Analytical Services Division

Rocky Flats Environmental Technology Site

Statement of Work
For Analytical Measurements

WHOLE EFFLUENT TOXICITY TESTING

MODULE SS11-A

October 22, 1998

Approved: _____

Analytical Services

Reviewed For Classification

By: Roger S. Cichorz U/NU

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WHOLE EFFLUENT TOXICITY TESTING MODULE

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WHOLE EFFLUENT TOXICITY PARAMETERS MODULE

INTRODUCTION

This module provides the technical requirements, analytical chemistry services, quality control procedures, and an analysis structure that will generate data of known and documented quality for the identification and quantitation of whole effluent toxicity, to include acute tests, chronic tests, toxicity reduction evaluation (TRE) and/or toxicity identification evaluation (TIE) parameters. The samples to be analyzed may contain potentially hazardous radioactive, inorganic, and/or organic materials.

Procedures specified herein shall be used in the preparation and analysis of effluents and receiving waters for the presence and quantitation of Whole Effluent Toxicity, to include Acute Tests, Chronic Tests, Toxicity Reduction Evaluation (TRE) and/or Toxicity Identification Evaluation (TIE) Parameters, and includes guidelines on laboratory safety, quality assurance, facilities and equipment, dilution water, effluent sampling methods and holding times, test specific selection, data analysis, report preparation, organism culturing and handling. The acute toxicity tests generally involve exposure of 20 test organisms to each of five effluent concentrations and a control water. The test duration ranges from 24-96 hours. This module contains specified test conditions for 2 commonly used freshwater organisms, Ceriodaphnia dubia and fathead minnows (Pimephales promelas). The chronic toxicity test measures lethal and sublethal toxicity with growth or reproduction as the sublethal endpoints.

This module describes acute and chronic toxicity tests for use in the National Pollutant Discharge Elimination System (NPDES) Permit Program to identify effluents and receiving waters containing toxic materials in acutely and chronically toxic concentrations. The methods included in this module are referenced 40 CFR Part 136 regulations and therefore, constitute approved methods for acute and chronic toxicity tests. They are also suitable for determining the toxicity of specific compounds contained in discharges.

The data are used for NPDES permit development and to determine compliance with permit toxicity limits. Data can also be used to determine potential acute and chronic toxicity in the receiving water, based on the LC50 and appropriate dilution, application, and persistence factors. The tests are performed as a part of self-monitoring permit requirements, compliance biomonitoring inspections, toxics sampling inspections, and special investigations. Data from acute and chronic toxicity tests performed as part of permit requirements are evaluated during compliance evaluation inspections and performance audit inspections.

Modifications of these tests are also used in toxicity reduction evaluations (TRE), and toxicity identification evaluations (TIE), to identify the toxic components of an effluent, to aid in the development and implementation of toxicity reduction plans, and to compare and control the effectiveness of various treatment technologies for a given type of industry, irrespective of the receiving water (USEPA, 1988a; USEPA, 1988b; USEPA, 1989a; USEPA, 1989b; USEPA, 1991a).

Guidance for the implementation of toxicity tests in the NPDES program is provided in the Technical Support Document for Water Quality-based Toxics Control (USEPA, 1991c). Further guidance for TIE and TRE can be found in EPA/600/6-91/005F and EPA/600/R-92/081.

The use of any test species or test conditions other than those described in this module or referenced in 40 CFR 136.3, shall be considered a major modification to the module and subject to application and approval of alternate test procedures under 40 CFR 136.4 and 40 CFR 136.5.

These methods are restricted to use by, or under the supervision of, analysts experienced in the use or conduct of, and interpretation of data from, aquatic toxicity tests. Each analyst shall demonstrate the ability to generate acceptable test results with the methods using the procedures described in this module. Updates to these methods shall be used by the Laboratory once the methods are promulgated and approved for use by the Site. The CTR shall provide the Laboratory with implementation dates for any new methods.

The following modules are required for the analysis of Whole Effluent Toxicity, to include acute tests, chronic tests, toxicity reduction evaluation (TIE) and/or toxicity identification evaluation (TRE) Parameters under this subcontract: the General Laboratory Requirement Module, GR01; and Whole Effluent Toxicity, to include acute tests, chronic tests, toxicity reduction evaluation (TRE) and/or toxicity identification evaluation (TIE) parameters Module, SS11. The specifications in SS11 shall supersede any GR01 specifications in the case of conflicting requirements.

EXHIBIT A

SUMMARY OF REQUIREMENTS

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WHOLE EFFLUENT TOXICITY PARAMETERS

SUMMARY OF REQUIREMENTS

1. GENERAL REQUIREMENTS

This module comprises the eight exhibits and one appendix, which define requirements for the determination of Whole Effluent Toxicity Parameters (WETP) and (NPDES). Exhibit A provides an overview of the Whole Effluent Toxicity Parameters module and its general requirements. Exhibit B contains reporting and deliverables requirements. Exhibit C contains the Target Analyte List (TAL) for all target analytes. Exhibit D specifies analytical procedures, defines the application of these procedures, and contains method specific QA/QC requirements. Exhibit E contains general and specific laboratory QA/QC requirements. Exhibit F contains the evidentiary requirements including, chain-of-custody and evidentiary document control requirements that the Laboratory shall follow in processing samples under this subcontract, and specifies requirements for written laboratory Standard Operating Procedures (SOPs). To ensure proper understanding of language utilized in this subcontract, Exhibit G contains a glossary of terms, which supplement the glossary found in GR01 Exhibit G. When a term is used in the text without definition, the glossary meaning shall be applicable. Exhibit H contains references applicable to SS11.

2. FACILITY, INSTRUMENTATION AND KEY POSITION REQUIREMENTS

2.1. **Facility:** The Laboratory facility shall meet all requirements of base analytical methods and the Laboratory Health and Safety Program specified in GR01. The analytical and sample storage areas of the Laboratory shall be isolated from all potential sources of contamination and cross contamination. Separate test organism culturing and toxicity testing areas shall be provided to avoid possible loss of cultures due to cross-contamination. The facilities shall be well ventilated and free of toxic fumes. Ventilation systems shall be designed and operated to prevent recirculation or leakage of air from chemical analysis laboratories or sample storage and preparation areas into organism culturing or toxicity testing areas, and from toxicity test laboratories and sample preparation areas into culture rooms. Organisms shall be shielded from extreme disturbance.

2.1.1. Laboratory and toxicity test temperature control equipment shall be adequate to maintain recommended test water temperatures.

2.1.2. Effluent toxicity tests may be performed in a fixed or mobile laboratory. Facilities shall include equipment for rearing and/or holding organisms.

2.1.3. Control of test solution temperature shall be achieved using circulating water baths, heat exchangers, or environmental chambers. Photoperiod shall be controlled using automatic timers in the laboratory or environmental chambers.

2.1.4. Water used for rearing, holding, and testing organisms shall be reconstituted synthetic water, ground water, surface water, or dechlorinated tap water. Dechlorination shall be accomplished by carbon filtration, laboratory water conditioning units, or the use of sodium thiosulfate. After dechlorination, total residual chlorine shall be non-detectable. Sodium thiosulfate may be toxic to the test organisms, and if used for dechlorination,

paired controls with and without sodium thiosulfate shall be incorporated in effluent toxicity tests. Use of 3.6-mg (anhydrous) sodium thiosulfate will reduce 1.0-mg chlorine/L. After dechlorination, total residual chlorine shall be non-detectable.

2.1.5. A deionizing system providing 18 mega-ohm, laboratory grade water shall be provided with sufficient capacity for laboratory needs. If large quantities of high quality deionized water are needed, it may be advisable to supply the laboratory grade water deionizer with preconditioned water from a Culligen®, Continental®, or equivalent, mixed-bed water treatment system.

2.1.6. Air used for aeration shall be free of oil and fumes. Oil-free air pumps shall be used where possible. Particulates shall be removed from the air using BALSTON® Grade BX or equivalent filters (Balston, Inc., Lexington, Massachusetts), and oil and other organic vapors can be removed using activated carbon filters, (RALSTON®, C-l filter, or equivalent).

2.1.7. During rearing, holding, and testing, test organisms shall be shielded from external disturbances such as rapidly changing light conditions (especially salmonids) and pedestrian traffic.

2.1.8. Materials used for exposure chambers, tubing, etc., that come in contact with the effluent and dilution water shall be carefully chosen. Tempered glass and perfluorocarbon plastics (TEFLON®) shall be used whenever possible to minimize sorption and leaching of toxic substances, and may be reused after cleaning. Containers made of plastics, such as polyethylene, polypropylene, polyvinyl chloride, TYGON®, etc., may be used to ship, store, and transfer effluents and receiving waters, but they shall not be reused unless absolutely necessary, because they could carry over adsorbed toxicants from one test to another. However, these containers may be repeatedly reused for storing uncontaminated waters such as deionized or laboratory-prepared dilution waters and receiving waters. Glass or disposable polystyrene containers can be used as test chambers. The use of large (> 20 L) glass carboys is discouraged for safety reasons.

2.1.9. New plastic products shall be tested for toxicity before general use by exposing organisms to them under ordinary test conditions.

2.1.10. Equipment, which cannot be discarded after each use because of cost, shall be decontaminated. Fiberglass, in addition to the previously mentioned materials, can be used for holding and dilution water storage tanks, and in the water delivery system. All material shall be flushed or rinsed thoroughly with dilution water before using in the test.

2.1.11. Copper, galvanized material, rubber, brass, and lead shall not come in contact with holding or dilution water, or with effluent samples and test solutions. Some materials, such as neoprene rubber (commonly used for stoppers), may be toxic and shall be tested before use.

2.1.12. Silicone adhesive used to construct glass test chambers absorbs some organochlorine and organophosphorus pesticides, which are difficult to remove. Therefore, as little of the adhesive as possible shall be in contact with water. Extra beads of adhesive inside the containers shall be removed.

2.1.13. New plasticware used for effluent or dilution water collection or organism test chambers does not require thorough cleaning before use. It is sufficient to rinse new sample containers once with sample dilution water before use. New glassware shall be soaked overnight in 10% acid and rinsed well in deionized water and dilution water.

2.1.14. All non-disposable sample containers, test vessels, tanks, and other equipment that has come in contact with effluent shall be washed after use to remove surface contaminants.

