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| Quality Assurance Project Plan |
| Withers Swash Microbial Source Tracking Program |
| Lead Organization: Coastal Carolina University  Burroughs and Chapin Center for Marine and Wetland Studies  Waccamaw Watershed Academy  Environmental Quality Lab (Lab ID 26001)  Project Manager: John Michael Trapp, EQL Laboratory Director  CCU, Environmental Quality Lab  Principal Investigators: John Michael Trapp, EQL Laboratory Director  CCU, Environmental Quality Lab  Erin Burge, Associate Professor of Marine Science, CCU  Susan M. Libes, EQL Program Director  CCU, Waccamaw Watershed Academy |
| **April 2012** |

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| **Management Approvals:**  Signature indicates that this QAPP is approved and will be implemented in conducting the research of this project.  John Michael Trapp, PhD  Project Manager and Liaison *Signature* *Date*  Coastal Carolina University  Burroughs and Chapin Center for Marine and Wetland Studies  Environmental Quality Lab    Susan M. Libes, PhD  EQL Program Director *Signature Date*  Coastal Carolina University  Waccamaw Watershed Academy  Burroughs and Chapin Center for Marine and Wetland Studies  Environmental Quality Lab  **Quality Assurance:**  Signature indicates that this QAPP meets the quality requirements of USEPA and SCDHEC. |

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# Project Management

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## Background and Project Objectives

This QAPP covers the work to be performed in Phase II of a two phase U.S. Army Corps of Engineers Planning Assistance to States (USACOE PAS) project funded and conducted collaboratively by the USACOE-SC District, Horry County, Georgetown County and the cities of Myrtle Beach and North Myrtle Beach. The goal of phase I was to develop local capacity for use of genotypic source tracking tools to identify mammalian hosts of fecal indicator bacteria (FIB). These microbial source tracking (MST) tools are designed to be applied in the context of a multi-tracer, targeted watershed-based investigation conducted collaboratively with local SMS4 stormwater staff and CCU’s Environmental Quality Laboratory (EQL). During phase II, this approach is implemented in the Withers Swash watershed, which lies within the jurisdictions of the City of Myrtle Beach and unincorporated Horry County.

Withers Swash is one of 14 tidal creeks located along the Grand Strand. Most of these tidal creeks are on the federal 303(d) list of impaired water bodies for recreational usage due to contraventions of Enterococcus water quality standards (SC DHEC 2010). The Grand Strand lies along the coast of northeastern South Carolina and stretches for more than 60 miles across Horry and Georgetown Counties. Eighty-three (83) sites in Horry and Georgetown counties have been documented by the South Carolina Department of Health and Environmental Control (SCDHEC) as having fecal indicator bacteria impairments.

An important approach to remedying these impairments is to reduce the input of FIB into the waterways. This requires knowledge of FIB bacteria sources in time and space. An important aspect of source tracking is identifying the host animal from which the FIB emanated. Phase I of this PAS study adapted peer-reviewed quantitative Polymerase Chain Reaction (qPCR) Bacteroides assays for use as a tracer of human sources of this FIB. Bacteroides spp. are a pervasive component of the gut microfauna in mammals, including humans, and are a component of the total fecal Bacteroidales community. Since qPCR provides a quantitative estimate of human-associated Bacteroides, this information can be used to infer the relative importance of human inputs via comparison to the total fecal Bacteroidales content of a given water sample.

The field work for Phase II will be completed between April and September 2012 with both dry and wet weather sampling in Withers Swash.

## Project/Task Description and Schedule

The tasks of the EQL in the Withers Swash MST project are to conduct regulatory-level sampling and analysis for water-quality parameters at fourteen sites in the Withers Swash watershed, sites are shown in Table 1 and Figure 1. Samples will be analyzed for the following parameters:

1. Total coliforms
2. *E. coli*
3. Enterococcus
4. Bacteriodes thetaiotamicron (GenBac)
5. Bacteriodes dorei (BacHum)
6. Bacteriodes canine (BacCan)
7. 5-day biochemical oxygen demand (BOD5)
8. Turbidity
9. Total Suspended Solids (TSS)
10. Volatile Suspended Solids (VSS)
11. Ammonia-nitrogen (NH3-N)
12. Optical brighteners
13. Toxicity
14. Dissolved Oxygen (DO)
15. pH
16. Conductivity
17. Temperature
18. Salinity

The project schedule is as follows:

**April 2012 – September 2012:** Sampling of Withers Swash. Checkpoint meeting(s) with project team if sampling scheme changes. All analyses completed within hold times as listed in Table 3.

**November 2012:** Team meeting to discuss end of sampling efforts and next steps to produce Watershed Assessment Report.

**January 15, 2013:** Produce draft Watershed Assessment Report in collaboration with the City of Myrtle Beach.

**February 1, 2013:** Project team submits comments back to Coastal Carolina University and the City of Myrtle Beach.

**February 14, 2013:** Coastal Carolina University and the City of Myrtle Beach produce final Watershed Assessment Report and protocol for future work.

**February 28, 2013:** Meeting with SCDHEC and other interested parties to show project results and protocol for future work.

Table 1. Sampling Sites

|  |  |  |  |
| --- | --- | --- | --- |
| **Site** | **Description** | **Latitude** | **Longitude** |
| MST-1 | 17th Ave. S. at Bent Oak Estates | 33.683625 o N | -78.911129 o W |
| MST-2 | 17th Ave. S., across from AVX | 33.680438 o N | -78.908253 o W |
| MST-3 | 11th Ave. S., BMP spillover | 33.683055 o N | -78.903416 o W |
| MST-4 | KOA at 5th Ave. S., pipe spillover | 33.685207 o N | -78.897461 o W |
| MST-5 | Beaver Rd. Ext. | 33.681399 o N | -78.904257 o W |
| MST-6 | BMP on Mister Joe White Ave. | 33.700243 o N | -78.887095 o W |
| MST-7 | Off Robert Grissom Pkwy., behind Coastal Electric | 33.704416 o N | -78.895698 o W |
| MST-8 | Street Reach on Osceola St. | 33.707382 o N | -78.893127 o W |
| MST-9 | Canal St., behind Shields Chapel | 33.699196 o N | -78.894043 o W |
| MST-10 | Holly Dr., at end of cul-de-sac | 33.694623 o N | -78.902775 o W |
| MST-11 | 3rd Ave. N. at Alder St. | 33.692301 o N | -78.892234 o W |
| MST-12 | KOA at 5th Ave. S., tidal creek | 33.685212 o N | -78.897352 o W |
| MST-13 | Cedar St. | 33.693397 o N | -78.895228 o W |
| MST-14 | Off of Cannon. Rd., accessed via Holly Rd. from MST-10 | 33.69406 o N | -78.902492 o W |

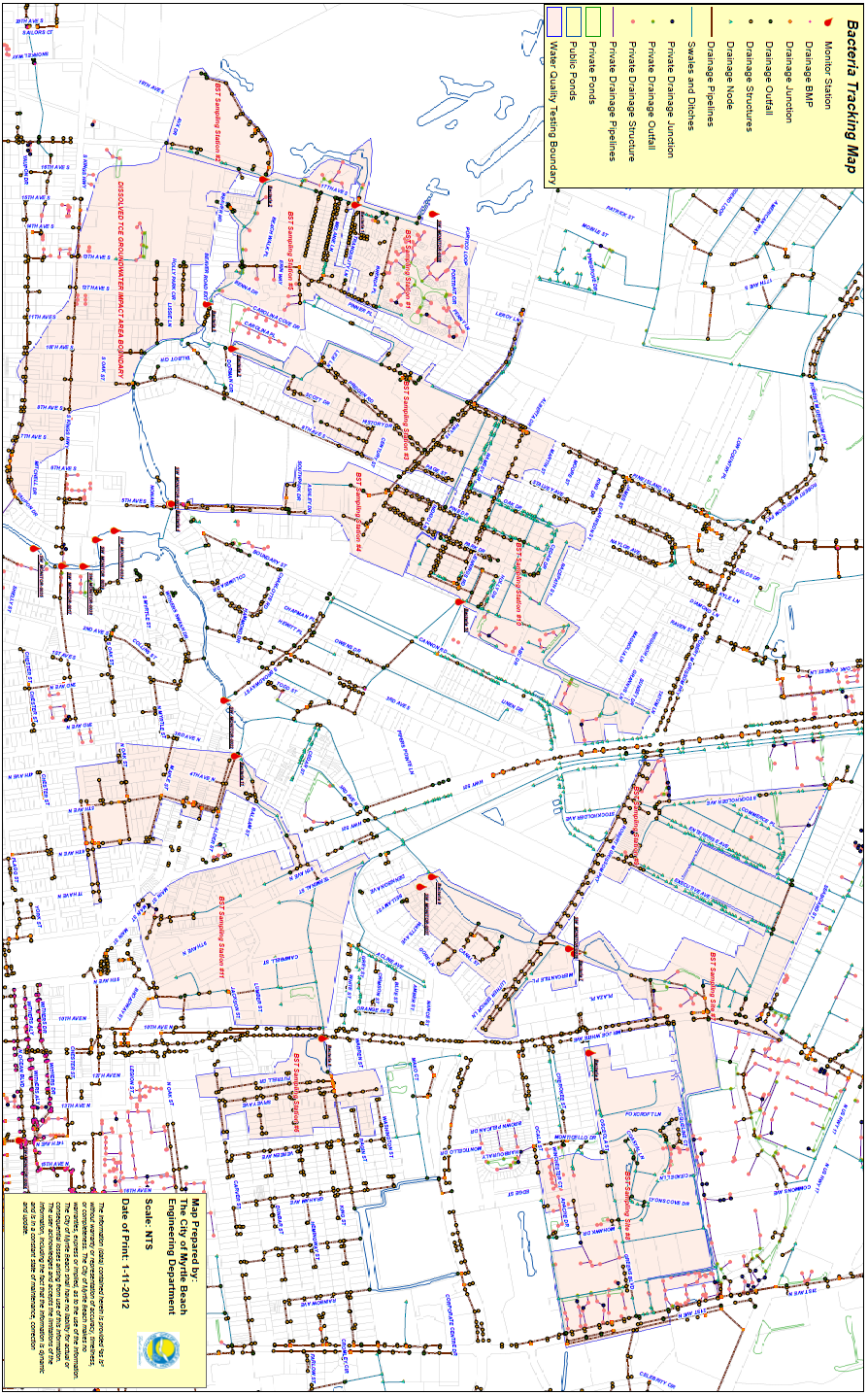


Figure 1. Project Monitoring Sites

## Data Quality Objectives and Criteria for Data Measurement

### The DQO Process

1. **State the Problem:** The goal of this project was to use genotypic source tracking tools to identify mammalian hosts of fecal indicator bacteria (FIB) in the Withers Swash watershed, which lies within the jurisdictions of the City of Myrtle Beach and unincorporated Horry County. These microbial source tracking (MST) tools will be applied in the context of a multi-tracer, targeted, watershed-based investigation conducted collaboratively with local small municipal separate storm sewer system (SMS4) stormwater staff and CCU’s Environmental Quality Laboratory.
2. **Identify the Decision-** Data from this study will be used to determine where to focus bacterial remediation efforts in the City of Myrtle Beach and unincorporated Horry County.
3. **Inputs to the Decision-** Lab and field data, in addition to baseline data from Phase 1 of the USACOE PAS study will be used to pinpoint the study sites most contaminated by bacteria.
4. **Define the Study Boundaries-** The study boundaries are delineated in Figure 1. At each sampling site (Table 1) within the study boundaries, water samples will be collected at a depth of 0.3 m.
5. **Develop an analytical approach and a decision rule-** Sites with greatest bacterial contamination, as demonstrated by lab and field data, will be the focus of remediation efforts.
6. **Specify Limits on Decision Error**- See Section 2.5 for information on error-minimization strategies used in this study.
7. **Optimize the design for obtaining the data**- The quality of measurements made for the project by this laboratory is determined by the following data quality indicators (DQIs), or characteristics: representativeness, accuracy, precision, detectability, completeness, and comparability. Specific criteria for each characteristic were established to assist in the selection of appropriate sampling and analytical protocols and to identify applicable documentation, sample handling procedures, and measurement system procedures. These DQI criteria were established based on site conditions, requirements of the project, and knowledge of available measurement systems, and were addressed whenever appropriate for the data generated.

### Representativeness

Representativeness is a qualitative measure of the extent to which a sample acquired from a matrix describes the chemical or physical characteristics of that matrix. Sample collection, handling (e.g., splitting, preservation, storage), and measurements are all conducted according to protocols allowing for the highest degree of representativeness possible for the sample media (air, soil, water, etc.). Recording procedures are utilized which document adherence to proper protocols and maintain sample identification and integrity.

