QUALITY ASSURANCE PROGRAM PLAN

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Table of Contents

1. INTRODUCTION

Aquatic Testing Laboratories (ATL) is dedicated to providing quality aquatic toxicity testing to its clients. This document describes ATL's Quality Assurance policies and procedures as they relate to biological monitoring for environmental pollutants.

Purpose of Document

This Quality Assurance Program Plan (QAPP) is intended to ensure that precision, accuracy, completeness, comparability, and representativeness of data are known and documented.

The QAPP presents an overview of the essential elements of ATL's QA program. This plan has been modeled along EPA guidelines as outlined in "Interim Guideline and Specifications for Preparing Quality Assurance Program Plans," QAMS-004/80, December 29,1980; "Interim Guideline and Specifications for Preparing Quality Assurance Project Plans," QAMS-005/80, February 1983; "Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms," EPA/600/4-89/001; and "Manual for the Evaluation of Laboratories Performing Aquatic Toxicity Tests," EPA/600/4-90/031. All of these documents have been issued by the Office of Monitoring Systems and Quality Assurance, Office of Research and Development, U.S. Environmental Protection Agency (U.S. EPA). Primary guidance was obtained from "Enseco Incorporate Quality Assurance Program Plan for Environmental Biology," Revision 3.1, July, 1988, written by Enseco Inc. with additional guidance provided from the Environmental Laboratory Accreditation Program, (State of California Department of Health Services and Department of Fish and Game).

QA Objectives

This QA Program Plan is designed to control and monitor the quality of data generated at ATL. The described QA program is geared toward generating data that comply with federal regulatory requirements specified under the National Pollutant Discharge Elimination System (NPDES) as well as the State of California Department of Health Services Environmental Laboratory Accreditation Program (DOHS ELAP) and other state equivalents. Although the QC requirements of these various programs are not completely consistent, each of the programs base data quality judgments on two types of information:

* Data that indicate the overall qualifications of the laboratory to perform environmental analyses;

* Data that measure the laboratory's daily performance using a specific method.

The operational elements that are involved in making each of these assessments are described in TABLE 1 along with the pertinent section number from this document in which each is discussed.

TABLE 1
DATA QUALITY ASSESSMENT

Evaluation Criteria	Operational Elements	Section of QA Plan
LABORATORY QUALIFICATIONS	Facilities/Equipment/Staff	SOQ*
	Written SOP's for all laboratory procedures	15
	Sample custody	5
	Calibration procedures	6
	Testing procedures	7
	Data validation	8
	Documented QA program	1-15
	Laboratory certifications	10
LABORATORY PERFORMANCE	Calibration data	6
	Check samples	10
	Reference toxicant data	9
	Control charts	9

* SOQ (Statement Of Qualifications) described in a separate document.

2. DEFINITIONS

Definition of Terms

Protocol: the actual plan for scientific testing. A protocol may refer to several SOP's to complete the plan.

Quality Assurance (QA): the total integrated program for assuring the reliability of data generated in the laboratory.

Quality Assurance Program Plan (QAPP): an assemblage of management policies, objectives, principles, and general procedures outlining the techniques by which the laboratory produces data of known and accepted quality.

Quality Assurance Project Plan (QAPjP): an assemblage of detailed SOP's describing how the laboratory will generate data that meet the data quality objective of a specific project.

Quality Control (QC): the routine application of specific, well documented procedures to ensure the generation of data of known and accepted quality, thus fulfilling the objectives of the QA program.

Quality Control Manual: an assemblage of detailed SOP's describing the laboratory implementation of the QAPP.

Standard Operating Procedure (SOP): a detailed, written description of a procedure designed to systematize and standardize the performance of the procedure.

3. ORGANIZATION, RESPONSIBILITIES AND AUTHORITIES

Executing an effective QA program demands the commitment and attention of both management and staff. The QA effort at ATL is managed by the Laboratory Director who serves as the QA Officer and as such, has the responsibility of overseeing and regulating all laboratory functions. The QA program operates independently of all areas, generating analytical data to ensure complete objectivity in the evaluation of laboratory operations.

QA Officer Responsibilities

The QA officer is responsible for:

* Developing and implementing a QA program that ensures that all data generated are scientifically sound, legally defensible, and of known precision and accuracy;

* Monitoring the QA Plan to ensure compliance with QA objectives;

* Ensuring that all employees are complying with the QA Plan;

* Developing and implementing new QA procedures to improve data quality;

* Conducting in-house audits and inspections of all laboratories on a regular basis and applying corrective actions as needed to ensure compliance with the QA Plan;

* Maintaining copies of all SOP'S;

* Assist in the writing of SOP's;

* Distributing current SOP's to the laboratory staff;

* Monitoring laboratory performance in the areas of holding times, turn-around times, and meeting contractual obligations;

* Performing statistical analyses of QC data and establishing data bases that accurately reflect the performance of the laboratory;

* Maintaining reference toxicant control charts on all testing done at ATL;

* Maintain records and archives of all QA/QC data, PE results, audit comments, and client inquiries concerning data quality;

* Conducting seminars on QA issues for both clients and laboratory staff; and

* Promoting sound QA practices within the environmental regulatory and analytical communities.

QA Officer Authority

The QA officer has the final authority on all issues dealing with data quality and has the authority to require that procedures be amended or discontinued, or analyses suspended or repeated. He also has the authority to suspend or terminate employees on the grounds of dishonesty, incompetence, or repeated non-compliance with QA procedures.

Laboratory Personnel Responsibilities

All laboratory personnel involved in the generation and reporting of data have a responsibility to understand and follow the ATL QA Plan. Laboratory personnel are responsible for:

* Have a working knowledge of the ATL QA Plan;

* Ensuring that all work is generated in compliance with the QA Plan;

* Performing all work according to written SOP's;

* Ensuring that all documentation related to their work is complete and accurate; and

* Providing management with immediate notification of quality problems.

Laboratory Personnel Authority

Laboratory personnel have the authority to accept or reject data based on compliance with well-defined QC acceptance criteria. The acceptance of data that fall outside QC criteria must be approved by laboratory management. The authority of the laboratory personnel flows from the Laboratory Director.

4. SAMPLING PROCEDURES

The generation of quality data begins with the collection of the effluent, water or sediment sample. Therefore the integrity of the sample collection process is of concern to the laboratory. Samples must be collected in such a way that no foreign material is introduced into the sample and no material of interest escapes from the sample prior to analysis. To ensure sample integrity, the following must be considered:

* Samples must be collected in appropriate containers. In general, glass containers are used for soils and solids, while plastic "cubitainers" are used for effluents and surface waters;

* The sample containers must be properly cleaned to ensure that the sample is not contaminated during the collection process;

* Appropriate volumes of sample must be collected to ensure that the required testing may completed and QC samples may be analyzed;

* Samples must be cooled to the appropriate holding temperature (4°C) prior to shipping;

* Samples must be properly shipped to the laboratory, in the appropriate time frame, to ensure that holding times can be met.

ATL can assist in the sample collection process by providing consultation and assistance to clients designing sampling programs and also by making available to the client a set of appropriate sample containers that are properly cleaned for use in sample collection.

The maximum holding times recommended by ATL, appropriate containers, and minimum sample volumes required for routine testing are given in Appendix I. These holding times are in general agreement with EPA and the State of California recommended holding times, as stated in the National Pollutant Discharge Elimination System (NPDES) and the California Environmental Laboratory Accreditation Program (ELAP) programs. Other holding times can be honored if special arrangements are made with the laboratory.

5. SAMPLE CUSTODY

Upon receipt by ATL, samples proceed through an orderly processing sequence specifically designed to ensure continuous integrity of both the sample and its documentation.

All samples are received by ATL's sample control personnel and are carefully checked for label identification, and completed, accurate chain-of-custody records. Photographs may be used to document the condition of samples. Each sample is then assigned an unique laboratory identification number. The date received, the condition upon receipt, the temperature upon receipt, the new laboratory identification number, as well as the client and the client's sample identification are recorded in the sample control log book. A sample file is then generated in which all documentation, including testing results, are kept. The sample itself is labeled with the laboratory identification number and stored in a secured refrigerated storage facility with temperature maintained at 4°C until analysis. The total residual chlorine (TRC) of effluent samples is measured and recorded. Any unused sample is returned to refrigerated storage with little headspace as possible, until all analyses are complete. Samples are then either returned to the client, properly disposed of, or at the request of the client, stored for an extended length of time.

6. CALIBRATION PROCEDURES AND FREQUENCY

Standard/Reagent Preparation

A critical element in the generation of quality data is the purity/quality and traceability of the standard solutions and reagents used in the analytical and/or biological operations. ATL continually monitors the quality of reagents and standard solutions through a series of well-documented procedures.

To ensure the highest purity possible, all primary reference standards and standard solutions are obtained from the EPA laboratory in Cincinnati, Ohio, or other reliable commercial sources. All standards and standard solutions are recorded into a log book that identifies the supplier, lot number, purity/concentration, receipt/preparation date, preparer's name, method of preparation, expiration date, and all other pertinent information.

Care is exercised in the proper storage and handling of standard solutions, and all containers are labeled as to compound, concentration, solvent, expiration date, and preparation data (initials of preparer/date of preparation).

Instrument Calibration and Tuning

Calibration of instrumentation is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity. Instruments used for routine measurements of chemical and physical parameters such as pH, DO, temperature, conductivity, salinity, alkalinity, and hardness, must be calibrated and standardized according to the instrument manufacturer's procedures prior to any uninterrupted use. The light meter is certified calibrated biannually per manufacturer's recommendation. Analytical balances are calibrated annually by a certified technician and verified monthly by laboratory personnel.

Dissolved oxygen probes are calibrated daily by use of the moist air technique, however, comparison to the Winkler titrimetric method may be performed as needed.

Wet chemical methods used to measure hardness and alkalinity must be standardized according to EPA Methods 130.2 and 310.1.

7. TESTING PROCEDURES

Test Organisms

The fish and invertebrates used in toxicity testing should appear healthy, behave normally, feed well, and have low mortality in cultures, holding tanks, and test controls. Test organisms should be disease-free and should be positively identified to species.

The sensitivity (quality) of test organisms obtained from an outside of the laboratory source is to be tested by conducting a reference toxicant test on organisms from each batch received by the laboratory or at a minimum on a monthly basis when more than one batch of organisms are received during the month from the same provider provided that the organisms have preformed satisfactory in the previous five monthly reference toxicant tests (value not well outside the expected range). The sensitivity of test organisms obtained from an in-lab breeding culture is to be tested by conducting a reference toxicant test on the cultured organisms on a monthly basis. Reference toxicant tests may be performed concurrently with an effluent toxicity test.

Facilities, Equipment, and Test Chambers

Laboratory and bioassay temperature control equipment must be adequate to maintain recommended test water temperatures. Surfaces that come in contact with the sample, such as test chambers, must be made of recommended materials. See individual testing SOP's and protocols for recommended materials and testing regimes.

Dilution Water

The dilution water used in toxicity tests will depend on the objectives of the study and client requirements. Hazardous waste testing utilize synthetic, soft (hardness: 40-48 mg/l $CaCO_3$) water. EPA NPDES toxicity test utilizes synthetic, moderately hard water or 20% diluted mineral water (DMW). Some tests will require the use of client-supplied dilution water.

The dilution water used for internal quality assurance tests with organisms, food, and reference toxicants should be water routinely used with success in the laboratory.

Testing Conditions

Water temperature must be maintained within the limits specified for each test. Dissolved

oxygen (DO) concentration and pH in fish and invertebrate test chambers should be checked daily throughout the test period, as described in the test SOP.

Food Quality

The quality of the food for fish and invertebrates is an important factor in toxicity tests. Suitable fish food flakes, brine shrimp cysts, and other foods must be obtained as described in the test SOP's and protocols. The suitability of each new supply of food should be determined in a side-by-side test, using two treatments with four replicates per treatment. In this test, the response of control test organisms fed with the new food is compared with the response of organisms fed a reference food or a previously used, satisfactory food.

Test Methods

Most tests performed by ATL are driven by regulatory concerns. Therefore, methods used at ATL predominately originate from regulatory agencies. Generally the methods used are those specified by the U.S. EPA and other federal agencies, state agencies, and professional organizations, as provided in the following references:

* California Department of Health Services. 1988. Static Acute Bioassay Procedures for Hazardous Waste Samples. Prepared by J.M. Polisini and R.G. Miller. California Department of Fish and Game Water Pollution Control Laboratory.

* California State Water Resources Control Board (CSWRCB). 1996. Procedures Manual for Conducting Toxicity Tests Developed by the Marine Bioassay Project. CSWRCB, Sacramento, CA. 96-1WQ

* U.S.EPA. 1993. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. 4th ed. EPA/600/4-90/027F.

* U.S.EPA. 1994. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. 3rd ed.EPA600-4-91-002.

* U.S.EPA. 1994. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. 2nd ed. EPA-600-4-91-003.

* U.S.EPA. 1995. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to West Coast Marine and Estuarine Organisms. EPA/600/R-95R/136.

* U.S.EPA. 2002. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. 5th ed. EPA-821-R-02-012.

* U.S.EPA. 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. 4th ed. EPA-821-R-02-013.

* U.S.EPA. 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. 3rd ed. EPA-821-R-02-014.

The choice of method is dependent on the objectives of the study in terms of qualitative certainty, quantitative sensitivity, precision and accuracy, and the type of matrix to be analyzed. Each method used routinely is documented in the form of an SOP. The SOP contains detailed instructions concerning both the use and the expected performance of the method. Any deviations from the published methodology are documented and explained in the SOP. A complete description of the contents of laboratory SOP's is given in Section 15.

Before any methods are routinely used to generate analytical and/or biological data, the method is validated. Validation criteria consists of:

* Method selection by a senior staff member;

* Documentation of the method in a SOP. This includes a summary of the method, detailed description of the procedure, calculations, reporting formats, safety concerns, and special remarks;

* Testing of the method to verify detection limits and linear range and establish precision and accuracy criteria; and

* Establishment of data acceptance criteria that must be approved by a senior staff member and the QA Officer.

8. DATA REDUCTION, VALIDATION, AND REPORTING

All data generated by ATL are extensively checked for accuracy and completeness. The data validation process consists of data generation, reduction, and two levels of review, as described below.

The analyst who generates the data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following methods specified in laboratory SOP's. Each analyst reviews the quality of his work based on an established set of guidelines. The analyst reviews the data package to ensure that:

* The protocol has been followed exactly; if not, any deviations are properly noted;

* Sample preparation information is correct and complete;

* Analyst information is correct and complete;

* The appropriate SOP's have been followed;

* Analytical/biological results are correct and complete;

* QC (reference toxicant) results are within established control limits;

* Special sample preparation and analytical requirements have been met; and

* Documentation is complete.

The data reduction and validation steps are documented signed and dated by the analyst. This initial review step, performed by the analyst, is designated as Level 1 review. The analyst then passes the data package to the QA Officer, who performs a Level 2 review.

Level 2 review is conducted to an established set of guidelines and is structured to ensure that:

* Calibration data are scientifically sound, appropriate to the method, and completely documented;

* QC samples (reference toxicants) are within established guidelines;

* Qualitative identification of sample components is correct;

* Quantitative results are correct;

* Documentation is complete and accurate;

* The data are ready for incorporation into the final report; and

* The data package is complete and ready for data archive.

Level 2 review is structured so that all calibration and QC data are reviewed and all of the analytical and biological results are checked back to the bench sheet. The review is complete when the data package has been reviewed in its entirety.

An important element of Level 2 review is the documentation of any errors that have been identified and corrected during the review process. Errors that are found are documented and transmitted to the appropriate supervisor. The cause of the errors is then addressed with additional training or clarification of procedures to ensure that quality data will be generated at the bench.

Data Reduction

Many toxicity tests require the calculation of an LC50, EC50, NOEC, LOEC, or percent survival calculations. ATL primarily utilizes the computer statistical program TOXCALC to calculate these values. Other statistical packages may be utilized to evaluate the data when appropriate. Proper statistical procedures, such as examining homogeneity of variance prior to ANOVA analyses, or data transformations when required, are conducted according to the method being tested. Proper statistical analyses are outlined in each test method SOP.

Data that do not appear to be in conformance with the substantial majority are often referred to as "outliers", and may be due to random variation, clerical errors, or experimental errors. Statistical outlier detection procedures are screening procedures that indicate whether a value is extreme enough to be considered not due just to random variation and thereby excluded from statistical analysis of the remaining testing data. When outliers are not known to be erroneous values, data analyses are performed with and without the questionable values in order to assess their importance.

Data Reporting

A final report will be generated after successful completion of Level 1 and 2 reviews. The report will include, but not be limited to, the following items:

* Summary, which includes: client name, client sample description, title and description of test, laboratory identification number, test dates, a description of the test organism, water, a definition of the effect criteria, and calculated endpoints.

* Material and Methods, which include: protocol, test dates, laboratory personnel, raw data and/or bench sheets, a description of the test methods and any deviation from the protocol, identification and source of test organisms, description of holding conditions, description and chemical/physical characterization of diluent water, description of analytical methods, counting procedures and statistical techniques.

* Results, which include: all observations, and endpoint determinations.

* References.

* Appendices, where appropriate:

    A. Raw data, including all biological observations and analytical results.

    B. Certification of good laboratory practices signed by all personnel involved in the study and the QA Officer. The certification will include the location and the period for data archiving.

* Client Services: Special services including data interpretation, special consultation, and raw data packages, when requested are included in the final report.

9. INTERNAL QC CHECKS

The QA/QC program monitors data quality with internal QC checks which are used to determine if all laboratory operations are "in control," (i.e., operating within acceptable QC guidelines), during data generation.

Responsibility for internal QC checks rests with the QA Officer and with the individual

analyst. These QC checks include instrument calibration checks, chemical monitoring of dilution waters, specific test validity requirements, and a reference toxicant monitoring program which includes the generation of test control charts.

Instrument QC Checks

All analytical instruments will be calibrated prior to use as set forth in ATL SOP's. Whenever calibration cannot be achieved or measurement of a calibration standard is not within specified limits, the instrument will be considered malfunctioning and will be reported to the Laboratory Director. Any malfunctioning instrument will not be used until appropriate maintenance or repairs are performed and documented.

Chemical Monitoring Of Dilution Waters

In order to establish and continuously monitor the acceptability of the dilution waters utilized in toxicity tests, the dilution waters will be monitored continuously, for deionized water, or at least twice per year for field collected seawater. Dilution waters are to be analyzed to the parameters listed in the appropriate SOP. Results of such analyses are to be maintained in appropriate dilution log books.

Test Validity Requirements

Due to the wide range of test guidelines utilized in toxicity testing, the requirements to determine the validity of any test conducted will be stated in the appropriate SOP. Generally, all acute toxicity tests will be required to meet the following criteria for acceptability:

* No more than a total of 10 percent of the control organisms may appear to be diseased, stressed, or die in a test.

* Appropriate testing conditions, (i.e., temperature, light/dark cycles), are maintained during the course of testing.

Reference Toxicant Monitoring Program

The QA Officer will obtain reference toxicants from the EPA Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, (Telephone No.: (513) 569-7325), or from another reputable commercial supplier. Generally, sodium dodecylsulfate (SDS) will be the reference toxicant of choice, however, in some instances or for certain species other reference toxicants may be utilized.

Reference toxicant tests will be performed on each new batch of test organisms received at the laboratory or on a monthly basis for organisms cultured in-house. Appropriate reference toxicant testing will be conducted concurrently with sample testing when required by test methodology or by the client.

Control Charts

Control charts are to be established and continuously maintained for each organism and test conducted at ATL. Control charts should monitor appropriate test endpoints such as LC50 and NOEC values obtained from the reference toxicant testing program. The control chart is used to evaluate the cumulative trend of the statistics from a series of tests. For point estimation techniques, the mean and upper and lower control limits (+/- 2 times the mean toxicity value standard deviation) are re-calculated with each successive point, until the statistics stabilize. Outliers, which are values which fall outside the upper and lower control limits, and trends of increasing or decreasing sensitivity are readily identified. Note: at the 0.05 probability level, one in 20 tests would be expected to fall outside of the control limits by chance alone. For hypothesis testing results, the same concentrations of reference toxicants are used for each toxicity test. The NOEC from each successive test is entered on the control chart, and the values should fall within one concentration interval above or below the central tendency.

Control charts are to be established based on five successfully completed reference toxicant tests with control limits recalculated with each successive valid reference toxicant test data endpoint. Control charts are used to monitor test organism sensitivity for both commercially obtained and in-house cultured test organisms. If a control chart data point falls outside the established control limits, corrective action must be taken to determine the cause of the discrepancy.

Laboratory Performance QC Program

Laboratory Performance QC is provided as a standard part of every analysis. The main elements of Laboratory Performance QC are:

* Organism survival and reproduction;

* The analysis of reference toxicants

* The generation of daily calibration data.

Satisfactory laboratory performance is demonstrated by performing at least one acceptable reference toxicant test per month for each of the toxicity test methods commonly used in the laboratory. Reference toxicant tests are to be conducted concurrently with less frequently performed tests. If the toxicity value from a given test with the reference toxicant does not fall in 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 should be examined for defects and should be repeated with a different batch of test organisms.

Please refer to section 6 of this manual for a discussion of calibration procedures.

10. PERFORMANCE AND SYSTEM AUDITS

ATL participates in a variety of federal and state certification programs, (i.e., EPA's DMR study and California's ELAP program), that subject the laboratory to stringent system and performance audits on a regular basis. A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff and procedures in place to generate acceptable data. A performance audit verifies the ability of the laboratory to correctly identify toxicity in blind check samples submitted by the auditing agency. The purpose of these audits is to identify those laboratories that are capable of generating scientifically sound data. A list of current ATL certifications is available upon request.

