





## 1.0 Introduction of the Laboratory Quality System and Organizational Structure

This Quality Assurance Manual for the laboratory serves as the Quality Assurance Project Plan (QAPP) for the Guam EPA Laboratory. This document now replaces the QAPP for the Microbiology and Chemistry Laboratories.

### 1.1 Laboratory Quality Mission statement

The Guam Environmental Protection Agency (GEPA) Laboratory is the principal state laboratory for Guam. The laboratory was established to provide analytical support to the GEPA Water Division (Safe Drinking Water (SDW), Water Resources, and National Pollution Discharge Elimination System (NDPES) programs); and the GEPA Environmental Monitoring and Analytical Services (EMAS) Division (Monitoring program); the laboratory may also serve any future requirements that GEPA may require. The purpose of this document is to assure that GEPA laboratory's program generates data that are of acceptable quality, known level of confidence, scientifically valid, and legally defensible as required by the different Quality Assurance Project Plans (QAPP) of the GEPA programs requiring the laboratory's services. If there are no specified requirements found in a QAPP, the laboratory will use requirements specified in US EPA approved methods or the American Public Health Association (APHA) standard methods for the examination of water and wastewater.

Briefly, the Guam EPA laboratory has two primary functions: (1) it assures that proper quality control practices are implemented in day-to-day laboratory tasks, and (2) it assures that reported data are valid and complies with the levels specified by the different GEPA QAPPs or approved standard methods.

In addition to this, and in compliance with the primacy requirements of the Safe Drinking Water Act (SDWA), GEPA laboratory personnel are required to attend and pass the US Environmental Protection Agency's Laboratory Certification Officers (LCO) Course. This certification allows GEPA to administer the certification process of Drinking Water Laboratories who would like to provide analytical services to Guam. Details of this are covered in the Laboratory Certification Program Manual.

### 1.2 Quality Policy

Guam EPA Laboratory is committed to providing the Agency with data of the highest possible quality. This is accomplished by following a documented quality system and adherence to those quality control procedures. This quality system is documented in this Quality Assurance Manual and other project specific QAPPs. This process will assist the Agency's decision makers in making sound decisions for the protection of the environment.



All Guam EPA laboratory personnel are required to be familiar with all facets of the quality system relevant to their position and implement these policies and procedures in their daily work.

GEPA management demonstrates its commitment to quality by providing the resources, to include facilities, equipment, and personnel, to ensure the adherence to these documented policies and procedures and to promote the continuous improvement of the quality system.

### 1.3 Laboratory Organization and Responsibility

An overview of the Guam EPA laboratory's organization is provided in Appendix A, Guam EPA Laboratory Organization chart. The chart shows both the laboratory organization and the monitoring group which together is the Environmental Monitoring and Analytical Services (EMAS) Division. The following is a description of key employees of the laboratory or Analytical Services group; and their responsibilities:

*Environmental Monitoring and Analytical Services (EMAS) Division Administrator* – responsible for the general operations and supervision of the EMAS Division which includes the GEPA laboratory. This includes administration, regulatory and technical operations. He or she also acts as the Quality Assurance Officer (QAO) of the laboratory. The EMAS administrator should have a Bachelor's degree in chemistry or a related field and should have at least two years of experience managing a laboratory.

The EMAS Division Administrator should also have undergone at least two weeks training in chemical analysis of drinking water at a Federal or State Agency, or on-the-job training at a certified laboratory.

*Chemist III* – currently the position is vacant. The Chemist III is responsible for the scheduling and organizing of laboratory activities. The Chemist III ensures that all laboratory personnel have demonstrated the ability to satisfactorily perform analysis and that all data produced meet quality assurance and regulatory criteria. In addition to this, the Chemist III is also part of the team of chemists doing analytical tests and day to day operations. The final review and revisions of quality documents and SOPs is one of the main duties of the Chemist III. The Chemist III also provides the main input in any quality decisions that the laboratory has to make. The Chemist III does the job of the Chemist II or Chemist I in times of personnel leaves or any vacancies. The Chemist III must also be able to participate in a successful analysis of PT samples.



The Chemist III has the same qualifications as the Chemist II including having undergone at least two weeks training in chemical analysis of drinking water at a Federal or State Agency, or on-the-job training at a certified laboratory. In addition to this the Chemist III is qualified for this position by seniority, length of service and other factors.

Specific responsibilities include but not limited to:

- Schedules and organize analytical activities with consideration to holding times and due dates in response to programs sampling and other Agency requirements.
- Enforce QA/QC procedures and requirements within the laboratory
- Makes the final review of analytical reports, quality document issues and revisions prior to final approval by EMAS Administrator/QAO
- Write or update Quality Documents as needed.
- Perform annual audit review to evaluate suitability and effectiveness of the quality system in place and make necessary changes or improvements
- Works as a team with other team members in any day to day laboratory operation.

*Chemist II* – directly involved in the day-to-day operation of the laboratory and performs analytical tests with minimal supervision. In addition to this, the Chemist II is also part of the team of chemists doing analytical tests and day to day operations.

The Chemist II should also have a bachelor's degree in chemistry or related field or at least one-college level laboratory course in environmental chemistry. In addition, he/she should have undergone at least two weeks training in chemical analysis of drinking water at a Federal or State Agency, or an on-the-job training or equivalent experience at a certified laboratory. The Chemist II should also have the work experience and training of a Chemist I or its equivalent. Before analyzing compliance samples for drinking water, the Chemist II must demonstrate acceptable results on unknown proficiency test (PT) samples.

Specific responsibilities include but not limited to:

- Performs analysis with consideration to holding times and due dates
- Implement QA/QC procedures and requirements within the laboratory
- Review analytical reports prior to final review by the Chemist III and approval by EMAS Administrator/QAO
- Write or update Quality Documents and SOPs as needed.
- Perform annual audit review to evaluate suitability and effectiveness of the quality system in place and make necessary changes or improvements

*Chemist I* – should perform analytical tests with supervision by the Chemist II and/or Chemist III and have at least a college degree in Chemistry or the equivalent. The Chemist I will be directly involved in the day-to-day operation of the laboratory. The Chemist I should also have training in chemical analysis including a minimum of 30 days on-the-job training. Before analyzing compliance samples



for drinking water, the Chemist I must demonstrate acceptable results on unknown performance test (PT) samples. As with the Chemist II, the Chemist I must also be able to participate in a successful analysis of PT samples. The chemist's responsibilities include but not limited to:

- Implement required QA/QC criteria in the laboratory
- Notify the Chemist III and Chemist II and the QAO of any quality problems
- Identify and implement corrective actions within his/her scope of responsibility
- Participate in the laboratory's training program such as review of standard operating procedures (SOPs), QA Manual, etc.

*Quality Assurance (QA) Officer* – reports directly to the Administrator of GEPA and is responsible for maintaining and implementing the quality assurance in the laboratory. Currently, this is performed by the EMAS Division Administrator. Responsibilities include but not limited to:

- Implement and monitor compliance of the laboratory's Quality Manual (QAM)
- Review and update the laboratory's QAM at least annually
- Conduct audits of the laboratory's operation on a periodic basis to evaluate its compliance with the other programs' QAPPs. Recommend corrective actions if necessary.
- Ensure that data generated by the laboratory is scientifically valid, legally defensible and of known precision and accuracy
- Coordinate the analysis of performance evaluation samples to maintain drinking water certification
- Develop and implement new QA procedures
- Review all results prior to final approval by the EMAS Administrator
- Maintain the repository of all Standard Operating Procedures (SOPs) and training logs

## 2.0 Quality Assurance Objectives

Data Quality Objectives (DQOs) are qualitative and quantitative measures that are used in environmental decision making. These are action limits upon which decisions are based and should be defined by each GEPA programs in their QAPPs or in Guam regulations. The laboratory's detection limits and its decisions concerning methods to use are dependent on program defined criteria that are in the QAPPs or in Guam regulations.

