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Guidance for Preparing Standard Operating Procedures (SOPs)

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FOREWORD

The U.S. Environmental Protection (EPA) Agency has developed an Agency-wide program of quality assurance for environmental data. EPA's Quality System requires documentation of both management and technical activities. This guidance document, *Guidance for Preparing Standard Operating Procedures (SOPs)*, provides a standardized working tool that can be used to document routine quality system management and technical activities.

This document is one of the *U.S. Environmental Protection Agency Quality System Series* documents. These documents describe the EPA policies and procedures for planning, implementing, and assessing the effectiveness of the Quality System. As required by EPA Manual 5360 A1 (May 2000), this document is valid for a period of up to five years from the official date of publication. After five years, this document will be reissued without change, revised, or withdrawn from the *U.S. Environmental Protection Agency Quality System Series* documents. Questions regarding this document or other *Quality System Series* documents should be directed to the Quality Staff at:

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GUIDANCE FOR PREPARING STANDARD OPERATING PROCEDURES

1. INTRODUCTION

1.1 Overview

A Standard Operating Procedure (SOP) is a set of written instructions that document a routine or repetitive activity followed by an organization. The development and use of SOPs are an integral part of a successful quality system as it provides individuals with the information to perform a job properly, and facilitates consistency in the quality and integrity of a product or end-result. SOPs describe both technical and administrative operational elements of an organization that would be managed under a work plan or a Quality Assurance (QA) Project Plan [*EPA Requirements for QA Project Plans (QA/R-5)* (EPA 2001a), or Chapter 5 of the *EPA Quality Manual for Environmental Programs, EPA Manual 5360 A1*] and under an organization's Quality Management Plan [*EPA Requirements for Quality Management Plans (QA/R-2)* (EPA 2001b), or Chapter 3 of the EPA Quality Manual]. This document is designed to provide guidance in the preparation and use of an SOP within a quality system.

1.2 Purpose

SOPs detail the work processes that are to be conducted or followed within an organization. They document the way activities are to be performed to facilitate consistent conformance to technical and quality system requirements and to support data quality. SOPs are intended to be specific to the organization or facility whose activities are described and assist that organization to maintain their quality control and quality assurance processes and ensure compliance with governmental regulations.

If not written correctly, SOPs are of limited value. In addition, the best written SOPs will fail if they are not followed. Therefore, the use of SOPs needs to be reviewed and re-enforced by management, preferably the direct supervisor. Current copies of the SOPs also need to be readily accessible for reference in the work areas of those individuals actually performing the activity, either in hard copy or electronic format, otherwise SOPs serve little purpose.

1.3 Benefits

The development and use of SOPs promotes quality through consistent implementation of a process or procedure within the organization, even if there are temporary or permanent personnel changes. SOPs can be used as a part of a personnel training program, since they should provide detailed work instructions. It minimizes opportunities for miscommunication. When historical data are being evaluated for current use, SOPs can also be valuable for reconstructing project activities when no other references are available. In addition, SOPs are frequently used as checklists by inspectors when auditing procedures. Ultimately, the benefits of a valid SOP are reduced work effort, along with improved data comparability, credibility, and legal defensibility.

SOPs are needed even when published methods are being utilized. For example, if a SOP is written for a standard analytical method, the SOP should specify the procedures to be followed in greater detail than appear in the published method. It also should detail how, if at all, the SOP differs from the standard method and any options that this organization follows. As noted in ASTM D5172-91 (1999), *Standard Guide for Documenting the Standard Operating Procedures Used for the Analysis of Water,* "A significant part of the variability of results generated by different laboratories analyzing the same samples and citing the same general reference is due to differences in the way the analytical test methods and procedures are actually performed in each laboratory. These differences are often caused by the slight changes or adjustments allowed by the general reference, but that can affect the final results." Using a correctly well-written SOP can minimize such differences.

1.4 Writing Styles

SOPs should be written in a concise, step-by-step, easy-to-read format. The information presented should be unambiguous and not overly complicated. The active voice and present verb tense should be used. The term "you" should not be used, but implied. The document should not be wordy, redundant, or overly lengthy.

2. SOP PROCESS

2.1 SOP Preparation

The organization should have a procedure in place for determining what procedures or processes need to be documented. Those SOPs should then be written by individuals knowledgeable with the activity and the organization's internal structure. These individuals are essentially subject-matter experts who actually perform the work or use the process. A team approach can be followed, especially for multi-tasked processes where the experiences of a number of individuals are critical, which also promotes “buy-in” from potential users of the SOP.

SOPs should be written with sufficient detail so that someone with limited experience with or knowledge of the procedure, but with a basic understanding, can successfully reproduce the procedure when unsupervised. The experience requirement for performing an activity should be noted in the section on personnel qualifications. For example, if a basic chemistry or biological course experience or additional training is required that requirement should be indicated.

2.2 SOP Review and Approval

SOPs should be reviewed (that is, validated) by one or more individuals with appropriate training and experience with the process. It is especially helpful if the draft SOPs are actually tested by an individual other than the original writer before the SOPs are finalized.

The finalized SOPs should be approved as described in the organization’s Quality Management Plan. Generally the immediate supervisor, such as a section or branch chief, and the

organization's quality assurance officer review and approve each SOP. Signature approval indicates that a SOP has been both reviewed and approved by management. As per the Government Paperwork Elimination Act of 1998, use of electronic signatures, as well as electronic maintenance and submission, is an acceptable substitution for paper, when practical.

2.3 Frequency of Revisions and Reviews

SOPs need to remain current. Therefore, whenever procedures are changed, SOPs should be updated and re-approved. If desired, modify only the pertinent section of a SOP and indicate the change date/revision number for that section in the Table of Contents and the document control notation.

SOPs should be also systematically reviewed on a periodic basis to ensure that the policies and procedures remain current and appropriate, or to determine whether SOPs are even needed. The review date should be added to each SOP that has been reviewed. If a SOP describes a process that is no longer followed, it should be withdrawn from the current file and archived.

The review process should not be overly cumbersome or SOPs will never get reviewed. The frequency of review should be indicated by management in the organization's Quality Management Plan. That plan should also indicate the individual(s) responsible for ensuring that SOPs are current.

2.4 Checklists

Many activities use checklists to ensure that steps are followed in order. Checklists are also used to document completed actions. Any checklists or forms that are included as part of an activity should be referenced at the points in the procedure where they are to be used and then attached to the SOP.

In some cases, detailed checklists are prepared specifically for a given activity. In those cases, the SOP should describe, at least generally, how the checklist is to be prepared, or on what it is to be based. Copies of specific checklists should be then maintained in the file with the activity results and/or with the SOP.

Remember that the checklist is not the SOP, but a part of the SOP.

2.5 Document Control

Each organization should develop a numbering system to systematically identify and label their SOPs, and the document control should be described in its Quality Management Plan. Generally, each page of a SOP should have control documentation notation, similar to that illustrated below. A short title and identification (ID) number can serve as a reference designation. The revision number and date are very useful in identifying the SOP in use when

reviewing historical data and is critical when the need for evidentiary records is involved and when the activity is being reviewed. When the number of pages is indicated, the user can quickly check if the SOP is complete. Generally this type of document control notation is located in the upper right-hand corner of each document page following the title page.

Short Title/ID # Rev. #: Date: Page 1 of

2.6 SOP Document Tracking and Archival

The organization should maintain a master list of all SOPs, and this file should minimally include the date of the current version. The QA Manager (or designee) is generally the individual responsible for maintaining a file listing all current quality-related SOPs used within the organization. This list may be used when audits are being considered or when questions are raised as to practices being followed within the organization.

As noted above in Section 2.3, the Quality Management Plan should indicate the individual(s) responsible for assuring that only the current version is used. That plan should also designated where, and how, outdated versions are to be maintained or archived in a manner to prevent their continued use, as well as to be available for historical data review.

Electronic storage and retrieval mechanisms are usually easier to access than a hard-copy document format. For the user, electronic access can be limited to a read-only format, thereby protecting against unauthorized changes made to the document.

3. SOP GENERAL FORMAT

SOPs should be organized to ensure ease and efficiency in use and to be specific to the organization which develops it. There is no one "correct" format; and internal formatting will vary with each organization and with the type of SOP being written. A generalized format is discussed next.

3.1 Title Page

The first page or cover page of each SOP should contain the following information: a title that clearly identifies the activity or procedure, a SOP identification (ID) number, date of issue and/or revision, the name of the applicable agency, division, and/or branch to which this SOP applies, and the signatures and signature dates of those individuals who prepared and approved the SOP. Electronic signatures are acceptable for SOPs maintained on a computerized database.

