QUALITY ASSURANCE PROGRAM

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FOREWARD

The role of the analytical laboratory is to provide qualitative and quantitative data to be used in decision making. To be valuable, the data must accurately describe the characteristics or the concentration of constituents in the sample submitted to the laboratory.

Due to the importance of laboratory analyses and the resulting actions which they produce, a program to insure the reliability of the data is essential, thus the establishment of a routine control program is applied to all aspects of the laboratory to assure the reliability of the results produced - The Quality Assurance/Quality Control Program.

The program has two primary functions. First, the program should monitor the reliability (truth) of the results, "measurement of quality". Secondly, control of quality in order to meet requirements of reliability.

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SIERRA ANALYTICAL LABORATORIES, INC.

QUALITY ASSURANCE POLICY

1. QUALITY ASSURANCE OBJECTIVES

Sierra Analytical Laboratories, Inc. is committed to providing quality analytical results to our clients. The policy at Sierra is to provide QA support to satisfy the six primary data quality objectives (DQO's) established by USEPA in 1980. These DQO's are: precision, accuracy, representatives, completeness, comparability, and detectability. In addition, it is our policy to demonstrate that all data collected and processed is scientifically valid, defensible, and accurate.

1.1 Precision and Accuracy

Precision is an estimate of variability; that is, an estimate of agreement among individual measurements of the same physical and chemical property, under prescribed similar conditions. Precision is expressed as relative standard deviation (RDS). Accuracy is the degree of agreement between a measurement and the true or expected value or between the average of a number of measurements and the true or expected value. For chemical properties, accuracy is expressed either as a percent recovery or as a percent bias.

Precision and accuracy are determined in part by analyzing data from quality assessment and quality control samples. The precision and accuracy of results shall be examined to ensure compliance of laboratory procedures with the contract requirements and good laboratory practices.

1.2 Representativeness

Provisions for sampling, sampling handling, and measurement are to be made to ensure that samples remain representative of the population or condition being measured.

1.3 Completeness

Provisions for equipment, availability, maintenance, information handling, analytical methods, etc., are to be made to ensure that all intended measurements are completed.

1.4 Comparability

Uniform procedures and a uniform set of units for collecting, analyzing, and reporting data are to be used to allow comparability of data bases and other organizations reporting similar data.

1.5 Detectability

Detectability refers to the minimum concentration of an analyte that can be measured with a stated level of confidence. It is determined by assessing the variability of replicate measurements at zero or near zero analyte concentration. The limit of detection is the point at which a measured value becomes believable; i.e. the point at which the value is larger than the uncertainty associated with it. This point is defined arbitrarily as 3s. The limit of quantitation is the lowest level at which measurements become quantitatively meaningful and is defined as 10s.

1.6 Applicability of QA Plan

This QA Plan is applicable to all projects in which Sierra is involved with.

2. FACILITY, EQUIPMENT, AND MATERIALS

2.1 Facility and Equipment

Sierra Analytical Laboratories, Inc. is located in Laguna Hills, California, and maintains a 10,000 square foot facility that houses our stationary and mobile laboratory. The laboratory is divided into five main departments: Metals, Organics, Wet Chem, Microbiology, and Mobile Laboratory. A floor diagram of the facility is enclosed in the appendices.

Sierra is fully equipped and staffed to meet the needs of our clients. A list of equipment is outlined in the appendices.

2.2 Material Procurement and Control

Supplies Management

To assure the quality of supplies used for various laboratory analyses, the following items are taken into account:

Materials, reagents, standard, solvent, and gases are carefully selected to meet specifications defined in the method analyses. Each new supply of these items is verified for their performance capabilities, freedom from impurities that interfere with the analysis, and background levels measured to check the degree of contamination. Solvent is tested for impurities whenever a new lot number is used.

Primary standards are obtained from a reliable, certifiable source, and of highest purity. Inorganic and organic analytical standards used for instrument/methodological calibration and preparation of QC samples are traceable to EPA standards and/or standard reference materials.

Materials are dated upon receipt to establish their order of use, “as first in, first out basis,” and to minimize the possibility of exceeding their shelf- life. Pertinent information such as name of supplier, lot number, expiration date, concentration, date opened, and date of preparation is documented in the standard preparation logbook.

Stock and working standards solutions are prepared fresh as required by their stability. These are checked for signs of deterioration (e.g., formation of precipitates,

discoloration, and changes of concentration through calibration results). Standard solutions are properly labeled as to name of solution, concentration, solvent, date of preparation, and preparer. Standard preparation is documented in the standard preparation logbook. These items are stored in places where they are protected from degradation and contamination.

Acids and bases are segregated in terms of storage. Various types of solvent are stored in flammable storage cabinets. Dry chemicals used for inorganic and organic analyses are stored in the chemical storage cabinet. Primary standards and working standards prepared for organic analysis are stored in the standard refrigerator/freezer.

Services such as electricity, water, air, gas, and vacuum are checked for proper specifications for efficient and reliable performance of the instruments.

Equipment Management

Information on the actual performance of the equipment is obtained before purchase request for a piece of equipment is made. The availability of the supplier's service to install and test it against specifications as part of purchase price is also considered. When first installed, an internal calibration of the instrument is performed using the manufacturer's manual. Analytical reference standards are analyzed for qualitative and quantitative checks on the instrument performance during the sample run. Routine preventive maintenance of the instruments/or equipment is done on a regular scheduled basis.

Preventive Maintenance Activities and Schedules

Instruments are maintained according to the Standard Operating Procedures using the manufacturer documentation. Repairs are conducted as needed, either by manufacturer representatives or by in-house personnel. Routine maintenance (lamp replacement, etc.) is conducted as needed to maintain instrument integrity.

Critical equipment and instrumentation are maintained on a scheduled basis to minimize the downtime of measurement systems. Maintenance logbooks are kept for each instrument. The logbooks must contain at a minimum: title page with equipment name manufacturer, date of acquisition, serial number, and reference to manuals provided with the equipment. Each entry must contain at a minimum: date, initials of person doing maintenance, and description of action taken.

Waste Disposal

Laboratory generated wastes are classified into various waste streams and are disposed according to the local, state, and federal regulations.

3. LABORATORY ORGANIZATION AND STAFF RESPONSIBILITIES

3.1 Introduction

Sierra was established in 1998 as an environmental stationary and mobile analytical laboratory which provides services to industries, private citizens, consultants, and Federal, State and Local Governments in a variety of technical areas. The technical areas served include: chemical and biological analyses, as well as monitoring programs for drinking water, surface water, groundwater, soil, hazardous waste, and effluent discharges.

3.2 Quality Assurance Policy

A laboratory which serves a wide variety of industries encounters many different sample types and matrices. In order to ensure validity of data regardless of the matrix encountered, Sierra has developed and implemented a stringent Quality Control program. The program was derived from guidelines established by both the Environmental Protection Agency and the California Department of Health Services Environmental Laboratory Accreditation Program.

Sierra is dedicated to reporting sound, viable, scientific data, regardless of the size or scope of the project. In order to fulfill this goal, Sierra has devised a QC program for each analyst to follow when generating and reporting data. This QC program yields data, which are reproducible and traceable to certified standards, as well as being legally defensible. All methods of analyses performed at Sierra undergo the appropriate quality control procedures which include: analysis of laboratory control standards, standard reference material, matrix spikes, surrogates, duplicates, method blanks, calibration verification, and data confirmation where applicable. All data is reported at the level of precision and accuracy defined in this manual, unless otherwise requested by the client being served. The steps documented in this manual present the end user of the data a definable set of limits to ensure the completeness and accuracy of the report generated. The Quality Assurance Policy has the complete support of Management.

3.3 Goals

Sierra's goal is to provide high quality customer service, as well as a legally defensible data packet at a reasonable cost to the end user.

3.4 Laboratory Staff

Sierra presently maintains a staff of eighteen (18), with the key personnel representing at least 75 years of experience in the environmental laboratory field. The resumes are available upon request.

The implementation of the Quality Assurance Plan is achieved through a laboratory-wide effort, which includes the entire staff. Sierra's organizational structure and personnel are geared toward meeting the goals and objectives of the Quality Assurance and Quality Control Plan.

The responsibility for the quality of all laboratory services resides with the Laboratory Director. To assist the Laboratory Director in maintaining quality services, the laboratory QA Coordinator coordinates the laboratory's QA activities and initiate efforts to provide quality control parameters. To promote objectivity in the management and the operation of the QA program, all management and supervisory personnel in the laboratory are directly involved in the QA program. This team work ensures laboratory operations adhere to generally accepted quality control practices which are designed to provide an analytical product of known, documented, and acceptable quality.

Laboratory Director

The Director is responsible for the overall operation of the laboratory. The responsibilities of the Director include personnel selection, client contact, development and upgrading of all physical facilities, methodologies, and equipment. In addition, the Laboratory Director is directly involved in all financial decisions, checking and maintaining laboratory quality assurance policy, the laboratory information management system, and all other operations in the laboratory.

Quality Assurance Manager

The Quality Assurance Manager reports directly to the Laboratory Director. The Quality Assurance Manager directs the QA Program of the laboratory. The duties of the QA manager include the development and upgrading of QA/QC procedures and manuals, incorporating Quality Control Procedures into the Laboratory's Standard Operating Procedures (SOP's), diagnosing quality control defects, resolving problems with the analytical QC process, as well as assisting in the QC training program for each area to ensure a cohesive, well-managed QA/QC program.

Client Services Manager

The Client Services Manager serves as a liaison between the clients and the laboratory staff. Any questions regarding sample reports, data, sampling procedures, appropriate sample containers and pricing can be addressed to the Client Services Manager.

Metals Department Supervisor

The Metals Department Supervisor is responsible for the day to day operation within the metals area. The Supervisor schedules, evaluates, and trains personnel on instrumentation and methods, and is the primary chemist for method development in this area.

