NEBRASKA DEPARTMENT
OF HEALTH AND HUMAN SERVICES
PUBLIC HEALTH
ENVIRONMENTAL LABORATORY

QUALITY ASSURANCE PLAN

October 2014

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Annual Review Documentation

Revision # and Date	Laboratory Manager Review	DHHS Environmental Administrator Review	QA Manager Review and Approval	Comments and/or Changes
Revision 3 6/30/03	Dalton Johnson	Bob Leopold	Sandy Irons	Update Management Duties and titles
Revision 4 Dated June 2004 9-14-2004 Replacement	Dalton Johnson	Bob Leopold	Sandy Irons	Updated Appendices D, L1, L2, N, & Q, Made grammar & spelling corrections, added data quality objective section
Revision 4 Dated June 2004 11-08-2004 Replacement 11-14-2005	Dalton Johnson	Bob Leopold	Sandy Irons	Updated Staff charts & duties due to new staff hired in October & November
Revision 6 Dated March 2007	Dalton Johnson	Bob Leopold	Sandy Irons	Updated staff charts & duties due to new staff hired, update equipment , and other Appendix's D, L1, L2, N, & Q
Revision 7 Dated November 2007	Dalton Johnson	Bob Leopold	Sandy Irons	Updated QA plan as required by EPA during their on-site audit
Revision 8 Dated May 2008	Mary Boden	Sue Semerena	Sandy Irons	Update laboratory name change through out document and organization and staff changes.
Revision 8 Dated May 2008	Mary Boden	Sue Semerena	Sandy Irons	Updated Staffing Attachments as of February 10, 2009
Revision 9 Dated July 2010	Mary Boden 04-14-11	Sue Semerena	Sandy Irons 02-12-2011	Add comments to clarify 2010 Audit response
Revision 10 Dated January 2012	Mary Boden 01-31-12	Sue Semerena	Sandy Irons 01-21-2012	Removed last sentence second paragraph Page 55, Section 12 QC Tracking. Updated Appendix A, C, D, L1, L2, Q, T
August 2013	Mary Boden 08-20-2013	Sue Semerena 08-20-2013	Laurie Wieting 08-20-2013	Updated Management Titles and duties, updated Appendices to reflect staff, ancillary and SOP Table of Content changes. Made changes to the content and forms.
October 2014	Mary Boden 10-30-2014	Sue Semerena 11-20-2014	Laurie Wieting 10-31-2014	Made changes requested by EPA 2013 audit report Attachment E. Add Linearity studies
November 2015	Mary Boden	Sue Semerena	Laurie Wieting	No changes made except ongoing equipment updates
December 2017	Mary Boden	Sue Semerena	Laurie Wieting	No changes made except ongoing staff and equipment updates

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Mission Statement & Operating Principles

Mission: ***"We help people live better lives through effective health and human services."***

Operating Principles:

There are five Cs, which serve as the NPHE Lab System's operating principles:

Communication

What we mean: Communication means keeping people informed; listening actively; being open and accessible; and ensuring we are accurate, timely, and complete in all we say and write.

The results we want: Our customers, the people we work with and the public, see us as open and honest in our communication, believe that we hear and understand what they say, and view us as a source of valid and reliable information that is easily accessible.

Cooperation

What we mean: Cooperation means a willingness to work with others in good faith; assisting them and accepting assistance from them.

The results we want: Our customers and the people we work with join us in seeking solutions and improvements.

Collaboration

What we mean: Collaboration means a willingness and ability to work together with others as equals in the pursuit of common goals.

The results we want: Our customers and the people we work with trust that we will work with them as partners in the pursuit of common goals.

Customer Service

What we mean: Customer Service means responding to our customers in a respectful, timely, and effective manner.

The results we want: Our customers know we value them and are considerate of their needs.

Confidence

What we mean: Confidence means reliance on us to do our jobs effectively and efficiently with integrity and fairness.

The results we want: Our customers, the people we work with and the public, learn that we do our jobs with commitment, professionalism, efficiency, and through accountable programs and accurate systems.

"Helping people live better lives."

1. Quality Assurance Policy

The Nebraska Department of Health and Human Services Public Health Environmental Lab (NPHE Lab), a part of the Nebraska Department of Health and Human Services Environmental Health Unit, provides analytical chemistry and microbiological services to clients concerned with the public's health in compliance with the Safe Drinking Water Standards. The NPHE Lab's goal is to meet and exceed the needs of our clients by providing quality services that set the standard for excellence in the environmental testing industry. The NPHE Lab will continue to maintain a Quality Assurance System that will satisfy all of the elements in the NPHE Lab Quality Policy as follows:

- Meet current local, state, and federal drinking water regulations
- Comply with EPA standards
- Define laboratory analytical capabilities within the division and for all clients
- Ensure that client needs are understood and can be met
- Provide analytical and microbiological data that is valid, defensible, and of known quality
- Maintain a workplace atmosphere that ensures conformance to the NPHE Lab Code of Ethics

The NPHE Lab is committed to performing analyses using sound professional practice. In all aspects of the laboratory, quality is the highest priority. All NPHE Lab personnel are required to read and understand this Quality Assurance Plan (QAP), and its supporting documents, and to adhere to the documented procedures and policies. Each staff member will be required to sign documentation stating that they have read this current QA plan and that they agree to follow this QA plan. The written documentation will be added to the training file for each lab staff, which is located in the QA office.

The NPHE Lab maintains an organizational environment that fosters individual motivation, innovation, and continuous improvement of our processes, systems, and structure to achieve division goals and meet the needs of our clients.

1.1. Ethics Policy

The Nebraska Department of Health and Human Services Public Health Environmental Laboratory is committed to conformance with ethical standards, ensuring the integrity of data which it provides to its clients, meeting the quality needs of our clients, and providing our employees with guidelines and an understanding of their ethical, legal, and quality responsibilities in the performance of their work. Analysts are instructed not to compromise the quality of results over production or personal interest. All employees are instructed to consult with the Unit Administrator, the Laboratory Manager, Quality Assurance Manager (Laboratory Assistant Manager), or Human Resources Manager when situations arise which may bring into question adherence to ethical issues, proper quality assurance procedures, analytical techniques and reporting of test results.

The NPHE Lab Laboratory Manager or QA Manager presents and reviews the NPHE Lab Code of Ethics with all new employees. Management also conducts annual training of its Code of Ethics with each employee, and documents their understanding and agreement to follow the code. The Code of Ethics is also reviewed periodically at NPHE Lab staff

meetings. Each NPHE Lab employee must sign a Code of Ethics form. The original signed documentation is retained on file by the Human Resources Department in the employee's personnel file. A copy is placed in the employee's training file and a second copy is given to the employee for their own reference. *The current Code of Ethics form can be found in Appendix A.*

1.2. Quality Assurance and Quality Control

Quality is the level of excellence needed to meet an established standard. Generally, quality refers to the superiority of end results and/or the excellence of performance required in achieving the established standards.

Quality Assurance (QA) is defined as those planned and systematic activities necessary to provide sufficient confidence to the NPHE Lab, its clients, and regulatory agencies that NPHE Lab's data and services meet or exceed mutually accepted quality standards consistent with the analytical methods which are used and with client and regulatory agency quality standards.

Quality Control (QC) is defined as the technical activities which are employed to quantitatively measure and evaluate the success of operating practices, procedures, and services against standards of performance established to meet standards of quality and the needs of the customer. Performance criteria are defined for all areas of the laboratory. These include:

- Administrative and technical policies, methods, and procedures
- Personnel accountability, authority, and responsibilities
- Performance monitoring
- Peer and supervisory review, checks, and approvals

This Quality Assurance Plan describes the NPHE Lab organization, structure and departmental QA/QC responsibilities. It identifies the Quality Assurance (QA) systems, policies, and activities performed to ensure that the NPHE Lab meets its Mission and QA objectives. This manual addresses the systems that monitor the QA activities for the purpose of achieving continuous improvement and identifies the QC records that are maintained and the reports that are generated.

The following documents have been used as guidance for writing and revising this manual:

- Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth Edition, January 2005
- NELAC Quality System Document, Approved July 12, 2002 Effective July 1, 2004
- Nebraska Title 179, Public Water Systems, Chapter 20, Laboratory Certification Requirements for Testing Drinking Water in Nebraska
- EPA Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA–600/4-79-019, March 1979
- EPA Methods for Determination of Organic Compounds in Drinking Water, 1991 and appropriate Method revisions

- EPA Methods for Chemical Analysis of Water and Wastes, March 1983, and appropriate Method revisions
- EPA Guidance in Assessing Quality Systems, EPA QA/G-3, March 2003
- EPA Guidance for Preparing Standard Operating Procedures, EPA/G6, March 2001
- EPA's Good Automated Laboratory Practices Manual, 1995
- EPA 40 CFR Parts 136-149
- Guidance on Environmental Data Verification and Data Validation. EPA/240/R-02/004 November 2002
- Standard Methods for the Examination of Water & Wastewater, 21st Edition, 2005 and 22nd Edition, 2012
- Guidance on Technical Audits and Related Assessments for Environmental Data Operation, EPA/600/R-99/080 January 2000

The NPHE Lab Quality Assurance Manager is responsible for writing this document. It is reviewed at least annually by the QA Manager and Laboratory Manager and revised as necessary. It must be approved by the NDHHS Environmental Health Unit Administrator and the Laboratory Manager. The review process is meant to evaluate the manual's ability to meet the NPHE Lab's Quality Objectives, address any process changes and meet new Federal and State regulatory requirements. This review and approval is documented by the approval signature of the NDHHS Environmental Health Unit Administrator, the Laboratory Manager, and the Quality Assurance Manager. All employees are required to read this document annually to ensure that they understand their ethical and quality assurance responsibilities. The effectiveness of the NPHE Lab Quality Assurance program depends on the adherence to these policies and procedures by the entire laboratory organization.

Employees of the NPHE Lab, permanent, temporary or contract, must adhere to ethical and legal standards, abide by the law, preserve the Lab's integrity and reputation, and preserve the confidentiality of its clients. Failure to adhere to this policy will result in disciplinary action, up to and including discharge from employment.

1.4. Certifications and Reference Methods

The NPHE Lab maintains EPA Drinking Water certification. Our current certificate lists the methods and/or analytes that the NPHE Lab is certified to perform. The NPHE Lab Fee Schedule lists most of the analytes and methods that are performed at the NPHE Lab including all the methods and/or analytes for which the lab holds a current EPA certification.

1.5. Customer Confidentiality Rights

All information received from NPHE Lab customers or potential customers and data generated for these customers are to be considered confidential by all Laboratory employees. Laboratory reports and accompanying documents contain confidential information intended for use by the individual or entity requesting the analytical service. Except when required by law, no information relating to a report is released to another person or party without written permission unless the information is public information.

Since the laboratory has state agencies and programs as clients, the majority of the data produced for these clients are considered public record. However, the data will be released to the primary data user initially so that he/she can perform data quality assessment before results are released to the public.

Analyses completed for private citizens will be considered confidential at all times. Private sample results are not routinely given out over the phone or fax unless the customer has made prior arrangements with the laboratory. The Laboratory Manager, the QA Manager and/or other designated staff in emergencies can release results, but great care is taken to ensure that the appropriate customer has granted approval for the release of the data.

Permission to release information to other entities must be in writing (Fax, e-mail PDF or letter). Faxes clearly must identify the intended recipient. The fax coversheet should contain a statement at the bottom of the sheet stating that the contents of the fax are confidential and intended for a specific recipient. It should also instruct the recipient to contact the laboratory if the information was received in error.

2. Organization and Responsibilities

It is the objective of the NPHE Lab to provide an organizational structure that enables its employees to achieve its quality goals and meet the requirements of its clients. Organizational support to accomplish the quality goals is derived from NPHE Lab Public Health Directives and Standard Operating Procedures.

The QA Manager oversees the quality achievement and quality verification processes through observations, internal/external audits, spot checks, data and process reviews. The individuals directing, managing, conducting, reviewing, monitoring, and approving the laboratory data are individually and collectively accountable for the quality of the data. The leadership and support of the NPHE Lab's Management ensures that lab practices and policies are effectively implemented. The Laboratory Manager provides administrative expertise, as well as technical guidance and support to staff and the QA Manager to ensure that QA issues are given appropriate attention and addressed in a timely fashion.

