

City of Santa Cruz Environmental Laboratory WWTF



WASTEWATER LABORATORY QUALITY ASSURANCE PERFORMANCE PLAN (QAPP)

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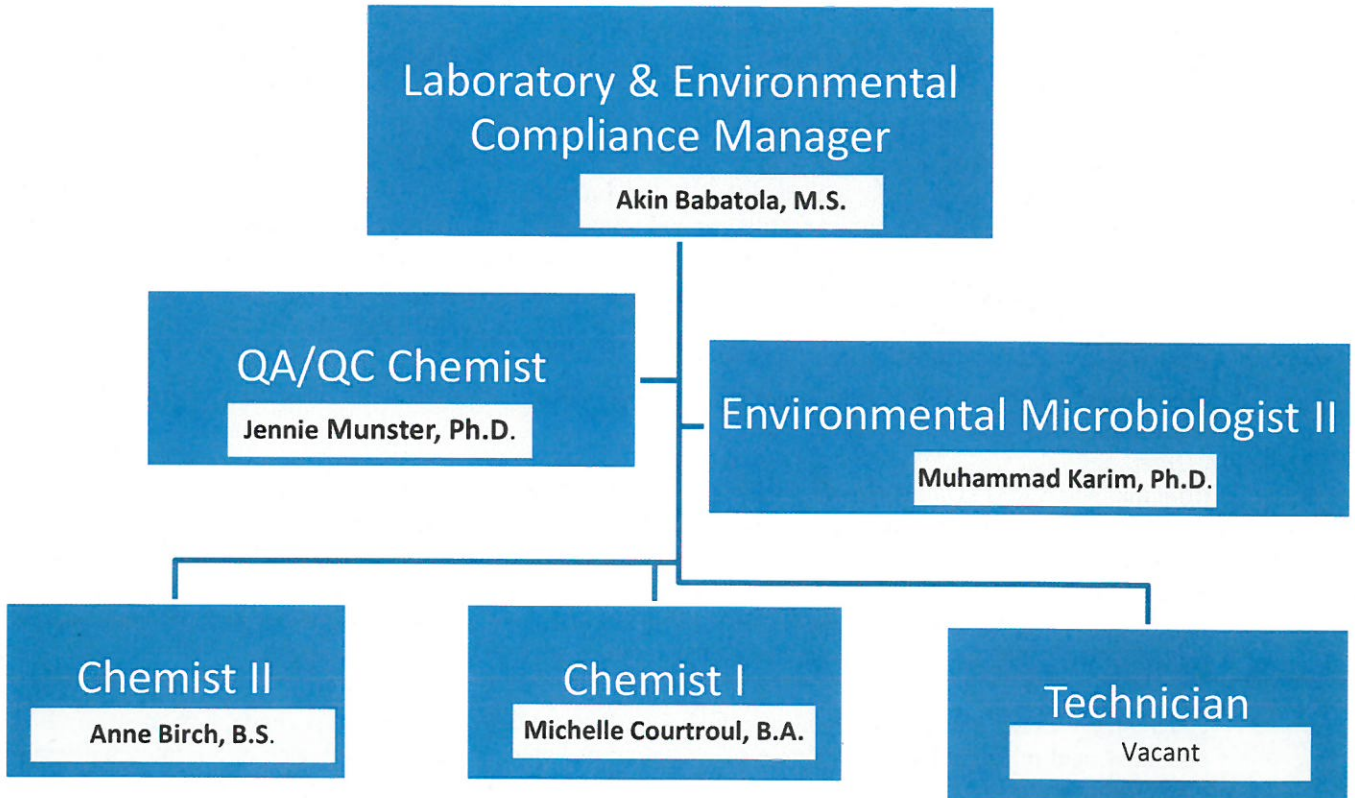
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Introduction

The objective of the laboratory Quality Assurance Program Plan (QAPP) is to assure the accuracy and precision, as well as the reliability of all laboratory results produced. This document serves as an operations charter defining objectives, principles, organization, and general procedures on how the laboratory produces data of known, documented, and accepted quality. While the goal of all quality control procedures is to generate the highest quality, defensible data, the application of those procedures varies from method to method, with sample matrices, and between chemical and microbiological analyses. All laboratory personnel are required to possess a thorough understanding of the goals of the Quality Assurance Performance Plan, as well as how the program is implemented for laboratory procedure.

Organization and Responsibility



The following is a brief description of the responsibilities of key personnel.

Laboratory Manager- Responsible for planning, organizing and directing the operations of a public environmental and municipal chemical, microbiological laboratory at the Wastewater Treatment Facility Laboratory through professional staff of chemists, microbiologists and technicians. Administrative guidance

is received from the Superintendent of the Wastewater Treatment Facility. The Laboratory Manager is the final person responsible for the quality of data and laboratory reports and invoices. He oversees all QA programs, signs and certifies performance evaluation and self-monitoring report results, and, if necessary, resolves any issues concerning data quality. This position gives the final approval on any changes made to the QAPP, and is the site manager for the laboratory's information management system (LIMS). The Laboratory/Environmental Compliance Manager directs the QA/QC Chemist, who is the coordinator and the project officer for the QAPP. In addition, he may designate duties including preparing QA Reports and Systems and Performance audits, reporting performance evaluation results to regulatory agencies, and overseeing laboratory accreditation documentation and site visits.