In addition to external audits conducted by certifying agencies or by clients, the QA Officer periodically conducts system and performance audits of the laboratory to verify that only quality, scientifically sound, data are being generated.

11. PREVENTIVE MAINTENANCE

To minimize downtime and interruption of analytical and/or biological work, preventive maintenance is routinely performed. Designated laboratory personnel are trained in routine maintenance procedures for all major equipment. When repairs are necessary, they are performed by either trained staff or trained service engineers employed by the manufacturer or qualified service company personnel.

Detailed SOP's are on file that describes preventive maintenance procedures. The laboratory also maintains a detailed logbook documenting the preventive maintenance and repairs performed on each analytical instrument or piece of equipment.

12. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA QUALITY

The effectiveness of a QA program is measured by the quality of data generated by the laboratory. Data quality is judged in terms of its precision, accuracy, representativeness, completeness and comparability. These terms are described as follows:

Precision

Precision is the degree to which the measurement is reproducible. Precision can be assessed by replicate measurements of reference toxicants or environmental samples. The standard deviation of replicate measurements of a single sample is commonly used in estimating precision. The sample coefficient of variation or CV, (also known as the relative standard deviation), expresses the standard deviation as a percentage of the mean, where CV = 100(std. dev./mean).

In the case of duplicates, the relative percent difference (RPD) between two samples may be used to estimate precision. $RPD = [|X_1 - X_2| / ((X_1 + X_2)/2)]*100$

The ability of the laboratory personnel to obtain consistent, precise results must be demonstrated with reference toxicants before they attempt to measure effluent toxicity. The single laboratory precision of each type of test to be used in a laboratory should be determined by performing at least five or more toxicity tests with a reference toxicant. In cases where the test data are used to obtain point estimates, such as LCs, ECs, or ICs, precision can be described by the mean, standard deviation, and relative standard deviation (percent coefficient of variation, or CV) of the calculated endpoints from the replicated tests. However, in cases where the results are reported in terms of the NOEC and LOEC, precision can only be described by listing the NOEC-LOEC interval for each test. In this case, it is not possible to express precision in terms of a commonly used statistic. For instance, when all tests of the same toxicant yield the same NOEC-LOEC interval, maximum precision has been attained. However, the "true" no effect concentration could fall anywhere within the interval, NOEC +/- (NOEC-LOEC).

The dilution factor selected for a test determines the width of the NOEC-LOEC interval and the inherent maximum precision of the test. As the absolute value of the dilution factor decreases, the width of the NOEC-LOEC interval increases, and the inherent maximum precision of the test decreases. Other factors which can affect test precision include test organism age, condition, and sensitivity, and temperature control and feeding.

Replication and Test Sensitivity

The sensitivity of the tests will depend in part on the number of replicates, the probability level selected, and the type of statistical analysis. The minimum recommended number of replicates varies with the test and statistical method used. If the variability remains constant, the sensitivity of the test will increase as the number of replicates is increased. See individual test SOP's and protocols for additional information on replication.

Accuracy

Accuracy is a determination of how close the measurement is to the true value. Accuracy can be assessed by comparing testing data to standard reference materials of a known toxicity or value.

Representativeness

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Analytical and/or biological data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix.

Completeness

Completeness is a measurement of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. In addition, all data are reviewed in terms of stated goals in order to determine if the data base is sufficient.

Comparability

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability is ensured through the use of established and approved analytical/biological methods, consistency in the basis of analysis (wet weight, volume, etc.), and consistency in reporting units (ppm, ppb, etc.).

13. CORRECTIVE ACTION

When errors, deficiencies, or out-of-control situations exist, the QA program provides systematic procedures, called "corrective actions," to resolve problems and restore proper functioning to the analytical and/or biological system.

Laboratory personnel are alerted that corrective actions may be necessary if:

* QC data are outside the warning or acceptable limits for precision and accuracy;

* Deficiencies are detected during QA internal or external audits or from the results of performance check samples.

* Inquiries concerning data quality are received from clients.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation procedure for possible errors, checks the instrument calibration, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, director or QA Officer for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA Officer and recorded in the corrective action log book.

14. QUALITY ASSURANCE REPORTS

The reporting system is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. The QA Officer periodically prepares QA reports which include:

* The results of system audits including corrective actions taken;
* Performance evaluation scores and commentaries;
* Results of site visits and audits by regulatory agencies and clients;
* Performance on major contracts;
* Problems encountered and corrective actions taken;
* Holding time violations; and
* Comments and recommendations.

QA Reports are submitted to the Laboratory Director for review and action if necessary.

15. LABORATORY DOCUMENTATION

Complete and accurate documentation of analytical, biological and procedural information is an important part of the QA program. Bound notebooks should be used to maintain detailed records of the test organisms such as species, source, age, date of receipt, and other pertinent information relating to their history and health, and information on the calibration of equipment and instruments, test conditions employed, and test results. Annotations should be made on a real-time basis to prevent loss of information. The following describes different types of documentation used at ATL.

Standard Operating Procedures (SOP's)

Details of analytical, biological and QC protocols are contained in SOP's. SOP's are documents that contain detailed information on the requirements for the correct performance of a laboratory procedure. ATL has five categories of laboratory SOP's:

* Performance of an Analytical Testing Method
* Performance of a Biological Testing Method
* Preparation of Standards and Reagents
* Equipment Operation, Calibration, and Maintenance; and
* General Laboratory Procedures.

Formats for these SOP's are shown in Appendix II.

All SOP's are approved by the QA Officer before being implemented. The distribution of current SOP's and archiving of outdated ones is controlled by the QA Officer who also serves as the Document Custodian.

Laboratory Bench Sheets

Laboratory bench sheets are used to document information from routine laboratory operations, including sample preparation and analysis. Bench sheets are used to ensure that the information is recorded in a complete and organized manner and that the analysis can be reconstructed, if necessary.

Laboratory Notebooks

Laboratory notebooks are used to document information that cannot easily be recorded on bench sheets such as methods development information. Each data entry in a laboratory notebook in initialed and dated by the analyst as the data is being entered.

Control Charts

Control charts are used to visually track precision and accuracy data. These control charts are used to identify trends in the analyses which may indicate a problem with the analytical procedure. When an adverse trend or data point is detected corrective action is performed.

Project Files

The project file consists of a project summary and raw data records. The project summary records includes correspondence from the client, (letters, phone logs, contracts, project plans), copies of preliminary and final reports, chain of custody records, air bills, photographs of samples, QA review checklists when applicable, and the summary file inventory check list. Raw data records include original sample raw data, QC data, bench sheets, and instrument logbook pages pertinent to the project. Contracts, project plans, calibration data and QC data may be stored separately from the project record. All project records must contain cross-references to this information. When a project is complete, all records are passed to the Document Custodian who inventories the file, checks for completeness, and puts the file into document archive.

APPENDIX I

SAMPLE HOLDING TIMES AND COLLECTION INFORMATION

Sample Holding Times And Collection Information

TEST	Container	Volume	Holding Time
Hazardous Waste Tests			
CCR Title 22 (Calif. DOHS 1988)	glass	Screen: 25 gm Definitive: 55 gm	NA* "
NPDES Acute Tests			
Fathead Minnow, *Menidia*, Topsmelt, Mysid	plastic/cubitainer	% Survival: 1 gallon Full (LC50): 2.5 gallons	36 Hours "
Rainbow Trout	plastic/cubitainer	% Survival: 5 gallons Full (LC50): 10 gallons	36 Hours "
Ceriodaphnia, *Daphnia*	plastic/cubitainer	% Survival: 1 liter Full (LC50): 1 liter	36 Hours "
NPDES Chronic Tests			
Fathead Minnow	plastic/cubitainer	2.5 liters/day	36 Hours
Ceriodaphnia	" "	1 liter/day	"
Selenastrum	" "	1 liter	"
3 Species Freshwater Chronics	" "	2.5 gal./2 days	"
Red Abalone Larvae	plastic/cubitainer	1 liter	36 Hours
Giant Kelp	" "	1 liter	"
Topsmelt	" "	1 gal./day	"
Menidia	" "	1 gal./day	"
3 Species Marine Chronics	" "	2.5 gal./2 days	"

* No holding time specified in protocol.

Note: Static-renewal tests may require more than one sample. Chronic static-renewal tests may require multiple day sampling, ie. collecting samples on a Monday, Wednesday and Friday.

APPENDIX II

FORMATS FOR STANDARD OPERATING PROCEDURES (SOP's)

FORMAT FOR SOP - LABORATORY ANALYTICAL METHOD

Title (includes method number)

1.0 Scope and Application

- 1.1 Analytes
- 1.2 Detection limit (instrument and method)
- 1.3 Applicable matrices
- 1.4 Dynamic range
- 1.5 Approximate analytical time

2.0 Method Summary

Generic description of method and chemistry behind it.

3.0 Comments

- 3.1 Interferences
- 3.2 Helpful hints

4.0 Safety Issues

5.0 Sample Collection, Preservation, Containers, and Holding Times

6.0 Apparatus

7.0 Reagents and Standards

8.0 Procedure (detailed step-by-step)

- 8.1 Sample preparation
- 8.2 Calibration
- 8.3 Analysis

9.0 QA/QC Requirements

- 9.1 QC samples
- 9.2 Acceptance criteria (precision and accuracy)
- 9.3 Corrective action required (reference current QC manual)

10.0 Calculations

11.0 Reporting

- 11.1 Reporting units
- 11.2 Reporting limits
- 11.3 Significant figures

12.0 References

- 12.1 Method source
- 12.2 Deviations from source method and rationale

13.0 Appendices (optional)

Additional information may be placed in appendices. This may include supporting data (e.g. method validation information), tables, flow charts, etc.

FORMAT FOR SOP - LABORATORY BIOLOGICAL METHOD

Title (includes method number, if applicable)

1.0 Scope and Application

- 1.1 Organism(s)
  - 1.1.1 Source
  - 1.1.2 How identified
  - 1.1.3 Authority
- 1.2 Response
- 1.3 Analysis
- 1.4 Approximate analytical time

2.0 Method Summary

Generic description of method and chemistry behind it.

3.0 Comments

- 3.1 Definitions
- 3.2 Helpful hints
- 3.3 Comments

4.0 Safety Issues

5.0 Sample Collection, Preservation, Containers, and Holding Times

- 5.1 Toxicant
- 5.2 Preservation
- 5.3 Containers
- 5.4 Holding Time

6.0 Equipment

7.0 Reagents and Standards

- 7.1 Reagents
- 7.2 Standards

8.0 Procedure (detailed step-by-step)

- 8.1 Sample preparation
- 8.2 Organism preparation
- 8.3 Equipment and calibration of equipment
- 8.4 Analysis
- 8.5 Monitoring parameters
- 8.6 Organism disposal
- 8.7 Data analysis
  - 8.7.1 Statistics required
  - 8.7.2 Technique
  - 8.7.3 Reasoning and interpretation
- 8.9 Other

9.0 Record Keeping

- 9.1 Lab notebooks
- 9.2 Bench sheets
- 9.3 Other

10.0 Reporting

- 10.1 Reporting units
- 10.2 Reporting limits
- 10.3 Significance of values
- 10.4 Other

11.0 QA/QC Requirements

- 11.1 QC controls
- 11.2 Reference Toxicant
- 11.3 QC Acceptance criteria
  - 11.3.1 Precision and accuracy
  - 11.3.2 Water Quality parameters
  - 11.3.3 Other

11.4 Inspections
11.5 Audits
11.6 Special considerations (client requests)
11.7 Corrective action required (reference current QC manual)
11.8 Other

12.0 References

12.1 Method source
12.2 Deviations from source method and rationale

13.0 Responsibilities

14.0 Appendices (optional)

Additional information may be placed in appendices. This may include supporting data (e.g. method validation information), tables, flow charts, etc.

FORMAT FOR SOP - LABORATORY PROCEDURE

Title (includes method number)

1.0 Purpose

2.0 Policies

3.0 Safety Issues

4.0 Procedure (detailed step-by-step)

5.0 Responsibilities

6.0 Comments

7.0 Definitions

8.0 References

FORMAT FOR SOP - LABORATORY STANDARDS AND REAGENTS

Title

1.0 Reagent/standard name

2.0 Type

3.0 Constituents/concentration

4.0 Solvent

5.0 Safety Issues

6.0 Shelf life and storage

- 6.1 Neat material
- 6.2 Prepared solution
- 6.3 Other

7.0 Procedure (detailed step-by-step)

- 7.1 Preparation for use
- 7.2 Documentation
  - 7.2.1 Purchase date
  - 7.2.2 Source
  - 7.2.3 Purity
  - 7.2.4 Date opened
  - 7.2.5 Labeling
  - 7.2.6 Other
- 7.3 Verification
- 7.4 Usage

8.0 Responsibilities

9.0 Comments

10.0 Definitions

11.0 References

Aquatic
Testing
Laboratories

REFERENCE TOXICANT DATA

FATHEAD MINNOW ACUTE
Method 2000.0
Reference Toxicant - SDS

Aquatic Testing Laboratories

QA/QC Batch No.: RT-100202

TEST SUMMARY

Species: *Pimephales promel*as.
Age: 13 days old.
Regulations: NPDES.
Test chamber volume: 250 ml.
Feeding: Prior to renewal at 48 hrs.
Temperature: 20 +/- 1°C.
Number of replicates: 2.
Dilution water: MHSF.

Source: In-lab culture.
Test type: Static-Renewal.
Test Protocol: EPA-821-R-02-012.
Endpoints: LC50 at 96 hrs.
Test chamber: 600 ml beakers.
Aeration: None.
Number of organisms per chamber: 10.
Photoperiod: 16/8 hrs light/dark.

TEST DATA

	INITIAL			24 Hr					48 Hr				
Date/Time:	2-2-10 1200			2-3-10 1300					2-4-10 1200				
Analyst:													
	°C	DO	pH	°C	DO	pH	# Dead A	# Dead B	°C	DO	pH	# Dead A	# Dead B
Control	19.6	8.4	7.6	19.4	7.9	7.4	0	0	19.2	7.1	7.9	0	0
1.0 mg/l	19.6	8.5	7.6	19.2	8.0	7.4	0	0	19.2	7.3	7.7	0	0
2.0 mg/l	19.6	8.5	7.7	19.1	8.0	7.4	0	0	19.1	7.2	7.6	0	0
4.0 mg/l	19.6	8.5	7.7	19.1	7.6	7.4	0	0	19.1	7.2	7.6	0	0
8.0 mg/l	19.6	8.6	7.7	19.0	6.8	7.3	10	10	—	—	—	—	—

	RENEWAL			72 Hr					96 Hr				
Date/Time:	2-4-10 1200			2-5-10 1200					2-6-10 1130				
Analyst:													
	°C	DO	pH	°C	DO	pH	# Dead A	# Dead B	°C	DO	pH	# Dead A	# Dead B
Control	19.5	8.8	7.8	19.5	7.4	7.4	0	0	20.6	6.3	7.4	0	0
1.0 mg/l	19.5	8.8	7.8	19.4	7.4	7.4	0	0	20.6	6.6	7.4	0	0
2.0 mg/l	19.5	8.9	7.8	19.2	7.4	7.4	0	0	20.6	6.5	7.4	0	0
4.0 mg/l	19.5	8.9	7.8	19.2	7.3	7.4	0	0	20.5	6.4	7.4	0	0
8.0 mg/l	—	—	—	—	—	—	—	—	—	—	—	—	—

Comments: Control: Alkalinity: 69 mg/l; Hardness: 94 mg/l; Conductivity: 330 umho.
SDS: Alkalinity: 68 mg/l; Hardness: 94 mg/l; Conductivity: 333 umho.

Concentration-response relationship acceptable? (see attached computer analysis):
Yes (response curve normal)
No (dose interrupted indicated or non-normal)

Acute Fish Test-96 Hr Survival

Start Date:	2/2/2010 12:00	Test ID:	RT100202f	Sample ID:	REF-Ref Toxicant
End Date:	2/6/2010 11:30	Lab ID:	CAATL-Aquatic Testing Labs	Sample Type:	SDS-Sodium dodecyl sulfate
Sample Date:	2/2/2010	Protocol:	ACUTE-EPA-821-R-02-012	Test Species:	PP-Pimephales promelas
Comments:					

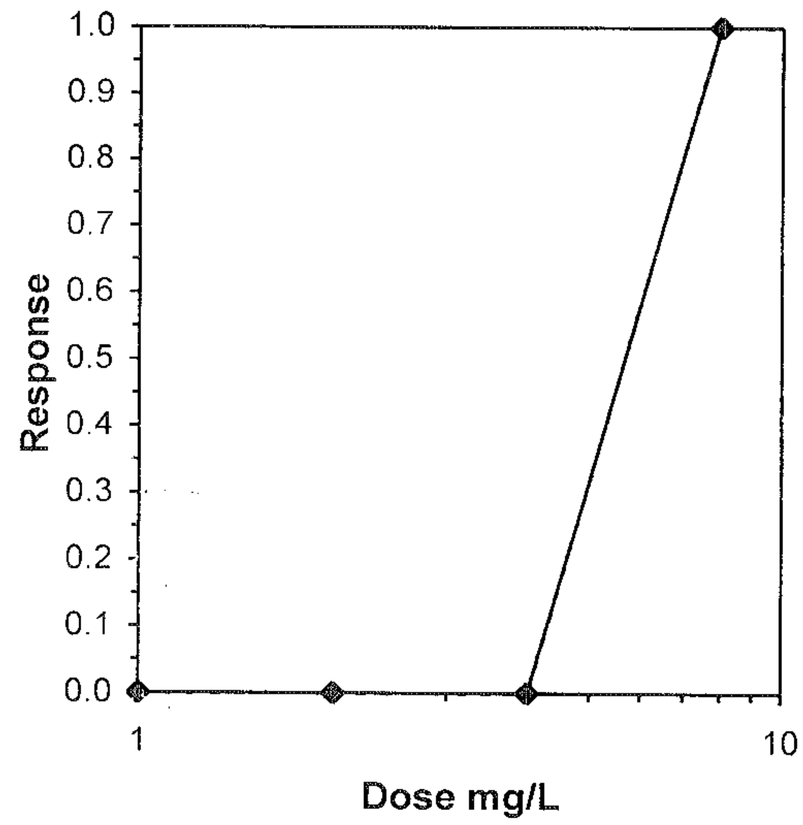
Conc-mg/L	1	2
D-Control	1.0000	1.0000
1	1.0000	1.0000
2	1.0000	1.0000
4	1.0000	1.0000
8	0.0000	0.0000

			Transform: Arcsin Square Root					Number	Total
Conc-mg/L	Mean	N-Mean	Mean	Min	Max	CV%	N	Resp	Number
D-Control	1.0000	1.0000	1.4120	1.4120	1.4120	0.000	2	0	20
1	1.0000	1.0000	1.4120	1.4120	1.4120	0.000	2	0	20
2	1.0000	1.0000	1.4120	1.4120	1.4120	0.000	2	0	20
4	1.0000	1.0000	1.4120	1.4120	1.4120	0.000	2	0	20
8	0.0000	0.0000	0.1588	0.1588	0.1588	0.000	2	20	20

Auxiliary Tests	Statistic	Critical	Skew	Kurt
Normality of the data set cannot be confirmed				
Equality of variance cannot be confirmed				

Graphical Method

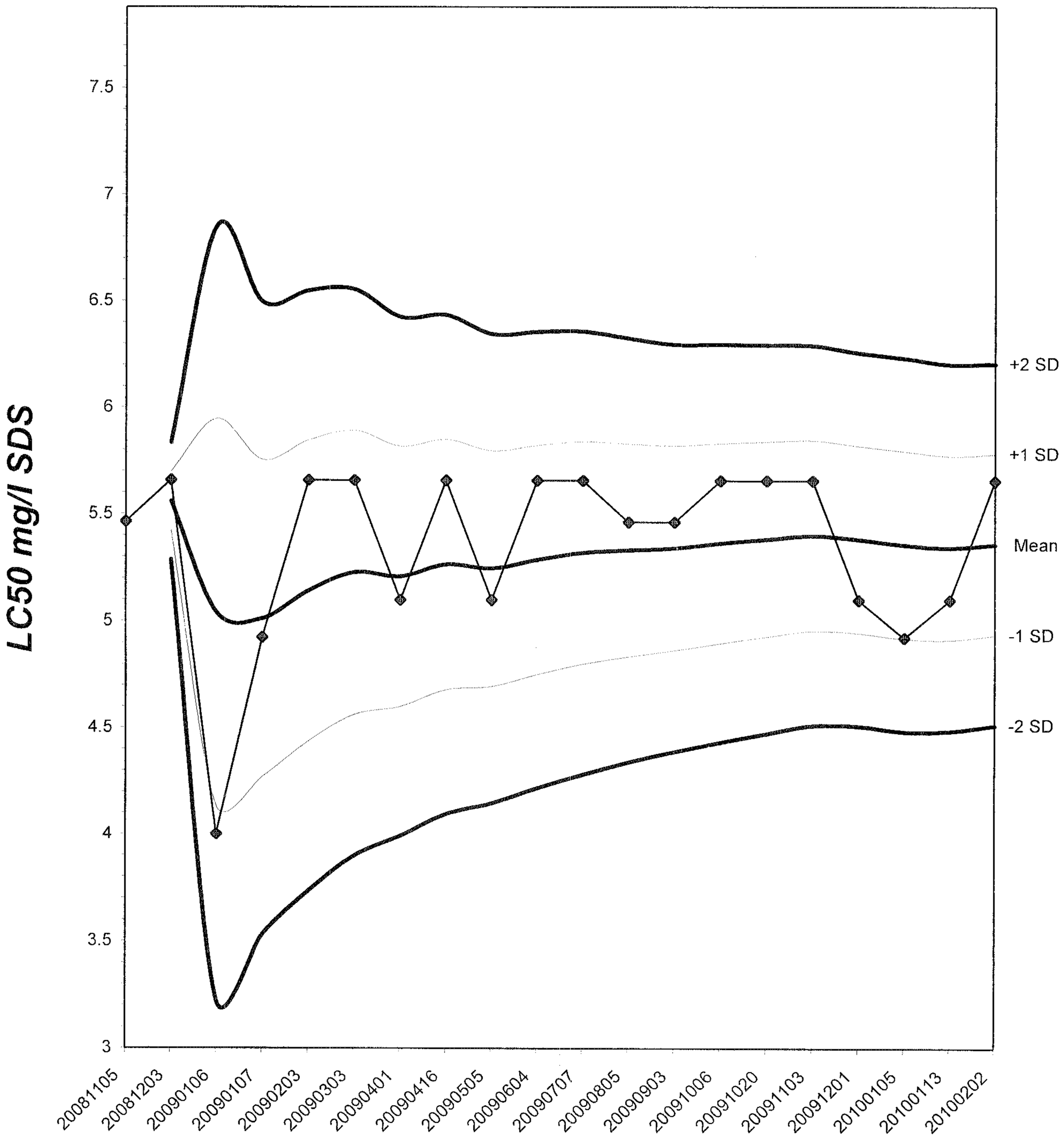
Trim Level	EC50
0.0%	5.6569
	5.6569



Reviewed by:

Fathead Minnow Acute Laboratory Control Chart

CV% = 7.91



TEST ORGANISM LOG

Aquatic Testing Laboratories

FATHEAD MINNOW - LARVAL
(*Pimephales promelas*)

QA/QC BATCH NO.: RT-100202

SOURCE: In-Lab Culture

DATE HATCHED: 1-20-10

APPROXIMATE QUANTITY: 400

GENERAL APPEARANCE: Good

 # MORTALITIES 48 HOURS PRIOR TO TO USE IN TESTING: 0

DATE USED IN LAB: 1 / 5 / 10

AVERAGE FISH WEIGHT: 0.006 **gm**

LOADING LIMITS: 0.65 gm/liter @ 20°C, 0.40 gm/liter @ 25°C

Approximately 1000 fish per 10 liters limit if held overnight for acclimation without filtration @ 20°C for fish with a mean weight of 0.006 gm.