3.2 Table of Contents

A Table of Contents is needed for quick reference for locating information and to denote changes or revisions made only to certain sections of a SOP.

3.3 Text

Well-written SOPs should first briefly describe the purpose of the work or process, including any regulatory information or standards that are appropriate to the SOP process. Define any specialized or unusual terms either in a separate definition section or in the appropriate discussion section. Then denote the sequential procedures to be followed, divided into significant sections; e.g., equipment needed, personnel qualifications, and safety considerations. Describe next all appropriate QA and quality control (QC) activities for that procedure, and list any cited or significant references.

As noted above, SOPs should be clearly worded so as to be readily understandable by a person knowledgeable with the general concept of the procedure, and the procedures should be written in a format that clearly describes the steps in order. Use of diagrams and flow charts help to break up long sections of text and to briefly summarize a series of steps for the reader.

Attach any appropriate information; e.g., a SOP may reference other SOPs. In such a case, the following should be included:

1. Cite the other SOP and attach a copy, or reference where it may be easily located.
2. If the referenced SOP is not to be followed exactly, the required modification should be specified in the SOP at the section where the other SOP is cited.

More information on text is contained in Section 4.1 for Technical SOPs and Section 4.2 for Administrative SOPs.

4. TYPES OF SOPs

SOPs may be written for any repetitive technical activity, as well as for any administrative procedure, that is being followed within an organization. General guidance for preparing both technical and administrative SOPs follows and examples of each are located in the Appendix.

4.1 Technical SOP Text Information Guidelines

Technical SOPs can be written for a wide variety of activities. Examples are SOPs instructing the user how to perform a specific analytical method to be followed in the laboratory or field (such as field testing using an immunoassay kit), or how to collect a sample in order to preserve the sample integrity and representativeness of the area of concern (such as collection of samples for future analysis of volatile organic compounds or trace metals), or how to conduct a bioassessment of a freshwater site. Technical SOPs are also needed to cover activities such as

data processing and evaluation (including verification and validation), modeling, risk assessment, and auditing of equipment operation.

Note that the Agency has prescribed a format for documenting environmental monitoring analytical methods entitled *Environmental Monitoring Management Council (EMMC) Methods Format* (see Appendix A). This methods format is sometimes confused with SOPs, perhaps because methods also include step-wise procedures that are to be followed by an analyst. However, this methods format contains information that is not essential to performing a repetitive technical activity, e.g., sections on method sensitivity, method performance, validation data, and pollution prevention.

Citing published methods in SOPs is not always acceptable, because cited published methods may not contain pertinent information for conducting the procedure-in-house. Technical SOPs need to include the specific steps aimed at initiating, coordinating, and recording and/or reporting the results of the activity, and should be tailored only to that activity. Technical SOPs should fit within the framework presented here, but this format can be modified, reduced, or expanded as required. Examples of technical SOPs describing a sampling activity and chemical and biological processes are located in the Appendices B, C, and D, respectively.

In general, technical SOPs will consist of five elements: Title page, Table of Contents, Procedures, Quality Assurance/Quality Control, and References:

1. Title Page - See Section 3.1.

2. Table of Contents - See Section 3.2.

3. Procedures - The following are topics that may be appropriate for inclusion in technical SOPs. Not all will apply to every procedure or work process being detailed.

    a. Scope & Applicability (describing the purpose of the process or procedure and any organizational or regulatory requirements),

    b. Summary of Method (briefly summarizing the procedure),

    c. Definitions (identifying any acronyms, abbreviations, or specialized terms used),

    d. Health & Safety Warnings (indicating operations that could result in personal injury or loss of life and explaining what will happen if the procedure is not followed or is followed incorrectly; listed here and at the critical steps in the procedure),

e. Cautions (indicating activities that could result in equipment damage, degradation of sample, or possible invalidation of results; listed here and at the critical steps in the procedure),

f. Interferences (describing any component of the process that may interfere with the accuracy of the final product),

g. Personnel Qualifications (denoting the minimal experience the SOP follower should have to complete the task satisfactorily, and citing any applicable requirements, like certification or "inherently governmental function"),

h. Equipment and Supplies (listing and specifying, where necessary, equipment, materials, reagents, chemical standards, and biological specimens),

i. Procedure (identifying all pertinent steps, in order, and materials need to accomplish the procedure such as:

- Instrument or Method Calibration and Standardization
- Sample Collection
- Sample Handling and Preservation
- Sample Preparation and Analysis (such as extraction, digestion, analysis, identification, and counting procedures)
- Troubleshooting
- Data Acquisition, Calculations & Data Reduction Requirements (such as listing any mathematical steps to be followed)
- Computer Hardware & Software (used to store field sampling records, manipulate analytical results, and/or report data), and

j. Data and Records Management (e.g., identifying any forms to be used, reports to be written, and data and record storage information).

4. Quality Control and Quality Assurance Section - QC activities are designed to allow self-verification of the quality and consistency of the work. Describe here the preparation of appropriate QC procedures (self-checks, such as calibrations, recounting, reidentification) and QC material (such as blanks - rinsate, trip, field, or method; replicates; splits; spikes; and performance evaluation samples) that are required to demonstrate successful performance of the method. Specific criteria for each should be included. Describe the frequency of required calibration and QC checks and discuss the rationale for decisions. Describe the limits/criteria for QC data/results and actions required when QC data exceed QC limits or appear in the warning zone. Describe the procedures for reporting QC data and results.

5. Reference Section - Documents or procedures that interface with the SOP should be fully referenced (including version), such as related SOPs, published literature, or methods manuals. Citations cannot substitute for the description of the method being followed in the organization. Attach any that are not readily available.

4.2 Administrative SOP Text Information Guidelines

As with the technical SOPs, administrative SOPs can be written for a wide variety of activities, e.g., reviewing documentation such as contracts, QA Project Plans and Quality Management Plans; inspecting (auditing) the work of others; determining organizational training needs; developing information on records maintenance; validating data packages; or describing office correspondence procedures. Administrative SOPs need to include a number of specific steps aimed at initiating the activity, coordinating the activity, and recording and/or reporting the results of the activity, tailored to that activity. For example, audit or assessment SOPs should specify the authority for the assessment, how auditees are to be selected, what will be done with the results, and who is responsible for corrective action. Administrative SOPs should fit within the framework presented here, but this format can be modified, reduced, or expanded. An example of administrative SOPs can be found in Appendix E.

In general, administrative SOPs will consist of five elements: Title page, Table of Contents, Procedures, Quality Assurance/Quality Control, and References.

1. Title Page - See Section 3.1.

2. Table of Contents - See Section 3.2.

3. Procedures - The following are topics that may be appropriate for inclusion in administrative SOPs:

    a. Purpose,
    b. Applicability (identifying when the procedure is to be followed),
    c. Summary,
    d. Definitions (defining any words, phrases, or acronyms having special meaning or application),
    e. Personnel Qualifications/Responsibilities (identifying any special qualifications users must have such as certification or training experience and/or any individual or positions having responsibility for the activity being described),
    f. Procedure,
    g. Criteria, checklists, or other standards that are to be applied during the procedure (such as citing this document as guidance for reviewing SOPs), and
    h. Records Management (specifically, e.g., as forms to be used and locations of files).

4. Quality Control and Quality Assurance Section - Describe any control steps and provisions for review or oversight prior to acceptance of the product or deliverable. This can include test plans such as verification and validation plans for software or running a "spell-check" program on the finished document.

5. Reference Section - Cite all references noted in the body of the SOP. A copy of any cited references not readily available should be attached to the SOP.

5. EXAMPLE SOPS

Appendices B-E contain examples of SOPs. These examples are not purported to be perfect or complete in content, nor is their use endorsed or recommended. They are provided merely to illustrate application of SOP format to technical and administrative subjects. They should not be cited or followed as actual procedure specification or guidance.

6. REFERENCES

ANSI/ASQC E4-1994, *Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs*. 1994. American Society for Quality. Milwaukee, Wisconsin.

ASTM D 5172-91 (1999), *Standard Guide for Documenting the Standard Operating Procedures Used for the Analysis of Water*. 2000. American Society for Testing and Materials. West Conshohocken, Pennsylvania.

Code of Federal Regulations. July 1, 1999. 40 CFR Part 160. Good Laboratory Practice Standards.

Escoe, Adrienne. 1997. *Nimble Documentation. The Practical Guide for World-Class Organizations.* Milwaukee, Wisconsin: American Society for Quality, Quality Press.