QA/QC Chemist- The QA/QC Chemist is the project officer for the QAPP. The incumbent works in collaboration with the laboratory manager to update and maintain the currency of the QAPP. In addition the incumbent supervises the work of chemists and technicians in the laboratory. The incumbent is responsible for ensuring the QAPP is followed at the bench level by reviewing and validating all data that go through LIMS, maintaining quality control charts and accurate SOPs and resolving any issues concerning data quality. This position also performs advanced analytical techniques and acts as back up for the other laboratory staff. Data produced by this position is checked for accuracy and precision and validated by the Laboratory Manager.

The QA/QC Chemist is a Principal Analyst position as defined in California Code of Regulations Title 22 at CCR Chapter 19.

Environmental Microbiologist II - Under direction of the Laboratory Manager, the incumbent plans, reviews and direct microbiological analyses at the Laboratory. Organizes molecular and culture based studies of samples for compliance; regulatory and treatment plant support. The incumbent also directs the sampling and analyses of the effluent toxicity monitoring program for NPDES analyses. The microbiologist works intimately with the QA/QC Chemist to ensure the currency and integrity of the microbiological elements and section of the QAPP as well as all the proficiency monitoring actions in microbiology required for laboratory certification.

The Environmental Microbiologist II is a Principal Analyst position as defined in California Code of Regulations Title 22 at CCR Chapter 19.

Principal Analyst(s) - Under direction, plans, reviews and directs routine sampling and analytical activities of other staff of the laboratory. Operates specific modules of the LIMS and performs professional and technical work; leading professional staff comprised of other chemists and technicians in the qualitative and quantitative microbiological, chemical and physical analyses of water, wastewater, biosolids, and industrial waste and related materials. The Principal Analyst also performs analyses on sophisticated analytical instruments and is tasked with development of sophisticated analytical methods for use in the laboratory.

Chemist(s) - Under direct and general supervision, performs standardized qualitative and quantitative physical, chemical, and microbiological analyses of water, wastewater, and industrial waste and related materials. Chemists operate specific modules of the LIMS and perform professional and technical work; they may lead other professional staff including newer chemists and technicians in the qualitative and quantitative microbiological, chemical and physical analyses of water, wastewater, biosolids, and industrial waste and related materials. Chemists may also be assigned other work-related functions as necessary. Laboratory Chemists are responsible for carrying out all quality control procedures in all their assignments as described in the QAPP. As the group primarily responsible for performing routine analyses, their understanding of the Quality Assurance Performance Plan and participation in charting QC indices for analyses they perform is essential to the success of the laboratory.

Lab Technician- Under direct and general supervision, performs routine and standardized qualitative and quantitative physical, chemical, and microbiological analyses of water, wastewater, and industrial waste and related materials. Technicians operate specific meters and bench instruments in support of the laboratory's mission. They also operate specified modules of the LIMS and perform technical work; they may lead other technicians in the qualitative and quantitative microbiological, chemical and physical analyses of water, wastewater, and biosolids. Technicians are responsible for maintaining and standardizing stock reagents and chemicals for their analytical assignments. Technicians may also be assigned other work-related functions as necessary. The Lab Technician is the first cadre responsible for carrying out specific quality control procedures at the bench level, as described in the QAPP. This applies solely to analyses they perform.

Sampling Procedures

The objective of sampling is to collect a portion of material that accurately represents the material being sampled while being small enough in volume to be readily transported and conveniently handled in the laboratory. Consequently, specific procedures are followed to help ensure that the analyte originally present in the sample matrix has not undergone significant volatilization, biological or chemical degradation, potentiation or concentration, and contaminants which might interfere with the analysis have not been added during the sampling process.

Environmental Laboratory and Environmental Compliance professionals routinely collect samples. The Environmental Laboratory utilizes three standard sampling methodologies, two of which represent snapshot methodologies and are conventionally described as Grab and Composite sampling. The third and newest standard methodology employs integrative sampling techniques using equilibrium-based characteristics of trace organic compounds in the water for effective and representative sampling.

Sample containers are purchased in lots from various sources and are equivalent to those specified in 40 CFR Part 136. Cleanliness checks, container integrity and validation checks are run by the manufacturer and/or the vendor. All certificates of analyses for the checks are kept within the laboratory and are integral to the final report. The documentation is reviewed before any new sampling equipment is used, and in the alternative, equipment blanks are analyzed in the laboratory. Sampling containers are often dedicated to a specific purpose, and upon re-use they are scrupulously cleaned according to EPA guidelines to prevent carry over from previous samples. Sample containers cleaned in the laboratory are periodically checked for contamination using blank determinations. Sampling for trace organic compounds and other ultra-trace environmental analytes are done through validated devices manufactured under licenses granted by USGS under its authority as specified in 40 CFR Part 136.