### Accuracy

Accuracy describes the degree of agreement between an observed value and an accepted reference (true) value. It includes a combination of random error (precision) and systematic error (bias) components which are introduced in sampling and analytical operations. DQI criteria for accuracy are established through quality control limits for each parameter measured and for each analytical technique, per matrix where applicable. These objectives are assessed through the analysis of sterility checks, positive and negative culture checks, blanks, matrix spike (MS)/matrix spike duplicates (MSDs), and laboratory control samples (LCSs), as specified by the analytical method, required by the project, or generated and updated from data acquired through required quality control measurements. Nominal quality control limits for each parameter and analytical technique are specified in the analytical methods.

### Precision

Precision is a measure of the reproducibility of an analysis under a given set of conditions, regardless of the true value of the target analyte in a sample. The overall precision of a sampling event has both a sampling and an analytical component. DQI criteria for precision are established through quality control limits for each parameter measured and for each analytical technique, per matrix where applicable. These objectives are assessed through the analysis of MSDs (if practical), LCS duplicates (if available), field duplicates, laboratory replicates, and split laboratory samples, as specified by the analytical method, required by the project, or generated and updated from data acquired through required quality control measurements. Nominal quality control limits are specified for each parameter and analytical technique in the analytical methods.

### Detectability

Method detectability objectives define the lowest concentration or quantities required of the measurement system for each analyte or parameter. The laboratory has established reporting limits (RLs) which are the minimum concentrations to be reported without qualification for routine laboratory conditions. Data quality indicator criteria for detectability (i.e., RLs) are established for each parameter measured and for each analytical technique. These criteria are specified by the analytical method, required by the project, or determined and updated from data acquired through required quality control measurements (e.g., the replicate analyses of samples or standards containing low concentrations of the analyte of concern).

The RL for an analyte is a function of the specific analytical procedures and can vary substantially as a result of dilutions and similar procedure modifications. In all cases, the RL necessary to fulfill data quality objectives is confirmed by laboratory measurements. Nominal RLs for each parameter and analytical technique are listed in the analytical methods and on the report of analysis.

### Completeness

The characteristic of completeness is a measure of the amount of valid data obtained compared to the amount that was expected to be obtained under normal conditions. The amount of valid data expected is based on the measurements required to accomplish project objectives.

### Comparability

The characteristic of comparability reflects both internal consistency of measurements and expression of results in units consistent with other organizations reporting similar data. The generation of comparable data requires operating within the calibrated range of an instrument and utilizing analytical methodologies which produce comparable results. Appropriate standard units for measurement values are utilized for each measurement system, which yields internally and externally comparable results assuming other comparability criteria are met.***DQO****s establish the data users requirements for precision, accuracy, completeness, representativeness, and comparability.With a list of specific goals the research scientist first decides what information is needed to answer the questions posed. This discussion should include aspects of data quality and can usefully be captured as a statement of* ***data quality objectives****. Some of this information lends itself to presentation in a table format, but representativeness and comparability are the products of an appropriate experimental design and are understood better in that discussion.*

*One method to determine the required data quality objectives (DQOs) is to make a list of all measurements needed to answer the questions posed in the research. Determine the level of precision, accuracy, and completeness needed to accomplish your goals. This can often be presented in a table format (example table 1). It is important to remember that the values presented as your data quality objectives will be the standards for evaluating the data collected. Make sure that the DQOs reflect the needs of the project and are not too restrictive or too lax. Remember that the DQOs may be changed if in the course of the project you have been too optimistic regarding your measurement ability or that your analysis requires greater precision, accuracy or completeness.*

### Project DQIs

Because of the intended data uses, the general philosophy for determining the project’s DQI criteria was that data quality should meet current industry standards for such measurement data. In general, measurement DQI criteria are based on the published analytical method for each parameter. A list of these methods can be found in Section 2.4 of this QAPP.

Specific criteria for measurement DQIs for the analyses to be performed are summarized inTable 2**.**

Table 2. Specific criteria for measurement DQIs

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Parameter** | **Units** | **Accuracya**  **(LCS)** | **Accuracya**  **(Matrix Spike)** | **Precisiona**  **(RSD or RPD)** | **MDLb** | **RLc** | **Complete-ness (%)** |
| Total coliform | CFU/100ml | NA | NA | RPD≤ 200% for <150 CFU/100 ml  RPD≤ 100% for ≥ 150 CFU/100 ml | 1 CFU/100 mL | 1 CFU/100 mL  if sample is not diluted |  |
| E. coli | CFU/100ml | NA | NA | RPD≤ 200% for <150 CFU/100 ml  RPD≤ 100% for ≥ 150 CFU/100 ml | 1 C1 CFU/100 mL FU | 1 CFU/100 mL  if sample is not diluted |  |
| Enterococcus | CFU/100ml | NA | NA | RPD≤ 200% for <150 CFU/100 ml  RPD≤ 100% for ≥ 150 CFU/100 ml | 1 C1 CFU/100 mL FU | 1 CFU/100 mL  if sample is not diluted |  |
| Bacteriodes thetaiotamicron (GenBac) | genomes/  100ml | 3% (>0.97R2) | NA | NA | <10 genomes | <10 genomes |  |
| Bacteriodes dorei (BacHum) | genomes/  100ml | 3% (>0.97R2) | NA | NA | <10 genomes | <10 genomes |  |
| Bacteriodes canine (BacCan) | genomes/  100ml | 3% (>0.97R2) | NA | NA | <10 genomes | <10 genomes |  |
| 5-Day Biochemical Oxygen Demand | mg/L | 85-115% | 85-115% | <25% | <2.0 | <2.0d | 95 |
| Turbidity | NTU | 90-110% | NA | <25% | <0.5 | <0.5 | 95 |
| Total Suspended Solids (TSS) | mg/L | 90-110% | NA | ≤5% | ≥2.5 mg to ≤200 mg | ≥2.5 mg to ≤200 mg |  |
| Volatile Suspended Solids (VSS) | mg/L | 90-110% | NA | ≤5% | ≥2.5 mg to ≤200 mg | ≥2.5 mg to ≤200 mg |  |
| Ammonia-nitrogen (NH3-N) | nM/L | 90-110% | RPD± 25% | RPD± 5% | 100 nM | 100 nM |  |
| Optical Brighteners | POS/NEG,  OB-28 ppb | 90-110% | 80-120% | RPD <25% | 1.29 ppb OB-28 | 1.5% of % reduction ratio indicative of OB |  |
| Toxicity | pos/neg | NA | NA | RPD≤ 25% | NA | NA |  |
| Dissolved Oxygen | mg/L | 90-110% | NA | <25% | <0.3 | <0.3 | 95 |
| pH | Standard | ±0.1 S.U. | NA | ±0.1 pH | NA | NA | 95 |
| Conductivity  <200 μS/cm | μS/cm | 90-110% | NA | <25% | <50 | <50 | 95 |
| Conductivity  >200 μS/cm | μS/cm | 95-105% | NA | <20% | NA | NA | 95 |
| Water Temperature | °C | ± 0.5°C | NA | ± 0.5°C | NA | NA | 95 |
| Air Temperature | °C | ± 1.0°C | NA | ± 1.0°C | NA | NA | 95 |
| Salinity | ppt | 90-110% R for <100/00  95-105 R for ≥ 100/00 | 80-120% R for <100/00  90-110 R for ≥ 100/00 | RPD ≤ 25% for < 100/00  RPD ≤ 20% ≥ for 100/00 | 0.01 0/00 |  |  |
| LCS = laboratory control sample % R = percent recovery  MDL = method detection limit RL = reporting limit  MS = matrix spike RPD = relative percent difference  NA = not applicable % RSD = percent relative standard deviation    a Criteria apply to concentrations > RL.  b For undiluted samples.  c For undiluted samples. If sample is diluted, RL is proportionally higher.  d Method constraint for valid measurement. Many river samples are below this method constraint, which was designed primarily for waste water discharges, so measured values for river samples will be reported. | | | | | | | |

## Special Training Requirements and Certifications

The Certificate issued by the SC DHEC Office of Environmental Laboratory Certification is 26001002.

The generation of reliable data by a laboratory requires that all operations are conducted by knowledgeable and trained personnel. The laboratory requires the accomplishment of a prescribed sequence of training objectives by a staff member before that individual is designated as qualified and permitted to independently conduct any assignment or analyses. The indoctrination and qualification process includes as a minimum:

* Reading and understanding applicable laboratory SOP,
* Reading and understanding applicable reference documents,
* Hands-on training under the supervision of an experienced and qualified individual, and
* For analytical methods used for measurements, a successful initial demonstration of analytical capability (i.e., IDC) by performing four replicate measurements which satisfy precision and accuracy criteria for the method (IDC Form 1020, Appendix A: Forms) as well as an MDL study.

Training records for staff are maintained by the Laboratory Director, and training files are kept for each staff member in the training and qualification files. Lab analysts also collect samples and perform field measurements. A summary of training accomplishments is recorded on a Personnel Qualification Record, Form 110 (Appendix A: Forms).

## Documentation and Records

Personnel on the distribution list will receive the QAPP electronically.

All records and documents generated by EQL specifically for this project are described and listed in Table 22 of Section 2.5.7.5. The formats of the records are illustrated in the copies of all applicable forms provided in Appendix A: Forms. The server is backed up weekly to an external hard drive. Annually, electronic records are backed up onto disk and kept for a minimum of 10 years. Hardcopies are bound and stored for a minimum of 10 years. All records are kept onsite.

EQL document control procedures are described in the Section 2.5.7.2 of this QAPP, and controlled copies of this QAPP are provided to the addressees listed in Section 1.1.

### Data Reporting

After completion of analyses, analysts enter results for both samples and QC measurements into the laboratory's computer-based report templates (i.e., spreadsheets). After peer review of the data is completed and the results are acceptable, the Laboratory Director reviews the preliminary report and works with necessary laboratory personnel to make any needed corrections. A final report is then produced and submitted to the customer, either electronically or by mail depending on the contract. For this project, excerpts from laboratory’s results database (Microsoft Excel) containing completed, reviewed, and approved project results are periodically sent to the EQL Program Director for distribution to all project customers.

If a hard copy report is requested, the procedures for preparation and distribution of a report are as follows: (1) assemble, (2) paginate, (3) final review, (4) approval and signature by Laboratory Director, (5) copy, (6) mail to customer. The copy of the data package provided to the client and all associated raw data are typically kept for period of at least 10 years. The retention period may be different if requested by the client as determined by the uses and recipients of the data. These records are stored in the laboratory for approximately two years, and then transferred to locked university storage room for secure, long term storage.

For electronic data deliverables (EDDs) in Microsoft Excel or similar formats, files are maintained on the laboratory’s desk top computers and the university’s intranet, with access restricted to the Laboratory Director, Laboratory Master Technician, and Laboratory Technicians. Backup copies of the electronic files are prepared at least annually and stored in a secure area off-site.

# Measurement/Data Acquisition

## 2.1. Sampling Process Design (Experimental Design)

Sites representing distinct drainage areas within the larger Withers Swash drainage basin that are likely to exhibit high instances of bacterial contamination were chosen by the City of Myrtle Beach. Dry and wet weather samples will be collected so that both baseline and runoff-influenced water quality conditions can be understood. In order to sample the most contaminated water at each site, wet weather sampling will be focused on capturing the first flush (rising limb of the hydrograph) of each rain event at each site.

## Sampling Methods

Sampling will occur from April to September 2012, as fecal indicator bacteria are known to be more active during warmer months. Within this time, samples will be collected during two dry and three wet weather events. Based on hydrographic data generated prior to the sampling period, this study will require 0.63 cm of accumulated rainfall during a single rain event, and 72 hours of antecedent dry conditions for any wet weather sampling event. Dry condition sampling events will similarly require 72 hours of antecedent dry weather for the area before sampling can occur (USEPA 1992).

Sampling will be conducted as per SOP 302. Water samples will be collected at a depth of 0.3 m. In order to insure that water is sampled during the rising limb of the hydrograph at each site, Nalgene first flush samplers will be deployed at sites with especially “flashy” (quickly rising and draining) hydrographs. At these sites, two first flush samplers will be deployed at different heights in order to sample water at different points during the rising limb of the hydrograph. The 2 L of water collected by the two first flush samplers at a site are then composited and sub-sampled for all laboratory analyses.

Sampling efforts will involve the collection of water samples for analyses including Bacteriodes thetaiotamicron (GenBac), Bacteriodes dorei (BacHum), Bacteriodes canine (BacCan), five-day biological oxygen demand (BOD5), turbidity, ammonia-nitrogen (NH3), total suspended solids (TSS), volatile suspended solids (VSS), toxicity, *E. coli*, total coliforms, *Enterococci,* salinity,and optical brighteners. At the time of sample collection, in situ measurements will also be made for pH, temperature, and dissolved oxygen (DO) at each sampling location.

Field measurement procedures and sample collection, handling, receiving, storage, and associated record keeping procedures are integral parts of the EQL’s QA program. The policies are designed to ensure that each measurement result and each sample are accounted for at all times. The primary objectives of EQL measurement and sample control procedures are as follows:

* Each field measurement is recorded and uniquely identified at the time of measurement,
* Each sample received for analysis is uniquely identified,
* The correct samples are analyzed and are traceable to the applicable data records,
* Important and necessary sample characteristics are preserved,
* Samples are protected from loss, damage, or tampering,
* Any alteration of samples during collection or transport (e.g., filtration, preservation, breakage) is documented,
* Records of field measurements and sample custody (i.e., chain of custody) and integrity are established which will satisfy legal scrutiny, and
* A record of ultimate sample disposition (i.e., disposal or release from laboratory) is established.