The NPHE Lab is divided into specialized testing work groups. These work groups include Organics, Inorganics, Microbiology, Metals, Air, Blood Alcohol, and Customer Service. Each work group consists of multiple analysts. Many analysts are members of two or more work groups. Work group members provide technical expertise, secondary data review, and analytical support to the rest of the testing group. Team members from each work group make up the members of the NPHE Lab's QA team and Safety team. These Laboratory teams provide valuable expertise, support, and guidance to Lab Management relating to specific QA, customer service, safety, technical and/or analytical needs of the laboratory as a whole.

Organizational charts for the NDHHS System, including the NPHE Lab, are available in Appendix B-D. Job descriptions for NPHE Lab personnel are maintained in the Human Resources Department and a brief summary of the positions and classifications can be found in *Appendix E-L.*

2.1. QA/QC Responsibilities

In general, each employee affects the quality of service provided by the NPHE Lab and is obligated to perform specific documented procedures in a professional and ethical manner and to document all discrepancies that may arise. All personnel must understand their responsibilities in the QA effort as described in this Manual and acknowledge their understanding by signing required training forms.

2.1.1. Policy and Procedures Departures

All NPHE Lab employees are required to adhere to all policies and procedures. Should it be necessary to depart from the requirements of a Standard Operating Procedure, the analyst must first receive approval from the appropriate manager to make the departure. Second, the analyst must clearly document the nature of the departure and the justification for the departure.

2.2. NDHHS Environmental Health Unit Administrator

The NDHHS Environmental Health Unit Administrator has the overall responsibility for approving all NPHE Lab policies and supporting the quality activities described in this QA Manual. He/She provides direction in establishing the NPHE Lab's Quality Mission and ensuring that the NPHE Lab's QA Manager and Laboratory Manager understand their responsibilities for maintaining the NPHE Lab's conformance to its quality policies.

2.3. Quality Assurance Manager

The Quality Assurance Manager is responsible for establishing, administering, monitoring, and improving the QA program and all the QA Systems which comprise the program. He/she has primary reporting responsibility to the NDHHS Environmental Health Unit Administrator but works closely with the Lab Manager to cover lab administrative and technical duties when the Lab Manager is out of the building. The Quality Assurance Manager has the following specific responsibilities and authorities:

- Supervision of the Quality Assurance activities of the NPHE Lab
- Identify, recommend or provide solutions to problems affecting quality
- Identify the need for corrective actions affecting quality, initiate corrective action and notify appropriate personnel of requirements for corrective actions, and verify and document implementation of needed corrective action
- Review and approve all Quality Assurance reports, and/or assign the responsibility to additional staff in their absence
- Notify the Laboratory Manager and NDHHS Environmental Health Unit Administrator (when appropriate) of deficiencies in the quality program
- Orient new personnel to the Quality Program
- Perform or schedule and provide support to QA team members to complete internal audits of all technical and non-technical laboratory operations and provide verbal and/or

written reports regarding the outcomes of those audits to the Laboratory Manager and NDHHS Environmental Health Unit Administrator
- Maintain current status of all certifications
- Assure that, when necessary, future work is stopped or controlled until proper resolution has been achieved for a nonconformance, deficiency, or another unacceptable quality issue
- Review and approve sub-contractor laboratory submissions
- Monitor QA NPHE Lab activities on a routine basis, through spot checks, blind sampling audits, QC chart inspections, data review checks, and other similar activities as deemed necessary to ensure continued compliance to QA objectives
- Prepare QA reports as required by NPHE Lab SOPs and this QA Manual
- Guide the activities of the NPHE Lab QA Team
- Act as State of Nebraska Drinking Water Certification Officer, providing guidance, support, and expertise to in-state and out state laboratories requesting Nebraska Drinking Water Certification status
- Oversee Lab equipment inventory
- Oversee Lab monthly invoicing and statement processing
- Oversee the BSL III Laboratory operation
- Oversee the Blood Alcohol testing area of the Laboratory

2.3.1 Management Review

At least annually the Quality Assurance Manager reviews the state of the quality of the Laboratory QA system, the corrective actions taken over the last year, the results of PT studies, and other QA/QC issues of importance. Laboratory Management will discuss any problems found and use the information gathered to determine future QA system priorities.

2.4 Laboratory Manager

The NPHE Lab Laboratory Manager has technical and administrative responsibility for the laboratory and its operations. He/she reports directly to the NDHHS Environmental Health Unit Administrator but works closely with the Quality Assurance Manager (Assistant Lab Manager) to cover lab QA activities and duties when the QA Manager is out of the building. They have the responsibility and authority to ensure conformance to all NPHE Lab policies and procedures so that all data produced by the NPHE Lab is technically valid and conform to all NPHE Lab and client quality requirements. He/she is responsible for addressing laboratory capacity issues, productivity goals, and due dates within the operations of the Lab. The Laboratory Manager is responsible for the technical aspects of the operations of the laboratory. Specifically the responsibilities include:
- Responsibility for the technical performance of the laboratory
- Responsibility for work schedules
- Responsibility for capacity issues, productivity goals, and due dates within the operations of the Lab

- Responsibility for the technical aspects of operations including standard operating procedures
- Responsibility for ensuring that method requirements are met
- Responsibility for assisting analysts to address quality control issues
- Responsibility for maintaining certification compliance facilitating the implementation of all corrective actions
- Responsibility for plans for technology improvement within the Lab Operations
- Defining the qualifications required for technical personnel
- Certifying that all technical personnel have the appropriate education and experience to execute their job responsibilities
- Gathering technical requirements, writing and working with purchasing to prepare Requests for Bid of Proposal (RFP) (as necessary)
- Ensuring that appropriate policies and procedures are implemented so that NPHE Lab produces valid data which conforms with applicable quality requirements
- Designate appropriate test area staffing and define their job responsibilities
- Ensuring that all laboratory personnel are trained in and follow the Chemical Hygiene Plan and other safety policies, ensure that all staff use appropriate safety equipment and procedures at all time, and that sample disposal follows State and Federal guidelines
- Ensuring that there is appropriate training and backup to cover the work processes of each laboratory area
- Provide technical support and advice to NDHHS staff, DHHS Management, DEQ Program Managers, NDHHS Environmental Health, Drinking Water program and clients as requested.
- Developing and updating an annual laboratory fee database and maintaining that fee structure
- Provide technical support for the Laboratory Information Management System (LIMS)
- Review and approve sub-contractor laboratory submissions
- Review of Client Contracts (as necessary)

2.5 Finance Manager

The Finance Manager provides support for various financial and security issues at the laboratory, including purchasing, record management, building space and security. These duties are routinely divided between the Laboratory Manager and QA Manager but can be handled by either as time permits. Specific responsibilities of the Finance Manager include, but are not limited to:

- Preparing budgets, fees and financial reports
- Interacting with staff and state purchasing to meet supply and equipment needs
- Preparing and monitoring equipment maintenance contracts
- Entering required contract information into the Nebraska Account System (NIS)
- Reviewing and approving Accounts Payable & Accounts Receivable documents
- Obtaining and providing information to the NDHHS Environmental Health Unit Administrator regarding clients' payment records, and contracts
- Reviewing client contracts, as necessary

- Overseeing compliance to State and Federal records management requirements and policies
- Overseeing building space and security issues as they arise
- Oversee records management

2.6. Analytical Team Members

The analytical team members report to the Laboratory Manager, except for the Blood Alcohol team members who report to the QA Manager. The analytical team members are responsible for implementation of NPHE Lab policies and procedures that ensure only data of the highest attainable quality is produced, that all quality control procedures are followed and that all tasks performed in the Laboratory are conducted in accordance with this manual and applicable SOPs. They have authority commensurate with their responsibilities for the day to day management and monitoring of laboratory activities in their analytical area.

The analysts are responsible for scheduling their own work, performing analysis, reducing the data, and performing a PREV of the data for conformance to QC requirements. They are responsible for recording any pertinent observations and reporting any QC failures to the Laboratory Manager and/or Quality Assurance Manager. They are responsible for understanding and correctly adhering to all applicable SOPs and Quality Control Procedures. The analysts are also responsible for performing QA peer review of data packages and/or electronic LIMS data produced by fellow analytical staff members prior to the data being reported to the customer.

Analytical Team Members have the following specific responsibilities and authorities for their designated areas:

- Schedule work to meet established turnaround times and sample holding times
- Designate specific personnel to act as backup for personnel who are absent
- Train alternate staff to perform the assignments and be responsible for the duties of the absent staff member
- Provide adequate personnel training, close monitoring, and adequate support for assigned analyses to new team members
- Maintain equipment and instrumentation in proper working order and document the process in a organized manner
- Ensure proper continuous calibration of laboratory equipment and instrumentation
- Review analytical data for conformance to all NPHE Lab QC and client requirements
- Initiate and/or implement and document all necessary corrective actions in an appropriate and timely manner
- Ensure that an Initial Demonstration of Capability (IDC), MDL, and blind audit sample is completed prior to testing and reporting results of compliance samples in conformance with method requirements and specific Laboratory SOPs
- Ensure that annual Method Detection Limit (MDL) Studies are completed on time and in conformance with method requirements and specific SOPs, when applicable
- Provide or review comments regarding data for which there has been a QC failure both which is judged usable and reportable or non-useable to the client

- Write, review, and update SOPs applicable to their primary analytical testing area at least once a year or more often if needed or if changes in equipment, methods, or procedures occur
- Ensure method meets EPA and Federal requirements for Federal programs
- Follow the NPHE Lab Chemical Hygiene Plan and any other Lab Safety policies
- Follow the NPHE Lab QA plan, related SOPS, and NPHE Lab personnel policies as written

3. Personnel Qualifications and Training

All personnel must meet the minimum education and experience requirements defined for their assigned responsibilities. State education classification requirements for each position, is used as a guideline. Expected job duties dictate part of those minimum qualifications. Experience in a given area of analysis also plays a part in the education process. During the employee recruitment process, the education and experience of all personnel selected for employment is verified prior to an employment offer. This verification includes employment reference checks and confirmation of college and advance degrees. This task is performed by the DHHS Human Resources Department.

3.1 Training System

All new personnel are given an orientation during their first few weeks of employment. This includes review of NPHE Lab Policies and Procedures, CHP and initial training in the use of the computer network and Laboratory Information Management System (LIMS). Additionally, they are introduced to NPHE Lab's Quality Program and Safety Procedures. All personnel receive training in the analytical procedures and operating practices which are specific to their job responsibilities. This training is directed either by the Laboratory Manager, the QA Manager or members of the analytical testing area team who are experienced in the applicable procedures.

Analysts must complete specified training and performance requirements before they are permitted to analyze samples for clients without direct supervision. Job training requirements are documented by utilizing the departmental training checklist. Training checklists and any documentation required to comply with the training checklists are signed by the trainer and trainee and filed in the employee's training file in the QA Office along with other internal and external training documents. Other training documents include Initial Demonstration of Capability Certification Statements, resumes, diplomas, and PT results. A variety of equipment manufacturer seminars, monthly laboratory staff meetings, quarterly DHHS Environmental drinking water department meetings, and semi-annual State water operator meetings, provide information and help update staff on regulations changes and/or methodology. Records of all external or internal training or continuing education, as well as all other miscellaneous training are maintained in the employee's file in the QA Office. Additional information regarding training and staffing qualifications can be found in the current NPHE Lab *SOP # 1100.1, Training and Qualifications.*

3.2 Initial Demonstration of Capability

Analysts, as part of their training, must successfully complete an initial demonstration of capability (IDC) study as described in each analytical SOP. At a minimum, the analysts must demonstrate that they can meet the accuracy and precision requirements of the method (if appropriate to the method) by analyzing replicate samples, which have been fortified with the analyte(s) of interest, at the appropriate level. The number of replicates performed is usually listed in the appropriate individual SOP. A new IDC must be completed when there is a change in the method, analyst, or equipment. Additional instructions for performing IDCs can be found in the current version of *SOP 8200.1, Documenting Data Quality.*

Some analytical tests, e.g., color, temperature, and pH do not allow for the creation of initial demonstration of capability samples, by spiking reagent water. In these cases, an experienced analyst should analyze a series of samples with a range of results. The analyst in training then should perform the procedure on the same samples analyzed by the experienced analyst and the QA Manager should review the results. Based on agreement of results, the QA Manager documents the acceptability of the IDC for the new analyst.