Sampling equipment is inspected before each use to ensure proper function and field instruments are calibrated regularly. Inspections and calibrations are kept in a logbook to provide an accurate record of performance. The preservation techniques used in the laboratory strictly adhere to those listed in 40 CFR Part 136.

Sample Custody

Because of the potential evidentiary nature of samples and analytical data, sample chain-of-custody must be controlled and documented in the laboratory. Sample custody and document control procedures function to identify and document tracking and handling of samples and documents.

Non-routine samples are delivered to the lab with a "Chain-of-Custody" form. Laboratory personnel sign and date the form during receipt of the sample(s) and sampling personnel sign and date the form when sample(s) are relinquished. The COC documents the sample identification, including a unique sample identity number (generated by LIMS); dates and times collected and received, sample type (i.e. grab; integrative or composite), identity of the sampler, the analyses requested, as well as the use of proper containers and preservatives. Laboratory personnel note the temperature of samples received for microbiological analysis. All samples received with a COC are properly labeled when received and after being logged into LIMS the chemist or technician records the LIMS identification number on the label and the COC. The sample information is recorded in a laboratory sample record book, in addition to being logged into LIMS, and the permanent record of the sample history in the lab is initiated. Any deviations from required sampling techniques (e.g. wrong container type, headspace in VOAs, not enough sample, leaky containers, and unusual sample temperature) are noted under "comments" in LIMS and the "Chain-of-Custody" form.

Operations personnel deliver routine composited process samples daily to a secured, refrigerated storage area located in the laboratory. Grab samples from the various process areas (trickling filters, UV, headworks, sludge, mixed liquor, etc.) are delivered directly to the Laboratory Chemists or collected by the Chemists, Microbiologists and Technicians themselves. These samples are tracked and permanently recorded using the sample record book and LIMS. Sample refrigerators are monitored for temperature, and the record is tracked and maintained in the laboratory.

Sample type, location, date, and special circumstances are used to identify and differentiate these samples. All samples collected at the plant for shipment to contract laboratories generate a COC initiated by the laboratory, and transferred along with the sample to the couriers for the contract laboratory.

Samples from the High Volume Integrative Sampling systems including SPMD are associated with a specifically designed Chain-of-Custody with XML features that allow the multiple contract labs to complete the same forms.

Sample storage, temperatures and holding times adhere to criteria as presented in 40 CFR Parts 136 and 503.

Documentation

All laboratory data are subject to rigorous standards to ensure validity and reliability. The following rules are maintained to ensure the integrity of the data and laboratory records that are manually generated:

- (1) Data produced by laboratory staff and by contract laboratories are entered in the LIMS.
- (2) Data entry is performed by the Chemist as soon as possible after testing. All paper entries shall be made in indelible ink in the laboratory workbooks and/or approved forms as are applicable.
- (3) All corrections shall be done by neat cross-over of the wrong entry; the initials of the personnel making the entry; date and the explanation of the cause(s) of the error.
- (4) The most recent analytical data sheets are stored in laboratory method binders or clipboards and archived thereafter for at least 5 years.
- (5) The use of correction tape, labels or correction fluid in the completion of laboratory documentation is expressly forbidden.
- (6) Laboratory Manager, the QA/QC Chemist or designate shall review all entries for

conformance with these and other approved criteria and indicate so by initialing.

Analytical Procedures

The primary sources of laboratory analytical methods are Standard Methods for the Examination of Water and Wastewater, published by the American Public Health Association/American Water Works Association/Wastewater Treatment Facility Federation (APHA/AWWA/WPCF); Methods published and validated by the US Environmental Protection Agency (EPA); The American Society for Testing and Materials (ASTM) and the United States Geological Survey (USGS). These agencies develop, validate, and publish laboratory and field protocols, most of which have regulatory and legal acceptance. The protocols used in this laboratory are specified in the laboratory's Standard Operating Procedures (SOPs), and may also be found in method binders for each particular analysis, and on the Laboratory Drive of the Facility's electronic network. Method binders also contain logs for sample analysis, MDL information, PT results, corrective actions, quality control data and control charts and standard/reagent preparation logs. Appropriate procedures for sample extraction, preparation, and clean-up are referenced in the individual methods and are followed accordingly. Any exceptions to these standard methods are clearly documented in the SOPs.

"Good Laboratory Practices" (GLP) are required for all analytical procedures performed in the laboratory. This includes proper cleaning of sample containers and glassware, use of acceptable grades of reagents and laboratory pure water, maintenance and archiving of records, and proper waste disposal.

- Purchased reagents are labeled with the date received and the date the container is opened.
- Daily checks include, but are not limited to: incubator, laminar flow hoods, oven, refrigerator, and water bath temperatures, as well as pH, and conductivity meters.
- Buffers for pH calibration and conductivity standards are replaced daily.
- Dissolved oxygen probes are checked by a Winkler titration each analytical batch.
- Balances are checked against Class S weights monthly and calibrated annually, and thermometers are calibrated to an NIST certified thermometer annually.
- Water suitability tests are performed annually.