2.1.15. All test chambers and equipment shall be thoroughly rinsed with the dilution water immediately prior to use in each test.

2.1.16. Water purification system- MILLIPORE® MILLI-Q®, MILLIPORE® QPAKTM2, or equivalent. Depending on the quantity of high-grade water needed, a first-stage pre-conditioner deionizer, such as a CULLIGEN® or CONTINENTAL® System, or equivalent, may be needed to provide feed water to the high-purity system.

2.2. **Instrumentation:** The Laboratory shall have sufficient analytical equipment and capability to meet all terms and conditions of this module and GR01, including all equipment requirements specified in base methods used to perform the analyses. At a minimum, the Laboratory shall have all of the following equipment and instruments operative at the time of the on-site evaluation and committed for the full duration of the subcontract:

2.2.1. Refrigerator

2.2.2. ASTM Type II water supply

2.2.3. Meters for pH, conductivity.

2.2.4. Ion Selective Electrode (ISE) for dissolved oxygen (DO), and ammonia

2.2.5. Autotitrator, or appropriate titration glassware, for hardness and alkalinity

2.3. **Key Position Requirements:** The Laboratory shall designate and use technical key positions listed below and in GR01 to perform the minimum functional requirements necessary to meet the terms and conditions of this subcontract. Minimum academic training and experience qualifications for positions specific to SS11 are identified below. All positions listed in GR01 Exhibit A Section 1, the Sample Custodian and Document Control Officer specified in GR01 Exhibit F Section 4, and all of the positions listed below are considered key positions for this subcontract.

2.3.1. <u>WET Classical Techniques Chemist Qualifications</u>

Responsibility:	Responsible for WET analysis procedures; maintenance of classical techniques, instrumentation and equipment; and review and approval of data.
Academic Training:	A minimum of a bachelor's degree in a science discipline.
Experience:	A minimum of three years of experience in performing pH, conductivity, dissolved oxygen (DO), ammonia, gravimetric, potentiometric, titrimetric, and turbidimetric analyses; and maintaining the instrumentation and equipment for these analyses.

2.3.2. WET Classical Techniques Analyst Qualifications

Responsibility:	Responsible for preparation and analysis of environmental samples.
Academic Training:	A minimum of a high school diploma and a college-level course in general chemistry.
Experience:	A minimum of one year of experience in an analytical laboratory with experience in sample preparation and analysis with WET analysis methods.

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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REPORTING AND DELIVERABLES REQUIREMENTS

1. INTRODUCTION

SS11 Exhibit B contains reporting and deliverables requirements applicable to all Whole Effluent Toxicity Parameter line item codes defined in SS11 Exhibit C. This exhibit supplements and augments the fundamental deliverable requirements specified in GR01 Exhibit B. Requirements for Sample Data Packages, Supporting Data Packages, and other deliverables specific to Whole Effluent Toxicity Parameters are detailed. Four tables in SS11, Exhibit B further define some of the required deliverables specified in GR01 Exhibit B.

Table B1. Schedule for Sample Data Package Deliverables
Table B2. Sample Data Package Deliverables
Table B3. Other Deliverables
Table B4. Supporting Documentation Package Deliverables

These tables define major deliverable components as *Deliverable Sections*, which are each assigned titles. Text accompanying the tables provides structural and content requirements. Tables and text also provide reference lists for Whole Effluent Toxicity Parameter deliverable requirements found in this module and other modules of this SOW.

SS11 Exhibit B also contains requirements for Document Inventories for Whole Effluent Toxicity Parameters.

Deliverable requirements and schedules specified in the General Laboratory Requirements Module, GR01, and its tables shall be met. However, where requirements and schedules contained in SS11 are in conflict with Module GR01, requirements in SS11 shall take precedence.

2. SAMPLE DATA PACKAGE DELIVERABLE REQUIREMENTS

The sample data package deliverable requirements for Whole Effluent Toxicity Parameters Priority and Rush processing differ from those specified in GR01. The priority and rush processing schedule for Whole Effluent Toxicity Parameters sample data package delivery is provided in Table B1.

2.1.1. All deliverables contained in Table B1 shall be transmitted to the CTR.

2.1.2. The final report shall be completed and in the Site's possession by the requested turn around time unless otherwise agreed upon in writing by the CTR.

TABLE B1 SCHEDULE FOR SAMPLE DATA PACKAGE DELIVERABLES

Processing	Item	Copies	Schedule	Reference
Priority: Acute	Quick-turn packet (Narrative, COC copies, and results only)	1	10 days after VTSR	SS11, Exhibit B/Section 3
	Sample data package	1	31 days after VTSR	SS11, Exhibit B/Section 2
Priority: Chronic	Quick-turn packet (Narrative, COC copies, and results only)	1	18 days after VTSR	SS11, Exhibit B/Section 3
	Sample data package	1	31 days after VTSR	SS11, Exhibit B/Section 2
Rush: Acute	Quick-turn packet (Narrative, COC copies, and results only)	1	5 days after VTSR	SS11, Exhibit B/Section 3
	Sample data package	1	14 days after VTSR	SS11, Exhibit B/Section 2
Rush: Chronic	Quick-turn packet (Narrative, COC copies, and results only)	1	9 days after VTSR	SS11, Exhibit B/Section 3
	Sample data package	1	14 days after VTSR	SS11, Exhibit B/Section 2

2.2. **Sample Data Package Components**: Table B2 lists the required deliverable sections for a Sample Data Package for Whole Effluent Toxicity Parameters. Each deliverable section is numbered and assigned a title. These *Deliverable Section Numbers* and *Titles* are referenced in the remainder of the accompanying text of this SS11 Exhibit B Section 2. The *Reference* column in Table B2 contains designators, which refer to modules, exhibits, and sections where more details may be found. This reference column is intended as an aid in locating requirements, but is not expected to be all-inclusive.

TABLE B2 SAMPLE DATA PACKAGE DELIVERABLES

Deliverable Section Number	Deliverable Section Title	Reference (Module, Exhibit/Section Letter or Title)
1	Sample Data Package Cover Page	GR01, Exhibit B/Section 4
2	Table of Contents	GR01, Exhibit B/Section 4
3	Data Review Checklist	GR01, Exhibit B/Section 4 SS11, Appendix A SS11, Exhibit B/Section 2
4	COC(s)	GR01, Exhibit B/Section 4 GR01, Exhibit F/Section 2

TABLE B2 SAMPLE DATA PACKAGE DELIVERABLES (continued)

Deliverable Section Number	Deliverable Section Title	Reference (Module, Exhibit/Section Letter or Title)
5	Narrative	GR01, Exhibit B/Section 4 SS11, Exhibit B/Section 2 SS11. Appendix A/Section 5
6	Sample and QC Result Summaries	GR01, Exhibit B/Section 4, 7, 8 SS11, Exhibit B/Section 2 SS11, Exhibit C SS11, Exhibit E/Section 4 SS11, Appendix A/B
7	Preparation Raw Data	GR01, Exhibit B/Section 4 GR01, Exhibit E/Section 5 GR01, Exhibit F/Section 4 SS11, Exhibit B/Section 2 SS11, Exhibit F/Section 4
8	Standards Summary	GR01, Exhibit B/Section 4 GR01, Exhibit E/Section 6 SS11, Exhibit E/Section 4
9	Instrument Raw Data	GR01, Exhibit B/Section 4 GR01, Exhibit F/Section 4 SS11, Exhibit B/Section 2 SS11, Exhibit F/Section 4

2.3. Sample Data Package General Requirements

2.3.1. All Sample Deliverable Sections shall meet the general and specific requirements listed in GR01 Exhibit B Section 4.

2.3.2. All Sample Deliverable Sections shall appear in the Sample Data Package in numerical order by *Deliverable Section Number*.

2.3.3. *Deliverable Section Numbers* 6 through 9 shall each be preceded by a Cover Sheet which is titled exactly as given under the *Deliverable Section Title* column of Table B2. These Cover Sheets shall be paginated along with the rest of the Sample Data Package. Each of these Deliverable Section Cover Sheets shall comply with the following structural requirements:

2.3.3.1. The Deliverable Section Title shall appear at the top.

2.3.3.2. If the locations of required items within the Sample Data Package differ from the specified location, then these discrepancies shall be mapped in table form on the deliverable section Cover Sheet for the specified location. This table shall include mapping information in columns labeled "Required Item," "Specified Location," and "Actual Location."

2.3.3.3. If identifiers for required items differ from specified identifiers, then these discrepancies shall also be mapped in table form on the Cover Sheet of the affected deliverable section. This table shall include mapping information in columns labeled "Specified Identifier" and "Identifier Used."

2.3.4. Any undocumented misplaced items shall be considered incomplete.

2.3.5. All raw data from failed analytical batches or from failed individual analyses shall be clearly labeled as "Data Not Used" and shall be included in the appropriate Deliverable Section.

2.3.6. Refer to GR01 Exhibit B and SS11 Exhibits D and E for additional requirements for Sample Data Packages.

2.4. Sample Data Package Cover Page Requirements

(Requirements for Sample Data Package Deliverable Section Number 1)

Sample Data Package Cover Pages shall be included as specified in GR01.

2.5. Table of Contents

(Requirements for Sample Data Package Deliverable Section Number 2)

A Table of Contents for the Sample Data Package shall be included as specified in GR01.

2.6. Data Review Checklist Requirements

(Requirements for Sample Data Package Deliverable Section Number 3)

Data Review Checklists document the completeness and the quality control status of the Sample Data Package. SS11 Appendix A contains the form, which shall be used to complete this check for Whole Effluent Toxicity Parameter analyses. A completed Data Review Checklist shall be submitted with each Sample Data Package and shall be in strict conformance with the formatting and content of the form contained in SS11 Appendix A. Refer to General Laboratory Requirements Module GR01 Exhibit B Section 4 for more details.

2.7. Chain of Custody (COC)

(Requirements for Sample Data Package Deliverable Section Number 4)

COC documentation shall be included in the Sample Data Package as specified in GR01.

2.8. Sample Data Package Narrative

(Requirements for Sample Data Package Deliverable Section Number 5)

2.8.1. Sample Data Package Narratives shall be included in the Sample Data Package as specified in GR01.

2.8.2. The narrative shall contain a table that lists the approved method used for each parameter analyzed.

2.8.3. Additional content requirements are listed in GR01 Exhibit B, SS11 Exhibit D, SS11 Appendix A, and in other SS11 exhibits.