Inorganics Department Supervisor

The Inorganics Department Supervisor is responsible for the day to day operation within the wet chemistry, bacteriological and toxicity areas. The Supervisor schedules, evaluates and trains personnel on instrumentation and methods, and acts as the primary chemist for method development in these areas.

GCMS/Volatile and Semi Volatile (Extractables) Organics Department Supervisor

The GCMS/Volatile and Semi Volatile (Extractables) Organics Department Supervisor is responsible for the day to day operation within both the GCMS and the other GC areas. The Supervisor schedules, evaluates, and trains personnel on the instrumentation and methods, and acts as the primary chemist for method development within these areas.

Analyst/Technician

The Analyst/Technician performs analytical tests and data recording according to client's technical requirements or lab standard operation procedures. The analyst also performs and documents calibration and preventive maintenance of instruments, as necessary, and reduces analytical data and validates data. It is also the analyst's responsibility to report out-of-control situations, instrument malfunctions, calibration failure, or other non-conformance to lab supervisor or lab management.

3.5 Training

The in-house training program consists of many steps. A general laboratory tour is given to all new employees. Safety procedures and guidelines are discussed by the laboratory director. The method analysis is briefly discussed and an over-all description of the test is given. The published method of analysis is given to the individual to read prior to learning the test. The published method is reviewed with the individual, and any in-house changes are also reviewed. QA/QC information and procedures are reviewed. MSDS sheets are reviewed with the individual. The individual observes an experienced analyst perform the test. Blanks, calibration standards and control standards are analyzed by the trainee. Results obtained are compared to established data. If results are satisfactory, comparison and reference samples are analyzed. If results are acceptable, the trainee is allowed to analyze samples independently.

3.6 Staff Responsibilities

In order to ensure that a cohesive, well run Quality Control Program is carried out, all personnel within the laboratory must be involved. Analysts are responsible for analyzing samples utilizing Standard Operating Procedures. The analyst is also responsible for following the proper quality control parameters outlined in this manual. The management staff is responsible for ensuring that analysts are conforming to these guidelines when performing the analyses.

4. QUALITY ASSURANCE OBJECTIVES

The primary quality assurance objectives followed when generating laboratory data are: precision, accuracy, integrity, and documentation. When these objectives are followed, the laboratory can ensure that the data generated meet the data quality objectives set by both the laboratory as well as each individual client.

4.1 Precision

Precision refers to the measure of the degree of agreement among replicate analysis of a sample, usually expressed as the standard deviation. If the precision is poor, it can be assumed that the associated data will be different from the true value.

4.2 Accuracy

Accuracy refers to the combination of biasness and precision of an analytical procedure, which reflects the closeness of a measured value to a true value. The measurement of any analytical bias due to systematic errors encountered along with the precision data will show overall accuracy of the data.

4.3 Integrity

Integrity refers to the overall completeness, comparability, and representation of the data. Good data integrity is shown when the data has passed all of the quality control policies stated in this manual. Any data that has exceeded the quality control parameters should be reanalyzed if still within holding time, and a representative sample is still available. Integrity also implies that the data is comparable and consistent, having been generated according to SOP, analyzed within holding time, and the subsample analyzed is representative of the entire sample.

4.4 Documentation

Clear and complete documentation is essential in supporting data integrity. Documentation starts during the field sampling and/or sample receiving process and is carried through the analytical procedures and finally through the final report. All processes are documented on a preprinted form, in a bound notebook, or in the Laboratory Information Management System (LIMS).

5. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

One of the most important parts of the entire analytical process is correct sample collection, handling, and storage. The proper container, preservative, handling procedure, and sampling technique will ensure that the sample is not contaminated nor degraded.

5.1 Sample Collection

It is very important to follow proper sample collection protocol. The EPA, DOHS, and other regulatory agencies have set up standards to follow when sampling. Good representation of the sampling area is vital for ensuring that the sample collection procedure has fulfilled the sampling plan for the project. Sierra has in-house, 40-hour OSHA trained personnel available for field sampling. However, many clients perform their own sampling. Sierra can provide various types of sampling utilizing ISCO equipment.

5.2 Sample Containers

Upon request, Sierra will supply the correct sampling containers, cooler with blue ice, chain-of-custody forms, and preservatives. Refer to the preservation/holdtime list in the Appendix for the correct containers. All of the sample containers used at Sierra for all analyses are purchased precleaned, and no containers are reused.

5.3 Sample Preservation

For most parameters it is more advantageous to preserve the sample when collected rather than after relinquishing the sample to the laboratory. For parameters that may degrade by chemical, physical, or bacteriological means, it is important to preserve the samples as soon as possible. Refer to the Preservation/Holdtime List in the Appendix for the proper preservation or Sierra's brochure which outlines all of the above.

5.4 Sample Storage

Samples for Inorganic, Organic, and Bacteriological analyses are kept at $4 \pm 2°C$. All samples are retained for 30 days past the report generation date except when specifically requested by the client.

6. SAMPLE CUSTODY

6.1 Sample Receiving and Log-In

The Sample Receiving department is notified when samples are expected to arrive. If samples are expected to arrive after 5 p.m. Monday through Friday, or during the weekend, special arrangements are made with the Client Manager and the Sample Custodian (or a designated alternate) to receive the samples. Samples which arrive in the laboratory are received according to the following procedure. Sample receiving protocol is outlined in the Appendix.

6.1.1 Shipping Container
The shipping container should be opened with caution if samples are suspected of containing high concentrations of hazardous materials. If possible, use a well ventilated area or fume hood.

6.1.2 Temperature
Upon receipt, the temperature of the sample containers is taken. Proper techniques should be followed in order to minimize the possibility of contamination to the samples. The temperature should be documented on the Chain-of-Custody form.

6.1.3 Inventory Sample Documents

The shipping container is opened and inventoried by the Sample Custodian according to the accompanying Chain-of-Custody form. If custody seals are in place on the samples, their condition is examined and documented on the Chain-of-Custody form and/or the shipping manifest, and the entry initialed. These Chain-of-Custody forms and/or shipping manifests are retained in the client's file.

6.1.4 Sample Condition

The samples are removed from the shipping container by the Sample Custodian and the condition of the samples documented on the Chain-of-Custody form. (I.e., intact, broken, leaking, properly iced, etc.)

6.1.5 Presence of Sample Identification Labels

Sample identification numbers are recorded on the Chain-of-Custody form. A single sample may arrive in more than one container (water samples, in particular, may have more than one container for one sample.) All samples which have similar identification numbers must be grouped together and recorded as one number. Samples are recorded in ascending numerical order. Sample identification labels are compared with the Chain-of-Custody record. All information (sample location, project code, analyses required, preservative, etc.) recorded on the Chain-of-Custody are checked to see that they are in agreement with the sample identification labels. Any discrepancies are documented.

6.2 Agreement of Documentation

Information contained on the Chain-of-Custody records, sample labels, and shipping manifests (if present) are compared and verified by the Sample Custodian. Any discrepancies found on the Chain-of-Custody form are documented in the comments section. If discrepancies are found, the Laboratory Director or Client Manager is notified so that the client can be contacted immediately for clarification.

6.3 Sign-Off

If all samples recorded on the Chain-of-Custody record are received by the Laboratory, and there are no problems observed with the sample shipment, the Sample Custodian signs the correct date and time of accepting custody of samples on the Chain-of -Custody record in the "received for laboratory by" box on the document. If problems are discovered, the shipment is signed for and the problems are noted in the comment box. The Client Manager is notified of any problems immediately, so the client may be contacted. Sierra utilizes a notification form in the case of any problems in sample receiving.

6.4 Sample Log-In

After samples and containers are inspected and observations recorded, the samples are logged-in by the Sample Custodian using the sample receiving logbook. Each sample will receive a separate laboratory number. A Sample Receipt is given to the client (copy of C.O.C.). The original C.O.C. is retained by the Sample Custodian with the sample (s). The appropriate sample preparation personnel are then notified that the samples have been received. Upon completion of the project, the original laboratory reports are sent to the client while a copy of the C.O.C. and report are retained in the client files.

6.5 Internal Chain-of-Custody

A copy of the C.O.C. record is generated and distributed to the appropriate departments by the Sample Custodian. The copied C.O.C. is the work order on the submitted samples.

6.6 Sample Tracking (Laboratory Information Management System)

All samples received by Sierra are entered into the LIMS.
This system will contain the following information: Sierra Project No., Sierra Sample No., Client Name, Client Project No., Date/Time Sampled, Sample Collector, Date/Time Received, Name of person who logged in samples, Sample Matrix, Analyses, and Turn Around Time.

6.7 Number System

All samples are entered into the LIMS when they are received. A unique *Work Order Number* is assigned to the sample delivery group and a *Sample Number* is assigned to each sample within the sample delivery group. The Work Order Number will incorporate the year and month of receipt and a sequential three-digit serial number indicating the order of receipt within each month. The format of the Work Order Number is YYMM###. (The Work Order Number is automatically assigned by Element whenever a new work order is "Added" or "Copied" into the LIMS system).

Example 1: The first project received in October 2002 would be assigned the Work Order Number 0210001.

Example 2: The two hundred twentieth project received in June 2003 would be assigned the Work Order Number 0306220.

The *Sample Number* for each sample will incorporate the Work Order Number followed by a hyphen and a sequential two digit serial number assigned to each sample in the project. Sample Numbers reset with each new Work Order. The format of the complete Sample Number is YYMM###-##. Please note that the Sample Number is distinct and different from the *Sample Name* which is the sample identification assigned by the Client. (The Sample Number is automatically assigned by Element whenever a new sample is "Added" or "Copied" into the LIMS system).

Example 3: The first sample of the twentieth project received in December 2002 would be assigned the Sample Number 0212020-01.

Example 4: The sixth sample of the one hundred thirteenth project received in March 2003 would be assigned the Sample Number 0303130-06.