Approximately 650 fish per 10 liters limit if held overnight for acclimation without filtration @ 25°C for fish with a mean weight of 0.006 gm.

200 ml test solution volume = 0.013 gm mean fish weight limit @ 20°C; 0.008 @ 25°C
250 ml test solution volume = 0.016 gm mean fish weight limit @ 20°C; 0.010 @ 25°C

ACCLIMATION WATER QUALITY:

Temp.: 19.6 **°C** **pH:** 7.6 **Ammonia:** <0.1 **mg/l NH_3-N**

DO: 8.4 **mg/l** **Alkalinity:** 69 **mg/l** **Hardness:** 94 **mg/l**

READINGS RECORDED BY: _____ **DATE:** 2-3-10

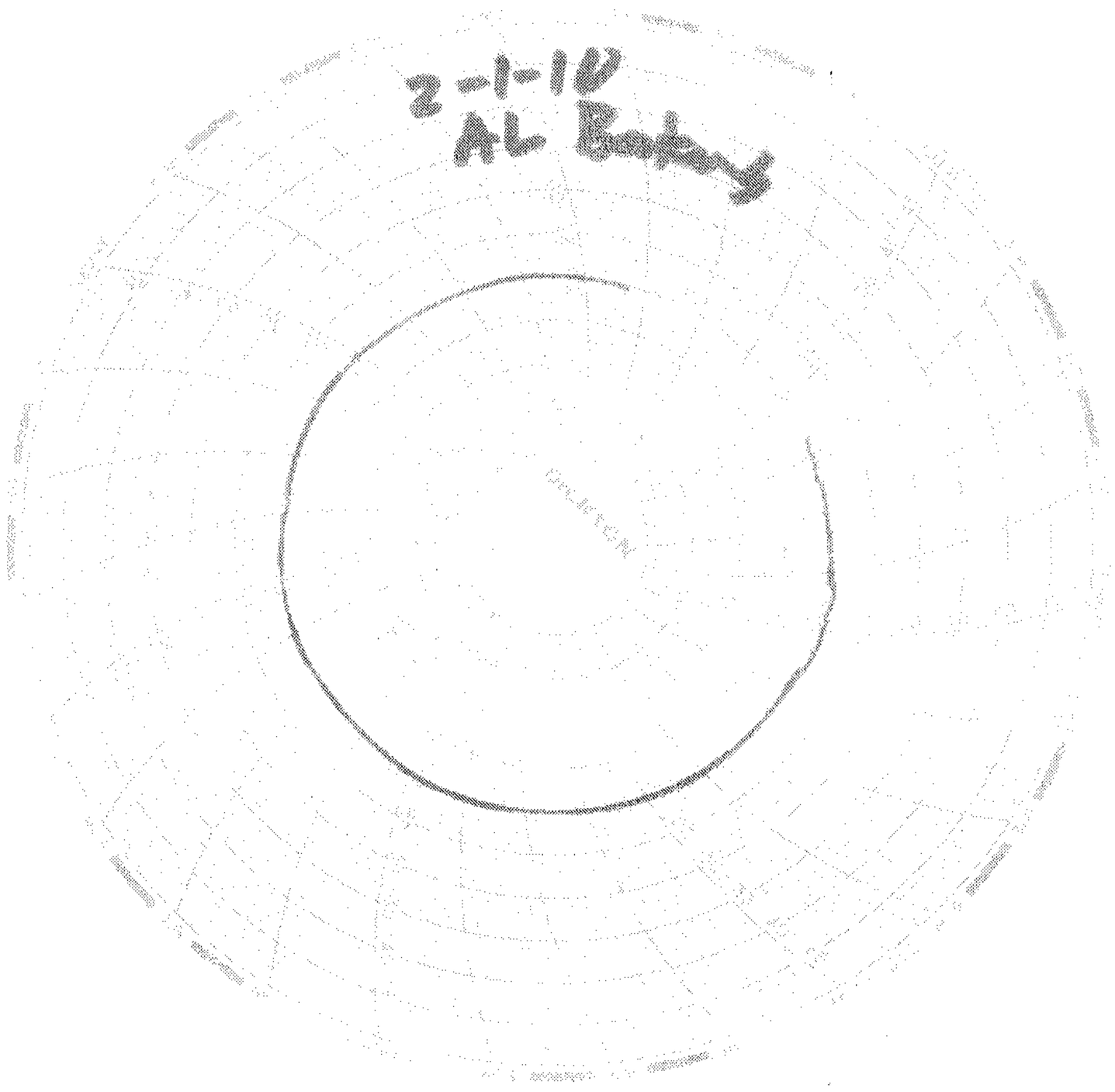
Aquatic Testing Laboratories

Test Temperature Chart

Test No: RT-100202

Date Tested: 02/02/10 to 02/06/10

Acceptable Range: 20+/- 1°C



Aquatic
Testing
Laboratories

Ceriodaphnia dubia
Chronic Toxicity Test
Reference
Toxicant
Data

CERIODAPHNIA CHRONIC BIOASSAY
EPA METHOD 1002.0
REFERENCE TOXICANT - NaCl

Aquatic Testing Laboratories

QA/QC Batch No.: RT-100207

Date Tested: 02/07/10 to 02/14/10

TEST SUMMARY

Test type: Daily static-renewal.
Species: *Ceriodaphnia dubia.*
Age: <24 hrs; all released within 8 hrs.
Test vessel size: 30 ml.
Number of test organisms per vessel: 1.
Temperature: 25 +/- 1°C.
Dilution water: Mod. hard reconstituted (MHRW).
Reference Toxicant: Sodium chloride (NaCl).

Endpoints: Survival and Reproduction.
Source: In-laboratory culture.
Food: .1 ml YTC, algae per day.
Test solution volume: 20 ml.
Number of replicates: 10.
Photoperiod: 16/8 hrs. light/dark cycle.
Test duration: 7 days.
Statistics: ToxCalc computer program.

RESULTS SUMMARY

Sample Concentration	Percent Survival		Mean Number of Young Per Female	
Control	100%		28.5	
0.25 g/l	100%		30.9	
0.5 g/l	100%		25.5	
1.0 g/l	100%		15.4	*
2.0 g/l	100%		2.9	*
4.0 g/l	0%	*	0	**

* Statistically significantly less than control at P = 0.05 level
** Reproduction data from concentrations greater than survival NOEC are excluded from statistical analysis.

CHRONIC TOXICITY

Survival LC50	2.8 g/l
Reproduction IC25	0.66 g/l

QA/QC TEST ACCEPTABILITY

Parameter	Result
Control survival ≥80%	Pass (100% Survival)
≥15 young per surviving control female	Pass (28.5 young)
≥60% surviving controls had 3 broods	Pass (100% with 3 broods)
PMSD <47% for reproduction	Pass (PMSD = 14.7%)
Stat. sig. diff. conc. relative difference >13%	Pass (Stat. sig. diff. conc. Relative difference= 46.0%)
Concentration response relationship acceptable	Pass (Response curve normal)

Ceriodaphnia Survival and Reproduction Test-7 Day Survival

Start Date:	2/7/2010 15:00	Test ID:	RT100207c	Sample ID:	REF-Ref Toxicant
End Date:	2/14/2010 14:00	Lab ID:	CAATL-Aquatic Testing Labs	Sample Type:	NACL-Sodium chloride
Sample Date:	2/7/2010	Protocol:	FWCH EPA	Test Species:	CD-Ceriodaphnia dubia
Comments:					

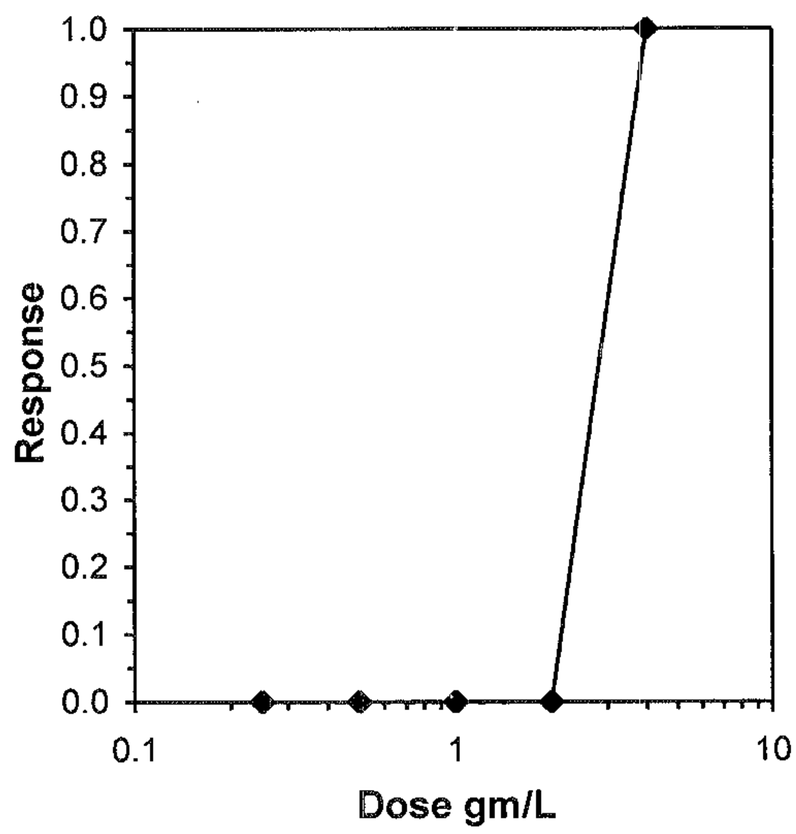
Conc-gm/L	1	2	3	4	5	6	7	8	9	10
D-Control	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
0.25	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
0.5	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
1	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
4	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Conc-gm/L	Mean	N-Mean	Not Resp	Resp	Total	N	Fisher's Exact P	1-Tailed Critical	Number Resp	Total Number
D-Control	1.0000	1.0000	0	10	10	10			0	10
0.25	1.0000	1.0000	0	10	10	10	1.0000	0.0500	0	10
0.5	1.0000	1.0000	0	10	10	10	1.0000	0.0500	0	10
1	1.0000	1.0000	0	10	10	10	1.0000	0.0500	0	10
2	1.0000	1.0000	0	10	10	10	1.0000	0.0500	0	10
4	0.0000	0.0000	10	0	10	10			10	10

Hypothesis Test (1-tail, 0.05)	NOEC	LOEC	ChV	TU
Fisher's Exact Test	2	4	2.82843	
Treatments vs D-Control				

Graphical Method

Trim Level	EC50
0.0%	2.8284
	2.8284

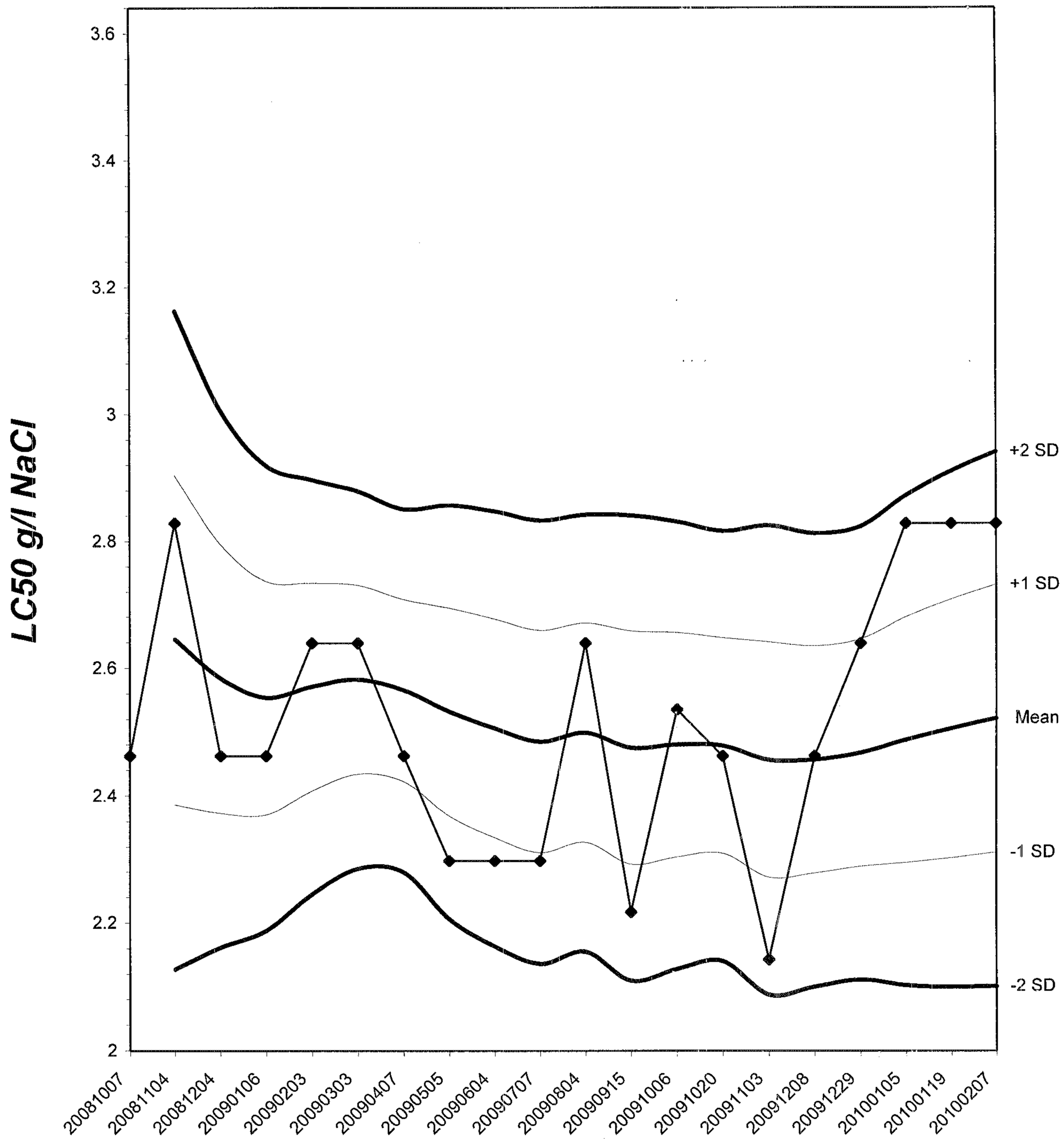


ToxCalc v5.0.23

Reviewed by:

Ceriodaphnia Chronic Survival Laboratory Control Chart

CV% = 8.34



Ceriodaphnia Survival and Reproduction Test-Reproduction

Start Date:	2/7/2010 15:00	Test ID:	RT100207c	Sample ID:	REF-Ref Toxicant
End Date:	2/14/2010 14:00	Lab ID:	CAATL-Aquatic Testing Labs	Sample Type:	NACL-Sodium chloride
Sample Date:	2/7/2010	Protocol:	FWCH EPA	Test Species:	CD-Ceriodaphnia dubia
Comments:					

Conc-gm/L	1	2	3	4	5	6	7	8	9	10
D-Control	30.000	29.000	30.000	32.000	29.000	30.000	30.000	25.000	26.000	24.000
0.25	48.000	29.000	31.000	31.000	27.000	27.000	28.000	36.000	25.000	27.000
0.5	27.000	26.000	26.000	28.000	25.000	25.000	30.000	25.000	18.000	25.000
1	24.000	13.000	15.000	19.000	24.000	13.000	11.000	13.000	11.000	11.000
2	3.000	3.000	2.000	3.000	2.000	3.000	4.000	4.000	2.000	3.000
4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

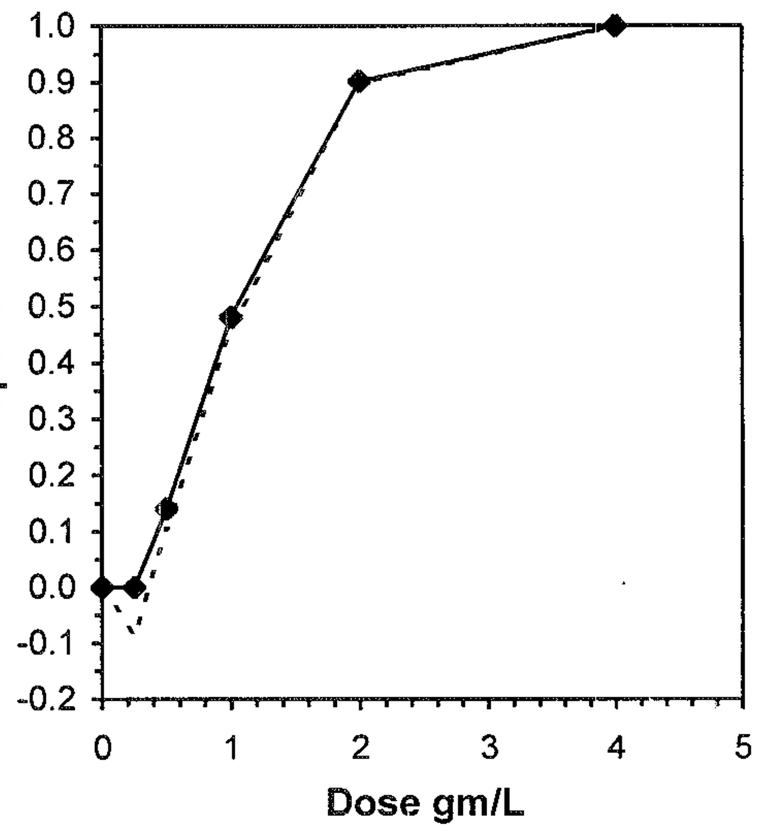
			Transform: Untransformed					Rank	1-Tailed	Isotonic	
Conc-gm/L	Mean	N-Mean	Mean	Min	Max	CV%	N	Sum	Critical	Mean	N-Mean
D-Control	28.500	1.0000	28.500	24.000	32.000	9.097	10			29.700	1.0000
0.25	30.900	1.0842	30.900	25.000	48.000	21.867	10	110.50	76.00	29.700	1.0000
0.5	25.500	0.8947	25.500	18.000	30.000	12.158	10	79.00	76.00	25.500	0.8586
*1	15.400	0.5404	15.400	11.000	24.000	33.280	10	56.00	76.00	15.400	0.5185
*2	2.900	0.1018	2.900	2.000	4.000	25.444	10	55.00	76.00	2.900	0.0976
4	0.000	0.0000	0.000	0.000	0.000	0.000	10			0.000	0.0000

Auxiliary Tests	Statistic	Critical	Skew	Kurt
Shapiro-Wilk's Test indicates non-normal distribution (p <= 0.05)	0.87968	0.947	1.72192	5.90298
Bartlett's Test indicates unequal variances (p = 1.75E-06)	32.1843	13.2767		

