibration range and the data reported are accurate.

All the pertinent information for the calibrations will be recorded in the instrument log-books. The initial calibration's day to day validity will be determined by analyzing either a calibration verification standard or a continuing calibration standard as required by the method. The initial calibration will be considered to be in control when the verification or the continuing calibrations, analyzed with reference to the initial calibration, meet the QC limits set in the method or the project specific SAP and the QAPP.

2.6.2.1 Organic Methods Calibration

Organic methods are primarily based on chromatography. Different methods based on chromatography use different chromatographic detectors. Organic method calibrations are method specific, and are discussed here in general terms. Method specific calibration procedures will be found in the corresponding method, and project specific QAPP and SAP.

MARRS Chemical Data Quality Management Plan

ICAL is based on the analysis of minimum of five calibration standards of varying concentrations and computing the average response factor. It is not uncommon that an ICAL is analyzed only once every 6 months under normal circumstances. A new ICAL is performed whenever a new capillary column is installed, the GC undergoes maintenance or continuing calibration fails repeatedly in a row. Initial calibration verification (ICV) is analyzed prior to daily sample analysis. The ICV is analyzed at the mid-point concentration of the initial calibration to verify that the ICAL is valid. The ICAL is valid when the response factor of the ICV falls within the defined acceptance limits. The continuing calibration (CCAL) is analyzed with the samples at a predetermined frequency. The function of the CCAL is to validate the ICAL throughout the sample analysis. The ICAL is valid when the response factor of the CCAL falls within the defined QAPP and SAP limits.

The sample quantitation will be based on validated ICAL. An ICAL can be constructed using internal or external calibration standards. The external calibration standard method is based on the instrument response to the straight reference standards and the target analyte's response from the sample. The analyte's response from the sample is compared with the response of the reference standards' response from an ICAL. ICAL is the calibration curve constructed by plotting the reference standard concentration versus the instrument response. This method of quantitation is primarily applied to GC analysis with non-mass spectrometric detectors. The internal standard method of quantitation is primarily applicable to GCMS. The calibration is based on the addition of an internal standard to all the reference standards used for calibration. The response is determined as a ratio of the response of the internal standard and the corresponding sample analyte. The internal standard is added to the final sample extract just prior to the injection into the GCMS. Both the methods should meet the required QC criteria as defined in the corresponding methods, SOPs, and project specific QAPP and SAP.

Gas Chromatography

EPA SW-846 Methods 8015B, 8021B, 8081A, 8082, 8141A, 8151A, and 8310 are based on capillary GC with non-MS detectors. Corresponding ICALs are based on certified reference standards. Primarily, GC with non-MS detectors are calibrated with external standards, but internal standard calibration method also can be used.

Prior to establishing an initial calibration curve, the operating conditions of the GC are optimized, and an instrument blank is analyzed to confirm that the GC system is free of contamination. A new ICAL is performed whenever a new capillary column is installed, the GC undergoes maintenance or ICV/CCV calibrations fail repeatedly in a row.

The concentration range of the calibration standards used will be decided by the responses of individual target analytes and the expected concentrations in the field samples, probably based on field screening results or the historical data. The lowest calibration limit (LCL) should be at or close to PQL. The concentration of the upper calibration limit should be such that the response falls within the linear range of the curve. The sample extract will require dilution if the analyte response exceeds the UCL.

The ICAL is used to determine calibration factor (CF) for each target analyte. The CF of each analyte is used to calculate the concentration of the analyte in the field sample. The response of each target analyte is represented by the area of the discrete peak the analyte generates in GC analysis. A number of target analytes generate complex, multiple peaks, such as chlordane, toxaphene, gasoline, and diesel. For these analytes, total area is summed up for calculating the sample concentration. Similarly, the ICALs for these analytes will be based on sum of the total

area.

The instrument response must be linear within the concentration range of interest. The linearity is evaluated by the correlation coefficient (r) or the percent relative standard deviation (%RSD). Linearity through the origin is assumed when the %RSD of the calibration curve is <20% and the average CF can be used. The correlation coefficient (r) must be ≥ 0.995 to be acceptable for the quantitation of the target analytes.

The ICAL will be validated daily by analyzing ICV. The percent difference (%D) between the mean CF of the ICAL and the CF of the ICV must be +/- 15 percent to be acceptable. The validity of the ICAL during the analysis is monitored by analyzing CCAL, at a frequency stated in the method, SAP and QAPP, as a part of sample analytical sequence. The percent difference (%D) between the mean CF of the ICAL and the CF of the CCV must be +/- 15 percent to be acceptable. The analyst will be responsible for evaluating all calibration related QC. It will be the analyst's responsibility to identify and resolve the problem if any QC element is non-compliant.

Gas Chromatography/Mass Spectrometry

EPA SW-846 Methods 8260, 8270, and 8280 use Mass Spectrometer (MS) as a detector. MS require calibrations to ensure that the mass analyzer calibration and the electron multiplier response to a target analyte. A MS is optimized for operation by process called 'tuning'. MS tuning is performed to optimize the MS performance for the required analysis. Bromofluorobenzene (BFB) is used to tune the MS for volatile compound analysis. Decafluorotriphenylphosphine (DFTPP) is used for the semivolatiles compounds. The MS must meet the corresponding method specific tuning criteria prior to calibration and the analysis of samples can begin. The MS must be retuned if any of the specified criteria are not met.

Initial calibration of GCMS is performed when the instrument is initially installed, when any continuing calibration fails, or following a major maintenance of the MS is undertaken. ICAL for a GCMS system is based on the analysis of 5 calibration standards encompassing the concentrations of interest, with the lowest standard being close to the MDL of the target analyte. Each calibration standard is made up of all the method specific target analytes and the internal standards. A relative response factor (RF) is calculated for each analyte, at each level of concentration, relative to the associated internal standard.

The ICAL must be evaluated for conformance once every 12 hours. The performance is evaluated by checking the response of certain key analytes that monitor system sensitivity and system linearity. System Performance Calibration Compound (SPCC) monitors the system sensitivity and Calibration Check Compounds (CCC) monitors the calibration linearity. The SPCC compounds must meet a method specified average RF, and the CCC compounds must meet method specified %RSD criteria. Non-compliance will result in identifying and resolving the problem that has caused the non-compliance.