6.8 Post Analysis Storage

All samples are kept in the proper environmental control until after holding times have expired and there are no QA/QC problems with any analysis on that sample. If reanalysis is not anticipated, environmental conditions will not be observed and samples will be stored at room temperature.

After analysis is complete and no further analysis is required, the analyst is responsible for placing the sample into the long term storage area. Samples remain in the long term storage for 30 days after data reporting and are then disposed. Samples are stored for longer periods of time if specified by the customer or if they are legal samples.

7. CALIBRATION PROCEDURES AND FREQUENCY

Instrument calibration is performed to ensure that the instrument is functionally correct and that the instrument sensitivity is at an appropriate level to achieve the method detection limits. All instrumentation is calibrated using analytical standards at a concentration level to effectively fit the instrument's linear range.

7.1 Instrument Calibration

7.1.1 GC/MS Calibration

Daily calibration of the instrument begins with a sensitivity check with (BFB) for volatile compounds and (DFTPP) for semi-volatile compounds. This must be performed daily, or every 12 hours of analysis. If the ion abundance criteria is not met the instrument must be retuned, and the sensitivity check repeated (See 7.3). A 50 mg/l. 624 standard is analyzed for volatile analysis if the criteria for the instrument tuning is met. This standard includes all of the 624 target compounds plus the surrogate and internal standard. The System performance Check Compounds (SPCCs) must have a minimum response factor $\geq$ 0.30, and Bromoform must have a minimum response factor of $\geq$ 0.25. In addition, the Continuing Calibration Check (CCCs) may only have a maxiumum % RSD $\leq$ 30%. For semi-volatile compounds a 50 mg/l 625 standard is analyzed. This standard includes all of the 625 target compounds plus the surrogate and internal standard. The SPCCs must have a minimum response of $\geq$ 0.050. The CCCs can have maxiumum %RSD of $\leq$ 30%. A five point calibration is performed every 6 months, or more frequently if major work is performed on the instrument.

The calibration points are specified in each method S.O.P. and as a rule, the lowest calibration point will be set at the Reporting Limit to ensure that results reports down to that limit are within the instrument's linear range.

7.1.1.1 GC/MS Ion Abundance Criteria

Bromfluorobenzene (BFB)

Mass	Ion Abundance Criteria
50	15-40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	Less than 1% of mass 95
174	Greater than 50% of mass 95
175	5-9% of mass 174
176	95-101% of mass 174
177	5-9% of mass 176

Decafluorotriphenylphospine (DFTPP)

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	less than 2% of mass 69
69	reference only
70	less than 2% of mass 69
127	40-60% of mass 198
197	less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198

365	greater than 1% of mass 198
441	0-100% of mass 443
442	greater than 40% of mass 198
443	17-23% of mass 442

7.1.2 GC Calibration

All 600 series methods require a working three point calibration curve, 8000 series methods require a 5 point calibration curve. The 500 series methods should have a 3 to 5 point curve but a 1 point calibration is a viable alternative as long as the response in the sample is within 20% of the response in the standard.

The calibration curve is checked for linearity. If a point-slope equation is used to calculate analyte concentrations, then the correlation coefficient acceptance limit is $\geq$0.995. If an average response factor is used to calculate analyte concentrations, then the % RSD acceptance limit is $\leq$ 10 to 20% depending on individual method requirements.

A daily calibration verification standard may be used to document the validity of a working calibration curve. The acceptance limit for this daily standard is generally $\pm$15% but is method dependent (502.2 is $\pm$20% and 601/602 has individual compound requirements). A new calibration curve may be performed daily.

Retention time windows are calculated as $\pm$3 standard deviations of a mid point standard analyzed three times over a 72 hour period, or may be calculated as $\pm$ standard deviations of the retention times of standards throughout the course of the analytical run.

A new calibration curve is performed every 6 months or more frequently if required.

The calibration points are specified in each method S.O.P. and as a rule, the lowest calibration point will be set at the Reporting Limit to ensure that results reports down to that limit are within the instrument's linear range.

7.1.3 ICP Standardization

The ICP standardization is updated daily according to the specifications in the instrument manual. The standardization consists of analyzing a calibration blank and a high standard. A Continuing Calibration Verification Standard (CCV) is analyzed immediately following the standardization update. This standard must fall within $\pm$ 15% of the calibration curve.

7.2 Calibration Standards

All standards used for calibration are ACS reagent grade or better. Standards certified by the National Institute for Standards and Technology (NIST) are used whenever possible. Standard logbooks are maintained in the laboratory.

7.3 Corrective Action

If the calibration criteria for any of the above instrumentation is not met, the calibration is repeated. If the second attempt does not pass, new calibration standards are then made. If the

calibration criteria is not met after making new standards, the analyst troubleshoots the instrument. If this fails, the service representative is contacted. If the continuing calibration verification is outside the acceptable criteria, the samples which are bracketed between the standard and the standard analyzed previously which passed are to be reanalyzed. This corrective action procedure generally applies to any instrument. However, some methods have specific requirements. Please refer to the appropriate method.

8. ANALYTICAL PROCEDURES

8.1 Method Sources

Most of the methods performed at Sierra originate from regulatory agencies. The methods used are specified by the United State Environmental Protection Agency, California Department of Health Services, various local agencies, as well as professional organizations. The following reference are used at Sierra:

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, 40 CFR, Part 136.

U.S. Environmental Protection Agency, *Methods for Chemical Analysis of Water and Wastes,* EPA-600/4-79-020, March 1983.

U.S. Environmental Protection Agency, *Methods for the Determination of Inorganic Substances in Environmental Samples,* EPA-600/R-93/1 00, August 1993.

U.S. Environmental Protection Agency, *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater,* EPA-600/4-82-057, July, 1982.

U.S. Environmental Protection Agency, *Methods for the Determination of Organic compounds in Drinking Water,* EPA-600/4-881039, December 1988.

U.S. Environmental Protection Agency, *Methods for the Determination of Organic Compounds in Drinking Water, Supplement I,* EPA-600/4-90/020, July 1990.

U.S. Environmental Protection Agency, *Methods for the Determination of Organic compounds in Drinking Water, Supplement II,* EPA-600/R-92/1 29, August 1992.

U.S. Environmental Protection Agency, *Methods for the Determination of Organic Compounds in Drinking Water, Supplement Ill,* EPA-6001R-95/131, August 1995.

U.S. Environmental Protection Agency. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd. Edition. 1992.

U.S. Environmental Protection Agency, *Methods for the Determination of Metals in Environmental Samples,* EPN6OOI4-91-010, June 1991.

U.S. Environmental Protection Agency, *Methods for the Determination of Metals in*

Environmental Samples, Supplement I, EPA-6001R-94/1 11, May 1994.

U.S. Environmental Protection Agency, *Procedures for Handling and Chemical Analysis of Sediment of Water Samples,* EPA/CE-81-1, May, 1981.

U.S. Environmental Protection Agency, *Microbiological Methods for Monitoring the Environment; Water and Wastes,* EPA6OO/8-78/017, December$_1$ 1978.

APHA-AWWA-WEF, *Standard Methods for The Examination of Water and Wastewater, 18th and 19th Editions*

Office of Administrative Hearings, Department of General Services, *Criteria for identification of Hazardous and Extremely Hazardous Wastes,* Article II, Title 22, Section 66696, California Code of Regulations

9. DATA REDUCTION, VALIDATION AND REPORTING

9.1 Data Reduction

Data reduction is the procedure in which the raw data from the analysis is transferred into reportable data. This includes the comparison of the raw data to calibration data, as well as the conversion of any digestion, extraction, or dilution factors. Data reduction is the primary responsibility of the analyst who performs the analysis.

9.2 Data Validation

After an analyst has completed an analysis and has reduced the data, the raw data and quality control data are then validated.

9.2..1 Analyst Review

The Analyst who generates the data has the primary responsibility to deem the data correct and complete. Each analyst is to review his/her data using the following guidelines and ensure that these guidelines are adhered to: sample preparation information is documented and complete (i.e., sample preparation sheets, log, etc.) all analysis information is documented and complete (i.e. run logs, analysis worksheets, etc.), the SOP has been followed and any deviations are documented, the data results are correct and complete, the quality control data is in compliance, the method blank is in compliance, the preparation and analysis holding times are met, and all of the documentation is initialed and dated.

9.2.2 Peer/Supervisor Review

Another analyst or supervisor will review all of the QC data calculations, as well as 10% of the sample calculations. In addition to the parameters listed in 9.2.1, the reviewer will check: that the calibration data is acceptable, and that the quantitative results and calculations are correct. If any of the criteria is not met, corrective action should be taken (See Section 14.0). If the sample can be reanalyzed it is done at this time, if not, the supervisor, in conjunction with the analyst, shall document the corrective action taken and state justification for validating the data. The client is contacted if there are any problems with the analysis and/or data, to determine if additional sampling or other actions will be

needed. Out-of-control QC information will be included in a case narrative. If any problems are found in the sample data review, an additional 10% of the samples are to be checked. This process will continue until no errors are detected or until the entire data package has been checked. The data is then validated in the LIMS by the Department Supervisor, or a Senior Analyst qualified to do so.

9.3 Reporting

9.3.1 Preliminary Report

A preliminary report is generated through the analyst and/or department supervisor, a secretary, or a member of upper management authorized to do so. The preliminary report is screened. This review is to ensure that the data is complete and correct. The following items will be checked: transcription and typographical errors from the chain of custody, documentation of any out-of-control situations or special problems, client information (i.e., project #, contract #, P.O#, location, etc.) is properly documented, verification of overall accuracy and reasonableness of data, and checking the report format for clarity.

9.3.2 Final Report

The final report format is reviewed and signed by the Data Review manager and/or the Laboratory Director.

9.3.3 Report Reviews

There are periodic reviews of the complete report package as well as the raw data associated with the report (chromatograms, calibration curves, etc.) by the Quality Assurance Manager, or another member of the management staff.

9.4 Data Storage

The data for the current year as well as the previous five years are maintained at the lab or at an off-site storage unit.