Hypothesis Test (1-tail, 0.05)	NOEC	LOEC	ChV	TU
Steel's Many-One Rank Test	0.5	1	0.70711	
Treatments vs D-Control				

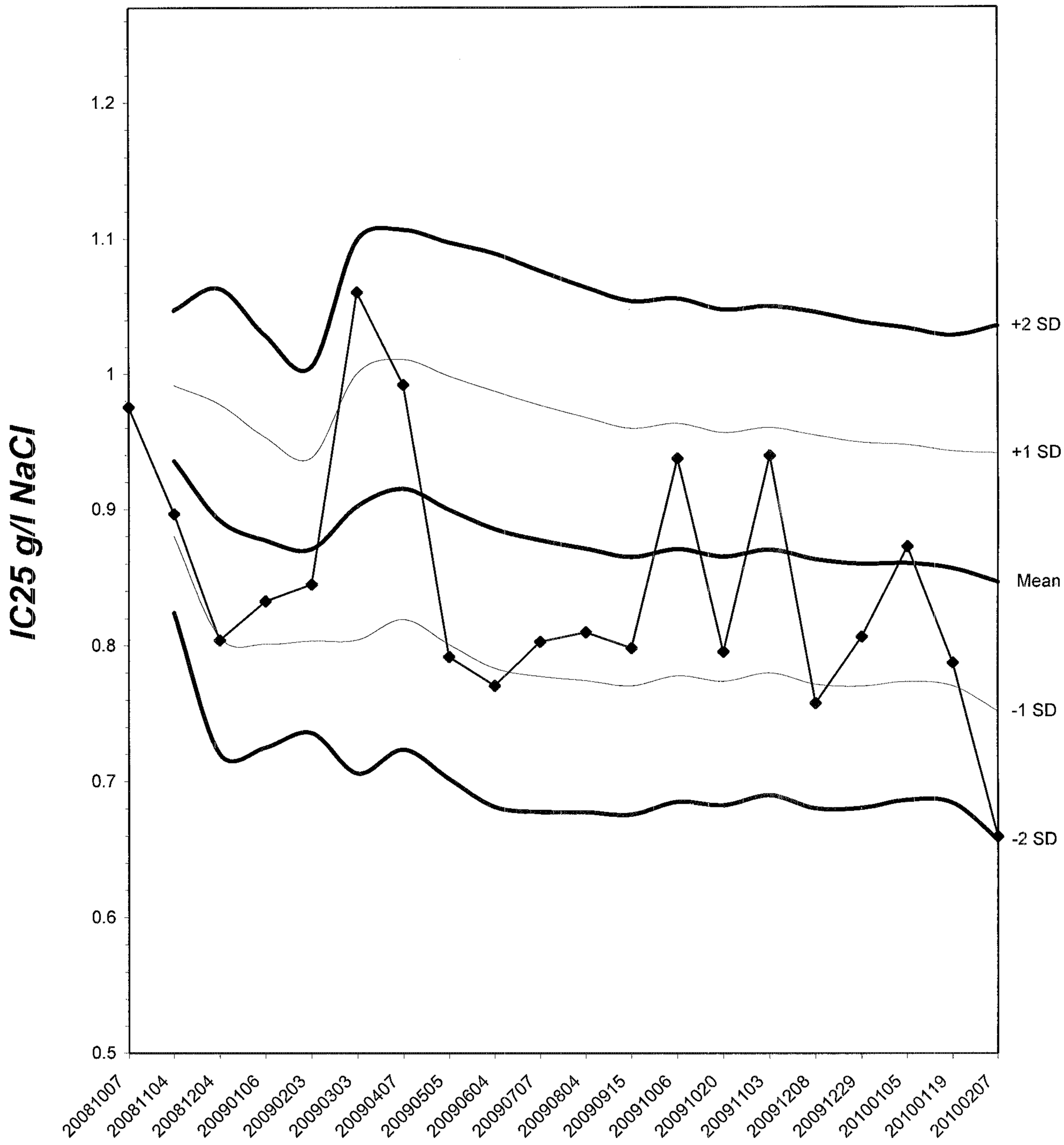
Linear Interpolation (200 Resamples)

Point	gm/L	SD	95% CL		Skew
IC05	0.3384	0.0442	0.2691	0.4525	0.4001
IC10	0.4268	0.0548	0.3537	0.5444	0.4118
IC15	0.5126	0.0553	0.4160	0.6069	0.0105
IC20	0.5861	0.0571	0.4714	0.6748	-0.2745
IC25	0.6597	0.0572	0.5402	0.7608	-0.3338
IC40	0.8802	0.0645	0.7629	1.0101	0.4008
IC50	1.0440	0.0882	0.8903	1.2112	0.2244



Ceriodaphnia Chronic Reproduction Laboratory Control Chart

CV% = 11.2



CERIODAPHNIA DUBIA CHRONIC BIOASSAY
Reference Toxicant - NaCl
Reproduction and Survival Raw Data Sheet

Aquatic Testing Laboratories

QA/QC No.: RT-100207

Start Date:02/07/2010

Sample	Day	Number of Young Produced										Total Live Young	No. Live Adults	Analyst Initials
		A	B	C	D	E	F	G	H	I	J			
Control	1	0	0	0	0	0	0	0	0	0	0	0	10	
	2	0	0	0	0	0	0	0	0	0	0	0	10	
	3	5	0	4	4	3	4	4	4	3	4	35	10	
	4	0	5	0	0	0	9	10	7	9	9	49	10	
	5	8	8	12	11	10	0	16	14	14	11	104	10	
	6	0	0	0	0	0	17	(17)	(15)	(17)	(12)	17	10	
	7	17	16	14	17	16	(15)	0	0	0	0	80	10	
	Total	30	29	30	32	29	30	30	25	26	24	285	10	
0.25 g/l	1	0	0	0	0	0	0	0	0	0	0	0	10	
	2	0	0	0	0	0	0	0	0	0	0	0	10	
	3	0	4	4	4	5	3	4	0	4	3	31	10	
	4	0	0	0	0	9	8	11	10	9	0	47	10	
	5	11	8	8	10	13	0	13	11	12	8	94	10	
	6	18	17	19	17	(15)	16	(15)	0	(17)	16	103	10	
	7	19	0	(17)	(16)	0	(17)	0	15	0	(15)	34	10	
	Total	88	29	31	31	27	27	28	36	25	27	309	10	
0.5 g/l	1	0	0	0	0	0	0	0	0	0	0	0	10	
	2	0	0	0	0	0	0	0	0	0	0	0	10	
	3	2	0	3	0	3	3	0	0	4	3	18	10	
	4	0	4	4	2	5	0	6	4	6	5	36	10	
	5	7	5	0	0	0	7	8	6	8	0	41	10	
	6	18	17	19	12	17	0	16	0	0	0	99	10	
	7	0	0	0	14	(16)	15	0	15	(14)	17	61	10	
	Total	27	26	26	28	25	25	30	25	18	25	255	10	

Circled fourth brood not used in statistical analysis.
7th day only used if <60% of the surviving control females have produced their third brood.

CERIODAPHNIA DUBIA CHRONIC BIOASSAY
Reference Toxicant - NaCl
Reproduction and Survival Raw Data Sheet

Aquatic Testing Laboratories

QA/QC No.: RT-100207

Start Date:02/07/2010

Sample	Day	Number of Young Produced										Total Live Young	No. Live Adults	Analyst Initials
		A	B	C	D	E	F	G	H	I	J			
1.0 g/l	1	0	0	0	0	0	0	0	0	0	0	0	10	
	2	0	0	0	0	0	0	0	0	0	0	0	10	
	3	(3)	0	2	3	3	0	0	2	2	0	15	10	
	4	0	2	5	2	4	0	0	3	3	0	19	10	
	5	5	4	0	0	0	6	4	0	0	0	19	10	
	6	0	0	0	14	17	0	0	0	0	4	35	10	
	7	16	7	8	0	0	7	7	8	6	7	66	10	
	Total	24	13	15	19	24	13	11	13	11	11	154	10	
2.0 g/l	1	0	0	0	0	0	0	0	0	0	0	0	10	
	2	0	0	0	0	0	0	0	0	0	0	0	10	
	3	0	0	0	0	0	0	0	0	0	0	0	10	
	4	0	0	0	0	0	0	0	0	0	0	0	10	
	5	0	0	0	0	0	0	0	0	0	0	0	10	
	6	0	0	2	0	0	0	0	3	0	0	5	10	
	7	3	3	0	3	2	3	4	1	2	3	24	10	
	Total	3	3	2	3	2	3	4	4	2	3	29	10	
4.0 g/l	1	(0)	(0)	(0)	(0)	X	X	X	X	X	~	0	0	
	2	—	—	—	—	—	—	—	—	—	—	—	—	
	3	—	—	—	—	—	—	—	—	—	—	—	—	
	4	—	—	—	—	—	—	—	—	—	—	—	—	
	5	—	—	—	—	—	—	—	—	—	—	—	—	
	6	—	—	—	—	—	—	—	—	—	—	—	—	
	7	—	—	—	—	—	—	—	—	—	—	—	—	
	Total	(0)	0	0	0	0	0	0	0	0	0	0	0	

Circled fourth brood not used in statistical analysis.
7th day only used if <60% of the surviving control females have produced their third brood.

C\RIODAPHNIA DUBIA CHRONIC BIOASSAY
Reference Toxicant - NaCl
Water Chemistries Raw Data Sheet

Aquatic Testing Laboratories

QA/QC No.: RT-100207

Start Date:02/07/2010

		DAY 1		DAY 2		DAY 3		DAY 4		DAY 5		DAY 6		DAY 7	
		Initial	Final	Initial	Final	Initial	Final	Initial	Final	Initial	Final	Initial	Final	Initial	Final
Analyst Initials:															
Time of Readings:		1540	1430	1430	1500	1500	1400	1400	1400	1400	1500	1500	1600	1600	1400
Control	DO	8.3	8.3	8.1	8.4	8.2	8.3	8.3	8.2	8.4	8.2	8.1	7.9	8.0	8.0
	pH	7.7	8.0	8.2	8.0	8.0	7.8	8.0	7.8	7.7	7.7	7.7	7.8	7.5	7.6
	Temp	24.3	24.2	24.7	25.0	25.7	25.1	24.4	24.0	25.7	24.8	25.4	25.2	25.9	24.5
0.25 g/l	DO	8.4	8.4	8.2	8.4	8.2	8.3	8.3	8.2	8.4	8.2	8.1	8.0	8.0	7.9
	pH	8.0	7.8	8.0	8.0	8.0	7.8	8.0	7.8	7.7	7.7	7.7	7.8	7.5	7.5
	Temp	24.4	24.2	24.6	25.1	25.8	25.2	24.5	24.2	25.7	24.9	25.4	25.3	25.9	25.0
0.5 g/l	DO	8.2	8.3	8.2	8.3	8.2	8.3	8.3	8.1	8.4	8.2	8.1	8.0	8.0	8.1
	pH	7.9	7.8	7.8	8.0	8.1	7.8	7.8	7.8	7.7	7.7	7.7	7.8	7.6	7.5
	Temp	24.4	24.6	24.4	25.2	25.8	25.4	24.5	24.2	25.7	25.0	25.5	25.4	25.8	24.7
1.0 g/l	DO	8.3	8.4	8.4	8.3	8.3	8.2	8.3	8.1	8.3	8.3	8.2	7.9	8.0	8.0
	pH	7.9	7.8	7.8	8.0	8.1	7.8	7.8	7.8	7.7	7.7	7.7	7.8	7.6	7.6
	Temp	24.4	24.6	24.5	25.2	25.9	25.4	24.6	24.1	25.8	25.0	25.6	25.4	25.8	24.6
2.0 g/l	DO	8.2	8.0	8.4	8.5	8.3	8.2	8.3	8.1	8.3	8.3	8.2	8.1	8.0	8.3
	pH	7.9	7.8	7.7	8.0	8.1	7.8	7.8	7.8	7.7	7.7	7.8	7.8	7.7	7.6
	Temp	24.6	24.8	24.5	25.2	26.0	25.3	24.8	24.1	25.9	25.1	25.8	25.3	25.6	24.7
4.0 g/l	DO	8.3	8.0	—	—	—	—	—	—	—	—	—	—	—	—
	pH	8.1	7.7	—	—	—	—	—	—	—	—	—	—	—	—
	Temp	24.5	25.1	—	—	—	—	—	—	—	—	—	—	—	—

Dissolved Oxygen (DO) readings are in mg/l O_2; Temperature (Temp) readings are in °C.

Additional Parameters	Control			High Concentration		
	Day 1	Day 3	Day 5	Day 1	Day 3	Day 5
Conductivity (µS)	349	335	341	6240	3390	3510
Alkalinity (mg/l $CaCO_3$)	67	68	67	67	68	68
Hardness (mg/l $CaCO_3$)	90	93	92	90	92	92

Source of Neonates

Replicate:	A	B	C	D	E	F	G	H	I	J
Brood ID:	3A	3B	2C	2D	1E	1F	3G	2H	3I	1J

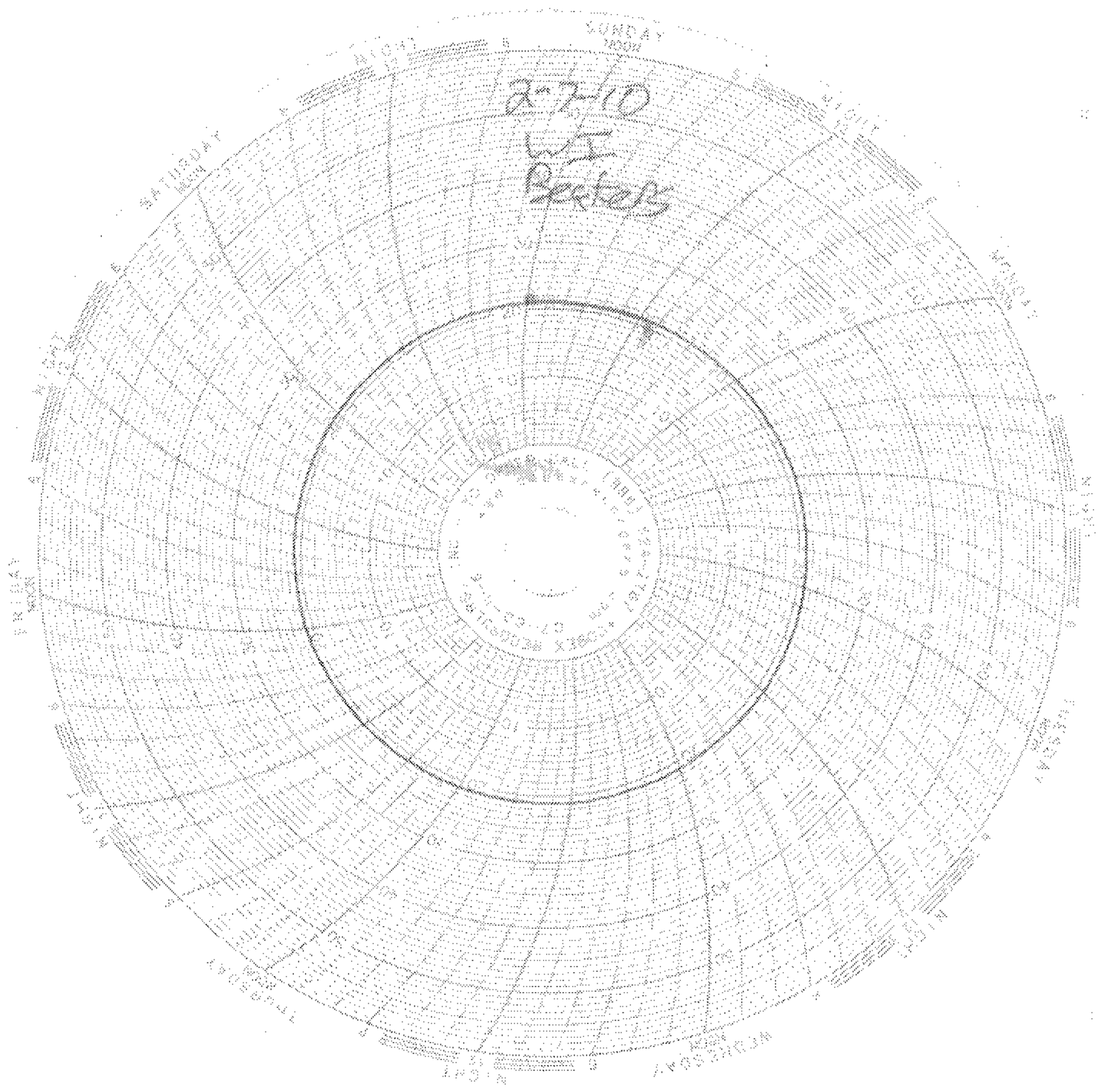
Aquatic Testing Laboratories

Test Temperature Chart

Test No: RT-100207

Date Tested: 02/07/10 to 02/14/10

Acceptable Range: 25+/- 1°C



EMS LABORATORIES, INC.

QUALITY MANUAL

_____
A. J. Kolk, Jr.
Technical Director

Revision #18
September, 2009

ANNUAL REVIEW OF QUALITY MANUAL

Date	Name	Revision Made	
		Yes	No
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_____	_____	☐	☐
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STAFF REVIEW OF QUALITY MANUAL

Date	Name	Comments

CONFIDENTIALITY

Verbal results are given to the customers by the laboratory director, technical director. quality assurance manager or department managers. In their absence, analysts in that department give the results. Verbal results are documented on the Laboratory Tracking Form with name, date, time.

For the sake of confidentiality note the following when giving verbal results:

Only the **Contact Person** indicated by the customer in the submittal/receiving form will be contacted by one of the above mentioned persons from EMS, unless otherwise instructed.

If the customer calls in for the result, get their phone number, verify the number and get back to them.

Only give the results to **EMS Customers**, and not the customer of the customer.

The customers can only get their samples back if it has been agreed upon in the contract, otherwise a written request and lab director's approval is needed.

EMS personnel are only responsible for EMS customers. Those customers that are not directly involved, **must** communicate with EMS through their intermediate companies. No results (verbal, fax, or reports) and/or samples are given out unless the company has a contract with EMS.

To protect electronic transmission of information via fax or email, following statement is placed in the first page as a footnote to protect customer's confidential information and proprietary rights:

"The documents that accompany this FACSIMILE (or EMAIL) transmission may contain confidential information which belongs to EMS Laboratories, Inc. This information is intended only for the use of the individual or entity named above. If you are not the intended recipient of this information or the person responsible for delivering it to the intended recipient, you are hereby notified that any disclosure, copying, distribution or use of any of the information contained in this FAX (or EMAIL) is strictly prohibited. If you have received this FAX (or EMAIL) in error, please notify us immediately by telephone and arrange for the original FAX to be returned to us. Thank you."

I received, read and understood EMS' Confidentiality Policy

_____ _____
Signature Date

Revised 1/8/08

POLICY STATEMENT

EMS Laboratories, Inc., is dedicated to producing quality laboratory services to all its customers. The laboratory will ensure quality data by providing a stable analytic staff with extensive experience in their respective specialties. The laboratory is involved in proficiency testing, internal quality checks, interlaboratory analysis and good laboratory practices as prescribed by accrediting agencies. The laboratory's management is committed to compliance with ISO/IEC 17025.

Management is committed to the best laboratory practices, and to perform all services in a timely manner. The accuracy and the precision of the analytical results will be monitored by sound statistical methods.

The purpose of the management system is to provide high quality data, accurate and precise results, legally defensible, to the customer at reasonable cost and turnaround time.

Every individual engaged in the conduct or responsible for an analysis has the education, training and experience to perform the assigned analysis competently. The staff includes degreed chemists, geologists and biologists.

The laboratory management is committed to comply with the International Standard ISO/IEC 17025 and will continue to improve the effectiveness of the management system. All analytical tests shall be carried out in accordance with stated methods and customers' requirements.

Management reviews of the quality assurance program shall be conducted annually with the Directors, Laboratory Managers, and Quality Assurance Manager. The review is to ensure that EMS' quality system is capable of meeting the laboratory's goal of producing quality data. All aspects will be reviewed in addition to manuals, such as audit results and proficiency evaluation results. Future plans regarding staff and equipment shall be reviewed. Review findings will be documented. The Quality Manager will implement the corrective actions and document that the implementation occurred. All review findings will be kept on file with the QA Analyst.

ACCREDITATIONS AND CERTIFICATIONS

EMS Laboratories is accredited for lead analysis (air, paint, dust, soil, water) under NLLAP. EMS participates in the American Industrial Hygiene Association ELPAT program (Lab No. 101634) for analysis of lead in soils, wipes and paint samples. EMS has always been proficient since the beginning of this program.

EMS Laboratories has been accredited by the American Industrial Hygiene Association since March 1, 1986 (Accreditation 297, Lab No. 101634). EMS Laboratories, Inc., participates in the AIHA PAT program and has been rated proficient for all the analytes in the proficiency program.

EMS Laboratories has been accredited by the California Department of Public Health for Environmental Laboratory Accreditation as an environmental testing laboratory since February 1, 1990 (Certificate No. 1119).

EMS Laboratories is also accredited by U. S. Department of Commerce, National Institute of Standards and Technology through National Voluntary Laboratory Accreditation Program (NVLAP) accreditation for bulk asbestos by PLM since 1989 and airborne asbestos fiber analysis by TEM (Lab Code 101218) since 1990. EMS has passed every round of proficiency testing.