Once the MS tune and ICAL are found to be acceptable, the sample analysis can begin. During the sample analysis phase, a tuning standard and calibration standards must be analyzed every 12 hours to monitor the sensitivity and the linearity of the GCMS system using Continuing Calibration Verification (CCV) standard at a concentration near the mid-point concentration of the calibration standards. The CCV is checked for the responses of SPCC and CCC compounds against the ICAL. Corrective action must be implemented if either SPCC or CCC compounds are found to be non-compliant and prior to continuing further analysis.

MARRS Chemical Data Quality Management Plan

The GCMS methods use internal standard for calibration, quantitation and qualitative identification of sample analytes. The internal standard must elute within +/- 30 seconds of the previous CCAL and the peak areas must be within a factor of two compared to the previous CCAL.

Corrective actions must be implemented and any problems affecting the analysis resolved before continuing the sample analysis.

Samples with high concentration may contaminate the chromatographic system. The contamination may 'carry over' to the next sample in the sequence. The laboratory will take all precautions to eliminate such carryover. Routine corrective action will be to flush the system with an organic solvent to remove the contamination before further analysis is continued. It may become necessary to reanalyze the samples which follow the sample with high concentration of an analyte in the analytical sequence

2.6.2.2 Metals Methods Calibration

Graphite Furnace Atomic Absorption (GFAA) and Inductively Coupled Plasma Emission Spectroscopy are the two primary analytical methods used for metals analysis. The methods use initial calibration (ICAL), Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV) and Continuing Calibration Blanks.

Inductively Coupled Plasma

Inductively Coupled Plasma (ICP) ICAL curve is based on one blank and a minimum of three certified calibration standards. Alternatively, the ICP is standardized with a blank and one standard. Following the standardization, a solution spiked with analytes at contract limiting concentration (CRI) at PQL and a high calibration standard are analyzed. The response of CRI at PQL must be +/- 50 % of the expected PQL and for high standard response must be +/- 5 % of the expected value. Once calibrated, the ICV and CCV from a different source than calibration standards are analyzed to verify the calibration. The ICV and CCV solutions are made up of known target analytes at known concentrations. The analyzed concentrations must fall within +/- 15 % of the known concentrations. The sample analysis must be performed within the daily established linear range based on the high standard.

CCV and CCB are used to monitor the ICP calibration during the analysis as per the method, and project specific SAP and QAPP requirements. The analysis will be stopped whenever either of these two QC elements fails to meet the required criteria. The analysis will be resumed after corrective actions have been taken to resolve the problem. In the analytical sample analysis sequence, the samples must be bracketed with CCV and CCB which meet the established criteria. The corrective action will be the reanalysis of all samples impacted by non-compliant CCB or CCV.

An inter-element check standard is analyzed at the beginning and end of the sample analytical run. This standard is run to ensure that the inter-element and background correction factors have remained constant through the analysis. Non-compliance to the established criteria will require reanalysis of the affected samples.

Inductively Coupled Plasma/Mass Spectrometry

Inductively Coupled Plasma/Mass Spectrometry (ICP-MS) method for metals analysis uses a MS as detector. Prior to analysis, the MS is tuned to optimize the analytical performance of the system. MS is checked for tuning parameters, mass calibration and mass resolution on daily basis.

MARRS Chemical Data Quality Management Plan

The tune and calibration is performed using the criteria specified in the method. Sample analysis will not begin if these operations do not meet the specified criteria.

The ICP-MS calibration is based on minimum of two standards, a blank and a high concentration calibration standard. The blank and the standard are fortified with internal standards which can be used to monitor and correct for matrix interference. The interferences are automatically corrected for by the computer software, and are verified by interference check standard analyzed every 12 hours to monitor the isobaric interferences. The CCV and CCB are used to monitor the ICP calibration during the analysis as per the method, and project specific SAP and QAPP requirements. The analysis will be stopped whenever either of these two QC elements fails to meet the required criteria. The analysis will be resumed after corrective actions have been taken to resolve the problem. In the analytical sample analysis sequence, the samples will be bracketed with CCV and CCB which meet the established criteria. The corrective action will be the reanalysis of all samples impacted by non-compliant CCB or CCV.

The instrument will be flushed with rinse solution following the analysis of a sample with very high concentration to prevent any carry over to the next sample.

Atomic Absorption

Atomic Absorption Spectrophotometer (AA) is calibrated before the sample analysis is started. The AA is calibrated using a blank and a minimum of three calibration standards, followed by analyzing ICV to verify the calibration curve. The CCV and CCB are used to monitor the AA calibration during the analysis as per the method, and project specific SAP and QAPP requirements. The analysis will be stopped whenever either of these two QC elements fails to meet the required criteria. The analysis will be resumed after corrective actions have been taken to resolve the problem. In the analytical sample analysis sequence, the samples must be bracketed with CCV and CCB which meet the established criteria. The corrective action will be the reanalysis of all samples impacted by non-compliant CCB or CCV.

For GFAA, all samples are spiked with appropriate standards post-digestion to monitor the matrix effect and effects from interferences. The method of standard addition or serial dilution is used when matrix interferences are evident. Chemical modifiers are added to the digestate to reduce the matrix interferences.

2.6.2.3 Wet Chemistry and Other Methods Calibrations

Wet chemistry and other related methods require use of a number of different analytical instruments and wet chemistry. Examples are pH meters, balances, conductivity meters, field monitoring systems, and gravimetric analysis. All these methods require calibration. Each system needs to be calibrated prior to sample analysis. For each method, the calibration needs to be monitored during sample analysis. The initial calibration ranges will be method specific, and monitored at a frequency specified by the method. Non-compliance will require suspension of the analysis, application of corrective action to resolve the problem, and recalibration and reanalysis of the samples. The decisions for reanalysis and recalibration will be taken in consultations with the group leader and/or project manager. These methods do not require CCV or ICV to monitor the calibrations.