Garner, Willa Y. and Maureen S. Barge, editors, *"Good Laboratory Practices. An Agrochemical Perspective," ACS Symposium Series 369*, American Chemical Society.

U.S. Environmental Protection Agency, 2001a. *EPA Requirements for Quality Assurance Project Plans (QA/R-5)*, EPA/240/B-01/003, Office of Environmental Information.

U.S. Environmental Protection Agency, 2001b. *EPA Requirements for Quality Management Plans (QA/R-2)*, EPA/240/B-01/002, Office of Environmental Information.

U.S. Environmental Protection Agency, 2000. *EPA Quality Manual for Environmental Programs (EPA Manual 5360 A1).*

U.S. Environmental Protection Agency, 1997. *Manual for the Certification of Laboratories Analyzing Drinking Water. Criteria and Procedures/Quality Assurance*. EPA 815-B-97-001. Washington, DC.

APPENDIX A

Environmental Monitoring Management Council (EMMC) Methods Format

1.0 Scope and Application

Use a tabular format whenever possible for:

- Analyte list(s)
- Chemical Abstract Service (CAS) numbers
- Matrices
- Method Sensitivity (expressed as mass and as concentration with a specific sample size)

Include a list of analytes (by common name) and their CAS registry numbers, the matrices to which the method applies, a generic description of method sensitivity (expressed both as the mass of analyte that can be quantified and as the concentration for a specific sample volume or size), and the data quality objectives which the method is designed to meet. Much of this material may be presented in a tabular format.

2.0 Summary of Method

Sample volume requirements

- Extraction
- Digestion
- Concentration, and other preparation steps employed
- Analytical instrumentation and detector system(s), and
- Techniques used for quantitative determinations

Summarize the method in a few paragraphs. The purpose of the summary is to provide a succinct overview of the technique to aid the reviewer or data user in evaluating the method and the data. List sample volume, extraction, digestion, concentration, other preparation steps employed, the analytical instrumentation and detector system(s), and the techniques used for quantitative determinations.

3.0 Definitions of Method

Include the definitions of all method-specific terms here. For extensive lists of definitions, this section may simply refer to a glossary attached at the end of the method document.

4.0 Interferences

This section should discuss any known interferences, especially those that are specific to the performance-based method. If know interferences in the reference method are not interferences in the performance-based method, this should be clearly stated.

5.0 Safety

- Above and beyond good laboratory practices
- Disclaimer statement (look at ASTM disclaimer)
- Special precautions
- Specific toxicity of target analytes or reagents
- Not appropriate for general safety statements

This section should discuss only those safety issues specific to the method and beyond the scope of routine laboratory practices. Target analytes or reagents that pose specific toxicity or safety issues should be addressed in this section.

6.0 Equipment and Supplies

Use generic language wherever possible. However, for specific equipment such as GC (gas chromatograph) columns, do not assume equivalency of equipment that was not specifically evaluated, and clearly state what equipment and supplies were tested.

7.0 Reagents and Standards

Provide sufficient details on the concentration and preparation of reagents and standards to allow the work to be duplicated, but avoid lengthy discussions of common procedures.

8.0 Sample Collection, Preservation and Storage

- Provide information on sample collection, preservation, shipment, and storage conditions

- Holding times, if evaluated

If effects of holding time were specifically evaluated, provide reference to relevant data, otherwise, do not establish specific holding times.

9.0 Quality Control

Describe specific quality control steps, including such procedures as method blanks, laboratory control samples, QC check samples, instrument checks, etc., defining all terms in Section 3.0. Include frequencies for each such QC operation.

10.0 Calibration and Standardization

Discuss initial calibration procedures here. Indicate frequency of such calibrations, refer to performance specifications, and indicate corrective actions that must be taken when performance specifications are not met. This Section may also include procedures for calibration verification or continuing calibration, or these steps may be included in Section 11.0.

11.0 Procedure

Provide a general description of the sample processing and instrumental analysis steps. Discuss those steps that are essential to the process, and avoid unnecessarily restrictive instructions.

12.0 Data Analysis and Calculations

Describe qualitative and quantitative aspects of the method. List identification criteria used. Provide equations used to derive final sample results from typical instrument data. Provide discussion of estimating detection limits, if appropriate.

13.0 Method Performance

A precision/bias statement should be incorporated in the Section, including:

- detection limits
- source/limitations of data

Provide detailed description of method performance, including data on precision, bias, detection limits (including the method by which they were determined and matrices to which they apply), statistical procedures used to develop performance specification, etc. Where performance is tested relative to the reference method, provide a side-by-side comparison of performance versus reference method specifications.

14.0 Pollution Prevention

Describe aspects of this method that minimize or prevent pollution that may be attributable to the reference method.

15.0 Waste Management

Cite how waste and samples are minimized and properly disposed.

16.0 References

- Source documents
- Publications

17.0 Tables, Diagrams, Flowcharts and Validation Data

Additional information may be presented at the end of the method. Lengthy tables may be included here and referred to elsewhere in the text by number. Diagrams should only include new or unusual equipment or aspects of the method.

APPENDIX B

STANDARD OPERATING PROCEDURE FOR SURFACE WATER SAMPLING

DRAFT EXAMPLE - DO NOT QUOTE OR CITE

Prepared by: _____ Date:_____
Environmental Engineer

Reviewed by: _____ Date:_____
Monitoring Section Chief

Approved by: _____ Date: _____
Quality Assurance Officer

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION XI

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PROCEDURES

1.0 Scope & Application

1.1 This Standard Operating Procedure is applicable to the collection of representative aqueous samples from streams, rivers, lakes, ponds, lagoons, and surface impoundments. It includes samples collected from depth, as well as samples collected from the surface.

2.0 Summary of Method

2.1 Sampling situations vary widely and therefore no universal sampling procedure can be recommended. However, sampling of liquids from the above mentioned sources is generally accomplished through the use of one of the following samplers or techniques:

- Kemmerer bottle
- bacon bomb sampler
- dip sampler
- direct method

2.2 These sampling techniques will allow for the collection of representative samples from the majority of surface waters and impoundments encountered.

3.0 Health and Safety Warnings

3.1 When working with potentially hazardous materials, follow EPA, OSHA, and specific health and safety procedures.

3.2 When sampling lagoons or surface impoundments containing known or suspected hazardous substances, take adequate precautions. The sampling team member collecting the sample should not get too close to the edge of the impoundment, where bank failure may cause them to lose their balance. The person performing the sampling should be on a lifeline and be wearing adequate protective equipment.

3.3 When conducting sampling from a boat in an impoundment or flowing waters, follow appropriate boating safety procedures.

4.0 Interferences

4.1 There are two primary interferences or potential problems with surface water sampling. These include cross-contamination of samples and improper sample collection.

- Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Refer to SOP R11-200, Sampling Equipment Decontamination.

- Improper sample collection can involve using contaminated equipment, disturbance of the stream or impoundment substrate, and sampling in an obviously disturbed area.

4.2 Following proper decontamination procedures and minimizing disturbance of the sample site will eliminate these problems.

5.0 Personnel Qualifications

5.1 All field samplers are required to take the 40-hour health and safety training course and regular refresher courses prior to engaging in any field collection activities.

6.0 Equipment and Supplies

6.1 Equipment needed for collection of surface water samples includes:

- Kemmerer bottles*
- bacon bomb sampler*
- line and messengers
- sample bottle preservatives as specified by the analysis to be performed
- plastic zip-sealed bags
- ice
- cooler(s)
- chain of custody forms, field data sheets
- decontamination equipment and reagents (decontamination solutions are specified in SOP R11 #200, Sampling Equipment Decontamination)
- maps/plot plan
- safety equipment
- compass
- tape measure
- Global Positioning System (GPS)
- survey stakes, flags, or buoys and anchors
- camera and film
- logbook and waterproof pen
- sample bottle labels

- approved QA project plan
- approved field health and safety plan

* The appropriate sampling device must be of proper composition. Samplers constructed of glass, stainless steel, PVC or PFTE (Teflon) should be used based upon the analyses to be performed.

7.0 Sample Collection - Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and which equipment and supplies are needed.
2 Obtain necessary sampling and monitoring equipment.
3. Decontaminate or pre-clean equipment, and ensure that it is in working order.
4. Prepare scheduling and coordinate with staff, clients, and regulatory agency, if appropriate.
5. Perform a general site survey prior to site entry in accordance with the site-specific health and safety plan and QA Project Plan.
6. Use stakes, flags, or buoys to identify and mark all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

8.0 Sample Collection - Secondary Parameters

1. Water quality data should be collected in impoundments to determine if stratification is present. Measurements of dissolved oxygen, pH, and temperature can indicate if strata exist which would effect analytical results. Measurements should be collected at 1-meter intervals from the substrate to the surface using an appropriate instrument, such as a Hydrolab (or equivalent).