Some reference methods require an MDL study to be performed by each analyst. See SOP 1100.1 Training and Qualifications, Attachment 6 and Attachment 7 for MDL and IDC requirements and documentation forms. The documentation for all IDCs is also shown in *Appendix M1 of the QAP.* The Quality Assurance Manager or the analytical section maintains the IDC record and a copy is placed in the staff training file.

3.3 Continuing Demonstration of Capability

The primary analyst for each method must continue to demonstrate their capability at least annually by successfully fulfilling both of the following criteria:

- Performing a MDL study or successfully analyzing at least 4 samples previously analyzed by an experienced analyst. The later criterion only applies to analytes, which cannot be readily spiked into reagent water (e.g., color, temperature, and pH).
- Acceptable performance on at least one PT Sample for each analyte by each method in any 12 month period. The primary analyst should perform the first round of PT samples in the Spring. They must pass the PT or complete a quick response PT and pass it.

Each secondary analyst for each method should maintain proficiency by:

- Performing and passing a PT in the fall if the primary analyst has already passed a PT in the Spring. (In methods that are covered by many analysts PTs may need to be split or rotated over several years to ensure a PT can be passed by each analyst) Blind samples may be prepared to verify continuing demonstration of capability in some situations.

3.4 Initial Demonstration of Capability Exclusions

Not all laboratory procedures require performing an IDC. Some analysts or technicians may be exempt from IDC requirements at the discretion of the QA Manager. Analysts are allowed to perform certain procedures without performing a complete IDC. However,

anyone who performs any of these procedures must receive proper training. Some of these procedures are listed below:

- Simple volumetric measurements
- Gravimetric measurements
- Non-critical filtration
- Date entry
- Loading autosamplers with samples
- Spiking samples
- Preparing standards
- Assisting the primary analyst in sample preparation steps.

If there is a question regarding the need for IDC documentation the analyst should consult with the QA Manager or the Laboratory Manager for guidance.

4. Facilities and Equipment

The NPHE Lab is located at 3701 South 14th Street, Lincoln Nebraska. The laboratory building contains the NPHE Lab and the State Agricultural Laboratory. The NPHE Lab is committed to providing staff with a safe environmentally controlled working space and appropriate, well maintained equipment.

4.1 Facility Space, Description, and Environmental Conditions

The NPHE Lab has approximately 12,000 square feet of floor space in the building. A separate chemical storage building is located outside of the main building on the southwest corner near the back dock. The NPHE Lab and the Agricultural Lab share this building for storage of bulk chemicals and hazardous wastes waiting for disposal.

It is important to maintain a controlled environmental condition in the laboratory in order to ensure proper equipment operation and provide a safe and comfortable work environment for the employees. Temperature, humidity and pressure are controlled by a HVAC unit. Temperature is maintained between 65-75 degrees F and the relative percent humidity is maintained between approximately 25-35%, depending on the area and the season. A new mechanical wing, air handling equipment and ducts, as well as new strobic fans for all the laboratory hoods were added to the present building. The project was completed in May of 2011. Monitoring of environmental conditions is done when the conditions could adversely affect test results.

The laboratory is designed to separate incompatible test areas and provide adequate lighting and space to perform analyses. The laboratory fume hoods are located a significant distance from the Volatile Organic Chemical (VOC) analysis area of the laboratory. The VOC analysis test area has its own conditioned filtered air system. The airflow in the area is directed in a manner to reduce possible contamination of VOC samples by solvents used in other parts of the building.

The Air PM 2.5 laboratory area is temperature and humidity controlled. An automatic continuous monitoring system is in place to detect any failures in the temperature or humidity requirements. An audible system alerts staff if the preset requirements are not being met and a strip chart recorder is used to document the temperature and humidity conditions in the area at all times. The recorder is calibrated yearly to ensure that it is in good working order. A whole room hepa air filter system is used to help eliminate contamination of the filters in the laboratory environment.

All employees are expected to keep their individual work areas neat and clean. The maintenance department maintains a contract to provide routine daily professional cleaning of the building as well as non-routine monthly, quarterly, and yearly cleaning tasks, such as washing windows, waxing floors, cleaning air ducts, shampooing carpets, and other miscellaneous cleaning tasks.
Short and long-term facility building upkeep and remodeling project schedules are provided to the staff as a means to communicate the ongoing building maintenance activities that may cause adverse testing problems. Short-term projects are those which are expected to be completed in a relatively short period of time and can usually be completed by the building Maintenance Department. Long term projects may require the use of an outside contractor(s). The contractor(s) will coordinate the project completion with the laboratory staff, as well as the Building Division staff and will also provide the appropriate individuals with the status on each project as it progresses.

4.2 Building Security

Access to the building is limited to employees and scheduled escorted visitors. The front main entrance and lobby is unlocked between 7:50 AM and 5:10 PM, Monday through Friday, except on holidays. The dock receiving area door and all side doors are locked and monitored by electronic security devices at all times. A receptionist at the front desk monitors the main entrance into the building and lobby area, as well as the entrance into the laboratory area during business hours. The entrance into the laboratory area of the building from the dock and front office entry way is controlled by an electronic card reader at all times. A member of the customer service staff is usually available to monitor individuals needing entrance to the back receiving dock entrance to the building. A phone on the dock rings into customer service during business hours.

Visitors to the front entrance lobby, who need to enter the laboratory area must sign in with the receptionist, and are then issued a visitor's badge. A NPHE Lab employee is called to escort them in the facility. Visitors to the laboratory receiving dock entrance are typically Department of Environmental Quality staff dropping off samples, vendors delivering supplies, and occasionally outside contractors. Visitors to the back dock must use the outside call system to alert staff that they are at the dock entrance, unless they have a building security access card. Customer Service Staff or Office staff will proceed to open the dock door and determine if the visitor should be allowed to enter the building. If they need to proceed past the dock area, they must first check in at the customer service office and sign in. They are issued a visitor's badge and are escorted in the facility by a NPHE Lab employee.

Due to the strict security access to the building, the samples are considered under control at all times

The NPHE Lab Visitor Policy requires that all visitors must be at least 10 years old to enter any of the laboratory testing areas or customer service areas of the laboratory and must be attended by NPHE Lab staff at all times. They are only permitted in the break room, front office area, and restroom without supervision.

The main building and the chemical safety building are both equipped with an electronic security system managed by Capitol Security. The system allows access to the building through some of the outside secured doors for those persons who have an authorized picture security card which has been programmed to grant access to the door in question. Every access card is individually assigned and programmed to grant access though specific doors during specific hours of the day. The access granted to each card is based on the duties of the individual and the specific work schedule of that individual. Each use of the card is automatically recorded into the State of Nebraska security database so door entry can be traced and documented. The NPHE Lab Manager and the QA Manager both work directly with the Nebraska State Patrol Building Security staff and DAS to approve security access for the building.

All contractors or other individuals requesting unescorted security access cards to secure parts of the laboratory building must first pass a DAS background security check prior to getting a security clearance which allows them access to secure building sites with their access card. Contractors and all other visitors to the building must display their security or visitor badges when they are inside the building. Any contractor or visitor without a visible badge will be asked to leave the building immediately and if they do not comply with the verbal request, building security or state patrol will be contacted to escort the individual from the building.

The security system also monitors the building's smoke and fire detectors. If an unauthorized access is made to the building, or a fire emergency is detected, the system notifies the State Security command center and if appropriate, the Fire Department. A facility chemical safety lock box is located just inside the front door to aid the fire department in determining where hazardous chemicals are located in the building, in case a fire occurs. State Security staff also patrols the laboratory facility during off-hours to monitor for suspicious activity. An emergency NPHE Lab call list is maintained with State Security, so that in the event of a security or other emergency, the appropriate NPHE Lab personnel can be notified.

The building has an emergency backup generator that is able to take over several sections of the electrical power functions of the building. The emergency power for the BSL suite, the microbiological incubators in the Micro area, and the computer servers is connected to this generator.

4.3 Reagent Water

The NPHE Lab maintains three reagent water systems. One system is a Barnstead NANOpure Diamond system and the other two are Millipore Elix 10's, one which feeds water through a Millipore Academic system. The Barnstead Unit uses a four-stage deionization process and a 0.2 micron filter to polish suitable feed water. The Millipore Elix system utilizes a pretreatment filter, RO, and electrodeionization technology. These units produce ASTM Type II water. The Academic system produces Type I water.

The conductivity, pH, total residual chlorine, and HPC of each system is measured monthly by staff. Nitrate, fluoride and metals are all measured annually. Results for monthly and yearly testing is documented and maintained in the Reagent Water Logbook located in the QA office.

Maintenance records for all systems are kept in notebooks located by each unit. All readings out of acceptance range should be investigated immediately and documented. Specific Reagent Water requirements and documentation procedures can be found in the current version of *SOP # 8100.1, Ancillary Equipment Documentation.*

4.4 Glassware Cleaning Procedures

The procedures for cleaning all sample containers and laboratory glassware are described in appropriate SOPs for each method. Most sample containers are purchased on contract from an environmental sample container vendor. The vendor is required to provide sample containers that meet or exceed minimum requirements set forth by EPA for each method. The vendors usually provide certification documentation for the sample containers they ship to the lab. The NPHE Lab staff maintains all certification documentation for future reference. Some method SOPs require that one or more container(s) from each box or lot of containers shipped to us, be checked for acceptability prior to using them for analysis. See individual Method SOPs for specific requirements.

Glassware must be handled according to all requirements stated in the specific method SOP. Reusable glassware may be washed by hand or in the laboratory dishwasher. One automatic laboratory grade dishwasher is maintained at the NPHE Lab which provides a final rinse with Type II water. It is currently maintained as needed by calling for service from the vendor if the building maintenance staff can not complete the required service. All maintenance is documented and recorded in the maintenance logbook located by the dishwasher.

Disposable laboratory glassware may be recycled, if it meets the State recycling vendor's requirements and Federal and State environmental regulations. If the glassware can not be recycled, it is disposed of in the building trash receptacles once it has been decontaminated, neutralized, or rendered environmentally safe by some other method to be disposed of in the sanitary landfill.

4.5 Reagent Storage

All laboratory chemicals are segregated according to critical chemical properties. For example, strong acids are stored separately from strong bases. Flammable materials are stored separately from oxidizing agents. Dry granular or powder reagents are in the storage cabinets in the appropriate chemistry and microbiology laboratories. Chemicals, standards, and solutions, which require refrigeration or freezing, are stored within the appropriate temperature ranges. A chemical storage building is located within 50 ft of the back dock of the main Laboratory building. This chemical storage building is for bulk storage of chemicals and solvents and for chemical wastes waiting for disposal.

All laboratory chemicals, reagents, standards, media, and solutions are labeled, dated when received and/or made and dated when first opened or used. All related data bases are updated appropriately, as required by SOP, method, and/or certification requirements to reflect receipt and/or usage information and any QC performed prior to use, for each of the above items.

4.6 Equipment

The NPHE Lab understands that well maintained equipment is required to produce quality data. A list of equipment used at the laboratory can be found in *Appendix N*. Routine preventative maintenance requirements for major laboratory equipment are described in the applicable method SOP and/or the equipment maintenance logbook.

4.6.1 Equipment and Instrument Maintenance

Instruments and other laboratory equipment require routine maintenance. This can either be routine maintenance for preventive reasons, or maintenance to correct malfunctions of the equipment. All maintenance activities are documented in the associated instrument maintenance logs. These logs may be electronic or paper, as long as they are complete and easily accessible. Paper logs are kept near the equipment so they are readily available to all personnel. Electronic logbooks are kept on the lab's shared computer drive. At a minimum, the information that is recorded includes:

- Instrument Identification
- Date of maintenance
- Maintenance Performed
- Name (or initials) of person who performed the maintenance

4.6.2. Service Agreements

The NPHE Lab maintains some partial or full service maintenance contracts on most of the major instruments and instrument software packages. An inventory of some critical spare parts is maintained to enable a quick return to service of malfunctioning equipment. Most of the analysts have some training on how to perform basic equipment trouble shooting and maintenance procedures. A master list and notebook of current equipment service agreements is maintained in the Lab Manager's office.