### Sample Collection

A summary of sample collection, handling, and preservation activities is provided inTable 3.

Samples collected by EQL personnel are placed in appropriate containers, having the required preservatives or additives, and labeled with site-specific information to uniquely identify each container at the time of collection. Conditions of sampling sites, sample IDs, number of samples, dates/times of collection, equipment calibrations, etc., are recorded on site in field logbooks or on EQL chain of custody forms as appropriate. Unless otherwise specified, samples are stored on ice in coolers at 1-6 °C until their receipt at the laboratory. EQL samplers may be the Laboratory Director, Laboratory Master Technician, Laboratory Technicians, or CCU students trained in sampling. In general, samples collected are grab samples (i.e., sample collected at a specific time and place) and collected manually. For bacteria analysis, samples are collected using sterile glass or sterile plastic sample bottles and collected carefully at 0.3 m. as to not contaminate by touching the inside of either the bottle or its lid. The bottle is filled with sample to approximately one-inch from the top, and then the lid is replaced. The bottle is then placed in a snap and seal plastic bag and a cooler with ice for storage and transport to laboratory. For analyses other than bacteria, samples are collected in plastic bottles. Bottles are rinsed with river water at the site three times, carefully filled with river, capped, and then placed in a cooler for storage and transport to the laboratory. Specific procedures for sample container preparation and sample collection are provided in EQL SOP 301, “Sample Container Preparation” and EQL SOP 302, “Sample Collection” (Appendix D: Environmental Quality Lab Standard Operating Procedures).

If issues occur in the field, the sample collector will handle these and record the issue and the corrective action on the Field Measurement Page. If the sample collector cannot fix the situation, then the Project Manager, Dr. Trapp, is contacted.

Table 3. Sample collection, handling, and preservation activities

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sample Type** | **Parameter Measured** | **Sample Container** | **Minimum Sample Size** | **Preservation Method/ Storage** |
| Urban stream/ditch water, collected via grab samples and first flush sampler samples | Total coliform and E. coli | Sterile plastic with sodium thiosulfate | 100 mL | Field: store in cooler at 1-6 °C  Lab: store in refrigerator at 1-6 °C and start analysis within 6 hours |
| Urban stream/ditch water, collected via grab samples and first flush sampler samples | Enterococcus | plastic | 100 mL | Field: store in cooler at 1-6 °C  Lab: store in refrigerator at 1-6 °C and start analysis within 6 hours |
| Urban stream/ditch water, collected via grab samples and first flush sampler samples | Bacteriodes thetaiotamicron (GenBac), Bacteriodes dorei (BacHum), Bacteriodes canine (BacCan) | plastic | 100 mL | Field: store in cooler at 1-6 °C  Lab: filter within 24 hours, but as close to time of sample collection as possible; until filtered, store in refrigerator at 1-6 °C; store filtrate in -80oC freezer for up to 30 days until analysis |
| Urban stream/ditch water, collected via grab samples and first flush sampler samples | 5-day biochemical oxygen demand | plastic | 1,000 mL | Field: store in cooler at 1-6 °C or at ambient temperature if to be analyzed immediately at lab  Lab: store at room temperature and start analysis within six hours of collection or store in refrigerator at 1-6 °C and start analysis within 24 hours |
| Urban stream/ditch water, collected via grab samples and first flush sampler samples | Turbidity | plastic | 100 mL | Field: store in cooler at 1-6 °C |
| Urban stream/ditch water, collected via grab samples and first flush sampler samples | Total Suspended Solids (TSS) | plastic | 500 mL | Field: store in cooler at 1-6 °C  Lab: store in refrigerator at 1-6 °C and start analysis within 7 days |
| Urban stream/ditch water, collected via grab samples and first flush sampler samples | Volatile Suspended Solids (VSS) | plastic | 500 mL (use same water as TSS) | Field: store in cooler at 1-6 °C  Lab: store in refrigerator at 1-6 °C and start analysis within 7 days |
| Urban stream/ditch water, collected via grab samples and first flush sampler samples | Ammonia-nitrogen (NH3-N) | plastic | 125 mL | Field: store in cooler at 1-6 °C  Lab: store in refrigerator at 1-6 °C and start analysis within 48 hours |
| Urban stream/ditch water, collected via grab samples and first flush sampler samples | Optical brighteners | amber glass | 500 mL | Field: store in cooler at 1-6 °C  Lab: store in refrigerator at 1-6 °C and start analysis within 8 days |
| Urban stream/ditch water, collected via grab samples and first flush sampler samples | Toxicity | plastic | 125 mL | Field: store in cooler at 1-6 °C  Lab: store in refrigerator at 1-6 °C and start analysis within 24 hours |

## Sample Handling and Custody Requirements

For EQL samplers at the time of sampling, a chain of custody (COC) Form 218 (Appendix A: Forms) must be filled out. The following information must be recorded by samplers:

* Date sample was collected
* Time sample was collected
* Location of sample: city, general location, and specific location.
* Example for a river sample: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
* Name of sampler
* ID of sampling bottle is the site name and the date collected.
* Analysis (e.g., bacteria) to be conducted, which must also be written in indelible ink on the sample bottle
* Environmental conditions (e.g., waves, currents, tide, wind, sky, rain, runoff)
* Describe in comments section any problems encountered during sampling and corrective actions taken

The sample collector is considered to have custody of the sample until relinquishing the sample. This sample is properly in the custody of the sampler as long as the sample is in possession of the sampler, within sight of the sampler, or locked in a secure place. When the sampler relinquishes custody he/she should sign, date, and write the time the sample was relinquished on the COC form. The person receiving the sample should then sign, date, and write the time the sample was received on the same line. The sample can be relinquished to other qualified individuals in the same manner. Sample receipt in the laboratory is indicated by the Laboratory Director, Laboratory Master Technician, or a Laboratory Technician accepting the sample and documenting it on the COC form. If the same individual transports the sample to the lab and processes that sample in the laboratory, then that person will record both accepting and relinquishing the sample on the COC form. In addition to the COC, the sample collector also has a Field Measurement Form (2000F RG). This form is attached in .

### Sample Receiving and Storage

Samples must be delivered to the laboratory in coolers packed in ice less than six hours after sample collection. Analysis of the samples must begin within the stated hold times for each parameter from the time of sample collection. At the beginning of sampling, a sample bottle containing water should be placed in the cooler with ice, and then upon delivery of the cooler to the laboratory, the water in this bottle is measured to determine the sample receipt temperature.

Prior to accepting custody and signing for the samples, the laboratory representative verifies that all samples submitted are listed on the COC and that the COC documentation is complete. Received samples and corresponding documentation are carefully reviewed for compliance with regard to condition of containers, sample preservation and temperature (i.e., reading temperature of water blank in cooler), holding times (collection date/time), and accurate identification on the COC.

Once the COC has been verified against the delivered samples, sample information is entered into the laboratory receipt log. The receipt log for samples is kept as a Microsoft Excel spreadsheet. The file is password protected.

Samples received by the laboratory are identified by unique laboratory identification numbers. The first character is an “E” which identifies it as an EQL sample. The next two characters identify the year in which the sample was received (i.e., 08 represents the year 2008). The final four characters are numbers assigned sequentially to identify the sample relative to the order that the sample was received. The sample number E11-0023 therefore is the 23rd sample received in 2011 by EQL for analysis.

The sample’s EQL laboratory number is transcribed to each container associated with that sample using an indelible marker. Numbered samples are stored in secured areas according to aliquot preservation requirements.

At the end of the day or as soon as practical, the receipt log for all samples received on a day is printed, Form 220, and placed in a logbook in chronological order. The printed sheet(s) must be reviewed for correctness and then initialed at the bottom of the sheet where it states:

“Printed (date of printing) by \_\_\_\_\_” and “Approved (date of printing) by \_\_\_\_”

These hard copy pages of the receipt log are numbered sequentially. In the event an error is later found in the receipt log, the change must be made in the spreadsheet and then corrected on the appropriate hard copy page. The hard copy corrections must be made by drawing a single line through the error, writing the correct data above or to the side, and initialing and dating the entry.

### Sample Distribution and Handling

Samples retrieved from their designated storage areas must be documented in an internal COC record, Form 217. Personnel removing samples from the storage areas are required to record the sample numbers removed, date, time, and their initials on the form. Staff must also document on that form the date and time samples are returned to storage. Several coolers and a refrigerator in the laboratory are for temporary storage of samples requiring refrigeration and awaiting preparation or analysis.

Notification of samples with parameters with critically short hold times (i.e., less than 48 hours) is provided verbally or in writing to the laboratory analytical staff on the day of receipt of such samples. Once notified, it is the responsibility of the analyst to perform the requested analysis within the appropriate hold time.

### Sample Disposal

In general, samples are disposed of 24 hours after results have been reported to the client. Arrangements for shorter or longer storage times are made with client approval based on specific project requirements. All sample container labels are removed or obliterated prior to disposal. Destruction of samples are noted on internal COC forms.

All samples suspected to be bacterially hazardous, incubated samples, used media, and bacteria control samples are sterilized by autoclaving for 30 minutes at 121 °C. In general, other samples found to be hazardous, or RCRA “D” listed, are returned to the client for disposal. Other hazardous wastes are disposed of by the science building staff by sending directly to an in-state permitted landfill.

Sterilized and non-hazardous aqueous samples are disposed of by pouring the sterilized, neutralized, or non-hazardous sample into a conventional drain to the municipal sewage treatment system. Non-hazardous solid wastes (including emptied disposable containers from aqueous samples) are disposed of by placing in a dumpster for municipal landfill disposal. The date of sample disposal is recorded on the internal COC form, Form 217, and autoclave sterilization log, Form 1070ASL.

## Analytical Methods

### Control of Analytical Processes

All aspects of laboratory operations are controlled by the key documents, the quality assurance manual (QAM) and standard operating procedures (SOPs). The SOPs detail and document the procedures which implement the activities and requirements specified in the QAM.

To perform the tasks described in this QAPP, the EQL uses 5 field and 14 laboratory analysis procedures:

* Total coliform and E. coli by IDEXX Colilert-18TM QuantiTrayTM method , based on IDEXX 06-02027-18
* Enterococcus by IDEXX EnterolertTM Quanti-TrayTM,method, based on IDEXX 06-02150-07
* Bacteriodes thetaiotamicron (GenBac), Bacteriodes dorei (BacHum), and Bacteriodes canine (BacCan) by qPCR based on Siefring et al. 2008 and EPA Method B, 2010
* 5-day biochemical oxygen demand (BOD5) by measuring oxygen consumed in incubated samples in a 5-day period, based on Method 5210 B. of *Standard Methods*
* Turbidity by nephelometry, based on Method 2130 B. of *Standard Methods*
* Total Suspended Solids (TSS) by gravimetric measurement, based on Method 2540 D. of *Standard Methods*
* Volatile Suspended Solids (VSS) by ignition at 550oC, based on Method 2540 E. of *Standard Methods*
* Ammonia-nitrogen (NH3-N) by fluorometry, based on Holmes et al. 1999.
* Optical Brighteners by fluorometry, based on Cao et al. 2009, Dickerson et al. 2007, Hartel et al. 2007a, Hartel et al. 2007b
* Water toxicity by Kingwood Diagnostics IQ Toxicity Test™, <http://www.kingwooddiagnostics.com/> verified by the USEPA under their Environmental Technology Verification Program
* Dissolved oxygen by membrane electrode method, based on Method 4500-O G. of *Standard Methods*
* pH (hydrogen ion concentration) by electrometric method, based on Method 4500-H+ B. of *Standard Methods*
* Conductivity by electrical conductivity, based on Method 2510 B. of *Standard Methods*
* Water and air temperature by thermometer or thermistor, based on Method 2550 B. of *Standard Methods*
* Salinity by electrical conductivity, based on Method 2510 B. of *Standard Methods*

The step-by-step procedures of these techniques are provided in EQL SOPs:

* 503 (Total coliform and E. coli)
* 504, 505, 506, 507 (Bacteriodes thetaiotamicron (GenBac))
* 504, 505, 506, 508 (Bacteriodes dorei (BacHum))
* 504, 505, 506, & one pending (Bacteriodes canine (BacCan))
* 430 (BOD5)
* 406 (Turbidity)
* 435 (Total Suspended Solids)
* 436 (Volatile Suspended Solids)
* 470 (Ammonia-nitrogen)
* 602 (Optical Brighteners)
* 601 (IQ Toxicity Test)
* 420, 422, and 423 (field measurement of pH, DO, temperature, and conductivity)
* 404 (Salinity)

All EQL SOPs referenced in this QAPP are provided in Appendix D: Environmental Quality Lab Standard Operating Procedures.

