



Sacramento Regional County Sanitation District

SRCSD Environmental Laboratory

QUALITY ASSURANCE PROGRAM MANUAL

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August 2010

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8/18/10

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SECTION 1. INTRODUCTION

The SRCSD Environmental Laboratory (SRCSD EL) is located at the Sacramento Regional Wastewater Treatment Plant (SRWTP) in Elk Grove. The SRCSD EL is a fully certified environmental laboratory that serves its customers with analytical and field sampling/monitoring services. It offers its environmental services to various groups within and outside the County of Sacramento organization structure. The laboratory operates seven days per week with normal working hours from 7 AM to 3:30 PM.

This Quality Assurance Program Manual describes the essential components and practices incorporated into the daily operation of the laboratory to assure that reported data will be error-free, accurate, legally defensible, and timely. Quality assurance (QA) is those planned and systematic actions necessary to provide sufficient confidence that a laboratory's product or service will satisfy given (internal) requirements for quality. A subset of QA is quality control (QC). Quality control refers to internal activities or activities according to externally established standards used to monitor the quality of analytical data to ensure that it satisfies customer and method (where applicable) specified acceptance criteria.

The QA program incorporates requirements and guidelines set forth by the US Environmental Protection Agency (EPA) and the California Department of Public Health Services (DPH). The SRCSD EL maintains its laboratory accreditation through the DHS by participating in mandatory periodic performance evaluations and audit inspections through its Environmental Laboratory Accreditation Program (ELAP) and following their guidelines and requirements. It is the responsibility of the SRCSD EL to carry out its testing and calibration activities in such a way as to meet the requirements of this QA program, to satisfy clients needs, regulatory authorities or organizations providing certification recognition. A copy of the SRCSD EL's DPH ELAP certification, including a list of tests the laboratory is certified for is presented in Appendix 4.

This Quality Assurance Program Manual provides laboratory employees and its customers with a description of the laboratory's quality assurance (QA) policy and program. The written procedures described herein are binding on all laboratory personnel. They shall be adhered to implicitly.

This manual is available to all laboratory employees, its customers, and management. The Laboratory QA Officer is responsible for providing the documented policy and ensuring that all staff familiarize themselves and comply with the policies and procedures in the manual and associated documentation.

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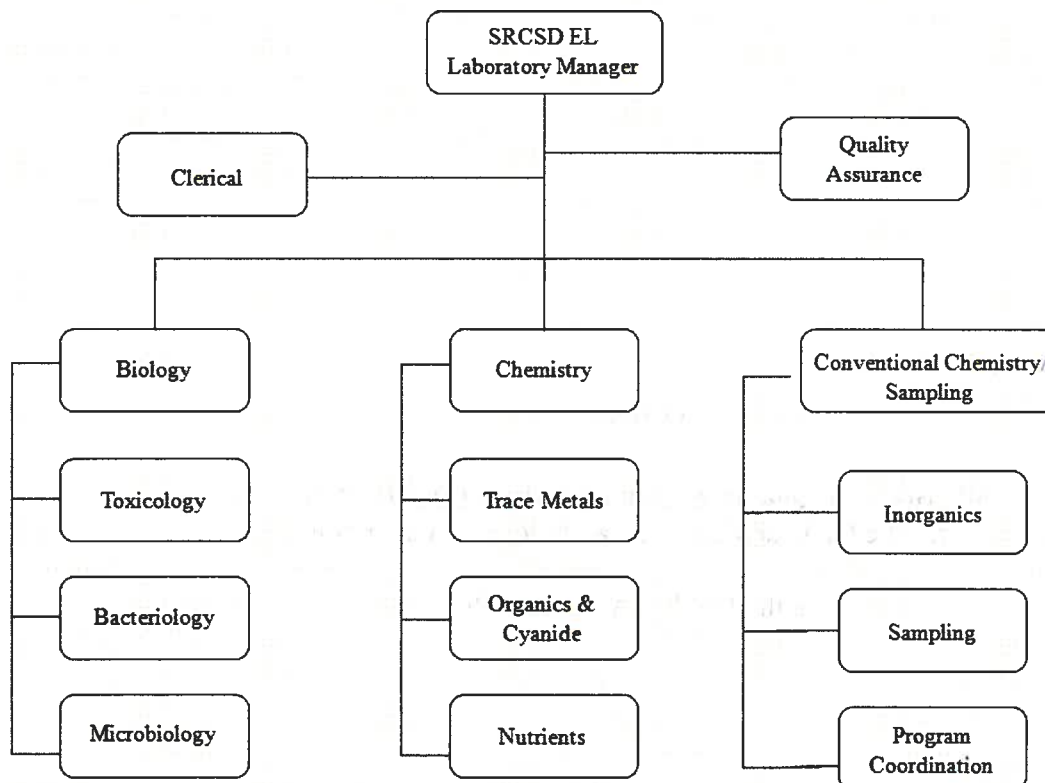
SECTION 2. LABORATORY ORGANIZATION AND PERSONNEL RESPONSIBILITIES

2.1. ORGANIZATION

The Sacramento Regional County Sanitation District Environmental Laboratory (SRCSD EL) is a full service environmental laboratory facility that provides for analytical testing, field monitoring, and sampling services. Approximately 35,000 samples are processed through the SRCSD EL yearly with over 120,000 related tests. Services include field sampling, field monitoring, chemical, physical, biological, and toxicological analyses, for mandated monitoring programs, or for Sacramento Regional Wastewater Treatment Plant (SRWTP) process control and optimization, and for special studies or projects.

There are three discrete work units in the laboratory. The organization chart for the SRCSD EL is shown in Figure 1. It presents the work unit relationships and illustrates the placement of the quality function, through the Quality Control Coordinator, within the organization.

Figure 1. SRCSD EL functional groups organization chart.



2.2. ORGANIZATIONAL ROLES AND RESPONSIBILITIES

Laboratory personnel are selected for employment based on professional qualifications, including education and relevant experience. Staffing levels are sufficient to maintain effective processing of the customer workload, quality control, and activities required for California DHS accreditation. Job descriptions are on file in the laboratory and available through the County of Sacramento, Public Works Agency's Department of Human Resources. The following paragraphs give a brief overview of the laboratory functional levels relative to the administration of the QA program.

Laboratory management shall ensure the competence of all employees who operate specific equipment, perform tests and/or calibrations, evaluate results and sign reports. Personnel performing complex tasks are pre-qualified based on appropriate education, training, experience, and/or demonstrated skills. The laboratory maintains records of the relevant authorization(s), competence, educational and professional qualifications, training, demonstrated skill, and experience of all technical personnel. This information is readily available and includes the date on which authorization and/or competence is confirmed.

2.2.1. Laboratory Manager

The Laboratory Manager is responsible for the daily operation of the SRCSD EL. He or she has the ultimate and overall responsibility for ensuring that all laboratory employees have demonstrated proficiency for their assigned functions and that all data reported by the laboratory meet the required QA criteria and regulatory requirements. The Manager oversees the development, implementation, approval, and continued operation of the laboratory. The Manager is responsible for initiating the QA/QC Program, assures that all laboratory employees are aware of and compliant with provisions set forth in the Quality Assurance Program Manual, and enforces applicable good laboratory practices. He or she sees to it that QA/QC information and training is provided to laboratory employees.

2.2.2. Quality Assurance Officer (QAO)

The SRCSD EL's Quality Assurance Officer (QAO), reports directly to the Laboratory Manager. The QAO advises the Laboratory Manager concerning QA/QC issues, reviews and approves all SRCSD EL Standard Operating Procedures (SOPs). QAO annually reviews and updates the Quality Assurance Program Manual and submits it to the Laboratory Manager for approval. The QAO serves as the focal point for all QA/QC activities within the SRCSD EL and has overall responsibility and authority for the implementation, management, and maintenance of the quality assurance program. The QAO works directly with section supervisors in determining the adequacy of corrective actions to all audits (both internal and external). The QAO has delegated administrative responsibility to coordinate the laboratory's certification through the California State Department of Public Health. The QAO administers, and/or coordinates all QA related

activities that include, but not limited to, the following:

- Special QA studies (e.g., troubleshooting blank contamination, corrective action investigation, etc.).
- New method/technology validation studies.
- Audit reports and follow-up investigations.
- Performance evaluation testing records.
- Files of National Institute of Standards and Testing (NIST) traceability records for weights, thermometers, volumetric syringes, etc.
- Initial Demonstration of Capability (IDOCs).
- Method Detection Limits (MDLs).
- Initial Instrument Detection Limits (IDLs).
- Summaries of spikes, replicates, surrogates, and updates of acceptance limits.
- Initiate and coordinate internal performance evaluation (PE) studies and QA audits for SRCSD EL analytical operations.
- Safety operations in the laboratory as the Chemical Hygiene Officer.

2.2.3. Laboratory Supervisors

The Laboratory Supervisors direct technical personnel in the proper performance of laboratory procedures and the reporting of results. Supervisors insure that appropriate corrective action is taken based on quality control indicators, or internal and external audits within their work units. They communicate regularly with the Laboratory Manager, the QAO, and analysts on technical issues and problems; insure that appropriate methods are used and equipment is properly maintained and operated; and plan activities leading to testing and modification of laboratory procedures. The incumbents provide for resources and adjust workloads for laboratory staff to facilitate completion of assigned tasks in a timely manner. They see to it that employees are provided a safe work environment supplied with appropriate personal protective equipment. They are responsible for all data produced by the analytical units within their sections. Supervisors are responsible for performing a final review of each work product (data, written reports, etc.) to make certain that all quality control information is complete, properly utilized, documented and maintained within the various analytical work units within their section.

2.2.4. Program Coordinators

Program Coordinators are senior-level, or above, laboratory employees that act as liaisons with external customers, including Program Managers, project leads, and special study leads. All analytical and field service activities provided by the laboratory in support of customer programs are channeled through the Program Coordinators. Duties and responsibilities assigned these positions are described as, but not limited to, the following:

- Assist external customers in setting up Laboratory Service Agreements for

environmental laboratory services; review and assist, as needed, to develop data quality objectives (DQOs) and quality assurance program plans (QAPPs).

- Coordinate customer schedules and workloads with Section Supervisors so that laboratory workloads and program needs are maintained at a manageable level.
- Arrange for analytical service and shipment of samples to outside laboratories for overflow work or specialized analytical procedures not available at the SRCSD EL.
- Assure that all program objectives are met, that reports are generated on time, and all data is complete and correct.

2.2.5. Sample Custodian/Coordinator (SC/C)

The Sample Custodian/Coordinator (SC/C) is a laboratory assigned position whose responsibilities include sample receiving, sample login, sample disposition, and reporting functions. In addition, this person also organizes and keeps records of sample receiving area refrigerator temperatures, sample locations, and notifies appropriate section staff when samples have arrived for analysis. If samples are not needed immediately for testing, the SC/C puts the sample in the appropriate laboratory sample refrigerator for future access by the analysts.

2.2.6. Technical and Analytical Staff

Technical and analytical level employees are non-supervisory, apprentice and journey, classes that have clearly defined areas of responsibility. Their work assignments are commensurate with their specific levels of skill, training, education, expertise, and experience. These employees work in the Biology, Chemistry, or Conventional Chemistry Sections of the laboratory. Job classes included in this group are the Biologists, Chemists, Environmental Laboratory Analysts (ELAs), Senior ELAs, and, to a limited extent, Laboratory Assistants.

Biologists, Chemists, and Sr. ELAs (as part of their job descriptions) may be required to act in a lead person capacity. As a lead person assignment, employees have limited oversight authority and responsibilities to assist the Section Supervisor in maintaining the work area, and to provide support in technical or scheduling capacities. A lead person may also head a special study or organize field investigations for routine and unscheduled programs or studies.

The Biologist and Chemist classes (as well as the Laboratory Supervisors and Laboratory Manager classes) are considered “professional” level positions in that it requires a Baccalaureate degree in a science related field. As such, they may be called upon to act

as expert witnesses in regulatory or legal matters pertaining to water quality or environmental analytical issues.

Technical and analytical class employee work assignments include, but are not limited to, the following:

- Collect field samples;
- Conduct field-monitoring tasks;
- Perform tests on given dates and times following prescribed procedures;
- Be aware of and insure that all applicable QC activities are performed;
- Take necessary action when quality indicators do not meet established acceptance criteria, assure that corrective action is implemented when warranted;
- Have a working knowledge of all policies, procedures, and QC activities within their respective work areas;
- Insure that documentation of work performed is complete, accurate, and analytical data is reported in a timely manner;
- Notify their immediate supervisor of any issues/problems with any work product so that any necessary corrective action be performed when indicated;
- Follow the appropriate SOPs for their assigned work;
- Insure that complete and accurate postings of all individual analytical data points, data qualifier flags, and explanatory footnotes are properly accounted for and documented in the LIMS.

2.2.7. Analytical Support

The basic support level employee classification used in the laboratory is the Laboratory Assistant. Employees holding this position are most often responsible for sample collection, reagent/media preparation, assist in sample check-in, maintain common laboratory supplies, carry out housekeeping duties, and under direction, perform rudimentary analytical functions. These employees are responsible for having a working knowledge of fundamental analytical methodologies, including QA/QC requirements, used within their work areas.

2.2.8. Laboratory Administrative Support

Employees not assigned to any of the three analytical sections of the laboratory serve at various levels in the Administrative Support Section. The support section functions include clerical, LIMS administration, program coordination, field monitoring, specialized sample collection, records management, sample shipping, sample receipt, procurement, and sample management and disposition. Job classes assigned these functions include Laboratory Supervisor, Sr. ELA, Sr. Office Assistant, ELA, and Laboratory Assistant.

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SECTION 3. QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT OF DATA

The quality assurance policy reflects the laboratory management's commitment to quality assurance throughout the data generating and processing operations. The SRCSD EL conducts all business activities (sampling and measurements) under prescribed conditions and by using techniques that achieve results to a high degree of reliability and accuracy. The measurements follow generally recognized good laboratory practices and documented protocols. The purpose of the Laboratory Quality Assurance Program Manual is to provide policy and oversight for the administration and maintenance of quality assurance (QA) and quality control (QC) within the SRCSD EL.

Quality assurance (QA) is an integrated system of management activities to ensure that a process or service meets the customer requirements. QA ensures that the facility, equipment, personnel, testing methods, data, and QC procedures are compliant with regulatory and internal policies so that the reportable results are appropriate for its intended use.

Quality control (QC) is the routine technical activities that quantitatively measure the success of a process or service against defined standards of performance established to meet the needs of the customer. It is the overall system of operations designed to control the particular analytical process or service.

QC indicates nothing about the systems while QA tells you nothing specific about the validation of the results.

Quality of the work is the responsibility of every employee. Meeting this commitment will result in continued customer satisfaction and improved quality of life for employees.

Specific QA **program objectives** for the SRCSD EL are:

- To develop and put into service methods and procedures capable of meeting the end user's needs for precision, accuracy, sensitivity, defensibility, and specificity.
- To ensure that all laboratory employees receive appropriate training in quality technology, sufficient in depth, to enable them to carry out the provisions of this manual.
- To establish the level of quality of the laboratory's routine performance as a baseline in which to measure the effectiveness of quality improvement efforts.

- To make any changes in routine methodology found necessary to make it compatible with established performance criteria as established in the previous item above.
- To monitor the routine operational performance of the laboratory through participation in appropriate inter-laboratory testing programs and to provide for corrective actions as necessary.

SECTION 4. SAMPLING PROCEDURES

4.1. GENERAL SAMPLING INFORMATION

Documentation of sample scheduling, collection, and handling is of critical importance for any regulatory monitoring efforts. Record management of samples collected and delivered to the laboratory for analysis follow a formal chain-of-custody (COC) procedure to provide a written record of sample traceability, accountability, and serves to validate sample integrity. A sample of the COC form used by the SRCSD EL is in Appendix 7 of this manual. All samples delivered to the SRCSD EL (outside of samples collected by laboratory employees) are controlled by the COC procedures. It is the responsibility of the SC/C to effectively maintain sample receipt records, control the sample receiving and custody areas, and insure that the receiving area is maintained in a clean, orderly, and secure manner. Depending on the nature of the samples, they will either be stored or delivered to the appropriate analyst(s) for immediate testing.

4.2. SAMPLE COLLECTION BY LABORATORY EMPLOYEES

Samples collected by laboratory staff employ techniques that are consistent with the requirements for the application and parameters tested. They are collected in appropriate containers guided by the methods being applied. Sample holding time, preservation, and appropriate container guidance are well documented in EPA records that can be found in Table II, Part 136 of 40 CFR. A facsimile of the current table is presented as Appendix 2 at the end of this Manual (Make sure this is updated).

Training in proper sampling techniques and sample handling is provided to laboratory employees responsible for collecting samples to ensure they are representative. Training includes user instructions on the proper operation and maintenance of sampling equipment used in the collection process. Care is taken in sample collection to minimize the possibility that foreign material is introduced into the sample by the sample collector or sampling device. Equipment blanks, trip blanks, container blanks, and field blanks may be employed to check for contamination due to sampling.

4.3. REPRESENTATIVE SAMPLE

A representative sample is one that reflects the characteristics of a population. Samples are collected in such a way that they are typical of the lot from which they are taken. Laboratory personnel who collect samples have been adequately trained in proper sampling, preservation, and transportation techniques, to protect against loss, contamination, misidentification, tampering, or other possible errors that may be introduced.

There are three types of variability which must be measured or otherwise accounted for in field sampling:

- **Temporal Variability** – The range of results due to changes in analyte concentrations over time. An example would be the range of concentrations obtained for total suspended solids in wastewater samples collected at different times from an outfall.
- **Spatial Variability** – The range of results due to changes in contaminant concentrations as a function of their location. An example would be the range of concentrations for a given parameter from a site where discrete “hot spots” are present due to localized mechanical effects of turbulence in a treatment process.
- **Sample Handling Variability** – The range of results due to the effects of sample collection and handling procedures. This variability manifests itself as a positive bias due to errors such as unclean sampling equipment, cross contamination, or a negative bias due to improper containers or sample preservation and preparation.

4.4. SAMPLE INFORMATION AND DOCUMENTATION

All samples and sample containers delivered to the laboratory must have appropriate labels and documentation. The collecting organization is responsible to include a COC record that accompanies the sample(s). The COC is a legal document that provides information as to the type of sample(s) (grab or composite), who collected the sample(s), sample collection time(s), preservation, transportation, any relevant notes concerning the collection procedure, and sample delivery information. The label attached to each sample container must have, as a minimum, the following information:

- Sample location;
- Sample date and time;
- Sample collector;
- Laboratory number, if sample is pre-logged;
- Chemical preservatives, if added.