Logs are maintained for all of these standardizations. In addition, there is a continual review process of all aspects of laboratory operation.

Calibration Procedures and Frequency

The WWTF Laboratory utilizes the calibration procedure as a referential portion of the analytical process that is identified specifically with the quantitation of the analyte and the performance of the measurement system. Instruments used by the laboratory are operated, calibrated and maintained in accordance with the manufacturer's guidelines and recommendations and approved methods.

In general, the initial calibration consists of developing a calibration curve using reference standards for each parameter analyzed. A blank and, preferably, 4 to 5 serial dilutions of a stock standard are analyzed, which upon plotting will yield values with a high degree of correlation, and generate a calibration curve. Sample values that fall outside the linear range of the standards must be diluted and reanalyzed until they are within the linear range established by the standard curve. Calibration often includes a low level standard to verify detection limit sensitivity. The initial calibration may be verified by comparing instrument parameters to manufacturer criteria or previous calibrations to help ensure optimal performance of the instrument. Once

established, calibration ranges and method detection limits (MDLs) are re-evaluated annually, or as required by the method or changes in the analytical protocol and personnel.

Initial calibration is performed on a frequency required by the analytical method. Ion selective probes, pH meters and the turbidimeter are calibrated daily. The ion chromatography instrument, following EPA 300.0, requires a calibration curve be performed every 6 months or when a change in the instrument or analyst occurs. The total organic carbon instrument following SM 5310 B is calibrated yearly or when a change in the instrument or analyst occurs.

Continuing calibration is performed during the analytical process to verify that the initial calibration is still applicable. Generally, continuing calibration is performed using check standard(s) and blank, though a replication of the initial calibration may be required instead. Check standards are run after every 10 samples, or as required by the method. The concentration is selected to be in the midrange of the calibration curve. Calibration range criteria are applied to determine if the instrument is performing optimally and measuring acceptably. The criteria are often expressed as a range of percent recovery of the initial calibration value, typically no worse than 90 to 110 percent or as required by the standard method, for example, EPA 300.0 requires the check standard to be within $\pm 10\%$ of calibration. When continuing calibration satisfies criteria, the normal measurement process continues. When criteria are not met, the problem is investigated, corrected and verified before recalibrating and reanalyzing the samples analyzed since the last in-control, initial or continuing calibration standard. The results of all calibration procedures, initial and continuing, are recorded in method binders and/or instruments electronic logbooks.

Standards for instrument calibration are obtained from a variety of sources traceable to the National Institute of Standards and Technology (NIST) or certified by the EPA. Certifications are kept in the method binders. Elemental standards are purchased from commercial suppliers, dated on receipt and replaced according to specified criteria. Standards logs are maintained, containing date of preparation, supplier, lot number, concentration(s) and volume(s) prepared.

Quality Assurance and Quality Control for Chemical Measurement Data

The term Quality Assurance (QA) refers to the overall program for assuring the reliability of analytical data generated by a laboratory. Quality Control (QC) refers to the specific routine procedures performed as part of the field activities and laboratory analyses to help ensure that the quality of the measurements meet criteria considered appropriate for the intended use of the data. The lab adheres to the quality assurance and quality control procedures described for each particular method, and as outlined in Standard Methods Table 4020:1 and 5020:1. Each specific analytical method employed within the laboratory may specify more stringent limits than the broad range acceptable for a group of methods. Method references include, but are not limited to:

1. 40 CFR Part 136, the EPA's Methods for Chemical Analysis of Water and Wastes (EPA 600/4-79-020).
2. Standard Methods for the Examination of Water and Wastewater.
3. EPA's Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms (EPA 600/4-85/013).
4. Short-Term Methods for Estimating the Chronic Toxicity of Effluent and Receiving Water to Freshwater Organisms (EPA 600/4-91/002).

5. EPA's Microbiological Methods for Monitoring the Environment, Water and Wastes (EPA 600/8-78/017).
6. 40 CFR Part 503 and
7. Annual Book of ASTM Standards (Section 11).

Accuracy and Precision.

This QAPP emphasizes the values of accuracy and precision in evaluating the acceptance of laboratory data and studies. Accuracy is a measure of the closeness or degree of agreement of a measured value (or mean of set of values) to the true value. Analyzing certified reference materials purchased from a quality vendor, matrix spikes and blank spikes per batch meet the needs of monitoring accuracy in the laboratory analyses at the WWTF. Recoveries are generally 90-110% for inorganic measurements and 80-120% for organic measurements, unless specified by the method. Precision is a measure of the agreement between a set of replicate measurements without assumption and knowledge of the true value. Precision measurements are performed at the same frequency as accuracy measurements by repeating the matrix spike and/or analyzing the sample in replicate. The relative percent difference (RPD) is the index of precision used by the laboratory and is critical in validating most samples and their associated analytical processes; data is accepted when the RPD is $\pm 10\%$ for inorganics and $\pm 25\%$ for organic analysis. All accuracy and precision results are documented and charted in the method binders.