When issues occur in the laboratory they are handled by the analyst. Appropriate corrective actions are given in SOP 201.

When samples are completely used or destroyed a notation is made on the internal chain of custody.

Laboratory turnaround time is generally associated with meeting holding times for samples.

## Quality Control (QC)

### Dissemination of Quality Requirements

The laboratory uses several means of communication to ensure staff is informed of all quality requirements. Routine operational requirements are communicated to applicable staff through distribution of the QAPP and laboratory SOPs. All these documents are controlled internally and are issued to selected laboratory staff on an individual basis, depending on staff assignment, task responsibilities, and work location. The QAPP and all SOPs are available to all laboratory staff on the laboratory's computer network. Changes in requirements are communicated to laboratory staff by distribution of revisions to this QAPP and applicable SOPs.

Any laboratory staff member observing any occurrence (e.g., equipment failure) that impacts laboratory capabilities or schedule of deliverables (i.e., analysis results are to be reported to SC DHEC and clients within 24 hours of completion of analysis) must immediately bring that observation to the attention of the Laboratory Director. The Laboratory Director shall immediately communicate the situation to the affected customer. These communications shall be recorded in the Laboratory Director's telephone logbook, and a copy shall be placed in the project files. The Laboratory Director determines necessary corrective actions for such occurrences.

Make a list of the instruments, the parameters measured, and the inferences to be made. This list will clarify the measurement procedure and be useful for the person making the measurements and the person reviewing the plan. This may be presented in a table (see Table 3), a list or in paragraph format.The intent of this section is to identify the needs and procedures for calibration. Describe how and when the instrument will be calibrated (example Table 4). **Describe the traceability of the calibration standard to some authenticated system and describe the procedure for maintaining standards**. It is important to distinguish the difference between calibrating and checking accuracy. To calibrate an instrument is to adjust the output so that the reading is accurate. For a balance, this may be accomplished by adjusting the tension on a spring or adjusting a potentiometer. For a spectrophotometer this may be accomplished by making a standard curve of a dilution series and applying the mathematical fit (calibration curve) to the measurements of unknown samples. Some instruments need calibration frequently (ie. a pH meter is calibrated each time it is used), while others need calibration rarely (ie. a balance). Some instruments require calibration at several concentrations. Often instrument detection limits determine the accuracy of measurements at the extremes of instrument sensitivity. Although values may be recorded by some instruments, those values may have no meaning if they fall outside detection limits. For those measurement systems with either high or low limitations a description of how to deal with values that fall outside acceptable limits must be described a

Quality control (QC) procedures for EQL measurements in this project are summarized in Table 4 through Table 17. When recording results of QC measurements on samples (e.g., duplicate analysis), an acronym suffix is added to the sample number; the suffixes are as follows:

duplicate = D or DUP replicates = R# or REP#

matrix spike = MS matrix spike duplicate = MSD

Acronyms for recording other QC measurements are as follows:

blank = B or BLK method blank = MB

calibration standard = CAL or CALIB calibration verification standard = CV

initial calibration verification standard = ICV primary standard = PS

working standard = WS laboratory control sample = LCS

Table 4. Summary of QC requirements for Total Coliform and E. coli analysis by Colilert-18

|  |  |  |  |
| --- | --- | --- | --- |
| **QC Sample or Activity** | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | Criteria for LCS recovery and duplicate precision | Repeat until acceptable |
| Media sterility check | Prior to use of new lot of Colilert-18 and weekly | No fluorescence | Investigate problem. Eliminate contaminations. Obtain new lot of Colilert-18 if necessary. Repeat until successful before using Colilert-18 lot. |
| Media positive check with control culture | Prior to use of new lot of Colilert-18 and weekly | Fluorescence | Investigate problem. Obtain new lot of Colilert-18 if necessary. Repeat until successful before using Colilert-18 lot. |
| Media negative checks with control cultures (gram+ and  gram-) | Prior to use of new lot of Colilert-18 | No fluorescence | Investigate problem. Eliminate contaminations. Obtain new lot of Colilert-18 if necessary. Repeat until successful before using Colilert-18 lot. |
| Method blank | At least weekly,  prior to sample analysis | < 20 CFU/100 mL | Clean analytical system and repeat MB analysis. Identify and eliminate source of contamination. |
| Sample duplicate or matrix spike duplicate | At least one (1) weekly, and one with all large sample batches (~20 samples) | RPD < 200% for <150 CFU/100 mL  RPD < 100% for > 150 CFU/100 mL | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | Criteria for LCS recovery and duplicate precision | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |
| LCS = laboratory control sample QC = quality control  MB = method blank %R = percent recovery  MDL = method detection limit RL = reporting limit  PE = performance evaluation RPD = relative percent difference | | | |

Table 5. Summary of QC requirements for Enterococci analysis by Enterolert

|  |  |  |  |
| --- | --- | --- | --- |
| **QC Sample or Activity** | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | Criteria for LCS recovery and duplicate precision | Repeat until acceptable |
| Media sterility check | Prior to use of new lot of Colilert18 and weekly | No fluorescence | Investigate problem. Eliminate contaminations. Obtain new lot of Enterolert if necessary. Repeat until successful before using Enterolert lot. |
| Media positive check with control culture | Prior to use of new lot of Enterolert and weekly | Fluorescence | Investigate problem. Obtain new lot of Enterolert if necessary. Repeat until successful before using Enterolert lot. |
| Media negative checks with control cultures (gram+ and gram-) | Prior to use of new lot of Enterolert | No fluorescence | Investigate problem. Eliminate contaminations. Obtain new lot of Enterolert if necessary. Repeat until successful before using Enterolert lot. |
| Method blank | At least weekly,  prior to sample analysis | < 20 CFU/100 mL | Clean analytical system and repeat MB analysis. Identify and eliminate source of contamination. |
| Sample duplicate or matrix spike duplicate | At least one (1) weekly, and one with all large sample batches (~20 samples) | RPD < 200% for <150 CFU/100 mL  RPD < 100% for > 150 CFU/100 mL | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | Criteria for LCS recovery and duplicate precision | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |
| LCS = laboratory control sample QC = quality control  MB = method blank %R = percent recovery  MDL = method detection limit RL = reporting limit  PE = performance evaluation RPD = relative percent difference | | | |

Table 6. Summary of QC requirements for 5-day BOD analysis

|  |  |  |  |
| --- | --- | --- | --- |
| **QC Sample or Activity** | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | Criteria for LCS recovery and duplicate precision | Repeat until acceptable |
| Dilution water blank | Daily prior to sample analysis | < 0.2 mg/L DO depletion | Clean analytical system and repeat MB analysis. Identify and eliminate source of contamination. |
| Minimum residual DO and minimum DO depletion | For all measurements | Minimum DO depletion 2.0 mg/L  Residual DO in bottle > 1.0 mg/L | Results not considered to be valid |
| Seed control | For every preparation batch | DO uptake attributable to seed added to each bottle generally 0.6 to 1.0 mg/L but seed amount must provide acceptable GGA recovery | Investigate and identify the problem. If system is out of control, reanalyze entire batch. |
| Glucose-glutamic acid (GGA) check standard | One (1) per preparation batch | 198 ± 30.5 mg/L | Investigate and identify the problem. If system is out of control, reanalyze entire batch. |
| Matrix spike (GGA) | When suspect matrix interference | 75-125% R | Investigate problem. If system accuracy is in control, qualify results. If system accuracy is out of control, reanalyze entire batch. |
| Sample duplicate or matrix spike duplicate (GGA) | One (1) per preparation batch | RPD < 25% | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | Criteria for LCS recovery and duplicate precision | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects. Once a year a successful analysis. | Determined by PE provider | Investigate all unacceptable results. |
| LCS = laboratory control sample QC = quality control  MB = method blank %R = percent recovery  MDL = method detection limit RL = reporting limit  MS = matrix spike RPD = relative percent difference  PE = performance evaluation | | | |

Table 7. Summary of QC requirements for turbidity analysis by Hach meter

|  |  |  |  |
| --- | --- | --- | --- |
| QC Sample or Activity | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | Criteria for duplicate precision  RPD < 25% | Repeat until acceptable |
| 4-Point calibration plus deionized water blank | At least quarterly | 90-110% R (measured value of primary standards when analyzed as samples within 10% of expected values) | Investigate problem. Correct any obvious problems. Repeat calibration until acceptable. |
| Assign values to permanent transfer standards using formazin primary standards | At least quarterly | Measurement after acceptable 4-point calibration and values within 10% of previous established values | Investigate problem. Correct any obvious problems including replacing transfer standards if necessary. Repeat until acceptable. |
| Daily calibration check | Immediately prior to and after sample analysis | GELEX Secondary Turbidity Standards should read within 10% of assigned values | Investigate problem. Correct any obvious problems. If necessary reassignment of GELEX values and reanalyze samples. Repeat calibration check until acceptable. |
| Method blank | Daily prior to sample analysis | <1.0 NTU (i.e., < RL) | Clean analytical system and repeat MB analysis. Identify and eliminate source of contamination. |
| Sample analysis | For all sample analyses | Direct sample reading within acceptable measurement range (i.e., 1.00 NTU to 4000 NTU) | If reading below range report result as < RL. If result above range dilute sample. |
| Sample duplicate | One (1) per preparation batch | RPD < 25% | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | 75-125% R  RPD < 25% | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |
| LCS = laboratory control sample QC = quality control  MB = method blank %R = percent recovery  MDL = method detection limit RL = reporting limit  MS = matrix spike RPD = relative percent difference  PE = performance evaluation | | | |

Table 8. Summary of QC requirements for TSS

|  |  |  |  |
| --- | --- | --- | --- |
| QC Sample or Activity | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | 90 – 110% R  < 10% RSD | Repeat until acceptable |
| Balance Calibration Check | Prior to weighing any sample filters | Weight of certified 200 mg weight:  0.1998 – 0.2002 g | Investigate problem including cleaning weight and balance. If balance is out of calibration attempt recalibration or use another balance until obtain acceptable calibration check. |
| Method Blank | At least one (1) per analysis batch of up to 10 samples | For 1.0 L blank filtered: < 1.0 mg/L | Investigate, identify, and correct the problem. If system accuracy is in control, qualify results. If system accuracy is out of control, correct problem before analyzing samples |
| Sample analysis | For all sample analyses | Total residue on filter:  >2.5 mg to < 200 mg | If total residue on filter < 2.5 mg report result as < RL  If total residue on filter > 200 mg filter a smaller volume of sample. |
| Laboratory Control Sample | At least one (1) per year | 90 – 110% R | Investigate, identify, and correct problem. If system accuracy is in control, qualify results. If system accuracy is out of control, correct problem before analyzing samples. |
| Sample duplicate | One (1) per preparation batch of up to 10 samples | RPD ≤ 5% | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | Criteria for LCS recovery and duplicate precision | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |
| LCS = laboratory control sample QC = quality control  MB = method blank %R = percent recovery  MDL = method detection limit RL = reporting limit where RL = (2.5 mg /mL filtered) x 1000 mL  MS = matrix spike RPD = relative percent difference  PE = performance evaluation RSD = relative standard deviation | | | |

Table 9. Summary of QC requirements for VSS

|  |  |  |  |
| --- | --- | --- | --- |
| QC Sample or Activity | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | 90 – 110% R  < 10% RSD | Repeat until acceptable |
| Balance Calibration Check | Prior to weighing any sample filters | Weight of certified 200 mg weight:  0.1998 – 0.2002 g | Investigate problem including cleaning weight and balance. If balance is out of calibration attempt recalibration or use another balance until obtain acceptable calibration check. |
| Method Blank | At least one (1) per analysis batch of up to 10 samples | For 1.0 L blank filtered: < 1.0 mg/L | Investigate, identify, and correct the problem. If system accuracy is in control, qualify results. If system accuracy is out of control, correct problem before analyzing samples |
| Sample analysis | For all sample analyses | Total residue on filter:  >2.5 mg to < 200 mg | If total residue on filter < 2.5 mg report result as < RL  If total residue on filter > 200 mg filter a smaller volume of sample. |
| Sample duplicate | One (1) per preparation batch of up to 10 samples | RPD ≤ 5% | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | Criteria for LCS recovery and duplicate precision | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |
| LCS = laboratory control sample QC = quality control  MB = method blank %R = percent recovery  MDL = method detection limit RL = reporting limit where RL = (2.5 mg /mL filtered) x 1000 mL  MS = matrix spike RPD = relative percent difference  PE = performance evaluation RSD = relative standard deviation | | | |