2.9. Sample And QC Sample Results Summary

(Requirements for Sample Data Package Deliverable Section Number 6)

2.9.1. The sample and QC sample results summary shall include forms providing observed results and information required to verify calculations for Whole Effluent Toxicity Parameter QC analyses. SS11 Exhibit D contains many clarifications and protocols for data, which are reported in this section of the deliverable.

2.9.2. At a minimum, forms shall be included which provide summaries for all of the following items if applicable:

- Sample results for all parameters involved
- Initial and continuing calibration verification (ICV, CCV) results and recoveries
- Preparation blanks
- Initial and continuing calibration blank (ICB, CCB) results
- Spike sample results and recoveries
- Replicate sample results
- Laboratory Control Sample results and recoveries
- Sample Holding Time Summaries

2.10. Preparation Raw Data Deliverable Requirements

(Requirements for Sample Data Package Deliverable Section Number 7)

2.10.1. Sample preparation raw data shall be documented in the form of preparation bench sheets and/or preparation logs. These documents shall follow the requirements defined in SS11 Exhibit F Section 4 and the General Laboratory Requirements Module GR01 Exhibit F Section 4.

2.10.2. Preparation Logs shall be included in the deliverable in order by parameter as listed in SS11 Exhibit C Table C1.

2.10.3. These logs shall include the following if applicable:

- Analytical Batch identifier (see the definition of Analytical Batch in GR01 Exhibit G)
- Date and time of preparation
- Identifier for the laboratory SOP for the preparation performed
- Identifiers for all samples, sample QC, and QC solutions prepared
- Balance identifiers with dates of use or balance verification bench sheet so that all measurements can be traced to the documented balance verifications performed according to GR01 Appendix E Section 5.
- Initial and final weights and volumes for all samples and QC samples including gross weights and tare weights where applicable
- All bench sheets
- Dilutions
- Receipt Logs
- Recirculating System Water Logs
- Reconstituted Freshwater Logs
- Pipette identifiers and dates of use (if applicable)
- Comments describing any significant sample changes or reactions which occur during preparation and storage
- Signatures and dates of all analysts and reviewers

2.11. Standards Summary

(Requirements for Sample Data Package Deliverable Section Number 8)

Each Sample Data Package shall include documentation which lists identification numbers and expiration dates for all standards used for data reported in the Report Identification Number (RIN). This documentation shall include secondary standard identification numbers for all secondary standards

used for calibration, calibration verifications, matrix spikes, and laboratory control solutions. Primary standards used as received shall be identified by their unique identifier. The standards summary documentation included in the Sample Data Package shall also include identifiers for all primary standards used to make secondary standards. These identifiers shall be traceable to standard and reference materials certificates included in the Supporting Documentation Package (see SS11 Exhibit B Section 5).

2.12. Instrument Raw Data

(Requirements for Sample Data Package Deliverable Section Number 9)

For each reported value, the Laboratory shall include all raw data from each instrument used to obtain reported values for all samples and QC solutions. These raw data shall include instrument readouts and run logs. Raw data shall contain all instrument readouts used for the sample results, including those readings that may fall below the Required Detection Limit (RDL). All instruments shall provide a legible hard copy of the direct real-time instrument readout. Numerical readouts shall be printed so that actual calculated or raw values are printed for those readings falling below zero. Originals of the chronological instrument readout shall be included. (Copies of original documents may be substituted if the original data were previously submitted with another RIN.)

2.12.1. Raw Data shall be included in order by parameter as listed in SS11 Exhibit C Table C1.

2.12.2. Raw data shall meet labeling and other requirements defined in SS11 Exhibit F Section 4 and the GR01 Exhibit F Section 4. All of the following items shall be included in the instrument raw data or in run logs.

- Calibration data, including instrument readouts, slopes, intercepts, correlation coefficients
- All sample data
- Effluent dilutions
- Contents of reconstituted water
- Initial and continuing calibration blanks
- Initial and continuing calibration verification standards
- Laboratory spikes, duplicates, and laboratory control samples
- Retention times for ion chromatography, if applicable
- Autosampler positions, if applicable
- Instrument identifier, instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation.
- Time and date of each analysis. If the instrument output cannot be set to automatically provide the time of analysis, the dates and times of instrument calibration/standardization and final calibration verification shall be recorded.
- Dilutions and volumes applied during the instrument analysis.

3. ELECTRONIC DATA DELIVERABLE

An electronic data deliverable is not required for PSA Module SS11.

4. REQUIREMENTS FOR OTHER DELIVERABLES

The following table defines other required deliverables in addition to the Sample Data Package deliverables for Whole Effluent Toxicity Parameters.

TABLE B3 OTHER DELIVERABLES

Deliverable Title	Schedule	Recipient	Reference (Module, Exhibit/Section)
Performance Evaluation Sample Analysis Results	7 days from receipt of results. Minimum frequency quarterly.	CTR	GR01, Exhibit B/Sections 2, 9 GR01, Exhibit E/Section 9 SS11 Exhibit E/Section 7

5. SUPPORTING DOCUMENTATION PACKAGE REQUIREMENTS

5.1. **Support Package Schedules and Maintenance**: See GR01 Exhibit B Section 5 for delivery schedules and maintenance requirements for Supporting Documentation Packages (Support Packages).

5.2. **Support Package Components:** Table B4 lists required components for a Support Package for Whole Effluent Toxicity Parameters. Each section is assigned a title. These titles are referenced in the remainder of the accompanying text in SS11 Exhibit B Section 5. The *Reference* column in Table B4 refers to the module, exhibit, and section number where more details may be found. The information in this column is intended as an aid in locating specifics, but may not be all-inclusive. Refer to Exhibits B, E, and F of the General Laboratory Requirements Module GR01 for details about these requirements.

TABLE B4 SUPPORTING DOCUMENTATION PACKAGE

Deliverable Section Title	Reference (Module, Exhibit/Section Number)
Document Inventory	SS11, Exhibit B/Section 4
Sample receipt, storage, tracking and internal COC records	GR01, Exhibit F/Sections 2, 3 SS11, Exhibit D/Section 3 SS11, Exhibit F/Section 3
Copy of Sample Data Package	GR01, Exhibit B/Sections 3, 4, 5 SS11, Exhibit B/Sections 3, 4
Original SRM Certificates	GR01, Exhibit E/Section 6 SS11, Exhibit D/Section 4
Original logs and/or logbooks	GR01, Exhibit F/Section 4
Standard Operating Procedures	GR01, Exhibit F/Section 6 SS11, Exhibit F/Section 5
Software Quality Assurance	GR01, Exhibit F/Section 5 SS11, Exhibit E/Section 2

5.3. **Document Inventory Requirements:**

5.3.1. The Document Inventory shall include a list of all documents in the Support Package file and the locations of all documents that are not physically in the Support Package file, but are listed in Table B4.

5.3.2. The Document Inventory shall appear at the beginning of the Support Package file and shall be the first item in the Support Package. All items listed in the Document Inventory shall be titled exactly as given under the "Deliverable Section Title" column of Table B4. All other items listed in Table B4 may follow in any order, however, the other documents shall appear in the order listed on the Laboratory's Document Inventory.

5.3.3. See GR01 Module B Section 5 for additional Document Inventory requirements for Supporting Data Packages.

5.4. **Support Package Internal COC Requirements**: The Support Package shall include sample receipt, storage, tracking, and internal COC records required in GR01 Exhibit F Sections 2 and 3.

5.5. **Support Package Copy of the Sample Data Package**: The Support Package shall include a photocopy of the Sample Data Package.

5.6. **Support Package Original SRM Certificate Requirements**: The Support Package shall include the originals of all standard certificates required for meeting GR01 Exhibit E Section 6 and SS11 Exhibit E Section 3. It is expected these originals will be included by referencing their location on the Document Inventory.

5.7. **Support Package Logs and Logbook Requirements**: Logs and/or logbooks include, but are not limited to, preparation logs, instrument run logs, standard dilution logs, balance calibration logs, pipette calibration logs, instrument maintenance logs, and ASTM II water logs shall all be included in the Support Package. (It is expected that many of these items will be included by referencing their location on the Document Inventory.)

5.8. **Support Package Standard Operating Procedures**: The Support Package shall include all SOPs required for meeting SS11 Exhibit F Section 5. (It is expected these procedures will be included by referencing their identifiers and location on the Document Inventory.)

5.9. **Support Package Software Quality Assurance**: The Support Package shall include Software Quality Assurance Documentation required by GR01, Exhibit F Section 5. (It is expected that this information will be included by referencing its location on the Document Inventory.)

EXHIBIT C

WHOLE EFFLUENT TOXICITY PARAMETERS

AND

REQUIREMENTS

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1. WHOLE EFFLUENT TOXICITY PARAMETER REQUIREMENTS LISTs..... C-2

1. WHOLE EFFLUENT TOXICITY PARAMETER REQUIREMENTS LISTS

Table C1 specify required parameter identifiers, required method sources and method type descriptions for each WET line item code.

Table C1 WHOLE EFFLUENT TOXICITY PARAMETERS

Line Item Codes	Parameter Identifier	Parameter Name	Approved Methods
SS11B001	WET-A-1	Whole Effluent Toxicity-Acute (Two Species)	EPA 600/4-90/027F
SS11B002	WET-C-1	Whole Effluent Toxicity-Chronic (Two Species)	EPA 600/4-91/002
By Task Order*	WET-T-1	Whole Effluent Toxicity TRE/TIR	EPA 600/6-91/005F 600/R-92/081

* Should acute or chronic toxicity be detected, task orders may be issued to the subcontractor by the CTR to characterize and/or identify the toxicant(s) as part of the TRE/TIR.

EXHIBIT D

ANALYTICAL METHODS

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ANALYTICAL METHODS

1. INTRODUCTION

This Exhibit contains the analytical methods requirements for Whole Effluent Toxicity Parameter analysis. The purpose of this section is to provide specific guidance for analyses specified in SS11 Exhibit C. This exhibit is not intended to be a comprehensive requirements document, rather it is intended to clarify and supplement requirements found in approved method sources. In many cases, these instructions apply specific protocols to requirements and recommendations of approved method sources. The objective is to increase uniformity between requirements of different approved method sources while still meeting or exceeding minimum method requirements

2. METHOD SELECTION AND APPLICATION

2.1. **Selection of Base Method**: Methods used for analysis shall meet the following requirements:

2.1.1. Methods shall be selected from Standard Methods for the Examination of Water and Wastewater, approved EPA methods, EPA 600/4-90/027F, EPA 600/4-91/002 and EPA/600/R-92/081 or procedures set out in 40 CFR 136 for acute and chronic toxicity.

