Chapter 17 Quality Assurance

Lab management really boils down to quality assurance. Quality assurance is the program/plan designed by the laboratory that specifies the methods and procedures required to produce defensible data and acceptable precision and accuracy. Each person in the laboratory should be familiar with the laboratory's Quality Assurance (QA) plan but it is the lab manager who is primarily assigned the task of facilitating the plan and keeping it up to date.

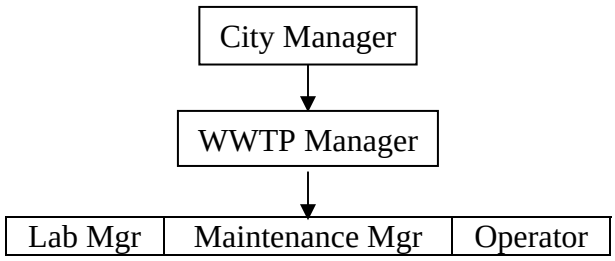
Laboratory QA Plan

The laboratory QA plan is a written document that includes:

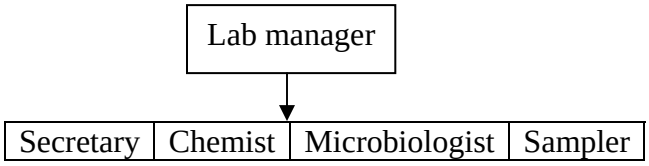
1. Laboratory organizational chart describing the function and responsibilities of each laboratory personnel and identifies the line of responsibility;
2. Field sampling procedures;
3. Laboratory sample handling procedures;
4. Standard Operating Procedures for each analytical method used;
5. Quality control procedures;
6. Data reduction, validation, reporting and verification;
7. Preventative maintenance procedures;
8. Corrective action contingencies; and
9. Recordkeeping procedures.

Organizational Chart

For most small systems, the organizational chart is pretty simple. The laboratory may be assigned to the WWTP manager.



As the system gets larger, the organizational chart will expand and the lines of communication become less clear.



Each position in the laboratory should have a well defined job description. Each person in the organizational chart should know the job duties of all the other personnel. A clear job description will minimize laboratory communication problems and provide an efficient movement of samples through the laboratory.

Training Procedures

The lab manager is responsible for assuring the personnel are performing the job duties correctly. This requires training to assure the technician is up to date on regulations and methodology. Cross-training is very valuable in the laboratory. Vacations, and illness can interfere with proper sample processing. The lab manager can set monthly, quarterly, or annual training goals for each employee as well as periodic performance evaluation samples. Each laboratory technician must demonstrate proficiency in the methods they perform.

Field Sampling Procedures

The lab manager is responsible for the Sampling Plan discussed in chapter 7. The sampling plan is a written description of:

- Sampling procedures;
- Sample locations;
- Safety concerns;
- Preservation;
- Types of containers;
- Cleaning and sterilization procedures;

- Holding times; and
- Shipping and storage procedures.

Sample Handling Procedures

The lab manager should prepare sample labels or forms that will contain the necessary collection information required by the laboratory. The QA plan should address how samples are checked in when they arrive at the laboratory. Questions that need to be addressed in the QA plan include:

- legibility;
- temperature;
- sample volume;
- type of container;
- preservation;
- holding times; and
- chain of custody for samples likely to be the basis for legal action.

Laboratory Sample Handling Procedures

Once the laboratory receives the sample, the QA plan should address how samples are processed within the laboratory from storage to analysis to disposal. Questions that need to be addressed in the QA plan include

- Legibility and laboratory notebooks;
- Storage temperatures;
- Isolation from laboratory contaminants;
- Specific sample rejection criteria;
- Holding times;
- Preservation; and
- Chain of custody.

Standard Operating Procedures

Standard operating procedures describe the analytical procedures used by the laboratory in very specific detail. SOPs should be in sufficient detail that a laboratory technician who is unfamiliar with the method can produce acceptable results. SOPs should describe:

1. Method title and referenced documents;
2. Scope of method and application;
3. Method summary;
4. Materials and equipment needed;
5. Health and safety precautions, including personal protective equipment;
6. Personnel qualifications;
7. Calibration or standardization methods;
8. Sample handling and preservation;
9. Detailed analytical procedure;
10. Calculations and data reduction;
11. Bench sheet and record handling; and
12. Quality control parameters, frequency, corrective actions and troubleshooting.

Quality Control Procedures

The laboratory QA manual should identify procedures used by the laboratory to demonstrate the laboratory data is valid and defensible. The QA manual will define control samples used and their frequency of use.

Field Control Blanks

Field control blanks are taken in the field during the sample collection process then taken through the entire analytical process. Field control blanks are usually laboratory pure water that is taken through the entire collection procedure. Field blanks can be used to verify the sampling equipment, the collection environment, the storage, transport, and sample preparation are not introducing errors to actual field samples.

Field Duplicates

Field duplicates are two separate samples collected at the same time and place under identical circumstances and treated exactly the same through field and laboratory procedures. Results of the duplicates give a measure of the precision associated with the collection and analytical processes.

Split Samples
Split samples are portions of the sample taken from the same sample container after thorough mixing or compositing. They are analyzed independently and the results used to document precision.

Negative Controls
Method blank (reagent blank) is a solution prepared and diluted with the same reagents and solvents (usually DI water) used in the samples. The method blank can be used to samples calibrate instruments. The method blank is used to provide a zero reading when running a calibration curve on an instrument. A method blank can also indicate if contamination from reagents interferes with the analysis. For instance, the unseeded blanks in the BOD test could be considered a method blank.