Table 10. Summary of QC requirements for determination of ammonia by Turner fluorometer

|  |  |  |  |
| --- | --- | --- | --- |
| QC Sample or Activity | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Calibration | With each run | r2 value near 0.999 | Investigate problem. Correct any obvious problems. Repeat calibration until acceptable. |
| Method Blank | Before any run and between sample sets | <RL | Rinse cuvette and repeat until criteria is met. |
| Sample Duplicate | One (1) per sample set (10 samples) | RPD + or - 5% | Investigate problem. Report to Laboratory Director if problem is unknown. |
| Matrix Spike | One (1) per sample set (10 samples) | RPD + or - 25% | Investigate problem. Report to Laboratory Director if problem is unknown. |
| Laboratory Control Standard | One (1) with calibration and with each calibration check | 90-110%R | Investigate problem. Correct any obvious problems. Repeat until acceptable. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | 75-125% R  RPD < 25% | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |
| LCS = laboratory control sample QC = quality control  MB = method blank %R = percent recovery  MDL = method detection limit RL = reporting limit  MS = matrix spike RPD = relative percent difference  PE = performance evaluation | | | |

Table 11. Summary of QC requirements for determination of optical brighteners by Turner fluorometer

|  |  |  |  |
| --- | --- | --- | --- |
| QC Sample or Activity | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | Criteria for duplicate precision | Repeat until acceptable |
| 5-Point calibration | Quarterly | 90-110% R (measured value of all standards when analyzed as samples within 10% of expected value) | Investigate problem. Correct any obvious problems. Repeat calibration until acceptable. |
| Solid secondary standard value establishment | Quarterly | Measurement after acceptable 5-point calibration and value within 10% of previous established value | Investigate problem. Correct any obvious problems including obtain new solid secondary standards if necessary |
| Calibration check with solid secondary standards | Daily prior to sample analysis | 90-110% R | Investigate problem. Correct any obvious problems including new 5-point calibration if necessary. Repeat calibration check until acceptable. |
| Method blank | Daily prior to sample analysis | < RL | Clean analytical system and repeat MB analysis. Identify and eliminate source of contamination. |
| Sample analysis | For all sample analyses | Direct sample reading within calibration range (i.e., lowest and highest calibration standard concentrations) | If reading below range report result as < RL. If result above range dilute sample. |
| Sample duplicate | One (1) per preparation batch | RPD < 25% | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | 75-125% R  RPD < 25% | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |
| LCS = laboratory control sample QC = quality control  MB = method blank %R = percent recovery  MDL = method detection limit RL = reporting limit  MS = matrix spike RPD = relative percent difference  PE = performance evaluation | | | |

Table 12. Summary of QC requirements for water toxicity analysis

|  |  |  |  |
| --- | --- | --- | --- |
| QC Sample or Activity | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Initial Demonstration of Capability | All technician performing test must perform once at end of training prior to actual sample analysis | The RPD of four (4) samples utilizing the same matrix must be within 90-110% of the results obtained by a trained technician reading an independently setup sample | Investigate issue. Observe technician-in-training for proper technique. Retrain as indicated. Repeat IDC’s until results within acceptable criteria. |
| Negative Method Control | One (1) sets of triplicate per each batch analysis | ≥15 fluorescing organisms for each set of three (3) Method Blank cells | Investigate issue. Re-run Method Blanks in a known “virgin” testing cell. Repeat until results are within acceptable limits. |
| Positive Method Control | One (1) sets of triplicate per each batch analysis | ≥4 non-fluorescing organisms for each set of three (3) Method Blank cells | Investigate issue. Re-run Method Blanks in a known “virgin” testing cell. Repeat until results are within acceptable limits. |
| Sample Analysis | For each sample analyzed | Three (3) test chambers filled with six (6) Daphnia magna per each sample analyzed | Qualify any results that fail to meet criteria due to sample or testing reagent issues. |
| Laboratory Duplicate | One (1) per ten (10) samples analyzed | RPD ≤ 25% | Investigate issue. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch associated with specific QC duplicate. |
| Field Duplicate | One (1) per ten (10) samples analyzed | RPD ≤ 25% | Investigate issue. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch associated with specific QC duplicate. |

Table 13. Summary of QC requirements for measurements with Hach ruggedized probes

|  |  |  |  |
| --- | --- | --- | --- |
| QC Sample or Activity | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | LDO 97-104% of theoretical DO  pH ± 0.1 of expected  Others RPD < 25% | Repeat until acceptable. |
| Calibration stability monitoring | Immediately before calibration measure standards | Not applicable. | Not applicable. Results are used to monitor stability of probes and evaluate need for maintenance. |
| Calibration | Daily prior to sample analysis and after every 8 hours | After calibration, measure calibration standards (conductivity, pH, DO % saturation of water saturated air) as sample  pH ± 0.1 of expected, others 99-101% R | Investigate and fix any obvious problems. Repeat until acceptable. |
| Calibration check | Immediately following calibration | Measurement of calibration standards or LCS (conductivity, pH, DO % saturation of LCS or of water saturated air)  Cond. 90-110% R, pH ± 0.1 of expected, DO 97-104% sat  \*\***LDO method requires LCS to be read in duplicate with each calib. event**\*\* | Investigate and fix any obvious problems. Recalibrate and repeat until acceptable. |
| Field duplicate (duplicate sample collected at one of sampling sites | One (1) per sampling event | RPD < 25% | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze all sampling sites if possible. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | 75-125% R  RPD < 25% | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |

Table 14. Summary of QC requirements for Oakton pH meter

|  |  |  |  |
| --- | --- | --- | --- |
| **QC Activity** | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Standardize using two pH buffers that are within 3 pH units of each other | Before each meter use | +/- 0.1 pH units  of actual buffer value\* | Recalibrate the meter with new buffer solution. If this doesn’t solve the problem, the electrode should be cleaned, repaired, or replaced. |
| Measure a pH buffer bracketed by the two buffers used for standardization as a post-calibration check | Before each meter use | +/- 0.1 pH units  of actual buffer value\* |
| Periodically measure pH of standard buffer solution when measuring pH of samples | Every 10 samples | +/- 0.1 pH units of actual buffer value\* | Recalibrate the meter and reanalyze the samples measured after the last acceptable check measurement. |
| Check ATC thermometer against NIST-traceable thermometer | Quarterly (4 times/year) | +/- 0.05oC of a NIST-traceable thermometer | Recalibrate the ATC thermometer to a NIST-traceable thermometer |

*\*NOTE: Actual pH standard values are temperature dependent. See 5.6.3. in SOP 422.*

Table 15. Summary of QC requirements for YSI Pro Plus probes

|  |  |  |  |
| --- | --- | --- | --- |
| QC Sample or Activity | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | DO 97-104% of theoretical DO  Others 75-125% R  Others RPD < 25% | Repeat until acceptable. |
| Calibration stability monitoring | Immediately before calibration measure standards | Not applicable. | Not applicable. Results are used to monitor stability of probes and evaluate need for maintenance. |
| Calibration | Daily prior to sample analysis and after every 8 hours | After calibration, measure calibration standards (conductivity, pH, DO % saturation of water saturated air) as sample  pH ± 0.1 of expected, others 99-101% R | Investigate and fix any obvious problems. Repeat until acceptable. |
| Calibration check | Immediately following calibration | Measurement of calibration standards or LCS (conductivity, pH, DO % saturation of LCS or of water saturated air)  Cond. 90-110% R, pH ± 0.1 of expected, DO 97-104% sat  \*\***DO method requires LCS to be read in duplicate with each calib. event**\*\* | Investigate and fix any obvious problems. Recalibrate and repeat until acceptable. |
| Field duplicate (duplicate sample collected at one of sampling sites | One (1) per sampling event | RPD < 25% | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze all sampling sites if possible. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | 75-125% R  RPD < 25% | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |

Table 16. Summary of QC requirements for salinity measurement by Hach HQ40d conductivity meter

|  |  |  |  |
| --- | --- | --- | --- |
| **QC Sample or Activity** | **Minimum Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | Criteria for LCS recovery and duplicate precision | Repeat until acceptable |
| Method blank | One (1) per preparation batch | < 0.2 0/00 | Clean analytical system and repeat MB analysis. Identify and eliminate source of contamination. |
| Laboratory control sample | One (1) per preparation batch | 90-110% R for <100/00  95-105% R for > 100/00 | Investigate and identify the problem. If system accuracy is in control (e.g., MS acceptable), no corrective action needed. If system is out of control, reanalyze entire batch. |
| Matrix spike | When suspect matrix interference | 80-120% R for <100/00  90-110% R for > 100/00 | Investigate problem. If system accuracy is in control, qualify results. If system accuracy is out of control, reanalyze entire batch. |
| Sample duplicate or matrix spike duplicate | One (1) per preparation batch | RPD < 25% for <100/00  RPD < 20% for >100/00 | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | Criteria for LCS recovery and duplicate precision | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |
| LCS = laboratory control sample QC = quality control  MB = method blank %R = percent recovery  MDL = method detection limit RL = reporting limit  MS = matrix spike RPD = relative percent difference  PE = performance evaluation | | | |

Table 17. Summary of QC requirements for GenBac, BacHum, and BacCan

|  |  |  |
| --- | --- | --- |
| **QC Sample or Activity** | **Acceptance Criteria** | **Corrective Action** |
| QC sample analysis | Any QC sample analysis (e.g., method blank, laboratory replicate, field replicate) should be subjected to exactly the same analytical procedures as those used on individual sample analyses. | Repeat until acceptable |
| Standard curves | Standard curve runs should be evaluated for agreement between duplicates (curve R2 > 0.97) and overall linearity. Slope values for standard curves are considered acceptable for unknown sample quantitation when between -3.75 and -3.20. These slopes represent PCR efficiency values between 85% and 105%. | Repeat until acceptable |
| Sample analysis | All unknown samples should be analyzed in duplicate. Duplicate samples >100% different should be analyzed again. | Repeat until acceptable |

### Verification Methods and Calculations

During the data review process, standardized methods and calculations are used to examine the measurement process against the specified QC requirements. These general methods and calculations, organized by DQI characteristics outlined in Section A, are described in the remainder of this section.

#### Representativeness

The appearance and records for samples should, at a minimum, be checked against project requirements for the following:

* Sampling protocols
* Sample types
* Sample containers
* Sample sizes
* Sample numbers
* Sample preservation
* Sample storage
* Sample analysis hold time
* Maintenance of sample chain-of-custody

#### Accuracy

Accuracy (bias) is a measurement of the extent to which a measured value of a quantity (parameter or analyte) agrees with the accepted value of that quantity. It is assessed by the analysis of samples of known concentration for the analytes of concern.

For LCSs, calibration standards, field reference standards, or additional QC samples of known concentration, accuracy is quantified by calculating the *percent recovery* (%R) of analyte from a known quantity of analyte as follows:



where:

Vm = measured value (concentration determined by analysis)

Vt = true value (concentration or quantity as calculated or certified by the manufacturer)

A matrix spike (MS) sample or a matrix spike duplicate (MSD) sample is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology. A known amount of the analyte of interest is added to a sample prior to sample preparation and instrumental analysis. To assess the effect of sample matrix on accuracy, the %R for the analyte of interest in the spiked sample is calculated as follows:



where:

SSR = spiked sample result

SR = sample result

SA = spike added

#### Precision

Precision is a measurement of the random error in an analytical measurement process. It reflects the degree of agreement between independent measurements determined by the analysis of replicate samples. When calculated for duplicate sample analyses, precision is expressed as the *relative percent difference* (RPD), which is calculated as:



where:

S = first sample value (original result)

D = second sample value (duplicate result)

When precision is calculated for three or more replicate determinations, the *relative standard deviation* (RSD), also known as the coefficient of variation, expressed in units of percentage, is used. This is an expression of the spread of the data relative to the mean value of the determinations. The specific formulas used for calculating the RSD are:







where:

 = mean of n measurements

xi = result value for the ith measurement

n = total number of measurements

s = standard deviation

#### Method Detection Limits

Method detection limits (MDLs) are determined for each analyte for each method used. These MDLs are determined by (a) conducting replicate analyses of standards at quantities approximately one to five times the estimated MDL, (b) determining the standard deviation, s, of the replicate measurements, and then (c) calculating the MDL from:

MDL = t (n-1, 1 - ∝ = 0.99)  ×s

where:

n = number of replicate analyses

t(n-1,1 - ∝ = 0.99) = t distribution value appropriate to a 99% confidence level (one-tailed) and standard deviation estimate with n - 1 degrees of freedom

s = standard deviation of the data set

The MDL calculated in this manner represents the minimum amount of a substance that can be measured and reported, with 99% confidence that the analyte quantity is greater than zero.

The MDL does *not* represent the analyte quantity for which there is a 99% probability that the analyte will be detected; there is a 50% probability of detection and reporting of the analyte whose actual amount is at the MDL. The analyte quantity at which there is a 99% probability that the analyte will be detected and reported is twice the MDL.

Because MDLs are usually determined using standards in a clean matrix, they represent optimum obtainable performance. MDLs for actual sample matrices are likely to be higher than those determined using clean matrices.

#### Quantitation/Reporting Limits

Because of significant uncertainty (about 33% RSD) associated with MDLs determined in a "clean" matrix, plus possible additional variability due to actual sample matrix, EQL uses higher levels, referred to as "limits of quantitation" or "reporting limits", down to which it routinely reports measured values.

The *limit of quantitation* (LOQ) is defined as 10 times the standard deviation (s) from the MDL determination. Therefore, the LOQ is roughly 3.33 times the MDL, since the MDL is usually about three times s.