2. Water quality measurements such as dissolved oxygen, pH, temperature, conductivity, and oxidation-reduction potential can assist in the interpretation of analytical data and the selection of sampling sites, and depths anytime surface water samples are collected.

3. Generally, the deciding factors in the selection of a sampling device for sampling liquids in streams, rivers, lakes, ponds, lagoons, and surface impoundments are:

   Will the sample be collected from the shore or from a boat on the impoundment?
   What is the desired depth at which the sample is to be collected?

What is the overall depth and flow direction of the river or stream?

9.0 Sample Collection - Method Options

9.1 Kemmerer Bottle

A Kemmerer bottle may be used in most situations where site access is from a boat or structure such as a bridge or pier, and where samples at depth are required. Sampling procedures are as follows:

1. Using a properly decontaminated Kemmerer bottle, set the sampling device so that the sampling end pieces are pulled away from the sampling tube, allowing the substance to be sampled to pass through this tube.

2. Lower the pre-set sampling device to the pre-determined depth. Avoid bottom disturbance.

3. When the Kemmerer bottle is at the required depth, send down the messenger, closing the sampling device.

4. Retrieve the sampler and discharge the first 10 to 20 ml to clear any potential contamination on the valve. Transfer the sample to the appropriate sample container.

9.2 Bacon Bomb Sampler

A bacon bomb sampler may be used in similar situations to those outlined for the Kemmerer bottle. Sampling procedures are as follows:

1. Lower the bacon bomb sampler carefully to the desired depth, allowing the line for the trigger to remain slack at all times. When the desired depth is reached, pull the trigger line until taut.

2. Release the trigger line and retrieve the sampler.

3. Transfer the sample to the appropriate sample container by pulling the trigger.

9.3. Dip Sampler

A dip sampler is useful for situations where a sample is to be recovered from an outfall pipe or along a lagoon bank where direct access is limited. The long handle on such a device allows access from a discrete location. Sampling procedures are as follows:

1. Assemble the device in accordance with the manufacturer's instructions.
2. Extend the device to the sample location and collect the sample.
3. Retrieve the sampler and transfer the sample to the appropriate sample container.

9.4 Direct Method

For streams, rivers, lakes, and other surface waters, the direct method may be utilized to collect water samples from the surface. This method is not to be used for sampling lagoons or other impoundments where contact with contaminants is a concern.

Using adequate protective clothing, access the sampling station by appropriate means. For shallow stream stations, collect the sample under the water surface pointing the sample container upstream. The container must be upstream of the collector. Avoid disturbing the substrate. For lakes and other impoundments, collect the sample under the water surface avoiding surface debris and the boat wake.

When using the direct method, do not use pre-preserved sample bottles as the collection method may dilute the concentration of preservative necessary for proper sample preservation.

10.0 Sample Handling and Preservation

10.1 Once samples have been collected:

1. Transfer the sample(s) into suitable labeled sample containers.
2. Preserve the sample or use pre-preserved sample bottles, when appropriate.
3. Cap container, tape the cap securely to the container and then place container into plastic zip-locked plastic bag. If the latter is unavailable, use plastic bags and secure closure with tape.
4. Load all sample containers into cooler(s) ensuring that bottles are not totally immersed in ice.
5. Record all pertinent data in the site logbook and on a field data sheet.
6. Complete the chain-of-custody form.
7. Attach custody seals to the cooler prior to shipment.

8. Decontaminate all sampling equipment prior to the collection of additional samples.

11.0 Data and Records Management

All data and information (e.g., sample collection method used) must be documented on field data sheets or within site logbooks with permanent ink.

12.0 Quality Control And Quality Assurance

12.1 Representative samples are required. In order to collect a representative sample, the hydrology and morphometrics, (e.g., measurements of volume, depth, etc.) of a stream or impoundment should be determined prior to sampling. This will aid in determining the presence of phases or layers in lagoons or impoundments, flow patterns in streams, and appropriate sample locations and depths.

12.2 All field QC samples required in the QA Project Plan must be followed; these may involve field blanks, trip blanks, and collection of replicate samples.

13.0 References

SOP R11 #200 Sampling Equipment Decontamination, Version 1.1.

APPENDIX C

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF COLOR

DRAFT EXAMPLE - DO NOT QUOTE OR CITE

Prepared by: _____ **Date:**_____
Chemist

Reviewed by: _____ **Date:**_____
Section Chief

Approved by: _____ **Date:** _____
Laboratory Quality Assurance Coordinator

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION XI
REGIONAL LABORATORY

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1.0 Scope and Application

1.1 The Platinum-Cobalt method is useful for measuring color of water derived from naturally occurring material, i.e., vegetable residues such as leaves, barks, roots, humus, and peat materials. The method is not suitable for color measurement on waters containing highly colored industrial wastes.

1.2 Detection limit is 5 color units.

1.3 The range is from 5 to 70 units. Higher values may be measured by dilution of the samples.

1.4 Note: The spectrophotometric and Tristimulus methods are useful for detecting specific color problems. The use of these methods, however, is laborious and unless determination of the hue, purity, and luminance is desired, they are of limited value.

2.0 Summary of Method

2.1 Color is measured by visual comparison of the sample with platinum-cobalt standards. One unit of color is that produced by 1mg/L platinum in the form of the chloroplatinate ion.

3.0 Health and Safety Warnings

3.1 Standard laboratory protective clothing and eye covering is required.

4.0 Cautions

4.1 Reagent standards must be prepared fresh on the day of analysis.

4.2 Determination must be made within 48 hours of collection and sample stored at 4^{o}C.

5.0 Interferences

5.1 Since very slight amounts of turbidity interfere with the determination, samples showing visible turbidity should be clarified by centrifugation. Alternately, samples may be filtered. If turbidity is removed, the results are reported as "true color" otherwise the results are reported as "apparent color."

5.2 The color value of water may be extremely pH-dependent and may increase as the pH of the water is raised. When reporting a color value, specify the pH at which color is determined.

5.3 Absorption of ammonia by the standards will cause an increase in color.

6.0 Personnel Qualifications

6.1 Technician should be trained at least one week in the method before initiating the procedure alone.

7.0 Equipment and Supplies

7.1 Nessler tubes: Matched, tall form, 50 ml capacity.

7.2 Racks for Nessler tubes.

7.3 Miscellaneous lab glassware.

8.0 Instrument Calibration and Standardization

8.1 Chloroplatinate Stock Standard, 500 units: Add 100 ml concentrated HCl to 500 ml reagent grade deionized water. Dissolve 1.246g Potassium Chloroplatinate and 1.0g Cobaltous Chloride Monohydrate in this mixture and dilute to 1000 ml. This may be purchased from Fisher Scientific as Platinum Cobalt Standard and is equivalent to 500 color units.

8.2 Prepare the following series of standards, fresh on the day of the analysis.

MLs of Standard Solution Diluted to 50 ml with Reagent Grade Deionized Water	Color in Chloroplatinate Units
0.0	0
0.5	5
1.0	10
1.5	15
2.0	20
2.5	25
3.0	30
3.5	35
4.0	40
4.5	45
5.0	50
6.0	60
7.0	70

9.0 Sample Collection

9.1 Representative samples shall be taken in scrupulously clean containers. Both glass and plastic containers are acceptable.

10.0 Sample Handling and Preservation

10.1 Since biological activity may change the sample color characteristics, the determination must be made within 48 hours of collection.

10.2 Samples should be stored at 4EC.

11.0 Sample Preparation and Analysis

11.1 Apparent Color: Observe the color of the sample by filling a matched Nessler tube to the 50 ml mark with the sample and compare with standards. This comparison is made by looking vertically downward through the tubes toward a white or specular surface placed at such an angle that light is reflected upward through the columns of liquid. If turbidity has not been removed by the procedure given in 7.2, report color as "apparent color."

11.2 True Color: Remove turbidity by centrifuging until supernatant is clear; up to one hour may be required. Samples can also be filtered through a Whatman #541 filter paper. Results are reported as "true color" if steps are taken to remove turbidity.

11.3 Measure and record pH of each sample (see SOP C-24).

11.4 Dilute any sample with more than 70 units of color and reanalyze.

12.0 Data Analysis and Calculations

12.1 Calculate the color units by means of the following equation:

$$\text{Color units} = \frac{A \times 50}{V}$$

where:

A = estimated color of diluted sample.
V = ml sample taken for dilution.