2.1.2. Routine chemical and physical analyses for culture and dilution water, food, and test solutions, shall include established quality assurance practices outlined in Agency methods manuals (USEPA, 1979a; USEPA 1979b).

2.1.3. Reagent containers shall be dated when received from the supplier, and the shelf life shall not be exceeded. Also, working solutions shall be dated when prepared, and the recommended shelf life shall be observed.

2.1.4. The Laboratory shall conduct an acute 48-hour static toxicity test using *Ceriodaphnia sp.* and an acute 96-hour static toxicity test using fathead minnows.

2.1.5. Chronic toxicity test shall be conducted in general accordance with the above procedures and the latest revision of associated guidance documents.

2.1.6. Should acute or chronic toxicity be detected a Toxicity Reduction Evaluation (TRE)/Toxicity Identification Evaluation (TIE) may be undertaken to establish the cause of the toxicity, locate the source(s) of the toxicity and develop control of, or treatment for the toxicity. The CTR must authorize implementation of the TRE and/or TIE.

2.1.7. Sample pretreatment, digestion, and analysis procedures shall be appropriate to the regulatory requirements.

2.1.8. The method employed shall be from the Approved Method Source and shall comply with all other requirements.

2.2. **Interpretation of Base Method Requirements**: Analyses performed shall meet requirements of the methods selected from the Approved Method Source.

2.2.1. The Laboratory shall be in strict compliance with all requirements in the these methods which are stated as 'shall.'

2.2.2. Additional method requirements and quality assurance measures are listed in SS11 Exhibits D and E. In the event method requirements and quality assurance measures in approved methods sources appear to be less stringent than those specified in SS11 Exhibits D or E, the requirements of SS11 Exhibits D and E shall prevail.

2.3. **EPA-600 Methods:** For the analysis of samples specifying EPA methods, the methods shall be chosen from the most-recently promulgated revision of the EPA Methods.

2.4. **Base Method Modifications:** Standard methods may be modified or alternative methods substituted only with the written consent of the CTR.

3. SAMPLE HOLDING TIMES AND PRESERVATION REQUIREMENTS

3.1. **Holding Times:** Sample holding time begins when the last grab sample in a series is taken (i.e., when a series of four grab samples are taken over a 24-h period), or when a 24-h composite sampling period is complete. Since the data from the samples are to be acceptable for use in the NPDES Program, the lapsed time (holding time) from sample collection to first use of the sample in test initiation shall not exceed 36 hours. In no case shall more than 72 hours elapse between collection and first use of the sample.

3.2. **Sampling:** To minimize the loss of toxicity due to volatilization of toxic constituents, all sample containers shall be completely filled, leaving no air space between the contents and the lid.

3.3. **Preservation:** Samples collected for off-site toxicity testing are to be chilled to 4 degrees C during or immediately after collection, and shipped iced to the performing laboratory. Sufficient ice shall be placed with the sample in the shipping container to ensure that the ice will be present when the sample arrives at the Laboratory and is unpacked. Insulating material shall not be placed between the ice and the sample in the shipping container.

3.4. **Containers:** Samples should be collected and shipped in one or more 4-L (1-gal) CUBITAINERS or new plastic “milk” jugs. All sample containers shall be rinsed with source water before being filled with sample.

3.5. **Temperature:** The temperature of the samples shall be checked immediately upon their receipt at the lab. If the temperature is 10 degrees C or greater the CTR shall be contacted immediately.

3.5.1. If samples are not immediately used for testing, they shall be kept under refrigeration at 4 degrees C until they are warmed prior to test preparation.

4. GENERAL QUALITY REQUIREMENTS

The following requirements apply to all determinations regardless of the Approved Method Source:

4.1. **Quality Assurance Practices.** QA practices within the laboratory shall address all the activities that affect the quality of the final effluent toxicity data, such as: (1) effluent sampling and handling; (2) the source and condition of the test organisms; (3) condition and operation of equipment; (4) test conditions; (5) instrument calibration; (6) replication; (7) use of reference toxicants; (8) record keeping: and (9) data evaluation.

Guidance for the evaluation of laboratories performing toxicity tests and laboratory evaluation criteria may be found in USEPA (1991a).

4.2. **Minimum QC Requirements:** Analyses shall be performed with the minimum QC requirements of the base methods and the requirements listed in SS11 Exhibit D. When there is a conflict between SS11 QC requirements, the requirements of the base methods take precedence.

4.3. **Measurement Range:** All results shall be taken from measurements made within the calibration curve of the instrument. All analyzed solutions with readings exceeding the highest calibration standard shall be diluted and reanalyzed.

4.4. **Cleaning protocols:** Glassware cleaning shall be performed in accordance with EPA guidelines for test chambers and laboratory apparatus. Cleaning protocols for new and used labware shall meet all requirements specified in base methods.

4.5. **Reference Toxicant Testing:** In house culture organisms shall be tested at least monthly in a reference toxicant test. Each batch of animals received from outside commercial sources shall be tested against a reference toxicant. All test results shall be used to update control charts maintained for each organism.

4.6. **Hazardous Additions:** If separation techniques are required for reduction of matrix interference, these techniques shall not introduce hazardous constituents over and beyond those which may be inherent to the sample, when alternative separation methods are available.

4.7. **Standard Traceability**: Standard documentation shall be maintained as stated in GR01 Exhibit B Section 4.

4.8. **Laboratory Water Used For Culturing and Test Dilution Water:** The quality of water used for test organism culturing and for dilution water used in toxicity tests is extremely important. Water for these two uses shall come from the same source. The dilution water used in effluent toxicity tests will depend in part on the objectives of the study and logistical constraints. For tests performed to meet NPDES objectives, synthetic, moderately hard water shall be used. The dilution water used for internal qua1ity assurance tests with organisms, food, and reference toxicants shall be the water routinely used with success in the laboratory. Water used for culturing and test dilution shall be analyzed for toxic metals and organics at least annually or whenever difficulty is encountered in meeting minimal acceptability criteria for control survival, and/or reproduction or growth. The concentration of the metals, Al, As, Cr, Cu, Fe, Pb, Ni, Zn, expressed as total metal, shall not exceed 1 µg/L each, and Cd, Hg, and Ag, expressed as total metal, shall not exceed 100 ng/L each. Total organochlorine pesticides plus PCBs shall be less than 50 ng/L.

4.9. **Test Organisms:** The organisms shall appear healthy, behave normally, be eating well, and have low mortality in cultures, during holding, and in test controls. Test organisms shall be positively identified to species.

4.10. **Test Conditions:** The temperature of test solutions shall be measured by placing the thermometer or probe directly into the test solutions, or by placing the thermometer in equivalent volumes of water in surrogate vessels positioned at appropriate locations among the test vessels. Test solution temperatures shall be maintained within the limits specified for each test. DO concentration and pH in test chambers shall be checked daily throughout the test period.

4.11. **Quality Of Test Organisms:** Where acute or short-term chronic toxicity tests are performed with effluents or receiving waters using test organisms obtained from outside the test laboratory, concurrent toxicity tests of the same type shall be performed with a reference toxicant, unless the test organism supplier provides control chart data from at least the last five monthly acute toxicity tests using the same reference toxicity and test conditions.

4.11.1. The supplier shall also certify the species identification of the test organisms, and provide the taxonomic reference (citation and page) or name(s) of the taxonomic expert(s) consulted.

4.11.2. If the laboratory performing toxicity tests maintains its own stock cultures, the sensitivity of the offspring shall be determined in a toxicity test performed with a reference toxicant at least once each month. If preferred, this reference toxicant test may be performed concurrently with each effluent toxicity test. However, if a given species of test organism produced by in house cultures is used only monthly, or less frequently, in effluent toxicity tests, a reference toxicant test shall be performed concurrently with effluent toxicity test.

4.11.3. If a routine reference toxicant test fails to meet acceptability criteria, the test shall be immediately repeated. If the failed referenced toxicant test was being performed concurrently with an effluent or receiving water toxicity test, both tests shall be repeated.

4.12. **Food Quality:** The nutritional quality of the food used in culturing and testing fish and invertebrates is an important factor in the quality of the toxicity test data. This is especially true for the unsaturated fatty acid content of brine shrimp nauplii, *Artemia*.

4.12.1. Problems with the nutritional suitability of the food will be reflected in the survival, growth, and reproduction of the test organisms in cultures and toxicity tests. If a batch of food is suspected to be defective, the performance of organisms fed with the new food can be compared with the performance of organisms fed with a food of known quality in side-by-side tests. If the food is used for culturing, its suitability shall be determined using a short-term chronic test which will determine the affect of food quality on growth or reproduction of each of the relevant test species in culture.

4.12.2. New batches of food used in culturing and testing shall also be analyzed for toxic organics and metals or whenever difficulty is encountered in meeting minimum acceptability criteria for control survival and reproduction or growth. If the concentration of total organic chlorine exceeds 0.15 µg/g wet weight, or the total concentration of organochlorine pesticides plus PCBs exceeds 0.30 µg/g wet weight, or toxic metals exceed 20 µg/g wet weight, the food shall not be used (for analytical methods see AOAC, 1990; USDA, 1989).

4.13. **Acceptability of Acute And Chronic Toxicity Test Results**: For the test results to be acceptable for Acute Toxicity, control survival shall equal or exceed 90%. For the test results to be acceptable for Chronic Toxicity, control survival shall equal or exceed 80%**.**

4.14. **Whole Effluent Toxicity Testing- (TIE/TRE):** Should acute or chronic toxicity be detected in the discharge, a Toxicity Reduction Evaluation (TRE)/Toxicity Identification Evaluation (TIE) shall be undertaken by permission of the CTR to establish the cause of the toxicity, locate the source(s) of the toxicity, and develop control of, or treatment for the toxicity. Failure to initiate, or conduct an adequate TIE/TRE, or delays in the conduct of such tests, shall not be considered a justification for noncompliance with the whole effluent toxicity limits.