The *reporting limit* (RL) is not as rigidly, and usually not as conservatively, defined as the LOQ. It is usually chosen at a level two to 10 times higher that the MDL. As much as possible, it is also chosen at a level which is below applicable regulatory action levels and which simplifies data review and reporting (e.g., RL of 1.0 μg/L for numerous parameters of similar chemical behavior, MDLs, and regulatory action levels).

#### Completeness

The characteristic of completeness is a measure of the amount of valid analytical data obtained compared to the total number of analyses performed. Valid analytical data are those for which all QC specifications are met. Completeness of the reported data (expressed as a percentage) is calculated as:



where:

Mv = number of measurements judged to be valid (meets all QC specifications)

Mt = total number of measurements performed (based upon number of samples submitted)

#### Comparability

Comparability of analysis results is evaluated by at a minimum checking the following against project requirements:

* Analysis method utilized
* Analysis QC measurement results
* Units utilized for reporting measurement values

#### Rejection of Data

Rejection of an analytical result for a sample may be required if established quality control acceptance criteria are not satisfied at any point during the course of analysis. Nominal quality control decision criteria are provided in analytical method SOPs and the corresponding data review checklists.

Additionally, the EQL uses a statistical outlier test (*Standard Methods*, 1010 B. Statistics, 17th through 21st Editions) for evaluation of a questionable value from a group of replicate readings, measurements, results, etc., for an individual sample or standard. Briefly, the test involves dividing the difference between the questionable value and the replicates' mean value by the standard deviation for all replicate values, to calculate a quotient, T. The questionable value is rejected if the calculated T is greater than an established rejection T. The outlier test is conducted at the 99% confidence level, which means if the calculated T exceeds the rejection T0.99, then the questionable value may be rejected with 99% probability that it is significantly different from the other values (**Table #**).

Table 18. Outlier test for evaluation of a questionable group from a group of replicate values

Rejection

Formula for Number of Quotient

Questionable Valuea Calculating Tb Values T0.99

Xave – X1

Smallest value (X1) T = ⎯⎯⎯⎯ 3 1.15

s

4 1.49

5 1.75

Xn – Xave 6 1.94

Largest value (Xn) T = ⎯⎯⎯⎯

s 7 2.10

8 2.22

9 2.32

10 2.41

1. 2.55
2. 2.66
3. 2.75

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

a Arrange values in order of increasing magnitude.

b If T > T0.99 reject questionable value.

Xave= average value for all replicates.

s = standard deviation for all replicates, where s = [∑(Xn - Xave)2/(n - 1)]1/2

### Instrument/Equipment Testing, Inspection, Maintenance Requirements

The term “equipment”, as used in this manual, includes equipment or instrumentation used in the areas of sample collection, preparation, or analysis. The laboratory utilizes all equipment (Table 19) as appropriate and necessary for a given technique, as specified in a referenced method, or as required by regulatory programs. The equipment investment and subsequent capabilities are sufficient for the laboratory’s field and laboratory tasks for this project. Except for the autoclave, Quanti-Tray sealer, and qPCR analysis system, there is a backup instrument for every critical instrument. There is a rapid response maintenance contract for the autoclave. In the event that EQL’s qPCR analysis system malfunctions, EQL has access to the qPCR analysis system at Hollings Marine Lab in Charleston, SC.

Table 19. Equipment list

|  |  |
| --- | --- |
| **Instrument** | **Number of Units** |
| Analytical Balance | 3 |
| Autoclave | 1 |
| Conductivity/Dissolved Oxygen/pH Field Meter | 3 |
| Fluorometer | 2 |
| Incubator | 3 |
| pH Meter | 3 |
| Oven | 3 |
| Refrigerator/Freezer | 10 |
| Spectrophotometer | 2 |
| Turbidity Meter | 2 |
| Water deionizing system | 4 |
| qPCR analysis system | 1 |
| Salinity meter | 2 |
| Quanti-Tray sealer | 2 |
| Water Bath | 2 |

#### Preventative Maintenance

Manufacturer recommended preventative maintenance schedules are performed internally for all equipment, in all lab areas. Additionally, some equipment, such as autoclave and analytical balances, require service checks by the commercial vendor. Service calls of this nature are scheduled by the Quality Assurance Officer or science building staff according to the maintenance schedule.

Maintenance logs are used to document any procedures performed either internally, or by vendor service technicians. These logs also document maintenance or repair which may be necessary as a part of corrective action resulting from QC failures. Documentation in the logs is the responsibility of the analyst or technician operating the instrument or equipment.

A summary of preventive maintenance activities for equipment utilized for this project is provided in Table 20**.**

Table 20. Instrument and equipment preventative maintenance and testing

|  |  |  |
| --- | --- | --- |
| **Instrument** | **Frequency** | **Preventive Maintenance** |
| Autoclave | Each use | Clean drain screen, measure maximum temperature |
| Monthly | Check timer, test sterility |
| Quarterly | Quarterly maintenance service |
| Balance | Each use | Check level and adjust if needed, clean after use, calibration verification |
| Monthly | Clean, level, calibration verification |
| Annual | Annual maintenance service, check electrical cord |
| Conductivity/Dissolved Oxygen/pH field meter | Each use | Insert batteries and turn on; after use rinse probes, clean meter, replace pH probe storage solution, and remove batteries. |
| Controlled temperature equipment | Daily | Check temperature and adjust if needed |
| Annual | Check temperature distribution, check electrical cord, clean instrument |
| Fluorometer | Each use | Plug in, turn on, allow 30 min. to warm up, check performance with secondary standards; after use turn off, unplug, and clean cuvettes |
| Annual | Check lenses and clean if needed, check electrical cord |
| Salinity meter | As needed | Clean the probe with a strong detergent solution and brush. Rinse thoroughly with DI water. |
| pH meter | Each use | Rinse probe, check probe electrolyte level, change electrode storage solution |
| As needed and annual | Clean probe, replace probe electrolyte, check electrical cord |
| Spectrophotometer | Each use | Plug in, turn on, allow 30 min. to warm up, check performance with blank and standards; after use turn off, unplug, and clean cuvettes |
| Annual | Check electrical cord |
| Quanti-Tray sealer | Monthly | Check sealer effectiveness by sealing 100 mL water colored with dye. If the colored water is observed outside the wells of the sealed Quanti-Tray, the sealer must be repaired or the back-up sealer used. |
| qPCR analysis system | Before initial use and as needed | Perform qualification plate test distributed by manufacturer. Check electrical cord. Replace lamp. |
| Thermometers | Annual | One-point or two-point calibration |
| Turbidity meter | Monthly | Turn on, allow 30 min. to warm up, check performance with secondary standards; after use turn off and clean cuvettes |
| Annual | Check electrical cord |
| Water deionizing system | Each use | Check water resistance |
| Semi-annual | Sterilize, change final filter |
| Annual | Check connections and electrical cord, change exchange cartridges if needed |

### Instrument Calibration and Frequency

Equipment requiring calibration must be calibrated according to manufacturer’s instructions or the analytical method. General guidelines for analytical instrument calibrations are covered in the corresponding analytical SOPs. A summary of instrument calibration procedures for this task’s measurements is provided in Table 21**.**

For equipment where documentation of the calibration can be obtained in the form of hardcopy printouts, the calibration data must be filed with the analytical run data. Where printouts are not possible, the following minimum information must be recorded in a calibration log or on the raw data sheet: equipment identification, calibration date, analyst initials, standard(s) used, certified concentration(s), equipment reading(s) per standard, calibration verification standard(s) results, due date for next calibration (if not “per use”).

It is the responsibility of the analyst performing calibration to record this information in the calibration log. Further, when persons who are not EQL staff perform calibration on any equipment, it is also the responsibility of the analyst to record the details of this work performed, and obtain any applicable certificates from the vendor.

### Inspection/Acceptance Requirements for Supplies and Consumables

To maintain efficient, safe, and high quality operations in a laboratory, it is essential that standardized and clearly understood procedures are used for ordering and receipt of materials and services. Consequently, the EQL requires its staff to follow CCU’s specific procurement procedures. These procedures include practices for source verification, ordering, receiving, inspection and testing, recordkeeping, and, if necessary, return to source.

The objectives of the laboratory's procurement procedures are as follows:

* Procurement procedures, including associated documentation, satisfy university and customer requirements
* Timely receipt of requested materials and services
* Received materials and services fulfill intended purposes
* Minimization of costs

*Where consumable items, such as solvents, standard gasses, reagents, etc. are involved, discuss acceptability rules and procedures used to inspect and evaluate.*

Specifically for this project, ordering information for critical equipment and supplies is listed in each SOP (Appendix D) for the applicable activity or analysis. Typically the Laboratory Director orders needed supplies using a CCU credit card or a purchase order. Supplies are received at the laboratory by the Laboratory Director or the Laboratory Master Technician or Laboratory Technician. They inspect the received supplies and log the supplies into the laboratory equipment and supplies inventory list. If received supplies do not fulfill advertised specifications or are damaged, the Laboratory Director contacts the supplier to discuss replacement.

Table 21. Instrument calibration procedures

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Instrument** | **Calibration Procedure** | **Frequency** | **Acceptance Criteria** | **Corrective Action if Unaccepatable** |
| Incubators and Water Bath | One-point or two-point calibration of thermometer with NIST traceable thermometer | Annual | + 0.5 ˚C | Replace thermometer |
| Refrigerators and pH Meters | One-point or two-point calibration of thermometer with NIST traceable thermometer | Annual | + 2.0 ˚C | Replace thermometer |
| Freezers and Ovens | One-point or two-point calibration of thermometer with NIST traceable thermometer | Annual | + 2.0 ˚C | Replace thermometer |
| Analytical Balance | Calibration verification using NIST traceable weights | Daily | + 0.1% | Clean and autocal or repair |
| Analytical Balance | Calibrated by service technician during annual maintenance | Annual | Professional service | Repair balance |
| Salinity meter | One-point calibration with certified standards | Every session | + 10% for <100/00  + 5% for > 100/00 | Investigate and correct problem. Repeat calibration until acceptable, if cannot recalibrate repair meter. |
| qPCR analysis system | Five to six point calibration curve near range of anticipated samples using standard of targeted assay | Every session or as needed | 85%-105% based on specific assay criteria | Investigate and correct problem. Repeat calibration until acceptable, if cannot recalibrate, repair instrument. |
| Quanti-Tray sealer | NA | NA | NA | NA |
| Fluorometer | Two-point calibration check with solid secondary standards | Every session | + 10% of established values | Investigate and correct problem. Perform new 5-pt calib. if necessary. |
| Fluorometer | Prepare series of liquid standards and preform five-point calibration, reestablish values of solid secondary standards | Quarterly | Acceptable 5-pt calibration and  + 10% of expected primary standard values | Investigate and correct problem. If necessary prepare new liquid stds or repair instrum. |
| pH meter | Two-point calibration with standard buffers | Every session | Slope 90-102%  pH + 0.1 | Clean probe, replace electrolyte, or replace probe as needed. Repeat calibration until acceptable. |
| Turbidity meter | Calibration check with gel secondary standards | Every session | + 10% of established values | Investigate and correct problem. Perform new 5-pt calib. if necessary. |
| Turbidity meter | Five-point calibration with liquid primary standards, reestablish values of gel secondary standards | Quarterly | + 10% of expected primary standard values | Investigate and correct problem. If necessary prepare new liquid stds or repair instrum. |
| Conductivity / Dissolved Oxygen / pH field meter | One-point conductivity calib., one-point dissolved oxygen calib. with water saturated air, two-point or three-point pH calib. | Weekly | Conductivity or salinity + 10%, dissolved oxygen + 5%, pH + 0.1 | Investigate and correct problem. Repeat calibration until acceptable, if cannot recalibrate repair meter. |
| Conductivity / Dissolved Oxygen / pH field meter | Repair by manufacturer or service technician | As needed | Per manufacturer | Repair meter |

*The intent of this section is to identify the needs and procedures for calibration. Describe how and when the instrument will be calibrated (example Table 4).* ***Describe the traceability of the calibration standard to some authenticated system and describe the procedure for maintaining standards****. It is important to distinguish the difference between calibrating and checking accuracy. To calibrate an instrument is to adjust the output so that the reading is accurate. For a balance, this may be accomplished by adjusting the tension on a spring or adjusting a potentiometer. For a spectrophotometer this may be accomplished by making a standard curve of a dilution series and applying the mathematical fit (calibration curve) to the measurements of unknown samples. Some instruments need calibration frequently (ie. a pH meter is calibrated each time it is used), while others need calibration rarely (ie. a balance). Some instruments require calibration at several concentrations. Often instrument detection limits determine the accuracy of measurements at the extremes of instrument sensitivity. Although values may be recorded by some instruments, those values may have no meaning if they fall outside detection limits. For those measurement systems with either high or low limitations a description of how to deal with values that fall outside acceptable limits must be described a priori..*

### Non-direct measurements

This is not applicable.