When accuracy or precision measurements are out of compliance it must be brought to the QA/QC Chemist or Laboratory Manager's attention. When evaluating all quality control parameters a decision can be made to (1) accept the batch with qualifiers, (2) rerun the batch (3) determine if a corrective action is needed or (4) another acceptable solution.

	Inorganic	Organic
<u>Accuracy:</u>		
Certified Reference Material	90<%R<110	80<%R<120
Matrix Spike	90<%R<110	80<%R<120
Blank Spike (Lab fortified Spike)	90<%R<110	80<%R<120
<u>Precision:</u>		
Matrix Spike Duplicate	$\pm 10\%$ RPD	$\pm 25\%$ RPD
Sample Duplicate	$\pm 10\%$ RPD	$\pm 25\%$ RPD

Examples of accuracy and precision parameters for routine analytical methods that fall outside of the above described limits:

- (1) High reproducibility of low level analysis, near the reporting limits, is difficult to obtain; for example of ammonia (SM4500-NH3-D/E) and fluoride (EPA 300.0). RPDs and recoveries of $\pm 20\%$ can be acceptable for low level analysis when other quality parameters are acceptable.
- (2) EPA 1664B has method defined recoveries for oil and grease. The precision and recovery sample (PAR) and matrix spike recoveries should be between 78-114%. The RPD of the matrix spike duplicate should be no greater than 18%.
- (3) Control charting is utilized by the QA/QC chemist to evaluate trends in accuracy and precision that might warrant different acceptable criteria.

Analytical blanks

A method blank or control is also run at the same frequency as accuracy and precision measurements. Method

blanks monitor the purity of reagents and verify that the instrument system and/or laboratory environment are clean. Controls are run for determinations where reagents are not added, such as total suspended solids. An acceptable method blank for Total Suspended Solids will yield +/- 0.0002 g (i.e. -0.0002 g < net weight of Blank < 0.0002 g). Controls are generally distilled and deionized water of reagent quality grade, and are not subtracted from the sample results.

To verify that no contamination occurs during sample transport or storage, field blanks are collected on a routine basis. Field blank results are recorded with the accompanying sample results. At least yearly bottle blanks are tested for contamination. All blank, control and field blank results are recorded in LIMS.

Method Detection Limit/Detection Limits

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. Measure a quantity estimated at 5 times the estimated true detection level in seven replicates ideally over a three day period. Recovery should be between 50-150% with the RSD \leq 20% and preferably \leq 10%. The MDL is calculated as 3.14 multiplied by the standard deviation of these measurements. For additional information and procedure for determination of MDL, refer to 40 CFR Part 136, Appendix B and Standard Methods 1020B.4. If the calculated MDL is not within a factor of 10 of the known addition then repeat determination at a different concentration. MDLs are performed annually, except for EPA 300.0 which is performed semi-annually, or when a change in the method, instrument or analyst occurs for all methods which require MDLs.

Detection limits are defined by the MDL, the lowest linear calibration standard or as defined by the method.

Proficiency testing

The laboratory participates yearly in proficiency testing, where samples of unknown quantity are purchased from a quality vendor and analyzed per usual methods.

Assessment of Precision and Accuracy

Precision is assessed by comparing results of replicate analyses of samples or matrix spike samples. The relative percent difference (RPD) is the difference between duplicates expressed as a percentage of the mean of the duplicates. RPD is calculated by:

$$\text{RPD \%} = [(A - B) / ((A+B)/2)] \times 100$$

Where: A = sample result (or spike)
B = duplicate sample result (or spike duplicate)

Precision is evaluated from replicate analyses with the statistically derived standard deviation. The standard deviation (s) is calculated by:

$$s = \sqrt{[(X_i - \bar{X})^2 / n - 1]}$$

Where: X_i = sample result

\bar{X} = the arithmetic mean

n = number of replicates

Accuracy is assessed by comparing concentration values with the "true concentration values" in mathematical terms of the recovery. The comparison is quantified by the calculation of the percent recovery (%R) of the true value. A known amount of compound (spike) is added to a sample of known concentration and the amount recovered by analysis is measured. For a method or matrix spike, the compound is added prior to sample preparation (e.g., digestion or extraction, cleanup) and analysis. An analytical or post-preparation spike is added just prior to analysis (e.g., post-digestion spike for metals analysis). %R is calculated by:

$$\%R = [(SSR-SR)/SA] \times 100$$

Where: SSR = spiked sample result

SR = sample result (unspiked)

SA = spike added to sample

Accuracy is also evaluated by the recovery of the known value of certified reference samples. The percent recovery for reference samples (%R) is calculated by:

$$\%R = [EV/CV] \times 100$$

Where: EV = experimental value

CV = certified value

Precision and accuracy data are plotted in control charts, which graphically demonstrate statistical control, monitor performance, diagnose problems, and aid in method development. The laboratory uses a type of Shewart control chart, the widely used x-bar. These control charts show time plotted QC data distributed around the arithmetic mean along with some kind of control limits (boundaries for data quality). The control limits can be non-statistical criteria [e.g., +/- 10% RPD, 85-115% spike recovery, and the MDL (for blanks)], or be statistically derived warning and control limits. Typically, the warning limits are the mean plus and minus two times the standard deviation (which should include 95 percent of the data) and the control limits are the mean plus and minus three times the standard deviation (which should include nearly all of the data). Control charts are updated annually in the method binders and quality is tracked daily using the graphing function in LIMS.