The SRCSD EL will provide its customers with the appropriate type and number of sample containers to meet the program requirements for specified analytes. Refer to Appendix 3 to identify the appropriate container and preservation requirements for specific analyte(s) to be tested.

4.5. SCHEDULED AND AD HOC SAMPLING EVENTS

Most sampling events for new or established programs, projects, or studies requiring analytical or field monitoring/sampling services are scheduled in advance and can be pre-logged into the LIMS by Program Coordinators, LIMS Administrator, or Section Supervisors. Each time samples are scheduled in advance, LIMS will provide unique laboratory identification numbers, printed labels with lab numbers, and sample information

for each container. Programs, projects, or studies are assigned a program name in LIMS, regardless of the source of the samples.

Often samples are collected and/or delivered to the SRCSD EL on an ad-hoc, or unscheduled, basis. Ad hoc samples delivered to the laboratory are first formally received by the SC/C then processed into LIMS. The required analysis information provided on the COC form is put into the LIMS at the time of receipt. The COC serves as the official record for analysis and reporting needs.

With prescheduled programs the Laboratory Program Coordinators will have advance knowledge of program, project, or study sampling events and are thus able to synchronize the available resources with the Laboratory Supervisor(s) to manage the section workloads. If resources are not available to do the testing in-house, the Program Coordinator will arrange to have samples shipped to, and tested by, an outside laboratory that is CA DPH ELAP certified, or qualified, for providing those services. If possible, the Program Coordinator may be able to negotiate a change in the sampling schedule with the Program Manager(s) that will enable the testing to be done in-house. It is the responsibility of the Program Coordinator to ensure that any work changes are first approved by the Program Manager and clearly communicated to affected parties. The Program Coordinator will insure that all change records are properly documented and kept on file.

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SECTION 5. SAMPLE MANAGEMENT

Sample management describes how the laboratory processes samples. Documentation of the sample collection and sample handling processes are an important aspect of the monitoring and reporting effort. COC records provide written documentation of sample traceability, accountability, and serves to validate sample integrity.

5.1. SAMPLE CUSTODY

Samples are considered officially received by the SRCSD EL when the person delivering the samples physically relinquishes them to the SC/C. The SC/C shall officially receive the samples by signing (**initials are not acceptable**), dating and recording the time of receipt on the COC form. At the time of delivery, the SC/C, or his/her designee, accepting the samples will take the following actions:

- 5.1.1. Record the condition of the samples. The SC/C will inspect the sample containers and document whether the individual samples and/or sample containers are in agreement with the information recorded on the COC. The SC/C will verify and record the following when samples are received and inspected:
 - Presence or absence and condition of custody seals on shipping and/or sample containers;
 - Presence or absence of sample container labels;
 - Sample containers listed or not listed on the COC;
 - Condition of the sample containers (leakage, broken custody tape, possible sources of contamination, etc.);
 - Date of receipt;
 - Time of receipt;
 - Problems and discrepancies.
- 5.1.2. If samples require a quick turnaround, the employee accepting the samples will notify the appropriate Section Supervisor immediately that the samples have been received and processing will be handled accordingly.
- 5.1.3. If a shipping container (e.g., ice chest, package) contains a temperature blank, the temperature is to be read immediately upon receipt and recorded on the COC form. The information may be used by the Program Manager to determine if the data quality objectives (DQOs) of the program, project, or study, are compromised. If a temperature blank is not included with the sample container, it shall be noted on the COC form. If the temperature blank is equal to or less than required, no further action is necessary. If the temperature blank of any ice chest is above the minimum required, the SC/C or designee will record the excursion on the COC form and notify the Section Supervisor and Program Coordinator.

It is generally recognized that on occasion samples may be collected locally and will be transported and received during normal business hours within short times of their collections. For samples received within a short time of sampling, measuring the temperature of a temperature blank may not be practical since the samples will not have reached equilibrium temperature with the ice. However, the SC/C, or designee, will note this in the comments section of the COC form.

- 5.1.4. Completed copies of the COC form will be made by the SC/C or his/her designee, for 1) the person delivering the samples, 2) the Program Coordinator, and 3) the respective Section Supervisor(s). The original completed COC form is filed in the SRCSD EL archives.
- 5.1.5. Ad-hoc samples delivered to the laboratory during normal business hours will be assigned laboratory identification numbers upon receipt by the SC/C. Samples that have been prescheduled (LIMS terminology refers to this as “pre-accession”) are auto-logged ahead by LIMS and have pre-printed labels for the containers. Each sample will have a unique identification number assigned by LIMS. The lab numbers are entered on the COC form by the SC/C, or designee, when they are delivered. Sample identification labels are placed on the containers by the SC/C or designee. The laboratory number for each sample is composed of ten unique characters that include the date and a sequential number for that date, starting each day with number one. (e.g., the lab number, in order of appearance, will read year xx, month xx, day xx, and number xxxx, so that 0302180049 represents the 49th sample received Feb. 18, 2003).

5.2. SAMPLE PRESERVATION AND STORAGE

5.2.1. Preservation

Sample containers that have preservative added will be noted on the label as to the chemical nature of the preservative. Preservation techniques are guided by the method being applied. Guidance for sample preservation and holding times is available in such references as: Standard Methods for the Examination of Water and Wastewater, American Society for Testing Materials (ASTM), Environmental Protection Agency (EPA) Methods for Chemical Analyses of Water and Waste, 40 Code of Federal Regulations (CFR), Parts 136 or 141. Appendix 3 presents guidance for preservatives and holding times extracted from the above mentioned references.

5.2.2. Storage

Samples requiring refrigeration for storage will be held in one of the designated sample refrigerators. Sample storage refrigerators are maintained at between

1°-6° C. Storage refrigerator temperatures are recorded from a thermometer kept in a bottle of 25% ethylene glycol solution that is calibrated against a certified thermometer. Storage refrigerator temperatures are measured daily and recorded on the refrigerator temperature log maintained by the SC/C, or designee.

5.2.3. Sample Storage Security

Samples are maintained in a secure status inside the confines of the SRCSD EL. Access to the laboratory is restricted to SRWTP employees. Visitors and vendors may access the facility only when approved and on official laboratory business. Visitors and vendors are escorted and remain with the respective laboratory employee(s) at all times when present in the laboratory.

Access to the sample storage area during non-operating hours is restricted (through keyed entry) to SRWTP and the janitorial service providers. Locked refrigerator or freezer space is available to customers upon request for samples that require locked storage.

Samples that have been properly received are delivered directly to where the analytical work will be performed or stored in a secure area within designated sample receiving area refrigerators or workstation storage areas.

5.3. SUBCONTRACTING

Sometimes samples will need to be sent to an outside laboratory for testing due to limitations on available resources (e.g., workload demands, staffing shortages, instrument down time). Other reasons might be where samples have specific testing requirements beyond our capabilities; or test procedures which our laboratory is not certified to perform. If an outside laboratory will be needed to perform the analytical procedure(s), the selection will be dependent on considerations such as program requirements, site visit evaluations, performance checks, costs, responsiveness, and reference checks. The ability of the outside laboratory to maintain a high level quality of work, that includes consistently meeting our own SRCSD EL QC standards, will also be taken into consideration as part the selection criteria.

5.4. SAMPLE SHIPPING

Whenever samples need to be shipped to outside laboratories for testing, the Program Coordinators will arrange for packaging, pick-up, and shipping via ground or air transportation. The Program Coordinator will see to it that samples will be properly preserved and protected from damage during shipping. Shipping containers will usually consist of ice-chests. They may be picked up by surface courier service, the outside

laboratory, or on-site courier service may be utilized through the Plant Procurement Section. Any shipping that goes by air will be processed through Fed-Ex or other county approved air freight carriers. The Plant's Administration Section has shipping supply documents and instructions for shipping samples by air freight.

5.5. SAMPLE DISPOSITION

Treatment plant samples and their extracts that are not part of an investigation will normally be disposed of after seven days and the satisfactory completion of the tests. The COC may indicate that any remaining sample(s) is/are not to be disposed of until official notification is given by the customer. Samples that need to be held longer than normal holding times will be tagged by the SC/C

Samples other than treatment plant samples (e.g., IWS, ambient monitoring, special studies, sewage spills, toxicity follow-up samples, samples related to matters for litigation, etc.) will be disposed of after 30 days, or held longer at the customer's request. The SC/C maintains a record of when samples are disposed and those held beyond the normal holding time.

While knowledge of hazardous waste handling and disposal is the primary responsibility of the Chemical Hygiene Officer, each SRCSD EL employee must know the chemicals used and present in their respective work areas and be familiar with basic policies and procedures for waste disposal as it pertains to his/her work area.

SECTION 6. CALIBRATION, PROCEDURES AND FREQUENCY

Calibration is the establishment of a quantitative relationship of the measurement system and the reliability of the reportable concentration of the material measured. The calibration procedure is performed at a prescribed frequency dictated by the method, and with a defined number of standard concentrations covering the entire working range of the method, whether it is single or multiple point calibrations. Calibration is of primary importance because it is the means by which instrument responses are translated into concentrations of materials present in samples.

Instrument calibration is performed before the actual sample analysis begins and during the course of sample analyses at intervals specified in the method and quality assurance plan. The calibration helps to ensure that data quality objectives (DQOs) established for a program, study, or project are met. If method specific or program calibration criteria are not met, a new calibration curve must be generated prior to the analysis of the samples to be tested. Acceptance criteria for all calibration procedures are specified in the individual methods and in the quality assurance project plans.

All instruments are calibrated and maintained according to the required EPA method protocols and manufacturers operations and maintenance guidelines. The analytical methods outline the basic calibration requirements for the analyses. All maintenance calibration and service information is kept in a bound and numbered page notebook assigned for each instrument.

Organic instrumentation is calibrated with internal standards. Some instruments, because of the complex nature of the multi-peak chromatograms produced by the method, necessitate the use of external standards. Surrogate compounds are included in the calibration processes for most organic analyses as well. Once the analytical method operating conditions have been established, the instrument is calibrated over its linear quantitation range with a number of points for the calibration curve. This curve is verified daily or after the completion of an analytical batch, which ever is more frequent. Individual methods may require additional calibration procedures.

Calibration for the majority of inorganic instrumentation employs the use of external standards. The establishment and verification of the calibration curve for inorganic instrumentation is much the same as for organic analyses. However, the EPA requires minimum-point calibrations for inorganic analytes measured by laboratory instrumentation.

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SECTION 7. ANALYTICAL AND SAMPLING PROCEDURES

As mentioned in Section 3, the SRCSD EL develops and follows standard operating procedures (SOPs) for all routinely used sampling or analytical laboratory methods. Generally, simply citing a published method is inadequate for a SOP. Published methods rarely have all the procedural details and usually are not totally applicable to the way in which tests are actually conducted in the SRCSD EL. Therefore, analytical and sampling SOPs are in place specifically for those activities routinely performed by laboratory employees.

Section Supervisors review, maintain, and update their respective SOPs. SOPs preferably originate from the analyst who performs the procedure. Before implementation, each SOP is reviewed, and approved, by the Section Supervisors and the QAO. Each time a new method is introduced into the Laboratory the respective Section Supervisor shall insure that all initial demonstrations of capabilities are properly performed, that the QAO has reviewed the technical data for the method implementation and finally, that the SOP is properly written and placed into the inventory of active SOPs. The effective date of the SOP shall be the date the last signature (Laboratory Supervisor) is placed on the cover sheet for the SOP. SOPs that are no longer used are to be maintained in a hard copy form for a period of three years by the specific Section for which the SOP was used.

Only the respective Section Supervisor(s) and QAO may make changes to existing SOPs. The Supervisor is responsible for insuring that SOPs are reviewed in detail if substantive changes are made to the existing procedures. Each review shall be approved and documented by the Supervisor and the QAO.

Each SOP documents the method in detail. Any deviation from the SOP must be coordinated and approved by the Section Supervisor prior to incorporating the change for a particular sample or project. Deviations will be case specific and not to be considered permanent. The rationale for a substantial deviation to an SOP shall be clearly documented in the project or program report.

7.1. ANALYTICAL PROCEDURES

7.1.1. SOP Format -- As a minimum, the following sections are included as part of the SOPs

- Scope and Applications
- Detection Limits
- Precision and Bias
- Working Range
- Summary of Method
- Definitions
- Sample Collection, Preservation, and Holding Times
- Interferences and Hints

- Safety Issues
- Instrumentation/Equipment
- Reagents and Standards
- Procedure
- QA/QC Requirements
- Calculations
- Reporting
- References

- 7.1.2. Analytical SOPs are titled by the EPA method numbers (including their revision date) when applicable. For methods that do not have an EPA number, SOPs are titled with the method number and/or title as appropriate to best describe the origin of the method (e.g., SM 4500-P, 18th Ed., 1992).

7.2. SAMPLING PROCEDURES

All samples collected by SRCSD EL staff will incorporate techniques consistent with the parameter(s) being determined. Sampling SOPs that are in place, or new ones being developed, describe standard practices and procedures used by SRCSD EL personnel for field operations and field sampling activities. Sampling SOPs are designed to ensure the collection of representative samples in a manner consistent with standard sampling and handling techniques. Sampling is conducted with the expectation that sample data and site information may be used for regulatory and/or enforcement purposes. Therefore, correct use and application of proper sampling procedures is essential.

Sampling SOPs exist for routine sampling activities in which SRCSD EL employees are responsible to collect those samples. Field or Plant sampling includes taking grab samples or setting up and collecting composite samples from autosamplers at customer prescribed locations defined in program plans. Sampling procedures are followed that a) ensure the safety of the sampler, b) provide enough volume to meet the analytical needs, c) minimize the possibility of sample contamination by either the sample collector or sampling device, and d) meet standards for preservation and holding times. Field sample collection procedures and record keeping requirements shall be detailed for each particular type of routine field sampling activity. Any unusual conditions experienced at the sampling location are noted in field sampling records (internally identified as the “Routing Lists”, see Sect. 8.1) and are documented in the LIMS.

SECTION 8. DATA HANDLING AND REPORTING

This section describes part of the Quality Assurance process that leads to reportable results. Presented at the end of this section is a process flow chart (Figure 2) that graphically illustrates how sample information is derived and reported. The process is detailed in the following paragraphs.

8.1. ACQUISITION OF DATA

Laboratory analytical data is generated at the workbench, or in the field. Analytical related data is either recorded on worksheets by analysts at the test station or derived from instrument printouts. Hardcopy worksheets, field records, and reports, are currently saved by the Laboratory in boxed files and held for a minimum of five years in a secure storage facility.

Analytical station worksheets for routine samples are generated electronically by LIMS after the samples are logged in at the receiving stage. Sampling site information, such as collection time, or unusual conditions, are documented on laboratory Routing Lists by laboratory staff when it is applicable. A Routing List is a pre-printed computer document from the LIMS that directs the laboratory sample collector to defined locations and serves as an internal Chain-Of-Custody record. An example of a Routing List is presented in Appendix 7.

Original handwritten laboratory worksheets and field records are written in blue, waterproof ink. The analyst or data recorder has the primary responsibility for the correctness and completeness of the data. Any corrections to worksheets or field records are noted by a single pen strike through the incorrect data so it remains legible. The correction is inserted near the strike-out and the initials of the corrector are added including the date the correction was made.

Each laboratory employee has responsibility for the quality of their work. They each must assess their work to ensure that:

- Sample preparation information is correct and complete.
- Analytical information is correct and complete.
- The appropriate SOP was followed.
- QC results meet acceptance criteria.
- Blanks are within established control limits.
- Analytical and preparation holding times are met.
- Documentation and recording of data is correct and complete.

8.2. REJECTION OF DATA

When one (or possibly more) of a set of results appears to differ unreasonably from the others in the data set, the measurement(s) is/are called an **outlier(s)**. The suspect result(s) should not be arbitrarily rejected as being “bad” because, in the absence of an investigation to determine the cause, the measurement may be legitimate. If a legitimate measurement is rejected, then bias is introduced, and the mean, while assumed to be correct, is damaged. If a cause is found, then the measurement should be rejected and reason(s) for the rejection documented. If cause is not found, it is advisable to repeat the analysis to verify if the anomaly repeats itself. If it does repeat itself and no cause is found, the situation is not resolved and further review is warranted.

For small data sets ($n < 10$), a simple method to determine if a suspect outlier is rejectable is to apply the Dixon’s Q test. The value for Q is calculated using the formula

$$Q = \frac{(\text{Suspect Value} - \text{Nearest Value})}{\text{Largest Value} - \text{Smallest Value}}$$

The ratio of these differences (without regard to sign) is known as Dixon’s Q. If the calculated Q is greater than the value from the table below, then the suspect value can be rejected and the mean is recalculated. If the calculated Q value is less, then the calculated mean should be reported. A list of Q values for the 95% confidence level is given below in Table 1.

Table 1. Critical values of Q at the 95% ($P=0.05$) confidence level for different numbers of measurements.

Sample Size	Critical Value (Q)
4	0.831
5	0.717
6	0.621
7	0.570
8	0.524
9	0.492
10	0.464

From E. P. King, *J. Am. Statist. Assoc.*, 1958, **48**, 531.

8.3. REDUCTION OF DATA

Essential analytical and field records are to be entered into the LIMS by the analysts and field technicians either manually and/or electronically. Electronically stored laboratory data includes:

- Reportable results of measurements recorded on worksheets and entered directly into LIMS,
- Data directly downloaded into the LIMS from instrument interfaces into the Plant server network, and

- Raw data that LIMS is programmed to calculate into reportable results. All electronically stored information is backed up daily on the Plant's network computer.

Direct access to LIMS is secure and restricted to authorized and licensed users. Any changes to validated data entries into LIMS must be approved by staff with appropriate authority and are tracked via an electronic audit trail.

The primary analyst shall, to the best of his/her ability, correct all mistakes and resolve all questionable issues before the results are subjected to validation. The review should at a minimum check to see that:

- All required documentation is included with the raw data,
- Proper QC protocols were followed,
- Documentation of any excursions from analysis requirements (e.g., QC acceptability, method, etc.),
- Check for math errors.

8.4. VALIDATION OF DATA

Data validation is the process in which data are checked and accepted or rejected based on a given set of criteria. As applied to the SRCSD EL, validating information consists of review of worksheets by lead analysts or supervisors to ensure that:

- Calibration data are appropriate to the method and completely documented.
- QC testing falls within the established guidelines.
- Qualitative identification of sample components is correct.
- Quantitative results are correct.
- Documentation is complete and correct.
- The data are ready for incorporation into the final report.
- The data package is complete and ready for data archiving.