### Data management

The data management scheme is as follows:

A lab staff member collects the sample and preserves it according to the SOPs. The samples are brought to the laboratory. If they are performing the analysis they relinquish them to themselves. If not, they relinquish them to sample custodian who logs and disseminates the samples. The samples are analyzed. The analyst verifies the sample calculations and then they make a hard copy of the data and submit it to Dr. Trapp. Dr. Trapp performs a second verification. Then Dr. Trapp gives it to the Master Technician who also reviews the QC and then enters the data into a Data Archive Spreadsheet. A preliminary report is submitted to Dr. Libes. Dr. Libes validates the data. If issues occur, Dr. Trapp will act as an assistant validator and will review any anomalies found to determine if the anomaly is valid. Once validation is complete, the data are released to SC DHEC.

Data integrity is ensured by the amount of verification that is performed. Hardware and software issues are also avoided by verification at several levels.

#### Document Review and Approval

All of the laboratory's key quality documents and SOPs receive initial and annual review by applicable laboratory staff and are approved by the Laboratory Director. Review and approval of each document are recorded by signatures in a review and approval section in each document.

#### Document Control

All of the laboratory's key quality documents, namely the microbiology QAM and SOPs, are controlled documents. A controlled document has been through the preparation, review, and approval cycle and may not be changed after release and issue without going through a formal review and change authorization process. Each controlled document contains a document assignment page that assigns the document to a named individual, office, or lab area, indicates the controlled document copy number, and instructs the document assignee on how to maintain the document and enter changes.

Revisions of controlled documents are identified by a consecutive revision number and the date of the revision on the document title page and page headers within the document. Within one month of final change approval, changes are distributed to those assigned a controlled copy of the applicable document. Each change transmittal is assigned a sequential issue number, which indicates the number of revisions the document has undergone. A record of revisions will accompany each change transmittal to indicate the number and type of changes to the document. Any document designated as an “Uncontrolled Copy” is not subject to updated revisions.

#### Analysis Methods

Analytical method SOPs are the key guidance documents for analysis activities in the laboratory. The analytical method SOPs use the following general format:

* SOP Title Number, Revision Number, Date and Page Number header
* Review and Approval Signature Block
* Scope/Application
* References
* Definitions
* Safety
* Method (Apparatus/Materials, Reagents, Procedures, Quality Control, Corrective Action)
* Calculations

Data and associated records from analysis of samples and from support activities in the EQL for this project are identified in Table 22**.**

All laboratory paper records are stored in file cabinets within the secure laboratory facility for a period of one to three years. After that period the records are placed in labeled boxes and transferred to a locked room in a nearby university storage room. Electronic data are stored in the laboratory's desktop computers and on a restricted access (i.e., access restricted to Laboratory Director, Laboratory Master Technician, and Laboratory Technicians) intra-university network. Backup copies of electronic media are prepared at least annually and stored in a secure area off-site.

#### Disposition of Records

Records are stored for a nominal period of at least ten years. Records are stored for longer periods if requested or required by the customer or regulatory authority.

#### Requests for Records

Access to recent (i.e., within the previous year) laboratory records is restricted to laboratory personnel. Access to archived laboratory records is restricted to the Laboratory Director, Laboratory Master Technician, and Laboratory Technicians. All requests for laboratory records should be directed to one of those individuals. Original documents shall not be taken from the file storage area without permission from one of the listed individuals, and copying and distribution of such documents must also have their authorization.

Table 22. Data and records generated by field measurements, sample collection, and laboratory sample analysis

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Activity** | **Data Generator** | **Data Type** | **Data Format** | **Formsa** | **Referenceb** |
| Field measurements | Field measurer | Field measurement results | Written field log sheets | Forms 2000F, 2100, 4500 HCAL | SOP 420 |
| Sample collection | Sampler | Field information | Written Chain-of-Custody | COC Form 1060 | SOP 302 |
| Sample receipt | Laboratory Director, Laboratory Technician | Receipt custody and temperature | Written Chain-of-Custody | COC Form 1060 | QAM 4.2 |
| Receipt Log Spreadsheet | Receipt Log, Form 220 | QAM 4.3 |
| Internal custody | Laboratory Director, Laboratory Technician, Student | Time and location of storage | Written Chain-of-Custody | Form 217 | QAM 4.4 |
| Analysis | Laboratory Director, Laboratory Technician, Student | Total coliform (TC), E. coli (EC), Enterococcus, Bacteriodes thetaiotamicron (GenBac), Bacteriodes dorei (BacHum), Bacteriodes canine (BacCan), 5-day BOD, Turbidity, Total Suspended Solids (TSS), Volatile Suspended Solids (VSS), Ammonia-nitrogen, Optical brighteners, Toxicity | Written log sheets and calculation spreadsheet printouts | Forms 9230 CAL (TC & EC), 59230 EAL (Enterococcus), 210 & 5220 (BOD5), 2130 (Turbidity), 2540 D DL (TSS), 2540 E DL (VSS), 602 (Optical brighteners), 8711 (Tox), 2520 (Salinity), NH3-N, | SOPs 501, 503, 430, 406, 435, 436, 423, 422, 420, 404, 602, 601, 504, 505, 506, 507, 508, 509, 470, |
| Data review, verification and validation | Laboratory Director, Laboratory Technician | Analysis results | Written log sheets, calculation spreadsheet printouts, and runlog spreadsheet | Run Log | QAM 7.1 |
| Report | Laboratory Director, Laboratory Technician | Analysis results | Electronic template | Excerpt from Run Log Spreadsheet | QAM 7.4 |
| **a**  All forms are provided in Appendix A: Forms  **b** Referenced SOPs are provided in Appendix D: Environmental Quality Lab Standard Operating Procedures and QAM = EQL QA Manual | | | | | |

# Assessment/Oversight

## Assessments and Response Actions

Table 23. Assessments and response actions

|  |  |  |  |
| --- | --- | --- | --- |
| **Assessment** | **Frequency** | **Description** | **Information reported to** |
| Initial demonstration of capability (IDC) | Initially, prior to reporting client data independently | The analyst must prepare four aliquots of a known level of the analyte of interest, analyze them according to the appropriate method, and demonstrate the ability to recover the analyte within established acceptance criteria. | Analyst, Laboratory Director, Program Director, SCDHEC, EPA Region 4 |
| Data generator review | Every time data is generated | Conduct real-time review and verification of 100% of the data resulting from their activities. | Laboratory Director |
| Peer review | Every time data is generated | The peer reviewer(s) must be a qualified individual other than the data generator and must meet the minimum training and qualifications requirements for analysts. Data is reviewed for technical correctness for a minimum of the method, proper units/significant digits, calculation verifications, variations documented, transcription errors, complete data package, QC measurments within limits or qualified, and hold times were met or exceptions documented. | Laboratory Director |
| Analysis of internal and/or external performance evaluation (PE) samples | Once per year or as required by specific client contract requirements. | Analysis of a blind sample for the analyte(s) of interest. Results are evaluated for accuracy by a third party. | Laboratory Director, PE provider, clients, Program Director, SCDHEC, EPA Region 4 |
| Internal audits | Quarterly | Review of SOPs for referenced method, review of procedure, review of data files, review of logbooks, review of compliance with QA policies | Analysts, Lab Director, Program Director |
| External audits | Per request | Review of entire scope of accreditation and project tasks by state, agency, or affiliations through whom EQL holds some form of certification or contract. | Lab Director, Program Director, |
| Lab Certification Evaluations | Minimum of three years | Review of entire scope of accreditation and project tasks by SCDHEC’s Office of Laboratory Certification | Laboratory Director, Program Director, SCDHEC, EPA Region 4 |

### Assessments

Assessments are tools used to examine laboratory systems as they normally operate and to determine if quality assurance needs of the project are being met by current policies. The laboratory is evaluated through surveillance (e.g., an analyst’s initial demonstration of capability (IDC) exercise), data generator review, peer review, analysis of internal and/or external performance evaluation (PE) samples, and both internal and external audits. Surveillance results may be evaluated as part of an audit. Lab Certification Evaluations occur a minimum of every three years which Review of entire scope of accreditation and project tasks by SCDHEC’s Office of Laboratory Certification.

### Demonstration of Capability

An analyst training on a given method must perform an initial demonstration of capability (IDC) exercise prior to reporting client data independently (i.e., without the supervision of a qualified analyst). The analyst must prepare four aliquots of a known level of the analyte of interest, analyze them according to the appropriate method, and demonstrate the ability to recover the analyte within established acceptance criteria. Acceptance criteria for IDCs, depend on analytical technique and are listed on the IDC form, Form 1020B. Calculation of IDC results is done through a standard spreadsheet and may be performed by either the analyst or the QAO. Results are filed in the employee’s technical training file and the IDC file in the QA records.

### Data Generator Review and Verification

Data generators (i.e., the analyst or personnel conducting analyses) are responsible for conducting real-time review and verification of 100% of the data resulting from their activities. This review must be documented by the data generator's signature and review date on the raw data and on a worksheet or data review checklist. Data generators are accountable for ensuring that all data they generate are complete, accurate, and compliant with applicable requirements (QAM, SOP, method, or client-specified criteria). Data generators are responsible for performing all data reduction required prior to independent technical review, reporting and for notifying the Laboratory Director and/or QAO of any problems encountered during analysis and data review that may potentially impact data quality. The Laboratory Director and/or the QAO then determine and assign necessary corrective actions (see “Corrective Actions” element of this QAPP).

### Peer Review

All laboratory data must also receive peer review (i.e., independent technical review and verification). The independent technical reviewer(s) must be a qualified individual other than the data generator (e.g., peer analyst or Laboratory Director). He/she must meet the minimum training and qualifications requirements for analysts. Individuals not qualified to perform data interpretation cannot perform independent technical review. The independent reviewer(s) must ensure that:

* Data generation and reduction were conducted in a technically correct manner in accordance with the methods used.
* Data are reported in the proper units and with the correct number of significant figures.
* Calculations were performed with a valid calculation program and are correct. Calculations are checked by a spot check of verified calculation programs or 100% check of all hand calculations.
* All variances from an accepted method and the rationale for the variations were documented and approved.
* Data were reviewed for transcription errors.
* Analytical data documentation file or data package is complete, including sample preparation/extraction records, analysis sequence list, raw data, calculations or calculation records, calibration data or records, QC measurement results, test results summary, and completed worksheet or data validation checklist.
* QC measurement results are within established program specification limits, or if not, the data are appropriately qualified.
* Analytical sample holding times were met, or exceptions are documented.

Independent technical review is required before any data are approved for release and submitted to the data reporting process. The independent technical review process is documented with a signed and dated worksheet or data review checklist. The worksheet or checklist is archived in the associated data package. The peer reviewer must notify the data generator and the Laboratory Director and/or QAO of any problems identified during peer review that may potentially impact data quality. The Laboratory Director and/or the QAO then evaluate and assign, if necessary, corrective actions (see “Corrective Actions” element of this QAPP).

### Performance Evaluations

Performance evaluation (PE) studies, also referred to as proficiency test (PT) sample analyses, involve the analysis of a blind sample (i.e., a sample whose true analyte concentrations and/or analyte identities are not known by the laboratory) for the analyte(s) of interest. The analysis results of the study are evaluated for accuracy by a third party. The majority of PEs are performed by the lab in order to maintain state or agency certifications. PE sample analysis may also be required by specific client contract requirements. PE samples may either be provided by the state, agency, or client independently, or ordered by the lab from approved vendors having established PE programs. In-house blind samples may be prepared or purchased and submitted to the lab by the QAO at any time.

For this project the EQL obtains and analyzes PE samples from an approved vendor at least annually as one of the requirements for maintaining its certification in the SC DHEC Laboratory Certification Program.

PEs are often received in the form of concentrates, which must be prepared according to the vendor’s instructions in order to obtain an aliquot that is ready for routine sample preparation and analysis. Preparation and analysis of PE samples are recorded in the PE preparation log. The reconstituted aliquot must be prepared and analyzed according to the applicable method in the same manner as routine samples. The PE sample results must be subjected to the same QC requirements as used for validating a routine sample result.

All PE raw data and results must be reviewed and approved (initialed and dated) by the Laboratory Director. Copies of raw data and final worksheets, showing the approval with results to be reported, are forwarded to a QAO for submittal to the evaluator. Scoring is performed by the provider, and the issued report is retained in the QAO files. These reports are available to all staff, auditing agents, and clients upon request. Any PE measurement result that is not within the acceptance range established for the measurement is reviewed by the Laboratory Director and/or the QAO, who then determine and assign necessary corrective actions (see “Corrective Actions” element of this QAPP).

### Internal Audits

Internal audits are conducted by the EQL Quality Assurance Officer (QAO). An audit may be performed by another designated staff member who is knowledgeable of the process. Activities of an internal audit include, but are not limited to the following:

* Review of the SOP against the referenced method(s)
* Review of the procedure with a staff member who routinely performs the process
* Review of data files for complete and proper documentation, calculations, and quality control frequency (examination may include all testing records showing standardization, spikes, duplicates, and QC samples from one or more analytical runs)
* Review of logbooks for accuracy and completeness
* Review of the process for compliance with laboratory QA policies including error corrections, corrective action, reagent labeling policies, etc.