10. INTERNAL QUALITY CONTROL CHECKS

Sierra routinely performs internal quality control checks with each batch of analyses. The quality control check samples are used to determine: if the system is “in control”, if there is any matrix effect with the sample and if any variables associated with the field sampling have a bearing on the data.

10.1 Method Performance Check Samples

10.1.1 Laboratory Control Sample (LCS) Frequency - Every Prep Batch

The LCS is an aqueous or solid matrix sample which is taken through the entire sample preparation process and analysis. The LCS consists of a known amount of analyte spiked into a blank. The LCS, in most cases, is a certified standard from a second manufacturer other than the calibration standard source. The

purpose of the LCS is to asses method performance. If the results for the LCS fall out of the control limits, the analysis is terminated and the problem is investigated. If the problem occurred during sample preparation, the sample batch associated with the LCS is redigested, reextracted, or redistilled, whichever is applicable, then reanalyzed.

10.1.2 Standard Reference Sample/Material (SRS/SRM) - Every Batch

The SRS/SRM is a known standard which is not taken through the whole sample preparation process. It is prepared from a source different from the calibration standard. It is used to verify the accuracy of the calibration curve and the instrument performance. An SRS/SRM is analyzed with every batch of samples analyzed in the Metals department. The Inorganics department analyzes an SRS/SRM with each batch of the analyses which do not require the calibration curve to be processed in the same manner as the samples (i.e.: Phenols, Cyanides and Ammonia.)

10.1.3. Method Blank (MB) Frequency - Every Prep Batch

At least one method blank is taken through the entire sample preparation process for each analysis performed. The method blank consists of deionized water (for aqueous sample determination), or clean sand (for solid sample determination) which is processed through the entire method. This data from the method blank is used to determine if there is any contamination within the analytical process.

10.2 Matrix Specific Check Samples

10.2.1 Matrix Spike (MS) Frequency - One Every 10 Samples

The Matrix Spike consists of a sample aliquot spiked with a known amount of analyte(s) to be tested. In most cases, the MS is a certified standard taken from a different manufacturer or lot number other than the calibration curve. (The same source can be used for both the LCS and the MS/MSD). The sample is spiked prior to the preparation of the sample, then the sample is processed through the entire preparation procedure and analyzed. The purpose of the matrix spike is to assess matrix problems (interferences). The matrix spike recovery is calculated. This value is an indicator of the effect of the sample matrix during the analysis. Typically, analyte(s) are added at the midrange concentration of the calibration curve.

10.2.2 Duplicates Frequency - One Every 20 Samples

Duplicate analyses are used to determine analytical precision in the analysis by calculating a Relative Percent Difference (RPD). Since no precision data can be calculated when one or both of the sample duplicates are non-detected, Matrix Spike Duplicate (MSD) is used for precision data calculation in Metals, Inorganic and Organic analyses. A MSD consists of a separate aliquot of the same sample as the matrix spike. It is prepared and analyzed in the same manner as the matrix spike. A Laboratory Control Sample Duplicate is analyzed for precision data calculation when there is insufficient sample volume to analyze MS/MSD samples.

10.2.3 Surrogates Frequency - Every Sample

Surrogates are organic compounds which are similar to the compounds of interest in chemical structure and behavior, but are not normally found in the samples being analyzed. Surrogates are used to monitor any matrix effect within the analysis. Surrogates are added to samples for most of the GC and GC/MS methods. Surrogates are not used to determine whether a sample batch is in control, but they may be used to indicate if single samples within the batch need reanalysis. When low surrogate recoveries are encountered, sample preparation and analysis errors such as evaporation of the extract, leaking septum, etc. must be evaluated.

10.3 Field QC Check Samples

10.3.1 Travel Blanks

Travel blanks are samples which contain analyte-free water which are transported to and from the sample site without being opened. The travel blanks are used when volatile organic compounds are being tested. The data for the travel blanks are used to determine cross-contamination from the container and preservative during sample transportation and handling in the field. One travel blank is sufficient for each day of sampling.

10.3.2 Equipment Blanks

Equipment blanks are blank water samples that are exposed to the sampling equipment to determine if the equipment was properly cleaned and rinsed so that no contamination or carryover is encountered during sampling.

10.3.3 Field Blanks

Field blanks are samples consisting of deionized water which are taken to the sampling site and exposed to the sampling environment. This may include transferring the sample from one container to another of simply opening the container for the duration of the sampling process. The field blank is then prepared and analyzed at the lab with the other samples. The field blank data is used to determine the amount of background contamination in the sampling environment.

10.3.4 Field Duplicates

Field duplicated consist of duplicate samples which are sampled at the same site. The field duplicates are prepared and analyzed as discrete samples. The data from the field duplicates determine the precision of the sampling technique.

10.4 Detection Limits

10.4.1 Instrument Detection Limit

The Instrument Detection Limit (IDL) is defined as the smallest signal above background noise that can be detected by the instrument as a 99% confidence interval. The IDL is calculated from the students'-t value (at the 99% confidence level for n-1 = 6 degrees of freedom) multiplied by the standard deviation of seven replicate analyses of a standard with a concentration of 3-5 time the instrument noise level.

10.4.2 Method Detection Limit

The Method Detection Limit (MDL) is defined as the constituent concentration that, when processed through the complete method, produces a signal with 99% probability that is different from the blank. The MDL is calculated from the student's-t value (at the 99% confidence level for n-1 = 6 degrees of freedom) multiplied by the standard deviation of seven replicates of the sample. Method detection limits are performed for each analysis according the the protocol in 40 CFR, Part 136, Appendix B. Unless otherwise specified, MDL's are performed on spiked deionized water blanks.

10.4.3 Practical Quantitation Limit

The Practical Quantitation Limit (PQL) can be defined as a value 5-10 times the MDL. The PQL provides a value that has very high certainty that the reported value is reliable.

10.4.4 Method Specific MDL

Method Detection Limit studies can be performed on a client's specific matrix. The MDL determined in this fashion is a true representation of the samples' background. This is the most accurate method for determining a MDL and should be considered when starting a project.

10.4.5 Reporting Limit

The Reporting Limit (RL) for most methods at Sierra are set at the MDL with an uncertainty level of ±100%. For example, a value reported at the detection limit of 3 ppm for instance can have an actual concentration from 0 ppm to 6 ppm.

10.5 Performance Evaluation Studies

Sierra's certification with the State of California Department of Health Services require that we participate twice a year in three EPA performance evaluation studies, the WP, WS and HW studies. These performance evaluation studies are applicable to the inorganic, organic and metal parameters for which we are certified. We also perform a bacterial performance evaluation sample once a year for the DOHS.

11. PREVENTATIVE MAINTENANCE

Sierra has preventative maintenance service contracts for most major instrumentation in the laboratory. In addition, daily maintenance is performed on most of the instrumentation, such as cleaning of contact rings on graphite furnace instrumentation, and cleaning injector ports and detectors for gas chromatography instrumentation. This information is documented in the instrument maintenance log. Log entries include the date, analyst name, the problem and the solution. If an instrument malfunctions and will be down for a prolonged period, samples are subcontracted to other certified laboratories.

12. ASSESSMENT OF PRECISION AND ACCURACY

12.1 Precision

Precision is the measurement of the difference between replicate analyses. The Relative Percent Difference (RPD) is used to measure this difference between replicates. The RPD is calculated from duplicate samples, MS/MSD or LCS/LCSD data as follows:

$$RPD = \frac{X - Y}{(X + Y)/2} \times 100 = RPD$$

Where X = Concentration of the Sample, MS or LCS and
Y = Concentration of the Sample Duplicate, MSD or LCSD

12.2 Accuracy

Accuracy if used to measure the difference of an analytical value from that of a known value. Types of QC samples associated with accuracy are: Laboratory Control Standards (LCS), Standard Reference Material (SRM), Matrix Spikes (MS), and Surrogate Spikes, Spike Recovery for the determination of accuracy is calculated as follow: % recovery = (A-B)/C x 100

Where A = spiked sample result, B = sample result, C = spike amount added

12.3 Control Charts

Control charts for both precision and accuracy are generated after 20 data points have been achieved. The charts indicate the mean recovery (accuracy), or RPD (precision), as well as the control limits ($\pm$3 standard deviations from the mean) of the 20 data points.

13. CORRECTIVE ACTION

The need for corrective action is identified by method QC data. The essential steps in the corrective action system are: identification and definition of the problem; assignment of responsibility for investigating the problem; investigation and determination of the cause of the problem; determination of corrective action to eliminate the problem; assigning and accepting responsibility for implementing the corrective action; implementing the corrective action and evaluating its effectiveness; and verifying that the corrective action has eliminated the problem.

Out-of-control situations are identified when a duplicate RPD (Relative Percent Difference), matrix Spike Recovery, Matrix Spike Duplicate Recovery, laboratory Control Sample (LCS) Recovery, Standard Reference Sample (SRS) Recovery, or Method Blanked exceeds control limits as defined by the method being used and/or as defined by control charts prepared from historical data. In general, when a non-conformance is incurred within normal laboratory protocol, the following procedures should be followed: stop the analysis immediately; review the data for calculation or integration errors; re-analyze the out-of-control sample(s) once. If the sample(s) are still out-of control, re-calibrate. If the sample in question is a reference sample, prepare new standards and repeat the analysis. For out-of-control duplicates, matrix spikes, or method blanks, re-digest or re-extract the sample and all other samples associated with the sample batch, if applicable, and reanalyze. the results should be reported to the Department Supervisor, the Laboratory Supervisor, or the QA manager. If the situation has not been corrected by the above procedures, review the method for specific technical problems, check for instrument or equipment malfunction and arrange for service if necessary. After the repair, recalibrate and analyze blanks and standard reference samples as defined in the method being used. Reanalyze the sample(s) in question and any samples associated with it.