5. Computer Systems

The laboratory purchased a new LIMS from Chemware and moved to the new Horizon software in March of 2010. The NPHE Lab uses its LIMS for many tasks. These include sample prelog, log-in, sample status, data reduction and reporting, electronic data archival and electronic communication with the Nebraska Drinking Water Program., with clients and entities outside the laboratory. The DHHS Public Health Computer Use Policy describes the company policies on the usage of computer accounts with emphasis on security issues and covers passwords, file naming, file protection, file placement, personal privacy, and company rights, as pertaining to e-mail usage, network programs, available disk space, and usage of monitors, printers, and the internet. The DHHS IS&T section controls the policies of the laboratory's computer systems. All employees have separate passwords which permits log-on to the user's computer network and the LIMS database.

5.1. Computer System Back-Up

Virtual Machine backups are taken once a night using Microsoft DPM. The backups are stored on disk on servers located in the DHHS datacenters in downtown Lincoln. The backup images retained on disk for 30 days. Due to limited site bandwidth, on occasion it takes up to 2-3 days for the backups to replicate back to the Microsoft DPM backup servers.

Backup tapes should be tested periodically to ensure data can be retrieved.

5.2. Computer System/Software Validation

The Information Technology Department maintains a computer software validation SOP that complies with the "The Environmental Protection Agency's (EPA) Good Automated Laboratory Practices Manual", 1995 edition. NPHE Lab validates software programs which are written by the NPHE Lab staff or miscellaneous vendors and which are utilized to generate or process data that are reported to our clients. This validation is performed prior to any use of these programs in calculating data to ensure the generation of the correct results. Data validation for the new Horizon data system covered a three week period of random double entry of samples into both the previous LIMS (Seedpak) and into the new LIMS (Horizon) system. During the validation period the laboratory worked out discrepancies throughout the process and compared the data reports for problems. When the Horizon system was accepted, cutover to Horizon took place in March 2010.

6. Bid, Proposal, and Contract Review

The Laboratory Manager and the QA Manager are both involved in the development and review of all Requests for Proposal (RFP). RFPs may also be reviewed by one or more of the following: the NDHHS Environmental Health Administrator, the DHHS Director, the DHHS Deputy Director, the DHHS Legal Department, and the State of Nebraska Purchasing Department Technical Services. This review focuses on technical, QC, and reporting requirements and

contract language including, but not limited to, insurance requirements, indemnification, and payment terms.

Prior to signing a contract, negotiation of contract language is conducted so that the resulting contract contains language acceptable to both the NPHE Lab- oratory and its client. The depth of this review depends upon the complexity of the contract terms. A contract review process developed by and for the State of Nebraska is used to document the review and approval process. This enables all NPHE Lab personnel to understand the details of the contract and client requirements.

7. Sample Management and Tracking

The custody procedures for shipping, sample collection, sample receipt, and sample tracking throughout the analytical process are described briefly in the following sections. Refer to the current version of NPHE Lab *SOP # 1900.1 Customer Service Procedures and SOP # 1910.1 DEQ Customer Services* for more specific sample management and/or tracking procedures.

7.1. Order Placement

In most cases, the first step for a private client in the laboratory process is a client phone call to the NPHE Lab customer service section. A customer service area staff member takes the order information, including the client identification, account number, requested analyses, type of sample matrix, turn around time needs and other pertinent information. This information along with any additional information is compiled into a sample request by using LIMS application for Order Processing. If the client is new to the NPHE Lab, the client account must be setup in both the LIMS and the NIS accounting system.

The Drinking Water Program staff can electronically submit SDWISIN files to the lab. These files are then processed into the LIMS and verified. The laboratory customer service staff then makes sure that the sample requests are filled as requested, at the appropriate time.

Clients may also request sample collection kits in person, at the front reception desk. These requests are processed much like the electronic or project requests described above. The client can pick up their order at the front reception desk or the laboratory customer service staff will mail it to the client once it has been prepared.

7.2 Shipping

The Customer Service area of the NPHE Lab, processes sample requests each day. The customer service staff closely works with the analytical staff, the Drinking Water Program staff, the Department of Environmental Quality program managers and samplers, as well as other clients to schedule the proper sample kits and timely shipment to the client. The NPHE Lab Customer Service staff ensures that the proper type, size, number of sample containers and appropriate preservatives are included in each order, as it is prepared for shipment. The required sample containers are packed into the shipping carton(s) with the required sample submission form (SSF) and any needed chain of custody documentation, instructions for

sample collection, and appropriate supplies to keep samples within the temperature requirements, during the return to the laboratory. See *Appendix O,* for a Sample Method Table, which includes the container type, preservation, holding time, and turnaround time for each method.

7.3 Chain of Custody

Completed Sample Submission Forms (SSF's) serve as COCs for all non-DEQ samples. These SSF's are then scanned into the LIMS and can be retrieved if needed. Chain of Custody (COC) records are maintained for all DEQ samples processed at the NPHE Lab. The purpose of COC document is to record the history of sample containers and samples during the container shipping, sample collection, transportation, and receipt process. Sample containers are submitted from DEQ samplers along with a COC form. The sampler is responsible for the information on the COC form. The client signs the COC form to verify that they are relinquishing the samples to the custody of the NPHE Lab. The samples arrive at the NPHE Lab by common carrier or they are driven to the NPHE Lab by the sampler. The NPHE Lab acknowledges the receipt of the samples by signing the COC form and logging the samples into the LIMS. A description of COC procedures are described in the current version *of SOP # 1910.1 DEQ Customer Service.* An example of a DEQ COC form is shown in *Appendix P.*

7.4 Evidentiary Chain of Custody

When an evidentiary COC is required, samples are secured under lock and key, once received at the NPHE Lab. An analyst in the section of the NPHE Lab in which the sample is being tested maintains access to the key. The custody of the sample containers for these projects are usually shipped by common carrier (UPS or Federal Express), and the representative of the common carrier will document receipt of the shipping and collection materials by signing the evidentiary chain of custody.

The evidentiary chain of custody procedure requires that when an analyst removes a sample for analysis that the date and time be entered in the appropriate place on the form. Additionally, the analyst must initial the form. The same procedures are required when returning the sample container to the storage area. If no sample remains after analysis, it must be so documented on the form with the date, time, and the initials of the analyst and witness.

When the samples are too numerous to store in the lock box in the walk-in cooler, the samples are stored in a locked refrigerator to enable the evidentiary chain-of-custody procedures and forms to be utilized. Records are maintained that account for time periods and identify individuals who handle the samples from receipt through sample preparation, analysis, and disposal.

7.5 Sample Collection

The NPHE Lab is never responsible for sample collection. The private client, public water operator or the field staff for the NDHHS Public Health Environmental Health Division, the Department for Environmental Quality, or County Health Departments collects the samples. When collected, the samples are labeled with a bar code, which matches the bar code on the Sample Submission Form (SSF). The SSF contains the following information: the client's name and account number, sample location, date and time of collection, collector's name, analysis required, and other pertinent information as prescribed in the applicable NPHE Lab Sampling and Shipping Instructions for sample collection and preservation. The sampling personnel enter all necessary information onto the SSF and transfer custody to the NPHE Lab receiving personnel when the samples are returned to the laboratory. The NPHE Lab Sampling and Shipping Instructions describe the specific collection procedures for the different analyses, as well as instructions for returning the samples to NPHE Lab or contract lab. Current copies of NPHE Laboratory sampling and shipping instructions are maintained in the Customer Service area. Samples collected by DEQ field samplers do not have SSFs. Instead they are submitted with COCs as described in section 7.3 above.

7.6 Sample Receipt

Upon receipt, the NPHE Lab customer service staff and other laboratory staff acknowledge receiving of the samples. The shipping containers are opened and the refrigerant (wet ice) is inspected to determine if the samples were maintained at the proper temperature during shipment. As long as there is wet ice present in the shipping container, the samples within the container are considered acceptable for meeting proper temperature shipment requirements. The actual temperatures of all Fecal coliform samples and any other temperature dependent samples, are measured using the IR temperature gun upon arrival at the lab. The temperature is recorded on the SSF or COC.

The sample kit(s) and request form(s) or COC(s) are compared to ensure that samples are received for all requested tests and that the appropriate bar code and information is included on the sample and request form. Clients are contacted whenever samples do not meet specific sample requirements. If there are document discrepancies, the samples are damaged, or any other problem is noted, the analytical staff and/or client is immediately notified for clarification and the appropriate corrective action is taken. The action directed by the client and/or analyst is recorded on the SSF or COC.

The sample demographic information is entered into the LIMS system under the assigned unique sample numbers. Sample numbers are recorded on the DEQ COC and recorded on the sample containers. If the client or project circumstance requires, an evidentiary COC is initialed, dated and all such samples are maintained secure in the walk-in cooler. The procedure for using the LIMS receiving application is described in the current version of *SOP #1800.1 Customer Service LIMS Login and Reporting.*

If any samples require chemical preservation upon receipt, the analytical staff is responsible for adding the preservative in accordance with the applicable method SOP. If samples were

collected in containers that had preservative, those samples are checked for proper preservation upon receipt at NDHHS, unless doing so may result in loss of analyte, such as for volatile compounds. In those cases, the preservation is checked once the test is completed. If the preservation technique is not sufficient to meet the criteria of the reference method, the sample is not considered valid, and the client is contacted to address the problem. Refer to *SOP 1900.1 Customer Service Procedures* or the method SOP for Rejection Criteria for samples.

Samples with short holding times are delivered immediately to the appropriate area of the laboratory, after log-in is completed. Other samples are stored in the assigned locations in the walk-in cooler, or in the appropriate area of the lab. All samples are stored at the appropriate temperature and away from laboratory contaminants, standards and known highly contaminated samples. Holding times may not be exceeded.

7.7. Sample Tracking

As described above, all samples are given a unique NPHE Lab sample number when they are prelogged or container logged in. These sample numbers are used to track samples through shipping, receipt, preparation, analysis, reporting, and archiving and are recorded on all documents relating to sample preparation, analysis, and reporting. The Inquiry and Backlog functions in the LIMS can be used to monitor the status of samples in the laboratory. The Inquiry function of the LIMS shows the dates and times of various process events (e.g., sample receipt, extraction, and analysis) and the initials of the employee who performed that step in the process.

7.8. Sample Disposal

Samples are retained for 30 days after the testing is completed by the analyst, unless those samples can no longer be re-tested. Those that can no longer be tested include samples for microbiological testing and volatile samples that are completely depleted during the initial testing process or compromised due to sample type and/or testing method. Samples may be held longer to meet client requests or specific contract requirements. Sample extracts and any other hazardous materials generated during sample preparation and analysis are disposed of by a commercial vendor who is licensed to do so. Samples requiring an evidentiary COC, require the client's approval prior to the disposal of the samples. Sample, extract and hazardous material disposal, is described in the current NPHE Lab *SOP # 7300.1, Management of Lab Wastes and NPHE Lab SOP # 7400.1 Sample Disposal.*

8. Analytical Procedures

All analyses for regulatory compliance are performed using methods approved under the Safe Drinking Water Act or Clean Water Act. Any variations to a referenced method are included in the applicable NPHE Lab Method SOPs. All procedures currently performed at the NPHE Lab have a written approved SOP. Each SOP has a unique control number which also denotes the revision number of the document. The SOPs currently utilized at the NPHE Lab are listed in the NPHE Lab Standard Operating Procedures Table of Contents. A current SOP Table of Contents

can be found in Appendix Q of this manual as of the time of approval of this QAP. A updated copy of Appendix Q SOP Table of Contents will be added to the front of each volume of SOPs at least once a year when all SOP' have been reviewed and/or updated.

The principal references for analytical methods employed at the NPHE Lab are:

- "US EPA Methods for the Determination of Organic Compounds in Drinking Water"
- "US EPA Methods for the Chemical Analysis of Water and Wastes"
- "Standard Methods for the Examination of Water and Wastewater,"20th Edition (1998), 21st Edition (2005) 22nd Edition (2012)
- Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-932, Aug. 1980
- "Eastern Environmental Radiation Facility Radiochemistry Procedures Manual," EPA-600/4-80-032, Aug. 1980
- "Manual for the Certification of Labs Analyzing Drinking Water" Fifth Edition, EPA 815-R-05-004 January 2005 & Supplement 1 to the Fifth Edition, EPA 815-F-08-006, June 2008

Copies of reference methods are maintained in the QA department, and in the technical operations areas of the laboratory. NPHE Lab employees continually monitor the Federal Register for regulatory activity, including the approval of new methods and revisions to old methods. The NPHE Lab also subscribes to EPA newsletters, Calibrate produced by Catalyst Information Resources and other industry publications to ensure that the most recent, approved versions of the test methods are being used.