EMS Laboratories, Inc. is accredited by the California Department of Health Services (DOHS), as a hazardous waste testing laboratory. Our industrial hygiene laboratory is accredited by the American Industrial Hygiene Association (AIHA). EMS is accredited for lead analysis under NLLAP.

As an environmental testing laboratory, EMS Laboratories is capable of analysis of water, wastewater, soil and sludge. Our industrial hygiene laboratory is capable of analysis of metals, organics and inorganics in

air samples (GC, HPLC, IC, AA, ICP, GFAA), qualitative and quantitative x-ray diffraction analysis (silica, asbestos), transmission and scanning electron microscopy with energy dispersive x-ray analysis for asbestos, particle analysis.

All the analysts that perform PCM analysis are enrolled in the AIHA Asbestos Analysts Registry (Lab No. 101634). They have been approved by the AIHA Board of Directors in asbestos analysis of air samples by PCM.

Interlaboratory comparisons are made quarterly for asbestos by PLM. Blind duplicates at least on 10% of the samples.

STAFF ASSIGNMENTS

Technical Director

The laboratory technical director is responsible for all technical operations, as well as the functioning and administration of the laboratory in such a manner that provides the necessary resources needed to ensure the required quality of the laboratory operations. The technical director is also responsible for the implementation of any corrective actions performed at the laboratory, address technical issues for laboratory staff and customers, and ensure compliance with ISO/IEC 17025.

QA Analyst

The QA Analyst must possess a bachelor's degree in science and have at least one year of nonacademic analytical experience appropriate to the analysis performed by the laboratory. The QA Analyst must also be trained in statistics and quality control procedures and apply statistical methods for proactively addressing data quality.

The QA Analyst is under the direction of the technical director and responsible for ensuring that the quality assurance and quality control program is implemented**,** followed and is in compliance with ISO/IEC 17025 at all times in the laboratory.

Analyst

The qualification requirement, training program, and responsibility for analyst are defined in each individual Standard Operation Procedures for Chemistry, TEM, PLM, and PCM.

In general the training procedure is as follows: new employees undergo an extensive training program, and the laboratory supervisor or a senior member of the staff conducts the training program. The QA/QC manager keeps all training records in a file.

The analysts are responsible for all aspects of assigned analytical procedures, including overseeing sample preparation and preservation, performing the analysis, reporting the results within the turnaround times. They must adhere to al QC procedures of that analytical method and documentation of these procedures. They are responsible for routine maintenance of their equipment and making certain there are sufficient supplies for analysis.

SAMPLE CUSTODY AND LOGGING

Sample Receipt

Samples are received in the Sample Receiving area. Designated laboratory staff is responsible for logging in the samples under the direction of the Log-in clerk. They are expected to be familiar with the SOP for sample receipt, sample custody, sample identification, and tracking sample procedures.

Samples are submitted to EMS by shippers (US Mail, FedEx, UPS, etc.), couriers, and by the customers themselves.

Because of the nature of the work at EMS, chain of custody (COC) procedures are followed. EMS recommends that all samples are delivered to the laboratory with a customer's COC or EMS provides its Submittal Form, EMS' COC. The COC provides the information with regard to the change in possession from sampling, delivery to receipt at the laboratory. Upon receipt of the samples at EMS, the chain-of custody form is signed, dated and time of receipt noted to establish the change in custody of the samples. In the laboratory, samples are considered secure for evidentiary purposes if the samples are in ones possession, within view or in a secured area.

On receipt, condition of the samples is noted. The customer is notified immediately if any problems are found with the condition of the package, chain of custody/seals, or samples at receipt. A laboratory identification number is assigned and logged into a bound, page numbered, "Logbook" with sample receipt conditions noted. Sample information is entered in a database system, which generates a Sample Tracking Form for the samples. Copies are made of customers COC and any accompanying paperwork from the customer. The originals of shipping bills, COCs and customer paperwork are filed in the Custody file by laboratory number. Copies of the paperwork are submitted with the samples to the appropriate department where the analyst signs the COC. Samples are then analyzed or stored at room temperature, refrigerator temperature ($4^{o}C$) or frozen. A log is kept in each department of the laboratory number, customer, date received, number of samples. After analysis and QC, the data package is assembled by the staff member who performed the work and is submitted for reporting and invoicing. The final report and data package are reviewed by a Director and submitted to the customer. The samples are stored as required.

Inspection of Incoming Samples

Upon receipt of samples at EMS by a shipper or courier, method of shipment and the condition of the shipping package are noted.

Bills of lading and shipping slips are retained.

If there is damage to the package, the customer and shipper are immediately notified before the shipment is unpacked, and we follow instructions from the customer.

After the package is opened and if EMS' Submittal Form has been used, delivery time and shipper are noted on the form. If the customer has used his own COC and not provided the Submittal Form, one will be filled out. The integrity of the package is also noted on the Submittal Form.

The ACM or unknown packages are opened in the Sample Receiving area. The contents are checked for breakage, leakage and integrity of the chain-of-custody seals, if present. Conditions are noted on the Submittal Form. The receiving area is monitored for asbestos fiber release.

If samples are submitted for asbestos analysis, the individual containers are damp wiped.

If these items are in order, the contents are checked against accompanying customer packing slips, chain-of-custody records.

Sample collection date, time and sampler's name are checked to see if they are present on the chain-of-custody. If they are not present, the customer is contacted for this information.

Preservatives, if used, must be noted on the chain-of-custody by the customer. Samples (i.e., water samples for asbestos analysis) are delivered immediately upon opening to the department to have the temperature measured which is recorded on the Submittal Form. Sample preservation, container types and holding times are posted in the sample receiving area.

Sample logging

Each set of samples is given a sequential laboratory number, in chronological order. The numbers are generated on a computer. Four labels with each number are printed. One label number is affixed to a log, which identifies the customer and date of sample submittal. The other 3 labels are attached to the 3 copies of the COC (Submittal Form).

Samples are logged by the sample log-in clerk into a bound, page numbered, "Logbook" after all the paper work is completed.

Every sample in the package is assigned a unique laboratory number, which is prefixed to part of the customer's sample identification number. For instance, if the next laboratory number is 30000, and a set of samples is submitted with the identification numbers 910034-1, 910034-2, the numbers assigned to the individual samples would be 30000-1 for Sample 910034-1, 30000-2 for Sample 910034-2, etc., or whatever suffix from the customer's identification number that would give the samples a unique identifier. If no unique suffix number is given by the customer or if only a description and no number is given, EMS will assign a number 1 through n (with n being the last sample in the set) for the samples.

If a sample is received where more than one analysis is performed in different departments, laboratory numbers are assigned for each department. Duplicate paper work is made out for the aliquot. Multiple water bottles of the same sample are indicated as Sample 1, Sample 2.or Dep. etc/. The sample that is analyzed (1, 2, dup or 3, etc.) is so designated on the data sheets and report.

If samples are submitted on one COC for various analyses, copies are made of all paper work for each set of samples and submitted to the appropriate department with the samples and each COC is signed by a staff member in that department. Invoices and reports are generated from each department.

Sample Tracking Forms are computer generated for each set of samples and are attached to the Submittal Form and the rest of the Sample Receiving paper package.

If the customer at a later date requests additional analysis, a new laboratory number is assigned with reference on the COC to the original number.

If there are more than 20 samples for any metal analyte, the samples for the metal analyte are broken down into batches of 20 or less samples, and each batch is given a separate laboratory number.

For EPA samples, Form DC1 is completed. (See Appendix for EPA submittals.)

Upon receipt of the samples, all paper work, i.e., chain-of custody is signed and dated on all forms that accompany samples. Copies are made of all incoming paper work. Two copies are for EMS' use.

The signed and dated chain-of-custody forms are returned to the customer.

One copy of all the paper work is attached to a EMS' copy of the Submittal Form along with a Sample Tracking Form and accompanies the samples until the analyses are finished at which time the paper work is returned to the secretary for the report and final filing with the report.

If samples are shipped to another laboratory, another chain-of-custody form is used which tracks sample from EMS Laboratories to the custody at the other laboratory. See Chain-of-Custody Form in Appendix 1.

One copy of all forms, receipts, shipping bills and other paper work is filed in a separate chain-of custody file, filed by laboratory number.

The following information is recorded in the bound logbook:

Entered into Bound Logbook and Submittal Form:

Laboratory Number
Date of package delivery
Customer's name
PO number (if provided)
Number of samples
Analyses required
Customer's sample number
Unique identifying number for each sample
Condition of sample, container or packaging

Also Entered onto Submittal Form or on Customer's COC:

Type of matrix
Sample preservatives and temperature, holding time (if applicable)
Date/time of sample collection and sampler's name
Condition of container, packaging, custody seals (if applicable)
Turnaround time
Contact person
Customer's comments and/or special instruction
Carrier's name
Shipping bill retained (if applicable)
Signature of sample receiver
Signature of staff member receiving samples
Disposition of samples after analysis

For other information that needs to be documented with EPA samples, see the appendix.

If samples arrive during the weekend, and weekend analysis is not required, the samples will be kept in safe storage until Monday morning. The complete logging procedure will be performed by the regular sample receiving staff.

If samples are dropped off by a customer during the weekend and weekend analysis is not requested but the customer needs a copy of the sample submittal form, weekend personnel will assign a sequential laboratory number to the submittal form, sign as the recipient, but may not necessarily enter the sample information into the bound receiving log book. The regular receiving staff will enter the information on Monday morning.

If samples are submitted on the weekend for weekend turnaround, the analyst will assign the sequential laboratory number but will not necessarily enter the information into the bound receiving logbook if time constraints do not permit it. The regular receiving staff will enter the information into the bound logbook.

Resolving Sample Receiving Problems

If problems are found during receiving such as samples with identical identification numbers, missing samples, identifications that do not match the samples, a Director or Manager is informed. The **customer** is immediately notified by phone or Fax. The problem is documented with the resolution by the **customer** on the COC Form.

Refusing Samples at Sample Receiving-Conditions

- Incompatible samples shipped in a single package (i.e., bulk and air for asbestos analysis)
- Samples collected on the wrong media

- Air cassettes or sample containers are broken open
- Sample has leaked out of the container
- Sample integrity appears compromised
- Chain-of-custody seals are broken

If there is a sampling error or other procedural error, the **customer** is contacted and told the samples have to be rejected. If the **customer** wishes to proceed with the analysis, the irregularity is documented in the report and on the chain-of-custody.

Terms Used to Describe Sample/Package Condition

The use of the terms "good "or "OK" means that the sample/samples were received in good condition, no broken or leaking bottles, air cassettes sealed. "Intact" means that the package shows no signs of damage or tampering. "Intact" and "Not Intact" also refers to chain-of-custody seals that have or have not been torn or removed.

Authorized Signatures

In special cases, customers are asked to provide a list of people authorized to submit samples with their signatures.

Transfer to and Custody of Samples in the Laboratory

The samples are transferred by sample receiving to the respective department along with copies of the chain-of-custody, Sample Tracking, Submittal forms and any other sample identification/descriptions provided by the customer. A staff member logs in the samples in that department. The Tracking Form is signed and filled out by the person performing the preparation/analysis. If samples are not analyzed immediately, they are stored under suitable conditions, such as a certain pH, temperature, etc.

The paperwork accompanying the samples consists of:
- Chain-of-Custody papers
- Sample Tracking Form
- Any other paperwork which was submitted with the samples

For true chain-of-custody samples that arrive with COC seals, the receiving person will deliver the samples to the appropriate department and store the samples in a secured storage area or refrigerator and deliver the paperwork to the analyst. If there are chain-of-custody seals on the separate sample containers, the analyst may break the seal for inspection of the sample if considered necessary. Otherwise the seal is not broken until the analysis is started. When the analyst is ready to begin, he will sign the Sample Tracking Form for the samples; and when the work is completed will return the samples to the person in charge of archiving and final disposal. See Sample Disposition Form in Appendix 1.

Sample custody is the responsibility of the staff member performing the analysis. The samples must be
- In their possession, or
- In their view after being in their possession, or
- The sample is in a designated secure storage area with access by authorized personnel

Rejection of Samples in the Laboratory

Samples are rejected upon opening in the departments if any of the following items occur:

- Collection media is inappropriate for the method.
- Volume collected is not appropriate for the analysis.
- Samples are overloaded.
- Samples were received where holding time has expired.
- Sample had leaked out of the container. *
- Samples were not shipped properly to the laboratory* i.e., proper temperature

*These items may have been addressed in Sample Receiving before they reach the department.

Logbook Review

The QC manager or a designee reviews receiving logbook and computer data entries by the following day. All other logbooks are reviewed weekly by the department managers.

Shipment Using Chain-of-Custody Seals

Shipping packages, where chain-of-custody seals are required, COC seals with all the appropriate information are filled out. Chain-of-custody with items/samples sent, sample number, where sent, date of submittal/delivery, and method of delivery is completed.

SAMPLE STORAGE

Sample storage is separate for each department: Chemistry, transmission electron microscopy, optical microscopy/x-ray diffraction.

In chemistry, the samples are stored as required for a particular analysis. Samples are stored at room temperature, in the freezer or refrigerator, as required.

Samples to be analyzed for volatile organics are stored in a separate refrigerator. Refrigerators and freezers, which are used for sample storage, are exclusively for storage. Standards are stored separately to avoid contamination.

In the optical microscopy/x-ray diffraction laboratory the samples are accessible to the optical personnel. When the analysis is completed, the samples are catalogued by laboratory number, and put into storage where they can be easily retrieved.

Air samples for the TEM laboratory are stored in the TEM sample preparation area. Prepared samples that are ready for analysis are stored with prepared paper work in the TEM area. After analysis, the grids are filed under the laboratory number, placed in boxes and stored above the TEM area that is accessible only to authorized personnel. Unused portions of air filters are stored in the original cassettes, unused portions of filters from water, microvac and bulk sample preparations are filed according to laboratory number and stored in the same area as the grid boxes.

Bulk samples for TEM asbestos analysis are stored with other bulk samples in the PLM area.

Samples are returned to the **customer**s upon their request. It will be documented with EMS' chain-of-custody.

SECURITY MEASURES FOR SAMPLE STORAGE

EMS Laboratories, Inc., is equipped with the DSC security system. Only authorized personnel have access to keys and security code information.

The computer systems used to track samples, create reports and store data are password protected.

Visitors, including delivery person, must sign-in to enter beyond the sample receiving area.

Samples are stored in the designated area of each department, and access to the storage areas is limited to authorized personnel. Authorized personnel are the Directors, Managers and designated analysts of each respective department.

When additional security measures are necessary by contractual agreement, appropriate measures are implemented and documented.

TRACKING SAMPLE ANALYSIS

The following documents are used in tracking sample analysis:

- Sample receipt - Submittal Form
- Sample Transfers - Chain-of-custody and Tracking Form
  - In-house transfer - See Submittal and/or Tracking Form
  - Transfer to another facility - See Laboratory Transmittal Form
  - Archiving -See section "Sample Storage " and accompanying forms
- Sample preparations - See individual SOP for each department
- Sample analysis - See individual SOP for each department
- QC/QA laboratory analysis -See individual SOP for each department
- Sample disposal - See section on "Hazardous Waste Management, Storage and Disposal Policy"

Samples are archived, after completion of analysis and before disposal, in designated storage areas for each of the departments. (See details in section "Sample Storage".) If samples need to be retrieved for quality control analysis or for legal purposes, a Director or Manager will direct a staff member to remove the sample from storage. After QC analysis, the sample will be returned to storage. If the sample is to ~~to~~ leave the laboratory, the Laboratory Transmittal Form will be filled out and noted so on the chain-of-custody form that accompanied the sample.

PURCHASING SERVICES AND SUPPLIES

Procedures for selection and purchasing of services and supplies that affect the quality of the test are described in SOP 1008: EMS standard operation procedure for purchasing.

Procedures for reception and storage of reagents and laboratory consumable materials relevant for the tests are in SOP 1007: EMS standard operation procedure for reception and storage.

Purchase Requests

Staff members order replacement of chemicals, glassware, small hardware, filters and other laboratory and office supplies. Repair and/or replacement of apparatus are reported to a supervisor. The Technical Director approves all orders.

Purchase Orders

All purchases are made by purchase order number, a majority verbally. The purchase order contains the date of order, supplier and the items purchased. The purchase order has the name of the recipient of the order. Any chemicals or consumables requiring certification will be obtained according to the specification for the material. The purchase order must reflect these requirements which is the usually the catalog number of the chemical, or item that is traceable through the catalog. Chemicals must be procured with reference to their standards. Where applicable, NIST traceability documents must be provided by the manufacturer.

Purchase of outside services shall be made with a written purchase order.

Qualified Vendors

To insure quality in the laboratory, all services and supplies are purchased from reputable sources with a historical record of providing service to EMS in a timely manner and at reasonable prices. QA shall maintain a list of qualified vendors.

Receiving of Chemicals and Supplies

Incoming materials are checked at receiving to assure that the material received corresponds with that ordered and the necessary labeling and certifications are included in all shipments. They are then sent to the appropriate department where they are inspected for content and conditions. Items received in damaged condition are reported to shippers by telephone (followed by written confirmation, if needed). Packing slips, signed and dated by the department receiving the items, are submitted to the business office for further processing with the invoice.

Upon receipt of the materials, the lot number from the manufacturer is recorded and the purity of the lot is established through a method blank and/or a calibration check, including filters for use in the optical and electron microscope laboratories. Records are kept in the respective departments.

REAGENTS AND STANDARDS

Only certified standards traceable to NIST are purchased and used. Reagents and standards shall be inspected, dated and initialed upon receipt. Calibration standards and analytical reagents shall have an expiration date assigned. Reagents and standards shall not be used beyond assigned expiration dates. Materials designated for reevaluation, which are determined to have adequate purity upon reevaluation, shall be assigned a new expiration date.

Documentation of calibration standards preparation **must** be strictly controlled. The documentation shall include a description of the content, the date of preparation, concentration and/or purity of parent material, manufacturer and lot number of parent material, assigned expiration date and the preparer's initials. Solution shall be adequately identified to trace back to preparation documentation.

A logbook is kept for the preparation of reagents solution. All reagents purchased from outside sources will be dated upon receipt and recorded in the logbook. The documentation for reagent preparation includes: date of preparation, a description of the preparation procedure, concentration, expiration date, initials of the person responsible.

For organics, standards are prepared daily with each batch of sample. The organic standard preparation is documented in chemist notebook, together with the sample analysis record.

ANALYTICAL METHOD

Refer to Standard Operation Procedure for each department for detailed analytical methods.

Standard procedures as specified by the EPA, NIOSH, OSHA, and DHS are followed. All methods are available in each department to the analysts.

All new methods adopted or revisions to an existed method are written in a standard format by the Methods Development analyst-QA/QC Manager and dated and approved for use by Technical Director. All the SOPs are reviewed by each of the analysts whenever they are revised.

Any modification of a method, such as required by the presence of an interfering substance, will be approved by a Manager or Directors. Methods are not used for sample analysis until competence has been demonstrated by EMS.

Where the standard method is modified for a particular non-routine sample, the modification and reasons for it are noted in the report to the customer.

Methods for media preparations are per standard published methods or methods developed and validated by EMS.

Methods of sample preservation will follow the guidelines of the standard methods whenever available. When the concentration of the analysis is expected to be in the low microgram range, the analysis will proceed as soon as possible after the samples are received.

Upon a request for a new method of analysis, the Technical Director or Laboratory Director will ensure that EMS has the proper method of analysis, instrumentation, standards, and quality control measures for the new method before commencing analysis of customer's sample.

EQUIPMENT CALIBRATION AND MAINTENANCE

See each department's Standard Operation Procedure for equipment calibration and maintenance.

Maintenance logs are kept for each instrument with dates of maintenance and person performing the maintenance. This will enable reconstruction of the run sequence of individual instruments.

DATA REDUCTION, VALIDATION, AND REPORTING

See each department's Standard Operation Procedure for details on data reduction and calculation for each specific method.

The laboratory documents that are directly related to all activities involved in the sample preparation and analysis are identified by EMS Laboratory Numbers. These documents are either in bound notebooks or preprinted forms with EMS' name and address. Bound notebooks are numbered sequentially. Logbook pages are sequentially numbered, ascending order front to back. Notebooks and logbooks are kept in chronological order.

In general, data is entered on NCR multiple copy forms, into notebooks or into the computer. All data sheets, notebook entries are signed and dated by the staff member performing the sample preparation and/or sample analysis. Unused sections of pages are lined-out.

Corrections are made on the original entries by neatly crossing out the original data, initialing, dating, and entering the new data in ink. New, corrected data is generated with a report to the customer if the mistake was not found before the report was sent. These corrected copies are kept in a file with a notation across that data that it has been corrected.

After all required analyses of a sample are completed, the documents are assembled and put in the data package file for that customer. The report/report cover letter and invoice and submitted to a director or manager for final QC and review.

(For sample data package file (CSF) - project specific for U.S. EPA see the Appendix.)

VERIFICATION OF REPORT

The data package file for the customer will contain:
A.Copy of final test report with sample analysis
B. Copy of invoice

C. Copy of the chain-of -custody
D. Original sample tracking records
E. Raw data and calculations (copies)
F. Computer printouts and chromatograms, strip charts, or other related instrument output (originals)
G. Results of QA/QC samples
H. Copies or original, as appropriate, of any correspondence and other written documents

Final test reports include the following information:

- ✓ Name, address and telephone of EMS
- ✓ Page number and total page numbers on each page
- ✓
- ✓ Unique identification number of the test report (Report No.)
- ✓ Name and address of the customer
- ✓ Description and identification of test sample/s, date collected by customer, and date received at EMS
- ✓ Identification of the test method, sample results, and reporting limit
- ✓ Modification to the test method if any
- ✓ Name, title and signature of the staff member accepting technical responsibility for the content of the original or corrected test report. This person at EMS is a Director or a designee
- ✓ Date of report issued and date of sample analyzed
- ✓ Report will indicate whether or not results were corrected for blank data. Report will include a note that calculations of the air results are based on the air volumes provided by the customer

Original pages of the report, cover letter (if used), invoice, and copies of the chain-of custody (EMS and customer's COC if provided) are sent to the customer. For TEM analysis, the original raw data sheets and summaries are provided. Copies are retained in the customer file under that report.