However, the above procedure is not practical for all analyses. Many analyses require a substantial amount of time. On occasion, a non-conformance may not be identified until the analysis is completed. With some of the more time consuming analyses, this may mean that the sample has exceeded holding time by the time the non-conformance is recognized. Also, some analyses require a large sample volume, thus there may not always be sufficient sample remaining to reanalyze the sample after the non-conformance is identified. In situations such as these, it is our practice to contact the client to make them aware of the problems with the analysis and/or data, and to determine if additional sampling or other actions will be needed. If the data is reported in these situations, narratives are included in the final report explaining the out-of-control conditions, the corrective action taken and the justification for accepting the data.

14. LABORATORY SAFETY

Sierra Analytical Laboratories, Inc. expects all members of our organization to have the highest regard for safety and safe work practices at all times. Each member of our staff is expected to follow all safety rules and establish good safety practices to prevent injury to him or herself, other personnel or visitors, and loss or damage to our facilities and equipment. Each new employee shall receive safety indoctrination by Laboratory Supervisor upon employment. Safety rules and regulations shall be reviewed and understood by all. It is the intent that the safety rules and regulations establish the minimum level of safety performance. Each member of our staff is expected to seek additional assistance or information whenever he/she may be in doubt of the appropriate safety measures or precautions to be taken in the conduct of work assignments.

14.1 Laboratory Safety Rules, Regulations and Practices

All laboratory personnel shall be responsible for familiarizing themselves with the following information and guidelines prior to beginning work in the laboratory.

1. Know the safety rules and procedures that apply to the work that is being done. New procedures or techniques should be evaluated for any particular safety precautions and reviewed with laboratory Supervisor before beginning work.

2. Know the location and use of all emergency equipment in the laboratory.

a. The emergency phone numbers for the Fire Department, Police Department and nearest Hospital Emergency Room shall be posted at the laboratory phone and receptionist desk.

b. Fire: In the event of a fire, the first course of action is to notify other personnel in the area and call the fire department at once. The phone number for the fire department is located both at the laboratory phone and at the receptionist desk. After the fire department has been notified, steps to extinguish the fire may be taken. Appropriate fire extinguishers are located throughout the laboratory. All personnel should familiarize themselves with their location and use.

c. Emergency eye wash and shower: An emergency eye wash and shower is provided. all personnel should familiarize themselves with its location and operation. Each person should be able to locate the eye wash and shower station with their eyes closed if needed.

d. The first-aid kit, located in the laboratory entrance shall be inspected monthly to ensure adequate first-aid supplies.

3. Know the types of protective equipment available and use the proper type in the correct manner for each job conducted.

a. Eye protection is provided and its use is required during any laboratory operation and process.

b. Protective clothing (lab coats) of flame-retardant, chemical resistant material should be worn at all times while conducting laboratory operations.

4. Be alert to unsafe conditions and practices. Unsafe conditions and practices should be remedied as soon as possible.

5. Any spilled materials must be cleaned up promptly and completely.

6. All laboratory accidents, resulting in injury or not, shall be reported to the Laboratory Supervisor immediately.

7. Post adequate warning signs and labels when special hazards exist.

8. Each laboratory employee is responsible for keeping the laboratory clean at all times.

9. No smoking, eating or drinking shall be allowed in the laboratory.

10. DO NOT PIPETTE BY MOUTH. Piping must be done with a rubber bulb or other mechanical means to prevent possible ingestion or reagents.

11. Working in the laboratory without at least one additional person on the premises is strictly prohibited.

12. Avoid startling or unnecessarily distracting the attention of other laboratory personnel.

13. Handling of toxic or flammable materials shall be done in the fume hood with the wearing of appropriate personal protective clothing and equipment.

a. Many of the chemical compounds used and handled in our laboratory have toxic or hazardous properties. Therefore, all chemicals are to be treated as potentially dangerous unless they are positively known to be safe. To assist in determining the particular toxicological effects of chemicals and their compounds, our reference library should be utilized.

b. Handling and Storage of flammable Liquids; flammable liquids constitute one of burn rapidly. Rules governing the handling and storage of flammable liquids in the laboratory are listed below. Any exceptions to these rules must be approved by the Laboratory Supervisor.

1.) Flammable liquids should never be stored in open containers.

2.) Flammable liquids shall be stored in metal cabinets provided for flammable liquid storage.

c. Fume hoods are provided in the laboratory for conducting experiments and laboratory used for such analysis and laboratory techniques whenever

possible. The flow of air into the enclosure sweeps the toxic and odoriferous vapor and dusts into the exhaust duct to be exhausted out-of-doors, thus protecting the person working in front of the hood and also preventing the toxic and odoriferous materials from passing into the air of the laboratory. No chemicals should be stored in the fume hood when an experiment or analysis is not in process.

14. Corrosive Liquids: Bottles of acids and other corrosive liquids should be stored and carried in acid carriers or in polyethylene or lead trays large enough to hold the contents of the bottles. Steps must be taken to ensure that mutually reactive chemicals cannot contact one another. Other chemicals, solvents or materials, such as steel wool, soap pads, or the like should not be stored in the same tray unless in an unbreakable, corrosion-resistant, secondary container.

15. Reactive Chemicals: Storage and use of spontaneously flammable materials require extreme care. The quantity of these materials handled or stored in our laboratory is routinely kept to a minimum. Additionally, some chemical combinations result in spontaneous ignition of this mixture, and conditions which might result in accidental contact of such materials must be avoided. Care must be taken to avoid ignition of vapors or dusts by soraks from static electricity, the collision of metal objects, etc.

16. All chemical containers shall be labeled as to their contents.

17. All chemicals and equipment shall be stored in their appropriate storage compartments and cabinets as provided when not immediately in use.

18. Caution shall be taken in the proper disposal of acids, corrosives and other hazardous waste materials.

19. Glassware: The handling of glassware is a leading cause of laboratory injuries. Many of these injuries can be prevented if employees use these proper techniques:

a. Wear sturdy gloves or hold glass tubing with a towel when breaking or inserting into a stopper, cork or rubber tubing.

b. Lubricate glass tubing when inserting it into a stopper, cork or tubing. Grasp the tube near the end being inserted.

c. Check glassware for stresses and relieve them if present.

d. Immediately and properly discard broken and chipped glassware.

20. The compressed cylinders used for the instrumentation shall remain stationary and chained to the wall at all times (chained separately).

21. Changing of the gas cylinders shall only be conducted under the direct supervision of the Section Supervisor.

22. Electrical equipment must be turned off and unplugged prior to any repair or maintenance work.

23. Electrical repairs, replacement, and adjustments must be made only by authorized service representatives.

24. Visitors entering the laboratory shall comply with all safety practices and shall be

supervised at all times while visiting the laboratory.

14.2 SAFETY EQUIPMENT LIST

1. Safety glasses
2. Rubber gloves
3. Laboratory Coats
4. Eye Wash and Deluge Shower
5. Fire Extinguisher, ABC
6. Pipefitting Bulbs
7. Acid Carriers
8. Respirators
9. Sodium Bicarbonate - Acid Spill Absorbent
10. First-Aid Kit
11. Vermiculite - Solvent Spill Absorbent
12. ToxBox - Solvent Hazardous Waste Disposal

14.3 EQUIPMENT INSPECTIONS

1. Fire Extinguisher - Check monthly to ensure adequate charge.
2. Eye Wash and Deluge Shower - Check monthly to ensure proper operation
3. First-Aid-Kit - Check monthly to ensure complete inventory of supplies.

SIERRA ANALYTICAL
MAJOR LABORATORY EQUIPMENT

Organic Department

- 5973 GC/MS equipped with Agilent 6890 Series GC
  OI 4560 purge & trap
  OI 4552 auto sampler
  split/splitless inlet
  MS Chemstation with enviroquant

- 5973 GC/MS equipped with Agilent 6890N series GC
  OI 4560 purge & trap
  OI 4552 auto sampler
  split/splitless inlet
  MS Chemstation with enviroquant

- Waters Alliance System HPLC
  2695 Separations Module
  2475 Fluorescence Detector
  2996 Photodiode Array Detector
  Empower Software

- 5972 GC/MS equipped with HP 5890 Series II plus GC
  HP 7673 Autosampler
  split/splitless inlet
  MS Chemstation Data Analysis with Enviroquant

- 5971A GC/MS equipped with HP 5890 Series II GC
  OI 4460 Purge & Trap
  OI MPM-16 Autosampler
  split/splitless inlet
  MS Chemstation with Enviroquant

- HP 6890 GC equipped with 6890 Autosampler
  Split/splitless inlet
  Dual ECD's

HP Chemstation for Data Analysis

- Two (2)- HP 5890 Series II GC's with HP 7673 Autosampler
  split/splitless inlet
  Dual ECD's
  PE Nelson Turbochrom 3.3 Data Analysis

- HP 5890 Series II GC with Dual HP 7673 Autosamplers
  Direct Inlet
  Dual FID's
  PE Nelson Turbochrom 3.3 Data Analysis

- HP 5890 Series II GC with OI 4460 Purge & Trap Concentrator
  OI MPM-16 Autosampler
  PID-FID in series
  HP Chemstation Data Analysis

- HP 5890 Series II GC with OI 4460 Purge & Trap Concentrator
  OI MPM-16 Autosampler
  PID-ELCD in series
  HP Chemstation Data Analysis

- Two (2-) HP 5890 Series II GC's with OI 4460 Purge & Trap Concentrator
  Dynatech PTA-30 Autosampler
  PID-FID in series
  HP Chemstation Data Analysis

Metals Department

- Perkin Elmer Corporation ICP/MS ELAN 6000 with
  AS-90 Autosampler and Hydride System

- Perkin-Elmer Corporation Optima 3000SC ICP with
  AS-90 Autosampler and ICP WinLab software

- Perkin-Elmer Corporation Optima 4300 DV ICP with
  AS-93p/us Autosampler and ICP WinLab software

- Perkin-Elmer Corporation Plasma 400 ICP with
  AS-90 Autosampler and QCExpert software