4.14.1. If a problem is established, and if prior knowledge or intuition is not helpful, initial efforts shall be expended on characterization and identification of the toxicant(s). Procedures exist for rapidly narrowing the possibilities to certain groups of pollutants such as metals, non-polar organics, oxidants, etc. In many cases, it is anticipated that the TRE may essentially terminate at this point if it is conclusively shown that the problem is due to one

distinct pollutant whose source, and method of correction, is known. This pollutant may be already controlled through a compliance schedule linked to a numerical limit.

4.14.2. Once the problem has been identified and located, the ultimate objective is to eliminate it by process controls, pretreatment, combined waste stream treatment, local enforcement, or whatever other measure may be necessary.

4.14.3. It is anticipated that toxicity problems will be controlled in most cases by following EPA 600/6-91/003, EPA/600/R-92/080, EPA/600/R-92/081, and EPA 600/6-91/005F.

5. REQUIREMENTS FOR CALIBRATION AND STANDARDIZATION

5.1. **Instruments:** Instruments used for routine measurements of chemical and physical parameters such as pH, DO, temperature, conductivity, salinity, alkalinity, and hardness, shall be calibrated and standardized prior to use each day according to the instrument manufacturer's procedures as indicated in the general section on quality assurance (see EPA Methods 150.1, 360.1; 170.1, and 120.1; USEPA, I 979b). Calibration data shall be recorded in a permanent log.

5.2. **Calibration Solutions:** Concentrations of preparation and preservation reagents in calibration standards and prepared samples shall be identical. The Laboratory shall introduce calibration solutions as described in the individual methods and record the readings.

5.3. **Wet Chemical Methods:** Wet chemical methods used to measure hardness, alkalinity, and total residual chlorine, shall be standardized prior to use each day according to the procedures for those specific EPA methods (see EPA Methods 130.2 and 310.1; USEPA 1979b).

5.4. **Titrimetric Standardization**

5.4.1. Titrants shall be standardized monthly and the Laboratory shall provide the last date of titrant standardization on the bench-sheet. Titrant concentration values shall be traceable to standardization data included in the Supporting Documentation Package (see SS11 Exhibit B, Section 5).

5.4.2. For alkalinity by electrometric endpoint, the pH probe shall be calibrated using two pH buffers (four and seven). The calibration shall be documented on the bench sheet and provided as part of the data package deliverable.

5.4.3. For all other titrimetric determinations, the Laboratory shall calibrate the instrument according to the specifications provided by the instrument manufacturer.

5.5. **pH Standardizations**

5.5.1. Calibration of the pH meter and electrode system shall consist of a two-point calibration per the instrument manufacturer’s instructions. The two-point calibration shall bracket the sample pH for pH measurement greater than 4 and less than 10. Standard pH buffer temperatures during calibration, and the slope and offset values of the calibration curve shall be recorded on the pH Logsheet. If the absolute value of the difference between the measured and true pH buffer values is greater than 0.1 S.U., the pH meter shall be recalibrated.

5.6. **Conductivity Standardizations**

5.6.1. For conductivity measurements, standardize the instrument with a 0.01 molar standard potassium chloride solution at 25°C per the instrument manufacturer's instructions. If a difference between the observed value and the standard value of less than 1% can not be obtained, clean the conductivity cell and recalibrate.

5.7. **Dissolved Oxygen Standardizations**

5.7.1. For dissolved oxygen (DO) measurements, calibrate the instrument per the instrument manufacturer's instructions.

6. REQUIREMENTS FOR CALIBRATION VERIFICATIONS

6.1. **Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)**

6.1.1. Immediately after calibration of each system, the accuracy of the initial calibration shall be verified and documented for every parameter by the analysis of the Initial Calibration Verification (ICV) solution(s).

6.1.2. A Continuing Calibration Verification (CCV) solution shall be analyzed and reported for each parameter, at a minimum frequency of one in every 20 measurements and after the last analytical sample.

6.1.3. The parameter concentrations in the ICV and CCV standards shall be at or near the midpoint of the calibration curve. The same standard may be used for both CCV and ICV. Concentrations of preparation and preservation reagents in CCV and ICV standards and prepared samples shall be similar.

6.1.4. A NIST-certified solution (or equivalent) shall be used for the ICV and CCV solutions. If certified ICV and CCV solutions are not available from any source, the ICV and CCV shall be prepared using an independent standard at a concentration other than that used for instrument calibration and near the midpoint of the calibration curve. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

6.1.5. ICV and CCV measurements shall reflect the analysis conditions (duration of analysis, rinses, and other related operations) of all associated analytical samples.

6.1.6. When the ICV percent recovery (%R) is outside the control limits of 85% to 115%, the analysis shall be stopped, the problem corrected, and the instrument recalibrated.

6.1.7. If the CCV percent recovery (%R) is outside the control limits of 85% to 115%, the analysis shall be stopped, the problem corrected, the instrument recalibrated, and affected samples reanalyzed. (Affected samples are defined as all samples analyzed since the last 'in control' ICV or CCV.)

6.2. **ICV & CCV For pH:** Calibration verification of the pH meter and electrode system shall consist of the analysis of a standard pH buffer near the mid-point of the calibration line. The temperature of the standard buffer at the time of analysis shall be recorded on the pH logsheet or benchsheet. If the absolute value of the difference between the measured and true pH buffer values is greater than 0.10 S.U., the calibration shall be repeated and affected samples reanalyzed. (Affected samples are defined as all samples analyzed since the last 'in control' ICV or CCV.)

6.3. **ICV & CCV For Conductivity:** A Calibration verification shall be performed with at least one standard potassium chloride solution other than the 0.01M KCl standard solution used for standardization. When possible, two standard KCl solutions should be analyzed that bracket the sample conductivity. If percent recovery (%R) is outside the control limits of 95% to105%, for either standard, the instrument shall be recalibrated and affected samples reanalyzed. (Affected samples are defined as all samples analyzed since the last 'in control' ICV or CCV.)

6.4. **Gravimetric Verifications:** For gravimetric determinations, the Laboratory shall provide the analytical balance standardization verification results for the balance check weight measurements prior to (ICV) and following (CCV) analytical batch measurements. The standardization verifications shall meet all requirements of GR01 Exhibit E Section 5.

7. DILUTION WATER

7.1. **Types of Dilution Water:** The type of dilution water used in effluent toxicity tests will depend largely on the objectives of the study:

7.1.1. If the objective of the test is to estimate the absolute acute toxicity of the effluent, which is a primary objective of NPDES permit-related toxicity testing, a synthetic (standard) dilution water is used. If the test organisms have been cultured in water which is different from the test dilution water, a second set of controls, using culture water, shall be included in the test.

7.1.2. If the objective of the test is to estimate the acute toxicity of the effluent in uncontaminated receiving water, the test may be conducted using dilution water consisting of a single grab sample of receiving water (if non-toxic), collected either upstream and outside the influence of the outfall, or with other uncontaminated natural water (ground or surface water) or standard dilution water having approximately the same characteristics (hardness and/or salinity) as the receiving water. Seasonal variations in the quality of surface waters may affect effluent toxicity. Therefore, the hardness of fresh receiving water and the salinity of saline receiving water samples shall be determined before each use. If the test organisms have been cultured in water which is different from the test dilution water, a second set of controls, using culture water, shall be included in the test.

7.1.3. If the objective of the test is to determine the additive or mitigating effects of the discharge on already contaminated receiving water, the test is performed using dilution water consisting of receiving water collected immediately upstream or outside the influence of the out fall. A second set of controls, using culture water, shall be included in the test.

7.2. **Standard, Synthetic Dilution Water:** Standard, synthetic dilution water is prepared with deionized water and reagent grade chemicals or mineral water. The source water for the deionizer can be natural water, or tap water.

7.2.1. Deionized water is obtained from a MILLIPORE® MILLI-Q® MILLIPORE® QPAKTM2, or equivalent system. It is advisable to provide a preconditioned (deionized) feed water by using a Culligan®, Continental®, or equivalent, system in front of the Ml LLI PORE® System to extend the life of the MILLIPORE® cartridges.

7.2.2. The recommended order of the cartridges in a four-cartridge deionizer (i.e., MILLI-Q® System or equivalent) is (1) ion exchange, (2) ion exchange, (3) carbon, and (4) organic

cleanup (such as ORGANEX-Q®, or equivalent), followed by a final bacteria filter. The QPAKTM2 water system is a sealed system which does not allow for the rearranging of the cartridges. However, the final cartridge is an ORGAN EX-Q® filter, followed by a final bacteria filter. Commercial laboratories using this system have not experienced any difficulty in using the water for culturing or testing.

7.3. **Use Of Receiving Water As Dilution Water**: If the objectives of the test require the use of uncontaminated surface water as dilution water, and the receiving water is uncontaminated, it may be possible to collect a sample of the receiving water close to the out fall, but upstream from or beyond the influence of the effluent. However, if the receiving water is contaminated, it may be necessary to collect the sample in an area "remote" from the discharge site, matching as closely as possible the physical and chemical characteristics of the receiving water near the out fall.

7.3.1. The sample should be collected immediately prior to the test, but never more than 96 h before the test begins. Except where it is used within 24 h, or in the case where large volumes are required for flow-through tests, the sample should be chilled to 4 degrees C during or immediately following collection, and maintained at that temperature prior to use in the test.

7.3.2. In the case of freshwaters, the regulatory authority may require that the hardness of the dilution water be comparable to the receiving water at the discharge site. This requirement can be satisfied by collecting uncontaminated surface water with a suitable hardness or adjusting the hardness of otherwise suitable surface water by addition of reagents.

7.4. **Use Of Tap Water As Dilution Water:** The use of tap water as dilution water is discouraged unless it is dechlorinated and fully treated. Deionization, carbon filtration, or the use of sodium thiosulfate can dechlorinate tap water. Use of 3.6-mg/L (anhydrous) sodium thiosulfate will reduce 1.0-mg chlorine/L (APHA, 1992, p.4-36). Following dechlorination, total residual chlorine shall not exceed 0.01 mg/L. Because of the possible toxicity of thiosulfate to test organisms, a control containing the same amount of sodium thiosulfate used to dechlorinate the effluent shall be included in toxicity tests utilizing thiosulfate-dechlorinated water. A second “blank” control shall also be included.