Before analytical data can be used, the quality of data produced by Guam EPA Laboratory is defined by the following Measurement Quality Objective (MQOs) characteristics: precision, accuracy, comparability, and sensitivity. Completeness, representativeness are functions more related to field sampling and sample design



and should be clarified by each program QAPPs. Documentation is more related to the laboratory's presentation of data in its reports. This may be specified in a program QAPP. As a general guideline a definition of each characteristic above follows below:

## 2.1 Precision

Analytical precision is an important component of overall data quality since it is a measure of how far an individual determination may be from the mean of replicate measurements (how well replicate analyses agree). If the precision of an analysis is poor, there is a good probability that the reported result will differ substantially from the true value even if there are no systematic errors leading to bias in the result. Precision is often directly related to concentration.

2.1.1 Guam EPA laboratory uses Relative Percent Difference (RPD) to measure agreement between duplicate analyses. RPD is calculated as follows:

$$RPD = [(S-D) \div (S+D)/2] \times 100$$

Where;

RPD = Relative Percent Difference

S = First Sample Value (original)

D = Second Sample Value (duplicate)

## 2.2 Accuracy

Accuracy is the agreement between an experimentally determined value and the accepted reference value (deviation of the analytical value from the "true or known value). Analytical accuracy is a measure of analytical bias due to systematic errors. A measure of this bias along with a measure of the precision will provide the overall accuracy of the results. The true value for field samples are never known, so accuracy measurements are made on the analysis of QC samples analyzed with field samples. The primary QC tools for assessing accuracy are laboratory fortified blanks (LFBs), laboratory control standards (LCSs), matrix spikes or spiked matrix duplicates (MS/MSD), and Proficiency Testing/Performance Evaluation (PT/PE) samples.

2.2.1 For accuracy, Guam EPA laboratory uses % recoveries (LCS) as %R which is calculated as follows:

$$\%R = \text{found concentration} \div \text{true concentration} \times 100$$



2.2.2 For spiked recoveries in samples (MS/MSDs) it is calculated as follows:

$$\%R = (SSR - SR) \div SA \times 100$$

Where:

%R = percent recovery  
SSR = spiked sample result  
SR = sample result  
SA = spiked amount

### 2.3 Comparability

The characteristic of comparability is achieved when a method is conducted in a consistent manner while the conditions in the laboratory is consistent. The supplies, reagents, and reagent water used in an analytical run under consistent laboratory conditions must be of the same quality thus assuring the analysis is done in a comparable manner.

### 2.4 Sensitivity

Sensitivity is the characteristic that measures the detection limits of a test method which in turn gives an idea as to how data can be reported with confidence. A discussion on method detection limits (MDL) as well as minimum reporting limits (MRL) is detailed in sections 3.2.2 and 3.2.3.

Detection limits of the instruments or equipment are specified by the manufacturer. This is the starting data that is used in determining the MDL during the initial startup of the method using the instrument or equipment. Other references for determining the MDL can be found in approved Standard or EPA methods.

### 2.5 Representativeness

Representativeness is the homogeneity of the samples. Unless a sample is known to be heterogeneous this is easily achieved especially for drinking water samples. For Microbiological samples it is a must that samples are shaken 25 times during enumeration methods assuring bacterial cells are well distributed in the samples. For chemistry shaking before an aliquot is taken is a required protocol.

### 2.6 Completeness

The characteristic of completeness is a measure of the percentage of specified data which are valid. Valid data are obtained when samples are



analyzed in accordance with the quality control procedures outlined in this manual and none of the quality control criteria is exceeded.

Sample data which does not meet the specified quality control criteria will automatically be reanalyzed if sufficient quantity of sample is available and analytical holding times have not been exceeded. The laboratory strives for a completeness percentage of 100%

## 2.7 Documentation

Documentation is accomplished by the availability of all raw data, Laboratory Information Management System (LIMS) report and Quality Control Check data for all analytical reports produced for compliance purposes. An analytical result in a sample can be traced through documentation from sampling, laboratory processing, analytical run and final laboratory report.

## 3.0 Quality of Test Results

As a general guideline the quality objectives of analytical runs for the laboratory are discussed in this section. The frequency and application of data quality objective as defined in each SOP is shown in Table 6.2.

### 3.1 Quality Control Checks

Test results are evaluated by the use of quality control checks (QC checks). These checks are designed to determine the bias and precision of a particular analytical run. In each test method (SOPs), this is specified by the use of negative controls, laboratory control standards (LCSs), which gives us an idea on the accuracy of the test results; duplicate testing (dups) for precision evaluation; and laboratory fortified matrix (LFM) or matrix spikes and matrix spike duplicates (MSs, MSDs) which give us an idea of the bias in test results. The following sections defines the different QC checks used in SOPs.

#### 3.1.1 Negative Controls

##### 3.1.1.1 Method Blanks

A method blank also known as laboratory reagent blank (LRB) consists of laboratory deionized water containing all of the reagents utilized in the analytical procedure. The method blank (MB) is prepared in the same manner as a sample and is processed through all of the analytical steps. All reagents are dated upon receipt in the laboratory and each new lot of reagents are checked by performance of method blanks.



Method blanks are processed along with the associated samples (minimum one MB per prep batch or analytical batch of  $\leq 20$  samples) performed to determine whether there is reagent contamination or instrument contamination due to sample carryover. The method blanks must remain below the minimum detection limit (MDL) of the test method.

In microbiological test methods, the method blanks are supplied with sterile water which is deionized water that is sterilized in an autoclave at  $\geq 121$  psi for at least 30 minutes. The sterility is verified by a nonselective media on a monthly basis.

When a blank is determined to be contaminated, the cause must be investigated and measures taken to minimize or eliminate the problem. Samples associated with a contaminated blank shall be evaluated as to the best corrective action for the samples (e.g. reprocessing or data qualifying codes).

Method blanks are analyzed as part of the initial or daily calibration process (calibration blanks) and after every 20 samples for each matrix type to monitor the overall procedural blank as well as the purity of the reagents

#### 3.1.1.2 Trip Blanks

The trip blank is required to be analyzed in the event of any detects in the associated field samples. This is prepared with sampling bottles with the applicable preservatives that is filled by laboratory deionized or sterile water in the laboratory environment.

If there is adequate holding time remaining the analyst may elect to not analyze the trip blank. However in this case, the data should be reduced immediately and if there are hits, the trip blank should be analyzed on the next run, still within holding time.

#### 3.1.1.3 Field Blanks

Field blanks are used to identify contamination that may have occurred during the sample collection process. Empty containers with the applicable preservatives are sent to the field and filled at the sampling location at the time of sampling from a bottle with analyte-free water that was prepared at the laboratory as per client's request. The empty sample bottle and field blank sample bottle will be provided.

#### 3.1.1.4 Sample Blanks

Sample blanks are used with spectrophotometric methods where sample characteristics such as color may give erroneous results. The absorbance of a sample is measured before and after the color development process. The absorbance before is subtracted from the absorbance after to give the true absorbance. Sample blanks are analyzed on an as needed basis



### 3.1.1.5 Calibration Blanks (CB)

Calibration blanks are prepared along with the calibration standards and differ from the standards only in that the calibration blank does not contain any of the analyte(s) of interest. The calibration blank, by definition, provides the "zero point" in the calibration curve.

### 3.1.2 Positive Controls

#### 3.1.2.1 Laboratory Control Sample (LCS)/ Laboratory Fortified Blank (LFB)

The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis.