- Perkin-Elmer Corporation Plasma 40 ICP with
  AS-50 Autosampler and QCExpert software

- Perkin-Elmer Corporation 5100 A.A. Spectrophotometer, Zeeman 5100 Graphite Furnace HGA600 system, with GEM desktop software

- Perkin-Elmer Corporation FIMS 400 Flow Injection Mercury System with
  AS-90 Autosampler and AA WinLab software

- CEM Corporation MDS 2100 Microwave Digestion System with computer software

- Metrohm Modular System

766 IC Sample Processor
766 IC Sample Processor
709 IC Pump
709 IC Pump
753 Suppressor Pump Module
752 Pump Unit
Lambda 1010
762 Interface
732 IC Detector
733 IC Separation Center
IC Net 2.1 software

Microbiology and Wet Chemistry

Microbiology

- Fisher-Scientific Low Temp Incubator, Model 307
- Kenmore Refrigerator, 18 cu. Ft.
- Precision Mechanical Convection Incubator
- Market Forge Sterilmatic Autoclave
- Precision Scientific Coliform Incubator Bath
- Fisher Scientific Waterbath
- Seward Laboratory Blender Stomacher 400
- Micro O Master Phase Contrast Microscope
- Fisher Scientific Microscope
- Fisher Scientific Touch Mixer
- Corning Hot Plates and Stirrers
- U.V. Light
- Automatic Pipeting Machine by Scientific Equip. Products
- Electric Pipetor

Wet Chemistry

- Metrohm Modular System
  766 IC Sample Processor
  766 IC Sample Processor
  709 IC Pump
  709 IC Pump
  753 Suppressor Pump Module
  752 Pump Unit
  Lambda 1010

762 Interface
732 IC Detector
733 IC Separation Center
IC Net 2.1 software

- Fisher ovens (2)- 180° and 103º
- Total Organic Carbon Analyzer Shimadzu 5000A w/Auto Sampler 5000 ASI
- Orion 960 Electrode Chemistry Station w/ Dispensing System, Orion 960SC Sample
- Changer and 7 Station Electrode Controller
- VWR 1350 U Oven
- Precision Oven Gravity Convection
- American Scientific Incubator Model
- Zero Headspace Extractor (Z.H.E.)
- Fume Hoods
- Dessicators (Fishers and Dry Keeper)
- Fisher Scientific Accumet pH Meter 915
- Orion Conductivity Meter Model 160
- Furnace 1400 Thermolyne
- New Brunswick Scientific Shaker
- Milton Roy Spectronic 601
- Hach 2000 Spectrophotometer
- Hach Heater Block
- US Standard Sieves
- Kjeltic System, System 20 Digestor, Model 1015 (TKN)
- Kjeltic System, 1002 Distilling Unit by Tecator
- Top Loader Balance, Fisher XL 1800 (3)
- Accumet pH Meter Model 915, Fisher
- Orion Model 160 Conductivity Meter
- YSI Oxygen Meter

- Orion Dissolved Oxygen Electrodes (3)
- HF Turbidimeter DRT 15-C w/Calibration Standards
- T.C.L.P. Rotator
- Pensky Martin Flash Point Apparatus

Mobile Laboratory

- Hewlett-Packard 5890 series II Gas Chromatograph equipped with four (4) detectors
- Two (2) OI Analytical Photo Ionization Detectors (PID)
- One (1) OI Analytical Flame Ionization Detector (FID)
- One (1) OI Analytical Electrolytic Conductivity Detector (ELCD)
- Hewlett Packard 7376 Automated Liquid AutoInjector
- Dynatech Precision PTA-30 Automated Volatile Organics Autosampler
- OI Analytical 4460A Purge and Trap Concentrator
- Wasson-ECE Injection port switching valve
- Buck Scientific HC-404 Infrared Hydrocarbon Analyzer
- 486DX-33 Mhz Computer with PE-Nelson TurboChrom Data Acquisition software
- Microsoft Office software for report generation
- Cellular telephone with data transmission capability
- Portable Facsimile Machine with Photocopy capability