Written standard operating procedures (SOPs) for conducting each of the analytical tests performed in the laboratory is available in the Laboratory Managers office, the laboratory testing area, and on the shared drive of the laboratory computer system. Each SOP includes or references, where applicable, the following:

- A cover page including SOP number, SOP title, date of preparation or revision, author, reviewer, and approver signatures
- Scope and application
- Summary of test method
- Definitions
- Interferences
- Safety and waste management
- Equipment and supplies
- Reagents and standards
- Sample collection
- Quality control requirements
- Calibration and standardization
- Analytical procedure
- Data acquisition
- Corrective action and maintenance
- Method performance
- Pollution prevention
- References

- Attachments

Additional information regarding preparation of standard operating procedures can be found in the current NPHE Lab *SOP # 1600.1, Preparation of Standard Operating Procedures.*

8.1 Subcontractor Approval

Subcontracting of tests occurs when the NPHE Lab is unable to perform a particular method or test for a certain analyte. The NPHE Lab Laboratory Manager or the QA Manager verifies that the subcontractor meets reporting limits and approved methodology as well as the client's regulatory needs, (i.e., state certifications or NELAC certification.) NPHE Lab maintains an information file for each subcontractor used to provide the analytical services listed below:

- Analysis for Dioxins
- Analysis for Radiochemical Parameters
- Analysis for Asbestos
- Analysis for Endothal
- Analysis for Total Organic Carbon
- Analysis for Disinfection Byproducts (ie, bromate, chlorite)
- Analysis for Microscopic Particulate Analysis
- Analysis for a variety of Miscellaneous Test Methods

A contract is in place with a contractor to cover the majority of the analytical services requested above. All contractors and subcontractors must maintain EPA and/or NELAP certification for the method requested, if certification is available in the United States and compliance samples are involved. The process for setting up contract services is a function of the Nebraska Department of Administrative Services Purchasing Department. That process involves input and review steps from the NPHE Lab and the NPHE Lab Legal Department to ensure that the contracts meet our client needs, as well as the State's legal obligations. An example of a COC for contract work can be found in *Appendix R* in this manual.

8.2. Sample Processing and Data Generation

8.2.1. Data Quality Objectives

Data quality objectives are qualitative and/or quantitative indicators used to define the quality of the data needed to support sample assessments. Achievement of these objectives helps to demonstrate that the data is scientifically valid and defensible. The data quality objectives for the laboratory are developed by the state agency using the data, such as the Drinking Water Program or the Department of Environmental Quality or by the private client ordering the testing prior to receiving the sample collection kit request. The data quality objectives differ with each program or client and are communicated to the laboratory prior to ordering sample kits. Most data quality objectives are based on preset program requirements that has been discussed with laboratory management prior to beginning a new project.

8.2.2. Data Acquisition

Data acquisition at the NPHE Lab includes both automated and manual entry. Some analysts use instruments which operate unattended and calculate results. The results are recorded in a data packet. When a computerized instrument is used, the raw data is usually saved to the LIMS and backed up by the LIMS at the end of the analysis. In some instances even though an instrument is computerized the data must be hand entered into the LIMS due to the inability of the instrument to interface with the LIMS.

8.2.3. Data Reduction

After raw data backup, an analyst reduces the data. This process may include inspection of chromatography and quality control performance, and checking the results for MCL violations. Printouts of the data may be generated and assembled into a data packet. The data packet may also contain a run log, any original calculations, results of calibration, and documentation of any sample preparation. For Organic analyses and some Inorganic analyses, the processed data is pushed into the LIMS system through the autopost pipe. The analyst records any comments regarding data qualification in the LIMS and those comments may or may not be reportable on the final report. Analytical bench sheets for the entire analysis are included with the printouts obtained using the instrument software. The assembled electronic data and/or data packet is reviewed by the analyst for completeness.

8.2.4 Manual Data Integration

The reduction of chromatographic data is typically performed utilizing the instrument data processing software. Once in awhile, this software does not process chromatographic responses in an accurate or representative manner. When this occurs, manual integrations of chromatographic peaks may be required. The manual adjustment of this data, for all chromatographic analyses is termed "manual integration". All such manual integrations undergo a peer review as part of the QA Review. The principal guidance in this can be summarized as follows:

- When integration of a chromatographic peak is incorrectly performed by the automated data processing system, the peak area is manually determined in such a way to most accurately reflect the correct and representative area response of the analyte peak under consideration.
- Manual integration must be made consistently in evaluation of calibration standards and sample responses.
- Manual integrations are identified on the data packet and must include a notation which describes why a manual integration was performed; the initials of who did the integration and the date must also be noted.
- Raw data for both integrations must be maintained with the results.

8.2.5. MCL/Action Limit Violations

An analytical result that equals or exceeds a Maximum Contaminant Level (MCL) or a Reporting Limit (RL) may need to be confirmed, depending on the analyte, method and/or SOP requirements. The analyst must give the highest priority to verification of critical data so that the client can be notified as soon as possible. The primary responsibility of reporting critical data to the Laboratory Manager is the responsibility of the analyst. All results, which show data which could be a critical health hazard, whether or not the client ordered that compound, are reported to the Laboratory Manager. The analyst may be asked to reanalyze the sample to verify the value. In the absence of the Laboratory Manager, the QA Manager should be notified of the critical value observed. The laboratory Manager or QA manager will report critical data to the program managers.

8.2.6. Other Compounds Detected

The NPHE Lab reports results for all compounds ordered by the client. NPHE Lab, in most cases, does not report results for other compounds detected unless they could be a critical health hazard.

8.2.7. Interfering Peaks

When a sample result cannot be calculated due to the presence of an interfering peak, and the opportunity to process the sample at a dilution does not exist, the comments in the Data Packet and the client Report Narrative must include data qualifier "M". The Report Data Qualifier List on the back of each report explains the "M" data qualifier as: "The analysis was inconclusive due to matrix interferences. The sample needs to be recollected."

8.3. Data Review

The primary analyst first reviews the data and any confirmation data. The results for the batch are entered into the LIMS either through the instrument and/or by manual entry, reviewed for accuracy, posted and released for second review. This step is defined as "Batch Review" ("BREV"). Any data packet and/or bench sheet and any confirmation data are given to a second analyst who performs a separate review of the data. This step is defined as "Peer Review" ("PREV"). During each of the data review processes, the entire data packet and/or LIMS documentation is inspected to verify that quality control requirements were met, the data is defensible and traceable, any manual integrations were properly documented, results were entered accurately in the LIMS database and any sample comments or flags are correct. Once all analysis for a sample are complete, the sample then moves to "Manager Review". Manager review may include one or two of three different types of review. "TECH" is a technical review for samples that may have a technical problem that must be corrected and/or approved prior to releasing results. "SREV" is supervisor review to verify that single batch reports are ready for printing. "WREV" is supervisor review to verify that workorder reports are complete and ready for printing. This final management review can be completed by the

following staff: the Laboratory Manager, the QA Manager, or other responsible staff as assigned by management. For more details refer to the NPHE Lab *SOP # 3230.1 Data Review and Handling.*

8.4. Data Reporting and Report Review

As analyses are completed, reviewed, and approved by the analyst and a second reviewer, a manager review is completed on the data. It is then released to the print daemon in the LIMS where it will be printed, faxed, e-mailed and/or placed in the laboratory's web portal for the client to access the results. All reports sent to the printer to be mailed to clients will be retrieved by customer service and compiled for mailing each day. For more details on data reporting and report review see current *SOP # 1800.1 Customer Service LIMS Login and Reporting.*

8.4.1. Significant Figures

All reported analytical results contain the number of digits (numbers) which are known to be valid. Significant figures reflect the limit in accuracy of the particular method of analysis and measuring instruments. The numbers of significant figures which are permitted to be reported in a valid result are limited to the number of significant figures which are associated with the least accurate determination in the measurement process.

Significant figures contain all digits that are accurately known and a last digit which is estimated. For example, if a concentration is reported as 18.8 mg/L, the first two digits must be accurately known and the decimal, 0.8, must reflect the best estimate of that value (i.e. it is more accurate than 0.7 or 0.9)

The number zero may or may not be significant. If it represents a measured quantity, zero is a significant figure. If a zero is merely a placeholder (i.e., it locates the decimal point), it is not a significant figure. Rules regarding zero as a significant figure are summarized below:

(a) A zero between two non-zero digits is significant.

(b) If there are no preceding non-zero digits, a zero before the decimal point is not significant. If there are no non-zero digits preceding a decimal point, the zeros after the decimal point but preceding other non-zero digits, are not significant.

(c) Final zeros to the right of the decimal point, at the end of a number (i.e., 11.00) are always meant to be significant. Final zeroes in a whole number (i.e., 11,000) may or may not be significant. The following table lists the guidelines for reporting results.

Result (units are example)	Report to Nearest
<1 mg/L	See Note Below
1.1-10 mg/L	.1 mg/L
11-100 mg/L	1 mg/L
101-1000 mg/L	10 mg/L
1001-10,000 mg/L	100 mg/L
10,001 – 100,000 mg/L	1000 mg/L
100,001 – 1,000,000 mg/L	10,000 mg/L

8.4.2. Rounding Rules

This section will describe the rules used at the NPHE Lab for rounding numbers. If the figure following those to be retained is greater than five, the figure is dropped, and the last retained figure is raised by one. For example 12.216 would be rounded to 12.22.

If the figure following those to be retained is less than five, the figure is dropped, and the last retained figure remains the same. For example, 10.224 would be rounded to 10.22. If the figure following those to be retained is five or more, the five is dropped and the last retained figure is raised by one figure. For example 3.305 would be rounded to 3.31.

If a series of calculations is to be performed, all figures are to be carried through the calculations. The final answer is then rounded to the correct number of significant figures.

8.4.3. Special Rounding Situations

In the special case where a group of analytes are reported separately and also as the "total" of the analytes, (e.g., Total Trihalomethanes) the individual results are rounded using the rounding rules first, and then the results are summed to produce the "total" result.

When comparing result percent recoveries to acceptance criteria, the percent recovery value is rounded to the nearest whole number prior to comparison to the acceptance window. This rounding is performed according to the rounding rules listed above, in Section 8.4.2.

At times, samples are diluted in order to get results within the calibration range, or to mitigate matrix affects. In these cases, the raw result of the diluted analysis is compared to the raw RL in order to determine if the result is reportable. If the procedure is one that employs a correction factor (an adjustment made due to the sample volume that was processed), the corrected result is compared to the corrected, unrounded RL. Alcohol results are truncated rather than rounded.

8.4.4. Electronic Data Reporting

Each day all of the completed Public Water System data is automatically reported to the DHHS, Drinking Water Program in an electronic format. The data format is based on the needs of the EPA Safe Drinking Water Information System (SDWIS). The data completed and reported to the customer each day is dumped into SDWIS at night and is available for the Drinking Water Staff the following morning.

8.5. Data Archival

All raw data, data packets, request forms, QC/QA information, equipment logs, and any other miscellaneous documents are archived for a minimum period of twelve (12) years after the report has been sent to the client. The data packets and all other possible documentation are scanned into the Horizon LIMS and are electronically linked to each sample in the batch. Anything not scanned into LIMS is filed in cabinets or boxes, which are usually located near the area where the analysis is performed. When these areas are full, the data is archived in boxes for long term storage and sent to Records Management. These boxes are clearly labeled and assigned a code. The codes and the contents are identified in the Record Management Archived Data Log. The boxes are kept in a secure, limited access area of the State Records Management facility. The procedure for storing data is described in the current method SOPS and NPHE Lab *SOP # 1500.1, Records Management.*

8.6. Invoicing

Once the data report has been finalized it is ready for invoicing. All invoicing is done by the NDHHS staff. Invoicing is done weekly for walk-in clients and once a month for Alcohol accounts, SDWIS accounts and interagency state accounts and includes all testing that was completed during the prior month. Invoicing is done using an extracted report from the NDHHS PHE LIMS. The invoice detail and totals are verified for accuracy. The NPHE Lab billing terms require payment within 30 calendar days of invoice receipt. NDHHS PHE management and customer service staff monitors Accounts Receivable (A/R) and begins follow-up with clients when the A/R exceeds 60 days. An A/R aging report is generated on a monthly basis, and is reviewed to determine what course of action is required. The actions range from a follow-up contact with the client by NDHHS Support Staff and in the extreme cases, corrective action may include getting the NDHHS Environmental Health Unit Administrator involved, setting up payment schedules, denying future testing until payment is received and/or writing off the amount due.