2.6.2.4 Analytical Calibrations and Results Calculations

All analytical instruments are calibrated to compute the final concentration of a target analyte in a sample. The calibrations must be as accurate as possible so that the final result quantitation is of known quality with high level of confidence that the result is accurate and can be used to make

environmental decisions. The ICAL and other calibrations must meet specific, statistically derived QC limits to be acceptable.

This section describes the mathematical formulae to compute Calibration Factors (CF), Response Factors (RF), and Relative Response Factors and QC limits for QC parameters. The calibration will be based on the use of external and internal standards.

2.6.2.4.1 Calibration Calculations

External Standards

Calibrations based on external standards are primarily used for GC with non-MS detectors.

External standards are pure, certified standards corresponding to each analyte of interest. The calibration standards will be prepared at various concentrations to reflect the expected range of concentrations in the sample and the dynamic range of the instrument. The lowest calibration level (LCL) will be at X3 to X5 of the MDL determined for the analyte. The upper calibration level (UCL) will be defined by the dynamic range of the instrument and will be such that the response is linear. Remaining of the standards will be prepared at concentrations between LCL and UCL.

To construct the calibration curve, each calibration standard at each concentration will be analyzed and instrument response recorded. For each analyte, a CF or RF is determined.

The CF is the ratio of the instrument response for the analyte (measured in area) and the concentration (Mass) of the standard injected in nanograms (ng):

$$CF = (A_s)/(M_s)$$

where:

A_s = Response of the standard (area)

M_s = Mass of the standard injected (ng)

The RF is the ratio of the standard concentration to its instrument response:

$$RF = (C_s)/(A_s)$$

where:

C_s = Concentration of the analyte in the standard (ng)

A_s = Response of the standard (area)

Internal Standard

Calibrations based on internal standards (IS) are used primarily for GC-MS methods. If necessary, internal standard based calibrations can be used for GC with non-MS detectors.

Internal standards are analogs of the target analytes that are not present in the samples, and exhibit similar chemical and analytical properties of the target analytes. Internal standards are added to each calibration standards, filed samples and QC samples.

The ICAL for each analyte is based on the ratio of the response to the internal standard and the corresponding target analyte at each calibration concentration. The ratio is defined as Relative

MARRS Chemical Data Quality Management Plan

Response Factor (RRF)

$$RRF = (A_s \times C_{is}) / (A_{is} \times C_s)$$

where:

A_s = Response of the calibration standard (area)

C_{is} = Concentration of the IS ($\mu\text{g/liter}$)

A_{is} = Area of the IS

C_s = Concentration of the calibration standard ($\mu\text{g/L}$)

A five point calibration curve is constructed for each target analyte. For each target analyte a RRF is determined at each concentration level and per cent Relative Standard Deviation (%RSD) is calculated:

$$\%RSD = (s/\text{mean of } x (100))$$

where:

mean of x = mean of the five RF of the analyte

s = standard deviation of RF of the analyte

Corrective actions will be implemented when %RSD for an analyte exceeds the method, SAP and QAPP specified upper limit. Some methods allow the use of linear regression type of calibration.

Linear regression is based on two factors, instrument response of an analyte and its concentration. The instrument response is a dependent variable (y) and the concentration of the standard is an independent variable (x). The regression will generate the slope and the intercept terms for a linear equation in the form:

$$y = ax + b$$

where:

y = Instrument response

a = Slope of the line (coefficient of x)

x = Concentration of the calibration standard

b = the intercept

The regression calculation will generate a coefficient correlation (R^2) which is a measure of "degree of fitness" of the regression line to the data. The degree of fitness is perfect when R^2 is 1.00. For quantitative purposes, the minimum acceptable R^2 must be 0.995 or greater.

A positive intercept of the slope indicates that there is some interference from the instrument that is a limiting factor in establishing the linearity. A negative intercept of the slope represents a threshold on lower limit of concentration. The positive intercept increases the probability of false positive if the instrument response of the analyte is 3 X less than the intercept value. If the intercept is negative, the results below the lowest calibration standard will be questionable.

The acceptance of a CCAL is determined by %D, which is the difference between the RF of the CCAL and the average RF of the ICAL for the analyte and is computed from the equation:

$$\%D = (RF_1 - RF_2) / 2 \times 100$$

Where:

$RF_1 = \text{Average RF of the standard analyte from the ICAL}$

$RF_2 = \text{RF of the standard from the CCAL}$

2.6.2.4.2 Result Quantitation

The concentration of the target analyte detected in a sample is calculated using CF, RF, or RRF depending on the analytical method. This section discusses the procedure for quantifying the concentration of detected target analyte in aqueous and soil samples.

Aqueous Sample

The concentration of each detected target analyte in aqueous sample is quantified using the following mathematical expression:

$$\text{Aqueous Concentration } (\mu\text{g/L}) = (A_x) (V_t) (D) / (CF) (V_i) (V_s)$$

where:

$A_x = \text{Response for the analyte detected (area)}$

$V_t = \text{Volume of extract injected } (\mu\text{L}). \text{ For purge \& Trap } V_t = 1$

$V_i = \text{Volume of total extract } (\mu\text{L}). \text{ For purge \& Trap } V_i = 1$

$V_s = \text{Volume of sample extracted or purged}$

$CF = \text{Calibration Factor}$

$D = \text{Dilution factor when applicable}$

Soil Samples

The concentration of each detected target analyte in soil sample is quantified using the following mathematical expression:

$$\text{Aqueous Concentration } (\mu\text{g/g}) = (A_x) (V_t) (D) / (CF) (V_i) (W) (P)$$

where:

$A_x = \text{Response for the analyte detected (area)}$

$V_t = \text{Volume of extract injected } (\mu\text{L}). \text{ For purge \& Trap } V_t = 1$

$V_i = \text{Volume of total extract } (\mu\text{L}). \text{ For purge \& Trap } V_i = 1$