APPENDICES

A. FLOOR PLAN OF LABORATORY

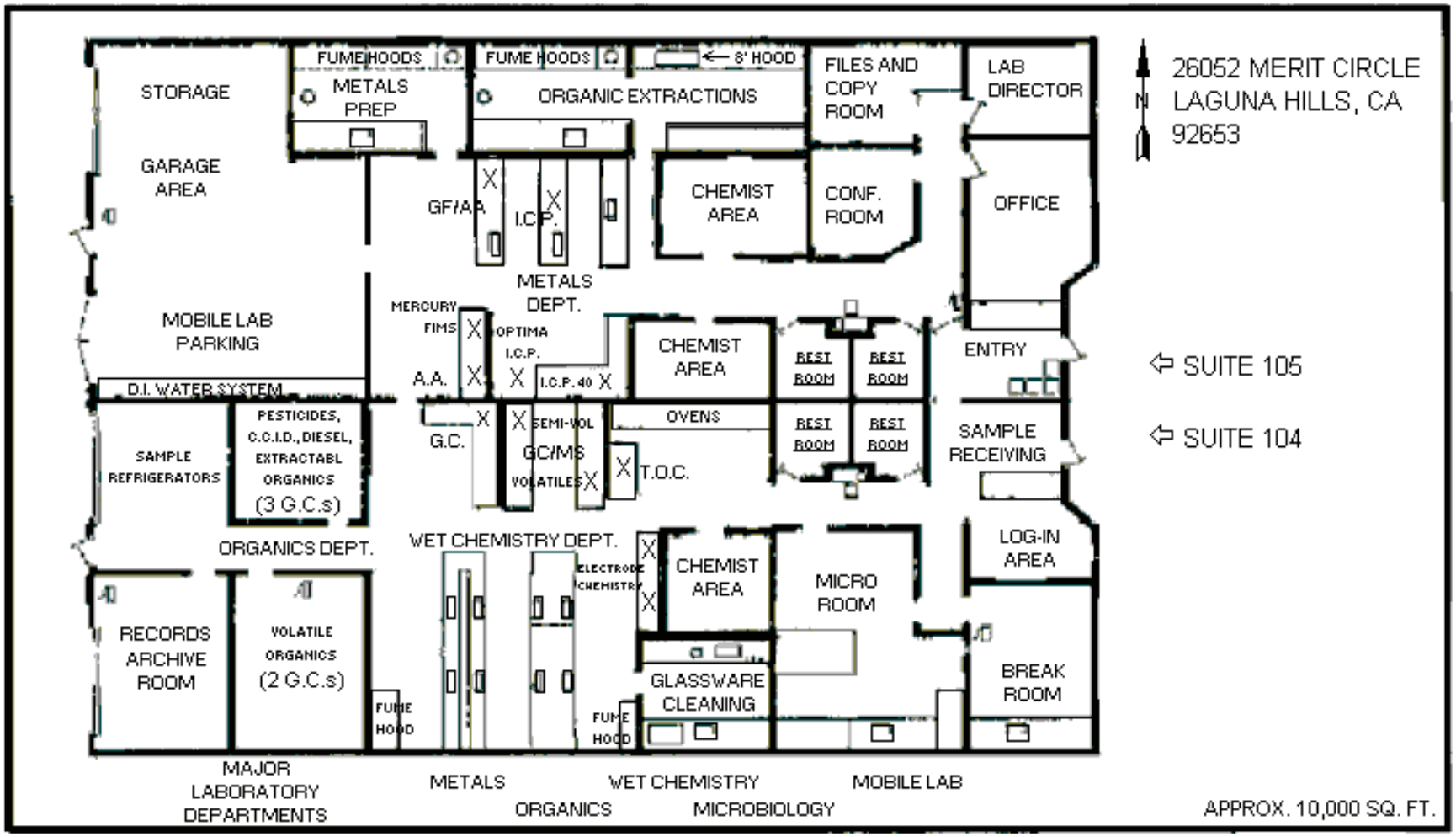
B. PRESERVATION AND HOLDING TIME LIST

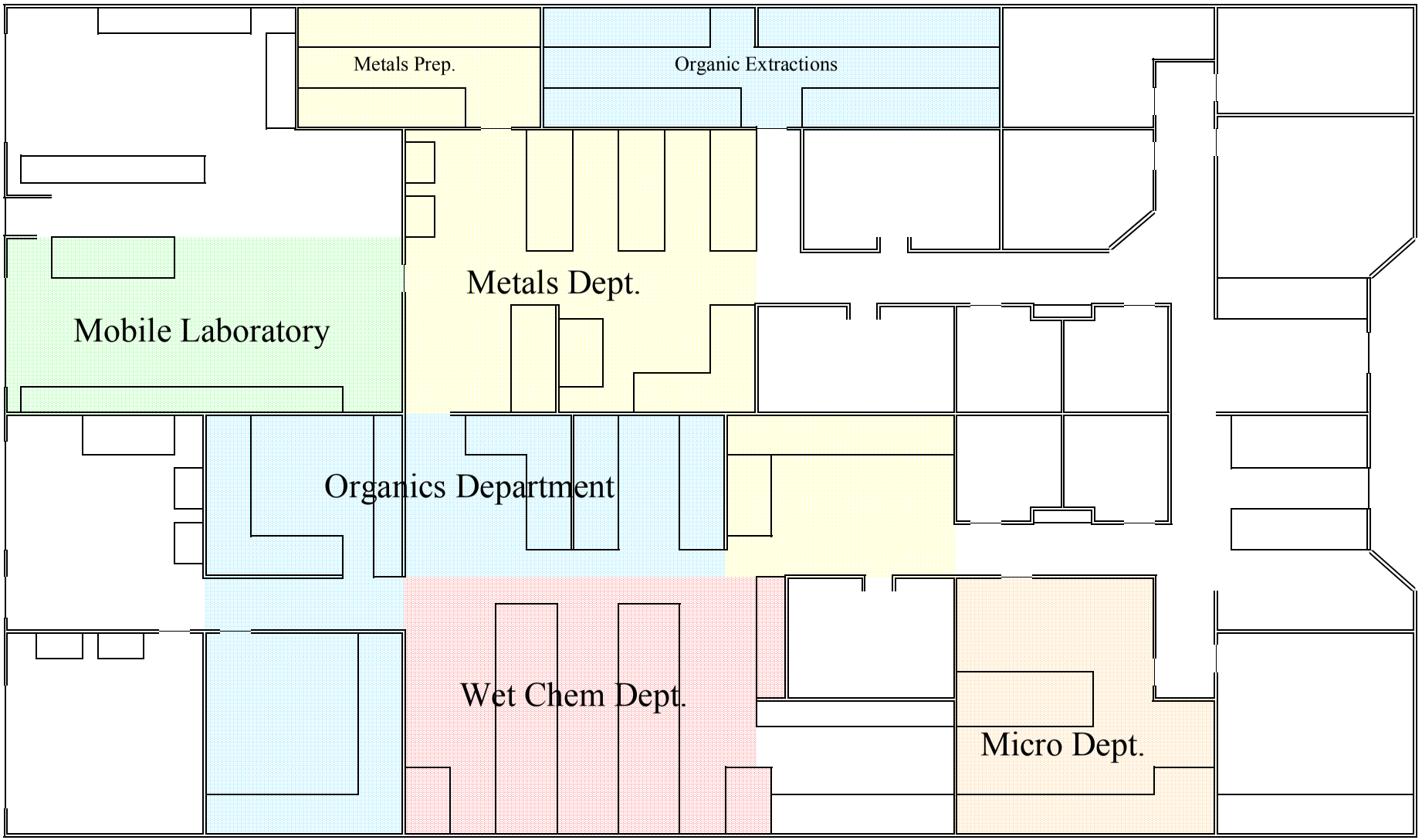
C. SAMPLE RECEIVING PROTOCOL

D. CHAIN OF CUSTODY FORM

APPENDIX A.
FLOOR PLAN OF LABORATORY

FLOOR PLAN
SIERRA LABORATORIES, INC.




Metals Prep.
Organic Extractions
Metals Dept.
Mobile Laboratory
Organics Department
Wet Chem Dept.
Micro Dept.

APPENDIX B.
PRESERVATION AND HOLDING TIME LIST

SIERRA ANALYTICAL

SAMPLE CONTAINER & PRESERVATIVE GUIDE

	METHOD	CONTAINER	VOLUME	PRESERVATIVE	HOLDING TIME
Volatile Organics					
Gasoline Range Organics	8015B GRO, 8015AZ GRO	VOA-glass	3 40 ml vials	Cool 4 C°, HCL, no HS	14 days
Gasoline Range Organics/BTEX	8015B GRO, 8021B, 8015AZ GRO/8021B	VOA-glass	3 40 ml vials	Cool 4 C°, HCL, no HS	14 days
BTEX,MTBE, or BTEX + MTBE	8021B	VOA-glass	3 40 ml vials	Cool 4 C°, HCL, no HS	14 days
EDB & DBCP	504.1, 8021B	VOA-glass	3 40 ml vials	Cool 4 C°, HCL ,sodium thiosulfate, no HS*	28 days
Halogenated Volatiles	601, 8021B	VOA-glass	3 40 ml vials	Cool 4 C°, HCL, no HS	14 days
Halogenated Volatiles/BTEX	601/602, 8010/8020, 8021B, 502.2	VOA-glass	3 40 ml vials	Cool 4 C°, HCL, no HS	14 days
Volatile Organics by GC/MS	624, 8260B, 524.2**	VOA-glass	3 40 ml vials	Cool 4 C°, HCL, no HS	14 days
	*If chlorine is present in water, add sodium thiosulfate before adding HCL			**If analyzing for 2-CVE, samples must be sent unpreserved	
	no HS = no Headspace	In Arizona, soils have a 48 hour to extraction holding time for volatile analyses (8015AZ GRO, 8021B, 8260B)			
Semi-Volatile Organics					
Diesel Range Organics	8015B DRO, 8015AZ DRO/ORO**	glass-amber	1L	Cool 4° C	7 days / 14 days soil**
Carbamate Pesticides	632	glass-amber	1L	Cool 4° C	7 days / 14 days soil
Chlorinated Herbicides	615, 8151 A	glass-amber	1L	Cool 4° C	7 days / 14 days soil
Formaldehyde	8351A	glass-amber	1L	Cool 4° C	72 Hours / 30 Days soil
Organophosphorous Pesticides	614, 8141A	glass-amber	1L	Cool 4° C	7 days / 14 days soil
Oil & Grease	413.1, 413.2	glass-amber	1L	Cool 4° C, HCL	28 days
PCB's	8082	glass-amber	1L	Cool 4° C	7 days / 14 days soil
Pesticides	8081A	glass-amber	1L	Cool 4° C	7 days / 14 days soil
Pesticides & PCB's	608, 8080	glass-amber	1L	Cool 4° C	7 days / 14 days soil
Phenolic Compounds	604, 8041, 8270C	glass-amber	1L	Cool 4° C	7 days / 14 days soil
Phthalates	8270C	glass-amber	1L	Cool 4° C	7 days / 14 days soil
Plynuclear Aromatic Hydrocarbons (PAHS)	8310, 8270C	glass-amber	1L	Cool 4° C*	7 days / 14 days soil
TRPH	418.1, 418.1AZ***	glass-amber	1L	Cool 4° C, HCL	28 days***
SemiVolatile Organics by GC/MS	625, 8270C, 525.2*	glass-amber	1L	Cool 4° C*	7 days / 14 days soil
	*If water is chlorinated, add soidum thiosulfate	**8025AZ DRO extraction requirement is 14 days for water.		***418.1AZ has a 14 days holding time to extraction requirement	
	Holding times are shown as "days until extraction". Samples have a 40 day holding time after extraction.				
Metals					
Chromium VI	7196A, SM 3500	poly or glass	500 ml	Cool 4° C	24 hours
Mercury	245.1, 7470A, 245.2, 7471A	poly or glass	500 ml	HN03	28 days
Organic Lead	CA DHS (LUFT)	glass-amber	500 ml	Cool 4° C	14 days
All Other Metals	200, 6010B, 7000A	poly or glass	500 ml	HN03	6 months
	Dissolved Metals must be filtered prior to preservation.				
Inorganics & Wet Chemistry					
Acidity	SM 2310B	poly or glass	500 ml	Cool 4°C	14 days
Alkalinities	310.1, SM 2320B	poly or glass	500 ml	Cool 4°C	14 days
Ammonia (as N)	350.3, SM 4500	poly or glass	500 ml	Cool 4°C, H2S04	28 days
Biochemical Oxygen Demand (BOD)	405.1, 405.2	poly	1L	Cool 4°C	48 hours
Carbon Dioxide	SM 4500	glass	500 ml	Cool 4°C	immediately
Chlorine	330.5, SM 4500	poly or glass	500 ml	Cool 4°C	immediately
Chemical Oxygen Demand (COD)	410.4, SM 5220	amber	500 ml	Cool 4°C, H2S04	28 days
Coliform (Total & Fecal)	SM 9221B, SM 9223B	bacti	100 ml	Cool 4°C, sodium thiosulfate	6-30 hours
Color	SM 2120	poly or glass	500 ml	Cool 4°C	48 hours
Conductivity	SM 2510B	poly or glass	500 ml	Cool 4°C	28 days
Cyanide (Total & Amenable)	SM 4500, 9014 (for Total)	poly or glass	500 ml	Cool 4°C, Na0H	14 days
Cyanide (Reactive)	SW 846 7.3.3.2	poly or glass	500 ml	Cool 4°C, Na0H	14 days

26052 MERIT CIRCLE SUITE 105, LAGUNA HILLS, CALIFORNIA 92653
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SIERRA ANALYTICAL

SAMPLE CONTAINER & PRESERVATIVE GUIDE

	METHOD	CONTAINER	VOLUME	PRESERVATIVE	HOLDING TIME
Inorganics & Wet Chemistry *(Continued)*					
Dissolved Oxygen	360.1, SM 4500	glass-amber	500 ml	none	immediately
Flashpoint / Ignitability	1010	glass-amber	1L	Cool 4°C	N/A
Hardness	200.7, 6010B, SM 2340C	poly or glass	500 ml	HN03	6 months
Hetertrophic Plate Count	SM 9215B	bacti	100 ml	Cool 4°C, sodium thiosulfate	30 hours
Ions by IC: FL, Br & Cl	300.0	poly or glass	500 ml	Cool 4°C	28 days
MBAS (Surfactants)	425.1, SM 5540C	poly or glass	1L	Cool 4°C	48 hours
Nitrate / Nitrite	300.0	poly or glass	500 ml	Cool 4°C	48 hours
Odor	SM 2150B	glass-amber	500 ml	Cool 4°C	24 hours
Perchlorate	300.0 Mod.	poly or glass	500 ml	Cool 4°C	28 days
pH	150.1, 9045C	poly or glass	500 ml	none	immediately
Phenols (Total)	420.1, 9065	glass-amber	500 ml	Cool 4°C, H2S04	28 days
Phosphate	300.0	glass-amber	500 ml	Cool 4°C	48 hours
Phosphorus (wet chem.)	365.3, SM 4500	poly or glass	500 ml	Cool 4°C, H2S04	28 days
Phosphorus (by ICP)	200.7, 6010B	poly or glass	500 ml	Cool 4°C, HN03	28 days
Settleable Solids(SS)	160.5, SM 2540F	poly or glass	1L	Cool 4°C	48 hours
Sulfate	300.0, SM 4500	poly or glass	500 ml	Cool 4°C	28 days
Sulfide (Dissolved, Soluble)	376.2, 9034, SM 4500	poly or glass	500 ml	Cool 4°C, Na0H	immediately**
Sulfide (Reactive)	SW 846 7.3.4.2	poly or glass	500ml	Cool 4°C, Na0H+zinc acetate	7 days
Sulfide (Total)	376.2, 9034, SM 4500	poly or glass	500 ml	Cool 4°C, Na0H+zinc acetate	7 days
Sulfite	377.1	poly or glass	100 ml	Cool 4°C	immediately
Thiosulfate	LACSD253B	poly	500ml	Cool 4°C	immediately
Total Dissolved Solids (TDS)	160.1, SM 2540	poly or glass	500ml	Cool 4°C	7 days
Total Suspended Solids (TSS)	160.2, SM 2540	poly or glass	500ml	Cool 4°C	7 days
Total Kjeldahl Nitrogen (TKN)	SM 4500	poly or glass	500 ml	Cool 4°C, H2S04	28 days
Total Organic Carbon (TOC)	415.1, SM 5310C	glass	500ml	Cool 4°C, HCL	28 days
Total Organic Halogens (TOX)	9020B	glass-amber	500ml	Cool 4°C, H2S04	28 days
Turbidity	180.1, SM 2130	poly or glass	500ml	Cool 4°C	48 hours
				**Separate and preserve immediately to extend holding time to 7 days.	
Bioassy					
Chronic	EPA 600 / 4 methods	poly	15 gal, 9 oz soil	Cool 4°C	36 hours
Effluent LC50	EPA 600 / 4 methods	poly	10 gal	Cool 4°C	36 hours
Effluent % Survival	EPA 600 / 4 methods	poly	5 gal	Cool 4°C	36 hours
Title 22 Hazardous Waste	CA Dept. of Fish & Game	N/A	1L, 9oz soil	Cool 4°C	N/A