Quality Assurance and Quality Control for Microbiological Measurement Data

Because microbiological analyses measure constantly changing living organisms, they are inherently variable, thus quality control tools available for microbiological analysis are different than those employed by chemical techniques. The QA/QC program in microbiological analyses is designed to control factors of influence within sample collection and all analytical processes through data reporting. These factors include (1) sampling techniques, (2) sample storage and holding, (3) facilities, (4) equipment and supplies, (5) culture media, (6)

quality of reagent water, (7) washing and sterilization, (8) analytical test procedures, (9) test conditions, including pH of culture media, temperature of incubation, oxygen requirements of different organisms (10) records and data reporting, (11) data validation.

Sampling Techniques/Storage and Holding

Samples are collected by WWTF staff in sterile whirlpak[®] bags or sterilized plastic bottles and kept cold until analyzed. Gloves are worn when collecting samples and care is taken not to contaminate the sampling collection area, often by sampling with a utility pole to avoid stepping into the water so as not to stir up the sand at the sampling point. Sample temperature is measured and recorded upon receipt in the laboratory on the COC and in the LIMS. Samples should be $\leq 10^{\circ}\text{C}$, and kept on ice during transport. Samples brought by those outside the WWTF are collected in sterile sample containers and noted on the COC if there is deviation from this.

Samples are analyzed as soon as possible after collection with sample incubation starting no later than 8 hours (Title 40 section 136.6 Table II) of collection. Plate counts are read within 22-24 hours for Total Coliforms, 24 ± 2 hours for Fecal Coliforms and 24 hours for Enterococcus. Date and times of sample collection, analysis initiation and time/date of plate count readings are recorded on the lab worksheets.

Facilities

Space for microbiological analysis at the WWTF laboratory is well ventilated, uncluttered and organized in a manner for chemists and technicians to produce high quality data. Staff maintains a high level of cleanliness by disinfecting surfaces before and after testing and sterilizing contaminated supplies and media promptly after use.

Equipment and Supplies

Equipment used for microbiological analysis are well maintained and regularly calibrated, all pertinent information is well documented in appropriate binders. Key quality control practices are outlined in Standard Methods Table 9020:I and Table 9020:II.

Analytical quality control procedures

Besides the good laboratory practices discussed above measures are taken to ensure reliable data quality per analytical batch by measuring positive and negative control cultures, sterility method blanks, and duplicates. Duplicate analysis is performed on 10% of the samples and on at least one sample per test run. High precision can be difficult to obtain if adequate sample volume isn't used, which commonly occurs when expecting high concentrations of colonies, or when colonies are not within the countable range.

Precision of duplicate analyses for each different type of sample examined is calculated by first obtaining logarithms of each result and then calculating the range (R) for each pair of transformed duplicates. If the range is greater than the precision criteria ($3.27R$, ref. Standard Methods Table 9020:VII and 9020B.9.e), chemist variability is excessive and corrective action should be initiated. For example, duplicate measurements of coliforms were 89 and 71 CFU/100mL, the logarithms of each is 1.9494 and 1.8513, yielding a range of 0.0981, a passing criteria.

Species of Microorganisms	Time Frame for 15 samples analyzed in duplicate	Precision Criteria
Fecal Coliforms	11/03/2015 through 02/27/2015	0.30
Total Coliforms	12/09/2014 through 03/01/2015	0.61
Enterococci	12/09/2014 through 02/27/2015/2013	0.45

Analyst colony counting variability is checked by the primary analyst and routinely with the secondary analysts. Replicate counts are within 5% for the same analyst and 10% between analysts, if not corrective action is initiated.

For each type of test conducted, the colonies are verified monthly from a known positive sample.

Colony Counting

Standard methods 9222.5 addresses the calculation of coliform density for plates that fall outside the countable range for coliforms and EPA 1600 Appendix B does the same for enterococci and outlined in the method SOPs. The WWTF laboratory abides by the following when counting colonies for total and fecal coliforms and enterococci

- 1) When multiple dilutions are performed and only one plate is within the countable range, report that value.
- 2) When multiple dilutions are performed and more than one dilution is within the countable range, average the values.
- 3) When all the dilutions are outside the countable range, but still countable, average all plates
- 4) When no colonies are counted on all plates for all dilutions, record the appropriate less than value based on the smallest volume used.
- 5) When all dilutions are too numerous to count (TNTC) report the data as TNTC. This includes plates that are confluent and plates that have colonies that overwhelm the plate but aren't considered confluent.