Laboratory control samples (LCSs) are defined as an interference free matrix sample that is spiked with a particular set of method-specific target compounds at a level 5-10 times above the minimum reporting limit. The matrix used to prepare aqueous LCS samples is laboratory reagent water. In Guam EPA laboratory, there are two types of LCS, the calibration verification (CV) and the Laboratory Fortified Blank (LFB). LCS spiked by a second or independent source is called the LFB while the LCS spiked by the calibration standards is called the CV. The CV is used to verify if the calibration curve is acceptable. CVs are used at a frequency of one per 10 samples while the LFB is one per analytical run (please see continuing calibration verification below).

In microbiological methods, the positive control is a sterile water contaminated with the organism being tested for. Other species but similar to the target organism is used to confirm that a similar type of bacteria will not give false positives in the media being used.

#### 3.1.2.2 Continuing Calibration Verification (CCV)

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

#### 3.1.2.3 Matrix Spike (MS) or Laboratory Fortified Matrix (LFM)

Matrix Spike (MS) or Laboratory Fortified Matrix (LFM) are field samples collected that are spiked with a known amount of a compound similar to the target analyte. MSs or LFM are used to ascertain the effects of the matrices of samples collected. The recoveries in these samples determine the accuracy and precision of the method in the matrices of samples collected. The frequency of MSs or LFM is method specific and is detailed in the SOPs.



## 3.2 Demonstration of Capability

In Guam EPA Laboratory, before a method is adopted, the method must be from a reputable source such as US EPA, Standard methods and others. Then the method undergoes evaluation as described in the following:

### 3.2.1 Method Detection Limit (MDL)/Limit of Detection (LOD)

3.2.1.1 Method Detection Limit or Limit of Detection is defined as the minimum concentration of an analyte that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero, but the exact concentration cannot be reliably quantified. For instance, if the true concentration of an analyte in a sample is equal to the MDL, there is a 50 percent chance that the analyte will be detected.

3.2.1.2 Method Detection Limits (MDLs) are determined as per 40CFR, part 136, Appendix B of the Code of Federal Regulations (CFR). Essentially, this requires that an estimate of the detection limit be determined for each target analyte based on analytical experience or published references. Seven replicates of DI water must then be spiked at this estimated MDL for each method analyte carried through the entire procedure over a minimum of 3 separate analysis/extraction days. The MDL is then calculated as the standard deviation of the 7 replicates multiplied by the statistical "t-value" associated with the actual number of replicates analyzed assuming N-1 degrees of freedom (for exactly 7 replicates, the t-value is 3.143; 40 CFR, Part 136).

3.2.1.3 MDLs are determined at least annually and by each analysts that uses the test method.

3.2.1.4 An MDL study must be repeated for each new analyst trained in a particular method, or if there is a change in the instrumentation or the test method that is used for the analysis in question. This is a necessary requirement to ensure that each new analyst has received sufficient training such that the data generated will be comparable to that of former analysts. It is necessary to repeat the MDL process with a change in instrumentation to ensure that the new instrumentation is capable of achieving equivalent sensitivity. An MDL study must also be repeated when there is any significant change in background or instrument response.

### 3.2.2 Minimum Reporting Limits (MRL) or Practical Quantitation Limits (PQL)



- 3.2.2.1 The Minimum Reporting Limit (MRL) is the lowest concentration normally reported. The MRL represents a conservative, nominal reporting limit designed to be representative of the minimum quantifiable concentration level for a particular analyte in a real environmental matrix as opposed to the statistically derived MDL calculation.
- 3.2.2.2 The MRL will generally be established by multiplying the statistically derived MDL by a factor of 2 or 3. The rationale for this approach is that the resultant value becomes approximately 10 times the standard deviation obtained during the MDL study; the EPA frequently refers to this concentration as the "Limit of Quantification (LOQ)", and defines it as the level above which accurate quantitation can be achieved. This level is also more similar to the SW-846 and SDWA concept of "Practical Quantitation Limits" (PQL). At a minimum, the MRL needs to be greater than or equal to the MDL.
- 3.2.2.3 Perform an MRL check and calculate the acceptance criteria for recovery of spiked analyte at MRL is 50-150 % or  $\pm 3$  standard deviations, whichever is greater if the method does not specify acceptance limits. An MRL Check is done every analysis day as per EPA Manual for the Certification of Laboratories Analyzing Drinking Water, 5th Edition.
- 3.2.2.4 If an analytical result indicates that the analyte level is between the MDL and MRL. This would be flagged or qualified with a letter "J".
- 3.2.3 Initial Demonstration of Capability (IDC) or Determination of Capability (DOC)
- 3.2.3.1 An IDC is a preliminary test done to test that test results in recoveries acceptable as specified in the SOP is performed for each instrument, new method, and analyst.
- 3.2.3.2 The IDC for each analyst includes a demonstration of the ability to achieve a low background, the precision and accuracy required by the method, the method detection limit (MDL) in accordance with procedure in 40 CFR 136, Appendix B and satisfactory performance on an unknown sample as on-going proficiency test result are also filed.
- 3.2.3.3 The IDC is repeated whenever there is a change in analyst, method, or instrument.
- 3.2.3.4 All IDC done are documented and filed by analyst, method-SOP, and instrument.
- 3.2.3.5 Continuing demonstration of method performance (such as laboratory control and matrix spike samples) is monitored by use of control charts.
- 3.2.3.6 The QC sample used for the IDC analysis is obtained from an outside source. If an external vendor is not available, the laboratory prepares the QC sample independent of the instrument calibration standard.



- 3.2.3.7 The QC sample concentration prepared for the IDC is approximately 1-4 times the MRL for spike concentration if not specified by the method or regulations. Four aliquots of the sample are analyzed concurrently (same day) or over a period of days. Average recovery and standard deviation for each parameter of interest are calculated in the units used for reporting to clients. The resulting average recovery and standard deviation must meet the acceptance criteria for the method.
- 3.2.3.8 When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory assesses performance against established and documented criteria. If there is no mandatory criteria in the method, either reference or laboratory generated limits are used.
- 3.2.3.9 If standards cannot be prepared, as for Microbiology, QC samples or PE samples obtained from NIST or other approved PT providers are used for the IDC. The laboratory retains all associated supporting data necessary to reproduce analytical results summarized in the IDC certification statement. The Microbiology DOC SOP provides the details of the DOC procedure. Please see Appendix B of this manual for Microbiological quality checks.
- 3.2.3.10 Analysis of actual samples is not done until all parameters of interest for the IDC meet acceptance criteria. If one or more of the test parameters do not meet the acceptance criteria, the problem is corrected, followed by repeated analysis of the four aliquots for those that failed to meet criteria. If the repeat analyses fail acceptance criteria the laboratory investigates, corrects the problem and repeats the test for all parameters.

#### 4.0 Sample Collection, Preservation, Identification, Handling , and Storage

Sample collection and sample handling techniques are important aspects of the overall sample analysis process and have a major impact on the validity of the results. Specific containers and preservatives are used to ensure that the analytes originally present in the sample are not lost through degradation or do not become more concentrated. In addition, contaminants that would interfere with the analysis or give erroneously high results must be mitigated. Because of this it is important that the different programs in Guam EPA adhere to their sampling techniques when submitting samples to the laboratory. The sampling method must be included in their respective QAPPs. The following gives us guidelines in the sample preservation, handling and storage of samples as they are transported to the laboratory.

##### 4.1 Sample Collection and Bottle Preparation

Quality analytical data requires that the collected sample is representative of the sampled area. Sampling procedures should adhere to the guidelines established by EPA and other



regulatory agencies and be appropriate for the sample matrix and types of analytical parameters to be determined. This should be specified in the different program QAPPs requiring laboratory analytical services. The laboratory can provide sampling instructions to the other programs in Guam EPA on the appropriate sample collection procedures.