7.4.1. To be adequate for general laboratory use following dechlorination, the tap water is passed through a deionizer and carbon filter to remove toxic metals and organics, and to control hardness and alkalinity.

7.5. **Dilution Water Holding:** A given batch of dilution water shall not be used for more than 14 days following preparation because of the possible build-up of bacterial, fungal, or algal slime growth and the problems associated with it. The container shall be kept covered and the contents protected from light.

8. REQUIREMENTS FOR LABORATORY DUPLICATES

8.1. **Laboratory Duplicate Sample Frequency:** The frequency of preparation and analysis of duplicate samples shall meet all requirements of base methods. At a minimum, one laboratory duplicate is required in each analytical batch (maximum batch size is twenty samples). Laboratory duplicates are required for each matrix or waste type within a batch. A duplicate sample is to be analyzed with each analytical batch regardless of whether sample preparation is required.

8.2. **Duplicate Sample Analysis**

8.2.1. Laboratory duplicates shall be designated before sample treatment or preparation begins. The laboratory duplicate shall be subjected to the same sample preparation, analytical methods and QA/QC procedures employed for the Site samples. Regardless of whether sample preparation is required, a duplicate sample is to be analyzed with each analytical batch.

8.2.2. Client samples identified as blanks cannot be used for duplicate sample analysis. The Site may require that a specific sample be used for the duplicate sample analysis.

8.3. **Laboratory Duplicate Agreement For Titrimetric, Potentiometric(ISE), Gravimetric, andTurbidimetric Techniques**

8.3.1. The results of the sample and duplicate of the sample shall be within a Relative Percent Difference (RPD) of 20% for sample concentrations greater than five times the analyte RDL or the absolute value of the difference between the sample and duplicate results shall be less than the RDL for sample concentrations less than five times the RDL.

8.3.2. If the RPD of a sample and duplicate involving sample preparation is not within the above control limits, the analyses shall be terminated, the problem corrected, and the samples associated with the Laboratory Duplicate reanalyzed (after preparation of another analytical batch, if necessary).

8.3.3. If the deviation of a sample and duplicate not involving sample preparation is not within the above control limits, the analyses shall be terminated, the problem corrected, and the analytical batch reanalyzed.

8.4. **Laboratory Duplicate Agreement For pH:** If the absolute value of the difference between the sample and duplicate sample pH values is greater than 0.10 S.U., the problem shall be corrected and the analysis of all affected samples repeated.

8.5. **Laboratory Duplicate Agreement Conductivity:** If the RPD between sample and duplicate sample conductivity values is greater than 5% the problem shall be corrected and the analysis of all affected samples repeated. (RPD is calculated as directed in SS11 Appendix B WQP Form 5 instructions.)

9. REQUIREMENTS FOR LABORATORY CONTROL SAMPLES

9.1. **Laboratory Control Sample Analysis For All Techniques**

9.1.1. A Laboratory Control Sample (LCS) shall be analyzed for each analyte using the same sample preparation, analytical methods and QA/QC procedures employed for Site samples. Regardless of whether sample preparation is required, a LCS is to be analyzed with each analytical batch for each analytical method and parameter.

9.1.2. The Laboratory shall obtain standards traceable to NIST (or equivalent). If such certified standards are not available, the LCS may be prepared using stock certified standards (salts) in ASTM Type II water, or standard grade soil at a different concentration and from a source independent from the ICV/CCV standard.

9.1.3. Documentation and reporting of LCS sources and concentrations shall meet all requirements of GR01 Exhibit E Section 6.

9.2. **Laboratory Control Sample Recovery For Potentiometric (ISE), Titrimetric, Gravimetric, andTurbidimetric Techniques**

9.2.1. The LCS concentration shall be within the limits of the calibration curve. For an analytical batch with results determined with autodilution, a LCS also requiring dilution shall be analyzed similar to the samples in the analytical batch.

9.2.2. If the %R of an LCS involving sample preparation is not within the control limits of 80% to 120%, the analyses shall be terminated, the problem corrected, and samples associated with that LCS reprepared and reanalyzed.

9.2.3. If the %R of an LCS not involving sample preparation is not within the control limits of 80% to 120%, the analyses shall be terminated, the problem corrected, and the analytical batch reanalyzed.

9.3. **Laboratory Control Sample Recovery For pH:** If the absolute value of the difference between the found and true LCS values is greater than 0.10 S.U., the calibration shall be repeated. No data associated with the out-of tolerance LCS may be reported.

9.4. **Laboratory Control Sample Recovery For Conductivity:** If the %R of an LCS is not within the control limits of 95% to 105%, the instrument shall be recalibrated. No data associated with the out-of tolerance LCS may be reported.

EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

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QUALITY ASSURANCE /QUALITY CONTROL REQUIREMENTS

1 INTRODUCTION

The purpose of this Exhibit is to describe the minimum QA/QC operations necessary to satisfy the analytical requirements associated with the determination of the Whole Effluent Toxicity Parameters analysis. These operations and those in the General Laboratory Requirements Module GR01 are designed to ensure the generation of comparable data from all laboratories. These requirements do not release the Laboratory from maintaining its own QC checks on method and instrument performance.

2 QUALITY ASSURANCE PLAN

Requirements for the quality assurance plan are specified in GR01 Exhibit E.

3 ANALYTICAL STANDARDS AND REAGENTS REQUIREMENTS

3.1 **Multi-parameter Standards**: Primary standards used for multi-parameter secondary standards shall include additional certification statements or tables quantifying or verifying the absence of all other parameters included in the multi-element standards.

3.2 **General Requirements**: Additional requirements for the analytical standards and reagents are specified in Exhibit E of the General Laboratory Requirements Module, GR01.

4 METHOD SPECIFIC QC REQUIREMENTS

4.1 **Default Protocols:** Where base methods do not require specific QC protocols, laboratory SOPs shall specify requirements which meet or exceed parallel requirements found in this SOW.

4.2 **General Requirements**: Other method specific QC requirements have been included in SS11 Exhibit D.

5 MEASURING AND TESTING EQUIPMENT REQUIREMENTS

Requirements for measuring and testing equipment are specified in GR01.

6 DATA MANAGEMENT

Requirements for site data management are specified in GR01.

7 LABORATORY EVALUATION SAMPLES

7.1 **Demonstrating Acceptable Laboratory Performance:** It is a laboratory's responsibility to demonstrate its ability to obtain consistent, precise results with reference toxicants before it performs toxicity tests with effluents for permit compliance purposes. To meet this requirement, the intra-laboratory precision, expressed as percent coefficient of variation (CV%), of each type of test to be used in a laboratory shall be determined by performing five or more tests with different batches of test organisms, using the same reference toxicant, at the same concentrations, with the same test conditions (i.e., the same test duration, type of dilution water, age of test organisms, feeding, etc.), and same data analysis methods. A reference toxicant concentration series (0.5 or higher) shall be selected that will consistently provide partial mortalities at two or more concentrations.

7.2 **Documenting Ongoing Laboratory Performance:** Satisfactory laboratory performance is demonstrated by performing at least one acceptable test per month with a reference toxicant for each toxicity test method commonly used in the laboratory. For a given test method, successive tests shall be performed with the same reference toxicant, at the same concentrations, in the same dilution water, using the same data analysis methods. Precision may vary with the test species, reference toxicant, and type of test.

7.3 **Control Charts:** A control chart shall be prepared for each combination of reference toxicant, test species, test condition, and end-point. Toxicity endpoints from five or six tests are adequate for establishing the control charts. In this technique, a running plot is maintained for the toxicity values from successive tests with a given reference toxicant. Endpoints (LC50s) are examined to determine if they are within prescribed limits. The types of control charts illustrated (see USEPA, 1979) are used to evaluate the cumulative trend of results from a series of samples. The mean and upper and lower control limits ($\pm$ 2S) are re-calculated with each successive test result. After two years of data collection, or a minimum of 20 data points, the control (cusum) chart shall be maintained using only the 20 most recent data points.

7.4 **Outliers:** The outliers, which are values falling outside the upper and lower control limits, and trends of increasing or decreasing sensitivity, are readily identified. At the $P_{0.05}$ probability level, one in 20 tests would be expected to fall outside of the control limits by chance alone. If more than one out of 20 reference toxicant tests fall outside the control limits, the effluent toxicity tests conducted during the month in which the second reference toxicant test failed are suspect, and shall be considered as provisional and subject to careful review.

7.5 **Toxicity Values:** If the toxicity value from a given test with the reference toxicant falls well outside the expected range for the test organisms when using the standard dilution water, the sensitivity of the organisms and the overall credibility of the test system are suspect. In this case, the test procedure shall be examined for defects and shall be repeated with a different batch of test organisms.

7.6 **Performance:** Performance should improve with experience, and the control limits for point estimates should gradually narrow. However, control limits of $\pm$ 2S, by definition, will be exceeded 5% of the time, regardless of how well a laboratory performs. Highly proficient laboratories which develop a very narrow control limit may be unfairly penalized if a test which falls just outside the control limits is rejected *defacto*.

7.7 **Inter-laboratory:** The Laboratory shall participate in an inter-laboratory comparison study on a quarterly basis that includes the following parameters at a minimum: total alkalinity, ammonia as N, chloride, hardness, conductivity, and pH.

7.8 **Regulatory Performance Evaluation (PE) Samples:** All NPDES, EPA and State of Colorado PE samples shall be analyzed when submitted.

7.9 **Other**: Requirements for laboratory evaluation samples are specified in the General Laboratory Requirements Module, GR01.

8 ON-SITE LABORATORY EVALUATIONS

Requirements for on-site laboratory evaluations are specified in the General Laboratory Requirements Module, GR01.

9 PERFORMANCE CRITERIA

Laboratory performance will be continually assessed by the CTR. Performance areas will include those outlined in the General Laboratory Requirements Module GR01 Exhibit E, Section 13, and those outlined below:

9.1 **Holding Time**: Hold-time shall be used as a performance measure for all applicable whole effluent toxicity parameters. The Site will monitor the Laboratory for compliance to holding time requirements as described in SS11 Exhibit D, Section 3.