Results are "validated" in the electronic LIMS as an approval step after the reviewer has completed all of the checks and is satisfied that internal laboratory acceptance criteria have been met. If errors are found with the documentation, the supervisor or lead person reviews them with the analyst who in turn is instructed and retrained, if necessary, to ensure that the problem has been corrected. If the error is systematic and thus accounted for, corrections are made where needed.

8.5. EXACT AND EXPERIMENTAL NUMBERS

Data used for laboratory calculations involve the use of exact and/or measured numbers. Exact numbers are those whose values are known without question. Exact numbers include result from fundamental definition of quantities or from counting objects. For example, 1 L

= 1000 ml; the 1000 is an exact number because its exact value is known. Exact numbers have an infinite number of significant figures.

Numbers that are experimentally measured are not exact because of small errors or uncertainties. All measured values derived from laboratory instruments and techniques have limited accuracy and uncertainty. The degree of uncertainty is indicated by the use of significant figures in the measured values. This is done by recording all digits that are known with certainty, and then adding an extra digit that has an uncertainty.

8.6. SIGNIFICANT FIGURES

Any figure that is necessary to define the specific value or quantity is said to be significant. When measured to the nearest 0.1 mg, a concentration may be recorded as 15.7 mg/L; this number has three significant figures. Reported analytical values should contain only significant figures. A value is made up of significant figures when it contains all digits known to be true and one last digit in doubt. The position of the decimal point is insignificant. Thus, 0.012345, 1.2345, 123.45, and 12,345 all contain five significant figures.

If there are no nonzero digits preceding a decimal point, the zeros after the decimal point but preceding other nonzero digits are not significant. These zeros only indicate the position of the decimal point. For example, 0.0008 gm contains one significant digit.

Zeros bounded by digits only on the left may or may not be significant. If the zeros are part of the number, they are significant. For example when mass is expressed as 0.0200 gm, the weight is known to three significant figures. If we express the volume of a 2L beaker as 2000 ml, the latter number will contain only one significant figure: the zeros simply indicate the order of magnitude. The identification of significant digits is only possible through knowledge of the circumstances.

A distinction must be made between those figures that have physical significance and those that are unknown or meaningless. The following rules apply in this regard:

- In any number, the digits 1 through 9 are all significant. Thus the numbers 456 and 0.45678 have three and five significant figures, respectively.
- The significant figures in a number comprise all those digits whose values are known with certainty plus the first digit whose value is uncertain. The position of the decimal is irrelevant. Thus 0.012345 and 123.45 contain five significant figures.
- Zeros are significant when they are part of the number; they are not significant when employed to indicate order of magnitude. Zeros bounded left and right by digits other than zero are always significant. Thus 21.03 and 20.03 each contain four significant figures.

In considering calculations involving the manipulation of numerical quantities that contain varying numbers of significant quantities the answer can be no more accurate than the least accurate term involved in the calculation. The following rules regarding significant figures in derived results are as follows:

- If a result is to be used for further calculations, retain one or more significant digits than is necessary. If the result is final, round it to the number of significant figures as described in Section 8.7.
- In any calculation involving multiplication or division of numbers that have been obtained as a result of a physical measurement, the answer can be no more accurate than the least accurate of these numbers.
- In operations involving only addition and subtraction, the answer can have no more decimal terms than are present in the number with the fewest decimal terms. Thus $142.7 + 0.081$ would be reported as 142.8.
- Evaluation of the number of significant figures to be shown in the result of a multistep calculation is accomplished by first considering any sums or differences and then the products or quotients.

8.7. ROUNDING-OFF OF NUMBERS

Rounding off of numbers is necessary in analytical areas. The number of significant figures given indicates the precision of the measurement. Specific rules must be observed when data are to be *added, subtracted, multiplied, or divided*.

- Rounding off numbers:

Round off by dropping digits that are not significant. If the digit 6, 7, 8, or 9 is dropped, increase the preceding digit by one unit. If the digit 0, 1, 2, 3, or 4 is dropped, do not alter the preceding digit. If the digit 5 is dropped, round off preceding digit to the nearest even number: thus 2.25 become 2.2 and 2.35 become 2.4.

- Rule for rounding-off in addition and subtraction operations:

The rule for addition and subtraction is that the answer shall contain no significant digits farther to the right than occurs in the least precise number. Rounding is done afterward.

- Rule for rounding-off in multiplication and division operations:

When two numbers are to be multiplied or divided, all digits are carried through the operation. Then the answer is rounded off to the number in the operation having the least significant figures.

The following multiplication operation illustrates this rule:

$$113.2 \times 1.43 = 161.876$$

is rounded to 162, since 1.43 contains only three significant digits

A good general rule is to keep one digit beyond the last significant figure and leave further rounding until the final result is reached. The same applies when the mean and standard deviation are used to apply a statistical test.

8.8. REPORTING OF DATA

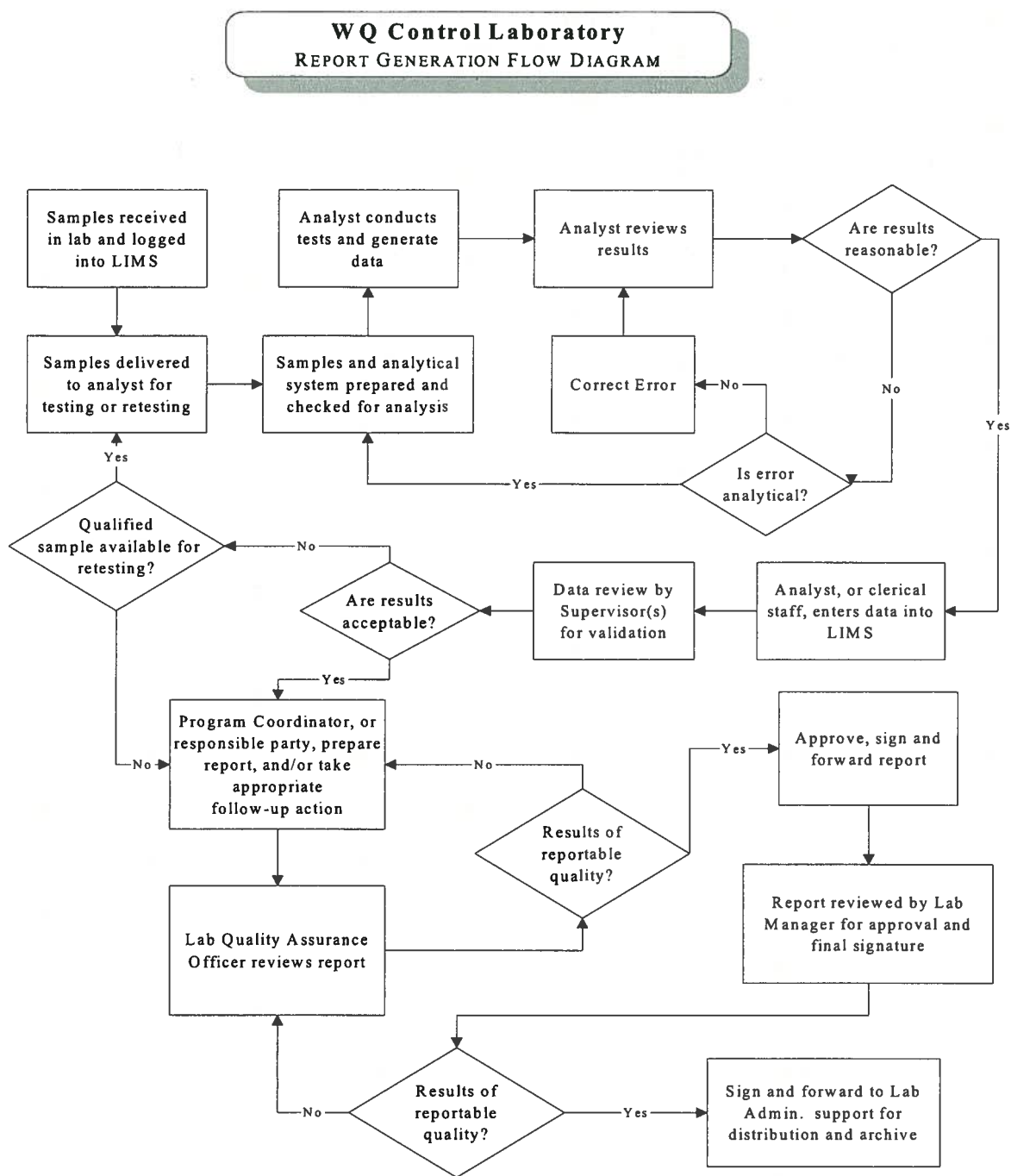
Samples that are analyzed as replicate (usually in duplicate) will have the result reported as an average. If samples are less than the detection or report limit (RL) but above the method detection limit (MDL) the results, by the LIMS default, are reported as <RL (less than the reporting limit). The results can also be manually entered as non detect (ND) or as DNQ, “detected but not quantified”, depending on the customer reporting requirements. If results are less than the MDL they will always be reported as <RL. Reports of analytical results will contain as a minimum the RLs for the parameters, reporting units, date of sample, date analyzed, method used, the laboratory numbers for the samples analyzed, and associated QC data.

Results for sample analyses are reported through LIMS or as a custom report prepared in a computer word processing program such as Microsoft Word. Currently, no laboratory customers are licensed users and thus data is not directly available to them through the LIMS. Reports can be sent to customers electronically as e-mail files, PDF files, and by hardcopy reports.

All official hardcopy reports must have three formal reviews and approval signatures. Reviews are steps taken by designated laboratory staff to assure the reported data is complete and correct, meets the program objectives, and conforms to laboratory standards. Approval signatures consist of the person responsible for reporting the data (usually the Program Coordinator or a designated responsible employee), the SRCSD EL’s Quality Assurance Officer, and the Laboratory Manager. Approved hardcopy laboratory reports are distributed to customer(s) by laboratory clerical support staff. Reports retained by the laboratory (originals or copies) are date stamped by the clerical staff when they are distributed.

Protocol for sending out or keeping the original report is dependent on the following conditions: If there is a single addressee for the report, the receiver is sent the original copy and the SRCSD EL keeps a copy on file. If there are multiple addressees, copies of the reports are sent and the SRCSD EL keeps the original copy of the report on file. All those designated for CCs on reports will receive photocopies of the original document with copies of attachments, if that is the case.

Figure 2. Data review and report generation flow diagram.



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SECTION 9. INTERNAL AND EXTERNAL QUALITY CONTROL CHECKS

9.1. APPLICATION OF CONTROL CHECKS

Quality control results are used for making decisions about the acceptability of analyses. The SRCSD EL's QA program gives guidance for accepting, rejecting, or flagging results. The guidelines include, but are not limited to acceptance limits based on method requirements, or historical data, or guidelines developed in-house for the area of interest, and method specific guidelines. Method guidance takes precedence.

Internal QA checks are applied to analytical procedures to answer the following:

- Are laboratory operations “in control” (i.e., operating within acceptable QC guidelines) during data generation?
- What affect does the sample matrix have on the data being generated?
- What affect do field conditions have on the analytical result?

The first question is answered by laboratory performance QC. Laboratory performance QC is based on the use of standards to generate precision and accuracy data that are compared, on a per use basis, to control limits. This information, in conjunction with method blank data, is used to assess the laboratory test performance.

The answer to the second question can be addressed by matrix specific QC. Matrix specific QC is based on the use of an actual environmental sample fortified with a known amount of the analyte(s) or compound(s) of interest.

The third question is addressed with the use of field QC samples. These samples include field blanks, trip blanks, equipment blanks, field duplicates, and field splits. They are used to evaluate the degree of contamination from the collection, transport, and storage of environmental samples.

9.2. TYPES OF INTERNAL QUALITY CONTROL SAMPLES

Internal QC is a way for our laboratory to assess the analytical measurement system and check whether it is in control. The analytical procedures will dictate the internal QC applied and varies with individual test methods. For internal QC, the following types of QC samples can be applied to each analytical batch or routine operation:

- 9.2.1. **Laboratory Blanks** – Laboratory blanks are prepared using laboratory reagent water and are treated exactly as a sample, including exposure to all glassware, equipment, solvents and reagent used with other samples. The laboratory blank is used to determine if the method analytes or other interferences are present in the lab environment, reagents, or equipment.

If contaminants are present that interfere with the determination of any analyte, detection limits must be elevated or affected compounds must be qualified accordingly. If a blank is contaminated, then the source of the contamination must be identified and eliminated.

9.2.2. **Laboratory Control Sample (LCS)** – A material of known composition (similar to the samples analyzed) used expressly for the purpose of monitoring a measurement process. They consist of a portion of analyte-free water or solid phase sample that is spiked with target analytes at a known concentration. The LCS is processed through the entire method procedure and the results examined for target analyte recovery. Precision evaluations can be determined by performing the LCS in duplicate (LCSD). The LCS and SCSD can be used in cases where the matrix spike and matrix spike duplicate have failed to achieve the acceptable recovery or precision.

9.2.3. **Initial Calibration Curve** (for Instrument Analysis) – A standard curve with concentrations bracketing the range of interest must be performed prior to sample analysis. Specific requirements are method dependent and discussed in detail in the method Standard Operating Procedures (SOPs).

9.2.4. **Initial and Continuing Calibration Verification (ICV & CCV)** – Standards for Instrument Analysis – An initial calibration verification (ICV) is performed to check the accuracy of the calibration curve immediately after it is prepared. The ICV comes from a different source than the initial calibration standard. Normally the ICV concentration obtained from the calibration curve should agree within $\pm 5\%$ of the true value of the ICV solution. Failure to obtain acceptable ICV results may be due to:

- Concentrations of either the calibration standard(s) or ICV solution are not accurate.
- There is a problem with the calculation of the calibration curve.
- The instrument developed a problem between the completion of the calibration and the analysis of the ICV.
- An instrument problem which existed during the calibration was corrected prior to the ICV analysis.
- The analysis of the ICV was botched.

The continuing calibration verification (CCV) is used to determine that the initial calibration is holding. The source of the CCV should be one of the calibration standards, the initial calibration standard at a different concentration other than that of the initial calibration, or from an independent source. The minimum frequency for the CCV will be specified by the method but at least once per use.

9.2.5. **Matrix Spike (MS) and Matrix Spike Duplicate (MSD)** – A MS or MSD are aliquots of environmental samples that are fortified in the laboratory with a

known amount of analyte(s) of interest. They are analyzed exactly like a sample. They are used to assess method bias and, when performed in duplicate, measure precision on “real” samples in the presence of matrix effects. Spike recoveries are used as indicators for potential sample matrix interference and may be used to monitor analyte losses or other matrix effects. Historical percent recoveries are used to calculate method bias and the confidence range of the bias. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS and MSD corrected for these background concentrations.

- 9.2.6. **Laboratory Duplicate Sample** – A laboratory duplicate sample is prepared with every sample batch to assess precision for each matrix type. The precision of the duplicate sample is reported as relative percent difference (RPD). RPD acceptance criteria are method specific. Duplicate samples that fall outside of the acceptance criteria must be reanalyzed or qualified if required.
- 9.2.7. **Internal Standards** – Pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.
- 9.2.8. **Surrogates** (for organic analysis) – Surrogates are organic compounds similar to the analytes of interest in chemical behavior, but not normally found in environmental samples. Surrogates are compounds added to samples to monitor method accuracy and check for gross inaccuracy (e.g., incorrect volumes, concentration problems, etc.) in all of the samples. Results are reported in terms of percent recovery

9.3. TYPES OF EXTERNAL QUALITY CONTROL SAMPLES

In addition to internal laboratory quality control samples, there are also field and proficiency quality control samples that are evaluated. External quality control samples are generally specified in sampling plans by outside sources for the purpose of assessing sample contamination or laboratory proficiency. External QC samples include:

- 9.3.1. **Field Duplicate Samples** – Two separate samples collected at the same time and from the same site under identical conditions and treated exactly the same through all field and laboratory procedures. Duplicate analysis gives a measure of the variability in the sample collection, shipment, storage, and analysis. Wide variations in the set of target analytes reported from the samples is a cause for concern.

- 9.3.2. **Field Blank Samples** – A reagent water blank prepared at the sample site by filling empty sample containers with analyte-free water, adding preservatives (if applicable) and then taking the samples to the lab for analysis. The samples are treated as regular samples including exposure to the sample site conditions, storage, preservation and lab procedures. It is used to determine if method analytes and/or other contaminants are present in the field sampling environment.
- 9.3.3. **Equipment Blank Sample** – A blank sample used to monitor the effectiveness of the cleaning procedures used on field sampling equipment. Equipment blanks are prepared by taking a quantity of analyte-free water to the sample site, rinse the piece of equipment with the analyte-free water directly into the sample container, add preservatives and then treat as a regular sample. At least one equipment blank is prepared for each type of analyte group collected with each item of equipment.
- 9.3.4. **Trip Blank Sample** – A sample container that is filled in the laboratory with reagent-free water that accompanies the other associated sample bottles to the sample collection site and back to the laboratory in the same containers as used for the other samples. The bottles remain closed and analyzed as regular samples when returned to the laboratory. These samples are used to monitor for possible airborne contamination due to volatile organic analytes capable of diffusing across septa in volatile organic sample vials. Used primary for VOC sampling.
- 9.3.5. **Field Spikes** – Fortified samples used to assess the stability and method performance of the analytes. These may be “blind” samples that are unlabelled as spikes and are therefore unknown to the laboratory personnel. This may be useful in determining true method performance.
- 9.3.6. **Proficiency Evaluation (PE)** – A fortified sample used to evaluate the performance of the laboratory. PE samples consist of solutions of known concentrations of target analytes sent to the laboratory to be analyzed as unknown. Based on either statistically derived or legislatively assigned acceptance criteria, the results are graded as “acceptable” or “non-acceptable”. Participation in PE sample studies provides a means by which the laboratory can discover analytical problems and improve performance. Samples may be used to evaluate an analyst or laboratory group. (See following Section 10.)

SECTION 10. PERFORMANCE AND SYSTEM QUALITY AUDITS

10.1. ABOUT QUALITY AUDITS

The laboratory's QA program is a management tool designed to examine and track overall performance, evaluate it in detail, and disclose the cause(s) of unsatisfactory conditions. Quality audits are an integral part of the QA program used to:

**IDENTIFY PROBLEMS THAT ARISE AS A RESULT OF ANY CUSTOMER
DISCOVERED ERRORS AND/OR DISCREPANT RESULTS
FROM THE ANALYSIS OF TEST DATA.
EVALUATE EVIDENCE FROM INTERNAL AUDITS (CONDUCTED
TRIANNUALLY) AND STATISTICAL CONTROL DATA.**

- Evaluate evidence from external audits (on-site assessments), participate in measurement assurance programs, proficiency testing for laboratory certification, round-robins, inter-laboratory collaborative studies, or calibrations.