Guam EPA laboratory staff does not routinely collect samples. The laboratory provides information on the proper collection and preservation of samples as described in Method 1060 of the Standard Methods for the Examination of Water and Wastewater, 20<sup>th</sup> edition. Sample collectors are required to be trained on these field sampling procedures before they can collect samples.

Sample bottles for all analyses except bacteriological are pre-cleaned according to EPA Protocol specifications. This is specified through guidelines set in Table 1V-1 of the US EPA Manual for the Certification of Laboratories Analyzing Drinking Water 5<sup>th</sup> edition (MCLADW 5<sup>th</sup> ed.).

#### 4.2 Sample Containers, Preservatives, Storage Conditions and Holding Times

The container types, storage conditions, preservatives and holding times are also important in assuring the quality of analytical data produced by the laboratory. They must be able to disallow interferences, preserve the amount of analytes, and allow the ease in subjecting the sample to the requirements of the analytical procedure. Storage conditions should be also right so as not to increase or decrease the amount of analyte in the sample from the time of sampling up to the time of analysis.

These are also specified in Table IV-1, and Table IV-6 of the MCLADW 5<sup>th</sup> ed.

#### 4.3 Sample Disposal

There are three streams of laboratory wastes identified by the laboratory. These are the nitrate/phosphate/silicates/ammonia Flow Injection Analyzer (FIA) wastes without mercury; the chloride FIA wastes which contain mercury ions; and the microbiological wastes.

The first two wastes are placed in separate plastic drums; one for FIA wastes without mercury and another for FIA wastes with mercury. These are then emptied by a hazardous waste handler/contractor.

The microbiological wastes are placed in autoclavable bags and are sterilized in an autoclave and disposed of in the trash.

For Extra Sample volumes that are not processed during analysis, the extra sample volumes are poured down the drain.

#### 5.0 Sample Management



The protocol in sample management is described in the SOP *Sample Registration, Acceptance and Log In*. The GEPA SOP number is MB-01-03. Data are stored in a Laboratory Information Management System (LIMS).

## 6.0 Laboratory Test Methods and SOPs

The laboratory test methods used by the laboratory are based on approved standard methods and EPA methods. They are written as SOPs. These methods are implemented in the laboratory as a requirement of the QAPPs needing analytical services. The table below shows the laboratory test methods employed by the laboratory.

Table 6.1 Laboratory Test Method and SOP

Parameter	Method Description	Approved Method	GEPA SOP	MDL	MRL
Total Coliform and E. coli	Simultaneous chromogenic detection, enumeration and confirmation of total coliforms and E. coli	SM 9223	MB-01-04	1 cfu/100mL	
Enterococcus	Chromogenic detection and enumeration and confirmation of <i>Enterococcus</i> genus	ASTM D6503-99	MB-01-05	1 cfu/100 mL	
Nitrate and/or Nitrite as N	Flow Injection Analysis of Nitrates and Nitrites in Water	EPA 353.2	CH-01-01	Nitrate: High 0.02 mg/L Low 2 µg/L	Nitrate: High 0.10 mg/L Low 10µg/L
				Nitrite: High 0.01 mg/L Low 2 µg/L	Nitrite: High 0.05 mg/L Low 10 µg/L
Orthophosphate as P	Flow Injection Analysis of Orthophosphates (as P) in Water	EPA 365.1	CH-01-02	High 0.005 mg/L Low 2 µg/L	High 0.025 mg/L Low 10 µg/L
Ammonia as N	Flow Injection Analysis of Ammonia-N in water	EPA 350.1	CH-01-03	0.01 mg/L	0.05 mg/L
Chlorides	Flow Injection Analysis of Chlorides in water	SM 4500 Cl E	CH-01-04	1 mg/L	5 mg/L
Conductivity	Conductivity Meter	EPA 120.1	CH-01-05	Instrument specified	Instrument specified



Parameter	Method Description	Approved Method	GEPA SOP	MDL	MRL
pH	pH meter	SM 4500-H <sup>+</sup>	CH-01-06	n/a	n/a
Turbidity	Nephelometric Measurement	EPA 180.1	CH-01-07	0.05 NTU	0.05 NTU
Total Nitrogen and Total Phosphorus	Peroxysulfate digestion in an autoclave followed with determination by Flow Injection Analysis	USGS-I-2650-03	CH-01-08	Nitrogen: 0.10 mg/L	Nitrogen: 0.30 mg/L
				Phosphorus: 5 µg/L	Phosphorus: 15 µg/L
Silica	Molybdate Reactive method determination by Flow Injection Analyzer	SM 4500-SiO <sub>2</sub> C	CH-01-09	0.10 mg/L	0.20 mg/L
Total Suspended Solids	Gravimetric Method	SM 2540 D	CH-01-10	1 mg/L	5 mg/L
Total Dissolved Solids	Gravimetric Method	SM 2540 C	CH-01-11	1 mg/L	5 mg/L

Copies of the SOPs are available to all analysts at a central location in the laboratory and a separate copy is kept at the office of the Quality Assurance officer. SOPs are also reviewed annually and revised if necessary to reflect the changes.

Table 6.2 Laboratory Test Method and Quality Objectives

Parameter/Methods	Quality Objective		Frequency
	Objective	Acceptable Result	
Microbiological Methods: Guam SOP MB-01-04 and MB-01-05 <i>Coliforms and Enterococci</i>	Laboratory Blanks	Negative	One per analytical run
	Field Blanks	Negative	One per sampling run
	Positive Controls	Positive for Target Test organism	One per new batch of media and quarterly afterwards.
	Quality Control Checks	Effectively implemented	Please see <b>Appendix B</b>
Chemistry Methods: Guam SOP CH-01-01 to -04; and CH-01-08 to -09 <i>Nitrates, Nitrites, Orthophosphates, Ammonia, Chlorides,</i>	Calibration Verification	90-110% Recovery	One per ten samples
	Calibration Blanks	< Minimum Reporting Limit (MRL) or Quantitation Limit (QL)	One per ten samples
	MRL or QL	50-150% Recovery	One per analytical run



Parameter/Methods	Quality Objective		Frequency
	Objective	Acceptable Result	
<i>Total Nitrogen and Phosphorus, and Silica</i>	Laboratory Reagent Blank (LRB)	≤ Minimum Detection Limit (MDL)	One per analytical run
	Laboratory Fortified Blank (LFB)	90-110% Recovery	One per analytical run
Chemistry Methods: Guam SOP CH-01-01 to -04; and CH-01-08 to -09 <i>Nitrates, Nitrites, Orthophosphates, Ammonia, Chlorides, Total Nitrogen and Phosphorus, and Silica</i>	Laboratory Duplicate (LD)	RPD ≤20% for samples with analyte levels ≥5X QL; For all others the difference between duplicate should be <QL; For samples < QL, RPD is N/A	One per 10 samples
	Matrix Spike (MS) or Laboratory Fortified Matrix (LFM)	75-125% recovery for samples with analyte level < 4X the added spike; for others 90-110% recovery	One per 10 samples.
Chemistry Methods: Guam SOP CH-01-05 <i>Conductivity</i>	If Meter is used for low level readings (0-199.9 μS) and/or at extreme temperatures, calibrate weekly	Result must be within 5% of standard otherwise re-calibrate.	Meter is calibrated with a standard within the range level of anticipated results. Once per analytical run
	If meter is used with a mid range (200-1999μS) calibrated at least monthly by wash probe with deionized water and store dry		
	LD	RPD ≤20%	One per 10 samples.
Chemistry Methods: Guam SOP CH-01-06 <i>pH</i>	Meter is calibrated with a three pH point buffers (4.0, 7.0, & 10.0)	Difference of pH reading from standard is ±0.1 pH unit)	Meter is calibrated before each use.