Microbiological negative controls are pure solutions of biological cultures prepared from ATCC control cultures which do not produce the desired growth. For example, Enterobacter Aerogenes is a negative control for fecal coliforms.

Positive Controls
Blank Spikes are prepared by adding known quantities of a standard to a volume of DI water. The concentration prepared should be typically mid-range for the method used. For example, a 1.0 mg/L spike could be prepared to verify a phosphate curve with a range of 0-3 mg/L.

Matrix Spikes
Matrix spikes are prepared by adding known quantities of a standard (blank spike) to a sample prior to analysis. This is also often called standard addition. For instance, a sample is analyzed and found to have a phosphate level of 1.6 mg/L. The 1.0 mgL blank spike could be added, the analysis repeated. The result would be expected to be 2.6 mg/L. The results of the matrix spike is reported as percent recovery. If the percent recovery is outside the established acceptable range, interferences with the method may be present.

Microbiological positive controls are pure solutions of biological cultures prepared from ATCC control cultures which produce the desired growth. For example, Enterobacter Aerogenes is a positive control for total coliforms.

Calibration Standards
Primary calibration standards are prepared from NIST traceable stock solutions which can be purchased or prepared in-house from reagent grade chemicals. These standards are used to calibrate instruments, prepare standard curves, and verify any other method of analysis including titration. Calibration standards should be used frequently to verify instrument calibration has been maintained.

Performance Evaluation Samples
Performance evaluation samples are samples that are generally purchased as a "blind sample" from an outside source. The sample is analyzed and the results are sent to the supplier for evaluation. These samples should be performed at least annually for each analytical method used.

Lab Duplicates
Lab duplicates are two portions taken from the same sample container that are processed and analyzed separately. For instance, running two TSS samples from the same container would be a lab duplicate. Duplicates are generally run on 5-10% of all samples.

Any quality control tests that are suspect, require the laboratory to evaluate the analytical process for errors. The errors

could be from the method used, the instrument used, or personal errors by the analyst. Error analysis will be discussed in another chapter. The QA manual should discuss what corrective actions the laboratory will take to remedy errors.

Data Reduction, Validation, Reporting and Verification Procedures

Data handling is the process of transforming the raw analytical numbers into reportable numbers. All written records are to be documented in permanent ink. Any errors during the analytical process should be corrected by drawing a single line through the error. The correct value is then entered beside the incorrect entry with the initials and date of the individual making the correction. The technician has the primary responsibility of assuring the data is correct and complete. The technician should verify quality controls are within the control range. If data is outside the acceptable control limits, the technician must either re-analyze the sample or invalidate. If the data is invalidated, it should not be reported. The QA plan should describe how invalidated samples are handled and should describe the procedures that are used to prevent the release of incorrect data. Large labs often have several analysts who are responsible for calculating and verifying data. The lab manager or his designee has the responsibility to review QC and bench sheet data prior to release. Small labs with only one technician do not have luxury so calculation and transcription errors are more likely. The QA plan can identify what corrective actions are to be performed when data cannot be validated. The laboratory manager or designee is responsible for the final verification and distribution of analytical results.

Preventative Maintenance Procedures

The lab manager is often responsible for setting up a preventative maintenance schedule. Instruments must be properly maintained to ensure they continue to work properly. The QA manual should describe preventative maintenance procedures needed to assure proper operation. Effective preventative maintenance will reduce down-time, poor performance, and "interruption of analysis". For instance, a DO meter that is constantly giving an error message may indicate the membrane needs to be replaced, the battery needs replaced, or the electrode is failing. Consumable materials such as batteries and membranes should be readily available. Replacing the expensive electrode could involve problems associated with budgeting, purchasing, or shipping delays that create a long "out of service" time. An effective lab manager must plan for replacement and repair of lab equipment. Routine maintenance performed by laboratory personnel should be recorded in a logbook for each major piece of equipment. The lab technician should document the problem and the repairs made.

Service contracts on major equipment should be documented.

Corrective action contingencies

The QA plan should describe how analytical sample results are handled when QC samples are unacceptable. Steps which should be taken in the corrective action process are:

- identify and define the problem
- assign responsibility for investigating the problem
- determine the cause of the problem
- determine actions needed to correct the problem
- implement the corrective action

- establish the corrective action is effective
- management verifies the corrective action effectiveness

Recordkeeping Procedures

Recordkeeping is often a weakness in laboratory management. The QA manual should describe recordkeeping for all aspects of the laboratory. Recordkeeping not only includes tracking sample and analytical information but also QC information, equipment maintenance, calibrations, reagent purchase/preparation, DMR reports, etc. Recordkeeping should cover the complete process:

Sample collection

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Sample analysis

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Data Verification

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Data Distribution

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Data Storage

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Data Disposal

After the data has been distributed, it must be stored for some time period. The QA plan should identify where the data will be stored, what form it will be stored in, and how long it will be stored. Most compliance/regulatory data must be stored for a minimum of 5 years and sometimes indefinitely (per state regulation). It can be stored as a hard copy or stored electronically. If it is stored electronically, the QA plan must identify where the copies will be stored, how many copies will be made, how long will the laboratory keep the copies before disposal, how the database be will secured?

The QA manual should address any differences in handling between process control samples, compliance samples and samples headed to court. The data for process control tests may not need to be as tightly documented as a compliance sample used for the DMR. Finally, facilities subject to legal action will need to have all laboratory information defensible. If samples are expected to be part of a legal action, chain of custody procedures will be needed.