12.2 Report the results in whole numbers as follows:

Color Units	Record to Nearest
1 - 50	1
51 - 100	5
101 - 250	10
251 - 500	20

13.0 Data and Records Management

13.1 All laboratory records must be maintained in the bound record book designated for the method.

13.2 All project records must be entered into the Laboratory Information Management System (LIMS) within seven days from the completion of the analysis.

14.0 Quality Control And Quality Assurance

14.1 There are no QC samples for color at this time.

14.2 Choose one sample per set of analyses and run in triplicate. RSD % should not be greater than 20%.

14.3 Spikes are not applicable to color determination.

15.0 References

Standard Methods for the Examination of Water and Wastewater, 20th Edition. 1995.

Methods for Chemical Analysis of Water and Wastewater, Method #110.2

APPENDIX D

STANDARD OPERATING PROCEDURE FOR CULTURING THE GREEN ALGA SELANASTRUM FOR USE AS CERIODAPHNIA FOOD

No. B23.0
December 1999

DRAFT EXAMPLE - DO NOT QUOTE OR CITE

Prepared by: _____ **Date:**_____
Regional Biologist

Reviewed by: _____ **Date:**_____
Branch Chief

Approved by: _____ **Date:** _____
Quality Assurance Officer

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION XI
REGIONAL LABORATORY

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1. Scope and Applicability

1.1 The purpose of this procedure is to present the recipe for a culture media to grow the green alga Selenastrum capricornutum, and to provide direction for preparing a final suspension for feeding to Ceriodaphnia dubia.

1.2 This green alga is known to be a preferred food of cladocerans and other herbivorous filter-feeding invertebrates in natural aquatic environments and is suitable for laboratory feeding of the invertebrate Ceriodaphnia dubia. A standard procedure is needed to consistently provide a healthy algal culture and obtain a cell density sufficient for use as a food source for Ceriodaphnia dubia, a test organism in toxicity testing.

1.3 This procedure is specially intended for use in culturing Selenastrum capricornutum in the laboratory when strict aseptic conditions cannot be met.

1.4 The media recipe is a modified Wood Hole Marine Biological Laboratory (mMBL) medium (ref. 1) and has been used successfully in this laboratory since October 1998.

2. Summary of Method

The green algae, Selenastrum capricornutum, is grown in a defined liquid medium, under set conditions, and concentrated by centrifugation for use as a food source to the invertebrate Ceriodaphnia dubia.

3. Health & Safety

No specific safety measures beyond good laboratory practices are necessary.

4. Equipment and Supplies

1. 2 L Erlenmeyer flask
2. Compound microscope, 10x ocular, 43x objective
3. Hemacytometer
4. Pipettor, adjustable
5. Sterile pipettes, 2 ml
6. Filtered air supply (carbon and 0.2 Fm capsule)
7. Light bank equipped with cool white fluorescent light bulbs capable of producing 400 ft-c of light intensity
8. Cheesecloth
9. Cotton

10. Centrifuge
11. Centrifuge bottles, 40 ml and 250 ml
12. Tygon of silicone tubing (for aeration)
13. Syringe needle on tip of sterile glass pipette
14. Light meter
15. Sterilized inoculation loop
16. Ceriodaphnia food preparation log book
17. Hydrochloric acid
18. Acetone
19. Purified water, Super-Q
20. Chemicals for MBL medium (see section 5.2)
21. Stock culture of Selenastrum capricornutum.

5. Procedure

5.1 Cleaning of Glassware

a. Glassware is cleaned according to SOP L0118.1, SOP for Cleaning Laboratory Glassware, except that the final rinse is with Super-Q water.

b. Glassware should always be oven dried to obtain partial sterilization.

c. Glassware used for algal culturing should be kept separate from general laboratory glassware and should be used only for cultivating algae.

5.2 Preparation of Stock Solutions

a. Prepare micronutrient stock solutions by adding the chemical(s) to Super-Q water to make 1L in a volumetric flask. For stock solution #3, add chemical in the order shown.

1.	Stock Solution #1:	4.36 g $Na_2EDTA\ H_2O$
2.	Stock Solution #2:	3.15 g $FeCl_3\ .6H_2O$
3.	Stock Solution #3:	0.01 g $CuSo_4.5H_2O^*$
		0.01 g $CuSO_4.6H_2O^*$
		0.022 g $ZnSO_4.7H_2O^{**}$
		0.18 g $MnCl_2.4H_2O$
		0.006 g $Na2MoO_4.2H2O^{***}$
		1.0 g H_3BO_3
		0.01 g $Na_2SeO_3^*$

[*] weigh out 100.0 mg and dilute to 100 ml. Add 10.0 ml of this solution to Stock #3**.**
[**] weigh out 220.0 mg and dilute to 100 ml. Add 10.0 ml of this solution to Stock #3.
[***] weigh out 60 mg and dilute to 100ml. Add 10.0 ml of this solution to Stock #3.

b. Prepare macronutrient stock solutions by adding the chemical to 1L Super-Q water in a volumetric flask.

1. Stock Solution #4: 36.76 g $CaCl_2.2H_2O$
2. Stock Solution #5: 36.97 g $MgSO_4.7H_2O$
3. Stock Solution #6: 12.60 g $NaHCO_3$
4. Stock Solution #7: 8.71 g K_2HPO_4
5. Stock Solution #8: 85.01 g $NaNO_3$

c. Add 1 ml of each stock solution per liter of medium, except add 2 ml of stock solution #2. Use a sterile pipette for each stock solution and dispose of after use.

d. Keep all stock solutions, except #3, in the dark at room temperature. Keep stock solution #3 at 4°C. Retain stock solutions for no longer than 24 months.

5.3 Starting an Algal Culture

a. Use a 2 L Erlenmeyer flask to prepare a 1.5 L batch of MBL medium. Add 1500 ml of Super-Q water to the cool oven-dried flask. Add 1.5 ml of each stock except #2, using a sterile 2.0 ml pipette for each solution. Add 3 ml of stock solution #2.

b. Inoculate the MBL medium with a slightly visible amount of inoculum from an agar slant using a sterile inoculating loop and aseptic technique. An alternative inoculum can be used by transferring 1-2 ml of a previous culture maintained at 4°C to the newly prepared mMBL medium.

c. Place a sterile pipette in a flask with a beveled syringe needle fixed to the pipette tip. Attach to a filtered air source. This is done to prevent the algae from settling and to provide adequate gas exchange. Stopper the culturing flask with a gauze/cotton plug.

d. Cultures are kept at 25 $\pm$ 2°C (or at ambient temperature) at a light intensity of approximately 400 ft-c. Lighting is continuous, i.e., 24 hours per day.

e. When cultures are very green, in 6-8 days, algal cells can be harvested.

f. A portion of this culture can be stored in the dark at 4°C (in a Nalgene bottle) and can be used as a future inoculum.

5.4 Selenastrum Culture Source

a. Algal slants can be purchased from the collection at the University of Texas, Austin, Texas.

b. Slants can be kept and used for up to 12 months when stored in the dark at 4°C.

5.5 Preparation of Algae for Use as Food

a. Centrifuge harvested algal culture to concentrate the cells and then decant the spent media. This can be done using either 40 ml aliquots in 40 ml centrifuge tubes at 3750 rpm for 12 minutes or in 200 ml aliquots in 250 ml centrifuge bottles at 2600 rpm at 40 minutes.

b. Rinse and resuspend centrifuged algal cells in reconstituted hard water (see SOP B13.1).

c. Determine the cell density (#cells/ml) of the cell concentrate by direct microscopic count at 400x using a hemacytometer chamber. (See SOP B14.0)

d. The goal is to produce an algal suspension with a final concentration of 3.5×10^7 cells per milliliter. If the cell concentrate is not dense enough, centrifuge again. Use the following formula to obtain the final volume needed to get this desired concentration:

$$\frac{\text{\# cells/ml in concentrate} \times \text{ml of concentrate}}{\text{\# cells of the desired concentration}} = \text{ml of final suspension}$$

Then, ml of final suspension - ml of concentrate = ml of diluent.

e. Increase the ml of algal concentrate using reconstituted hard water as the diluent.

f. Confirm the cell density of the final suspension (i.e., the diluted concentrate) using a microscope (400x) and hemacytometer to count and calculate the # cells/ml.

g. Store this suspension of algae in the dark at 4°C. Use for 4 weeks, then discard any remaining or use to feed backup Ceriodaphnia cultures.