Parameter/Methods	Quality Objective		Frequency
	Objective	Acceptable Result	
	LD	Difference between lab duplicate samples is $\pm 0.1$ pH unit	One per 10 samples.
Chemistry Methods: Guam SOP CH-01-07 <i>Turbidity</i>	Meter is calibrated according to Manufacturer's instruction with primary standards	Calibration accepted by instrument and instruments prints calibration data	Quarterly
	Check calibration with secondary standards	Difference must $\leq 5\%$	One per 10 samples
	LD	Difference must $\leq 5\%$	Once per analytical run
Chemistry Methods: Guam SOP CH-01-010 to -011 <i>Total Suspended Solids and Total Dissolved Solids</i>	LRB	$\leq$ Minimum Detection Limit (MDL)	One per analytical run or one per 20 samples whichever is lower
	LD	RPD $\leq 20\%$ for samples with analyte levels $\geq 5X$ QL; For all others the difference between duplicate should be $< QL$ ; For samples $< QL$ , RPD is N/A	One per 10 samples

## 7.0 Calibration Procedures

In each SOP calibration procedures of necessary instruments are detailed. These are verified by Continuing Calibration Verification (CCV) samples and Calibration Blanks (CB) for every 10 samples in an analytical run.

For microbiological samples, the target organism is used as a positive control and similar organisms that do not react with the selective media as negative controls. Sterile water is used as a blank to check for any interference from the sterile water used for dilution in the microbiological analytical test. These are also detailed in each SOP for microbiological parameters.

## 8.0 Data Reduction, Validation, and Reporting

The process of transforming raw analytical data into a finished report involves steps which are generally grouped into the categories of data reduction, data validation, and reporting. It involves mathematical modeling of the standard calibration curves, statistical analysis of



the acquired data, calculations to account for preparation steps and dilution, verification of adherence to quality assurance procedures, and the generation of hardcopy output.

### 8.1 Data Reduction

The analyst or team of analysts performing an analysis has the primary responsibility for reducing raw data. This process consists of converting raw data values into final, reportable values by comparing individual sample results to those obtained for calibration purposes and then accounting for any dilution or concentration procedures.

The extent to which raw data from the instrument needs to be mathematically processed varies depending on the analysis. For the following methods finished data is directly read from the instrument; pH, conductivity, spectrophotometric/colorimetric Flow Injection Analyzer (FIA) measurements (i.e.: nitrate/nitrite, orthophosphate), and presence/absence bacteriological tests. Mathematical processing of data occurs when samples are diluted to fit the standard calibration curves obtained.

### 8.2 Data Validation

Upon completion of each analytical run, the analyst checks the raw data and QC to determine if the run is acceptable for submission. Data are entered into the laboratory LIMS. The data package is submitted to the Peer analyst for review. The submitted package contains all relevant documentation such as chromatograms, instrument run logs, digestion logs, information about calibration or second source standards and reagents, and other printed pages from the instruments, result summary sheets, and/or a checklist.

The Reviewer is responsible for verifying the validity of the data by determining if all quality control parameters have been analyzed and are within method acceptance limits, checking calculations, assessing the acceptability of standard calibration curves, and addressing any inconsistencies in the data with the analyst. Deviations from the method should be documented by the analyst and/or reviewer. The documentation should explain the deviation, any flags or qualifiers associated with the deviation and the acceptability of the data. The review includes a perusal of the supporting documentation to ensure that the documentation is present and is complete. Before the Reviewer or Validator approves the data in the LIMS, data in the package is checked against the LIMS to ensure that there are no transcription errors, retests have been properly initiated, the appropriate flags have been added, and any comments regarding data/sample integrity have been added at the run/sample level.

### 8.3 Data Reporting

After all data has been reviewed and peer reviewed, the analytical report is produced by the LIMS or by excel reporting depending on the request of the program requiring the analysis. The report is approved by the EMAS Division Administrator or QAO.



## 8.4 MCL Notifications

Any exceedances to US EPA Maximum Contaminant Levels or the program's QAPP's criteria, should be reported immediately to the Program Managers, who in turn report them to their corresponding Division Managers. The Division Managers will take it up with Guam EPA Administrator for further administrative or enforceable action.

## 9.0 Control of Non-Conforming Work, Corrective Action, and Preventive Measures

When a failure to meet quality control criteria in analytical runs, or when internal or external audit samples are not acceptable, corrective action may be necessary. A list of possible corrective actions are listed in Table 9.1. These are general guidelines as to what to do when encountered with deviations in the different quality control standards on a method by method basis.

### 9.1 Selection and Implementation of Corrective Action

Failure to meet criteria of the LCS, surrogate spikes and continuing calibration standards, holding time exceedance, improperly preserved samples, method blank contamination, and not passing proficiency testing (PT) samples are QC failures that trigger the generation corrective actions to identify the root cause of the problem. Root causes of problems are documented in the analytical report as comments after they have been identified. The data produced in these runs are flagged to let the other program managers know of the problems and their causes.

### 9.2 Documentation of Corrective Actions

9.2.1 Ordinary corrective actions taken are documented as comments that are attached in the analytical reports. All other corrective actions entailing more system corrections are documented in corrective action reports (CAR) which are in the form of a report memo submitted to the EMAS Division Administrator or QAO.

9.2.2 Results are flagged or qualified for all QC failures that have may have an impact on the data quality of the result.

9.2.3 Data qualifiers are used by the laboratory in reporting analytical results to flag the user about the data. Some of the qualifiers are requested by a program manager as required in the Project's Quality Assurance Plan to ensure that the Data Quality Objectives of the project are met.



- 9.2.4 The comments attached to the analytical reports that outline any corrective actions also describes the non-conformances of the data and how it affects the final reduced data for the information of the QAPPs' program managers.
- 9.2.5 Depending on the significance of nonconformance, the program manager is notified by the Chemists and work recalled if necessary. The program manager must be informed immediately for possible re-sampling.
- 9.2.6 Where the identification of nonconformance or departure casts doubts on the laboratory's compliance with its own policies and procedures, or on its compliance with this Standard, the QAO shall ensure that the appropriate areas of activity are audited internally first and then externally if necessary. A CAR should be generated as a memo and QAO informed.
- 9.3 Monitoring Corrective Actions
- 9.3.1 Monitoring of corrective actions are necessary to determine whether they are effective in removing the problem.
- 9.3.2 The Quality Assurance Officer (QAO) does the monitoring of all corrective actions that are documented as a CAR for trends and notifies the analyst of any preventive actions to assure that the problem does not occur again.
- 9.4 Preventive Measures
- 9.4.1 Preventive action, rather than corrective action, aims at minimizing or eliminating inferior data quality or other non-conformance through scheduled maintenance and review, before the non-conformance occurs.
- 9.4.2 Preventive action includes, but is not limited to, review of QC data to identify quality trends, regularly scheduled staff quality meetings, annual budget reviews, annual managerial reviews, scheduled column trimming, running a new LIMS in tandem with the old system to assure at least one working system, and other actions taken to prevent problems.
- 9.5 Control Charts

Control Charts are used by the QAO to determine trends. If there are any out of control data output, corrective actions are immediately implemented to make the corrections. Control charts are generated by the LIMS or any manual computer spreadsheet file created by the needs of a QAPP. This is a tool that the QAO or the program manager may need to assure quality of data. The charts also provides an overview during audits and management review to implement systemic corrective actions or preventive measures to assure problems do not reoccur.