$CF = \text{Calibration Factor}$

$D = \text{Dilution factor when applicable}$

$W = \text{Weight of the sample extracted or purged}$

$P = \text{Percent dry weight of the sample } (W/100) \text{ or } 1 \text{ for wet-weight}$

Quantitation of target analyte detected in soil or aqueous sample, and based on RF of the calibration curve uses the following mathematical expression:

$$\text{Concentration } (\mu\text{g/L or } \mu\text{g/g}) = (\text{mean RF}) (\text{area of analyte response}) (\text{dilution Factor})$$

Quantitation of target analyte detected in soil or aqueous sample analyzed by GC-MS, and based on internal standards and RRF of the calibration curve uses the following mathematical expression:

$$\text{Aqueous concentration } (\mu\text{g/L}) = (A_x) (C_{is}) (D) / (A_{is}) (RRF) (V_s)$$

where:

A_x = Response for the characteristic quantitation ion of the target analyte (area)
 C_{is} = Concentration of the internal standard injected (ng)
 A_{is} = Response for the characteristic quantitation ion of the internal standard (area)
 RRF = Relative response factor from initial calibration
 V_s = Volume of the aqueous sample purged (consider any dilutions made)
 D = Dilution factor when applicable

$$\text{Soil Concentration } (\mu\text{g/g}) = (A_x) (C_{is}) (D) / (A_{is}) (RRF) (W) (P)$$

where:

A_x = Response for the characteristic quantitation ion of the target analyte (area)
 C_{is} = Concentration of the internal standard injected (ng)
 A_{is} = Response for the characteristic quantitation ion of the internal standard (area)
 RRF = Relative response factor from initial calibration
 W = Weight of sample extracted or purged
 D = Dilution factor when applicable
 P = Percent dry weight of the sample ($W/100$) or 1 for wet-weight

When using regression analysis to calculate the concentration, the equation is rearranged to solve for the concentration (x):

$$x = (y-b) / a$$

where:

x = Concentration
 y = Instrument response
 b = y-intercept
 a = Slope of the line (Coefficient of 'x')

2.7 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

Data acquisition is not limited to acquiring quantitative data from laboratory or field testing only. Assimilation of regulations and rulings of federal, state, and local authorities, historical background on the project site, methodologies available for various tasks, existing data for a specific region or site, and data generated on specific wastes, landfills, materials or chemical compound of interest also fall under this category. This section briefly outlines information required for the data acquisition that will be used for final decision making.

Any of the following information may be assimilated for the data acquisition process:

- Applicable federal, state, and local rulings and regulations
- Site specific Historical background
 - Future plans for the site
 - Site requirements and schedules
- Technology and methods available for:
 - Laboratory testing
 - Geophysical investigations, monitoring, and sampling
 - Volume reduction, isolation and disposal of radioactive/hazardous

MARRS Chemical Data Quality Management Plan

- Materials
 - Statistical analysis and design
- Assimilation of available site specific data
 - Demographical
 - Geological
 - Hydrological/meteorological
 - Geochemical
 - Past, present, and future development
 - Type, volume, and extent of contamination
 - Physical distribution and lay out of existing and past facilities
- Available data on wastes, materials and/or chemical compounds of concern
 - Processing facilities
 - Physical/chemical characteristics of chemical compounds of interest
 - Geochemical and radiological
 - Toxicity
 - Human/environmental risk
 - Remedial technology and treatability

Various sources may be used to assimilate the data acquisition information listed above:

- Regulations, guidance, rulings, standards published by the government and professional institutes
- Academic textbooks
- Geological, geophysical maps
- Past reports, guidance documents, and manuals published by federal, state, local and professional institutes and organizations
- Personnel interviews
- Aerial and satellite photographic and imagery
- Procurement and waste disposal documents

The assimilation of all the information will be well documented. The documentation process will include:

- Source of the information
- Name of the person interviewed
- Name of the publishing author, media in which published, date and the year of publication
- The identity of the federal, state or local authority when used as an information source
- Reference to the website (if internet used as information assimilation source)

Trip reports, minutes of meetings, telephone call logs, hard copy prints of e-mails and any other mode of project specific communication will be documented and included with the project file. Any amendments, addition or deletion of information relating to the site specific communication will be dated and signed by the responsible person. Verbal communications will be confirmed in writing.

The process of required data acquisition will be oriented to meet the goals set out in the DQO.

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3.0 ASSESSMENT OVERSIGHT

3.1 QUALITY CONTROL

A Quality Control Program (QC) will be implemented to allow Naval Facilities Engineering Command (NAVFAC) to evaluate the performance of a project. The QC program will allow NAVFAC to ensure that the project activities are performed in accordance to the approved SAP, Quality Assurance Project Plan (QAPP), and the project Health and Safety Plan (HASP).

The QC program will be based on three QC elements:

- Preparatory phase
- Initial phase
- Follow-up phase

The Program Manager will be responsible for the implementation of the QC program. The H&S manager will be responsible for implementing the HASP program. He/she will periodically visit the sites for H&S inspection to ensure compliance to the approved H&S plan.

3.1.1 Definable Features of Work

A feature of work is definable when its task is different and unique from other work tasks, and has its own QC elements.

Following are the examples of task with definable features:

- Field sampling activities
- On-site analysis using field laboratory
- Off-site analysis using off-site, fixed laboratory
- Data management
- Risk assessment
- Remedial action

The QC program based on the three phase QC elements will be implemented for each feature of work listed above. The procedures that will be used for implementing each phase QC control are described below:

3.1.2 Preparatory Phase

This phase of the three phase QC program will be implemented prior to any task related to site activity is initiated. The Contracting Officer (CO) will be delivered a preparatory phase out-line of the preparatory phase activities. The check-list inspection will address the following prior to beginning work at the site:

- The Project Manager will ensure that QAPP, SAP, and CDQMP have been approved
- The H&S Manager will ensure that site specific approved HASP plan available
- Ensure that all parties concerned have reviewed QAPP, SAP, , and HASP
- Will ensure that all required permits, clearances, and authorizations are in place