10.2. AUDIT ADMINISTRATION

Audits are administered on either a mandatory or voluntary basis.

10.2.1. Mandatory audits

Mandatory Audits are driven by laws and/or regulations and administered through governmental agencies to ensure the laboratory's ability to meet minimum standards when reporting analytical data that is required under mandated monitoring programs. These audits assess performance evaluations and on-site systems inspections. Regulatory audits are conducted by the State of California's Department of Public Health Services (DPH) under their Environmental Laboratory Accreditation Program (ELAP) on a scheduled basis. The audits serve to assess the laboratory's overall status for maintaining its certification. A list of tests the SRCSD EL is approved for under ELAP is presented in Appendix 5 (*Laboratory Certification*). Failure to meet the minimum requirements can result in a downgrading or loss of the laboratory's certification. The audit records and resulting corrective action reports are administered by the Laboratory's QAO, and maintained in laboratory files.

10.2.2. Voluntary audits

Voluntary audits are internal checks administered by the laboratory to self-assess performance and correct problems identified through a systematic review conducted by the QAO and/or section staff.

10.3. TYPES OF AUDITS

Audits fall into two general categories, performance and system audits. In both, the appropriate implementation of corrective action is assured to effect permanent solutions to problems that are detected. Corrective action is discussed in more detail in Section 13.

10.3.1. Performance Audits

A performance audit verifies the laboratory's ability to correctly identify and quantify substances in samples. It involves the analysis of a sample or reference material and comparing the results with either the results of other laboratories or with the true answer, or both. Performance audits include those required for certification and those routinely conducted on a voluntary basis as an internal check on performance to demonstrate competency. The QAO manages the activity and reports results to the referee laboratory.

The laboratory voluntarily conducts its own performance audits. The voluntary evaluation program implemented at the SRCSO EL involves the following activities:

- Internal laboratory quality control samples are analyzed at a frequency of once per batch, and no less than 5% of the number of samples analyzed (also see Section 9).
- Blind check samples provided from an independent commercial vendor are sent to the laboratory on a regular schedule, about once every four months or more frequent if warranted. The check samples include material tested for organic and inorganic analytes. Test results are compared to the true values and must fall within acceptance limits. For those samples that fall outside of acceptance limits, corrective action must be taken to identify where the problem lies and then subsequently fixed.

10.3.2. System Audits

A system audit includes examining all aspects of the laboratory. System audits include assessments about, but not necessarily limited to, the following:

- Personnel – Education, training and experience.
- Physical aspects of the laboratory – Examine adequate separation of activities, cleanliness, orderly, waste disposal operations.
- Standard Operating Procedures (SOPs) – Assess whether current and complete. Check text for technical errors.
- Equipment/Instruments used in the laboratory – Check if equipment/instruments are clean, well maintained and regularly inspected as evidenced by equipment/instrument log books. Check that calibration is done correctly, properly documented, and whether documentation is done in blue, waterproof ink.
- Test substances, Reagents, and Samples – Check notebooks, or logbooks to see if substances are properly identified, look at container labels for proper identification (identify material, concentration, composition, storage requirements, expiration date, and initials of person making up material) for sample.

- Chain of Custody (COC) – Look at procedures, documentation, and records management.
- The laboratory information management system (LIMS) – Inspect records and raw data, including notebooks, computer printouts, chromatograms, worksheets, COC sheets, protocol, and SOPs to confirm work was carried out according to customer requirements and in accordance with the prescribed methods.
- Random check of analytical values reported against numbers in the worksheets and electronic database.
- Review laboratory records to verify that sample holding times were met, calibration checks adequate, equipment monitoring records are performed as required (e.g., temperature records), and sample preservation records are maintained and correct.
- Corrective action documentation complete and current.

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SECTION 11. PREVENTIVE MAINTENANCE

To minimize downtime and interruption of analytical work, maximize longevity of the useful life of laboratory equipment and instruments, provide for reliable analyses and increase data completeness, the SRCSD EL uses a preventive maintenance approach for its equipment and instruments. Preventive maintenance is an orderly program of positive actions such as routine or scheduled equipment and instrument inspections, cleaning, lubricating, reconditioning, adjustment, timely replacement of consumables, or testing, to prevent instruments or equipment from failing during use.

Outside of scheduled maintenance needs, analysts are trained to recognize problems and expected to request equipment maintenance and repairs provided by in-house staff or vendor technicians. To this end, maintenance and service contracts are provided for major equipment and instruments used in the laboratory.

Each of the major pieces of equipment and instruments have manufacturer maintenance and operations manuals available on them to keep equipment and instruments running at optimum conditions. The maintenance manuals often provide preventive maintenance schedules and troubleshooting guides.

Laboratory instrument workstations have log books at each site so that the instrument or equipment operators can document the maintenance and operational history including what happened, when, and who, performed the maintenance and repairs. As a minimum, the instrument logs will contain a record of the routine performance check results and maintenance done on the instrument, as well as a record of the day-to-day use of the instrument. This instrument log book is clearly marked to show the instrument identification and kept near the instrument.

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SECTION 12. ASSESSMENT OF PRECISION AND ACCURACY

The effectiveness of the SRCSD EL QA Program is measured by the quality of the analytical data generated by the laboratory. Data quality information is obtained with the use of statistical tools. Statistics is the art of presenting data in a meaningful way. The quality is often judged in terms of its precision, accuracy, bias, completeness and comparability. These terms and their application are described below.

12.1. MEASURES OF CENTRAL TENDENCY

It is often useful to calculate a single value that will represent a body of data. Such a value is often referred to as a *measure of central tendency*. The arithmetic **mean (or average)**, the **median**, and the **mode** are such measures.

- 12.1.1. **Mean** – The most common measure of central tendency is the arithmetic mean or average (\bar{x}). It is the sum of the individual values in a set of numbers divided by the number of values calculated by the formula:

$$\bar{x} = \frac{\sum_n x}{n}$$

The average of several values should be calculated with at least one more figure than that of the data.

- 12.1.2. **Median** – The median is the middle value of a ranked set of data and is often a more meaningful location parameter than the mean. If there is an even number of values in the data set, the median is the arithmetic mean of the two middle values.
- 12.1.3. **Mode** – The value, or values, occurring most frequently in a sample of data is the mode, although it has little practical importance. Some data will have more than one mode. This distribution is called bimodal. In a symmetrical frequency curve the mode and median are equal.

12.2. BIAS

Bias measures systematic errors. Bias can be removed, once identified, by checks on equipment and experimental techniques. It occurs over and over again due to some fault of the measurement and thus cannot be averaged out by making more measurements. When measurements are bunched, not around the correct answer, they are precise and not accurate, and so a bias is indicated.

12.3. PRECISION

Precision is the degree to which a measurement is reproducible under the same conditions. It measures the extent of scatter in repeated measurements. The closer the results of the measurement are together, the greater is the precision. The standard deviation, s , is often used as an index of precision. When s is large, the measurements are imprecise. Making replicate measurements provides a means to quantify the measurement errors and evaluate their importance. The ability to duplicate results should not be construed as adequacy of the method or the analyst because a bad result can be duplicated just as easily as a good one.

Precision has no relation to accuracy. Precision relates to the random errors inherent in the analysis and can be impacted by sample inconsistency. It is normally calculated on spiked samples such as the matrix spike (MS) and a matrix spike duplicate (MSD), or as a laboratory control sample (LCS) and a laboratory control sample duplicate (LCSD). Differences can be expressed as follows:

12.2.1. Duplicate Analysis

Duplicates are performed on duplicate aliquots of actual samples, matrix spikes, or laboratory control samples. Results of laboratory duplicate samples are often reported as the average value. Comparison of the two measurements (duplicates) from two aliquots of the same sample for precision is expressed as the Relative Percent Difference (RPD). RPD is calculated as the absolute difference between the two measurements divided by their average. The decimal fraction is multiplied by 100 to convert it to a percent.

$$RPD = \frac{D}{X} \times 100$$

Acceptance limits for the RPD are calculated based on data generated on a minimum of 20 results from samples of the same matrix. The limits for RPD are the average RPD plus three times the standard deviation.

12.2.2. Deviation

Deviation is how much a measurement differs from the mean. A deviation is associated with each measurement, and if large compared to others in a series of identical measurements, may indicate a rejectable measurement that can be tested by statistical methods. Mathematically, deviation is calculated by the following equation:

$$d = \bar{x} - e$$

in which d is the deviation, \bar{x} is the mean, and e represents the individual experimental measurement.

12.2.3. Variance, Sample and Population

Sample variance is the sum of the squares of the differences between the individual values (x_i) of a set and the arithmetic mean of the set, divided by one less than the number of values. The divisor ($n - 1$) is used instead of n , so that the value of s^2 , the sample variance, is an unbiased estimate of the population variance and usually applied when measurements ≤ 20 .

$$s^2 = \frac{\sum (x_i - \bar{x})^2}{n - 1}$$

Population variance is used when the population is very large ($n > 20$) and is denoted by σ^2 . The variance of a population is the mean (\bar{x}) value of the square of such deviation taken over the whole population. It is a measure of how far any particular observation is from the mean and is calculated using the formula:

$$\sigma^2 = \frac{\sum (x_i - \bar{x})^2}{n}$$

12.2.4. Standard Deviation

Variability is a range or distribution of results around a mean value obtained from samples within a population. The standard deviation is the most common measure of variability. It is the square root of the ‘variance’ and it has the same units as the original measurements and as the mean. The significance of the **standard deviation** is that the smaller it is numerically, the more precise the data and thus presumably (if free from bias and determinate error) the more accurate the data. The standard deviation should be computed to at least three significant figures.

The **population standard deviation** is designated by the Greek character sigma (σ), and the **sample standard deviation**, which gives an estimate of σ , is denoted by s . It is the most common measure of the dispersion around the mean.

The population standard deviation, σ , applies if the data points are large (>20). It is a measure of spread of a large group of data and has the same units as the original measurements (x_i) and the mean (\bar{x}). It is calculated by the following equation:

$$\sigma = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n}}$$

For replicate analysis (any number >2) the equation for standard deviation of the sample, s , is the square root of the sample variance (s^2).

$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n - 1}}$$

In a normal or Gaussian distribution of the population from which the samples are drawn, 99.9% of the data points will fall within plus-or-minus three standard deviations from the mean. On each side of the mean (\bar{x}), the data points will fall as follows:

- ± 1 standard deviation – 68.27%
- ± 2 standard deviations – 95.447%
- ± 3 standard deviations – 99.745%

This can be used to predict what percentage points might fall within or outside of the distribution curve.

12.2.5. Relative Standard Deviation

The **relative standard deviation** (RSD), also known as the coefficient of variation, is the ratio of the standard deviation to the mean. It is a measure of precision when three or more replicates are used for an analysis. To express the fraction as a percentage, the result is multiplied by 100.

$$\% \text{ RSD} = \frac{\text{Standard Deviation, } s}{\text{Average, } \bar{x}} \times 100$$

The RSD relates the standard deviation to the value of the mean and represents a practical expression of data quality, similar to RPD. As an example, two analysts given the same sample have replicate analyses resulting in a %RSD of 3.6% and 10.2% respectively. The analyst with 3.6% RSD has demonstrated a greater degree of precision. Acceptance criteria applied to % RSDs must take into account sample variability and homogeneity. Acceptance criteria are calculated in a similar way to that used for the RPD.

12.2.6. Matrix Spike Duplicates

The **matrix spike duplicates** (MSD) are primarily designed to assess the precision of analytical results in a given matrix. The frequency of the analysis may be determined as part of a study plan, data quality objectives (DQOs) or as specified by the method. Results are compared to established limits for that specific matrix if available. The precision of MSDs is expressed as the relative percent difference (**RPD**) and calculated as:

$$RPD = \frac{2|MSR - MSRD|}{MSR + MSRD} \times 100$$

where: MSR = matrix spike percent recovery (%R)
MSRD = matrix spike duplicate percent recovery (%R)

Acceptance limits for MSDs are calculated based on data obtained from 20 results of samples of the same matrix to establish a “practical” limit. The limits for the RPD are the average of the 20 RPD results plus three times the standard deviation of the RPDs.

12.3. ACCURACY

Accuracy demonstrates the closeness of a result, or the arithmetic mean of a set of results, to the true, expected, or accepted value. A measurement, at best, is always going to be an estimate of the true value. Thus, it is important to note that accuracy in a measured value is a matter of degree rather than an absolute quantity. The reported value does not have to be dead on the mark to be considered ‘accurate’. When we classify a result as ‘accurate’ or ‘inaccurate’ it is determined by the width of pre-established zones of tolerance. The inability to obtain acceptable results for samples of known, certified, analyte concentrations is directly related to the inability to generate acceptable results for any sample.

Accuracy can be assessed using laboratory control samples (LCSs) or standard reference materials (SRMs). LCSs or SRMs can be spiked into environmental samples to measure accuracy. An LCS or SRM consists of a portion of analyte-free water, or solid phase sample spiked with target analytes at known concentrations. Accuracy can be determined through the analysis of an LCS, SRM, or matrix spike (MS). Matrix spike consists of the addition of a known amount of target analyte from an LCS or SRM to a portion of the sample, then analyzing the sample and comparing the result with the true added amount.

Matrix spike recoveries outside the acceptance limits of three standard deviation units from the mean of at least 20 historical matrix spike recovery tests will require corrective action to be taken. Standard Methods¹ suggests the following guidelines when results fall outside of the control chart limits:

- **Control Limit (3s)** – If one measurement exceeds the control limit, repeat the analysis immediately. If the repeat test result is within the control limit, continue the analysis; if it exceeds the control limit, discontinue the analyses and correct the problem.
- **Warning Limit (2s)** – If two out of three measurements exceed the warning limit, analyze another sample. If the next point is less than 2s, continue the

¹ American Public Health Assn., *Standard Methods for the Examination of Water and Wastewater*, 18th Ed., 1992, pg 1-6.

analysis; if the next point exceeds 2s, discontinue analyses and correct the problem.

Percent recovery determinations on spikes from LCSs or reference materials can be calculated and reported using the following formula:

$$\% R = \left(\frac{SSR}{SA} \right) \times 100$$

Where: SSR = spiked sample result
SA = spike amount added

Percent recoveries (%R) for matrix spike compounds are calculated as:

$$\% R = \frac{SSR - SR}{SA} \times 100$$

Where: SSR = spiked sample result
SR = unspiked sample result
SA = spike amount added

12.4. SOURCES OF ERRORS

Measurement systems are subject to error. Measurements of some physical, chemical, or biological characteristic that has some true value will always be estimates. In the statistical sense, error does not imply fault, mistake, or blunder. It refers to variation that is often unavoidable resulting from such factors as measurement fluctuations due to instrument conditioning, sampling imperfections, variation in ambient conditions, analyst skill, etc.

Errors can be classified as two types, systematic (or determinate) and random. The following paragraphs describe the error types in more detail.

- 12.4.1. **Systematic** (Determinate) Errors – Systematic errors have a variety of causes such as defects in the method, poor analyst practices, instrument malfunction, and contamination. These errors affect accuracy. They are usually constant for a given method and can be accounted for or measured.
- 12.4.2. **Random** (Indeterminate) Errors – Random errors are unavoidable and reflected in the unpredictable fluctuations that occur in the use of the method. They affect precision and can be described using statistical procedures.

12.5. COMPLETENESS

Completeness is a measure of the amount of valid data obtained from an analysis compared with the amount that was expected to be obtained. To be considered complete, the data set must contain all analytical results and data specified for the program study. In addition, reported data are compared to program or study requirements to ensure that the specifications were met. Any deviations are noted in the report narrative.

The percent completeness for each set of samples can be calculated as:

$$\% \text{ Completeness} = \left(\frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \right) \times 100$$

Where valid data are determined by the data acceptance criteria defined in the program or study plan.

12.6. REPRESENTATIVENESS

Representativeness is the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Analytical data should represent the conditions of the sample site. If a grab sample is likely not possess all of the characteristics of the site, a series of samples are obtained from different parts, or if temporal variation is a factor, samples might be taken at different times and analyzed separately or as a composite, depending upon the study or program needs and objectives.

12.7. COMPARABILITY

Comparability is the degree to which one data set can be compared to another. Comparability is ensured through the use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), consistency in reporting units (ppm, ppb, mg/L, etc.), and the analysis of LCSs and SRMs.

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SECTION 13. CORRECTIVE ACTION

13.1. CORRECTIVE ACTION OVERVIEW

Corrective action investigates suspect procedures and/or data. Corrective action is intended to prevent the recurrence of similar problems and to promote continuous improvement in the quality of service through training and education. The more formal corrective action process relates to dealing with non-conforming results. The American National Standard (ANSI/ASQC Z1.4-1981 defines “non-conformity” as:

“A departure of a quality characteristic from its intended level, or state that occurs with a severity sufficient to cause an associated product or service not to meet a specification requirement.”

Some type of corrective action, whether formal or not, is required whenever any of the following conditions exist:

- Suspicious results discovered during the testing or analytical operations.
- Suspicious results discovered during the data validating procedures.
- Suspicious results discovered during the internal report review procedures.
- Suspicious results discovered in independent audits.
- Suspicious results originating from customer complaints.
- Suspicious results from equipment or instrument failure.

The analyst at the bench has the primary responsibility for ensuring the quality and acceptability of test results. One of the most effective means of error prevention is to respond immediately to suspicious data or equipment malfunctions from the bench. Taking proper corrective action at this point can reduce or prevent producing erroneous or poor quality data that may have serious consequences in how the information is expected to be used. Specific control procedures, calibration checks, control charts, operational check lists, etc, are in place to detect instances in which corrective action might be necessary.

13.2. KINDS OF CORRECTIVE ACTION

There are basically **two types** of corrective actions available to the laboratory, on-the-spot or immediate, and long-term.

- 13.2.1. **On-the-spot** – Designed to correct or repair non conforming data, equipment, or materials. The analyst or operator has the responsibility for conducting immediate corrective action when the problem has been discovered and cause

identified. This normally precedes any data entry into LIMS, then corrected at the bench. This type of corrective action should be managed as normal operating procedures. This corrective action needs only to be documented in instrument logbooks or noted as applicable in LIMS. No further formal action is needed.

- 13.2.2. **Long term** – This is corrective action taken for (a) recurring problem(s). It is a long term solution to eliminate causes for non-conformance types of errors and requires formal documentation. A sample Corrective Action Form is provided in Appendix 16.6. The long term corrective action process and decision making steps are presented as a flow chart (see Figure 3) at the end of this section. The formal process assures that the appropriate corrective action steps are taken in a timely manner.