Assessments (internal audits, proficiency testing)

The WWTF Laboratory participates in the annual evaluation of proficiency of NPDES data reporting laboratories. The annual DMR-QA studies provide data for inter-laboratory ranking, method validation and personnel performance of regulatory methods.

Document control and recordkeeping requirements

Documentation and data validation is adhered to as described in this QAPP. Records of all quality control measures are maintained in appropriate binders for five years.

Corrective Action

Corrective action procedures are required as the result of audited or self-discovered nonconformance with predetermined QA/QC criteria. The corrective action system functions to identify, document, and prevent reoccurrence of out-of-control situations. The system applies to all situations which impact data quality. These situations include, but are not limited to: quality assurance acceptance limits being exceeded, deviations from normally expected results, divergence from SOPs, abnormalities in sample handling, etc.

The first level of responsibility for identifying nonconformance lies with laboratory staff, which is trained to recognize nonconformance and notify the QA/QC Chemist or Laboratory Manager. The second level of responsibility for identifying nonconformance lies with reviewing the data. The QA/QC Chemist is responsible for reviewing all analytical and QC data, monitoring control charts, determining the source of errors and correcting them. The Laboratory Manager is notified in all cases and will help develop and initiate corrective action and oversee the review of corrective action reports.

Each nonconformance is documented by recording the circumstances in a Corrective Action Report. Results of corrective actions are documented as well. Documentation of corrective action steps includes problem identification, investigation, action to eliminate the problem, and verification that the problem has been solved. Notification and investigation of nonconformance is the responsibility of staff; the QA/QC Chemist verifies that corrective actions have been completed and notifies the Laboratory Manager accordingly. These reports are stored in the method binders.

Examples of corrective action include resampling and/or reanalysis of samples, amending sampling and/or analytical procedures, or accepting the data and acknowledging the level of uncertainty with a written explanation.

Data Acquisition, Reduction, Validation and Reporting

Laboratory personnel are responsible for the recording and reduction of all raw data associated with the analyses. Equations and calculations for data reductions are performed in accordance with procedures detailed in the analytical protocol, and are transcribed into the analytical worksheets as appropriate. Some instruments, such as the balance and the ion chromatograph electronically transfer data into LIMS, leaving no hand written record. When possible, preference is given to LIMS for calculating results (such as ratios, RPD, etc.) over the analyst to reduce error. Computations and recorded results carry the common units of measurement from the methods. Report only such figures as are justified by the accuracy of the work.

Significant figures: Only significant figures should be used in recording analytical results. Significant figures by definition are those digits in a number that are known with certainty, plus the first uncertain digit. For reporting requirements, rounding off, ambiguous zeros and related issues in expression of results, refer to Section 1050B, Standard Methods, 21st Edition. Generally, data is recorded to the third significant figure unless otherwise noted in the method, such as 2 significant figures for pH, or more significant figures for methods of higher accuracy.

Calculations: As a practical operating rule, round off the result of a calculation in which several numbers are multiplied or divided to as few significant figures as are present in the factor with the fewest significant figures. When numbers are added or subtracted, the number that has the fewest decimal places (not necessarily the fewest significant figures) determines the number of places that justifiably may be carried in the sum or difference.

Zeros: In a number written as 5.000, it is understood that all zeros are significant, or else the number could have been rounded off to 5.00, 5.0, or 5, whichever was appropriate. The digit 0 is significant when it records a measured value of zero (e.g. 104 and 40.08), but is insignificant when it serves merely as a spacer to locate the decimal point, as in 0.003.

Less than detection values: When treating less than detection values for statistical purposes, such as computing an average, treat them as the value for inorganic measurements, such as <1 mg/L for ammonia would be treated as 1 mg/L. For averaging colonies of bacteria use the less than value minus one, so that <4 CFU/100mL would be treated as a 3 CFU/100mL.

All data are recorded in LIMS, and all paper documentation is in a clear and legible ink. For reasons of traceability, entries are dated and initialed on each line or page, as appropriate. Errors are lined out with a single line and corrections are initialed and dated.

Data review begins at the bench level and extends throughout the reporting process. Chemists review data for calculation and transcription errors before transference into LIMS. Unusually high results or those clearly in violation of discharge limits or hazardous waste standards, are reviewed carefully for any reporting unit errors and frequently trigger an examination of QA/QC binders and instrument printouts to check for calculation errors. The QA/QC Chemist and Laboratory Manager are responsible for the final review of data. Binders, worksheets, and copies of final reports are kept in a secure filing area for a minimum of five years.

Preventive Maintenance

Preventive and remedial maintenance procedures help maintain on-schedule operation of the laboratory through readiness of the equipment and supplies. Maintenance is performed in accordance with the direction and frequency detailed in instrument manufacturer's manuals and the laboratory's SOPs. Dated and signed instrument logbooks describe and document scheduled inspections, routine and non-routine maintenance, and major repairs.