MARRS Chemical Data Quality Management Plan

- Will ensure that everyone involved in performing the assigned tasks have received required training
- H&S Manager will ensure that medical monitoring program, emergency response program safety training have been adequately provided to everyone
- Will ensure that all team members are thoroughly briefed about the tasks to be performed
- Will ensure that all equipment related to sampling and field analysis is available, calibrated and in acceptable operational conditions
- Will ensure that all required preliminary work has been completed in accordance with the criteria set out in QC documents
- Will ensure that all materials and information as per H&S Plan is available on site, including emergency procedures, location and telephone numbers of the nearest hospital etc
- Ensure that all requirements for on-site sample storage, shipping containers and supplies, and courier services have been provided for
- Ensure that appropriate chain-of-communication has been set-up
- Ensure that daily briefings will be conducted by the field team leader and H&S Manager prior to the start of the day's field activities

3.1.3 Initial Phase

The initial phase inspection will be conducted when a certain part of the initial task has been completed. This inspection will ensure that the task completed so far has been in accordance with SAP, QAPP and the WP. The H&S Manager, at this point, will audit the site to ensure that the tasks are performed within the regulation set out in HASP. Any discrepancies will be resolved at this stage of the inspection, and corrective actions documented. The Team Leader will review the corrective actions taken and inform the Project Manager as appropriate.

3.1.4 Follow-up Phase

The follow-up inspections will be performed at a frequency of one or more depending on the length of the project. The goal of this inspection will be to ensure that all task related activities are performed in compliance with project QC requirements. Issues of non-compliance will be resolved by the Field Team Leader in consultation with the Project Manager, followed by another inspection to ensure that the non-compliant issues have been resolved.

3.1.5 Completion Inspection

A completion inspection will be performed at the conclusion of all the site specific tasks. The completion of the project will be reviewed and the project elements that do not confirm to the project requirements will be identified. Corrective actions will be implemented. A follow-up inspection will be performed by Program Manager to ensure that the issues identified have been resolved with appropriate corrective actions. A final completion inspection will be performed just prior to the closure of the site specific activities.

3.2 ASSESSMENT AND RESPONSE ACTION

The project assessment will be performed to ensure adherence to SAP and QAPP requirements. The process will include the review of readiness of the field teams and laboratories to perform the

assigned tasks, system audits to ensure adherence to QC protocols, and surveillance to review a specific activities to ensure adherence to established criteria to fulfill the project goals.

3.2.1 Readiness Review

Readiness review primarily will be performed to assess the readiness of the field team to perform all tasks associated with field investigations. This function will review field supply procurement and availability plans, contingency plans, sample management and shipping plans, training of the project team, site clearance, and ensure that H&S Plan is in-place and readily accessible to all parties concerned.

Readiness review will be conducted jointly by the Field Team Leader and the H&S Manager prior to mobilization. The review will be in a form of checklist specific to the project field activities. Any deficiencies will be communicated to the Project Manager prior to mobilization. Corrective actions will be implemented to address the deficiencies prior to the field team mobilization.

The readiness review will also ensure that analytical facilities and services required for the project are in place prior to mobilization. The contracted laboratory will be informed of the project schedule, and when the first batch of samples will be shipped. The laboratory will be informed ahead of time if sample delivery is expected to be made on a weekend.

3.2.2 System Audit

System audit is the process of evaluating all aspects of the project activities performed to achieve a desired goal. The system audit will be performed to evaluate the field sampling activities and laboratories contracted to perform the analytical tasks.

The field sampling audit will be performed as directed by the Project Manager. The audit function will evaluate the field sampling activities to ensure that the procedures used are according to those established in SAP and QAPP. This is especially critical because SAP will be dynamic in nature and may be amended, with necessary approvals, during the life of the site specific project to meet new unexpected challenges.

System audits of the contracted laboratories will be performed at various stages of the project activities. The suitability of the contracted laboratory for a project will be evaluated prior to field team mobilization and sampling activities are started. The initial evaluation will be to ensure that the contracted laboratory has been NAVFAC and NELAC certified, and can meet the analytical requirements of the project. The initial evaluation will be followed by on-site audit of the laboratory. The laboratory audits will be performed by the primary contractor at the direction of NAVFAC, or by the auditors assigned by NAVFAC.

The on-site laboratory audit will ensure that all procedures to support analytical needs of the specific project are established prior to the first samples received by the laboratory. The audit will include the review of sample receiving, logging and tracking procedures; review of the analytical procedures and SOPs, laboratory quality management program, inventory of consumables, reference and other standards and reagents; availability of analytical instruments and back-up systems, QA/QC program; data management and review programs; procedures to address QC non-conformance and corrective action programs, format of reporting analytical results; and the training and experience of the analysts and support personnel to perform assigned tasks satisfactorily to meet the project requirements.

When directed by NAVFAC, the laboratory system audit will be conducted by the Project Chemist to verify the laboratory's ability to perform the analysis of the project field samples in accordance with the QA/QC criteria established in QAPP and SAP to meet the DQO. The audit process will be initiated first by the submittal of audit notification letter to the laboratory. This will be followed by scheduling the times and dates for the audit with the laboratory management, the activities that will be audited, and the laboratory personnel who should be available during the audit. An audit report will be generated at the conclusion of the audit and instances of non-compliance brought to the attention of the laboratory management. The laboratory management will resolve discrepancies by full written explanations of the issues or implementing corrective action. The final close-out audit report will be submitted to the Project Manager.

3.2.3 Surveillance

During the project's lifetime, surveillance audits will be performed to monitor specific field sampling and analytical activities to ensure adherence to the established protocols. The surveillance functions will be documented describing the specific activities that were reviewed, interviews with the project team members, conclusions on compliance to the established criteria, instances of non-compliance and corrective actions, and recommendations submitted for any other deficiencies.

3.2.4 Performance Evaluation Samples/Data Tracking Audits

In addition to conducting system audits and surveillance, the on-going laboratory performance will be evaluated by submitting performance evaluation (PE) and blind samples for analysis at frequency specified in the project specific QAPP. The PE and blind samples will be certified and procured from an NAVFAC approved vendor. The samples will be used for data tracking functions as well, from the point of receipt and logging the samples into the laboratory's data system to the final generation of the analytical results. The sample results will be reviewed by the Project Chemist against the certified values and QC limits as submitted by the vendors. Any deficiencies will be brought to the attention of the laboratory QA/QC Manager for resolution. The final PE and blind analysis report and evaluation will be submitted to the Project Manager.