8.7. Correction of Errors

If a transcription error is found, the error is corrected by placing a single line through the incorrect text or number. The correct information is placed above or next to the error, and the initials of the person making the correction is recorded in the vicinity of the correction, along with the date of the correction action.

9. Customer Complaints

The NPHE Lab responds to customer complaints based on the specific details of the complaint. Complaints generally fall into two categories: technical and customer service. The QA Manager, the Laboratory Manager or any NPHE Lab staff member who is contacted by the customer is responsible for initiating Corrective Action Requests or Customer complaint forms, as necessary to resolve the cause of the complaint. The QA Manager, the Laboratory Manager, or other appropriate NPHE Lab staff contacts the client, depending on the severity of the complaint and of the action necessary to resolve the issue. The QA Office documents any corrective action taken due to the complaint and files the completed Corrective Action Request or Customer Complaint form generated as a result of a customer complaint.

10. Calibration, Traceability, and Instrument Maintenance

Calibration, verification, and traceability of all equipment, standards, reagents, chemicals, controls and other supplies used in the analytical process is extremely important in ensuring that all data is accurate and defensible. The documentation of these processes is equally important and can be found in this QA plan as well as specific administrative and analytical SOPs.

10.1. Initial Calibration

All instruments are calibrated prior to use to establish instrument response to a known amount of a reference material. The manner in which various instruments are calibrated is dependent on the type of instrument and its intended use. All sample data is reported from within the calibrated range of the instrument. Preparation of all calibration standards is documented in the applicable Standard Preparation Log. Instrument Calibration is performed using standards that are traceable, when available, to NIST Standards. At a minimum, all standards are traceable to the manufacturer's lot number and Certificate of Analysis. The number and concentration of calibration standards used are included in the individual method SOPs. Acceptance criteria for calibration data and calibration verification procedures are specified within individual method SOPs.

Initial calibration procedures establish the calibration range of the instrument and determine instrument response over that range. Typically, three to seven analyte concentrations are used to establish the instrument response over the concentration range of interest. All calibration standards that are analyzed in the initial calibration must be included in the calibration curve, if the curve meets the acceptance criteria stated in the SOP, unless one of the following situations occurs:

- The lowest point may be dropped only to produce an acceptable correlation coefficient. If dropping the lowest point results in a calibration range that does not include the MRL, the MRL must be raised to a concentration that is included in the new calibration range.
- The highest point may be dropped only to produce an acceptable correlation coefficient. No data that exceed the highest concentration of the new calibration range may be reported.
- A low point and a high point may not be dropped from the same calibration curve.
- Other calibration points may be dropped only if there is a gross physical error that is known to the analyst, such as a broken injection needle or an empty autosampler vial.

Dropping of calibration points must be noted in the data packet and the reason for dropping the point noted, dated and initialed. Once a calibration curve has been accepted and put into use, points may not be dropped or reinstated. The selection for the curve fit and the treatment of the origin are not changed from one calibration run to the next.

The laboratory will always run a low standard or a LFB at the method RL during each test run. If the lowest standard is not equal to or less than the method RL, the low level LFB at the method RL will be run before any samples and again after all samples are run to verify that the RL can be met during each run. The laboratory's reporting limit for all contaminants will be equal to or greater than the lowest calibration standard or lowest acceptable LFB used in the run. Each primary analyst will ensure that this meets method requirements for all contaminants for their respective method. All affected method SOPs will be updated to reflect any change in the makeup of the calibration curve, LFBs used and/or RL limits of contaminants. (Inserted January 31, 2011 as per EPA Audit Response)

10.2. On-Going Calibration Verification

Calibration curves are verified at the frequencies required in the individual SOP. For most methods, these verifications are performed by the analysis of Continuing Calibration Check (CCC), Continuing Calibration Verification (CCV), or Instrument Performance Check (IPC). These verifications may involve measurement of instrument response at the low, middle, or upper end of the calibration range and require that the responses conform to a specified limit relative to the initial calibration. Continuing calibration standards (from the same source as the initial calibration standard) are typically analyzed at the beginning, and sometimes the middle and end of an analytical sequence to verify stable calibration throughout the sample analysis. The calibration curves are also verified against a second source standard called a Quality Control Sample (QCS) at a frequency specified in the method SOP. Each analytical data packet includes calibration used with that data packet.

10.3 Linearity Studies

Linearity is defined as the ability of the analytical procedure to obtain test results within a given range, which are directly proportional to the concentration of analytes in the sample. Linearity studies are performed to determine the linear reportable range for an analyte. The linearity for each analyte is assessed by checking the performance of recovery throughout the manufacturer's stated range of the testing system. This is done using a set of standards containing various levels of an analyte in high and low enough concentrations so as to span the entire range of the test system. Linearity studies are performed as required in specific methods. Some are performed at least once a year, well others at least once every six months. The data from the linearity study should be reviewed for acceptable accuracy and precision as well as the true value of each standard used. Ideally all data should be within 10% of the expected values. The upper limit of the reportable range will be set at the concentration of the highest standard tested which exhibited acceptable results for linearity, accuracy, and precision. This concentration, however, may not exceed the manufacturer's stated linear range. Samples with concentrations higher than the reportable range must be diluted and

retested. The same is true for the lower limit. Samples with concentrations lower than the reportable range must be reported as less than the reporting limit.

10.4 Calibration and Validation of Other Equipment

All supporting equipment used in sample preparation and analysis (thermometers, automatic pipettes, balances, pH meters, etc.) is calibrated before being placed into service and on a continuing basis in accordance with the requirements of the manufacturer, the NPHE Lab Ancillary SOP, or method SOPs. The periodic calibration checks are performed by comparing the equipment to NIST calibrated equipment, when applicable. The analyst in the testing area or QA Manager in the QA Office maintains records of each calibration check. For more details regarding the required checks and documentation see the current version of *SOP # 8100.1, Ancillary Equipment Documentation.* An Ancillary Equipment Table, located in *Appendix S* summarizes most of the ancillary equipment calibration tasks, who is responsible for completing the tasks, and where the documentation for each task can be found.

10.4.1 Balances

Laboratory balances are calibrated and serviced annually by an authorized vendor. The balance calibration is verified daily when in use with two weights using a set of class "S" weights which are NIST traceable and have annual certification. The results of these daily checks are documented in the Balance Log.

10.4.2 Thermometers

Thermometers are used to monitor the temperature of refrigerators, freezers, incubators and water baths which are used for sample storage, preparation and analysis. Thermometers used for chemistry analyses are calibrated annually at the "Temperature of Use". All thermometers in use are labeled with the calibration date, initials and calibration factor. Calibration is performed with thermometers that are certified to be NIST traceable. NIST traceable thermometers are re-certified by an outside source, at least every five years or internally once a year. Thermometers used in chemistry, which differ by more than 1°C from the correct temperature, are taken out of service. Thermometers used in microbiology incubators and water baths, which differ by more than 0.5°C from the correct temperature, are taken out of service.

10.4.3 Refrigerators, Freezers, Incubators, and Water Baths

All refrigerator and freezer temperatures are monitored daily. Incubator and water bath temperatures are monitored when samples are analyzed and equipment is in use. Microbiological incubators and water bath temperatures are recorded twice daily when in use, at least four hours apart. All temperatures are recorded on the temperature log located on or near the unit. In the event that the temperature is outside the required range, the appropriate corrective action is taken and documented for future reference. If the temperature problem invalidates results or can not be resolved, the QA Manager

and/or the Laboratory Manager should be notified and results should be flagged appropriately. Past year temperature records are maintained in the QA Office.

10.4.4 Automatic Pipettes

Automatic pipettes used for preparation of samples and standards are calibrated quarterly. Each pipette is labeled with the date of calibration, initials of the person who performed the calibration and the date that re-certification is due. The pipette calibration data is maintained in an electronic database. A print out of the calibration history for each pipette is kept on file by the pipette calibration station.

10.5 Traceability of Standards, Reagents, Solvents and Other Supplies

Standards are traceable to NIST Standards whenever they are available. Other standards and reagents are traceable to the manufacturer's lot number and Certificate of Analysis. The Certificate of Analysis for standards and reagents are maintained in the areas of the laboratory where they are used.

All data packets must include the proper documentation to trace and identify the standards, solvents, reagents, and extraction material used to process the samples. This is accomplished by including standard and reagent lot numbers, or by other documentation in the data packet and/or LIMS. Various standard and reagent preparation logs are used to document any preparation steps used to prepare working standards and reagents. Some electronic logs are contained within the LIMS. All standards, reagents, chemicals, controls and all other laboratory supplies with an expiration date, such as media, or bottles, must not be used if they are past their expiration date.

10.6 Validation of Supplies and Standards

New standards received at the NPHE Lab should be verified before being used in the laboratory for compliance purposes. New standards are treated as a second source standard and analyzed against existing calibration standards to verify concentrations. These new standards are not extracted or processed unless a necessary chemical reaction such as derivation is required to compare the old standards. Agreement between the new and old standards should be determined prior to use as the primary standard source. New shipments or lots of reagents are validated prior to use. Analyzing a blank prepared with that reagent validates reagents that are critical to the procedure.

All chemicals used in reagents (including standards) are labeled and included on the chemical inventory list. The label information includes date received, date opened and expiration date, if applicable and available, along with the initials of the analyst dating the label. Reagents and standards used in sample preparation and analysis are recorded in the applicable standard or reagent preparation logbooks, data packets or the LIMS database.

10.7 Validation of New Equipment

New instruments must be validated according to the reference method requirements. At a minimum, the validation must include the analyses of blanks, calibration curves, MDLs, and manual calculation checks. Other supportive measuring devices (pipettes, balances) are validated according to the specific method SOP. Results from the old equipment should be compared to results from the new equipment to verify that appropriate equipment setup has been accomplished. This can include the use of old PT samples, standards, or other samples of known values.

11. Internal Quality Control Procedures

The daily quality of analytical data generated by the NPHE Lab is controlled by conformance to this Quality Assurance Manual and all applicable NPHE Lab Standard Operating Procedures (SOPs). The type, frequency, and acceptance criteria for internal quality control checks are described in this section and in NPHE Lab technical SOPs.

11.1 Quality Control Indicators

To assess the validity of analytical data, a number of Quality Control (QC) samples are included in the measurement system. Acceptance criteria and corrective actions for these samples are defined in the technical SOPs. NPHE Lab's Quality Objective is to only report sample results from analyses that have demonstrated that the calibration is acceptable, the sample preparation process was performed correctly, and no laboratory contamination is present. If these criteria are not met, the analyst is expected to take the necessary corrective action depending on the amount of sample available, holding time remaining for the test, and turn around time (TAT) requested by the client. If the sample is out of holding time, or if there is insufficient sample volume to repeat the process, NPHE Lab will either report data acceptance with qualifying flags or request recollection of sample based on sound scientific judgment.

Acceptance criteria for Quality Control Indicators are generally defined as either the limits required by the reference method or statistically determined control limits based on data generated by the NPHE Lab. Compounds analyzed at NPHE Lab that are not listed in reference methods will use limits as stated in the analytical SOP, established from NPHE Laboratory data. Descriptions of the different types of Quality Control Indicators are given in the following sections. All controls, solutions, reagents and standards must be used prior to their stated expiration date.

11.1.1 Chemical Analyses

Blank (Blank) - This is a sample of reagent water that is analyzed when specified in the method SOPs. The calibration blank is considered to be a zero standard.

Calibration Blank (CB) – This is a sample of reagent water that is analyzed as the first point of the calibration curve, when specified in the method SOPs. The calibration blank is considered to be a zero standard.

Calibration Standard (CAL) – A solution prepared from the dilution of stock standards in which the concentration of each analyte is known. The calibration standards are used to calibrate the instrument response with respect to analyte concentration.

Calibration Stock Standard – A concentrated solution containing one or more of the method analytes prepared in the laboratory from stock standard solutions obtained from a source different than that used for the preparation of the CCV stock standards or the quality control standards. This is used to prepare the calibration standards.

Continuing Calibration Blank (CCB) – A solution, the calibration blank, which is monitored periodically throughout the analysis. The CCB is used to verify the baseline stability of the instrument during analysis.