13.3. ADMINISTRATION OF LONG-TERM CORRECTIVE ACTION

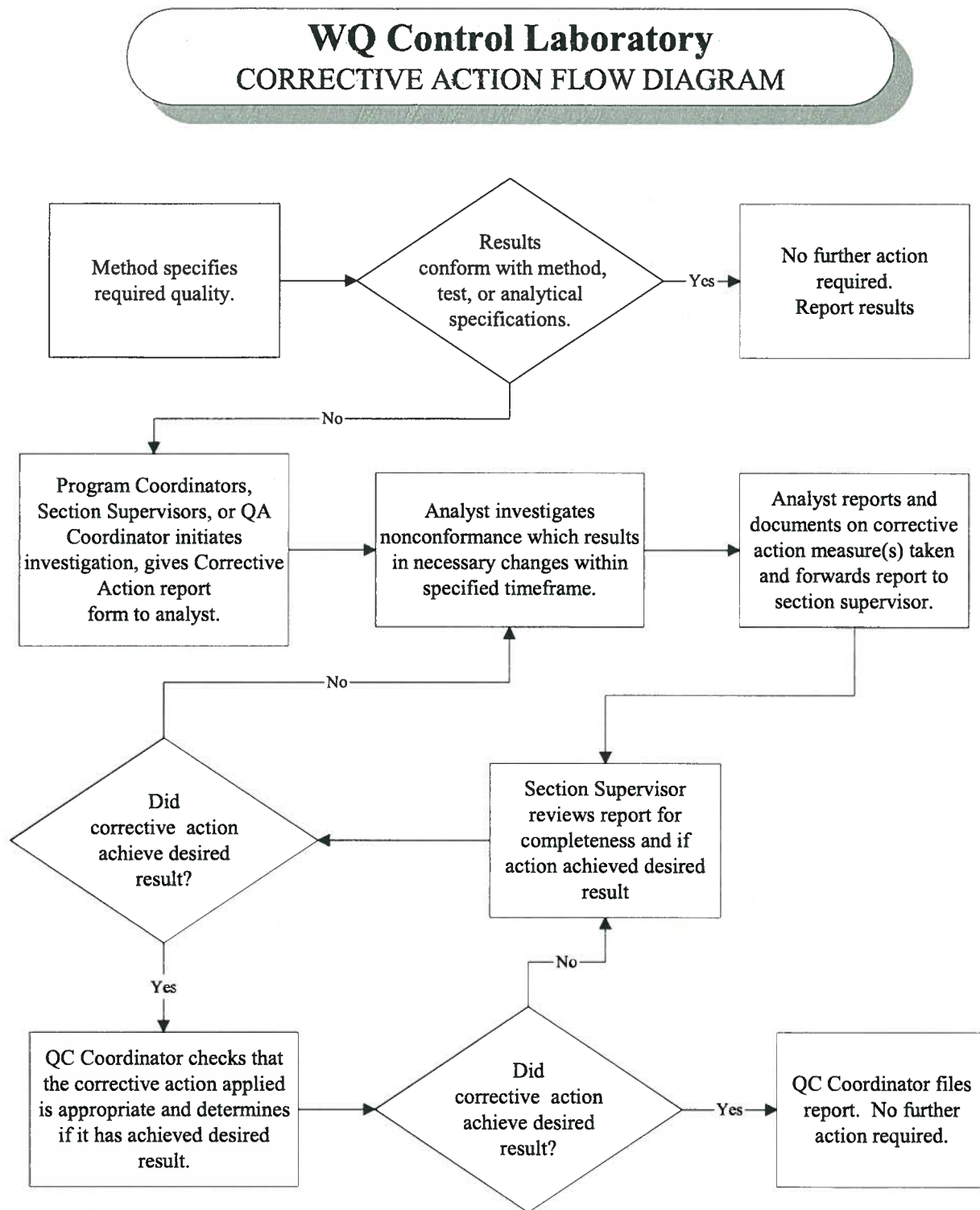
The QAO and/or respective Section Supervisor will formally initiate the investigation of the problem. The goal of the investigation is to determine the cause and develop a strategy to correct the problem and keep it from recurring. Either or both may oversee the implementation for the solution to the problem, verify the correction, and document the effectiveness. The QAO receives the final report and reviews it for approval. If applicable, the QAO will notify the respective Program Coordinator who will contact the customer regarding the outcome of the investigation and provide corrections or other options, if available.

The Corrective Action Report will include details of the investigation including:

- Relevant dates.
- Name(s) of the analyst(s) involved.
- Identification of affected samples.
- Status of equipment.
- Procedures used.
- Statement of problem, cause(s), corrective action(s) taken, and verification of corrective action.
- Complete report initialed and dated by analyst, supervisor, and QAO.

Regardless of who initiates the Corrective Action Report, the QAO is responsible for the maintenance of the Corrective Action program and for any periodic Corrective Action program report required by the Laboratory Manager.

Figure 3. Corrective action flow chart.



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SECTION 14. QUALITY ASSURANCE REPORTS AND RECORDS CONTROL

The quality assurance reporting system is a tool designed to measure the effectiveness of the quality assurance program. It serves to evaluate the program design, identifies problems and trends, and aids in the planning for future needs. In addition to reporting test results to program managers, extensive quality assurance documentation is provided with the reports which serves to support the quality of the reported data. The QAO reports directly to the Laboratory Manager on all matters pertaining to the status of the QA program. The QAO prepares and submits periodic reports to the laboratory manager including:

- Results of on-site audits by regulatory agencies and clients.
- Laboratory responses to audit deficiencies or action items required as a result of an audit by a regulatory agency.
- Performance evaluation sample results and associated corrective action reports.
- Summaries of certification activities including new certifications applied for, certification renewals, any actions taken by certifying agencies (suspensions, deficiency reports, probationary reports, reinstatements, etc.)

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SECTION 15. LABORATORY PERSONNEL TRAINING RECORDS

The SRCSD EL maintains training records for each employee in an Excel file. The records include the analyst's name, the method(s) and date(s) for which the analyst has completed training, the person(s) (supervisor) certifying completion of the training session, the date(s) recertification is needed and the date(s) recertification was/were completed (if appropriate). Only analysts who have completed training and demonstrated proficiency may conduct analytical methods independently. An analyst in training must be directly supervised by an analyst who has completed training.

The training record may also include additional educational courses, professional seminars attended, in house training courses, etc.

The SRWTP Administration Section also maintains an employee training records database that is maintained by Plant Administration staff. The Plant's database includes the laboratory employee training records.

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APPENDIX 1. GLOSSARY OF TERMS AND DEFINITIONS

Acceptance Criteria – Specified limits placed on characteristics of a quality control item as defined in required documents.

Accuracy – A measure of the agreement between the true value and the individual or average measured value of a parameter.

Acute Toxicity – A stimulus severe enough to rapidly induce an effect; in aquatic toxicity tests, an effect observed in 96 hours or less, not always measured in lethality.

Aliquot – A measured portion of a sample, or solution, taken for sample preparation or analysis.

Ambient Water – Waters in the natural environment (e.g., rivers, lakes, streams, and other receiving waters), as opposed to effluent discharges.

Analysis Matrix Spike – The subjection of a prepared sample, extract or digestate that has been fortified (spiked) with a known amount of the analyte of interest to the determinative step of an analytical method to estimate the bias imparted by the instrumental or determinative procedure.

Analyst – The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analyte – The chemical element or compound an analyst seeks to determine; the chemical element of interest.

Analytical Batch – A batch composed of prepared environmental samples (extracts, digestates, or concentrates) which are analyzed together as a group. It can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Areal Composite – Sample composited from individual, equal aliquots collected on an areal or horizontal cross-sectional basis. Each aliquot is collected in an identical manner.

Assessment – The evaluation process used to measure the performance or effectiveness of a system and its elements. As used here, assessment is an all-inclusive term used to denote any of the following: audit, performance evaluation, management systems review, peer review, inspection, or surveillance.

Audit – A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. An independent

review conducted to compare the various aspects of laboratory performance with a standard for performance. (Also see *Laboratory Audits*.)

Authenticate – The act of establishing an item as genuine, valid, or authoritative.

Background Sample – A sample (usually a grab) collected from an area, water body, or site similar to the one being studied, but located in an area known or thought to be free from pollutants of concern.

Batch – Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A batch can be classified as a preparation or analytical batch.

Bias – Consistent under or over-estimation of the true value due to systematic errors (sampling errors, sample handling errors, or analytical errors) in a procedure (i.e., the expected sample measurement is different from the sample's true value).

Bioassay – The use of living organisms to measure the effect of a substance, factor, or condition by comparing its effect on a living organism with the effect of a standard preparation on the same type of organism.

Blank – A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage, or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to correct or adjust routine analytical results.

Blind Sample – A proficiency test sample for which the analyst is unaware of the test nature of the sample at the time of analysis. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Blunder – A mistake that occurs on occasion and produces erroneous results.

Calibration – Comparison of a measurement standard, instrument, or item with a standard or instrument of higher accuracy to detect and quantify inaccuracies and to report or eliminate those inaccuracies by adjustments.

Calibration Blank – A volume of deionized water fortified with the same matrix as the calibration standards but without the analytes, internal standards, or surrogate analytes.

Calibration Curve – The graphical representation between known values (such as concentrations) of a series of calibration standards and their instrument response.

Calibration Drift – The deviation in instrument response from a reference value over a period of time before recalibration.

Calibration Method – A defined technical procedure for performing a calibration.

Calibration Standards – A series of known standard solutions used by the analyst for calibration of the instrument. The calibration standards are used to calibrate the instrument response with respect to analyte concentration.

Certification – The process of testing and evaluation against specifications designed to document, verify, and recognize the competence of a person, organization, or other entity to perform a function or service usually for a specified time.

Certified Reference Material – A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.

Chain of Custody – An unbroken trail of accountability that ensures the physical security of samples, data, and records, and includes the signatures of all who handle the samples, with the intent of legally demonstrating that custody remained intact and that tampering or substitution were precluded.

Characteristic – Any property or attribute of a datum, item, process, or service that is distinct, describable and/or measurable.

Chronic – A stimulus that lingers or continues for a relatively long period of time, often one-tenth of the life span or more.

Comparability – A measure of the confidence with which one data set or method can be compared to another.

Competency Test – The evaluation of a person's ability to perform work in any functional area prior to the performance of independent work.

Completeness – A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions.

Composite Sample – A sample that is collected over a temporal or spatial range that typically consists of a series of discrete, equal samples (or aliquots) which are combined.

Contamination – A component of a sample or an extract that is not representative of the environmental source of the sample.

Control Sample – Chemically, a standard of comparison for verifying or checking the findings of an experiment.

Control Sample Site – Typically a discrete grab sample collected to isolate a source of contamination. Isolation of a source could require the collection of both an upstream

sample at a location where the medium being studied is unaffected by the site studied, as well as a downstream control which could be affected by contaminants contributed from the site under study.

Corrective Action – Measures taken to rectify conditions adverse to quality and (where possible) to preclude their recurrence.

Correlation Coefficient – A number between -1 and +1 that indicates the degree of linearity between two variables, or sets of numbers. The closer to -1 or +1, the stronger the linear relationship between the two (i.e., better correlation). Values close to zero suggest no correlation.

Database – A collection of related information about a subject organized in a useful manner that provides a base or foundation for procedures such as retrieving information, drawing conclusions, and making decisions.

Data of Known Quality – Data that have the qualitative and quantitative components associated with their derivation documented appropriately for their intended use, and when such documentation is verifiable and defensible.

Data Quality Objectives (DQOs) – Qualitative and quantitative statements derived from the DQO process that clarify study objectives, define the appropriate type of data, and specify the tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions. (See EPA QA/G-5)

Data Quality Objectives Process – A systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use.

Data Reduction – The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useful form. Data reduction is irreversible and generally results in a reduced data set and an associated loss of detail.

Data Validation – A systematic process for reviewing a body of data against pre-established set of criteria to determine the quality of the data.

Detection Limit – The lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated level of probability. EPA: “The minimum concentration of an analyte (substance) that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero as determined by the procedure set forth in Appendix B of 40 CFR, Part 136.”

Director – The highest ranking manager within an individual laboratory.

Dissolved Solids – Those elements that will pass through a 0.45 µm membrane filter.

Document – Any written or pictorial information describing, defining, specifying, reporting, or certifying activities, requirements, procedures, or results.

Document Control – The policies and procedures used by an organization to ensure that its documents and their revisions are proposed, reviewed, approved for release, inventoried, distributed, archived, stored, and retrieved in accordance with the organization's requirements.

Duplicate Samples – Two samples collected from a common source and carried through all steps of the sampling and analytical procedures in an identical manner. They are used to assess variance of the total method including sampling and analysis.

Duty – An action, task, etc., required by or relating to one's occupation or position.

Effluent – Treated or untreated wastewater that flows out of a treatment plant, sewer or industrial outfall. Generally refers to wastes that are discharged into surface waters and are regulated under the Clean Water Act.

Equipment Blank – A sample collected using analyte-free water which has been run over/through sample collection equipment. These samples are used to determine if contaminants have been introduced by contact of the sample medium with sampling equipment.

External Standards – A method of quantifying chromatographic data in which standards of known concentrations are analyzed prior to unknown samples.

Extractable – A compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography.

Field Blank – A control sample used to provide information about contaminants that may be introduced during sample collection (atmospheric), as well as those activities listed under trip blank (storage, and transport). A clean sample, carried to the sampling site, exposed to sampling conditions and returned to the laboratory and treated as an environmental sample.

Field Duplicate Samples – Two separate samples that are collected at the same time and place under identical conditions and treated the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

Field Matrix Spike – A sample prepared at the sampling point (i.e., in the field) by adding a known mass of target analyte to a specified amount of sample. Field matrix spikes are used to determine the effect of the sample preservation, shipment, storage and sample preparation on analyte recovery efficiency (analytical bias).

Field Split Samples – Two or more representative portions taken from the same sample and submitted for analysis to different laboratories to estimate interlaboratory precision. (Also see “Laboratory Split Samples”.)

Flow Proportioned Composite – A sample collected proportional to the flow during the compositing period by either time-varying/constant volume (TVCV) or time-constant/varying volume method.

Grab Sample – An individual sample collected from a single location at a specific time, or period of time not to exceed 15 minutes. (US EPA, 1982.)

Hazardous Substance – Any material that poses a threat to human health and/or the environment. Typical hazardous substances are toxic, corrosive, ignitable, explosive, or chemically reactive.

Headspace – Any area in a container not completely filled by the sample in which gases can collect.

Holding Time – The period between sample collection and its required analysis when designated preservation and storage techniques are employed. While exceeding the holding time does not necessarily negate the veracity of analytical results, it causes the qualifying or “flagging” of the data for not meeting all of the specified acceptance criteria.

Instrument Blank – A blank designed to determine the level of contamination associated with the analytical instruments.

Instrument Check Standard – A multi-element standard of known concentrations prepared by the analyst to monitor and verify the instrument performance on a daily basis.

Instrument Detection Limit – The concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength.

Intercomparison Study – An exercise in which samples are prepared and split by a reference laboratory, then analyzed by one or more testing laboratories and the reference laboratory. The intercomparison, with a reputable laboratory as the reference laboratory, serves as the best test of the precision and accuracy of the analytes at natural environmental levels.

Internal Standard – A standard added to a test portion of a sample in a known amount and carried through the entire determination procedure as a reference for calibration and controlling the precision and bias of the applied analytical method.

Laboratory Audit – A systematic on site laboratory evaluation. It is used to determine the managerial and technical capability of the laboratory to perform analysis in conformance with specifications in contracts and approved analytical methods. Audits normally evaluate a laboratory's technical expertise, operating procedures, facility and equipment sufficiency, and possible sources of sample contamination.

Laboratory Blank – A that is blank taken through sample preparation and analysis only. It is a test for contamination in sample preparation and analysis.

Laboratory Control Sample – A material of known composition (similar to the samples analyzed) used expressly for the purpose of monitoring a measurement process.

Laboratory Duplicate – Two sample aliquots taken in the analytical laboratory and analyzed separately with identical procedures. Analysis of laboratory duplicates give a measure of the precision associated with the laboratory procedures, but not with sample collection, preservation, or storage procedures.

Laboratory Fortified Blank (LFB) – An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results.

Laboratory Fortified Sample Matrix (LFM) – See Matrix Spike.

Laboratory Fraud – The deliberate falsification during reporting of analytical and quality assurance results that failed method and contractual requirements to make them appear to have passed requirements.

Laboratory Reagent Blank (LRB) – An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the analytical apparatus.

Laboratory Split Samples – Two or more representative portions taken from the same sample and analyzed by different laboratories to estimate the interlaboratory precision or variability and data comparability.

Limit of Quantitation (LOQ) – The minimum concentration of an analyte or category of analytes in a specific matrix that can be identified and quantified above the method detection limit and within specified limits of precision and bias during routine and analytical operating conditions. CWRCB identifies it as ten standard deviations greater than the average of measured blank values in developing the method detection limit.

Log-In – The receipt and initial management of an environmental sample. It includes identifying who sent the sample; maintaining chain-of-custody; checking report and

invoice information; recording analyses requested, including methodology and special instructions; and assigning a discreet laboratory identification number.

Matrix – The predominant material of which the sample to be analyzed is composed.

Matrix Effect – In general, the effect of a particular matrix (water or soil/sediment) on the constituents with which it contacts.

Matrix Interference – The influence of the sample matrix or sample components upon the ability to qualitatively identify and quantitatively measure compounds in environmental samples.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD) – An MS or MSD are aliquots of environmental samples that are fortified in the laboratory with a known amount of analyte(s) of interest. They are analyzed exactly like a sample. They are used to indicate the appropriateness of the method, assess method bias and, when performed in duplicate, measure precision on “real” samples in the presence of matrix effects by measuring recovery. (Also known as Laboratory Fortified Sample Matrix)

May – When used in a sentence this denotes permission but not a requirement.

Mean (arithmetic) – The sum of all the values of a set of measurements divided by the number of values in the set. A measure of central tendency.

Median – The observation of a data set that is in the middle, that is, the number that is located such that half of the observations are less than in and half are greater.

Method – The course of action or technique followed in conducting a specific analysis or comparison leading to an analytical result. A method is systematically presented in the order in which the steps are to be executed.

Method Blank – A sample that consists of an analyte-free matrix into which all reagents used in the sample processing are added in the same volumes or proportions. It is carried through the complete sample preparation and analytical procedure.

Method Detection Limit (MDL) – The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. The procedure for the determination of the MDL is presented in Appendix B to Part 136 of 40 CFR.

Method of Standard Addition – The standard addition technique involves the use of the unknown and the unknown plus a known amount of standard.