5.6 Feeding Algae to Ceriodaphnia

a. The algal suspension is dispensed from an adjustable pipettor that has been designated for use only with algal suspension.

b. QC: The pipettor is calibrated monthly at the daily feeding volumes by using gravimetric measurements. See SOP B29.1.

c. The alga, Selenastrum capricornutum is fed at the daily rate of 100 Fl per 15 ml solution.

6.0 Data and Records Management

Record all algal procedures and calculations in the Ceriodaphnia Food Preparation logbook.

7.0 References

1. Nichols, H. W. In Handbook of Phycological Methods, J. R. Stein, Ed. Cambridge University Press, London, 1931, pp. 7-24.

2. Standard Operating Procedure for Cleaning Laboratory Glassware, SOP L0118.1. EPA Region IX, Regional Laboratory

3. Standard Operating Procedure for Centrifuging Algal Cells, SOP B13.1. EPA Region IX, Regional Laboratory.

4. Standard Operating Procedure for Use of a Hemacytometer, SOP B14.0. EPA Region IX, Regional Laboratory.

5. Standard Operating Procedure for Calibration of Algal Pipettor, SOP B29.1. EPA Region IX, Regional Laboratory.

APPENDIX E

Standard Operating Procedure
for the Review of Data Produced in the Analysis for Metals in Environmental Samples

ML 005 Version 0.0

United State Environmental Protection Agency
Region 11

Date: November 2000

DRAFT EXAMPLE - DO NOT QUOTE OR CITE

Concurrences:

Originator:_____

Quality Control Coordinator:_____

Laboratory Director: _____

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1. Purpose:

The purpose of this standard operating procedure is to ensure that all reviews of analyses for metals in environmental samples are of similar quality. Raw data review is a very critical part of the analytical process. A thorough review can prevent errors in reporting or shortcuts in procedure on the part of the analyst. It is imperative that a thorough review be conducted because some of the data may be used as evidence in court proceedings. Raw data review is also needed to ensure that the acceptance criteria for the project have been met.

2. Applicability:

All data produced by the Region 11 laboratory must be reviewed before its release to the client. Data produced in the analysis of metals must be reviewed by the procedure described herein.

3. Procedure Summary:

3.1 There are three levels of data review. These are done by the analyst, a peer, and the quality control (QC) coordinator. The analyst and peer do similar depth of profile review, while the QC coordinator does an in-depth review, on an as-needed basis. This would be done as a random spot-check, or selectively, as in the case of a high profile survey. There are three types of review for the analyst and peer. These are Package Overview, determining correctness of the reports and completeness of the package (Section 5); Quality Control Review, determining the quality of the data from the audits (Section 6); and Technical Review, determining that the data are not compromised by matrix effects (Section 7). The QC coordinator will do all of these, plus examine the electronic records.

3.2 A narrative prepared by the analyst is included with the data (Section 9). This document contains a description of the samples, the purpose of the analysis (acceptance criteria, if known), all operations with the samples, including corrective actions taken in the process, the filenames and paths for all electronic records pertaining to the analysis, and the result of the analyst's QC review with comments about the usability of the data.

3.3 Each reviewer is responsible for verifying each of the parts that are designated for their review and for completing the checklist (Attachment 1) associated with the data package.

3.4 Appropriate data qualifiers are used where they are appropriate. The data qualifiers that will be used in the data validation report (B, D, E, J, M, N, Q, R, and U) are described in Attachment 2.

4. Definitions

Acceptance Criteria: the specific quality objectives for a given project. These should be described in the QA Project Plan.

Peer: someone who has equal understanding of the chemical analysis of these samples.

Prep log: the laboratory record book having the sample preparation information.

Quality Assurance (QA) Project Plan: a document that describes project-specific information such as the necessary quality assurance, quality control, and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated acceptance criteria. All work funded by EPA that involves the acquisition or usage of environmental data must have an approved QA Project Plan. The Plan must be approved before samples or analysis is started.

RSD: relative standard deviation, a measure of dispersion in a frequency distribution, the standard deviation divided by the mean.

5. Data Package Overview Requirements

5.1 Verify that all components under Section 8, Raw Data File Requirements, are present.

5.2 Verify that results are reported for all samples and analytes requested. This includes:

- Sample Number and Station ID
- Sample Batch Number
- Facility or Study Name
- Detection Limits - Check if dilution used for reporting
- Sample reporting units are correct (Fg/L, mg/kg, Fg/m^3, etc.)
- Correct method for preparation
- Dates of Analyses
- All requested analytes are reported
- Concentrations reported are correct

- Correct number of significant figures are used
- If data value between reporting level and MDL are reported, data is properly flagged with an "M"

5.3 Verify that all special requests have been done. This should be evaluated against the QA Project Plan acceptance criteria received with the sample request. This includes detection limits, special analytes, and method requirements. If detection limits are elevated due to matrix effects, and the acceptance criteria are affected, this should be noted in the narrative.

5.4 Calculations: Verify that calculations used to arrive at the reported concentration are correct. This would be done by performing a sample of manual calculations, and by verifying that manual input to a computerized algorithm are correct. Spot-check to verify that such algorithms are applied correctly. This includes sample manipulations, dilutions, preconcentration, etc.

5.4.1 Verify that results reported from a dilution agree within expected error with previous analyses at different dilutions or of the original undiluted sample. It is best if two dilutions of different levels are made, one to confirm the other. If, for a multi analyte technique, results are reported from one dilution for some analytes, and from a different dilution for others, verify that the analytes reported from the lesser dilution are not affected by the analytes reported from the greater dilution (see Section 7.1.4). Samples diluted in the course of the analysis to obtain the reported value should be flagged with a "D," even if the dilution is done in the preparation due to suspected high solids.

5.4.2 If results are reported from a dilution, wherein the diluted concentration lies between the MDL and the reporting limit, the result must be flagged with an "M", and the number of significant figures used that is appropriate to the diluted concentration. This reporting of data between the MDL and the reporting limit should be consistent with the usage in the remainder of the report for undiluted samples.

5.5 Calibration: Verify that reported results do not exceed the calibration without explanation in the narrative. Data that exceed the calibration range are flagged with an "E."

5.6 Field Quality Control: Verify that the field QC (field duplicates and blanks) has no errors attributable to laboratory operations. No calculations are performed on the

field QC because there are variables beyond the laboratory's control. The reviewer still needs to verify that these QC audits provide a true picture of the field operations.

5.6.1 Field Blanks: Normally, field blanks should be of the same quality as laboratory blanks. Sometimes there will a signature note to indicate that the blank was contaminated before its arrival at the laboratory. High sodium may indicate that blank water was kept in a glass bottle before use. High copper may indicate the blank water was purchased at a grocery store. Since the blank is without solids, the analyst may confirm the blank result in the undigested aliquot to indicate to the client that the blank result was not from laboratory contamination. Some field blanks are actual washings of field equipment, and should be so designated on the station identifier. These blanks may well show some elements above detection. Unless there is some indication from the client to the contrary, these blanks are treated just as a blank originating in the laboratory would for use of the "B" flag.

5.6.2 Field Duplicates: Verify which samples are field duplicates by comparison of the sample and station identifiers. For multi-analyte tests, verify that the pattern of results is similar for the two samples. For single analyte tests, view the result as if it were a laboratory duplicate. Again, the sample description in the narrative is of aid in this evaluation. It sometimes occurs that the field duplicates have varying amounts of solids. It is important to communicate all variables to the client.

5.7 Turn in final reviewed package on or before agreed-upon due date.

6. Quality Control Review

6.1 This type of review is required of the analyst and the peer reviewer. The QC Coordinator may choose to do this on a spot check basis. Failure to meet some QC standards may affect usability of the data when evaluated against the acceptance criteria, so this review should be done as quickly as possible. Re-analysis or even re-preparation of the samples may be necessary in some situations, so this must be done before holding times expire, or before agreed upon turnaround times expire, whichever comes first. Except where noted, most of the QC failures discussed below will result in a flag of a "J." Use of a "Q" or "R" flag (with which no result is given) is expected to be rare, as most metals analyses are amenable to re-analysis within the holding time, and the entire sample is not consumed, except in rare instances. Any flag must be explained in the narrative.

6.2 Holding Times: Verify that the samples have been analyzed within the holding time for the analysis. The holding time for total metal analysis of aqueous samples (other than mercury: 28 days) is six months from date of collection. There are no data for a corresponding holding time in other matrices, but many QA Project Plans and the acceptance criteria will assume the same holding time as applies for water.