EQL internal audits occur at minimum of one laboratory area per quarter. Areas are defined by method or technique for analytical audits and by section for operational activities audits. Auditing in this manner allows for a comprehensive, on-going review of several areas throughout the year. The scheduling of the quarterly audits is at the discretion of the QAO and Laboratory Director.

Any deficiency identified by an audit is reviewed by the Laboratory Director then assigned to appropriate individual(s) for corrective action. The Laboratory Director establishes a corrective action completion date and monitors the corrective action until completed.

### External Audits

External audits are initiated primarily by states, agencies, or affiliations through whom EQL holds some form of certification or contract. For this project, external audits of EQL will be conducted by SC DHEC’s Office of Laboratory Certification and possibly the City of Conway, Georgetown County, and Horry County Stormwater Managers. Audits of this nature cover the entire scope of the accreditation and project tasks, including sample handling, preparation, analysis, and reporting for all parameters. Clients may also employ a qualified third-party assessor on their behalf to perform an external audit. The level of detail of an external audit is at the discretion of the auditor as related to the lab’s responsibilities and activities described in the project QAPP.

Any deficiency identified by an audit is reviewed by the Laboratory Director then assigned to appropriate individual(s) for corrective action. The Laboratory Director establishes a corrective action completion date and monitors the corrective action until completed.

### Corrective Actions

Any condition that adversely affects compliance with established QC requirements must be identified and corrected as soon as practical. Action taken to correct or preclude the recurrence of that condition is called “corrective action”. Some examples of corrective actions include repairs to equipment, revision of an SOP to eliminate a repetitive problem, or obtaining an approved variance to a procedure.

If severe issues are found then both Dr. Trapp and Dr. Libes have the authority to stop work.

#### Nonconformances

Nonconformances are items or conditions of a process which do not meet established QAM, SOP, method, or project requirements. As described in EQL SOP 201, "Nonconformance Identification and Corrective Action", all nonconformances, and the corrective actions taken, must be documented on a Non-Conformance/Corrective Action Report (NCR). Completion of a NCR should include not only a description of the problem and corrective actions but also copies of any documentation to support the same. NCRs must be routed through the QAO and Laboratory Director for approvals and closure.

Should a nonconformance affect the reportability of a client’s data or the ability to analyze a sample, it is the responsibility of the staff member documenting the nonconformance to notify the Laboratory Director immediately. The Laboratory Director must in turn contact the client, describe the details of the problem, act on any further instructions received, and follow up with written notice to the client of the problem and its resolution. A copy of the NCR may be used for this purpose.

Client inquiries concerning quality assurance are handled in a similar manner. When a client has a concern regarding laboratory results or procedures, it is the responsibility of the Laboratory Director to initiate a NCR. The Laboratory Director will review testing records for the sample (if applicable) and any circumstances surrounding the complaint. This review may include examination of bench sheets, compiled results, or applicable log books to check for errors. A copy of the NCR, detailing all findings and corrective actions, will be kept with the file copy of the formal result report for the sample in question. Review and approval of the NCR by the Laboratory Director and a QAO is required. Again, a written follow-up to the client is required. All NCRs are logged, and originals are retained in QAO files.

#### Variances

A variance is a type of corrective action involving an approved change to a process or procedure. A variance describes a deviation from a method, which affects the operation of the method, but not the method’s ability to achieve the performance standards or quality assurance objectives required. Variances must be requested in writing and receive approvals from the Laboratory Director and QAO.

#### Emergency Alternatives Policy

Under extreme or unavoidable circumstances (such as equipment failure, or irreconcilable matrix difficulties) samples may not be able to be analyzed by methods specified by the client or program. Alternative procedures may be acceptable. However, use of these procedures must be approved by the client. Laboratory staff identifying the problem must notify the Laboratory Director. The Laboratory Director is responsible for communicating the situation to the client. This communication must take place prior to reporting the results of the test by the alternate method and must be documented. The Laboratory Director may also inform the client if an option exists to sub-contract the samples to an appropriately certified laboratory. Sub-contracting options are also subject to client approval. Only labs that are certified for the parameters needed will be used.

### Quality Improvement

The EQL Laboratory Director, Laboratory Master Technician, and Laboratory Technicians meet periodically (as needed) as a quality improvement team to continually assess project work processes and laboratory operations, identify needed improvements, assign responsibilities for making improvements, and monitor progress on improvement actions.

The EQL quality improvement processes are summarized as follows:

* Nonconformance reporting
* Corrective actions
* Internal audits
* Management assessments
* Trend analysis
* Control charting

#### Trend Analysis

As described in EQL SOP 201, "Nonconformance Identification and Corrective Action", the laboratory uses trend analysis to monitor its analytical systems and associated activities. The goals of the trend analysis are as follows:

* To detect quality problems before they become significantly adverse to the quality of the products.
* To allow timely initiation of corrective actions to prevent development of significant quality problems.
* To ensure continuous quality improvement.

Control charting and frequency histograms are the main techniques EQL uses to conduct trend analysis; the parameters charted are nonconformance characteristics and QC measurement results.

#### Control Charts

Control charts are used by the EQL laboratory to monitor trends in analytical performance. As illustrated in **Figure 3** a control chart consists of a graph with the vertical axis labeled in units of the analysis or parameter of interest and the horizontal axis labeled in units of time or sequence of results. The upper and lower warning and control limits, which are statistically determined or specified by the method, may be used as criteria for instituting corrective actions. When the parameter being plotted is the relative percent difference (RPD) the lower limits do not apply (i.e., the minimum value of the RPD plotted is always zero and the limits plotted are upper limits).



Figure 2. Example of quality control charting

A basic principle in a QA program is the establishment of control limits. Such limits are utilized as decision criteria during analytical processes to reduce errors to acceptable levels and statistically characterize the results. Control limits are finite values which are comparable to the measurement values and can be used to statistically assess the acceptability of analytical measurements. There are two goals in establishing control limits. They should yield a narrow enough acceptance range so measurements that lie outside the upper or lower control limit indicate problems within the analytical system (i.e., the system is out of control). The limits, however, should not provide a range so narrow as to cause unnecessary adjustments of the analytical system and rejection of acceptably accurate and reliable results.

EQL's general policy is to utilize control limits where specified by the analytical method or where a sufficient data base exists (i.e., at least 20 data points) to establish control limits of ±3 from the mean value of replicate measurements, where "s" is the estimated standard deviation for replicate measurements for the system of concern. Measurements exceeding the control limits (either blank or control sample recovery measurements or precision measurements) usually require halting the analytical process, institution of corrective action measures necessary to obtain acceptable measurements, and documenting the corrective measures taken. This occurrence also normally requires rejection of any results generated between the last acceptable measurement and the unacceptable measurement or reporting those results with the notation that the analytical system was out of control. Warning limits of ±2 are utilized. Measurements inside the control limits but exceeding the warning limits require close examination of the measurement system by the analyst. Measurements in this category do not normally require halting the analytical process and rejection of data unless a significant problem is discovered.

## Reports to Management

### Annual Report

By mid-June of each year, the Laboratory Director prepares an annual activity report summarizing the following:

* Goals
* Financial summary and projections
* Measures and comparisons
* Major activities and accomplishments for year
* Needs

An important objective of the report is to address any unresolved quality issues pertinent to each area of lab operation, including any deficiencies identified by internal or external audits concerning equipment, systems, training, and/or staffing levels required to maintain or improve product quality. The report is submitted to EQL Program Director/Watershed Academy Director, B&C CMWS Director, Marine Science Department Chairman, and College of Natural and Applied Sciences Dean.

### Monthly Meeting/Report

Laboratory staff meets early each month to discuss and plan that month’s activities. The meeting agenda, which also serves as the meeting report for the previous month, lists accomplishments since the last meeting, the activities planned for the current month, review quality control issues, and important activities planned or expected in the near future. The meeting is always attended by the Laboratory Director and all Laboratory Technicians and is frequently attended by the Laboratory Program Director. The main purpose of the meeting is to finalize the assignments and scheduling of tasks for the upcoming month. Any issues that could adversely impact completeness, quality, or schedules of projects are thoroughly discussed and actions taken to ensure problems are avoided or reduced as much as possible.

### Bi-weekly Leadership Meeting

The Program and Technical Director meet every other week to discuss laboratory operations. This includes discussions of data QA/QC and data validation concerns and non-conformances. Additionally, overall programmatic goals, data reporting, and communications with funding and data reporting partners are discussed.

# Data Validation and Usability

## Data Review, Validation, and Verification Requirements

Table 24. Criteria for accepting, rejecting, or flagging data

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Item** | **Criteria** | **If not met sample is accepted, flagged or rejected?** | **Flag** | **Comments** |
| Sample not analyzed within hold time | Sample received in the lab within 6 hours of collection and analyzed within 2 hours of receipt appropriate hold time | Rejected | HT |  |
| Lost sample | Proper COC documentation not followed and sample is misplaced | (Unable to analyze) | LS |  |
| Unable to Collect Sample | Various circumstances (i.e., weather, lost sampling container) cause sample to not be collected | (Unable to analyze) | NS |  |
| Sample not held within required temperature range | Temperature blank within cooler indicates temperature above 6° C or proper storage equipment failed to read within range (refrigerator/freezer) | Rejected | T |  |
| Temperature blank not placed within cooler during sample transport | Unknown receipt temperature | Flagged | UT |  |
| Incorrect sampling container used for sample collection | Incorrect sampling container used for sample collection | Flagged | SC |  |
| Improper preservation | Improper preservation (i.e., acidification, filtering) | Flagged | IP |  |

## Validation and Verification Methods

All processes at EQL (sample receiving and handling, sample analysis, data reduction, data reporting, data review, etc.) are subject to examination to evaluate adherence to project specifications. This examination consists of several layers of technical and QA review. These reviews ensure that all data released by EQL were scrutinized by qualified independent reviewers and are scientifically sound, appropriate to the method, completely documented, and legally defensible.

All data receive analyst review and independent analyst (i.e., qualified peer) review. The Laboratory Director and QAO also review the data to varying degrees at different points in the review process. These review processes are appropriately documented before data are released from the laboratory.

*Data review* ensures that raw data are properly collected, reduced, and reported. *Data verification* confirms by examination of the measurement process and provision of evidence, that specified method, procedural, or contractual requirements have been met. For example, QC measurements must indicate that deviations between measured values and known values are smaller than the maximum allowable error (i.e., DQIs). *Data validation* is the process of substantiating that specified performance criteria were achieved for an entire data set or data reporting group, including comparisons between analytes and samples to see if relationships are scientifically reasonable.

At EQL, a worksheet or data review checklist (DRC) for each analytical process outlines the performance criteria for the process. The worksheet or checklist is completed and signed for each analysis batch by both the analyst and a qualified peer to document the process as described earlier in the “Data Generator Review and Verification” and “Peer Review” subsections of the “Assessments and Response Actions” element of this QAPP.

The EQL review process must examine as a minimum the following data recording requirements for analyses:

* All original data must be recorded, signed, and dated in black waterproof ink.
* All data must be recorded clearly and accurately in laboratory records, bench sheets, or logbooks, and include applicable sample identification numbers.
* All changes and additions to original data must be made with a single-line drawn through the error with the correction entered above or next to the line-out. **White-out, correction tape, or similar correction techniques must not be used for changing laboratory data.** The change must be initialed and dated by the individual making the change (an explanation of the change or addition must be included if the change or addition deals with rejecting data).
* All data used from logbooks and laboratory records must be transferred and reduced completely and accurately.
* All laboratory records shall be maintained in permanent files.
* Data shall be organized into standard formats.
* All electronic data shall be stored appropriately to ensure that sample and QC data are protected and readily retrievable. Corrections made to hardcopy data must also be made in electronic data files whenever possible.

The data review is documented by the Laboratory Director’s signature and date on the final reports and is done before the reports are released to the client.

### Project Management / Data Validation

The final step in the data validation and usability determination in the EQL analysis and reporting process is data validation or the project management review by the Laboratory Program Director, Dr, Susan Libes.

One hundred percent (100%) of the data reports must receive a relational technical review before being released to the client. The project management relational review occurs after the data have been entered and all analytical peer review has taken place.

This review must ensure that:

* Data are technically reasonable based on the technique used.
* Reported analytical data documentation or data package meets the clients’ data quality objectives (DQOs).
* Relationships between related parameters are scientifically reasonable. Anomalies in the data will be investigated.
* Notation of contravention of water quality standards.
* Site specific statistical rankings of data based on the entire project data set
* Entry of data in to the project data base

## Reconciliation with User Requirements

Reconciliation of data with DQI criteria to determine data usability is performed primarily by the Laboratory Program Director working in direct communication with the clients.

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# Revisions

# Appendix A: Forms

# Appendix B: Laboratory Certification Documentation

# Appendix C

# Appendix D: Environmental Quality Lab Standard Operating Procedures