3.2.5 The Nonconformance/Corrective Actions

Instances of nonconformance to the established QC criteria for all project related tasks will be documented. Maintenance of the documentation will be the responsibility of the Field Team Leader for field sampling activities, and the contracted laboratory QA/QC Manager for all laboratory related activities. Corrective actions will be applied to the nonconforming elements of the project. All pertinent information will be documented by the responsible party. The documented records of the nonconformance and the corrective actions taken will be communicated to the Program Manager. The Program Manager will review and file the documents for inclusion in the final project report if required.

The laboratory QA/QC Manager will make the documentation of nonconformance and corrective action available to the Project Chemist, when requested to do so.

3.3 Reports to Management

A project report for each defined work element will be submitted to the management that will discuss the project status and sampling and analytical activities at a frequency stated in the work plan. The reports will include the area of site sampled with sampling locations, number of samples collected and the type of matrices, non-conformance/corrective action reports (NCR/CAR), system audit and surveillance summaries, QA/QC summaries and future project

activity schedules with time frames. The reports will be submitted to the Program Manager within the time frames stated in the Work Plan.

3.3.1 Field Activities

The field team leader will provide a summary report of field activities, at a frequency established in the Work Plan, to the Project Manager or the Project Chemist as applicable. The summary will include site location, field laboratory activities, sampling activities, date and hours spent on activities, weather conditions, any difficulties encountered and the resolution of such difficulties.

3.3.2 Drilling Subcontractors

The drilling subcontractors will be responsible for submitting a summary of drilling activities to the Project Manager at a frequency established in the work plan. The summary will include the drilling activities in chronological order, hours spent on the project, difficulties encountered and resolution of the problems. The drilling subcontractors will inform the Field Team Leader and the Project Manager of any instances where an interruption in drilling activities will impact the project schedule (for example replacement for damaged drilling bits not available because the back up inventory got used up).

3.3.3 Subcontract Laboratory

Laboratories contracted to provide analytical services for NAVFAC programs will be required to be pre-qualified by NAVFAC by performing laboratory's system audit. The laboratory will address any concerns identified during the audit that may impact the performance of the project. A follow up audit will be performed to verify resolution of findings and observations and to review the corrective actions implemented. Laboratories found to be deficient in any aspect of the prequalification process will not be used for the program until the deficiencies are corrected and appropriate evidence of the resolution of the deficiencies is submitted to NAVFAC.

Laboratories pre-qualified within the past one year of the commencement of the program may not need to be re-qualified provided that the laboratory submits documentary evidence that the performance of the work done by the laboratory is acceptable to NAVFAC. The evidence could be in the form of the most recent report of the audit performed by another federal agency, records of PE sample analysis performed or other relevant documentation.

The contracted laboratory will provide summary reports of the tasks performed at a frequency established in the work plan. The summary will include the number of samples received, analyzed and reported, discrepancies noted, laboratory NCR/CAR, and the QC summary report. This summary report will be a separate document and will not replace the requirement for specific case narrative per SDG submitted with data packages.

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4.0 DATA VALIDATION AND USABILITY

The quality of the data generated for a project will determine the usability of the data. Function of data validation is to ensure that the data generated for a project are of known quality and legally defensible should a need arise to do so. This section of the CDQMP describes procedures used and information required for validating a data set.

4.1 DATA REVIEW, VALIDATION AND VERIFICATION

Critical environmental decisions are based on the on the data collected during project activities. To support the decision making process, it is important to ensure that the data is accurately reduced, validated and managed. The accuracy and quality of the data collection process will be evaluated through all measurements performed. The quality of the data will be determined by evaluating and assessing the QC requirements established in SAP and QAPP.

4.1.1 Field Sampling/Non-analytical Data

The field sampling activities, including drill boring activities, will be recorded in field log-books. All entries in the log-book will be dated and signed by the responsible field personnel. Any problems faced during the sampling activities will be recorded in the field log-book, with the measures taken to resolve the problem. Samples collected will be listed in the log-book with field ID numbers and cross-referenced with sample ID numbers in the COC. The log-book will be dated and signed at the conclusion of the day's sampling activities. The Field Team Leader will review the log-book on daily basis, and will sign and date the page of the log-book. Unused portions of the log-book pages will be crossed out with a single diagonal line.

The log-book will be included in the final data package submitted to the Project Manager.

4.1.2 Screening/Non-definitive Data

Screening data are generated in the field using field screening techniques to identify and differentiate overall contamination pattern at a given site. Screening data at best are qualitative and semi-quantitative, and do not generate definitive and quantitative data. The QC requirements for screening data are minimal but must be adhered to.

The Project Chemist will be responsible to review and verify the screening data based on the available QC information. At a minimum, the screening data must include instrument blank, system blank, initial calibration, instrument sensitivity to determine detection limit and initial calibration verification check. The acceptance criteria will be established in the SAP and QAPP. A pre-determined percent, routinely 10 %, splits of the samples subjected to field screening analysis will be submitted to a certified fixed laboratory for definitive analysis.

The Project Chemist will review the screening data for compliance with QC criteria established in SAP and QAPP. In addition, the quality of the screening data will be evaluated by comparison with definitive data from split sample analysis. A summarized review report of the QA/QC data and comparison data will be submitted to the Project Manager as a part of QC summary report for the project.

4.1.3 Definitive/Confirmatory Data

Definitive and confirmatory data will be generated by a certified, NAVFAC approved off-site fixed laboratory using approved analytical methods and stringent QC criteria established in SAP and QAPP. The data will be reviewed by the laboratory, NAVFAC or its designated contractor

Project Chemist, and an independent third party contractor.

Data verification will be performed on 90 percent of the results generated. Full Level IV data validation will be performed by a third party on a minimum of 10 percent of the results generated. Additional data validation will be performed if deemed necessary by the Project Chemist or Project Manager.