Checking of sample custody records is performed first in Sample Receiving and then at receiving in the appropriate department. Checking of laboratory bench sheets, personal and instrument run logs, sample tracking records, charts, strip charts, computer printouts, raw data summaries are the responsibility of the senior staff member assigned in each department.

After assembly of the above documents the project manager or director check the file for consistency and completeness of data. If everything is acceptable, the deliverable package is shipped using, U. S. Mail, Federal Express, or other courier service. Carrier is identified on Sample Tracking.

DISPOSAL POLICY FOR REPORTS

All reports and supporting documentation are destroyed starting three months before the completion of the seventh year unless there is a legal requirement for longer storage (lead reports require 10-year storage). A paper recycling company is called to shred all the documents.

When instructed otherwise, EMS will store documents for a longer period or destroy documents within the time specified by the customer or regulatory agency if a shorter period is consistent with legal requirements.

Chemistry reports and receiving logbooks are kept 10 years.

HAZARDOUS WASTE MANAGEMENT

Chemical Waste

All chemistry samples are kept for 90-120 days from the date of analysis or as required by the customer before they are treated as waste. For other samples see SOPs for each department. Samples are returned to the customers upon their request, or if they require special storage. It is documented with EMS' chain-of-custody. The results of the analysis are used as a guide to determine whether the sample **shall** be considered normal or hazardous waste. Samples are disposed with other compatible laboratory waste.

EMS generates different residual wastes from sample analysis. EMS is a small generator of waste. The waste comes from analysis of samples and the chemical reagents used. Samples are returned to the customer if a large amount is involved. If the samples are kept, then the following procedures are followed:

1. All samples and chemicals are sealed in individual containers compatible with their contents.

2. All containers are labeled "Hazardous Waste", with specific contents name, concentration, matrix, starting date of accumulation and the initial of the person in charge of waste handling in that department. The containers are labeled "Danger" or "Caution" if necessary.

3. All containers are stored in designated areas, according to their chemical toxicity. Asbestos bulk waste is stored in upstairs storage. In chemistry, solvent wastes are kept under a hood. Other chemical wastes such as mercury, arsenic, chromate, etc., are placed in different marked containers.

4. Incompatible wastes are never mixed and containers are carefully labeled. The hazard concentration is marked on the sample container after completion of the analysis in order to determine disposal requirements.

5. Each department head is in charge of the wastes to insure that the labeling and storage are performed properly. Every laboratory sink has pH paper and a labeled bottle of sodium bicarbonate for neutralizing small amounts of acid generated during sample preparation. Chemical storage area must be equipped with a fire extinguisher, spill kit, first aid kit.

6. Waste inventory is conducted every six months. A treatment storage disposal facility will be used before the 90-day limit is exceeded. (In California, the 90-day limit applies when 100 kg of waste has accumulated. One can keep the waste on site for 90 days after the day the 100 kg of waste has been accumulated at which time a permitted TSD facility is called to handle the waste.)

Asbestos Waste

The sample preparation technician and TEM laboratory manager are responsible for storage and disposal of waste in the TEM area. They coordinate with the Optical Manager for final waste disposal by an asbestos waste disposal firm.

Filters are stored in original cassettes in labeled plastic bags and stored in boxes with the name lab number and discarded as hazardous waste after 30 days (CFR Part 763, AHERA, 41868, Item II). 7/31/08.

Filters from water, bulk and vacuum samples are stored in petri dishes with laboratory and sample numbers on the petri dish covers. They are discarded after one year as asbestos hazardous waste.

Stock solutions from bulk asbestos sample preparations are stored in the sample-preparation area until the analyses and quality control-quality assurance procedures are completed. The stock solutions are stored in 5-gallon plastic containers and discarded by an asbestos hazardous-waste disposal company.

Grid boxes containing the prepared asbestos samples are filed under a systematic order and stored for 1 year and then the grids are discarded as asbestos hazardous-waste. (CFR Part 763, AHERA, 41868, Item II.)

Bulk asbestos samples are stored in the PLM area.

The optical laboratory supervisor is responsible for storage and disposal of samples.

Samples, after analysis, are placed in sealed plastic bags, labeled with the customer's name, lab number and stored in boxes. A packing list is sealed inside the box and a copy attached to the outside.

Bulk samples are kept in storage for 90 days unless a longer period of storage is contractually necessary. Samples that must be kept for longer periods of time are stored separately with the customer's name. Upon customer's request, the samples are returned by UPS or are picked up by the customer. Return of samples is documented. Otherwise the samples are double bagged in asbestos disposal bags and sent to a hazardous waste dumpsite.

CUSTOMER FEEDBACK

EMS sends with reports a customer satisfaction survey, which is used to analyze the management system, to improve our analytical activities and customer service.

The survey is included in all reports for one week, every 3 months. A review of reports with customers will also be included.

Management will review the survey with staff at monthly meetings and in the annual management review.

CUSTOMER'S

If a customer questions the validity of the test result of a specific sample or the results of a batch of samples, the laboratory director, manager and the quality control supervisor immediately address any question regarding the quality of the data, including turnaround time. The following standard procedure is followed by the Laboratory Manager/Quality Control Manager immediately upon receipt of the complaint:

- Calculations are re-checked
- Calibration data are re-checked
- Other QA/QC data are re-checked

If no source of error was detected, the sample(s) is reanalyzed within one working day of the complaint, if the sample is still available, or the customer is advised to submit new samples which will be run immediately without charge. If the result of reanalysis agrees with the previous result(s), the customer will be notified by the QA Analyst. If the result of the reanalysis of sample does not agree with the initial value, the reason will be investigated. The reanalysis will be completely documented in the corrective action file.

The final result of the investigation along with a revised final report will be submitted to the customer by the QA Analyst within three working days of the complaint. The corrective action must in all cases include a written response to the customer. It will be the responsibility of the QA Analyst to provide the written response after submitting the package to a laboratory director for approval.

The problem will be completely documented in the file for that report. A copy goes to the customer's complaints file. Also, if necessary, additional QA checks will be instituted so that the problem will not be repeated in the future.

The laboratory director or quality control manager addresses customers' requests for additional information regarding preparation methods, analytical methods, and/or presentation of the results.

Customers' requests for further analysis, running duplicate/replicate and/or sample re-preparation is approved and accommodated by the laboratory personnel upon instructions from the laboratory director or quality control manager. Documentation is sent expeditiously to the customer.

If the customer is still unsatisfied we will recommend an equivalent laboratory for further Q.C. or analysis.

The data entry personnel correct typing mistakes and the corrections are checked by the laboratory director or QC manager.

INTERNAL COMPLAINTS

Internal complaints are communicated by personnel to the manager of their departments. If the problem has not been resolved by the Manager, personnel are directed to the Laboratory or Technical Director.

Problems of safety are directed to the Chemical Hygiene Officer who either corrects it or notifies a department manager or director. EMS has a policy that it will comply with Federal, State and local regulations regarding employee health and safety. EMS' Chemical Hygiene and Safety Plan is given to every employee. All safety and health issues are periodically discussed at EMS' monthly meetings.

All complaints must be documented and are kept in the Safety Office file or the Laboratory Director's office and reviewed periodically.

CONTROL OF NONCONFORMING TESTING WORK

If the level of acceptance set by the methodology is not met (non-conforming work) departures from the policies and procedures in the management system or technical operations are identified, corrective action must be taken immediately. The following steps are taken to maintain the integrity of the data:

Identification of the problem
Assignment of responsibility for investigating the problem (manager)
Investigation and determination of the cause of the problem

- discussion with the analyst, QA Analyst, laboratory or technical director

Determination of a corrective action to eliminate the problem
Assigning and accepting responsibility; for taking corrective action
Implementing the correction action and evaluating its effectiveness
Verifying that the corrective action has eliminated the problem

The corrective action notification form is completed by the analyst, department manager or QA Analyst. QA Analyst will file a copy for review with management.

A summary report is filled out describing the initial indication of the problem, the cause that was discovered, and remedial actions taken. It is placed in the personnel file of the person or persons who are found to be negligent. The summary reports will be used in the employees next performance review.

Correction of Potential Non-Conformities and Identification of Opportunities for Improvement

The laboratory shall identify ways to improve technically and to improve the laboratory management system.

Potential sources of non-conformities may come from nonconforming procedures, nonconforming incoming supplies, mathematical error, quality control check samples that deviate from expected concentration, instrument calibration, instrument maintenance.

When any source of nonconformity is identified by internal or external audit, by analysts' audit performance, by customer complaints, remedial actions shall be developed, implemented and monitored to reduce the chance of the reoccurrence and to make future improvements.

A list of the identified non-conformities will be developed and checked periodically by department managers (overseen by the QA Analyst), to identify improvement opportunities.

CORRECTIVE ACTION

Upon identification through an internal audit, recalculations, QA/QC procedures or identifying a breach of policies or procedures of one of its analysis, written notification regarding poor data or recalls breach of policies will be sent to the customer or customers who received the data whether they have been verbally notified or not. Immediate corrective action will take place.

Corrective action starts with an investigation conducted by a director or QA Analyst to determine the root causes of the problem. If the root cause is not obvious, a careful analysis of all potential causes of the problem is required. Potential causes could include customer requirement, the samples, sample specifications, methods and procedures, staff skills and training, consumables, or equipment and its calibration.

Once the root cause is identified, reanalysis, rerunning new samples and issuing new reports will take priority. The written notification and the corrective action will be documented and kept in a separate file. If the identification of the root cause casts doubts on the laboratory's compliance with ISO/IEC 17025, an internal audit must be conducted as soon as possible to ensure that the corrective actions taken have been effective.

Every department of the laboratory must fill out a corrective action form when an analytical technique repeatedly exceeds the control limit, repeated errors in an analyst's calculations are found or other persistent problems with the data or analysis are discovered. See Appendix 1 – EMS Documents.

Interpretation and Utilization of Control Limits

If one measurement exceeds control limits, repeat the analysis immediately; if the reanalysis is within the control limits, continue; if the reanalysis exceeds the control limit, discontinue analyses and correct problem as described below.

If two out of three successive points exceed warning limits, analyze another sample. If the next point is less than warning limits, continue analyses; otherwise, discontinue analyses and correct problem as described below.

If measurements never or rarely exceed warning limits, at the next system audit recalculate warning limits and control limits with the most recent data points.

Situations that exceed the control limits and their resolution are documented.

Correcting the Problem

If the control limits indicate that a problem exists which must be corrected, the analyst will cease any analysis on that instrument and record the problem in the calibration log. The analyst will report the problem to the department manager and/or the quality control director. Data are not reported until problems requiring corrective action are resolved.

The analyst will be responsible for a complete check of the reagents and standards used for that analysis and determine whether the problem can be attributed to the reagents or standards. If a problem exists with either reagents or standards the analyst will determine the origin of the deficiency. The following actions will be taken:

1. If the problem is due to purely random factors such as accidental contamination, the standards or reagents will be discarded and replaced.
2. If the problem is found to be systematic, such as the grade or supplier of the reagent used, or a cleaning procedure, the analyst will request a change in the appropriate SOP to correct this cause.
3. When reagents and standards have been eliminated as the cause, the replaceable components of the instrument will be checked. If such a component, e.g. an AA lamp, are found to be the cause then the faulty component will be replaced.
4. If none of the above appears to be the problem, then an instrument will be serviced by a qualified person.

Upon correction of the problem, the analyst will run standards and QC samples to prove that the analytical method is giving the correct results. The results of the analysis of the standards and QC samples will be given to the chemistry manager who will authorize that analytical procedure to be resumed and who will document the authorization in the appropriate instrument or calibration log.

Root Cause Investigation

All corrective actions and infractions, which are usually less serious problems, are collected in files and examined for the "root" of the problems.

On-going problems are examined for the analyst, (i.e., calculation, dilution errors, methods of analysis, not responding in the correct turnaround time, etc.), clerical errors (no pagination, not following instructions on the chain-of-custody, billings and reports to wrong addresses, etc.), instrument problems that may be on-going on an intermittent basis to determine source and correction.

This paper work is examined collectively over weeks and months to ascertain any pattern by the QA Analyst and Directors. This information is used to examine analyst/s and clerical deficiencies, instruments, outside purchases of chemicals and supplies so that the root of the problem can be identified and resolved.

New vendors, updated instrumentation, sources of supplies, laboratory SOPs and retraining of employees are then addressed.

PREVENTIVE ACTIONS

Analysts will promptly report any malfunction, high noise levels or other changes in the performance of any of the instruments to the department manager who will contact the instrument repair services if it is required.

Each instrument has a manufacturer's operating manual for standard operating conditions and use. Any equipment that is out of calibration or is defective is taken out of service until repaired.

Records are maintained for all preventive maintenance and repairs. The instrument maintenance book describes the problem, service, dates, and person performing repair.

In the circumstances of corrective action, the quality control person immediately conducts an internal audit and the results are presented to the director and the department manager. Corrective measures are put in place to prevent further problems from arising.

The department manager will be notified immediately by the analyst when reagents, solvents, standards or other supplies are low.

See "Correction of Potential Non-Conformities…" section for descriptions.

SYSTEM AND PERFORMANCE AUDITS

1. QA/QC manager will annually review the quality control system to ensure that the requirements of the manual are being met. The annual review include following:

    a. Review of the daily calibration log for each instrument to ensure that it is being properly kept and to determine any trends or inconsistencies in the calibration data.
    b. Review of the control charts for the various laboratory procedures to see if the control limits **shall** be revised and if any trends must be addressed.
    c. Review of the instrument SOPs to insure that they are current.
    d. Review records for quality control samples, such as spike, duplicates, and blind samples for each instrument and method

    An audit checklist containing the required items for the entities accrediting the laboratory will be used for the laboratory audit and must be completely filled out at each annual audit.

    The checklist will be given to the laboratory director and a copy placed in the Internal Audit file. The laboratory director must review the audit checklist within two weeks of its completion. If the audit reveals that the requirements of an accrediting group were not properly addressed, appropriate changes in the organizational structure, instrumentation, or the quality procedure will be immediately instituted.

2. The annual audit may reveal deficiencies that affect a customer's reported results. If such a deficiency is identified, the laboratory director must

    a. Immediately determine whether the deficiency can be corrected without affecting the results reported to the customer. If the deficiency can be corrected then the appropriate action must be taken and the problem and solution documented in the file for that report.
    b. If the deficiency resulted in an erroneous value being reported to the customer and if the sample is still in the laboratory archives, the sample must be reanalyzed and a corrected report will be sent to the customer.
    c. If the sample which was incorrectly analyzed is no longer available then the customer must be advised that the reported value(s) might be erroneous and given the option of submitting another sample(s) or given a refund.
    d. The cause of the error must be carefully reviewed and, if possible, additional safeguards must be added to the system to prevent reoccurrence.

3. Review proficiency test results, which include all categories of the AIHA PAT program, ELPAT lead program, NVLAP bulk and airborne asbestos programs, and the EPA drinking water, wastewater, and hazardous waste. Any discrepancies revealed by these proficiency tests must be immediately addressed by the quality manager and the corrective action described in a memorandum.

4. The laboratory will maintain or pursue its accreditation status with the appropriate accrediting groups. These presently include AIHA, California ELAP and NVLAP. This will ensure that an outside audit of the quality system will occur no less than every two years and in most cases annually.

CONTROL OF RECORDS

The final hard copy reports include the invoice, the cover page (where applicable), typed results, copies of the raw data, quality records, sample tracking form, EMS' chain-of-custody form (Submittal Form), copies of all chain-of-custodies and other paper submissions from the customer. See "Assembly of Completed Data Package."

The final reports are filed alphabetically according to the customer's name and kept for at least two months to six months in steel file cabinets. When a customer's file becomes too large, those reports that are older than 2 months

are placed in file boxes and stored on shelves near sample receiving. These files are accessible to the staff. Two months after the beginning of the new year, all the reports are placed in file boxes alphabetically and stored on the second floor of the laboratory. The environment is such that there is no deterioration of the records. Access is by ladder. Only assigned staff members have access to these files.

All the quality records are stored chronically. The training records of the employee, together with all the proficiency testing result, are kept in steel file cabinets. The internal audits and management review, corrective action, together with all the quality control data from each department, are kept on the small bookshelf next to the chemistry department. These files are accessible to the analysts.

Logbooks are kept in a fireproof file.

Files are stored according to year.

See "Disposal Policy for Reports" for retention, etc.

The custodian of records maintains and has archived records retrieved when necessary.

Records are transferred only after the customer who submitted the samples to EMS Laboratories gives verbal or written instructions. Any third party that needs to access the records must have written consent from EMS' customer.

Personnel Policy 017, "Confidentiality," (also found in this Quality Manual), is observed in the release of the information.

All information regarding reports that need to be faxed are accompanied by the "EMS Laboratories, Inc., Confidential FAX Memo" cover.

ETHICAL AND LEGAL RESPONSIBILITIES

Personnel are educated in their ethical and legal responsibilities, which include the potential punishments and penalties for improper, unethical or illegal action. The procedures are reviewed whenever there is revision. The topics are covered in EMS' Quality Manual and EMS' personnel policies which are given to every member of the staff and include Safety in the Workplace - Work Assignments, Procedural Changes and Corrections, Confidentiality, and in the Quality Manual : Internal and external Pressures on EMS' Personnel and procedures including Health & Safety

UNDUE PRESSURES ON EMS' PERSONNEL

Under no circumstances are any of the personnel to be pressured regarding the quality or the context of any analysis presented to the customer. Management will not be influenced by the customer or financial considerations in subjecting any of its personnel to outside pressures regarding any analytical results.

Under no circumstances are the personnel of EMS to succumb to any external or internal pressures of any manager or supervisor regarding the quality or the context of any analysis presented to the customer.

Under no circumstances are the personnel of EMS to yield to any external or internal, commercial, financial or other undue pressures regarding the quality or the context of any analysis presented to the customer.

If instances of undue pressure are encountered from outside sources, they must be reported immediately to a Director who will deal with the customer directly.

If instances of undue pressure are encountered from inside sources, they must be reported immediately to a Director who will deal with the internal matter/staff directly.

Disregard of this policy, depending upon the severity, may result in termination of the person/s involved.

If a situation occurs where an employee alters the quality or context of an analysis, whether there is any profit directly or indirectly to them, termination will result.

See "Conflict of Interest", Policy and Document in Employee Handbook dated 1/08.

CONTROL AND MAINTENANCE OF DOCUMENTATION

All SOP and QA/QC manuals are reviewed annually or when it is necessary to change any part of these documents.

When changes are needed, they must be reviewed and approved by the laboratory director followed by revising the appropriate document. Initial draft may be made by an analyst, department manager, QA Analyst or a director, depending on the nature of the changes. The final revised document must by approved by a director. Every document has a revision number, approval signature and is dated when the director has approved it.

The altered new text shall be identified in the document by a strike-through the old text and bold/italic font for the new or altered text.

Hand written changes of controlled documents shall be made in the text by the appropriate person, i.e., department head, quality manager or director. These changes shall be initialed and dated. New revised documents shall be reissued as soon as possible if there are major changes. A copy of the document, with the text changes, shall be kept in the archived files. The review page found in every controlled document shall indicate any changes (in the comment column) of that particular document dated and signed by the originator.

The following procedure must be met when dealing with controlled documents:

The QA Analyst maintains a list of all controlled documents and distributes the revised document to the appropriate personnel or department. The QA/QC manager removes the old revision. When receiving ***or sending*** a new, revised document, the document receipt log shall include manual number, name and date of revision. All copies of the old versions are destroyed with the exception of one copy of the old version, which is archived in the file for historical purposes.

Documents that are controlled included: quality manual documents (Document ID number start with QM), reference material (document ID number start with R), Instrument manuals (document ID number start with M), computer software (document ID number start with S), and all EMS SOPs. All of these documents are listed in the document control list.

Every department, i.e., chemistry, transmission electron microscopy, industrial hygiene, optical microscopy, has within their area a current Quality Manual, and SOPs for the department.

Every analyst must review the most current documents annually. A log sheet is kept with the manuals. Each analyst will sign the log sheet after reviewing the documents.

REVIEW OF REQUESTS, TENDERS AND CONTRACTS

No new work can be accepted without the approval of a Manager or a Director. The review process will define the test method, evaluate the lab capability and resources, and negotiate a contract.

The review of the requests and tenders by a manager or a director will ensure that the methods to be used meet the needs of the customer and are appropriate for the tests it undertakes. Methods published in international, regional or national standards will preferably be used. When the customer does not specify the method to be used, EMS will select appropriate method that is capable of meeting the customers' requirements and inform the customer as to the method chosen. EMS will inform the customer when the method proposed by the customer is considered to be inappropriate or out of date.

The review of the request and tender will also ensure the EMS has the capability and resources to meet the requirement. Either the Manager or a Director reviews all projects before any new work can be received at EMS. If the work can be performed because one of the analysts' has a background in the analysis, but it has not been utilized at EMS and if the equipment is available or is such that EMS is interested in expanding into the field, a cost effectiveness and turnaround ability will be first assessed. Upon these decisions, either work will be accepted or referred to a laboratory with the capability.