Minimum Level (ML) – The lowest level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to

the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed. The ML is calculated by multiplying the MDL by 3.18 and rounding the result to the number nearest to $(1, 2, \text{ or } 5) \times 10^n$, where n is an integer. (Also, see Section 1.5 of EPA Method 1631e).

Must – This action, activity, or procedural step is required.

National Pollutant Discharge Elimination System (NPDES) – A provision of the Clean Water Act (CWA) that prohibits discharge of pollutants into waters within the U.S. unless a special permit is issued by the US EPA, a state (where delegated), or a tribal government on an Indian reservation.

Nutrient – Any substance assimilated by living organisms that promotes growth.

Outlier – An observed value that appears to be discordant from the other observations in a sample. One of a set of observations that appears to be discordant from the others.

Parameter (statistic) – A quantity, usually unknown, such as a mean or standard deviation characterizing a population.

Percent Recovery – The measured concentration of a standard reference material (e.g. PES) divided by the known concentration, multiplied by 100, expressed as a percentage.

Performance Audit – A quantitative evaluation of a measurement system that involves the analysis of standard reference samples or materials which are certified as to their chemical composition or physical characteristics.

Performance Evaluation (PE) – A type of audit in which the quantitative data generated in a measurement system are obtained independently and compared with routinely obtained data to evaluate the proficiency of an analyst or laboratory.

Performance Evaluation Sample (PES) – A test sample prepared with known concentrations of specific analytes, within specified limits of uncertainty, and submitted to a laboratory for chemical analysis, which yields quantitative data that can be used to evaluate the ability of the laboratory to successfully handle, analyze and identify the contaminants, and accurately report their concentrations.

Pollutant – Generally, any substance introduced into the environment that adversely affects the usefulness of a resource.

Practical Quantitation Level (PQL) – A quantitation limit that is approximately 3 to 10 times the MDL and that is greater than the concentration of a chemical that can be detected by current laboratory methods, thereby providing a less demanding, “practically-based” analytical limit.

Precision – Agreement among a set of individual measurements of identical samples, and usually be expressed in terms of variance without assumption of knowledge of the true value.

Preparation Batch – A batch composed of one to 20 environmental samples of the same matrix, with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours.

Procedure – A specified way to perform an activity. The manner in which an operation is performed; a set of directions for performing an examination or analysis; the actual parameters of the methods employed.

Process – A set of interrelated resources and activities which transforms inputs into outputs. Examples of processes include analysis, design, data collection, operation, fabrication, and calculation.

Protocol – A directive listing the procedures to be followed in performing a particular laboratory examination or operation.

Quality Assurance – Those planned and systematic action necessary to provide sufficient confidence that a laboratory's product or service will satisfy given requirements for quality.

Quality Audit – A management tool used to evaluate and confirm activities related to quality. Its primary purpose is to verify compliance with the operational requirements of the quality system.

Quality Control – Internal activities or activities according to externally established standards used to monitor the quality of analytical data and to ensure that it satisfies specific criteria.

Quality Control Samples – Samples collected during field studies for various purposes which include the isolation of site effects (control samples), define background conditions (background sample), and evaluate field/laboratory variability (spikes and blanks, trip blanks, duplicate, split samples).

Quality Manual – A document stating the quality policy and describing the various elements of the quality system and quality practices of an organization.

Quantitation Limit – The minimum concentration of a compound that can be reliably quantified;; very dependent upon sample matrix.

Range – The difference between the largest and smallest values in a data set.

Reagent Blank – A volume of deionized distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme. It is used to

determine the concentration of the target analyte(s) in the reagents used to prepare and analyze the samples.

Reagent Water – Water demonstrated to be free of the target analyte(s) at the method MDL. It is prepared from 18 MΩ ultra pure deionized water starting from a pre-purified source. Reagent water is used to wash bottles, as trip and field blanks, and in the preparation of standards and reagents.

Regulatory Compliance Limit – A limit on the concentration or amount of a pollutant or contaminant specified in a nationwide standard, in a permit, or otherwise established by a regulatory authority.

Repeatability – The degree of agreement between independent test results produced by the same analyst, using the same test method and equipment on random aliquots of the same sample within a sort time period.

Replicate Samples – A set of environmental samples collected in a manner such that the samples are thought to be essentially identical in composition. Replicate is the general case for which a duplicate is the special case consisting of two sequentially-collected samples and for which a split is the special case in which two or more samples are generated from one.

Reporting Limit (RL) – The lowest concentration or amount of the target analyte required to be reported from a data collection project. Reporting limits are generally greater than detection limits and are usually not associated with a probability level.

Representative Sample – A sample that reflects one or more characteristics of a population.

Reproducibility – The precision, usually expressed as variance that measures the variability among the results of measurements of the same sample at different laboratories.

Sample – Part of a larger lot, usually an area, a volume, or a period of time. A portion of material to be analyzed that is contained in a single or multiple containers and identified by a unique sample number.

Sample Representativeness – The degree to which a set of samples defines the characteristics of a population, where each sample has an equal probability of yielding the same result.

Shall – This action, activity, or procedure is required.

Should – This action, activity, or procedure is suggested, but not required.

Site Water – Upstream water, actual downstream water, or simulated downstream water in which a toxicity test is conducted side-by-side with the same toxicity test in a laboratory dilution water to determine a water-effect-ratio (WER).

Solvent – A substance, usually liquid, capable of dissolving or dispersing one or more other substances.

Spike – A sample with known concentrations of analytes. Spiked samples are used to measure the ability of test procedures to generate a correct result from the sample, or assess the performance of a laboratory.

Split Sample – A sample which has been portioned into two or more containers from a single sample container or sample mixing container. The primary purpose of a split sample is to measure sample handling variability.

Split Field Sample – A type of replicate sample in which one sample is split into two or more subsamples composited contemporaneously in two or more collection containers.

Standard Deviation – The square root of the variance.

Standard Operating Procedures (SOPs) – A written document that gives precise descriptions of routine procedures for operations, analyses, or actions. It is a written document which provides directions for the step-by-step execution of an operation, analysis, or action which is commonly accepted as the method for performing certain routine or repetitive tasks. The SOP is functional, clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts.

Standard Reference Material (SRM) – Quality control standards which are traceable to the National Institute of Standards and Testing (NIST) materials. NIST traceable materials are used for calibration and quality control of all US EPA approved testing protocols.

Statistically Significant – When the difference between a predicted and an observed value is so large that it is improbable it could be attributable to chance.

Stock Solution – A solution containing an analyte that is prepared from a reference material traceable to NIST, or a source that will attest to the purity and authenticity of the reference material.

Surrogate – An organic compound similar to the analyte of interest in chemical composition, extraction, and chromatography, but not normally found in environmental samples. Primarily used in chromatography techniques, the surrogate standard is spiked into quality control blanks, calibration and check standards, samples and spiked samples before analysis. A percent recovery is calculated for each surrogate.

System Blank – An analyte free sample used to determine contamination in the analytical system and in the reagents used to prepare the calibration standards.

Temperature Blanks – A container of water shipped with each cooler of samples requiring preservation by cooling to 4 °C. The temperature of the blanks is measured at the time of sample receipt by the laboratory.

Time Composite Sample – A sample comprised of a varying number of discrete samples collected at equal time intervals during the composite period.

Total Recoverable – The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

Trip Blanks – An analyte free water sample prepared in the laboratory prior to the sampling event and stored in the same container with the investigative samples throughout the sampling event. They are then packaged for shipment with the other samples and submitted for analysis as regular samples. At no time after their preparation are trip blanks to be opened before they reach the laboratory. Trip blanks are used to determine if samples were contaminated during storage and/or transport to the laboratory. They are typically used for volatile contaminants and mercury analysis where ever samples may be subject to vapor phase contamination.

Ultraclean Technique – Refers to those requirements or practices necessary to produce reliable analytical data in the part per trillion (ppt) range.

Uncensored Data – Data reported as the measured value (actual instrument response, or calculated quantity if derived by subtraction) and do not include data reported as “not detected” or “less than” a specified value.

Variability – The range or “distribution” of a result around the mean value obtained from samples within a population.

Variance – A measure of the dispersion of a set of values.

Water Effect Ratio (WER) – An approximate measure of the toxicity of a material obtained in a site water divided by the same measure of the toxicity of the same material obtained in a laboratory dilution water.

Water Sample – A sample taken from one of the following sources: drinking, surface, ground, sea, brackish, industrial, or domestic wastewater.

Wet Chemistry – Procedures that involve distillation, colorimetric determinations and titrimetric measurements.

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APPENDIX 2. SAMPLE CONTAINER AND HOLDING TIME GUIDANCE

MAXIMUM HOLDING TIMES AND SAMPLE COLLECTION/PRESERVATION INFORMATION

(Ref.: Federal Register / Vol. 27, No. 46 / Monday, March 12, 2007 / Rules and Regulations)

Parameter	Container ¹	Preservation ^{2,3}	Max Holding Time ⁴
Bacterial Tests:			
Coliform, total, fecal, and <i>E coli</i>	PA, G	Cool, <10 °C; 0.0008% Na ₂ S ₂ O ₃ 5	6 hours
Fecal streptococci	PA, G	Cool, <10 °C; 0.0008% Na ₂ S ₂ O ₃ 5	6 hours
Enterococci	PA, G	Cool, <10 °C; 0.0008% Na ₂ S ₂ O ₃ 5	6 hours
Protozoan Tests:			
Cryptosporidium	LDPE; Field filtration	0-8 °C	96 hours ²¹
Giardia	LDPE; Field filtration	0-8 °C	96 hours ²¹
Aquatic Toxicity Tests:			
Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C ¹⁶	36 hours
Inorganic Tests:			
Acidity	P, FP, G	Cool, ≤6 °C ¹⁸	14 days
Alkalinity	P, FP, G	Cool, ≤6 °C ¹⁸	14 days
Ammonia	P, FP, G	Cool, ≤6 °C ¹⁸ H ₂ SO ₄ to pH <2	28 days
Biochemical oxygen demand, total	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours
Biochemical oxygen demand, , carbonaceous	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours
Boron	P, FP, or Quartz	HNO ₃ to pH <2	6 months
Bromide	P, FP, G	None required	28 days
Chemical oxygen demand	P, FP, G	Cool, ≤6 °C ¹⁸ H ₂ SO ₄ to pH <2	28 days
Chloride	P, FP, G	None required	28 days
Chlorine, totals residual	P, G	None required	Analyze within 15 minutes
Color	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours
Cyanide, total or available	P, FP, G	Cool, ≤6 °C ¹⁸ , NaOH to pH >12, reducing agent	14 days
Fluoride	P	None required	28 days
Hardness	P, FP, G	HNO ₃ or H ₂ SO ₄ to pH <2	6 months
Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes
Parameter	Container ¹	Preservation ^{2,3}	Max Holding

			Time ⁴
Inorganic Tests:			
Kjeldahl and organic nitrogen	P, FP, G	Cool, ≤6 °C18 ,	28 days
Nitrate	P, FP, G	H2SO4 to pH <2 Cool, ≤6 °C18	48 hours
Nitrate -Nitrite	P, FP, G	Cool, ≤6 °C18, HCl or H2SO4 to pH <2	28 days
Nitrite	P, FP, G	Cool, ≤6 °C18	48 hours
Oil and grease	G	Cool, ≤6 °C18 , H2SO4 to pH <2	28 days
Organic carbon	P, FP, G	Cool, ≤6 °C18, HCl, H2SO4, or H3PO4 to pH <2	28 days
Orthophosphate	P, FP, G	Cool, ≤6 °C18	Filter within 15 minutes; Analyze within 48 hours
Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes
Oxygen, Winkler	G, Bottle and top	Fix on site and store in dark	8 hours
Phenols	G	Cool, ≤6 °C18	28 days
Phosphorous, elemental	G	Cool, ≤6 °C18	48 hours
Phosphorous, total	P, FP, G	Cool, ≤6 °C18 , H2SO4 to pH <2	28 days
Residue, Total (TS)	P, FP, G	Cool, ≤6 °C18	7 days
Residue, Filterable (TDS)	P, FP, G	Cool, ≤6 °C18	7 days
Residue, Non-filterable (TSS)	P, FP, G	Cool, ≤6 °C18	7 days
Residue, Settleable (SS)	P, FP, G	Cool, ≤6 °C18	48 hours
Residue, Volatile (VS)	P, FP, G	Cool, ≤6 °C18	7 days
Silica	P or Quartz	Cool, ≤6 °C18	28 days
Specific Conductance (EC)	P, FP, G	Cool, ≤6 °C18	28 days
Sulfate	P, FP, G	Cool, ≤6 °C18	28 days
Sulfide	P, FP, G	Cool, ≤6 °C18, add zinc acetate plus NaOH to pH >9	7 days
Sulfite	P, FP, G	None required	Analyze within 15 minutes
Surfactants	P, FP, G	Cool, ≤6 °C18	48 hours
Temperature	P, FP, G	None required	Analyze
Turbidity	P, FP, G	Cool, ≤6 °C18	48 hours
Table 1B – Metals Tests:⁷			
Chromium VI	P, FP, G	Cool, ≤6 °C18 , pH = 9.3 – 9.720	28 days
Mercury (CVAA)	P, FP, G	HNO3 to pH <2	28 days
Mercury (CVAFS)	FP, G; and FP-lined cap ¹⁷	5 mL/L 12N HCl ¹⁷ or 5 mL/L BrCl ¹⁷	90 days
Metals, except boron, chromium VI, and mercury	P, FP, G	HNO3 to pH <2, or at least 24 hours prior to analysis ¹⁹	6 months

Parameter	Container ¹	Preservation ^{2,3}	Max Holding Time ⁴
Table 1C – Organic Tests			
Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C ₁₈ ; 0.0008% Na ₂ S ₂ O _{3 5}	14 days
Purgeable aromatic hydrocarbons	G, FP-lined septum	Cool, ≤6 °C ₁₈ ; 0.0008% Na ₂ S ₂ O _{3 5}	14 days ⁹
Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C ₁₈ ; 0.0008% Na ₂ S ₂ O _{3 5}	14 days ¹⁰
Phenols ¹¹	G, FP-lined cap	Cool, ≤6 °C ₁₈ ; 0.0008% Na ₂ S ₂ O _{3 5}	7 days until extraction, 40 days after extraction
Benzidines ^{11, 12}	G, FP-lined cap	Cool, ≤6 °C ₁₈ ; 0.0008% Na ₂ S ₂ O _{3 5}	7 days until extraction ¹³
Phthalate esters ¹¹	G, FP-lined cap	Cool, ≤6 °C ₁₈	7 days until extraction, 40 days after extraction
Nitrosoamines ^{11, 14}	G, FP-lined cap	Cool, ≤6 °C ₁₈ ; store in dark, 0.0008% Na ₂ S ₂ O _{3 5}	7 days until extraction, 40 days after extraction
PCBs ¹¹	G, FP-lined cap	Cool, ≤6 °C ₁₈	1 year until extraction, 1 year after extraction
Nitroaromatics and isophorone ¹¹	G, FP-lined cap	Cool, ≤6 °C ₁₈ ; store in dark, 0.0008% Na ₂ S ₂ O _{3 5}	7 days until extraction, 40 days after extraction
Polynuclear aromatic hydrocarbons ¹¹	G, FP-lined cap	Cool, ≤6 °C ₁₈ ; store in dark, 0.0008% Na ₂ S ₂ O _{3 5}	7 days until extraction, 40 days after extraction
Haloethers ¹¹	G, FP-lined cap	Cool, ≤6 °C ₁₈ ; 0.0008% Na ₂ S ₂ O _{3 5}	7 days until extraction, 40 days after extraction
Chlorinated hydrocarbons ¹¹	G, FP-lined cap	Cool, ≤6 °C ₁₈	7 days until extraction, 40 days after extraction
CDDs/CDFs¹¹			
Aqueous Samples: Field and Lab Preservation	G	Cool, ≤6 °C ₁₈ ; 0.0008% Na ₂ S ₂ O _{3 5} , pH <9	1 year
Solids and Mixed-Phase Samples: Field Preservation	G	Cool, ≤6 °C ₁₈	7 days
Tissue Samples: Field Preservation	G	Cool, ≤6 °C ₁₈	24 hours
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation	G	Freeze, ≤ -10 °C	1 year

Parameter	Container ¹	Preservation ^{2,3}	Max Holding Time ⁴
Table 1D – Pesticide Tests:			
Pesticides ¹¹	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , pH 5-9 ¹⁵	7 days until extraction, 40 days after extraction
Table 1E – Radiological Tests:			
Alpha, beta, and radium	P, FP, G	HNO ₃ to pH <2	6 months

Table Footnotes:

1 “P” is polyethylene, “FP” is fluoropolymer (polytetrafluoroethylene, “G” is glass, “PA” is any plastic made of sterilizable material, “LPDE” is low density polyethylene.

2 Except where noted, preserve each sample within 15 minutes of collection. For composite samples, refrigerate sample at ≤6 °C during collection unless specified otherwise. Add the preservative to the sample container prior to collection when the preservative will not compromise the integrity of a sample, otherwise preserve the sample within 15 minutes of collection. Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of the results of analysis of individual grab samples.

3 When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Dept. of Transportation (DOT) Hazardous Materials Regulations (49 CFR Part 172). For the preservation requirements cited above, the DOT has determined that the Regulations do not apply to the following materials: hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and sodium hydroxide (NaOH) in water solutions at concentrations of 0.80% by weight or less (pH about 12.30 or less).

4 Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid. Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the grab last sample in the set. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and are collected across two calendar days, the date of collection is the two calendar days; e.g., November 14-15. For a composite sample collected automatically on a given date, the date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days.

5 Add reducing agent only if an oxidant (e.g., chlorine) is present. Reducing agents shown to be effective are sodium thiosulfate (Na₂S₂O₃), ascorbic acid, sodium arsenite (NaAsO₂), or sodium borohydride (NaBH₄). However some of these agents have been shown to produce a positive or negative cyanide bias, depending on other substances in the sample and the analytical method used. Therefore, do not add an excess of reducing agent. Methods recommending ascorbic acid (e.g., EPA Method 335.4) specify adding ascorbic acid crystals, 0.1 – 0.6 g, until a drop of sample produces no color on potassium iodide (KI) starch paper, then adding 0.06 g (60 mg) for each liter of sample volume. If NaBH₄ or NaAsO₂ is used, 25 mg/L NaBH₄ or 100 mg/L NaAsO₂ will reduce more than 50 mg/L of chlorine (see method (Kelada-01” and/or SM 4500-CN- for more information). After adding reducing agent, test the sample using KI paper, a test strip (e.g., for chlorine, SenSafe™ Total Chlorine Water Check 480010) moistened with acetate buffer solution, or a chlorine/oxidant test method (e.g., EPA Method 330.4 or 330.5), to make sure all oxidant is removed. If oxidant remains, add more reducing agent. Whatever agent is used, it should be tested to assure that cyanide results are not affected adversely.