4.2 Validation and Verification Methods

Data validation and verification will be performed at various levels within the full range of the project tasks. Program work plan, project-specific documents, and plans will provide specific details of the personnel responsible for verification and validation activities involved with data management.

4.2.1 Data Verification

Data verification is the process of evaluating the correctness, completeness, and conformance/compliance of a specific data set against the method, procedural, or contractual requirements. The verification will be performed by examination and provision of objective evidence, through appropriate documentation, that the project/contract specific requirements have been fulfilled. The following data verification will be performed on the complete set of analytical data to ensure that the established requirements have been satisfied:

- All documented records of sample tracking and COC
- The organic data will be reviewed for holding times, blank analysis performance, LCS, MS/MSD, surrogate recoveries, and method and instrument calibrations
- The inorganic data will be reviewed for holding times, blank analysis performance, pre-digestion matrix spike, sample duplicates and LCS
- Analytical results will be qualified based on data validation decisions in accordance with the flagging convention as will be defined in the data package narratives submitted by the laboratory and the third party data validator

The initial data verification will be performed at laboratory level by the laboratory analyst or the data reviewer, and the Project Chemist. This will be an on-going process applicable to each data package generated. The data will be thoroughly reviewed to ensure project/contractual QC compliance, calculations, QC recoveries, instrument performance, and completeness of the documentation. Any problems encountered during analysis and instances of noncompliance will be documented, including corrective actions implemented, and will be part of the case narrative submitted. Calculations will be checked for any variation. Source of the unexplainable variations will be determined, corrected, documented, and submitted with the project data package narrative. The laboratory's Project Manager will then review the data thoroughly for QC compliance and completeness, and generate a case narrative for the data package. The data package will then be for final printing according to the format described in the project Work Plan and the QAPP. The final, printed data package will be submitted to the laboratory QA/QC manager who will verify 100 % of the data for project compliance, QC performance and conformance of the data to the project data quality objective.

The Project Chemist will be notified of any problems that may impact the project activities. Data verification by the laboratory will be performed at various levels to ensure that the quality of the analytical data is in accordance with the criteria established in SAP and QAPP to satisfy the goals set in DQO.

MARRS Chemical Data Quality Management Plan

Level 1 Review

The laboratory generated data is thoroughly reviewed for accuracy and completeness. This process will begin at the bench level and is called Level 1 Review. The analyst will be responsible to ensure the completeness and accuracy of the data he/she generates and reduce the data following the established protocols in the applicable SOPs. The analyst will review the completed data package to ensure that:

- Sample tracking from preparation, extraction to analysis is correct and complete with required dates and signatures
- The tasks are performed according to applicable SOPs
- Analytical results are correct and complete. Calculations are accurate and based on the procedures described in applicable SOPs and QAPP.
- QC samples are analyzed at required frequencies and are within the established QC limits
- Any deviations from the established protocols are documented with explanations
- Unique sample preparation and analytical requirements are met
- Supporting documentation is complete and accurate

Level 2 Review

The Level 2 laboratory review provides an independent review of the data package and will be performed either by the Group Leader or the data review specialist.

The Level 2 Review will ensure that:

- All calibration data are as per required by the method, analyzed at the required frequency, meet the criteria as set out in the SOP, SAP and the QAPP, and are accurately documented
- QC samples are in control
- Qualitative identification of a detected target analyte is accurate
- Quantitation of the target analyte is accurate
- Correct dilution factors have been used when applicable
- Any deviations from the established protocols are documented with explanations
- Unique sample preparation and analytical requirements are met
- Supporting documentation is complete and accurate
- All documentation required is present and complete
- The data are correct and valid for incorporation with the final report
- The data package is complete for data archiving and ready for final review by QA/QC Manager prior to submittal to the client

Level 2 Data Review includes review of all calibrations and QC recoveries. At a minimum, 10 % of the data will be back reviewed to bench sheets and sample logging in the data base. Complete data package will be reviewed if problems are encountered with samples in 10 % review. At a satisfactory conclusion of the review, the reviewer will date and sign the review tracking

document, data entered in the data base, case narrative prepared and the package will be further reviewed by the QA/QC manager for final review. The QA/QC Manager will authorize the release of the data to the client.

The laboratory will qualify non-compliant data with conventional data qualifying system. The data qualifiers will be defined in the laboratory generated case narrative. The detection limits reported will be based on the sample type and the level of interference associated with the sample matrix. Results from diluted analysis will be reported at a higher detection limit, by multiplying the detection limit with the dilution factor. Every effort will be made to identify the source of any anomalous results, including possible problems with sampling procedures, sample preparation and analytical procedures. The results for the impacted samples will be reported with appropriate qualifier. The impact of the problem on the data will be estimated or determined. Failure of the data to meet the DQO goals will be thoroughly reviewed to identify possible sources of problems. The samples will be reanalyzed if deemed necessary, and with consultation with QA/QC Manager and the Project Manager.

The final data released by the laboratory will be submitted to the Project Chemist for review and to ensure that the data provided satisfies the project and DQO requirements.

4.2.2 Data Validation

Data validation is an analyte and sample specific process that extends the evaluation of the data evaluation beyond the procedural or contractual compliance (performed under data review) to determine the analytical quality of a specific set of data. Data validation will focus on evaluating the data to ensure that project specific data needs as stated in project-specific documents are fulfilled.

A third party, independent of the laboratory or data user, will perform the data validation process. The data validation criteria will be based upon the measurement quality (acceptance criteria) developed in the QAPP and the goals developed in DQO.

Level III validation will be performed on definitive analysis performed for each method of analysis using “Functional Guidelines for Evaluating Organic Analysis (US EPA)” January 2005) and “Functional Guidelines for Evaluating Inorganic Analysis (US EPA October 2004)”. Full validation at Level IV will be performed for a minimum of 10 percent of the data for each test method. Qualified data will be flagged using the convention stated in the guidelines.