A Manager or a Director routinely assesses capacity of the laboratory. Large scale jobs are analyzed for the capacity of the equipment and the availability of personnel or the ability to add personnel with adequate time for training and indoctrinating them into our QA/QC procedures.

If analyses cannot be performed because EMS does not have the capability or capacity, a form will be filled out indicating the reason for the rejection of the job. These cases will be reported to the Technical Director periodically for review.

The contract negotiation will be conducted in a practical and efficient manner, and the effect of financial, legal and time schedule aspects will be taken into account. Any difference between the request or tender and the contract will be resolved before any work commences. Each contract shall be acceptable both to the laboratory and the customer.

Records of reviews regarding the request, tender, and contract will be maintained. Pertinent discussions with a customer relating to the customer's requirements or the results of the work during the period of execution of the contract will be recorded on the sample chain of custody.

SUBCONTRACTING OF TESTS

If subcontracting is needed because of unforeseen reasons (e.g. workload, need further expertise or temporary incapacity), or on a continuing basis, the work shall be placed with a competent subcontractor. A competent subcontractor is one that complies with AIHA for industrial hygiene analysis, ELAP for environmental analysis, or NVLAP for asbestos analysis. Certificates from each subcontractor labs shall be maintained at EMS.

Customer shall be advised of the subcontracting arrangement and approval from the customer shall be recorded on the sample COC. The final report states that the work was done at the subcontracted laboratory.

QUALITY ASSURANCE REPORTS

On a quarterly basis, the QA Analyst provides a summary report to laboratory management regarding the quality assurance problems. All quality assurance problems are reviewed by the Technical Director within 3 working days of becoming aware of the problem.

The quarterly report shall include information on internal audits, proficiency program performance, nonconformances and corrective/preventive actions taken.

Internal audits must be conducted annually by the Quality Assurance Manager according to internal audit SOP 102. During the internal audit, control charts will be evaluated to monitor the validity of tests undertaken.

The audit can also be conducted using following checklist:

1. AIHA LAQP Application Review and Site Assessment Checklist
2. NVLAP specific operations checklist for TEM and PLM

The results of the audit is transmitted to the laboratory director for review and initiation of whatever corrective actions are required.

DOCUMENT REVIEW

The Quality Manual and all Standard Operation Procedures must be reviewed by personnel in the appropriate departments whenever there is a revision. These documents reflect the operations of EMS Laboratories.

Any discrepancies in, or changes to procedures, must be brought to the attention of the QC manager and the directors so that appropriate changes can be made to the documents.

There is also document control of these documents. These documents may not be copied or removed from the department. Every department will have a responsible person who will accept the document and who must return the document when a revised document is presented to them.

After reviewing the pertinent SOP, the staff must sign and date a log to indicate that they have reviewed it.

The one document that every staff member must read is the Quality Manual that covers the overall function of the laboratory.

Internal Quality Control

Quality control measurements verify the integrity of the analytical results. Quality control procedures vary from method to method. The analyst must have a thorough understanding of and is responsible for the quality control measurement as required by the method. All documentation is kept on file and securely stored. See "Disposal Policy for Reports' Section.

Instrument calibration is essential to the production of quality data. Strict calibration procedures are followed for each method, which are designed to determine and document the method detection limits, working range of that particular instrument and any fluctuations that may occur from day to day. See the SOPs for each department for details regarding calibration and maintenance of their analytical instrumentation.

In addition to instrument calibration, the quality control measurements of the analysis themselves is necessary. These involve measurement of blanks, accuracy and precision.

The number of QC samples to be analyzed with each batch of samples to meet the requirements of internal quality assurance, blank, spiked blank, and a duplicate spiked blank is determined as needed by contractual or accrediting body's requirements.

The principles of the internal quality control systems are given below. Some are general while some apply mainly to organic or inorganic analysis. See also the SOPs for the departments.

System Blank is run without a sample in the same manner as if a sample was present. It is used to verify that the background due to equipment contamination is below detection limit.

Method/Reagent Blank is a sample of reagent/water, which is processed exactly as if it was a sample. It is used to monitor the background due to reagents and laboratory glassware.

Calibration Standard is a sample prepared using a concentrated standard certified as traceable to NIST, which is diluted as directed, by the calibration section of the Chemistry SOP. This standard is used to quantify the compound in the sample.

Quality Control Check Standards are obtained from a second source which is different from the source of the calibration standard or the same source but with a different lot number compared to the lot of the calibration standard. Results of the analysis are compared with the calibration standard results. If the relative difference is 25% or greater, the instrument must be recalibrated.

Spiked Duplicates are prepared by addition to two aliquots of media material (soil, water) known amounts of the compounds being assayed from a laboratory reagent stock and analyzing these duplicate samples. The results from analysis of the sample and the spiked sample are used to calculate percent recovery of the spike. The results are used to monitor the precision of the measurement system.

Interference Check Samples contains both compounds of interest and interfering compounds at known concentrations to verify background and inter-element correction factor.

Control Chart is the basis for objective consideration of the analytical results. Construction of such charts assumes that the data approximate a normal distribution. The data can be plotted where the vertical scale represents the units of analytical results and the horizontal axis the results in the order in which they are obtained. The mean and the limits of dispersion, expressed in terms of the standard deviation are then calculated and plotted. The upper and lower control limits (UCL, LCL) are set at +3 and –3 standard deviations from the mean, respectively, and upper and lower warning limits (UWL, LWL) are set of +2 and –2 standard deviations. Results which fall outside the control limits signal an analysis which is out of control and indicate that analytical results for unknown samples obtained the same run are suspect. While results, which fall outside the warning limits, do not require strong action, a response may be needed if the results exceed these limits on a regular basis. (For detailed calculations see Chemistry SOP.)

Special Operational Procedures: Customer contracts, purchase orders and other specifications are checked periodically to provide for special or unusual requirements.

Precision is determined using data from the analysis of spiked duplicates. The precision control limits are based on the standard deviation of spike recovery data. The results of duplicate analyses to monitor precision (the Relative Percent Difference (RPD)) between the analysis is calculated as:

$$RPD = \frac{(s-d)}{s+d/2} \times 100$$

d = duplicate value
s = first sample value

Duplicate analyses which have values 5 times above the method detection limit and an RPD greater than 20% are considered to be insufficiently precise, and out of control procedures are initialed.

Quality Assurance Reports to Management by the quality assurance manager includes assessments of data accuracy, precision and summaries of standard control charts. Corrective actions and maintenance reports are also reported. These reports aid management to focus on areas that are not performing up to expectation. See more details in "Management Review of Quality System".

MANAGEMENT REVIEW OF QUALITY SYSTEM

EMS is committed to providing quality analytical services to all of its customers. In order to maintain a high level of quality services, EMS has implemented a quality assurance program with the goal of providing legally defensible analytical data of known and supportable quality that conforms to standards set by state and/or federal regulations, AIHA and ISO/IEC 17025. The QA program functions at the management level through company goals and management goals, and at the analytical level through standard operating procedures and quality control. These

functions are administered through data control and review process, which results in data that, is reproducible and technically accurate.

Annually, the management of EMS Laboratories will conduct a formal review of the quality program. This review will include the Laboratory Director, Technical Director, Laboratory Managers, and Quality Assurance Manager (or Analyst).

This review helps ensure that EMS' quality system is capable of meeting its goal of producing quality data. All aspects of the quality program will be reviewed. In addition to reviewing and updating the quality manual, results of audits, both internal and external will be reviewed along with proficiency evaluation results corrective action and complaint resolutions. Plans regarding future equipment and facilities, new analytical procedures and volume of work will be reviewed.

Feedback regarding EMS' quality of service from its customers will be reviewed.

The Quality Assurance Manger (QA) is responsible for ensuring that the review occurs (in June) and that all review findings are documented. Documentation will consist of meeting minutes and written documentation of any necessary corrective actions that are revealed during the review of the quality system. The QA Analyst will be responsible for implementing the corrective actions and documenting that implementation has occurred. All reviews and corrective action documentation are kept on file with QA Analyst.

The review will be shared with the staff within 2 weeks of completion of documentation or at the next monthly management meeting with staff.

EDDs DATA VERIFICATION PROCEDURE

Data and reports are sent electronically when requested by the customer. Signed reported are submitted in a PDF file. All reports go through the routine QC procedures before the documents are shipped.

For customers requesting data in a spreadsheet, a footnote is attached indicating that the integrity of the data cannot be guaranteed by EMS.

All documents have a paper trail.

REFERENCES

1. Chemistry Laboratory Standard Operation Procedure, Dec. 2007.
2. Standard Operation Procedures for Airborne Asbestos Analysis by Transmission Electron microscopy, Dec. 2007.
3. Standard Operation Procedure for Asbestos in Water by Transmission Electron Microscopy, April 26, 2000.
4. Standard Operation Procedure for Asbestos in Soil, Sludge, Bulk and Asbestos Water Method, EPA 600/R-93/116, EPA 600/4-83-043, Sep. 2000.
5. Polarized Light Microscopy Standard Operation Procedure, Dec. 2007.
6. PCM Standard Operation Procedure, Dec. 2007.
7. Microbiology Standard Operation Procedure, Dec. 2007.
8. EMS SOPs, all the technical methods generated by EMS are listed in EMS SOP Control List.
9. Reference Method, all the agency published technical methods are listed in EMS Reference Control List.
10. Instrument Manual, all the Instrument Manuals related to the instruments in use are listed in Manual Control List.
11. Computer software, all the computer software purchased separately or together with the instrument are listed in the Software Control List.
12. SOP 102 for Internal Audit.
13. SOP 108 for Purchasing.

14. General Requirements for the Competence of the testing and calibration laboratories, International Standard ISO/IEC 17025, 2005.
15. General Quality System Requirements, AIHA LQAP Policy Document Module 2A, 2007.
16. Industrial Hygiene Laboratory Accreditation Program (IHLAP) Specific Additional requirements, AIHA LQAP Policy Document Module 2B, 2007.
17. Environmental Lead Laboratory Accreditation Program (ELLAP) Specific Additional Requirements, AIHA LQAP Policy Document Module 2C, 2007.
18. Proficiency Testing (PT) for Industrial Hygiene Laboratories, AIHA LQAP Policy Document Module 6B, 2007.
19. EMS Employee Handbook, January, 2008.

APPENDIX 1

EMS DOCUMENTS

Sample Submittal (Chain-of-Custody)
Sample Tracking Form
PLM Sample Disposition Form
Corrective Action Forms
Laboratory Transmittal Form

Laboratory Submittal Form

Page 1 of 1

Date:	Time:	Relinquished by:	
Client:		Date of Shipment:	
Address:		Shipped from:	Carrier:
		Client P.O. No:	
Telephone:		Client Project ID:	
Contact:			
Results via: ☐ Fax No:		☐ Email address:	☐ Verbal

(Complete written reports will follow all analyses, in addition to any prior verbal, fax, or email results)

Turnaround Time:		Sample Preservatives:	
Number of Samples:		Sampler's Name:	
Date & Time of Sample Collection:		Holding Times:	Signature:

Type: ☐ Water ☐ Waste Water ☐ Soil ☐ Filter ☐ Impinger ☐ Sorbent Tube ☐ Other

EMS Only	Client Sample No.	Description/Location	Analysis	Volume/ Weight
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				

For EMS Only

Laboratory Number:		Received by:	Time:
Date of Package Delivery:		Shipping Bill Retained?	None
Condition of Package on Receipt:		Condition of Custody Seal:	None
Number of Samples:		Chain of Custody Signature:	
Disposition of Samples:	EMS LABS	Misc. Info:	SF 7/06

EMS Laboratories 117 West Bellevue Drive / Pasadena CA 91105-2503 / 626-568-4065

TRACKING SHEET

DATE DUE: _____TIME DUE: _____

EMS REPORT:

DATE IN: **PRIORITY:**

CLIENT:

ATTENTION:

CUSTOMER REFERENCE NO.:

NO. OF SAMPLES: DEPT:

FAX ☐ DATE/ TIME_____VERBAL ☐ DATE/TIME_____

EMAIL ☐ DATE/TIME_____

SUBJECT: _____

DATE TO ANALYST: _____ ANALYST: _____

ANALYTICAL METHODS: _____ DATE ANALYZED:_____

QA ANALYST: _____ QA DATE: _____

CORRECTION (1) by _____DATE: _____

CORRECTION (2) by _____DATE: _____

DATE RECEIVED FOR MAILING: _____

DATE MAILED: _____

Rev February 12, 2008

EMS LABORATORIES (PLM) TRACKING/DISPOSITION FORM

REPORT	**CUSTOMER**	REC. DATE	DUE DATE	PROGRESS	REMARK	SAMPLE NO.	TOTAL SAMPLE NO. IN THE DAY	ACCUMULATE SAMPE NO. IN THE DAY

CORRECTIVE ACTION REPORT

Date: **Client:**

Department: **Report #:**

Method: **Matrix:**

Identification and Definition of Problem:

Determination of the Cause of the Problem:

Corrective Action:

Follow-up:

QA/QC Analyst_____ **Date** _____

Lab ID 101634

Notice of Infraction

Employee Date

Client Report No./Sample No.

Completed by

Sample Control

- ☐ Lost Sample or COC
- ☐ Sample not logged in properly
- ☐ Improper interpretation of COC.
- ☐ Samples delayed in Log in
- ☐ Hazardous samples not labeled properly
- ☐ Failure to maintain proper documentation
- ☐ Special remarks left out on Analysis Log Sheet
- ☐ Samples labeled incorrectly
- ☐ Failure to maintain consistency
- ☐ Dissatisfied client
- ☐ Failure to maintain proper COC Documentation
- ☐ Other

Laboratory Personnel

Department

- ☐ Failure to meet holding time
- ☐ Missed analyses
- ☐ Analyzed wrong sample
- ☐ Improperly interpreted COC/instructions
- ☐ Failure to conform to methodology
- ☐ Incorrect units used
- ☐ Analytical interpretation error
- ☐ Calculation error
- ☐ Incorrect use of or failure to use dilution factors
- ☐ Rounding error
- ☐ Failure to report method used or detection limits
- ☐ Transcription error
- ☐ Failure to alert manager of problems which delay reporting
- ☐ Reporting late when results generated on time
- ☐ Failure to turn in QC data on a timely basis
- ☐ Corrective Action Reports not completed
- ☐ Improper documentaion
- ☐ Inconsistent date:
  - ☐ Results changed after review
  - ☐ Results not consistent within report
- ☐ Other

COMMENTS

_____

_____

_____

_____

_____

_____

EMS LABORATORIES
117 West Bellevue Dr. Pasadena, CA 91105-2503
Phone: (626) 568-4065
Fax: (626) 796-5282

Page ____ of ____ Date: _____ P O # : _____

Laboratory Transmittal Form

Laboratory: _____
Attention: _____
Address: _____
City: _____ State: _____ Zip: _____
Phone # ( ) _____

Please sign, date & return a copy of this form with the results to:
EMS Laboratories
Attn: _____
117 West Bellevue Dr.
Pasadena, CA 91104

EMS Lab #	Sample ID	Date	Time	Sample Matrix	Methods				Shipment Type:	Quantity
									Total # of containers	

Service Request:
☐ Standard (5-10 days) ☐ 48 HR TAT ☐ 24 HR TAT ☐ RUSH

Results needed by: _____

Sample Conditions:
Received on Ice? Yes / No Sealed? Yes / No
Special Shipment/ Handling or Storage Requirements:
_____

Relinquished by: _____ _____ _____ _____ am / pm
Signature Print Name Company Date Time

Received by: _____ _____ _____ _____ am / pm
Signature Print Name Company Date Time

Inhalelaboratory.doc: 02/2001

APPENDIX 2

EMS Organization Chart

ORGANIZATION CHART – January 15, 2008

Bernadine M. Kolk - *President*
Anthony J. Kolk, Jr. - *Secretary/Treasurer*

Bernadine M. Kolk - *Laboratory Director*
Anthony J. Kolk, Jr. - *Technical Director and*
Special Projects, Q. A. Coordinator, Laboratory Facilities
↓
Quality Assurance Manager and Method Development

Chemistry
Anthony J. Kolk, Jr.
(Director IH Chemistry)
↓
Supervisor , (GC, HPLC, AA, IR, UV-VIS)
↓
Chemist s, (AA, GFAA, IC, ICP)
↓
Technician (Gravimetric analysis, digestions)

PLM/PCM
Bernadine Kolk
↓
Manager Optical Microscopy
↓
Geologists
↓
Microscopist/Technician

Laboratory Safety Committee
Anthony Kolk
↓
Chemical Hygiene Officer
↓
Optical Dept. Representative
TEM Dept. Representative
Chemistry Representative
Environmental Biology Representative

Scanning Electron Microscopy and X-ray Diffraction
Anthony Kolk
↓
Analyst

Industrial Services
Anthony J. Kolk, Jr.
↓
Chemists
↓
Technicians

Transmission Electron Microscopy
Bernadine Kolk
↓
Supervisor
↓
Analysts
↓
Preparation Technician

Environmental Microbiology
Anthony Kolk
↓
Supervisor
↓
Technician

Sample Receiving
Bernadine Kolk
↓
Sample Receiving Staff

Support Staff
Bernadine Kolk
↓
Support staff

Network Administrator
Anthony Kolk
↓
Administrator

Some of the personnel are listed in two positions because of EMS' policy for cross training. All reports must be signed by a Director, Manager or a designee January 15, 2008

APPENDIX 3

EPA Specific Documentation

PROJECT SPECIFIC FOR US EPA

For EPA samples, additional information must be completed and included on a *DC-1 form*.

- Are the EPA chain-of-custody forms present
- Are airbills or airbill stickers present (make copies for EMS' files)
- Are traffic reports, packing lists present
- Are custody seals on shipping and/or sample containers
  Note their condition.
- If "yes" are custody seal numbers present
- Are airbill or airbill sticker number present
- Are sample tags present
- Are sample tag ID number present
- Is type and condition of the shipping container and sample container noted (on EPA forms) and noted on EMS' forms
- Verify agreement or non-agreement of information on receiving documents and sample containers
- Definition of terms used to describe sample condition upon receipt

METHOD USED TO VERIFY CONSISTENCY AND COMPLETENESS OF SAMPLE DATA PACKAGE FILE {CSF} – PROJECT SPECIFIC FOR U.S. EPA

A. Designation of document control officers (DCOs)

Cross-checking of sample tags, and custody records are performed first in Sample Receiving and then at receiving in the appropriate laboratory by

TEM staff preparation personnel
Senior optical staff member
Manager or senior chemist in chemistry

Laboratory bench sheets, personal and instrument run logs, sample tracking records, charts, strip charts, computer printouts, raw data summaries are the responsibility of the senior staff member assigned for each project by a Director.

B. The final assembly of the above documents, correspondence and other written documents is the responsibility of a director or manager.

C. An inventory of all documents relevant to each sample package is made by a director or manager.

D. The CSF is compiled with the original documents submitted to the Region or project liaison.

In all cases, the original copies are provided to the customer. Where originals cannot be submitted, a written explanation is attached by the project manager.

- Copies are marked as such when included instead of originals
- Copies are submitted when originals are in a bound logbook or originals were submitted with a previous package.

After all required analyses of a sample is completed the following documents are assembled and put in the data package file for that customer:

- Originals of log book pages (if in bound logbook or previous sent, copies)
- Originals of sample tracking records
- Originals of raw data and calculations (if inbound logbook or previously sent, copies)

- Originals of final results which includes computer printouts and chromatograms, strip charts, or other related instrument output
- Originals of results of QA/QC samples analyzed along with that sample on that particular day or 12 hour shift (if in bound logbook or previously sent, copies)
- Copies or original, as appropriate, of any correspondence and other written documents

After assembly of the above documents the project manager or director check the file for consistency and completeness of data. If everything is acceptable, the deliverable package is shipped using U.S. Mail, Federal Express, or other courier service.

SHIPMENT OF DELIVERABLE PACKAGE USING CHAIN-OF-CUSTODY SEAL

A. When shipping packages, where chain-of-custody seals are required, use EMS' chain-of-custody form, fill out seals with all the appropriate information and seal the sample appropriately. Fill out the chain-of-custody with items/samples sent, sample number, where sent, date of submittal/delivery, and method of delivery.

B. For EPA samples, submit a transmittal letter indicating that the CSF has been submitted.

SAMPLE LOG-IN SHEET

Lab Name EMS Laboratories			Page_____ of _____		
Received By (Print Name)			Log in Date		
Received by (Signature)					
	Sample Delivery Group No.				
		EPA Sample #	Corresponding Sample Tag#	Corresponding Assigned Tag#	Remarks Condition of Sample, Shipment etc
Remarks					
1. Custody Seal(s)	Present/Absent Intact/Broken				
2. Custody Seal Nos	_____				
3. Chain-of-Custody Records	Present/Absent				
4. Traffic Reports or Packing Lists	Present/Absent				
5. Air Bill	Airbill Sticker Present/Absent				
6. Air Bill No.	_____				
7. Sample Tags	Present/Absent				
Sample Tag Numbers	Listed/Not Listed on Chain-of-Custody				
8. Sample Condition	Intact/Broken/Leaking				
9 Does information on custody records, traffic reports and sample tags agree?	Yes/No				
10. Date Received at Lab	_____				
11. Time Received					
Sample Transfer					
Fraction	Fraction				
Area	Area				
By	By				
On	On				
Contract Client and Attach Records of Resolution					
Received By		Logbook No.			
Date		Logbook Page No.			