6.3 Preservation: Verify that the samples had been properly preserved prior to analysis. For aqueous samples, the preservation for total metals is a pH <2. This must be checked before an aliquot is taken for digestion, and indicated on the prep log. If pH is >2, either the pH must be adjusted with HNO_3 and time allowed to re-solublize analyte that may have adsorbed onto the container walls (16 hours minimum), or if the sample is extremely basic or highly buffered, this must be documented. For soil, sediment or sludge samples, the proper preservation is for the sample to be kept at 4°C until subampling. The sample custodian will document if samples are received without ice. The analyst must keep the sample refrigerated until subsampled for drying and grinding.

6.4 Digestion: Verify that the samples have been digested using a method appropriate to the request. The referenced digestion SOP must be noted on the prep log. The sample must be digested unless the samples are to be analyzed for dissolved metals, in which case the sample must be filtered through a 0.45 Fm filter in the field, and there must be documentation that matrix matching occurred between samples and standards. If the QA Project Plan states a requirement for a specific digestion, that digestion must be used. If there is no such statement, there may be Programmatic assumptions, e.g., RCRA samples use SW-846 methods.

6.5 Instrument Performance Checks: Some methods (ICP, ICP-MS) have instrument performance checks that are performed on a periodic basis to confirm that the instrument is operating properly before analysis proceeds.

6.5.1 Spectral Position Optimization: Documentation must be present that either the mercury re-alignment or the zero order search, carbon line auto search, and auto search on each analyte line has been performed.

6.5.2 Interference Check Samples: These are solutions containing known interferences that are analyzed to confirm that spectral interference corrections are being done properly. The analysis SOP describes the frequency at which these must be run.

6.5.3 ICP-MS Tuning: A copy of the tuning log should accompany the data package.

6.5.4 Short-Term Stability Check: Before an ICP-MS analysis begins, a short-term stability check must be run. An RSD of 3% or better must be obtained on the isotopes either used as internal standards or bracketing the isotopes used for data reporting.

6.6 Calibration: Verify that a fresh calibration curve has been performed the day of the analysis, and before analysis proceeds. For multiple-point calibrations (GFAA, ICP-MS), verify that the figures of merit for the calibration are acceptable when evaluated against the criteria in the analysis SOP. In addition, there are several calibration checks which must be present.

6.6.1 Reporting Level Check: For ICP, a reporting level check standard is analyzed following calibration. The concentration of this standard is the lowest concentration for reporting without qualifiers. The result for this standard must be within the statistic derived from historical data.

6.6.2 Instrument Blank (LCB): This blank must be run after calibration, and at the minimum frequency of one every 10 samples and at the end of the analysis run. The limit for analytes reported to the reporting level is the MDL. If all samples in the analysis run either in the grouping just before or just after a blank above the MDL are more than 10 times greater than the result in that blank for that analyte, the data are useable. The flag for the presence of the analyte in the blank is a "B."

6.6.3 Instrument Check Standard (LCM): This check must be run after calibration, and at the minimum frequency of one every 10 samples and at the end of the analysis run. The limit for this check is specified in the analysis SOP. Some tests will have more than one LCM for different analytes. Some deviation from the limits is allowable, as in the case of the element not being reported but analyzed only for interelement corrections, or if a result for this audit above the limit is obtained but the analyte is less than a reportable quantity.

6.7 Internal Standards: Some tests (e.g., ICP-MS) make routine use of internal standards. The limits on the recovery of the internal standards is given in the analysis SOP. Verify that these limits are met, and that the software is flagging those outside the limits.

6.8 Analytical Spikes: Some tests (e.g., GFAA) make routine use of analytical spikes. The limits on the recovery of the analytical spikes is given in the analysis SOP. Verify that these limits are met, and that the software is flagging those outside the limits.

6.9 Matrix Spikes: Verify that matrix spikes were analyzed at the required frequency, and recoveries are within the limits stated in the analysis SOP. Laboratory policy is to include a matrix spike/duplicate pair for every 10 samples, with a minimum of one for each batch from a survey. This frequency may be increased or decreased at the discretion of the client, group leader, or sample management. Preapproval is required. The limits for recoveries are evaluated against the required acceptance criteria in the QA Project Plan. A recovery outside of limits has a much greater weight when the result for the analyte is near the action level given in the QA Project Plan. The matrix duplicate has a bearing on the evaluation of the matrix spike, as is explained in the next section.

6.10 Matrix Duplicates: Verify that the matrix duplicates are analyzed at the required frequency, and RPDs or absolute differences are within the limits stated in the analytical SOP. As for spikes, laboratory policy is to include a matrix spike/duplicate pair for every 10 samples, with a minimum of one for each batch from a survey. This frequency may be increased or decreased at the discretion of the client, group leader, or sample management. Preapproval is required. The limits for RPDs or absolute differences are evaluated against the acceptance criteria in the QA Project Plan. A difference outside of limits has a much greater weight when the result for the analyte is near the action level given in the QA Project Plan. The matrix spike/duplicate pair should be evaluated together for evidence of sample homogeneity problems. The sample description in the narrative is of aid in this evaluation. A QA Project Plan may call for matrix spike duplicates in place of separate matrix spikes and duplicates. In that event, the two matrix spikes are evaluated separately as matrix spikes, and the pair is evaluated as above for a sample/duplicate pair.

6.11 Digestion Blanks (LRB): Verify that digestion blanks were prepared and analyzed at the required frequency. Ordinarily, this will be the same frequency as the matrix spikes and duplicates. For large batches, a lesser number of blanks may be prepared with pre-approval from the group leader, so long as it meets the needed acceptance criteria. The limit for analytes reported to the reporting level is the MDL. If all samples in the analysis run either in the grouping either just before or just after a blank above the MDL are more than 10 times greater than the result in

that blank for that analyte, the data are useable. The flag for the presence of the analyte in the blank is a "B."

6.12 Spike Blanks (LRB): Verify that digested spiked blanks are done at the frequency required by the analysis SOP. Evaluation of a spiked blank is similar to a matrix spike. The spiked blank may be the only accuracy audit for a batch where the sample greatly exceeds the spike amount added to the matrix spike.

6.13 Laboratory Control Sample (LCS): In addition to the spiked blank, preparation of matrix types other than simple water samples requires a preparation of a sample of known concentration for the analyte in question. Verify that this LCS has been digested and analyzed with recovery of the analytes within the limits specified in the SOP, or the QA Project Plan, if so specified.

6.14 Species Specific QC: Some methods (hydride generation AA, mercury) are sensitive to the oxidation state or the molecular species of the analyte. To ensure that the analysis is a true total analysis, audits are performed to verify that the sample preparation converts all forms of the analyte into the appropriate oxidation state for analysis. Verify that the audits specified by the analysis SOP have been performed with results within the limits given in the SOP.

7. Technical Review

7.1 Finally, the analyst and peer should use their knowledge and experience to verify that known interference and matrix problems do not compromise the data. Among these are known spectral, chemical and physical interferences. In the process of setting up instrumental methods, most of these are overcome or compensated for. However, it is in the nature of analyzing unknown samples that interferences beyond those foreseen are encountered, or it may be cost prohibitive to compensate for all interferences, however infrequent. It is best to be vigilant for such problems in the review. Where such interferences are suspected with reasonable certainty, the affected data should be flagged with a "J" and an explanation included in the narrative (Section 9.4). Among the types of interferences to be concerned about are:

7.1.1 Physical Interference: Analysis techniques dependent on aerosol formation for sample transport to the analytical system (ICP, ICP-MS, Flame AA) are prone to physical interference due to high solids or failure to match acid type or strength. If internal standards are not used, and some samples have widely varying solids concentrations, it may be wise to either dilute very

high solids samples or perform an analytical spike to demonstrate that solids are not causing an interference. The analysis SOP should give guidance regarding when such is necessary.

7.1.2 Chemical Interference: GFAA compensates for chemical interferences by using the analytical spike to verify that they are not causing interference. ICP can have ionization interference when, for example, a high sodium concentration provides excess electrons, permitting more neutral atom species for potassium, thereby biasing potassium high.

7.1.3 Spectral Interference: All spectral techniques are prone to interference, either from fluctuating background, spectral overlap, or isobaric overlap. The expected interferences are built into correction algorithms in the instrument method. The analyst and peer reviewer should be aware of the limitations of those correction algorithms and what evidence might indicate those limitations have been exceeded. For example, the presence of a result that is very negative for one analytical line in ICP analysis may be an indicator that an interferent is present in the sample that is not included in the IEC table or MSF model. Alternate lines can be used to verify the results obtained. An additional spectral interference that is unlike the usual overlap is possible with the X instrument: subarray saturation. It is possible that as the subarray is integrated using variable integration time, that another line on the subarray other than the one being analyzed for is much larger, thereby cutting short the integration time for the analyte and raising the detection limit for that line.