Table 9.1 Example Summary of Corrective Action Procedures

Method No.	Analysis Method	Control Limits	Acceptance Criteria	Corrective Action
SM2540 C	Total Dissolved Solids	Lab duplicate	For samples with TDS $\geq$ 50 mg/L RPD should be $\leq$ 20% For samples with TDS < 50 mg/L the absolute difference between duplicates should < MRL	If this exceeded, reanalyze sample and duplicate once if this exceeds again or if it is not possible to reanalyze flag all associated samples
		Reproducibility of weighing after drying	Weights should be < 4% or 0.5mg difference	Weigh until criteria is reached.
SM2540 D	Total Suspended Solids	Balance Check with weight set	Expected Value within 0.01% of balance	Recalibrate the balance
		Laboratory Reagent Blank	< MRL which is 5 mg/L	If MRL is exceeded, find root cause
		Lab duplicate	For samples with TDS $\geq$ 50 mg/L RPD should be $\leq$ 20% For samples with TDS < 50 mg/L the absolute difference between duplicates should < MRL	If this exceeded, reanalyze sample ad duplicate once if this exceeds again or id it is not possible to reanalyze flag all associated samples
		Reproducibility of weighing after drying	Weights should be < 4% or 0.5mg difference	Weigh until criteria is reached.
SM 4500 H <sup>+</sup> B/EPA 150.1 (DW Only)	pH	3 buffers (4,7,10)	within $\pm$ 0.1 pH unit of true value	Re-calibrate instrument
		Duplicates	RPD < than control limits	Re-prep duplicates and reanalyze or flag if reported



Method No.	Analysis Method	Control Limits	Acceptance Criteria	Corrective Action
		Laboratory control samples (LFB)	% R within control limits of the method	Re-analyze batch
SM2540 C	Total Dissolved Solids	Balance Check with weight set	Expected Value within 0.01% of balance	Recalibrate the balance
		Laboratory Reagent Blank	< MRL which is 5 mg/L	If MRL is exceeded, find root cause
Different methods (see specific SOP as there may be slight differences in each method)	FIA methods	Calibration curve	$r \geq 0.995$	Rerun calibration
		Continuing calibration Verification,/LCS/LFB	90-110% recovery	Recalibrate and rerun last batch affected
		Spiked samples/LFM	80-120% recovery	If LFB in control, no action taken flag results as probably matrix bias
		MRL Check (sometimes called Quantitation Limit (QL) check	50-150%	MRL check high, flag data. Ok to report high biased ND data. Out low adjust MRL or repeat test.
		Method Blank	< Minimum Detection Limit (MDL)	Identify and eliminate source of problem. Do not do further sample analysis until contamination problem is resolved. Repeat sample prep using another source of reagent if contamination is found to be due to the Reagents used.

Note: The above table shows an example of corrective action plans and may serve as a guideline. Detailed, Quality Control checks, Control limits, acceptance criteria and corrective actions are found in each method SOP.

#### 10.0 Performance and System Audits/ Management Review

The QAO is responsible for the ongoing program of internal system audits and performance evaluation samples, and for coordinating all external audits and PT samples. In addition, the QAO with the Chemists are responsible for maintaining state and agency certifications.

#### 10.1 Internal Audits



10.1.1 The audits are carried out by the QAO or designee(s) who is independent of the activity to be audited. These audits are conducted solely for the determination that the method being utilized by the lab is updated and serves as a review of all quality documents being utilized by the laboratory. It also serve as a means of continuous improvement of the laboratory's quality system.

10.1.2 It is also utilized to determine if any errors were made in any analytical data that may require corrective action either systemic or applied.

10.1.3 Internal audits are conducted at least annually by the QAO.

## 10.2 External Audits

External audits are conducted by the US EPA Region 9 team. It may in the future, at the initiative of the Guam EPA Administrator, utilize the National Environmental Laboratory Accreditation Program (NELAP) of The NELAC Institute (TNI) as an external auditing organization in the future.

## 10.3 Performance Audits

Proficiency Testing (PT) Samples are conducted to test the ability of the individual analysts in obtaining analytical data of acceptable quality. These samples serves as a measure of the analysts' ability to obtain analytical data of acceptable quality. Guam EPA laboratory analyzes internal PT samples as part of the Guam EPA laboratory's quality system, while external PT samples are analyzed as part of the certification and approval process for US EPA as well as for other organizations such as TNI.

### 10.3.1 Internal PT samples

Internal PT Program is conducted as part of the corrective action process for any PT reported as unacceptable and evaluated by the PT provider as "check for error" or did not pass the PT provider's warning limits. Internal PT samples are also provided as needed as part of the analyst's initial demonstration of capability and as part of training. The laboratory maintains a file of all blind PT samples for traceability of the true and reported values as well as results of individual analysts. PT samples after they have been evaluated in a PT study may also be used as a LFB in analytical runs.

Problem areas are reviewed as soon as they surface; the probable cause is determined as expeditiously as possible and corrective action implemented. If a severe problem with the analysis is evident, the analysis is halted until the cause is found and corrected.

### 10.3.2 External PT Samples



External Proficiency Testing (PT) samples are analyzed annually as part of the US EPA certification process. Blind PT samples are procured from US EPA Approved PT Providers that are accredited by the American Association for Laboratory Accreditation (A2LA).

### 10.3.3 Proficiency Testing Protocol

10.3.3.1 Internal PT samples are conducted by individual analysts. These samples are prepared by their peers and is done once every 3 years, or whenever there is a new test method being developed.

10.3.3.2 External PT samples are done annually by an analyst which is rotated annually among the Chemists. The external PT samples are part of the annual study being conducted by the PT provider who is A2LA accredited. These samples are purchased from the provider.

10.3.3.3 The External PT samples are handled according to the PT provider's instructions in preparing the samples for analysis and in reporting results.

10.3.3.4 Non acceptable PT results are handled in corrective action reports. The root cause determined, corrective action identified and implemented. An additional PT is analyzed. External PTs require reporting results and corrective action to US EPA or the external agency requiring the PT analyses.

## 10.4 System Audits and Management Review

In order to insure that the Quality Assurance program at the laboratory maintains a high profile, there are several mechanisms in place which insure that QA information is routinely conveyed to laboratory management. This includes corrective action reports, reports on internal and external PT samples, and verbal transmittal of QA information to the EMAS Division Administrator.

### 10.4.1 System Audits

System audits are performed by external agencies such as the US EPA or the TNI (a future possibility). In these audits they may find systemic weaknesses in the quality system that may require corrective or preventive actions. These findings are included in the annual QA/QC report by the QAO.

### 10.4.2 Management Review

The EMAS Division Administrator who is also the QA Officer prepares with the laboratory team an annual QA/QC report that is used for an annual review with US EPA region 9 program manager and the EMAS team which is called End of Year (EOY) report. This report is also reviewed by the Guam EPA Administrator and the



GEPA management team. The QA/QC report includes any CAR reports, PT performance reports, internal/ external audit reports. Trends are noted and any preventive actions or measures may be taken up and decisions to implement are made. Other matters are also taken up in this management review.