10 CERTIFICATIONS AND APPROVALS

Laboratories are encouraged to provide proof of certifications or approvals for any programs in which the Laboratory is participating. Proof of laboratory certifications (or approvals) delivered to the CTR according to the schedule in GR01 Exhibit B Table B2 may increase the potential sample load for Laboratories providing this proof.

10.1 Laboratories providing analytical services under Module SS11 are required to have the State of Colorado or USEPA certifications or approvals related to the determination of whole effluent toxicity parameters for samples related to US EPA National Pollutant Discharge Elimination System (NPDES) permits.

EXHIBIT F

EVIDENTIARY REQUIREMENTS

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2. SAMPLE CHAIN-OF-CUSTODY	F-2
3. SAMPLE RECEIVING REQUIREMENTS	F-2
4. DOCUMENT CONTROL PROCEDURES	F-2
5. STANDARD OPERATING PROCEDURES	F-2

EVIDENTIARY REQUIREMENTS

1. INTRODUCTION

The purpose of this Exhibit is to describe the evidentiary requirements that shall be followed for the preparation and analysis of Site samples for Whole Effluent Toxicity Parameters under this subcontract.

2. SAMPLE CHAIN-OF-CUSTODY

Requirements for sample chain-of-custody are specified in GR01.

3. SAMPLE RECEIVING REQUIREMENTS

Requirements for sample receiving are specified in GR01.

4. DOCUMENT CONTROL PROCEDURES

4.1. **General Requirements:** General requirements for document control are specified in GR01.

4.2. **Raw Data Labeling Requirements:** Results shall be presented with all raw data necessary for the CTR to assess their validity. This verification process involves such processes as recalculating reported values and qualifiers from raw data and tracing all standards to valid reference materials. In order to perform this verification, the following rules shall be followed for labeling all raw data submitted in Sample Data Packages and Supporting Documentation Packages (Support Packages). Preparation, instrument, and other raw data shall meet the following labeling requirements:

4.2.1. Sample data shall be labeled with the Site Sample Identifiers or with the lab identifiers cross-referenced to the Site Sample Identifiers on the Sample Data Package Cover Page.

4.2.2. All data generated by Material and Testing Equipment (M&TE) shall be labeled with the unique M&TE identifier.

4.2.3. Data labels shall clearly identify samples designated as samples, controls, and blanks. *Codes for Labeling Data* may be used without clarification. Use of other identification schemes will require all codes to be clarified in a description table on each affected Section Cover Sheet.

4.2.4. The identification scheme used (whether using SS11 or alternate codes) shall provide an unequivocal and unique link between all samples and QC samples prepared as an analytical batch. For example, a preparation blank labeled as ‘PBW’ appended by the Analytical Batch Identifier (see GR01 Exhibit G) would provide this link.

4.2.5. All standards referenced in raw data shall be identified by the unique identifier assigned as required in GR01 Exhibit E Section 6.

4.2.6. All numerical data shall be accompanied by applicable units.

5. STANDARD OPERATING PROCEDURES

5.1. **General Requirements:** Requirements for written SOPs are specified in GR01.

5.2. **Required Procedures:** For each analysis technique used by the Laboratory, each of the operations shall be included in one or more SOPs.

EXHIBIT G

GLOSSARY OF TERMS AND ACRONYMS

TABLE OF CONTENTS

SECTION **PAGE NO.**

1. INTRODUCTION G-2
2. GLOSSARY OF TERMS G-2

GLOSSARY OF TERMS AND ACRONYMS

1 INTRODUCTION

The glossary of terms and acronyms contained in parameter-specific analytical module(s) supplement those found in the General Laboratory Requirements Module, GR01. Any definitions provided in this Exhibit, however, shall supersede the definitions provided in GR01 in cases of conflicting definitions.

2 GLOSSARY OF TERMS

Refer to General Requirements Module GR01, Exhibit G for additional terms.

DOSE - The amount of toxicant that enters the organism. Dose and concentration are not interchangeable.

TOXICITY - The potential or capacity of a test material to cause adverse effects on living organisms, generally a poison or mixture of poisons. Toxicity is a result of dose or exposure concentration and exposure time, modified by variables such as temperature, chemical form and availability.

EXPOSURE TIME - The time of exposure of test organisms to test solution.

ACUTE TOXICITY - Relatively short term lethal or other effect, usually defined as occurring within 4 d for fish and (2 d) for macroinvertebrates.

CHRONIC TOXICITY - Toxicity involving a stimulus that lingers or continues for a relatively long period of time, often one-tenth of the life span or more. "Chronic" should be considered a relative term depending on the life span of an organism. A chronic toxic effect can be measured in terms of reduced growth, reduced reproduction, etc., in addition to lethality.

LETHAL CONCENTRATION (*LC*) - The toxicant concentration estimated to produce death in a specified proportion of test organisms. Usually defined as median (50%) lethal concentration, LC50, i.e., concentration killing 50 % of exposed organisms at a specific time of observation.

STATIC TEST - A test in which solutions and test organisms are placed in test chambers and kept there for the duration of the test.

RENEWAL TEST - Tests in which organisms are exposed to solutions of the same composition that are renewed periodically during the test period. This is accomplished by transferring test organisms or replacing test solutions.

FLOW THROUGH TEST - Test in which solution is replaced continuously in test chambers throughout the test duration.

ANALYSIS DATE/TIME - The date and time (24-hour clock) of the analytical measurement of the sample, standard, or blank by the analysis system.

ANALYSIS LOGSHEET/BENCHSHEET - A form with lab and analysis method identifiers; calibration information; sample identifiers; sample volumes, weights, dilution factors and measurement data; standard traceability information; date/times of analysis; and signatures of analyst and reviewer.

CALIBRATION BLANK - A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation. This blank is subjected to the preparation method, if applicable but is produced synthetically.

CALIBRATION/STANDARDIZATION DATE/TIME - The date and time (24-hour clock) that the analytical system calibration/standardization was completed.

CALIBRATION STANDARDS - A series of known standard solutions used by the analyst for calibration of the instrument (i.e. preparation of the analytical curve). For WET, the solutions may be subjected to the preparation method but contain the same matrix as the sample preparations to be analyzed.

CONTROL LIMITS - A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

INITIAL/CONTINUING CALIBRATION VERIFICATION (ICV/CCV) - A single element or multi-element standard solution prepared by the analyst to be used to verify the stability of the instrument calibration with time and the instrument performance during the analysis of samples. The initial/continuing calibration verification solution(s) is either the same solution or two different solutions that are independent of the calibrations standards source. However, all analyte elements being measured by the particular system must be represented in this standard and the standard must have the same matrix as the samples. The initial/continuing calibration solution(s) should have a concentration near the mid-point of the analytical calibration curve. The initial calibration verification solution must be analyzed immediately after calibration and prior to analysis of any samples. The continuing calibration verification solution must be analyzed every 20 analytical samples and after the last analytical sample to verify the calibration of the analytical system.

PREPARATION DATE/TIME - The date and time (24-hour clock) of the preparation of the sample, standard, or blank was started.

PREPARATION LOGSHEET/BENCHSHEET - A form with lab and analysis method identifiers; sample identifiers; sample volumes, weights, and dilution factors; standard traceability information; date/times of preparation; and signatures of analyst and reviewer.

EXHIBIT H

REFERENCES

TABLE OF CONTENTS

SECTION **PAGE NO.**

1 REFERENCES H-2

1 REFERENCES

USDA. 1989. Methods which detect multiple residues. Vol.1. Pesticide analysis manual. U.S. Department of Health and Human Services, Washington, DC.

USEPA. 1979a. Handbook for analytical quality assurance in water and wastewater laboratories. U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio. EPA-600/4-79-0 19.

USEPA. 1979b. Methods for chemical analysis of water and wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA-600/4-79A)20.

USEPA. 1979c. Interim NPDES compliance biomonitoring inspection manual. Office of Water Enforcement, U.S. Environmental Protection Agency, Washington, DC. (MCD-62).

USEPA. 1980a. Proposed good laboratory practice guidelines for toxicity testing. Paragraph 163.60-6. Fed. Reg. 45:26377-26382, April 18, 1980.

USEPA. 1980b. Physical, chemical, persistence, and ecological effects testing; good laboratory practice standards. 40 CFR 772, Fed. Reg. 45:77353-77365, November 21, 1980.

USEPA. 1981. Results: Interlaboratory comparison--acute toxicity tests using estuarine animals. S.C. Schimmel. Environmental Research Laboratory, U.S. Environmental Protection Agency, Gulf Breeze, Florida. 15 pp.

USEPA. 1983. Analysis of an interlaboratory comparative study of acute toxicity tests with freshwater aquatic organisms. S.J. Broderius. Environmental Research Laboratory, U.S. Environmental Protection Agency, Duluth, Minnesota. 54 pp.

USEPA. 1985. Ambient water quality criteria for ammonia - 1984. Office of Water Regulations and Standards, Office of Water, U.S. Environmental Protection Agency, Washington, DC. EPA/440/5-85/001.

USEPA. 1986. Occupational health and safety manual. Office of Administration, U.S. Environmental Protection Agency, Washington, DC.

USEPA. 1988a. Methods for aquatic toxicity identification evaluations: Phase III, toxicity confirmation procedures. D.I. Mount. Environmental Research Laboratory, U.S. Environmental Protection Agency, Duluth, Minnesota. EPA-60013-881036.

USEPA. 1988b. Methods for aquatic toxicity identification evaluations: Phase II, toxicity identification procedures. D.I. Mount, and L. Anderson-Carnahan. Environmental Research Laboratory, U.S. Environmental Protection Agency, Duluth, Minnesota. EPA-600/3-88/035.

USEPA. 1989a. Toxicity reduction evaluation protocol for municipal wastewater treatment plants. J.A.Botts, J.W. Braswell, J. Zyman, W.L. Goodfellow, and S.B. Moore. Risk Reduction Engineering Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268. EPA/600/2-881062.

USEPA. 1989b. Generalized methodology for conducting industrial toxicity reduction evaluations (TREs). 3.A. Fava, ID. Lindsay, W.H. Clement, R. Clark, G.M. DeGraeve, J.D. Cooney, S. Hansen, W. Rue, S. Moore, and P. Lankford. Risk Reduction Engineering Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268. EPA/600/2-88/070.