6 Sample collection and preservation: Collect a volume of sample appropriate to the analytical method in a bottle of the material specified. If the sample can be analyzed within 48 hours and sulfide is not present, adjust the pH to >12 with sodium hydroxide solution (e.g., 5 % w/v), refrigerate as specified, and analyze within 48 hours. Any procedure for removal or suppression of an interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide. Particulate cyanide (e.g., ferric ferrocyanide) or a strong cyanide complex (e.g., cobalt cyanide) are more accurately measured if the laboratory holds the sample at room temperature and pH >12 for a minimum of 4 hours prior to analysis, and performs UV digestion or dissolution under alkaline (pH=12) conditions, if necessary.

7 For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

8 Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

9 If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.

10 The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

11 When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observe for optimum safeguard of sample integrity (i.e., use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to ≤6 °C, reducing residual

chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnote 12, 13 (regarding the analysis of benzidine).

12 If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

13 Extracts may be stored up to 30 days at $<0^{\circ}\text{C}$

14. For the analysis of diphenylnitrosamine, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ and adjust pH to 7 – 10 with NaOH within 24 hours of sampling.

14 The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$.

15 Sufficient ice should be placed in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

16 Samples collected for the determination of trace level mercury ($<100\text{ ng/L}$) using EPA Method 1631 must be collected in tightly capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

17 Aqueous samples must be preserved at $\leq 6^{\circ}\text{C}$, and should not be frozen, unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of " $\leq 6^{\circ}\text{C}$ " is used in place of the " 4°C " sample temperature requirements listed in some methods. It is not necessary

to measure the sample temperature to three significant figures ($1/100$ of 1°C); rather, three significant figures are specified so that rounding down to 6°C may not be used to meet the $\leq 6^{\circ}\text{C}$ requirement. The preservation temperature does not apply to samples that are analyzed immediately (**less than 15 minutes**).

18 An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

19 To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

20 Holding time is calculated from the time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.

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APPENDIX 3. LABORATORY PERSONNEL BIOGRAPHIES

- 4.1. **LUCY BOEHM, SRCSD Laboratory Manager**, –Ms Boehm is the Laboratory Manager since October 2009. Lucy has been in the field of Environmental testing since 1987. She was the chemistry section supervisor at SRCSD EL from 1994 to 2005. The Chemistry Section is responsible for the organics, trace metals, and cyanide testing. Her primary duties in this position were as follows: Supervise and evaluate 4 chemists, 2 analysts and one student intern; prepare technical reports; prepare job duty statements and annual employee evaluations; schedule work; perform hiring tasks; manage and validate test results; order supplies; provide input to the lab's annual budget preparation; coordinate lab programs for other Sacramento County agencies; serve as Plant Radiation Safety officer (assure compliance with the Plant's Radioactive Materials License). Lucy earned a Baccalaureate Degree in Biological Science in 1972 from the University of California, Davis. Prior to working at the SRCSD EL, Lucy spent 13 years as a chemist and then a supervisor for Babcock Laboratory in Riverside, California, where she specialized in metals testing working with drinking water, water, agricultural and hazardous wastes samples.
- 4.2. **SRIVIDHYA RAMAMOORTHY, SRCSD EL Quality Assurance Officer (QAO)** – Srivi received her Ph.D in Microbiology in 2005, from University of Montana, with a major emphasis on Environmental Microbiology. She also has a B.S in Physics and M.S in Biophysics and Crystallography from University of Madras, India. Srivi coordinates the ELAP certification activities and has administrative responsibility for the QA Program. She is also the Laboratory safety officer and Chemical hygiene officer.
- 4.3. **TINA MARCUM, Sr. Office Assistant** – Tina works as the Administrative clerical support for the laboratory. She has worked for the County since 1991. She is charged with the daily management of laboratory records and organization of files. She manages and maintains the laboratory office equipment and supplies. Prior to SRCSD EL Ms Marcum was working as a Animal Control officer at Sacramento County Animal Control Agency
- 4.4. **CONVENTIONAL CHEMISTRY/ SAMPLING SECTION**
- 4.4.1. **DAN DIEHL, Laboratory Supervisor**, Conventional Chemistry Section – Mr. Diehl started working at the SRCSD EL as a Laboratory Assistant in 1994 where he was assigned to conduct inorganic conventional wet-testing procedures. Prior to working here he was with the California Department of Fish and Game as a scientist at their Aquatic Toxicology Laboratory in Elk Grove, CA for two years. In 1998 Dan was promoted to the

Supervisor position for the Conventional Chemistry Section. Dan earned a Bachelor's Degree in 1992, in Environmental Biology from California State University, Sacramento.

- 4.4.2. **TONY GONZALES, Sr. Environmental Laboratory Analyst** – Tony has worked in an environmental testing laboratory since 1975. He has experience in conventional chemical analysis of pollutants, trained in and application of correct sampling techniques for soil, wastewater, groundwater, and potable water. Mr. Gonzales has attended four years of college level classes with a major emphasis in biochemistry. In addition to his conventional testing responsibilities, he is the lead person in sample collection and field testing of the Groundwater Monitoring Program.
- 4.4.3. **Celeste C. Patena, Sr Environmental Laboratory Analyst** - Celeste has worked in laboratory test since 1981. She has experience in conventional chemical analysis of pollutants, trained in and application of correct sampling techniques for wastewater, groundwater, and potable water. Celeste obtained her Bachelor's degree in Agricultural science in 1980. Before joining SRCSD EL, Celeste worked as a Laboratory Assistant at West Sacramento WWTP. Currently she is the primary operator for Total Organic Carbon analyzer along with performing bench top WET chemistry analysis.
- 4.4.4. **MIKE COOK, Sr. Environmental Laboratory Analyst** – Mike Cook was hired by the SRCSD EL in the summer of 2003 acting in the capacity of a Program Coordinator for various internal (Plant) and external monitoring programs including the lead person for the Ambient Monitoring Program (AMP). For the AMP, Mike is the crew chief for the SRCSD EL's ambient monitoring boat the "Guardian". Prior to working at the SRCSD EL, Mike has five years of research experience as a scientist in an agricultural plant laboratory conducting field and bench level research on pest and pesticide resistant fruit crops. Mike graduated from the University of California at Davis in 1994 with a Bachelor's Degree in Environmental Toxicology. He also spent two years of graduate research work using GC technology in studying the fate and transport of pesticides in biological organism of lakes, rivers, and streams.
- 4.4.5. **KIM TUFTS, Sr. Environmental Laboratory Analyst** – Ms. Tufts received a Bachelor of Arts Degree in Biology from Humboldt State University in 1978. Kim began her career as a Laboratory Technician in a private analytical laboratory in Northern California. While there, she was promoted to Analytical Program Coordinator and Laboratory Supervisor. She also served as the laboratory's Safety Officer. Kim started work at the SRCSD EL in 1999 as a Laboratory Assistant. In 2000 she was promoted

to Environmental Laboratory Analyst and in 2001 to her current position. She has been trained and is experienced in applying correct sample collection techniques, conducting conventional wet chemistry analytical procedures, and has previously been the principal operator of the TOC analyzer. She is the lead person for the sample collection, receiving and disposition activities and the Sample Custodian/Coordinator. She has Program Coordination responsibilities for the SRWTP NPDES compliance process monitoring and a number of external customer programs.

- 4.4.6. **CHIHO CHAN, Environmental Laboratory Analyst** – Ms. Chan was hired in 2001 by the SRCSD EL. She is currently coordinator for various internal and external programs. Prior to this she worked as a Environmental laboratory Analyst where she was the principal operator for the Lachat auto-analyzer which uses segmented flow technology to do the automated nitrogen, chloride, phosphorous, and orthophosphate analyses. Chiho has also had bench level experience conducting bacteriological and microbiological testing while assigned to the Biology Section when she started with the SRCSD EL. In 1996 she received her Bachelor's Degree in Environmental Sciences for the University of Hong Kong. She started her career as a research assistant in 1998 working in the City University of Hong Kong.
- 4.4.7. **MICHAEL YERBY, Environmental Laboratory Analyst** – Michael started work at the SRCSD EL in 1993 as a Laboratory Assistant. His current assignments include various conventional chemistry tests including MBAS, UV 254. In 1991, Mr. Yerby earned a Bachelor's Degree in Plant Science from the University of California, at Berkeley. He holds a California Water Pollution Control Association Grade I Laboratory Technician certificate. He has experience in metals preparation, EPA Method 245.1 low level mercury analysis, operated the GFAA, inorganic testing, bacteriological and toxicological testing procedures.
- 4.4.8. **ROY FONG, Environmental Laboratory Analyst** – Mr. Fong is assigned to the Conventional Chemistry Section where he conducts conventional chemical procedures. He has worked at the SRCSD EL since 1974. His duties also include collecting samples and interacting with the LIMS. Roy earned and AA degree in 1972 from Jr. College and has done additional coursework towards a Bachelor's degree at California State University, Sacramento.
- 4.4.9. **STEVE (YIH-MIN) TSUI, Environmental Laboratory Analyst** – In 1989 Steve Tsui began his career at the SRCSD EL. He has extensive experience in most of the conventional wet chemistry methods. He

received a Bachelor's Degree in 1968 in Biological Science from Chung-Hsing University of Taichung, Taiwan and has done post graduate work in Biology at Texas Southern University, Houston, Texas. After graduation, Steve worked at the Hwa-Sha Institute in Taiwan where his tasks included the use of colorimeters, titration fixation, microtome, cell staining, cell culture, electron microscopy, chemical and microbiological analyses of water. In addition, he is the author of the book titled, "The Practice of Acupuncture".

- 4.4.10. **NAI SAETERN, Laboratory Assistant.** - Ms. Saetern started as a student intern at SRCSD EL before becoming a Laboratory Assistant. Her current work assignments include most of the in Plant sampling. She also performs the solids testing. Nai has a Associate degree in Biology and Social Sciences from Sacramento City College, CA. She is also currently working as Biology Laboratory Technician at Sacramento City College.

4.5. **CHEMISTRY (Metals & Organics) SECTION**

- 4.5.1. **HANOI KWONG, Laboratory Supervisor, Chemistry Section** – Ms. Kwong supervises the Chemistry Section of the laboratory. The Chemistry Section is responsible for the organics, trace metals, and cyanide testing. Ms. Kwong began her career as a Research Chemist at the NASA Ames Research Center in the SF Bay area. While there she operated gas chromatography analyzer system, an infrared spectrometer and other analytical instruments. She has been a Chemist in the SRCSD EL organization since 1981. She completed her Bachelor's Degree in Chemistry in 1977 while at California State University, Sacramento, and her Master's Degree in Chemistry in 1982 at California State University, San Jose. Hanoi is the Principal Analyst for trace metals determination using the Perkin-Elmer 6100 ICP/MS
- 4.5.2. **RON HARRIS, Chemist** – Dr. Ron Harris joined the organization in 1992. In 1979 he earned his Doctorate Degree in Chemistry from the University of Texas, Austin. He is a principal operator for the Flame AA, the Gas Furnace AA, the ICP/MS, and the UV/VIS spectrophotometer system used in the analysis of trace level mercury. He is also the lead person in training of staff for trace metals sample preparation, digestion, and cleaning. Ron had two years chemical research experience at Carus Chemical Company where his work involved researching and inventing water and air quality products. He worked as a chemist for one year at Bell Laboratories. In addition, he conducted research at the University of Texas, Austin, for one year, and research and training at the University of Purdue, Ft. Wayne, Indiana, for eight years. He taught chemistry at Northern Illinois University, in DeKalb, Illinois, Cosumnes River Jr. College, Sacramento, and University of Texas, Austin for a combined total

of 12 years.

- 4.5.3. **GURJIT DHILLON, Chemist** – Dr. Gurjit Dhillon began her career as a QC Chemist at American Garment Finishers and then at Sunbelt Solution in El Paso, Texas. While there she was responsible for quality control testing of raw materials in process and final products such as surfactants, wetting agents, softeners, and enzymes used to finish the garments like jeans by wet chemistry tests. Other testing include: BOD, COD, TDS, TSS of reuse and sewer water samples, cations, anions, and heavy metal analysis of well water samples with capillary electrophoresis. Prior to starting at SRCSD EL in 2005 she worked at Air Toxics as Analyst. She completed her Bachelor's Degree in Chemistry in 1980 from Punjab University, India. She completed her Master's Degree in Chemistry in 1982 and Ph.D in chemistry in 1987 from Guru Nanak University, India. Gurjit is the Principal Analyst for analysis semi-volatile organic compounds by GC/MS using the Varian Saturn 2200 GC/MS instrument.
- 4.5.4. **SCOTT BARMBY, Chemist** – Mr. Barmby has a B.A in chemistry from California State University, Sacramento. He started working at SRCSD EL as a Chemist from 2006. He is Principal analyst for testing metals using ICP-MS. He also performs organics testing. Before SRCSD EL, Scott worked as a chemist for 20 years at Severan Trent Laboratories, CA. At Severan Trent Laboratories he performed various analysis including volatiles, semivolatiles, extraction, and Low resolution dioxins. He also was responsible for creating a program for automated data entry.
- 4.5.5. **WAYNE HALOZAN, Chemist** – Wayne joined the organization in 2008. In 1982 he earned his Bachelor's Degree in Chemistry from the Stockton State College, NJ. He is the principal operator for the GC/MS-VOA, Pesticides and PCB testing. He has been in Environmental laboratory testing field since 1985. Prior to SRCSD EL Wayne worked at TestAmerica (formerly Enseco, Quanterra, and STL Laboratories) for 18 years. For TestAmerica he started as the Metals/Mobile Manager then became the Production manager for that facility. He later switched to Mobile Lab analysis where he performed environmental analysis in the field specializing in explosive analysis. He also setup the metals and PCB/Pesticide laboratories in Alaska for Test America. Wayne transferred to the West Sacramento facility where he was the VOA supervisor. For the last 5 years he had been working with Hi-Res Dioxin's and PCBs analysis.
- 4.5.6. **JOHN VREELAND, Environmental Laboratory Analyst** – John graduated from California State University, Sacramento, in 1984, with a Bachelor's Degree in Chemistry. He started work in the SRCSD EL in 1988 as a Laboratory Assistant and subsequently promoted to his current position. Through his career at the SRCSD EL Mr. Vreeland was assigned to work

in all three sections and thus is familiar and has working knowledge and experience with most of the analytical procedures employed in the laboratory. While in the Chemistry Section, John processed and prepared samples for metals analysis and was trained to operate the Flame AA spectrophotometer. He currently is assigned to conduct the conventional wet chemistry procedures. John has also conducted the bacteriological analyses in the Biology Section. He successfully completed a two year certificate course in Total Quality Management from the American River Jr. College, Sacramento.

- 4.5.7. **KARIN YEE, Environmental Laboratory Analyst** – Ms. Yee's has a degree in Biological sciences from UC, Davis. Her current work assignments include digestion for metals analysis and organic prep. She also performs cyanide and mercury analysis. Before starting at SRCSD EL she worked at Severan Trent Laboratories, CA for 21 yrs at various levels starting from Data technician to Scientist.
- 4.5.8. **ALEX, KLISHEVICH, Environmental Laboratory Analyst** – Mr. Klishevich started here at the SRCSD EL in 2006. His work assignments include digestion for metals analysis and organic prep. He also performs cyanide and mercury analysis. He has a B. A. in Zoology from Indiana University, and has done research on Bad Water at Death Valley through California State University, Los Angeles. Prior to SRCSD EL, he worked as a Water Treatment Plant Operator at Sacramento Water Treatment Plant and as a Public Health Microbiologist at Department of Health.
- 4.5.9. **CRAIG OTIS, Environmental Laboratory Assistant** – Prior to working at the SRCSD EL, Craig worked as a Laboratory Helper for 12 years at the Sacramento County Public Health Laboratory where he was responsible for preparing media, sterilizing media and instruments, and preparing specimens and reagents. In 1998 he promoted to Laboratory Assistant position and subsequently transferred to his current worksite as a SRCSD EL employee. Craig's current work assignments include sample collection, some conventional wet chemical procedures, sample processing, equipment monitoring, and maintaining the preparation room. In 1993 Craig earned a Bachelor's Degree in Human Behavior with special emphasis in Human Resources Management from National University, Sacramento. He also completed course requirements in 2003 for an Associate of Arts Degree in Computer Science from American River Jr. College.



4.6. **BIOLOGY SECTION**

- 4.6.1. **DON SCHWARTZ, Laboratory Supervisor, Biology Section** – Don Schwartz has been working in the environmental laboratory field for 23 years. He has had bench level experience in the inorganic chemical and

biological analysis of drinking water and water. Don has been the Biology Section supervisor of the Laboratory since 1998. As the section supervisor, Don oversees all of the Laboratory's bacteriological, microbiological, and toxicological work activities. He earned a Bachelor's Degree in Environmental and Systematic Biology in 1979 from California Polytechnic State University in San Luis Obispo.

- 4.6.2. **GISELA CLUSTER, Biologist** – Dr. Cluster joined the Biology Section in 2002 as an Environmental Laboratory Analyst. In December, 2003, she promoted to her current position in the SRCSD EL as a Biologist. Gisela graduated in 1988 with a Bachelor's Degree in Biology from the Eberhard-Karls University, Tübingen, Germany. In 1992 she earned her Ph. D. in Microbiology from the same institution. She spent two years as a post-doctoral scientist at the University of California, Davis, studying the role of complex lipids in nitrogen fixing symbioses of plants and bacteria. In 1996-97 she worked at the University of Kyoto, Japan, as a visiting scientist (JSPS Fellow). There she was part of a project characterizing the pathogenic relationship between a fungus and rice. During her years in academic research she worked mostly on bacterial membranes and cell walls, identifying, isolating and characterizing polysaccharides, lipids, and enzymes with a variety of methods.
- 4.6.3. **TONY COSTA, Biologist** – Mr. Costa received his Bachelor's Degree in Microbiology and Molecular Genetics, with minors in Chemistry and Philosophy, from California State University, Humboldt, in 1986. For the next seven years he was the lead chemist in the inorganic section of a private environmental laboratory (North Coast Laboratories) in Arcata, CA. In May of 2000 he received his Master's Degree from the University of Texas, Houston, with a major in Microbiology and Molecular Genetics. While at Texas, he worked as a research fellow on a project involving the bacterium *Bacillus anthracis*. He started work at the SRCSD EL in 2001 where his current duties include developing and conducting various methods for acute and chronic toxicity bioassays of water (ambient, industrial, and wastewater treatment plant effluent).
- 4.6.4. **Jane T. Adalid, Sr. Environmental Laboratory Analyst:** Jane Started working at SRCSD EL since 2008. She works in the Biology section as a lead worker providing support for various biological testing performed including but not limited to Bioassay, and various bacteriological testing. She obtained her AA degree in Industrial Chemistry in 1985 from Foundation University, Philippines. She has been working in the Environmental Laboratory testing since 1990. Prior to working at SRCSD EL Jane has performed various Laboratory testing including metal analyses using ICP, mercury testing, Selenium testing, organic testing using GC, WET chemistry, Bacteriological testing and Bioassays. She has a CWEA Grade 1 Laboratory certification.