Normal preventive maintenance is performed on a routine and as-needed basis. Typical procedures include cleaning, adjusting, and replacing easily serviced items. Specialized inspection, maintenance, and trained personnel from the Department or the manufacturer, as appropriate, perform repair. If needed, maintenance and repair may be provided through manufacturer's service contracts.

Remedial maintenance is performed for real and potential out-of-control situations. Quality control data are examined for trends or excursions toward or beyond acceptance limits to detect evidence of equipment malfunction. Maintenance is performed for decreases in resolution, shifts in calibration, decreased sensitivity, or failure to meet other QC criteria.

Having an adequate inventory of expendable supplies and critical instrument components available for use minimizes instrument downtime. Expendables are those items considered to have a lifetime of less than one year. Responsibility for performance of preventive and remedial maintenance lies with the instrument user, QA/QC chemist and ultimately the Laboratory Manager.

Performance and System Audits

Systems audits are qualitative inspections of all laboratory operations. The purpose of these audits is to determine whether the laboratory has the resources, capability, and capacity to perform the requisite work and to verify that protocols are followed appropriately during the course of the work. Adequacy of personnel, facilities and equipment, application of methodology and quality control procedures, and acceptability of data

handling and documentation are all evaluated in the systems audits, which are performed annually, or as needed.

Performance audits determine the accuracy of the measurement system by proficiency testing with the analysis of "unknown" samples. The laboratory receives samples from the United States Environmental Protection Agency as part of the Discharge Monitoring Report (DMR) Quality Assurance Program (analyzing for parameters routinely reported to government agencies), and the Water Pollution (WP) Performance Evaluation Program, in which samples are analyzed for all accredited parameters. In addition, the laboratory participates in a nationwide round robin performance evaluation program annually.

Quality Assurance Reports

On a as-needed basis, QA reports are prepared summarizing the status of the QA/QC program, documenting problematic conditions, and evaluating the attainment of QA/QC objectives of the program. These reports function as a feedback mechanism on QA/QC performance and data quality. Information is presented in combinations of narrative text, tabular summaries, statistical charts, or schedules. The reports include the following:

- Status of the program
- Assessment of analytical data quality for precision and accuracy
 - Status of performance and system audits
 - Significant quality control problems and recommended solutions
 - Corrective actions taken for any problems previously identified
 - Recommendations for potential changes in the quality assurance program

QA reports are prepared by a laboratory chemist, as designated and given to the QA/QC Chemist and laboratory staff. This feedback to program management helps to assure progress in attainment of objectives for data quality.

In addition, quality assurance issues are reviewed by the Laboratory Manager and QA/QC Chemist on an on-going basis through monthly reference sample result reports, nonconformance log updates, LIMS audits, and weekly meetings with the chemists and technicians.

QA/QC Flags


The Wastewater Treatment Facility Laboratory designates QA/QC flags on outgoing reports and in LIMS when reanalysis of the sample is not possible, for example, due to insufficient sample volume or lapse of holding times. It is the standard of the laboratory to use codes designated by SWAMP (Source Water Assessment and Monitoring Program), unless none are designated. Below is a list of possible flags, if the need for additional flags arises they will be added to the list.

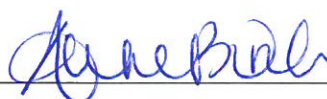
CODE	DESCRIPTION
AW (Army Corp.)	Detection limit increased due to dilution factor
BLM	Compound unidentified at a second dilution
BY	Sample received at improper temperature
BZ	Sample preserved improperly
CS	QC criteria not met due to analyte concentration near RL
CT	QC criteria not met due to high level of analyte concentration

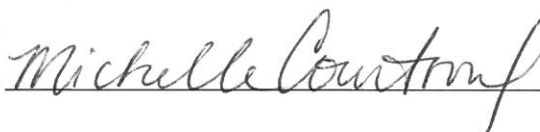
D	Analytes analyzed at a secondary dilution
DB	QA results outside of acceptance limits due to matrix effects
EU	Lab Control Spike is outside of acceptance limits. MS/DMS are accept., no corr.
EUM	Lab Control Spike is outside of control limits.
FX	Analyte present in the instrument blank
GB	Matrix spike recovery not within control limits
GBC	CRM analyte recovery not within control limits
H	A holding time violation has occurred.
HH	Result exceeds linear range; concentration may be understated
IL	RPD exceeds laboratory control limit
IP	Analyte detected in method, trip, or equipment blank
M	A matrix effect is present.
JA	Analyte positively identified but quantitation is an estimate
UJ	Analyte was not detected above the reported sample quantitation limit. Reported quantitation limit is approx. & may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

SIGNATURE PAGE

QA/QC Chemist:  Date: 01-06-2016

Environmental Microbiologist II:  Date: 1/20/16

Chemist II:  Date: 1.10.16

Chemist I/II:  Date: 1-7-16

Laboratory Technician: _____ Date: _____

Lab/Environmental Compliance Manager:  Date: 01.06.16