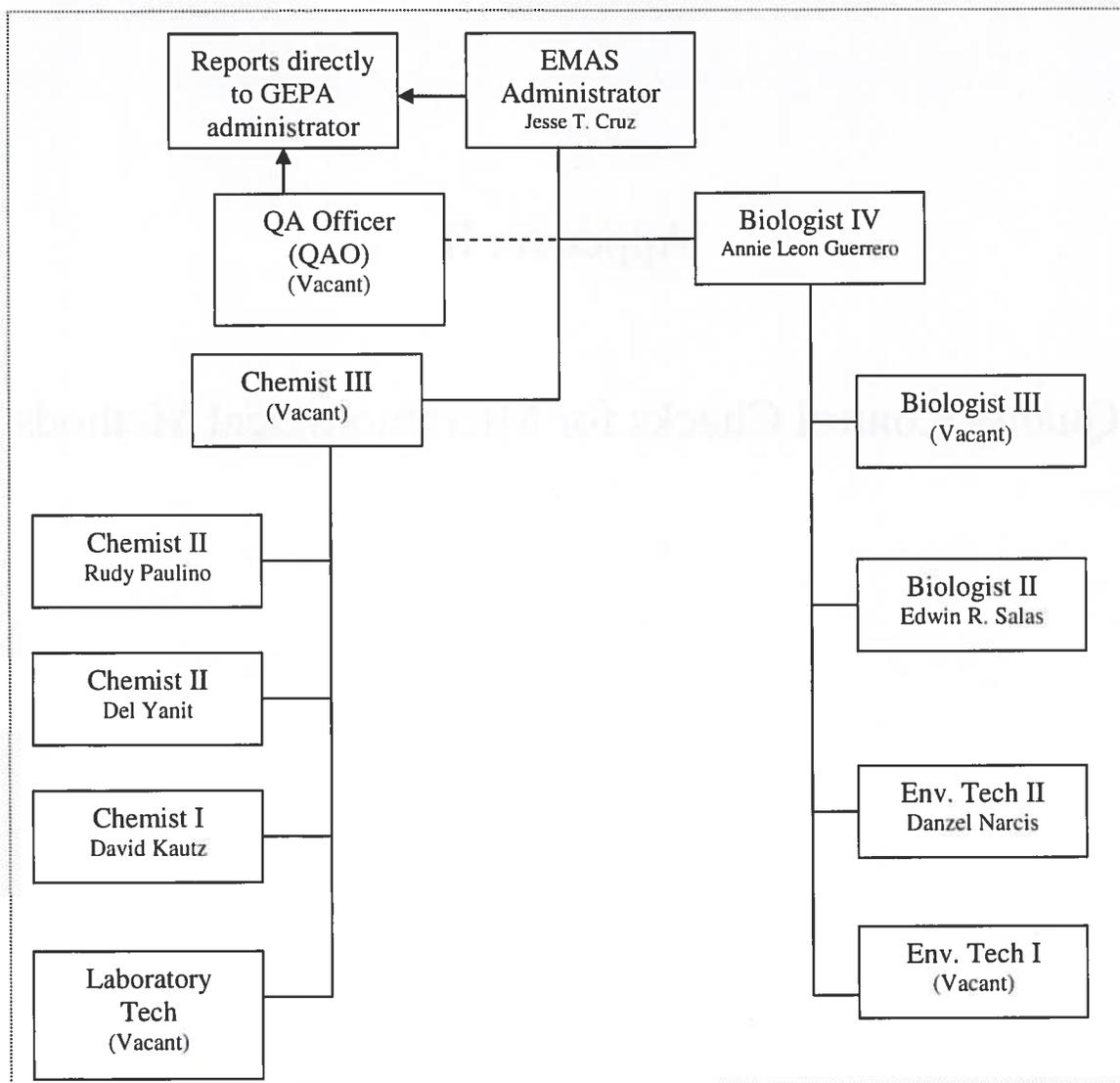
## 11.0 References

- 11.1 Eaton, Andrew D., L. S. Clesceri, and A.E. Greenberg, *20<sup>th</sup> Edition Standard Methods for the Examination of Water and Wastewater*, APHA, AWWA and WEF, 1995.
- 11.2 US EPA, *Manual for the Certification of Laboratories Analyzing -Criteria and Procedures Quality Assurance*, 5<sup>th</sup> edition, (EPA 815-R-05-004 Jan 2005).
- 11.3 US EPA QA/G-4, *Guidance for the Data Quality Objective Process* (EPA/600/R-96/055- Aug 2000).
- 11.4 MWH Laboratories, *Quality Manual*, 33<sup>rd</sup> edition, 3 Feb 2012.



### Appendix A

### Environmental Monitoring and Analytical Services (EMAS) Organizational Chart





Guam EPA Laboratory  
B-15-6101 Mariner Ave.  
Tiyan, Barrigada  
Guam 96921

Title:  
Number:  
Date:  
Rev. no.

QA Manual  
QAM  
04/08/2016  
001

## Appendix B

### Quality Control Checks for Microbiological Methods



## GUAM ENVIRONMENTAL PROTECTION AGENCY MICROBIOLOGY LABORATORY QUALITY CONTROL CHECKS FOR MICROBIOLOGY METHODS

### 1 Thermometers

- 1.1 Reference thermometers are recalibrated at least every five years by NIST, and calibration documentation is maintained with the reference thermometer in the thermometer kit.
- 1.2 Laboratory thermometers used to monitor incubators and refrigerators' temperature are recalibrated annually against a NIST- traceable reference thermometer, (or one that meets the requirements of NBS Monograph SP 250-23).
- 1.3 The calibration factor and the calibration date are indicated on the thermometer.
- 1.4 The following information is recorded in the Thermometer Annual Calibration Check Log (QC-01-01):
  - Serial number of the laboratory thermometer
  - Serial number of the NIST- traceable thermometer
  - Temperature of the laboratory thermometer
  - Temperature of the NIST-traceable thermometer
  - Correction (or calibration) factor
  - Date of check
  - Analyst's initial
- 1.5 The thermometer is discarded if it differs by more than 1°C from the reference thermometer.

### 2 Incubator Units

- 2.1 The calibration-corrected temperature is recorded for each thermometer being used at least twice per day during each day the incubator is in use. These readings are separated by at least four hours.
- 2.2 The date and time of reading, temperature and the analyst's initials are recorded in the temperature logbook for each incubator unit (QC-02-01/QC-02-02/QC-02-03).

### 3 Autoclave

- 3.1 A maximum-temperature-registering thermometer is used each time the autoclave is used to ensure that the proper temperature was reached.
- 3.2 The following information is recorded each time the autoclave is used (QC-03-01):
  - Date
  - Contents
  - Sterilization time



- Temperature
- Total cycle time
- Indicator Tape
- Analyst's initial

- 3.3 Spore strips or spore ampules are used monthly as bioindicators to confirm sterilization. This is documented in the Monthly Effectiveness Check Logbook for the autoclave (QC-03-02).
- 3.4 Automatic timing mechanisms are checked quarterly with a stopwatch, and the results are recorded and initialed in the Market Forge Sterilmatic Autoclave Quarterly Timer Check Logbook (QC-03-03).
  - 3.4.1 Elapsed time is recorded as the time when the indicator light turned on and turned off.
  - 3.4.2 Total cycle time is the time when the light turned on and the pressure gauge is down to zero psi.
  - 3.4.3 Note that the autoclave is set at SLOW exhaust selector when performing timer check.
  - 3.4.4 The required elapsed time and total cycle time are noted in the Quarterly Timer Check Logbook (QC-03-03).
- 3.5 Annual maintenance is conducted and recorded in the Market Forge Sterilmatic Autoclave Maintenance Logbook (GEN-02-01)
- 3.6 Copies of the service contracts are kept on file.

#### **4 Refrigerators**

- 4.1 Refrigerators maintain a constant temperature of 1 to 5 °C.
- 4.2 The calibrated-corrected temperature is recorded at least once per day for every refrigerator unit (QC-04-01/QC-04-02).
- 4.3 The refrigerator thermometer is graduated in at least 1°C increments and the thermometer bulb immersed in liquid.