- 4.6.5. **LEONORA ABELLANOSA, Sr. Environmental Laboratory Analyst** – Leonora has worked at the SRCSD EL since 1999. She started out in the Chemistry Section where she worked as an analyst for the trace mercury testing; did metals preparation (digestion and filtrations); assisted in cyanide analyses; assisted in or operated the GC analyzer for digester gas testing. While working at the SRCSD EL she received training and became proficient in a variety of inorganic and biological testing methods. Currently she is a Program Coordinator where she takes on the responsibility of coordinating customer program needs with laboratory resources and report preparation. Leonora began her career in environmental laboratories in 1978 working as a Chemist at the National Pollution Control Commission (a Philippine Government agency). Since then she has had bench experience as a principal operator of AAs, GFAAs, ICP, and performed a variety of inorganic analyses (BOD, TSS, TOC, IC, Cr(VI), TKN, etc.) From 1995 to 1998 she was a section supervisor in a private lab responsible for three chemists, three analysts, and a lab assistant.
- 4.6.6. **LOREN CHEW, Environmental Laboratory Analyst** – Loren Chew started work with the SRCSD EL in 2001. Prior to working here, he had 13 years experience working as a Laboratory Assistant at a medical laboratory (Physicians Clinical Laboratory) performing, processing, and testing of medical related samples in the Bacteriology Department. He also had 18 months experience as a Laboratory Technician at a veterinary laboratory (IDEXX Veterinary Services) performing IFA, ELISA, latex agglutination and immunodiffusion assays. Mr. Chew earned his Bachelor's Degree in Biological Conservation from California State University, Sacramento, in 1986.
- 4.6.7. **SHANNON FORRESTER, Environmental Laboratory Analyst** – Ms Forrester has a Bachelors degree in Physiology and Ecology. She started working here at SRCSD EL since 2004. Her work assignments include Bacteriology and toxicology testing. Her earlier work experience includes Metal Analyst for 2 years at Sequoia Analytical laboratory and Analyst at Air Toxics doing organic extraction for a year.
- 4.6.8. **MICHELLE PATE, Laboratory Assistant** – Michelle has been with SRCSD EL since 2005. She has a B.S degree in Biotechnology from UC Davis. She performs most of the Drinking water sampling. Her work assignments also include preparing culture media, performing bacteriological testing and helminth ova test.

APPENDIX 4. LABORATORY CERTIFICATION

CALIFORNIA STATE

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM BRANCH

CERTIFICATE OF ENVIRONMENTAL ACCREDITATION

Is hereby granted to

SRCSD ENVIRONMENTAL LABORATORY

8521 LAGUNA STATION ROAD
ELK GROVE, CA 95758-9550


Scope of the certificate is limited to the
"Fields of Testing"
which accompany this Certificate.

Continued accredited status depends on successful completion of on-site,
proficiency testing studies, and payment of applicable fees.

This Certificate is granted in accordance with provisions of
Section 100825, et seq. of the Health and Safety Code.

Certificate No.: 1100
Expiration Date: 01/31/2011
Effective Date: 01/01/2009

Richmond, California
subject to forfeiture or revocation


George C. Kulasingam, Ph.D., Chief
Environmental Laboratory Accreditation Program Branch

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**CALIFORNIA DEPARTMENT OF PUBLIC HEALTH
ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
Accredited Fields of Testing**

**SRCSD ENVIRONMENTAL LABORATORY**

Lab Phone (916) 875-9020

8521 LAGUNA STATION ROAD
ELK GROVE, CA 95758-9550

Certificate No: 1100 Renew Date: 01/31/2010

Field of Testing: 101 - Microbiology of Drinking Water

101.010	001	Heterotrophic Bacteria	SM9215B
101.011	001	Heterotrophic Bacteria	SimPlate
101.060	002	Total Coliform	SM9223
101.060	003	E. coli	SM9223
101.110	002	Total Coliform	m-ColiBlue24
101.110	003	E. coli	m-ColiBlue24
101.120	001	Total Coliform (Enumeration)	SM9221A,B,C
101.130	001	Fecal Coliform (Enumeration)	SM9221E (MTFEC)
101.131	001	Fecal Coliform (Enumeration)	SM9221E (A-1)
101.160	001	Total Coliform (Enumeration)	SM9223
101.200	001	E. coli (Enumeration)	SM9223B
101.210	001	E. coli (Enumeration)	SM9221B.1/SM9221F

Field of Testing: 102 - Inorganic Chemistry of Drinking Water

102.050	001	Cyanide	EPA 335.4
102.060	001	Nitrate calc.	EPA 353.2
102.061	001	Nitrite	EPA 353.2
102.070	001	Phosphate, Ortho	EPA 365.1
102.100	001	Alkalinity	SM2320B
102.120	001	Hardness	SM2340B
102.121	001	Hardness	SM2340C
102.130	001	Conductivity	SM2510B
102.140	001	Total Dissolved Solids	SM2540C
102.163	001	Chlorine, Free and Total	SM4500-Cl G
102.251	001	Sulfate	SM4500-SO4 E
102.260	001	Total Organic Carbon	SM5310B
102.261	001	DOC	SM5310B
102.270	001	Surfactants	SM5540C
102.280	001	UV254	SM5910B
102.520	001	Calcium	EPA 200.7
102.520	002	Magnesium	EPA 200.7
102.520	003	Potassium	EPA 200.7
102.520	004	Silica	EPA 200.7
102.520	005	Sodium	EPA 200.7
102.520	006	Hardness (calc.)	EPA 200.7
102.564	001	Cyanide	Quickchem 10-204-00-1-X

As of 10/23/2008, this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

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SRCSD ENVIRONMENTAL LABORATORY

Certificate No: 1100

Renew Date: 01/31/2010

Field of Testing: 103 - Toxic Chemical Elements of Drinking Water

103.130	003	Barium	EPA 200.7
103.130	004	Beryllium	EPA 200.7
103.130	005	Cadmium	EPA 200.7
103.130	007	Chromium	EPA 200.7
103.130	008	Copper	EPA 200.7
103.130	009	Iron	EPA 200.7
103.130	011	Manganese	EPA 200.7
103.130	012	Nickel	EPA 200.7
103.130	015	Silver	EPA 200.7
103.130	017	Zinc	EPA 200.7
103.130	018	Boron	EPA 200.7
103.140	001	Aluminum	EPA 200.8
103.140	002	Antimony	EPA 200.8
103.140	003	Arsenic	EPA 200.8
103.140	004	Barium	EPA 200.8
103.140	005	Beryllium	EPA 200.8
103.140	006	Cadmium	EPA 200.8
103.140	007	Chromium	EPA 200.8
103.140	008	Copper	EPA 200.8
103.140	009	Lead	EPA 200.8
103.140	010	Manganese	EPA 200.8
103.140	012	Nickel	EPA 200.8
103.140	013	Selenium	EPA 200.8
103.140	014	Silver	EPA 200.8
103.140	015	Thallium	EPA 200.8
103.140	016	Zinc	EPA 200.8
103.140	017	Boron	EPA 200.8
103.140	018	Vanadium	EPA 200.8
103.160	001	Mercury	EPA 245.1

Field of Testing: 107 - Microbiology of Wastewater

107.010	001	Heterotrophic Bacteria	SM9215B
107.020	001	Total Coliform	SM9221B
107.040	001	Fecal Coliform	SM9221C,E (MTF/EC)
107.041	001	Fecal Coliform	SM9221C,E (A-1)
107.060	001	Total Coliform	SM9222B
107.080	001	Fecal Coliform	SM9222D
107.100	001	Fecal Streptococci	SM9230B
107.100	002	Enterococci	SM9230B
107.242	001	Enterococci	Enterolert
107.245	001	E. coli	SM9223

Field of Testing: 108 - Inorganic Chemistry of Wastewater

108.020	001	Conductivity	EPA 120.1
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As of 10/23/2008, this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

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SRCSD ENVIRONMENTAL LABORATORY

Certificate No: 1100
Renew Date: 01/31/2010

108.090	001	Residue, Volatile	EPA 180.4
108.110	001	Turbidity	EPA 180.1
108.112	001	Boron	EPA 200.7
108.112	002	Calcium	EPA 200.7
108.112	003	Hardness (calc.)	EPA 200.7
108.112	004	Magnesium	EPA 200.7
108.112	005	Potassium	EPA 200.7
108.112	006	Silica	EPA 200.7
108.112	007	Sodium	EPA 200.7
108.113	001	Boron	EPA 200.8
108.113	002	Calcium	EPA 200.8
108.113	003	Magnesium	EPA 200.8
108.113	004	Potassium	EPA 200.8
108.113	006	Sodium	EPA 200.8
108.183	001	Cyanide, Total	EPA 335.4
108.200	001	Ammonia	EPA 350.1
108.211	001	Kjeldahl Nitrogen	EPA 351.2
108.232	001	Nitrate-nitrite	EPA 353.2
108.232	002	Nitrite	EPA 353.2
108.260	001	Phosphate, Ortho	EPA 365.1
108.266	001	Phosphorus, Total	EPA 365.4
108.410	001	Alkalinity	SM2320B
108.420	001	Hardness (calc.)	SM2340B
108.421	001	Hardness	SM2340C
108.440	001	Residue, Total	SM2540B
108.441	001	Residue, Filterable	SM2540C
108.442	001	Residue, Non-filterable	SM2540D
108.443	001	Residue, Settlesable	SM2540F
108.452	001	Chloride	SM4500-Cl-E
108.461	001	Chlorine	SM4500-Cl-C
108.465	001	Chlorine	SM4500-Cl-G
108.490	001	pH	SM4500-H+ B
108.531	001	Dissolved Oxygen	SM4500-O G
108.590	001	Biochemical Oxygen Demand	SM5210B
108.591	001	Carbonaceous BOD	SM5210B
108.610	001	Total Organic Carbon	SM5310B
108.640	001	Surfactants	SM5540C
108.660	001	Chemical Oxygen Demand	HACH8000
108.700	001	Sulfate	ASTM D516-90
108.926	001	Cyanide	Quickchem 10-204-00-1-X

Field of Testing: 109 - Toxic Chemical Elements of Wastewater

109.010	001	Aluminum	EPA 200.7
109.010	002	Antimony	EPA 200.7

As of 10/23/2008, this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

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SRCSD ENVIRONMENTAL LABORATORY

Certificate No: 1100
Renew Date: 01/31/2010

109.010	003	Arsenic	EPA 200.7
109.010	004	Barium	EPA 200.7
109.010	005	Beryllium	EPA 200.7
109.010	007	Cadmium	EPA 200.7
109.010	009	Chromium	EPA 200.7
109.010	010	Cobalt	EPA 200.7
109.010	010	Cobalt	EPA 200.7
109.010	011	Copper	EPA 200.7
109.010	012	Iron	EPA 200.7
109.010	013	Lead	EPA 200.7
109.010	015	Manganese	EPA 200.7
109.010	016	Molybdenum	EPA 200.7
109.010	017	Nickel	EPA 200.7
109.010	019	Selenium	EPA 200.7
109.010	021	Silver	EPA 200.7
109.010	023	Thallium	EPA 200.7
109.010	024	Tin	EPA 200.7
109.010	026	Vanadium	EPA 200.7
109.010	027	Zinc	EPA 200.7
109.020	001	Aluminum	EPA 200.8
109.020	002	Antimony	EPA 200.8
109.020	003	Arsenic	EPA 200.8
109.020	004	Barium	EPA 200.8
109.020	005	Beryllium	EPA 200.8
109.020	008	Cadmium	EPA 200.8
109.020	007	Chromium	EPA 200.8
109.020	008	Cobalt	EPA 200.8
109.020	009	Copper	EPA 200.8
109.020	010	Lead	EPA 200.8
109.020	011	Manganese	EPA 200.8
109.020	012	Molybdenum	EPA 200.8
109.020	013	Nickel	EPA 200.8
109.020	014	Selenium	EPA 200.8
109.020	015	Silver	EPA 200.8
109.020	016	Thallium	EPA 200.8
109.020	017	Vanadium	EPA 200.8
109.020	018	Zinc	EPA 200.8
109.020	021	Iron	EPA 200.8
109.020	022	Tin	EPA 200.8
109.020	023	Titanium	EPA 200.8
109.190	001	Mercury	EPA 245.1
109.361	001	Mercury	EPA 1631E

Field of Testing: 110 - Volatile Organic Chemistry of Wastewater

As of 10/23/2008, this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

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SRCSD ENVIRONMENTAL LABORATORY

Certificate No: 1100
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110.040	040	Halogenated Hydrocarbons	EPA 824
110.040	041	Aromatic Compounds	EPA 824
110.040	043	Other Volatile Organics	EPA 824

Field of Testing: 111 - Semi-volatile Organic Chemistry of Wastewater

111.101	032	Polynuclear Aromatic Hydrocarbons	EPA 825
111.101	034	Phthalates	EPA 825
111.101	036	Other Extractables	EPA 825
111.170	030	Organochlorine Pesticides	EPA 608
111.170	031	PCBs	EPA 608

Field of Testing: 113 - Whole Effluent Toxicity of Wastewater

113.010	001B	Fathead Minnow (P. promelas)	EPA 600/4-90/027F, Static Renewal
113.010	001C	Fathead Minnow (P. promelas)	EPA 600/4-90/027F, Continuous Flow
113.021	001B	Fathead Minnow (P. promelas)	EPA 2000 (EPA-821-R-02-012), Static Renewal
113.021	001C	Fathead Minnow (P. promelas)	EPA 2000 (EPA-821-R-02-012), Continuous Flow
113.040	001	Fathead Minnow (P. promelas)	EPA 1000 (EPA/600/4-91/002)
113.041	001	Fathead Minnow (P. promelas)	EPA 1000 (EPA-821-R-02-013)

Field of Testing: 114 - Inorganic Chemistry of Hazardous Waste

114.010	001	Antimony	EPA 60108
114.010	002	Arsenic	EPA 60108
114.010	003	Barium	EPA 60108
114.010	004	Beryllium	EPA 60108
114.010	005	Cadmium	EPA 60108
114.010	006	Chromium	EPA 60108
114.010	007	Cobalt	EPA 60108
114.010	008	Copper	EPA 60108
114.010	009	Lead	EPA 60108
114.010	010	Molybdenum	EPA 60108
114.010	011	Nickel	EPA 60108
114.010	012	Selenium	EPA 60108
114.010	013	Silver	EPA 60108
114.010	014	Thallium	EPA 60108
114.010	015	Vanadium	EPA 60108
114.010	016	Zinc	EPA 60108
114.020	001	Antimony	EPA 6020
114.020	002	Arsenic	EPA 6020
114.020	003	Barium	EPA 6020
114.020	004	Beryllium	EPA 6020
114.020	005	Cadmium	EPA 6020
114.020	006	Chromium	EPA 6020
114.020	007	Cobalt	EPA 6020
114.020	008	Copper	EPA 6020
114.020	009	Lead	EPA 6020
114.020	010	Molybdenum	EPA 6020

Aqueous only

As of 10/23/2008, this list supersedes all previous lists for this certificate number.
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SRCSD ENVIRONMENTAL LABORATORY**Certificate No: 1100****Renew Date: 01/31/2010**

114.020	011	Nickel	EPA 6020
114.020	012	Selenium	EPA 6020
114.020	013	Silver	EPA 6020
114.020	014	Thallium	EPA 6020
114.020	015	Vanadium	EPA 6020
114.020	016	Zinc	EPA 6020
114.025	001	Mercury	EPA 6020A
114.140	001	Mercury	EPA 7470A
114.141	001	Mercury	EPA 7471A

Field of Testing: 116 - Volatile Organic Chemistry of Hazardous Waste

116.080	000	Volatile Organic Compounds	EPA 8260B	Aqueous only
116.080	120	Oxygenates	EPA 8260B	Aqueous only

Field of Testing: 117 - Semi-volatile Organic Chemistry of Hazardous Waste

117.110	000	Extractable Organics	EPA 8270C	Aqueous only
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As of 10/23/2008, this list supersedes all previous lists for this certificate number.
 Customers: Please verify the current accreditation standing with the State.

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APPENDIX 5. CORRECTIVE ACTION REPORT FORM



**WATER QUALITY CONTROL LAB
CORRECTIVE ACTION REQUEST (CAR) FORM # 0511001**

1. Person Initiating: _____ Date: _____
2. Section Assigned to: _____
3. Section Supervisor: _____ Assigned To: _____
4. Completion Due Date: _____ If Other, Specify time: _____
5. Problem Identification: _____

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Attach additional sheet if needed.

6. Problem Identified by: _____
7. Is this a repeat problem? _____ If yes, previous CAR # reference: _____
8. Root Cause: *Why the issue originally occurred.* _____

Attach additional sheet if needed.

9. Corrective Action Taken: *What was done to correct the issue.*

--

Attach additional sheet if needed.

10. Completed by: _____ Date: _____
11. Completed & returned to Supervisor- Date: _____
12. Reviewed & Approved by: _____



**WATER QUALITY CONTROL LAB
CORRECTIVE ACTION REQUEST (CAR) FORM # 0511001**

Supervisor : _____

Date: _____

QA Officer : _____

Date: _____

Lab Manager: _____

Date: _____

13. File Date: _____

14. Comments:

EXAMPLE CHAIN-OF CUSTODY FORM

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APPENDIX 7. ROUTING LIST EXAMPLE

Sacramento Regional County Sanitation District
Water Quality Control Laboratory
8521 Laguna Station Road, Elk Grove, CA 95758

Sample Collection Routing List**EQS Run-1 Sampler**

For Thursday, April 12, 2007

Lab Sample	Mode	Sample Name	Sampled By	Sample Date/Time
0704120032	GRAB	SE Channel North grab (0472)	_____	_____
0704120033	GRAB	SE Channel South grab (0473)	_____	_____
0704120011	SAMP	Influent Grab 2 (0102-2)	_____	_____
0704120012	GRAB	PE Grab (0302)	_____	_____

Analyst Read / Date

Approved By / Date

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