7.1.4 As sometimes occurs in multi-analyte techniques, dilution may be necessary to obtain a valid result for one constituent, but the presence of that constituent may or may not affect the analysis of the other analytes. It is desirable to provide as much information to the end user of the data as possible. It is valid to report some data from one dilution, while reporting other data from a lesser dilution or even the undiluted sample. If the effect of the high level constituent is merely a physical effect on sample delivery, and it can be shown that the analytes that are above the reporting level both before and after the dilution are the same within expected error, then all the trace analytes may be reported from the lesser dilution. If the sample were spiked, direct evidence of the physical interference can be obtained. For a spectral interference, this is not conclusive evidence, so an evaluation of the relative error in each case, as determined from the IEC table (if used), is needed.

7.2 If a reviewer has prior knowledge of what might be expected from a sample type, either from the QA Project Plan or elsewhere, that knowledge should be applied in the review. This would not be used to qualify the data, but may suggest extra verification may be advisable if the analytical results differ strongly with the prior knowledge.

8. Raw Data Requirements:

The raw data should contain all information necessary for data review, and should be present with every package. This includes not only the raw instrument printout, but other hard copy records used to review the data. If multiple batches are analyzed together in a single run, and all batches can be submitted for review together, it is not necessary to copy all records for each batch. However, if they are submitted separately, copies should be made. The electronic records are maintained as described in the procedure for archival noted in the laboratory information system (LIMS) (SOP xxx). The electronic records are reviewed by the peer and subsequent reviewers only in special cases at the request of the Deputy Laboratory Director.

8.1 Hard Copy Raw Data: These are the raw instrument printout and intermediate calculations which are presented with the package, along with the finished data report, for the purposes of establishing the validity of the data.

8.1.1 Raw Instrument Printout: This is the raw output from the instrument in the course of the analysis run. Each page, if not from unburst fan-folded paper, needs to be sequentially numbered, with time and date stamp. This output needs to be initialed and dated by the analyst on the front page, and the filename written out, if it is not so stamped by the instrument software.

8.12 Analysis Summary: Depending on the method, this would be either a summary list of samples and QC checks analyzed in the run with time stamps, or a condensed summary of samples, results, and recoveries.

8.1.3 Preparation Log: A copy of the sample preparation log should be part of the data package. The original is kept in the ring binder in the prep laboratory. This log, which is given a unique number when it is created, should be initialed and dated by the sample preparer, and should contain sample and QC audit identifiers, any notes about the sample preparation, notations of sample weight (for solids), spike solutions used, and any unusually observations about the samples.

8.1.4 Quality Control Summary: A summary of all the instrument and matrix QC includes concentrations obtained for all the audits, and the appropriate calculations, such as relative percent difference and recovery calculations, with notations where limits in the analysis SOP are exceeded. This will include separate sections for blanks, laboratory control samples, spiked blanks, calibration checks, and matrix duplicates and spikes.

9. Records Management:

All important events that would have any bearing on subsequent review of this data package should be summarized in a narrative. This document contains, for a batch number and a parameter (or parameter class), the facility name, a description of the samples, the purpose of the analysis (acceptance criteria, if known), all operations with the samples, including corrective actions taken in the process, the filenames and paths for all electronic records pertaining to the analysis, and the result of the analyst's QC review with comments about the usability of the data. The necessary components of the narrative are as follows:

9.1 Header: This contains:

- Date of preparation of the narrative
- Analyst name preparing the narrative (with signature)
- Facility or study name
- Parameter or parameter class

9.2 Description of Samples and Operation:

9.2.1 The number of samples and the sample numbers and station identifiers are stated. The type of sample is described, along with anything noteworthy about the physical state of the samples. If the samples are quite different from one another, a tabular form may be most useful to present this information. A statement should be present stating whether the samples were properly preserved upon receipt, and what action was taken if they were not. The dates of sample collection and analysis are given, with a statement as to whether the holding times were met.

9.2.2 The operations conducted with the samples are described, with the methods for preparation and analysis referenced. If there are any deviations from these methods, or if any optional operations within those methods were used, and/or if corrective actions were taken in dealing with difficulties encountered in the process, those should be described. If one or more of the

methods used was specified by the QA Project Plan, note it here. If one or more of the methods used deviated for that specified by the QA Project Plan, note it here, and explain the deviation. If samples required re-preparation or re-analysis, note that here.

9.2.3 The purpose of the analysis, if known from the QA Project Plan, should be summarized in a sentence or two. Action limits, such as permit limits, or TCLP extract limits, should be stated, if known.

9.2.4 If analysts other than the preparer of the narrative participated in the analysis in any way, their contribution should be documented.

9.3 Quality Control: All QC audits detailed in Sections 6.5 to 6.14 should be within control limits, or if not, the outliers listed here. Significance of the outliers to the usability of the data is discussed with reference to the acceptance criteria. If no acceptance criteria are available for the batch, evaluate against the analysis SOP.

9.4 Technical Review: If any of the situations described in Section 7 affect the data and result in a data flag, describe the condition here.

9.5 Electronic Data Inventory: The file names and paths for all electronic files used in generating the final report is given. The paths given should direct the reviewer to all data placed on the laboratory information management system (LIMS) per the upload protocol (SOP xxxx).

10. References:

SOP xxxx, Protocol for Upload of Inorganic Data to the LIMS, Revision 5, October 2000.
SOP xxxx, Analysis of Metals using GFAA, Revision 1, September 1998.
SOP xxxx, Analysis of Metals using ICP, Revision 0, January 1999.

Attachment 1
Metals Data Review Checklist

Batch Number: _____ Facility Name: _____
Parameter: _____

Package Overview		
Raw Data Package Complete?		
Results Reported Correctly?		
Special Requests Done?		
Calculations Checked?		
Calibration Not Exceeded?		
Field QC Checked?		
Quality Control		
Holding Times Met?		
Preservation Checked?		
Proper Digestion Verified?		
Initial Instrument Performance Checks Verified?		
Calibration Verification Checked?		
Sample-Specific QC (Internal Standards or Analytical Spikes) Okay?		
Matrix QC Checked?		
Digestion Blanks Checked?		
Spiked Blank Checked?		
LCS (if applicable) Checked?		
Species QC (if applicable) Checked?		
Final Check		
Technical Review Done?		
Narrative Complete?		

Analyst: _____ Peer Reviewer: _____
Date: _____ Date: _____
Comments Attached? (Y/N): _____

Attachment 2

Region 11 Data Review Qualification Codes

Qualifier	Description
B	This flag is used when the analyte is found in the associated ***B***lank as well as the sample. It indicates possible blank contamination and warns the user to take appropriate action while assessing the data.
D	This flag is used when the analyte concentration results from a required ***D***ilution of the sample, extract or digestate.
E	This flag is used to identify analyte concentrations ***E***xceeding the upper calibration range of the analytical instrument after dilution of the sample, extract or digestate. The reported value is considered to be estimated.
J	This flag is used when the analyte is confirmed to be qualitatively present in the sample, extract or digestate, at or above the Region's reporting limit (RL) but the quantitated value is estimated due to quality control limit(s) being exceeded. This flag accompanies all GC/MS tentatively identified compounds (TICs). This flag also applies to a suspected, unidentified interference. (***J*** is the flag used in the Superfund CLP SOW and Data Review Functional Guidelines and is used by this laboratory for consistency.)
M	This flag is used when the analyte is confirmed to be qualitatively present in the sample, extract or digestate, at or above Region's Method Detection Limit (MDL) but below the Region's reporting limit (RL). This flag applies to all values in this concentration range and indicates the quantitated value is estimated due to its presence in this concentration range.
N	This flag applies to GC/MS tentatively identified compounds (TICs) that have a mass spectral library match.
Q	This flag applies to analyte data that are severely estimated due to quality control and/or ***Q***uantitation problems, but are confirmed to be qualitatively present in the sample. No value is reported with this qualification flag.
R	This flag applies to analyte data that are ***R***ejected and unusable due to severe quality control, quantitation and/or qualitative identification problems. No other qualification flags are reported for this analyte. No value is reported with the qualification flag.
U	This flag is used when the analyte was analyzed but ***U***ndetected in the sample. The Region's RL for the analyte accompanies this flag. As with sample results that are positive, the value is corrected for dry weight, dilution and/or sample weight or volume.