FORM DC-1

APPENDIX 4

Sample Preservation, Container Types, Holding Times

SAMPLE HOLDING TIMES, RECOMMENDED DIGESTION VOLUMES AND RECOMMENDED COLLECTION VOLUMES FOR INORGANIC DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES

Measurement	Digestion Volume. (mL)[a, c]	Collection Volume (mL)[a,c]	Treatment/ Preservative Holding Time[b]
Inorganic Analytes (except hexavalent chromium and mercury):			
Aqueous			
Total	100	600	HNO_3 to pH <2 6 months
Dissolved	100	600	Filter on site; HNO_3 to pH <2 6 months
Suspended	100	600	Filter on site 6 months
Solid			
Total	2 g	200 g	6 months
Hexavalent Chromium:			
Aqueous	100	400	24 hours Store at 4°± 2°C until analyzed
Solid	2.5 g	100 g	One month to extraction, 4 days after extraction Store at 4°± 2°C until analyzed
Mercury:			
Aqueous			
Total	100	400	HNO_3 to pH <2 28 days
Dissolved	100	400	Filter; HNO_3 to pH <2 28 days
Solid			
Total	0.2 g	200 g	28 days Store at 4°± 2°C until analyzed

[a] Unless stated otherwise.
[b] Either glass or plastic containers may be used.
[c] Any sample volume reduction from the reference method's instructions must be made in the exact proportion as described in the method and representative sampling must be maintained.

(EPA SW-486 Chapter 3)

CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR AQUEOUS MATRICES[A]

Name	Container[1]	Preservation	Maximum holding time
Inorganic Tests:			
Chloride	P, G	None required	28 days
Cyanide, total and amenable to chlorination	P, G	Cool to 4°C; if oxidizing agents present add 5 mL 0.1N $NaAsO_2$ per L or 0.06 g of ascorbic acid per L; adjust pH>12 with 50% NaOH. See Method 9010 for other interferences.	14 days
Hydrogen ion (pH)	P, G	None required	24 hours
Nitrate	P, G	Cool to 4°C	48 hours
Sulfate	P, G	Cool to 4°C	28 days
Sulfide	P, G	Cool to 4°C, add zinc acetate	7 days
Metals:			
Chromium VI	P, G	Cool to 4°C	24 hours
Mercury	P, G	HNO_3 to pH<2	28 days
Metals, except chromium VI and mercury	P, G	HNO_3 to pH<2	6 months
Organic Tests:			
Acrolein and acrylonitrile	G, PTFE-lined septum	Cool to 4°C, 0.008% $Na_2S_2O_3$[3], Adjust pH to 4-5	14 days
Benzidines	G, PTFE-lined cap	Cool to 4°C, 0.008% Na_2S_2O3[3]	7 days until extraction, 40 days after extraction
Chlorinated hydrocarbons	G, PTFE-lined cap	Cool to 4°C, 0.008% $Na_2S_2O_3$[3]	7 days until extraction, 40 days after extraction
Dioxins and Furans	G, PTFE-lined cap	Cool to 4°C, 0.008% $Na_2S_2O_3$[3]	30 days until extraction, 45 days after extraction
Haloethers	G, PTFE-lined cap	Cool to 4°C, 0.008% $Na_2S_2O_3$[3]	7 days until extraction, 40 days after extraction
Nitroaromatics and cyclic ketones	G, PTFE-lined cap	Cool to 4°C, 0.008% $Na_2S_2O_3$[3], store in dark	7 days until extraction, 40 days after extraction
Nitrosamines	G, PTFE-lined cap	Cool to 4°C, 0.008% $Na_2S_2O_3$[3], store in dark	7 days until extraction, 40 days after extraction

(Continued on next page)

Name	Container[1]	Preservation	Maximum holding time
Oil and grease	G	Cool to 4°C, add 5 mL diluted HCl	28 days
Organic carbon, total (TOC)	P, G	Cool to 4°C, store in dark[2]	28 days
Organochlorine pesticides	G, PTFE-lined cap	Cool to 4°C	7 days until extraction, 40 days after extraction
Organophosphorus pesticides	G, PTFE-lined cap	Cool to 4°C[4]	7 days until extraction, 40 days after extraction
PCBs	G, PTFE-lined cap	Cool to 4°C	7 days until extraction, 40 days after extraction
Phenols	G, PTFE-lined cap	Cool to 4°C, 0.008% Na_2S_2O3[3]	7 days until extraction, 40 days after extraction
Phthalate esters	G, PTFE-lined cap	Cool to 4°C	7 days until extraction, 40 days after extraction
Polynuclear aromatic hydrocarbons	G, PTFE-lined cap	Cool to 4°C, 0.008% $Na_2S_2O_3$[3], store in dark	7 days until extraction, 40 days after extraction
Purgeable aromatic hydrocarbons	G, PTFE-lined septum	Cool to 4°C, 0.008% $Na_2S_2O_3$[2,3]	14 days
Purgeable Halocarbons	G, PTFE-lined septum	Cool to 4°C, 0.008% $Na_2S_2O_3$[3]	14 days
Total organic halides (TOX)	G, PTFE-lined cap	Cool to 4°C, Adjust to pH<2 with H_2SO_4	28 days
Radiological Tests:			
Alpha, beta and radium	P, G	HNO_3 to pH<2	6 months

[A] Table originally excerpted, in part, from Table II, 49 **FR** 28, October 26, 1984, and revised as appropriate for SW-846. See Chapter Three, Chapter Four, or Section 6.0 of the individual methods for more information.
[1] Polyethylene (P) or Glass (G)
[2] Adjust to pH<2 with H_2SO_4, HCl or solid $NaHSO_4$. Free chlorine must be removed prior to adjustment.
[3] Free chlorine must be removed by the appropriate addition of $Na_2S_2O_3$.
[4] Adjust samples to pH 5-8 using NaOH or H_2SO_4.

(EPA SW-486 Chapter 2)

CONTINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES

	Container	Preservation	Maximum Holding Time
Asbestos in water	P, G	None allowed, cool, 4°C	48 hours
Asbestos bulk insulation	P, metal	None	Indefinite
Bacterial Test Coliform, Fecal and Total Fecal Streptococci	P, G	Cool, 4°C, 0.008% $Na_2S_2O_3$	6 hours
Inorganic Test			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days

Sampling Information for Industrial Hygiene Samples

Instrumentation	Reference Method	Sampling Media	Analyte	Sample Shipment	Preservation
AA	OSHA ID 121	MCE filter	Metal & Metalloid Particulates	Routine	None
AA	NIOSH 7082	MCE filter	Lead	Routine	None
Cold Vapor	NIOSH 6009	Sorbent Tube	Mercury	Routine	None
ICP-AES	NIOSH 7301	MCE filter	Elements	Routine	None
ICP-AES	OSHA ID 125G	MCE filter	Metal & Metalloid Particulates	Routine	None
ICP-AES	OSHA ID 206	MCE filter	Metal & Metalloid Particulates	Routine	None
GFAA	OSHA ID 105	MCE filter	Arsenic	Routine	None
GFAA	OSHA ID 189	MCE filter	Cadmium	Routine	None
GFAA	NIOSH 7102	MCE filter	Beryllium and compounds	Routine	None
GFAA	NIOSH 7105	MCE filter	Lead	Routine	None
GFAA	NIOSH 7900	MCE filter	Arsenic and Compounds	Routine	None
GFAA	NIOSH 7901	MCE filter	Arsenic Trioxide	Routine	None
GFAA	EPA 7740	MCE filter	Selenium	Routine	None
GC	OSHA ID 7	Sorbent Tube	Organic Solvents	Routine	5 °C
GC	OSHA 67	Sorbent Tube	Chlordane	Routine	5 °C
GC	OSHA 57	GFF w/H2SO4	Methyl Dianiline	Transfer filter to glass vial containing 2 ml of deionized water	5 °C
GC	OSHA 48	Sorbent Tube	Petroleum Distillate Fraction	Routine	5 °C
GC	OSHA 1001	Sorbent Tube	Tetrachloroethylene, Trichloroethylene	Routine	5 °C
GC	OSHA 99	Sorbent Tube	Propylene Glycol Monomethyl Ethers/Acetates	Routine	5 °C
GC	OSHA 69	Sorbent Tube	Acetone	Routine	5 °C
GC	OSHA 59	Sorbent Tube	Methylene Chloride	Routine	5 °C
GC	NIOSH 1003	CCT	Hologenated Hydrocarbons	Routine	5 °C
GC	NIOSH 1005	2 CCT	Methylene Chloride	Separate front and back tubes	5 °C
GC	NIOSH 1007	2 CCT	Vinyl Chloride	Separate primary and backup tubes and cape each tube	5 °C
GC	NIOSH 1010	CCT	Epichlorohydrin	Routine	5 °C
GC	NIOSH 1019	Sorbent Tube	1,1,2,2-Tetrachloroethane	Routine	5°C
GC	NIOSH 1022	CCT	Trichloroethylene	Routine	5 °C
GC	NIOSH 1300	CCT	Ketones I	MIBK must be refrigerated	5 °C

GC	NIOSH 1301	CCT	Ketones II	Routine	5 °C
GC	NIOSH 1302	CCT	n-Methyl-2-Pyrrolidinone	Keep cold, protected from prolonged exposure to light	5 °C
GC	NIOSH 1400	CCT	Alcohols I	Cooled	Store in freezer
GC	NIOSH 1401	CCT	Alcohols II	Routine	Store in freezer
GC	NIOSH 1402	CCT	Alcohols III	Routine	Store in freezer
GC	NIOSH 1403	CCT	Alcohols IV	Routine	5 °C
GC	NIOSH 1450	CCT	Esters I	Refrigerated	4 °C
GC	NIOSH 1453	MS	Vinyl Acetate	Routine	5 °C
GC	NIOSH 1454	CCT	Isopropyl Acetate	Routine	5 °C
GC	NIOSH 1457	CCT	Ethyl Acetate	Refrigerated	5 °C
GC	NIOSH 1458	CCT	Methyl Acetate	Refrigerated	5 °C
GC	NIOSH 1459	CCT	Methyl Acrylate	Routine	5 °C
GC	NIOSH 1460	CCT	Isopropyl Acetate	Routine	5 °C
GC	NIOSH 1500	CCT	Aliphatic Hydrocarbons	Routine	5 °C
GC	NIOSH 1501	CCT	Aromatic Hydrocarbons	Routine	5 °C
GC	NIOSH 1550	CCT	Naphthas	Routine	5 °C
GC	NIOSH 1551	CCT	Turpentine	Routine	5 °C
GC	NIOSH 1552	CCT	Terpenes	Routine	5 °C
GC	NIOSH 1602	CCT	Dioxane	Routine	5 °C
GC	NIOSH 1603	CCT	Acetic Acid	Routine	5 °C
GC	NIOSH 1604	CCT	Acrylonitrile	Routine	5 °C
GC	NIOSH 1606	CCT	Acetonitrile	Keep cool and pack securely for shipment	5 °C
GC	NIOSH 1609	CCT	Tetrahydrofuran	Routine	5 °C
GC	NIOSH 1613	CCT	Pyridine	Routine	5 °C
GC	NIOSH 1614	PCT W/HBr	Ethylene Oxide	Routine	5 °C
GC	NIOSH 1615	2 CCT	Methyl-tert-Butyl Ether	Routine	5 °C
GC	NIOS H 1616	CCT	Butyl Glycidul Ether	Routine	5 °C
GC	NIOSH 2000	Sorbent Tube	Methanol	Pack securely for shipment	5 °C
GC	NIOSH 2002	Sorbent Tube	Aromatic Amines	Routine	5 °C
GC	NIOSH 2004	Sorbent Tube	DMF	Routine	5 °C
GC	NIOSH 2500	Carbon beads	Methyl Ethyl Ketone	Routine	5 °C
GC	NIOSH 2519	2 CCT	Ethyl Chloride	Separate front and back tube	5 °C
GC	NIOSH 2538	XAD-2	Acetaldehyde	Routine	5 °C
GC	NIOSH 2539	XAD-2	Aldehydes Screening	Routine	5 °C
GC	NIOSH 2541	XAD-2	Formaldehyde	Routine	5 °C
GC	NIOSH 2546	Sorbent Tube	Phenol	Routine	5 °C
GC	NIOSH 2553	A-CMs	Ketones II	Routine	5 °C

GC	NIOSH 2555	A-CMs	Ketones I	Refrigerated	5 °C
GC	NIOSH 2557	A-CMs	Diacetyl	Ship cold (5 °C)	store in dark (5 °C)
GC	NIOSH 2558	A-CMs	Acetoin	Ship cold (5 °C)	store in dark (5 °C)
GC	NIOSH 5503	GFF & Florisll	Polychlorobiphenyls (PCB's)	Transfer filter to glass vial after sampling	5 °C
GC	NIOSH 5510	MCEF & Chrom102	Chlordane	Routine	5 °C
GC	NIOSH 5523	OVS-7	Glycols	Pack cold for shipment	5 °C
Gravimetric	NIOSH 0500	PVC filter	Particulates N.O.R.	Routine	None
Gravimetric	NIOSH 0600	PVC filter	Particulates N.O.R, Resp.	Routine	None
Gravimetric	NIOSH 5000	PVC filter	Carbon Black	Routine	None
Gravimetric	OSHA 58	Glass Fiber Filter	Coal Tar Pitch Volatiles (Benzene Soluable Fraction)	Each GFF must be transferred to a separate scintillation vial after sampling and the vial sealed with a PTFE-lined cap.	5 °C Samples must be protected from direct sunlight.
HPLC	OSHA 64	GFF w/DNPH	Glutaraldehyde	Ship with blue ice	5 °C
HPLC	OSHA 47	GFF w/12PP	Methylene Bispheny Isocyanate (MDI)	Routine	5 °C
HPLC	OSHA 42	GFF w/12PP	Toluene-2,4-diisocyanate and Toluene-2, 6-diisocyanate (TDI)	Routine	5 °C
HPLC	OSHA 42	GFF w/12PP	1,6-Hexamethylene Diisocyanate	Routine	5 °C
HPLC	NIOSH 5004	MCE	Hydroquinone	Transfer filter immediately to jar with 10ml 1% acetic acid and ship sample solution	None
HPLC	NIOSH 5506	PTFE & XAD-2	Polynuclear Aromatic Hydrocarbons	Transfer filter to culture tube and wrap sorbent and culture tube with Al foil. Ship at 0°C	Protect from heat and UV light
IC	NIOSH 7903	Sorbent Tube	Inorganic Acids	Routine	None
IC	NIOSH 7906	MCE+pad w/Na2CO3	Fluorides	Routine	None
IC	NIOSH 6011	Silver filter	Chlorine and Bromine	Routine	Protect from light

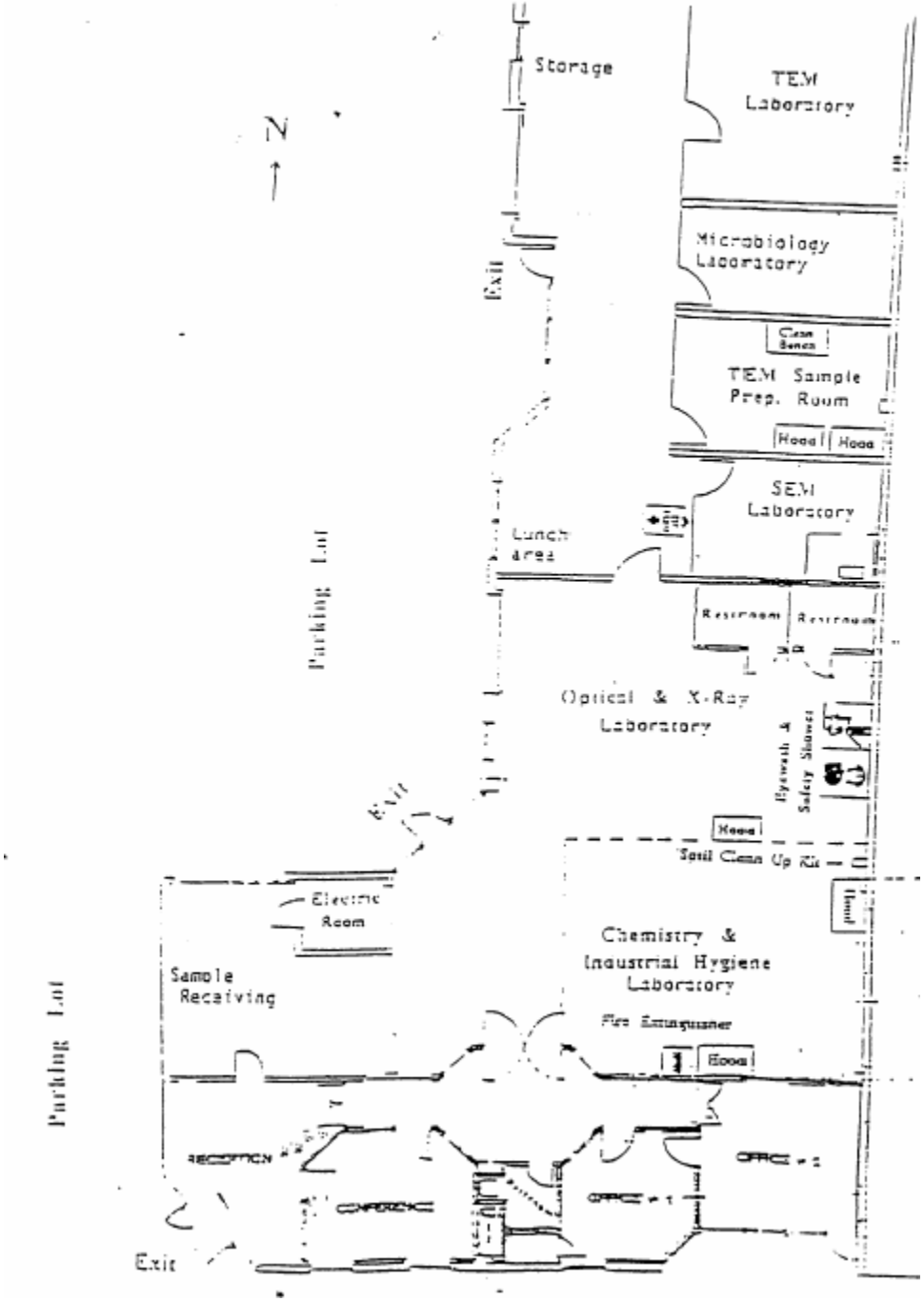
IC	OSHA ID 186SG	CCT	Acetic Acid	Routine	5 °C
IC	OSHA ID 165SG	Silica Gel Tube	Acid Mist	Routine	5 °C
IC	OSHA ID 174SG	Silica Gel Tube	Hydrogen Chloride	Routine	5 °C
IC	OSHA ID 190	MS w/triethanolamine	Nitric Oxide	Routine	5 °C
IC	OSHA ID 182	MS w/triethanolamine	Nitrogen Dioxide	Routine	5 °C
IC	OSHA ID 214	GFF w/sodium nitrite	Ozone	Routine	5 °C
IC	OSHA ID 111	MCE	Phosphoric Acid	Routine	5 °C
IC	OSHA ID 113	Silica Gel Tube	Sulfuric acid	Routine	5 °C
IR	NIOSH 5026	MCE or PVC	Mineral oil and mist	Routine	None
PCM	NIOSH 7400	MCE filter	Asbestos Fibers	Routine	None
PCM	OSHA ID 160	MCE filter	Asbestos Fibers	Routine	None
PLM	NIOSH 9002		Bulk Asbestos	Seal securely to prevent escape of asbestos	None
PLM	OSHA ID 191		Bulk Asbestos	Routine	None
PLM	EPA 600/R-93/116		Bulk Asbestos in Building Materials	Routine	None
TEM	NIOSH 7402	MCE filter	Asbestos Fibers	Pack to reduce shock	None
TEM	AHERA 40 CFR 763 for School clearances	MCE filter	Asbestos Fibers	Routine	None
TEM	ASTM D5755	Wipe or microvacs	Asbestos in dust & on wipe	Routine	None
Titration	NIOSH 7401	PTFE	Alkaline Dusts	Routine	None
UV-Vis	NIOSH 3500	PTFE & 2 IMP	Formaldehyde	Transfer sample to low density polyethylene bottles before shipping	None
UV-Vis	NIOSH 3503	Bub	Hydrazine	Routine	None
UV-Vis	NIOSH 3508	IMP	Methyl Ethyl Ketone Peroxide	Transfer to sanilized, foil wraped vials, ship in dry ice	Freezer
UV-Vis	NIOSH 6010	Soda Lime	Hydrogen Cyanid	Routine	None
UV-Vis	NIOSH 6014	MS w/TEA & Oxidizer	Nitric Oxide and Nitrogen Dioxide	Routine	None
UV-Vis	NIOSH 7600	PVC	Hexavalent Chromium	Ship to lab within 24 hrs of sampling	Analyze within 6 days
UV-Vis	NIOSH 7904	MCE & Bub	Aerosol and gas Cyanines	Routine	Analyze within 5

					days
XRD	NIOSH 7500	PVC	Crystalline Silica	Routine	None
XRD	NIOSH 9000		Bulk Asbestos, Chrysotile	Routine	None
XRD	OSHA ID 142	PVC	Quartz & Cristobalite	Routine	None

APPENDIX 5

Floor Plan

FLOOR PLAN OF EMS LABORATORIES



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