Continuing Calibration Check Standard (CCC) – The CCC is a working standard, which is prepared from the same standards used to calibrate the instrument. The CCC is used to validate the calibration curve at the beginning of each analytical sequence, and may also be analyzed throughout and at the end of the sequence. Some procedures refer to this QC type as a Continuing Calibration Verification (CCV) or an Instrument Performance Check (IPC).

Continuing Calibration Verification Stock Standards - A concentrated solution containing one or more of the method analytes prepared in the laboratory from stock standard solutions obtained from a source different than that used for the preparation of calibration stock standards or the quality control samples. This is used to prepare the CCV or CCC.

Field Duplicates (FD1 and FD2) – Two separate field samples collected at the same time and place under identical circumstances. Field duplicates are placed in separate sample containers and treated identically throughout field and laboratory procedures. Analysis of field duplicates provides a measure of the precision associated with sample collection, shipping, preservation, storage, and laboratory procedures.

Field Reagent Blank (FRB) – Reagent water placed in a sample container in the laboratory and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.

Field Trip Blank (FTB) – Reagent water from the laboratory is poured into a sample container in the field. FTBs are treated as samples in all respects, including exposure to sampling site conditions, transportation, storage, preservation, and all analytical

procedures. FTBs are not exposed to pumping and collection equipment. FTBs are used to determine if method analytes, or interfering substances, which may affect the accuracy of the analysis, are present in the field environment.

Internal Standards (IS) – A solution of compounds, which contains pure analyte(s) with properties similar to the analytes of interest, which are added to field samples, standards, extracts, and controls at a known concentration. They are used to measure the relative responses of other method analytes and surrogates in the sample. Some methods require the use of one IS. All analytes used for the internal standard must not be present in any of the samples or standard solutions.
Internal Stock Standard (ISS) – A concentrated solution containing one or more of the internal standard analytes prepared in the laboratory from assayed reference material or purchased from a commercial source.

Laboratory Control Sample (LCS) – A sample matrix containing a solution of method analytes in a water miscible solvent which is used to fortify reagent water. The LCS is obtained from a source external to the laboratory, and is used to check laboratory performance with externally prepared test materials.

Laboratory Duplicates (LD1 and LD2) – Two sample aliquots taken from the same field sample container in the laboratory and analyzed separately with identical procedures. Analysis of laboratory duplicates provides a measure of the precision of the laboratory procedures, but not with sample collection, preservation, and storage procedures. In our laboratory a duplicate of the LFM is used for the laboratory duplicate and thus would be referred to as a duplicate LFM or DUP LFM or spike duplicate sample.

Laboratory Fortified Blank (LFB) – An aliquot of reagent water to which known concentrations of the method analytes are added. LFBs are used to determine whether the method is in control. The purpose of the LFB is to assess accuracy and precision and to determine whether the methodology is within accepted control limits. Additionally, when surrogates and internal standards are added to samples as required by method requirements, they are also added to the LFB. The LFB may also be referred to as a method standard.

Laboratory Fortified Matrix (LFM) – An aliquot of a sample to which known quantities of the method analytes are added in the laboratory. The laboratory-fortified matrix is analyzed as if it were a sample. The purpose of analyzing a LFM is to assess whether the sample matrix contributes bias to the analytical results. The background concentrations of the method analytes in the sample matrix must be determined using a separate aliquot and the determined concentration valuations in the LFM must be corrected for the background concentrations. The LFM may be referred to as a spike sample or simply a spike.

Laboratory Instrument Blank (LIB) – An aliquot of reagent water, rinse water or solvent analyzed to determine if any background contamination exists in the instrument and its components.

Laboratory Method Blank (MB) – A sample of reagent water included in the sample batch analyzed in the same way as the associated field samples. The LMB is used to determine if method analytes or other background contamination have been introduced during the preparation or analytical procedure.

Laboratory Performance Check Standard (LPC) – A sample or solution of method analytes and surrogate which contains target compounds. It is designated to evaluate the performance of their instrument system and monitor analytical performance and sensitivity with respect to a defined set of method criteria.

Laboratory Reagent Blank (LRB) – Reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, plastic ware, equipment, solvents, and reagents. The lab reagent blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

Laboratory Trip Blank (LTB) – Reagent water is placed in a sample container in the laboratory and treated as a sample, including storage, preservation, and all analytical procedures. The laboratory trip blank container follows the collection bottles to and from the collection site but, the LTB is not opened at any time during the trip. The laboratory trip blank is primarily a travel blank used to verify that the samples were not contaminated during shipment.

Laboratory Matrix Spike/Laboratory Matrix Spike Duplicates Samples (LFM/LFMD) – Aliquots of an environmental sample to which known quantities of certain method analytes are added in the laboratory. The purpose is to demonstrate recovery of the analytes from a sample matrix to determine if the sample contributes bias to the analytical results and to demonstrate the reproducibility of these results.

Proficiency Testing (PT) Sample – Solution containing unknown quantities of various regulated and unregulated analytes which is provided from an outside vendor (e.g., RTC, Environmental Resource Associates, etc.). The identification and concentrations of the analytes are unknown to the analysts. PT samples must always be analyzed in the same manner and the same number of times as all field samples. NPHE Lab participates in a minimum of at least one and preferably two Drinking Water PT sample studies (WS) each year and at least one and preferably two Waste Water PT sample studies (WP) each year for each analyte and method performed by the lab.

Quality Control Assessment Sample (QCAS) – Any sample in which the determined analyte concentrations are used to assess the quality of the data.

Quality Control Standard or Sample (QCS) – Solution containing known concentrations of analytes prepared by a source external to the laboratory performing

the analysis and different from the source of the calibration standards. The QCS sample is analyzed using the same procedures as field samples. The QCS is used as a check on the calibration standards used in the method on a routine basis. The QCS is analyzed at least quarterly unless the method SOP requires a more frequent usage. The QCS may also be referred to as an external control sample or a known reference sample. Determining if the analytical results are within the acceptable control limits assesses the data quality

Stock Standard Solution - A concentrated solution containing one or more of the method analytes prepared in the laboratory from assayed reference material or purchased from a commercial source.

Surrogate Standard or Analyte (SS) or (SA) – Compound with properties similar to the analytes of interest that is added to the sample before sample preparation, extraction, or other processing and is measured with the same procedures used to measure other sample components. The SS is used to evaluate the efficiency of the sample preparation process and to monitor method performance with each sample.

Tuning Solution – Any solution composed of specific analytes that is used for tuning, optimization and/or calibration of instrument components prior to the analytical procedure. A tuning solution is also used to determine acceptable instrument performance prior to calibration and sample analysis.

11.1.2 Microbiological Analysis

This section summarizes some of the Internal Quality Control checks and the procedures which are performed in the microbiology laboratory. The type of QC sample, frequency, acceptance criteria and corrective action for QC failures are defined in the applicable microbiology method and SOPs.

Positive Growth Controls - Analyzed to demonstrate that the medium can support the growth of the test organism, and that the medium produces the specified or expected reaction to the test organism. It also shows that conditions in incubators, water baths, etc. are accurate for growth of the test organism.

Blank Control – An uninoculated control used to demonstrate that samples have not been contaminated by sample handling, preparation, or environmental exposure.

Negative Growth Control – Analyzed to demonstrate that the media does not support the growth of non-target organisms.

Autoclave – Each time the autoclave is used, the analyst records the date, contents, sterilization temperature, cycle time, sterilization time and pressure. Their initials are recorded with this data. A maximum temperature thermometer or continuous temperature recording device is used during every cycle to ensure that the proper temperature was reached and recorded. Spore strips or autoclave indicator tape is used

during every cycle to confirm sterilization. Automatic timing devices are checked quarterly and the temperature gauge is calibrated and tagged annually.

Culture Dishes – Sterility checks are performed on each new lot number.

Culture Tubes, Containers and Closures – Sterility checks are performed with new lot number.

Sample Containers – At least one sterile sample container is selected at random for sterility check from each lot of commercially prepared or laboratory prepared sample containers. The sterility is confirmed by adding approximately 25 ml of sterile non-selective broth (e.g., Tryptic Soy Broth.)

Glassware, Plastic ware and Metal Utensils – Sterility checks are conducted for plastic ware with each new lot number. An inhibitory residue test is performed with each new detergent, following standard methods procedure. Each batch of clean glassware is tested for acid or alkaline residue with bromothymol blue indicator.

Rinse Water – Each batch that is prepared is checked for sterility by adding 50 ml of water to a 50 ml volume of double strength non-selective broth (e.g., tryptic soy or nutrient broth). The solution is incubated at 35° +/- 0.5 C for 24 & 48 hours and checked for growth.

Analytical Media – Each batch is checked before use for sterility, pH, positive and negative growth controls. These checks are documented as follows:

(a) **Laboratory media**: Preparation is documented in a reagent log book to include: media manufacturer, type of media, lot number, date of preparation, time-in, time at sterilization temperature, time-out, total time autoclaved/boiled, temperature attained, final pH, positive/negative growth control results, sterility check, and date of expiration. The analyst's initials are also recorded.

(b) **Commercial Media documentation includes**: the media manufacturer, lot number, type of media, date of preparation, date of receipt, final pH, positive/negative growth control results, sterility check, date of expiration and the analyst's initials.

Lab Reagent Water – The reagent water is checked annually for nitrate, fluoride and metals. Additionally, the reagent water is tested monthly for pH, conductivity, free chlorine residual and HPC.

Thermometer – Thermometers are graduated in at least 0.5°increments (usually 0.1°increments for incubators) for uses at temperatures other than 44.5 °C and graduated in at a minimum of at least 0.2°C increments for tests which are incubated at 44.5°C. Thermometers are calibrated at least once a year with a NIST thermometer.

11.2 Method Detection Limits (MDLs)

Each Method Detection Limit (MDL) is defined as the lowest concentration which can be measured with a 99% confidence level for a given analytical method and sample matrix. MDLs are determined in accordance with the requirements of 40 CFR Part 136 Appendix B. A standard deviation and calculated 99% confidence level are usually determined by the analysis of seven replicate reagent water spikes fortified with the analyte(s) of interest. The determination of MDLs is performed on a frequency based on the analytical method requirement, and on all instruments used in the analysis. In the absence of any method requirement, MDLs will be performed annually. The results of all MDL studies are maintained by the area analysts and by the QA Office. The applicable technical SOPs include procedures and acceptance criteria for MDL studies. All analysts are responsible for maintaining current MDLs and IDCs for their areas of responsibility.

11.3 Reporting Limits (RL)

The NPHE Lab establishes a reporting limit (RL) for each analyte, as the lowest analyte concentration that can be quantified and reported by the laboratory. The RL is set at or above the lowest calibration standard used in all analyses or is verified by a LLLFB. Valid and current MDLs must support RLs. The RL is usually set at 5 to 10 times the MDL value and/or at detection limits required in the MCLADW (ex. VOCs). RLs are typically set below 1/5 of the MCLs set by EPA. RLs can be found in the LIMS, and/or the lab method SOP.

12. QC Tracking

QC tracking is currently completed by using Excel folders or by a NPHE Lab LIMS process. It is used to monitor the performance of quality control samples through the use of accuracy and/or means control charts, using at least one LFB in each test batch. Some analysts may also track LCSs and/or LFM recovery with QC charts. A change in QC results usually indicates that some variable in the test has changed. The method acceptance limits must be the control limits from the historical data provided by control chart or the control limits of the method, whichever is tighter.

Results must be charted on a routine basis, so that early detection of trends, shifts, or out-of-control situations can be identified and corrected. QC charting helps the analyst identify the variables that affect the precision and accuracy of the test method. The analyst must attempt to identify these variables and correct or control the variables when the QC results fall outside the established acceptance limits. When an outlier is suspected, the analyst must take a critical look at all aspects of the measurement process to determine if the data produced is valid and defensible.

Details for QC charting and handling QC failures are documented in each technical SOP. The NPHE Lab system for documenting data quality and QC charting corrective action is described in the current version of NPHE Lab *SOP # 8200, Documenting Data Quality* and NPHE Lab *SOP # 8500.1 Corrective Action Documentation and Procedures.*

The method limits are used to set the initial upper and lower control limits prior to the generation of in-house limits and when the established limits exceed the method limits.