#### **5 Glassware and Sample Containers**

- 5.1 Sterility check is performed on at least one sample container at random from each lot of IDEXX bottles by adding 25 ml of a sterile single-strength non selective broth (TSB), incubating at 35°C ±0.5°C, and checking for growth after 24 and 48 hours. These results are recorded in the Bottle/Quanti-Tray QC Logbook (QC-05-01).
  - 5.1.1 If growth is detected the entire lot should not be used.
- 5.2 Volume check is performed on at least one sample container at random from each lot of IDEXX bottles against a Class A 100-ml graduated cylinder. This is done by filling up the IDEXX bottle to the 100-ml line with water, and then transferred to a Class A 100-ml graduated cylinder. The volume should be accurate to within



- 100 ± 2.0 ml. This is documented in the Bottle/Quanti-Tray QC Logbook (QC-05-01).
- 5.3 Fluorescence check is performed on at least one bottle at random from each lot of IDEXX bottles by adding 100 ml of sterile water and then checked with a six watt 365-nm UV light . If it fluoresces, another lot of containers that does not fluoresce is used. This is recorded in the Bottle/Quanti-Tray QC Check Logbook (QC-05-01).
- 5.4 Fluorescence check is performed on at least one quanti-tray at random from each lot of IDEXX Quanti-trays by adding 100 ml sterile water, then sealed with IDEXX Quanti-Tray Sealer, and checked with a six watt 365-nm UV light. If it fluoresces, another lot of quanti-trays that does not fluoresce is used. This is recorded in the Bottle/Quanti-Tray QC Logbook (QC-05-01).
- 5.5 At least one quanti-tray from each lot is checked with 0.04% bromcresol purple to ensure they do not leak. This is done by adding several drops of 0.04% bromcresol purple to 100 ml distilled water, poured into a quanti-tray and sealed with IDEXX Quanti-Tray sealer (QC-05-01).
- 5.6 Quarterly pH check is performed on each lot of IDEXX bottles and glassware (i.e., graduated cylinders, test tubes, flasks) by adding few drops of 0.04% bromthymol blue(BTB). Bromthymol blue should be blue-green in the neutral range. If pH check fails, glassware rinsing procedure should be changed to ensure adequate rinsing. This is documented in the Quarterly Glassware pH Check Logbook (QC-05-02).

## 6 Media

- 6.1 Each lot of Colilert /Colilert-18 and Enterolert is checked for fluorescence before use. This is done by dissolving the medium into 100 ml sterile water filled IDEXX bottle and checked with a six watt 365-nm UV light.
- 6.2 Each new lot of Colilert/Colilert-18 media is checked before use for sterility and with positive and negative cultures controls.
- 6.2.1 To check for sterility, one ampoule of Colilert/Colilert-18 medium is dissolved in 100 ml sterile water filled IDEXX bottle, incubated at 35°C ±5°C and checked for growth after 24 hours. This sterility check is also called the laboratory blank that is being run concurrent with positive and negative culture controls.
- 6.2.2 To check with positive and negative culture controls, the expected results for various types of bacteria are as follows:

Organisms	Expected Results
<i>Escherichia coli</i> (fecal coliform)	Yellow, fluorescent
<i>Enterobacter aerogenes</i> (coliform, non-fecal)	Yellow, non-fluorescent
<i>Pseudomonas aeruginosa</i> (non-coliform)	Clear, non-fluorescent

- 6.2.3 Results of QC checks (6.2.1 and 6.2.2) are recorded in the Total Coliform and E. Coli in Drinking Water by Chromogenic Substrate Method Logbook (MB-02-01).



- 6.3 Each new lot of Enterolert media is checked before use for sterility and with positive and negative culture controls.
  - 6.3.1 To check for sterility, one ampule of Enterolert is dissolved in 100 ml sterile water filled IDEXX bottle, incubated at 41°C ±5°C and checked for growth after 24 hours. (see 6.2.1).
  - 6.3.2 To check with positive and negative culture controls, the expected results for various types of bacteria are as follows:

Organisms	Expected Results
<i>Enterococcus faecalis</i>	Fluorescent
<i>Streptococcus bovis</i> (Gram-positive)	Non-fluorescent
<i>Escherichia coli</i> (Gram-negative)	Non-fluorescent

- 6.3.3 Results of QC checks (6.3.1 and 6.3.2) are recorded in the GEPA Enterococci in Marine Waters by Chromogenic Substrate Method Logbook (MB-02-02).
- 6.4 Unused media are discarded appropriately by manufacturer’s printed expiration date.

**7 Reagent –Grade Water**

- 7.1 The quality of reagent water is tested and should meet the following criteria:

Parameters	Limits	Frequency
Conductivity	<2 micromhos/cm at 25°C	Monthly
Total Residual Chlorine	<0.1 mg/L	Monthly
Pb, Cd, Cr, Cu, Ni, Zn	Not > 0.05 mg/L per contaminant. Collectively not > 0.1 mg/L	Annually
Heterotrophic Plate Count	<500/mL CFU/mL	Monthly

**8 Dilution Water**

- 8.1 Monthly sterility check of dilution water is performed by adding 50 ml of sterile water to 50 ml double strength non-selective broth (TSB), incubating at 35°C ± 0.5°C, and checking for growth after 24 hours and 48 hours. These results are recorded in (QC-08-01).

**9 Glassware Washing**

- 9.1 Distilled or deionized water is used for final rinse.



- 9.2 Laboratory glassware is washed with a detergent designed for laboratory use. GEPA laboratory uses LIQUINOX<sup>®</sup> for washing.
- 9.3 A glassware inhibitory residue test (Standard Methods, Section 9020B, under Laboratory Supplies) is to be performed before initial use of a washing compound and whenever a different formulation of washing compound, or washing procedure, is used. This is to ensure that glassware is free of toxic residue. GEPA Lab has not changed its washing compound since it started using LIQUINOX<sup>®</sup>. An inhibitory residue test for LIQUINOX<sup>®</sup> was performed on its initial use and is recorded in Glassware Washing Compound Inhibitory Residue Test Logbook (QC-09-01).

## 10 Balances

- 10.1 Electronic balances are calibrated at least monthly using ASTM Class 1, 2, or 3 weights (minimum 3 traceable weights which bracket laboratory weighing needs, with a readability of 0.1g). Calibrations are recorded in the logbook for each balance with the date and initials of the individual performing the calibration (QC-10-01/QC-10-02/QC-10-03).
- 10.2 Non-reference weights are calibrated every six months with reference weights. Calibrations are recorded in a logbook with the date and initials of the individual performing the calibration (QC-10-04).
- 10.2 Reference weights are recertified every five years.
- 10.3 Maintenance, calibration, and cleaning is conducted annually by an approved calibration Laboratory. Service contracts and maintenance and calibration records are kept on file.

## 11 pH Meter

- 11.1 The pH meter is standardized at three points before each use period with pH 4.0, 7.0 and 10.0 standard buffers as recommended by the instrument's manufacturer. This is documented in the pH Electrometric Method Analytical Results Logbook (CH-02-01) with the date and initials of analyst.
- 11.2 pH buffer solutions are dated when received and when opened. The expiration date and the vendor's name are also recorded in the pH Buffers Inventory Log (GEN-01-03).
- 11.3 pH buffer solutions are discarded by the printed expiration date.

## 12 Conductivity Meter

- 12.1 The conductivity meter is calibrated at least monthly following the manufacturer's recommendation and using an appropriate certified and traceable low-level standard. This is recorded in the Conductivity Analytical Results Logbook (CH-02-02).



12.2 Alternatively, monthly conductivity cell calibration should be performed if the meter cannot be calibrated as noted in section 12.1. (Refer to SOP # CH-01-05, Conductivity by EPA 120.1, Attachment C, for conductivity cell calibration procedure).