USEPA. 1989c. Short-term Methods For estimating the chronic toxicity of effluents and surface waters to freshwater organisms. C.I. Weber, W.H. Peltier, T. J. Norherg-King, W.B. Horning, II, F.A. Kessler, J.R. Menkedick, T.W. Neiheisel, P.A. Lewis, D.J. KIemm, Q.H. Pickering, E.L. Robinson, J.M. Lazorchak, L.J. Wymer, and R.W. Freyberg. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA-600/4-89/001.

USEPA. 1990a. Macroinvertebrate field and laboratory methods for evaluating the biological integrity of surface waters. D.J. Klemm, P.A. Lewis, F. Kessler, and J.M. Lazorchak. Environmental Monitoring Systems Laboratory, Office of Modeling, Monitoring Systems, and Quality Assurance, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio. 45268 EPA/600/4-90/030.

USEPA. 1990b. Supplemental methods and status reports for short-term saltwater toxicity tests. G. Morrison, and G. Chapman. ERL Contrib. No. 1199. Environmental Research Laboratory, U.S. Environmental Protection Agency, Narragansett, Rhode Island. 127 pp.

USEPA. 1991a. Methods for aquatic toxicity identification evaluations: Phase I, toxicity characterization procedures. T. Norberg-King, D.I. Mount, E. Durhan, G. Ankley, L. Burkhard, J. Amato, M. Lukasewycz, M. Schubauer-Berigan, and L. Anderson-Carnahan. Environmental Research Laboratory, U.S. Environmental Protection Agency, Duluth, Minnesota. EPA/60016-91/003.

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USEPA. 1991c. Technical support document for water quality-based toxic control. Office of Water, U.S. Environmental Protection Agency, Washington, DC. EPAI5O5/2-90/00l. 387 pp.

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USEPA. 1993b. Short-term methods For estimating the chronic toxicity of effluents and surface waters to marine and estuarine organisms. 2nd edition. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA-60014-9 1/003.

USEPA. 1993c. Table 1. In: Guidelines establishing test procedures for the analysis of pollutants. Code of Federal Regulations, Vol.40, Part 136. U.S. Environmental Protection Agency, Washington, DC.

‘Standard Methods for the Examination of Water and Wastewater,’ 18th Edition, 1992, American Public Health Association (APHA), New York, NY.

‘Standard Specifications for Reagent Water,’ ASTM D 1198, American Society for Testing and Materials (ASTM).

‘Methods for Water Analysis,’ Volume 11.01 and 11.02, Water 1 and 2, 1993, American Society for Testing and Materials (ASTM) (or latest revision)

‘Standard Methods for the Examination of Water and Wastewater,’ 19th Edition, 1995, American Public Health Association (APHA), New York, NY.

Region VIII EPA NPDES Chronic Test Conditions – Static Renewal Whole Effluent Toxicity Test August 1997

A P P E N D I X A

Data Review Checklist

SS11 SAMPLE DATA PACKAGE

D a t a R e v i e w C h e c k l i s t

SS11 Sample Data Package

1. SAMPLE DATA PACKAGE COVER PAGE	***Reply***	***Ö***	***C#***
a) The laboratory name, code, subcontract number, RIN, Site sample numbers, analyses, and report dates are accurately recorded.			
b) All Site sample identifications are cross-referenced with all lab identifications.			
c) The verbatim compliance and authorization statement is present with the dated signature of the Laboratory Manager or designee.			
d) Any problems with the receipt are explained.			

2. TABLE OF CONTENTS	***Reply***	***Ö***	***C#***
a) The Table of Contents contains all Sample Data Package Deliverable Section Titles and the page numbers.			

3. DATA REVIEW CHECKLIST (DRC) and SS11 SAMPLE DATA PACKAGE (SDP)	Reply	√	C#
a) The SS11 DRC is present and in strict conformance with the formatting and content of the form contained in the current version of the SS11, Appendix A. All discrepancies were identified and documented, accordingly.			
b) All DRC ***Reply*** **blocks are completed with either a "Y", "N" or "N/A".**			
c) All DRC ***Reply*** **blocks completed with an "N"** are explained in the Narrative.			
d) The DRC footer is completed for each page; the laboratory manager or designee signed and dated the DRC.			
e) All SDP deliverable sections appear in the SDP in order by deliverable section number			
f) Only one SDP is submitted for each SS11 and RIN request.			N/A
g) All components of the SDP deliverables contain original documents where possible.			Φ
h) There is no inclusion of required items in the SDP by reference to another SDP.			Φ
i) Site samples are exclusively used for sample matrix QC			Φ
j) Site and non Site samples are not reported together in any way			Φ
k) The complete sample data package is single sided and consecutively paginated.			

4. CHAIN-OF-CUSTODY	***Reply***	***Ö***	***C#***
a) The continuity of each sample's custody is evidenced by the chain of the date, time and signatures of each transaction from sampling to final disposition.			
b) If the continuity was corrupted, there is documentation and evidence of correspondence with the CTR.			
c) All samples are identified on the COC with the corresponding analysis.			
d) Temperatures of samples were checked immediately upon receipt in the lab. All samples were 4 degrees C or below.			
e) The sample was refrigerated at 4 C upon arrival in the lab and the temperature recorded.			
f) Any conflicting, incorrect, or missing information are identified and documented, and there is documentation of the resolution.			

5. NARRATIVE	***Reply***	***Ö***	***C#***
a) Contains a synopsis of the methodology and analysis procedure for each parameter. The base method for each parameter is referenced with any deviations.			
b) Contains a description of the samples and which samples are of similar matrix.			

RIN: _____ **Lab Name:** _____ Initials: _____

Analytical Batch Identification No.(s): _____

Data Review Checklist

SS11 Sample Data Package

5.	Continued	***Reply***	***Ö***	***C#***
c)	Contains synopsis of original Analytical Batch QC assessment. All anomalies, caveats, deficiencies, interferences, reanalysis, and deviations from approved SOPs related to the analysis are explained.			
d)	Samples requiring reanalysis are identified with the reason for reanalysis, the original and reanalysis Analytical Batch Identification Numbers, and a synopsis of the reanalysis Analytical Batch QC assessment is included.			
e)	For any deviations that required CTR approval, the correspondence and approval are documented.			
f)	Identification of hold-times and any hold-time violations are described in Narrative.			

6.	SAMPLE AND QC RESULTS SUMMARY	***Reply***	***Ö***	***C#***
a)	All parameters for General Quality Requirements have been met as stated in Exhibit D, Section 4.			
b)	All requirements for Calibration and Standardization have been met as stated in Exhibit D, Section 5:			
c)	Correct results reported in the correct units for each requested analyte			
d)	All ICV and CCV results are reported for all applicable parameters			
e)	All ICV and CCV results are within limits.			
f)	All QC samples were counted and analyzed in the same manner as the samples in the Analytical Batch, in the same time frame, and using the same instrument calibration parameters, instrument analysis algorithms, etc.			
g)	Methods for analysis are in compliance with Colorado Water Quality Control Division and EPA Region VIII guidelines:			
h)	Hardness, alkalinity, conductivity, ammonia, pH, and dissolved oxygen analysis were performed on all samples.			

7.	PREPARATION RAW DATA	***Reply***	***Ö***	***C#***
a)	The preparation raw data (benchsheets and/or preparation logs) are included and document the required items as specified in the Preparation Summary Section of SS11, Exhibit B, Section 2.9.			
b)	Sufficient raw data are included to allow manual calculation of the final sample results and QC sample recoveries			

8.	STANDARDS SUMMARY	***Reply***	***Ö***	***C#***
a)	For primary standards that were diluted and used for LCS, ICV/CCV and any in-house prepared instrument calibration solutions, the required items as specified in the Standards Summary Section of SS11, Exhibit B are included.			
b)	All standard identifications are traceable to the primary certificates, which are traceable to NIST.			
c)	All standards and sources traceable to NIST were valid (not expired) at the time of use.			

9.	INSTRUMENT RAW DATA	***Reply***	***Ö***	***C#***
a)	The instrument raw data for the RIN are included and document the required items as specified in SS11, Exhibit B, Section 2.11. The reviewer verified all data entries as accurate.			
b)	The data were reviewed, signed and dated by an area specialist, and found to be acceptable			
c)	Sufficient raw data are included to allow manual calculation of the final sample results and QC sample recoveries if applicable.			
d)	All QC samples were prepared and analyzed in the same manner as the samples in the Analytical Batch, in the same time frame, and using the same instrument and instrument parameters if applicable.			

RIN: _____ **Lab Name:** _____ Initials: _____

Analytical Batch Identification No.(s): _____

Shaded areas are for Site use only.
Respond to each checklist item in the "Reply" column with a Y (yes), N (no), or NA (not applicable).
Complete footer information, including the initials of the laboratory manager or designee on each page.
Refer to Module GR01, Exhibit B, Section 4 for instructions to complete this form.

I certify that all responses to this checklist accurately reflect the completeness and quality aspects of this sample data package as outlined in GR01 and SS06. Furthermore, I understand that inaccuracies in the completion of this checklist will be considered a nonconformance to Subcontract Requirements as evidenced by the following signature of the laboratory manager or designee.

Print/Typed Name: _____ Title: _____

Signature _____ Date _____

For Data Assessment Use Only

DATA REVIEW CHECKLIST EXAMINATION

Assessment of this Sample Data Package is based on the following documents:

DA-GR01 Version ____ DA-SS11 Version ____

This Sample Data Package requires the following assessment level:

❑ No Further Assessment ❑ Partial Verification ❑ Complete Verification ❑ Validation

Assessment Level is based on: ❑ Examination ❑ Customer ❑ Laboratory Assessment Program

Notes:

Examiner	Signature	Date

RIN: _____ **Lab Name:** _____ Initials: _____

Analytical Batch Identification No.(s): _____

PARTIAL VERIFICATION

❑ **This Sample Data Package requires no further assessment (See attached Data Quality Assessment Report)**

❑ This Sample Data Package requires further assessment at the following level:

❑ Complete Verification ❑ Validation

Notes:

Data Verifier	Signature	Date

COMPLETE VERIFICATION

❑ **This Sample Data Package requires no further assessment (See attached Data Quality Assessment Report)**

❑ This Sample Data Package requires validation

Notes:

Data Verifier	Signature	Date

RIN: _____ **Lab Name:** _____ Initials: _____

Analytical Batch Identification No.(s): _____