13. Holding Time Policy

The applicable holding times for all analytical methods are included in the NPHE Lab Collection and Preservation Tables. This table can be found in Appendix O. The analysts are responsible for recognizing the time left in the sample or extract holding time. Samples are tracked in the database using the time and day that the samples were collected, extracted and analyzed, to ensure that the information can be provided to the client if any holding times are exceeded. The holding time requirement is typically measured from the date and hour of sample collection, and is figured by the LIMS automatically.

If the holding time requirement in 40 CFR, (method hold time), is expressed in days, then the holding time in the laboratory is calculated in calendar days from the day of sample collection. If the holding time requirement is expressed in hours, then the holding time in the laboratory is calculated in hours from the day of sample collection. For example, samples with a 14-day holding time and which are collected on 4/1/04 are within holding time, if analyzed anytime before the end of the 14th day from sample collection or midnight on 4/15/04. Samples with a 48-hour holding time which were collected on 4/1/04 at 2:00 p.m. are within holding time if analyzed anytime before 2:00 p.m. on 4/3/04. If the holding times are exceeded, the analyst must notify the Laboratory Manager and/or QA Manager. The analyst must also flag the data so that any holding time violation is clearly stated on the Result Report for the client.

The NPHE Lab will adhere to other sample holding time requirements when specifically requested or required in writing from clients or regulatory agencies.

All total coliform samples that exceed the 30 hour holding time **must be rejected** and a new sample must be requested.

14. New Method Development and Validation

Any work on a New Test Method requires approval by the Laboratory Manager and the QA Manager before work begins. The assigned analytical area is responsible for defining the analytical test method, writing the draft SOP, identifying collection and preservation requirements, and performing at a minimum, an acceptable IDC, MDL study, and a blind QC sample. Both the Laboratory Manager and QA Manager will then review this data.

Upon approval by Management, the appropriate documents are created (or updated) to include the requirements for sample collection and preservation, receiving, analysis and reporting. The appropriate reporting documentation and formats are created.

A price for the new test is established by the Laboratory Manager and/or QA Manager and approved by the NDHHS Environmental Health Unit Administrator. All data and documentation for the new test are provided to the QA Office. The Laboratory Manager ensures that appropriate documents for shipping, sample collection, preservation, sample receipt, and analysis are

updated. After completion of all these steps, the test is given a unit price and entered into the NPHE Lab database as an orderable test.

15. Performance and System Audits

Quality assurance audits are conducted to verify conformance with the NPHE Lab's Quality Assurance Program, to evaluate the effectiveness of the QA Program and to take the appropriate actions to continually improve the Laboratory Quality Assurance Program. Audits are conducted by either NPHE Laboratory employees (internal), or by other parties (external). Both internal and external audits are used to evaluate the laboratory's performance or systems.

Performance audits are conducted to assess the quantifiable aspects of the laboratory's processes. Performance can be assessed in a very objective manner, since the performance either conforms or doesn't conform to prescribed procedures or policies. Some of the most common ways to evaluate performance are by determining adherence to an SOP, ability to produce acceptable (passing) analytical results, or by obtaining acceptable results in PT studies.

System audits evaluate the operational details of the laboratory's QA Program. A system audit is more subjective in nature. The purpose of the system audit is to determine if the laboratory's overall quality system is effective in ensuring compliance and conformance to the Quality Objectives.

The QA Team randomly selects data packets and client reports and performs a quality review. This is in addition to the peer review that is performed in the laboratory. Internal performance is also audited as part of Internal System Audits.

15.1. Internal System Audits

The design of the Internal System Audit is intended to assure that all areas of the laboratory are audited annually. These audits consist of reviews of applicable laboratory systems, procedures, and documentation. The QA Manager with the help of the laboratory staff oversees the annual Internal System Audits. Various NPHE Lab staff members are assigned to perform the internal audits. An Internal Audit Schedule is prepared by the QA Manager. The Internal Audit Procedure is described in the current NPHE Lab *SOP # 8400.1, Audits and On-site Evaluations.*

15.2 Audit Response

Response to deficiencies found in any audit, is the responsibility of the appropriate method Primary analyst and the Laboratory Manager. Responses are compiled and documented, and forwarded to the QA Manager in writing. The QA Team and QA Manager, coordinates the necessary activities to ensure that any problems are clearly identified, the effect on analytical results is determined, and those clients whose samples were affected are identified. The QA Manager and Laboratory Manager will review this information and determine if the findings require any additional corrective action. Where the audit findings cast doubt on the

correctness or validity of the laboratory's test results, the laboratory must take immediate corrective action and must notify, in writing, any client whose data may have been affected.

15.3 PT Studies

The laboratory will perform and pass at a minimum at least one and preferably two PT studies for each method/analyte in any 12 month period. The laboratory must pass at least one WS PT for each drinking water method and at least one WP PT for most of the waste water methods they are analyzing samples for. Each PT sample must be treated the same as all laboratory samples are treated. They may not be handled in a different manner than any other laboratory sample. The primary analyst should complete the first PT study of the year and if it is passed the secondary analyst should complete the second PT study of the year. If the analyst misses two out of three PTs, they must pass two corrective action PTs in a row, at least 15 days apart or from two different vendors to continue testing routine samples for that method and analyte.

A PT is considered unacceptable in the following situations:

- The PT sample is for a single analyte and that analyte is not within the acceptable range.
- The PT sample contains multiple analytes and each analyte is reported as a single result. Such as each metal included in the Metals PT vial or each analyte in a Nutrient PT vial. Each analyte that is reported out of acceptable range is unacceptable.
- The PT sample is for a specific test method that contains multiple analytes that are evaluated together as a test method. This includes some of the organic methods such as VOC's, SOC's, and HAA's. These contain both regulated and unregulated compounds. Missing one regulated analyte is unacceptable unless the 80% rule is acceptable. In this case missing several regulated VOC's or SOC's is acceptable as long as the same analytes are not consistently missed. Exception to this rule: all analytes in THM PT's must be within acceptable ranges and Vinyl Chloride must be within acceptable range each PT ran.
- For microbiological methods using presence or absence in a sample, each PT set should contain 10 samples. To be acceptable, the lab must analyze a minimum of 9 out of 10 samples with no false negative results. One false positive result is acceptable however by EPA.

The QA manager will start a Corrective Action Report CAR) for each unacceptable PT. It is up to the QA manager and/or Lab manager to determine if a missed PT result is unacceptable and what follow-up actions if any must be taken by the analyst. In all cases, a CAR must be completed to acknowledge and document that a PT result reported to a PT vendor was unacceptable. The completed CAR must include the process taken to discover the cause of the error, a description of the error, and what steps are being taken to eliminate it from happening again.

16. Corrective Actions

The laboratory will take corrective action whenever unacceptable conditions exist. Corrective Actions may be necessary for many different reasons. The following indicators may be used to determine unacceptable conditions:

- QC samples outside of established acceptance criteria
- Calibrations outside acceptable criteria
- Equipment failure
- Deficiencies identified during internal audits, blinds, or spot checks
- Deficiencies identified during EPA on-site audits
- Deficiencies or problems identified after receiving a complaint
- PT failure
- QC chart outliers and trending

Once an unacceptable condition is identified, the laboratory will investigate the problem and outline a corrective action plan.

Corrective action may include any of the following:

- Re-analysis of samples
- Re-calculation of results
- Re-calibration of equipment
- Preparation of new standards, reagents, spikes, or other controls
- Re-analysis of blanks
- Dilution of samples
- Additional analyst training
- Replace equipment or supplies
- Re-sampling
- Validating a new technique

The extent of action necessary to correct a problem varies greatly. In some cases, the corrective action may be as simple as reanalyzing samples that were originally contained in an analytical batch with failed QC. Other times, the action may be as complicated as purchasing new equipment. See *SOP 8500.1 Corrective Action Documentation and Procedures* for additional information regarding the Corrective Action process and documentation.

16.1. Immediate Corrective Actions

Immediate corrective actions may be necessary because of an increased failure rate of analytical QC measurements on a particular instrument. In this situation, the analyst takes action to restore the instrument to acceptable working condition by performing maintenance or initiating a service call. The instrument must be put out of service until such time as it has been repaired and certified in good working order. An out of order tag must be placed on all out of service equipment until it is repaired. Written documentation must be available, which validates that the equipment has been repaired and that it has passed calibration requirements prior to being used for testing. All necessary repairs and part replacements must be

documented in an Instrument Maintenance Log. The instrument is not used to generate sample data until acceptable performance is documented. Any analyst as well as the QA Manager and/or the Laboratory Manager have the authority to stop further analyses until the issue is resolved.

Data generated with out of control QC measurement(s) are evaluated for usability in light of the nature of the deficiency. In any event, the QA Manager and/or Laboratory Manager must be notified of the problem, so it can be discussed. If the deficiency is judged not to impair the usability of the results, the data may be reported and the QC failure is noted in the report. In cases where the samples associated with the QC failure are for regulatory compliance, such qualified data may not be acceptable. When such data cannot be reported, corrective action includes sample reanalysis, if sufficient sample remains and it is still within holding time. Otherwise recollection may be the only option.

16.2. Long Term Corrective Action

Long term corrective action is generally initiated to address Quality Assurance issues that are often identified through internal or external audits. These involve more detailed investigation into the root cause of the nonconformance, and may take longer to resolve. Staff training, SOP revisions, or equipment replacement may be indicated as solutions for long term corrective action.

16.3. Corrective Action Report

A Corrective Action Report (CAR) is the mechanism by which an employee can identify a quality deficiency and document the necessary steps to remediate the problem or propose an improvement to current procedure. Any employee can initiate a CAR. When a CAR is initiated, the QA Manager or Laboratory Manager designates a responsible person. The designated person is responsible for constructing a corrective action plan, and setting a target date for completion of the action plan. The QA Manager reviews the finding and the corrective action plan, and either approves or denies the plan.

If the report and corrective action plan are approved, the plan is implemented as soon as possible. The QA Manager oversees the progress of the corrective action, but it is the responsibility of the designated person and the Laboratory Manager to ensure completion of the action. After the completion of the action, the system is reevaluated to determine the effectiveness of the corrective action. When the action is determined to be complete and effective, the CAR is closed. A NPHE Lab Corrective Action Report Form (CAR) is included in *Appendix T.*

16.4. Preventive Actions

When applicable, a preventive action plan is required as part of a corrective action request. The plan will state the steps that will be implemented to ensure that the deficiency does not reoccur. Other types of preventive actions are those taken to avoid QC failures in analytical

batches. Specific actions necessary for each analytical procedure may be listed in the technical SOP.

17. Document Control

Activities and information that have been identified as essential to the operation of the NPHE Lab are documented and included in the formal Document Control System to ensure their integrity. The Document Control System ensures that information or instructions are documented in a standardized format, receive management approval when created and revised, are issued to the appropriate personnel, and that previous revisions are removed from circulation. Effective control over the documents to prevent the use of incorrect or of out-dated information is critical. Annual review and/or revision of these documents are conducted. Annual performance audits are performed to ensure that these documents are accurate, understood, and followed by the appropriate personnel.

17.1 Revision of Controlled Documents

The QA documentation begins with the QA Manual. The next level of documents is the QA System SOPs, which utilize any number of additional controlled documents to make the system functional, efficient, and affective. The next level of documents consists of technical and non-technical SOPs. The lowest level of documents consists of tables, (e.g. QC tables, Collection/Preservation Tables) computer spreadsheets, and forms.

The Master SOP Table of Contents (Appendix Q) identifies all of the approved NPHE Lab SOPs. A unique number identifies each SOP. The Master SOP Table of Contents displays the SOP number, the method number, the effective date, the last revision date, and the author of each SOP. The Master SOP Table is maintained by the QA Manager on the computer shared drive in the current year QAP folder. A current copy of the table can be found in the front of the first Notebook of the Official Lab SOPs located in the Lab Manager's Office.

The current official signed SOP is always on file in the Lab Manager's office. Copies of SOPs are available in the appropriate departments. All SOPs are also available on the shared drive of the Lab computer network. SOPs are reviewed annually by the primary analyst and/or other individual responsible for those specific documents and the QA manager and/or Laboratory Manager. Documentation of the review process is maintained with the original SOP in the Lab Manager's office.

All administrative SOPs are reviewed by the QA Manager and the Laboratory Manager yearly. All Lab staff must then document that they have read the Administrative SOPs and the documentation added to their training file in the Lab Manager's office.