Data validation at Level III will be less intense compared to Level IV data validation process. Level III data validation, the level at which 90 percent of the data will be validated, will include the evaluation of case narratives, chain-of-custody, holding times, instrument run-logs, summary results and QC recoveries, calibrations, MS tuning, blanks, MS/MSD, LCS/LCSD and field blanks. Level IV data validation, performed on 10 percent of the sample data, will include all elements covered in Level III validation and sample calculations, random calculations of CF or RRF, review of chromatograms and MS spectra, and QC recovery calculations.

The data validation report will be submitted to the Project Chemist.

4.2.3 Data Validation Summary Report

The summary report will include the analytical criteria reviewed for each analytical method and qualifiers applied. The non-compliant surrogate recoveries will be identified.

The organic data will be reviewed for sample preparation and analysis holding times, calibrations, blanks, surrogates, internal standards, matrix spike, matrix spike duplicate, laboratory duplicates, ICP interference check, ICP serial dilution, post-digestion spikes, and MS tuning for VOC and SVOC. Second column confirmation data, when applicable, will be reviewed. The third party data validator will discuss all these elements in the data validation narrative, and tabulate non-compliant parameters with the qualifiers and the QC element on which the non-compliant qualification is based. Definitions of data qualifiers will be included in the summary data validation report.

4.2.4 Data Usability

When applicable, analytical data will be qualified as a result of non-compliance determined during data validation. The qualified data will be flagged according to the convention stated in the Functional Guidelines. The usability of qualified data will be based on the action levels established for the project and applicable, relevant and appropriate requirements (ARAR's). Data qualified as rejected due to gross non-compliance (such as improper sample identification or insufficient sample for the intended analysis) will be determined to be not usable for the intended purpose(s).

4.3 RECONCILIATION WITH DATA QUALITY OBJECTIVES

The validated data will be subjected to Data Quality Assessment (DQA). The report on DQA will present the evaluation of the entire data collection program and document the successful completion of the DQOs. The DQA will include relevant documentation of all reviews performed of project activities during data acquisition, validity of the data and recommendations for data use. The DQA report will include:

- Summary of Project DQOs
- Summary of Field QC operations
- Summary of laboratory QC operations
- Statistical summaries of precision, accuracy, representativeness, and completeness of the off-site, definitive data
- Summaries of non-compliant elements and impact on DQO
- Recommendations for data use.

DQA will provide documentation that the data acquisition program collected sufficient quantity and quality of data to accomplish the goals set for the project.

4.3.1 Analytical/Statistical Control Parameters

The quality of the measurement data will be evaluated to assess the quality of the data. The assessment will be based on accuracy, precision, completeness and representativeness.

Accuracy/Precision

Accuracy is the agreement between the measured and true value. Precision is the degree of variability in the agreement.

Accuracy of the analytical method, and the accuracy achieved by the analyst using the method is determined by applying the process of spike analysis. A known concentration of an analyte is spiked into an appropriate matrix and subjected to the analytical method. The result of the recovered spiked analyte from the spiked sample analysis is compared against the known concentration spiked into the sample. The percent recovery (%R) of the spiked analyte is the parameter for accuracy. The requirement for accuracy (%R) will be defined in the project specific

MARRS Chemical Data Quality Management Plan

QAPP and SAP and will be set at a value to meet the DQO goals. Percent recovery will be calculated using the following mathematical expression:

$$\%R = (C_1 - C_2) (100) \% / C_3$$

where:

%R = Percent recovery

C₁ = Total concentration of analyte in spiked sample

C₂ = Concentration of analyte in un-spiked sample

C₃ = Concentration of spike added

Precision of the analytical method, and the precision achieved by the analyst using the method is determined by performing replicate analysis for target analyte. The results generated by the replicate analysis are used to determine the relative percent difference (RPD) which is the measure for precision. The RPD will be calculated using the following mathematical expression:

$$RPD = \frac{X_1 - X_2}{(X_1 + X_2)/2} (100\%)$$

where:

RPD = Relative Percent Difference

X₁, X₂ = values of sample 1 and sample 2

Completeness

Completeness is the measure of the quantity of statistically accepted valid measurements that should be available to prevent misinterpretation and ambiguity, and to answer important decision making questions. Project specific data completeness objective will be 90 percent. The completeness for the holding times will be 100 percent. This means that minimum of 90 percent of the project specific measurement data will be adequate in quality to be used for final decision making, and that all samples will be analyzed within their holding time requirements. If, for any reason, initial analysis of a sample is not performed within the established holding time, the sample will be re-sampled and reanalyzed.

Representativeness

Representativeness is a qualitative parameter that reflects the extent to which a given sample is characteristic of a given population at a specific location or under a given environmental condition. To ensure that the collected samples are representative of the given population, the sampling locations will be designed based on statistics and sound logistics, and adequate number of samples will be collected using appropriate and established sampling techniques. Field duplicate samples will be collected to evaluate variations at a sampling point. To further ensure representativeness of sampling process, attention will be paid to the selection of the sampling sites, drilling sites and depths, and analytical parameters.

Comparability

Comparability is the extent to which comparisons among different measurements of the same quantity and quality will yield valid conclusions. Comparability amongst field measurements will be achieved by using standard procedures, standard field data sheets, and uniform measurement and concentration units. To ensure comparability in the field standardized field procedures and standard operating procedures will be adhered to. Laboratory data comparability will be achieved by use of established and approved analytical methods and procedures and consistency in reporting units based on consistent analytical basis (such as wet/dry weight and volume).

Sensitivity

Quantitative measurements at low concentrations and with acceptable level of confidence are technically a challenging problem. With low level measurements, the probability of reporting false positive or false negative increases significantly with significant consequences. Laboratory will report quantitative results above established reporting limits as definitive and below reporting limits as Not Detected (ND). Some projects may require that the results below the reporting limits should also be reported. Such results will be reported with qualification as "estimated results" by the laboratory.

Quantitation Limit is the minimum concentration of an analyte that can be measured and reported with 99 percent confidence that the value reported is above zero. The actual quantitation limit will be determined by the sensitivity of the analytical system used, and the matrix of the sample.

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