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APPENDIX C.
SAMPLE RECEIVING PROTOCOL

SIERRA ANALYTICAL

SAMPLE RECEIVING INSTRUCTIONS

It is imperative to maintain acceptable quality that samples be submitted to the laboratory in an orderly manner without doubts to the condition and integrity of the sample. Each sample or set of samples must be accompanied by a legibly completed Laboratory Submittal Form, Chain of Custody Form, or a reasonable facsimile of one of these forms. The forms must contain the following information:

1. Name of sample and number
2. Name of sample collector (person who obtained sample)
3. Name of agency or company (client)
4. Date and time of sampling
5. Location of sample source
6. Type of sample (grab, etc.)
7. Note on any preservation done prior to submittal
8. List of requested analyses to be performed

The Sierra employees receiving the samples will sign and date the submittal form and record the method of shipment. The samples, along with the necessary forms will be forwarded to the laboratory log-in room for processing.

The laboratory personnel who receive samples will be responsible to inspect the samples and submittal forms to assure the sample receipt policy has been followed. The physical condition, sample integrity, and correct sampling procedures will be checked.

Also, any preservation done or that needs to be done will be noted on the laboratory sample analysis form. The preservation must be performed immediately: Refer to preservation list.

Once the sample clerk has determined the sample submission is in acceptable condition, he or she assigns the sample a Sierra Analytical job number. The job number is written on the submittal forms, samples, and laboratory sample analysis request form. (Note: all water wastewater, and hazardous waste samples will be numbered independently).

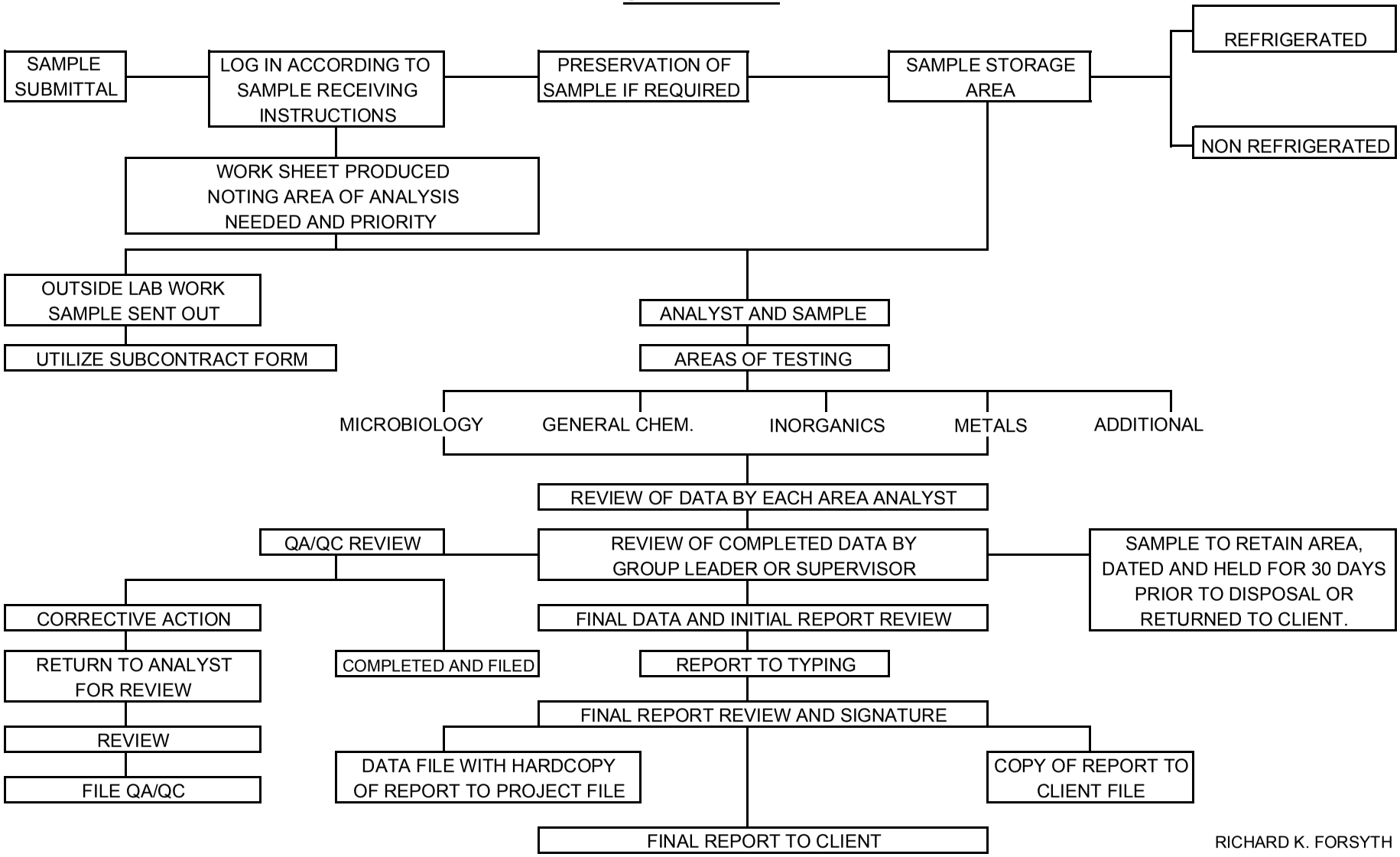
The sample is logged into the LIMS (Laboratory Information Management System) with the following information:

1. Sierra Lab ID No.
2. Sierra Sample No.
3. Client's Name
4. Time/Date Sampled
5. Sample Collector
6. Time/Date Sample Received
7. Sample Description
8. Analysis Requested
9. Preservation Performed
10. Date Report Needed
11. Logged in By

All samples are then stored in the appropriate secured area with the laboratory sample analysis request placed in the designated department bins in the log-in area. Samples on a rush basis will be marked and easily identifiable for immediate attention.

Upon completion of the analyses, the samples are placed in holding for 60 days, unless the client requests special holding.

SAMPLE FLOW DIAGRAM
GENERAL VIEW



RICHARD K. FORSYTH

APPENDIX D.
CHAIN OF CUSTODY FORM

SIERRA ANALYTICAL

TEL: 949 • 348 • 9389
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26052 Merit Circle • Suite 105 • Laguna Hills, CA • 92653

CHAIN OF CUSTODY RECORD

Lab Project No.: _____

Client:

Client Address:

Client Tel. No.:

Client Fax. No.:

Client Proj. Mgr.:

Client Project ID:

Turn Around Time Requested:
- ☐ Immediate
- ☐ 24 Hour
- ☐ 48 Hour
- ☐ 72 Hour
- ☐ 4 Day
- ☐ 5 Day
- ☐ Normal
- ☐ Mobile

Analyses Requested

Client Sample ID.	Sierra Sample No.	Date/Time	Matrix	Preservative	Container Type	No. of Containers												Comments

1 ☐
Sampler Signature:
Printed Name:

Shipped Via:
(Carrier/Waybill No.)

Total Number of Containers Submitted to Laboratory

Sample Disposal:
- ☐ **Return to Client**
- ☐ **Lab Disposal ***
- ☐ **Archive _____ mos.**
- ☐ **Other _____**

2 ☐
Relinquished By: Date: Time:
Company:

Received By: Date: 05/20/02 Time: 14:00
Company: **Sierra Analytical**

The delivery of samples and the signature on this chain of custody form constitutes authorization to perform the analyses specified above under SIERRA's Terms and Conditions, unless otherwise agreed upon in writing between SIERRA and CLIENT.
*** - Samples determined to be hazardous by SIERRA will be returned to CLIENT.**

3 ☐
Relinquished By: Date: Time:
Company:

Received By: Date:
Company: Time:

Total Number of Containers Received by Laboratory

4 ☐
Relinquished By: Date: Time:
Company:

Received By: Date:
Company: Time:

FOR LABORATORY USE ONLY - Sample Receipt Conditions:
- ☐ Intact
- ☐ Sample Seals
- ☐ Properly Labelled
- ☐ Appropriate Sample Container
- ☐ Chilled - Temp (°C) _____
- ☐ Preservatives - Verified By _____
- ☐ Other _____
- ☐ Storage Location _____

Special Instructions: