Quality Assurance Plan

Version 6

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SECTION 1.0: STATEMENT OF QUALITY ASSURANCE POLICY

1.1. The objective of the National Center for Water Quality Research (NCWQR) is to accurately characterize the concentrations of sediments, nutrients, and pesticides in natural waters, particularly in relation to non-point pollution from agricultural, urban, and forested land-use activities; to accurately characterize nutrient, sediment, and pesticide loading into lakes and/or export from watersheds of varying sizes; and to assess changes in concentrations and loading of non-point pollutants in response to changing management practices and/or climate.

1.2. To ensure the highest quality of data, all methods and procedures when specified by a study plan will adhere to the current Quality Assurance Plan (QAP).

SECTION 2.0: LABORATORY ORGANIZATION AND RESPONSIBILITY

2.1. The NCWQR consists of employees classified as either research scientists or laboratory personnel. In addition, as the NCWQR is located at Heidelberg University, Tiffin, Ohio, student interns and student employees (hereafter called Interns) are utilized during the academic year and summer session. The organizational structure for the laboratory is shown in Figure 2.1.

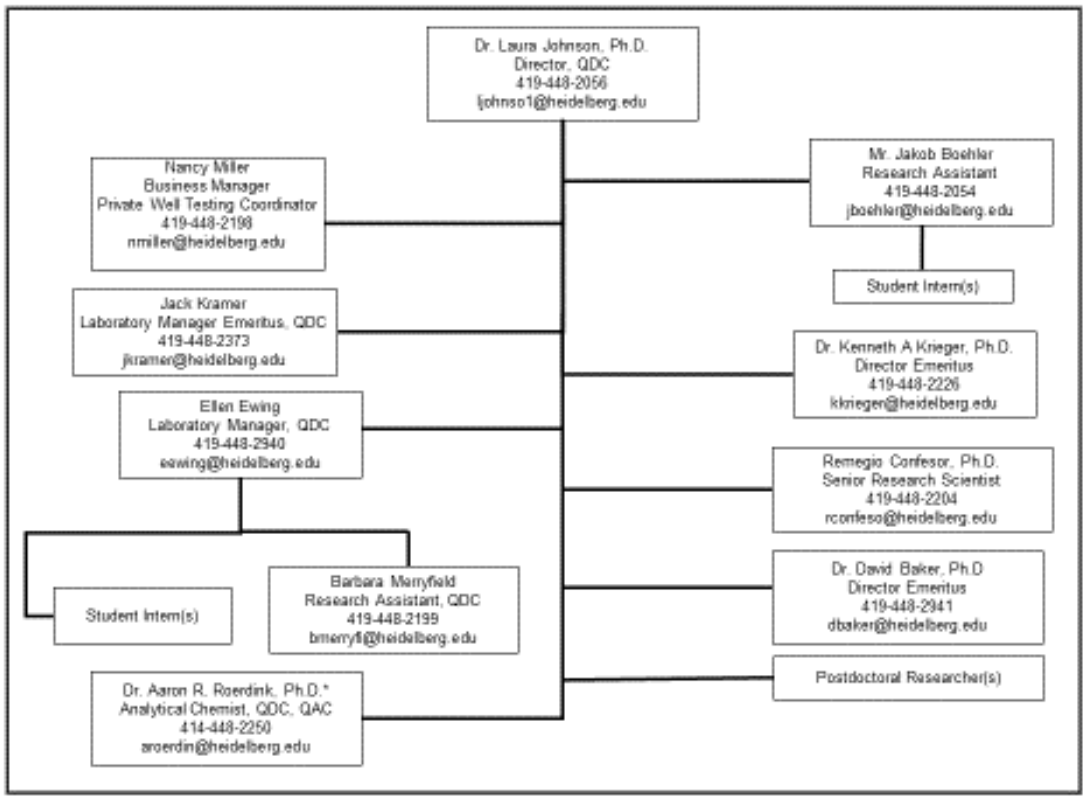

Figure 2.1
Organizational Chart

For the purposes of specific study plans, members of the NCWQR may be given alternative designations that are independent of their position within the NCWQR. These designations are indicated within the specific study plan.

2.1.1. The Director and research scientists are responsible for overall interpretation and presentation of data generated by the laboratory

2.1.1.1. Director: Oversees all laboratory functions. Calculates loads and flow or time weighted mean concentration. Manages and procures funding for the program as well as completes or delegates project reports. Seeks out funding and manages projects that examine reasons for patterns found in the long-term data collection in the laboratory. Emeritus designates past directors that provide assistance as needed.

2.1.1.2. Senior Research Scientist: Analyzes the data for watershed patterns and uses the information to develop watershed models. Calculates loads and flow or time weighted mean concentration. Conducts watershed scales mass balances comparing nonpoint and point sources of pollutants to the watershed. Seeks out funding and manages projects that examine reasons for patterns found in the long-term data collection in the laboratory.

2.1.1.3. Research Scientist: Independently leads and also assists the Senior Research Scientist and Director in all aspects of data interpretation and calculations. Helps conduct funded research projects and seeks out funding opportunities.

2.1.1.4. Post-doctoral Researcher: Assists the Senior Research Scientist, Research Scientist, and/or Director in data interpretation and calculations.

2.1.2. Laboratory personnel are responsible for ensuring successful analysis of all water chemistry and storage on laboratory databases.

2.1.2.1. Laboratory Manager: Oversees laboratory operations; manages new samples or method development; helps the QAC (see 2.1.3) ensure data meet QAP standards; assists with sample collection and field station maintenance; assists in sample receiving, login, chain of custody, and sample preparation; conducts specific analyses following all SOP requirements; and trains and supervises research assistants and interns.

2.1.2.2. Research Associate: n/a

2.1.2.3. Research Assistant: Assists the Laboratory Manager with sample collection and field station maintenance; assists in sample receiving, login, chain of custody, and sample preparation; conducts specific analyses following all SOP requirements; ensures data entry in to the database and maintains backup hardcopies of all data; and trains and supervises interns.

2.1.2.4. Interns: Assist the Laboratory Manager and Research Assistants in laboratory operations including sample preparation and analysis, sample collection and field station maintenance, and other miscellaneous tasks to keep the laboratory clean and organized.

2.1.3. NCWQR members who have retired, but work on a part-time basis, are titled with “emeritus” and are considered consultants in their capacities listed above.

2.1.4. At least one member of the NCWQR must hold the designation of Quality Assurance Coordinator (QAC). It is the responsibility QAC to ensure the implementation of this QAP and its various aspects stated herein. Additional

members may be designated as a QAC to assist in QAP implementation and Quality Control (QC) Data review; however, a Lead QAC must be distinguished. The Lead QAC is represented by a * on the organizational chart.

2.1.5. At least one member of the NCWQR must hold the designation of chemical Qualified Data Collector (QDC). Additional members may also be designated a QDC, but one QDC will be given the designation of Lead QDC (for the Lead QDC, see the relevant study plan). It is the responsibility of the Lead QDC to report data to the Surface Water Volunteer Monitoring Program as outlined by the Ohio Environmental Protection Agency.

2.1.6. One member of the NCWQR must hold the designation of Laboratory Manager. It is the responsibility of the Laboratory Manager to oversee testing in the Analytical Laboratory.

2.2. Training, education, experience, and a position description are maintained by a QAC for each employee in a personnel file. These files (hereby called Personnel Files) are stored in Gillmor Science Building Room 327 (hereafter referred to as Gillmor 327) and will be maintained for a minimum two years after the individual is no longer employed by the NCWQR. Any additions or changes to a Personnel File may be completed by using the form in Appendix A or annotating the CV.

2.2.1. Curriculum vitae (CV) and a position description for each member of the NCWQR will be stored in the individual's Personnel File. The CVs will be updated yearly with a notation by the QAC if no changes are made. Additions by members to their CV may include, but are not limited to, completion of degree, completion of short course, published papers, presentations, etc.

2.2.2. Personnel File Forms will be stored in the individual's Personnel File. Personnel File Forms document the date, data, certificates, etc., from successful completion of training. (See Appendix A, Form 1)

2.3. Training procedures for new employees are outlined within each method's SOP or the QA-SOP.

2.3.1. An analyst who has demonstrated competency for a specific method by successfully completing the Demonstration of Performance outlined in the QA-SOP is considered "trained". Any analyst who has yet to complete the performance assessment must work under the supervision of a trained analyst.

2.3.2. Individuals working within the NCWQR who have not completed training for a specific method (e.g., Interns) must work under the supervision of a trained NCWQR member when executing part of an analysis that is covered by the QAP, SOP, or QA-SOP. All work requiring a signature or initials will be signed by both the Intern and trained NCWQR member.

SECTION 3.0: DATA QUALITY OBJECTIVES

3.1. Data quality objectives such as, but not limited to, accuracy, precision, and reporting limits are listed in each method's Standard Operating Procedure (SOP). Evidence that data quality objectives are met must be produced at least once a year for each SOP and are outlined in the QA-SOP.

3.2. Specific data quality objectives may be set by a study plan, but the objectives must be within the ranges stated in the SOP or QA-SOP.

SECTION 4.0: ANALYTICAL METHODS AND LABORATORY EQUIPMENT

4.1. Analytical methods covered by the QAP are listed in Table 4.1

Instrument Name	Description	Manufacturer	Model Number(s)	Analyte(s)	EPA Method
AAIII	Segmented Flow Injection Analyzer	Seal Analytical	AutoAnalyzer III	TP	365.1
AAIII	Segmented Flow Injection Analyzer	Seal Analytical	AutoAnalyzer III	TKN	351.2
AAIII	Segmented Flow Injection Analyzer	Seal Analytical	AutoAnalyzer III	SRP	365.2
IC	Ion Chromatograph	Thermo Scientific	DX320, ICS2000	Nitrate, Chloride, Sulfate	300.1
Balance	Balance	Mettler	AB204, AE163, P163, PB303, PE3000, PG,3001, PJ6000	Suspended Solids	160.2

Table 4.1 Analytical Methods

4.2. Variances from the stated EPA methods in Table 4.1 along with any justifications can be found within a specific study plan.

4.3. Reagents are analytical reagent grade or equivalent unless otherwise specified in the method SOP.

4.4. Laboratory oven, muffle furnace, autoclave and refrigerators temperatures are verified semi-annually with a NIST-certified thermometer. Daily refrigerator temperatures and all verifications are recorded in a laboratory log.

4.5. Verification of analytical balance calibration is performed using a set of check weights each day before sample measurement. Each check weight is compared to a certified weight on an annual basis. Results of all verifications are recorded in a laboratory log.

4.6. A schematic of the Laboratory Floor Plan is exhibited in Appendix B.

SECTION 5.0 SAMPLE RECEIPT AND CHAIN OF CUSTODY

5.1. Sample collection requirements are specified in the individual Study Plans.

5.2. Sample storage requirements and holding times are specified in each method's SOP.

5.3. Chain of Custody requirements can be determined using the following criteria:

5.3.1. Samples collected by NCWQR personnel to comply with the Tributary Loading Study Plan will be accompanied by Form 1, Appendix C.

5.3.2. Samples collected by persons other than NCWQR personnel to comply with the Tributary Loading Study Plan will be accompanied by Form 2, Appendix C.

5.3.3. Samples received for surface water analysis, other than those covered by the Tributary Loading Study Plan, must be accompanied by Form 3, Appendix C.

5.4. Chain of Custody documents will be retained in the appropriate file in Gillmor 327 for a minimum of 2 years.

5.5. Samples will be rejected if the sample container has been damaged so that a representative sample cannot be obtained or if the handling of the sample does not meet the requirements of the appropriate Study Plan. Clients will be notified of any sample rejection and may provide a resample if desired.

5.6. The NCWQR reserves the right to reject any sample deemed inappropriate for analysis.

5.7. Samples will be disposed in an appropriate manner. In most cases surface water samples are poured down the drain. Samples used for analysis are disposed in the manner stated in the SOP.

SECTION 6.0: STANDARD OPERATING PROCEDURES

6.1. All analyses covered by this Quality Assurance Plan (QAP) will follow the method's Standard Operating Procedure (SOP). The SOP will contain at a minimum the requirements for equipment preparation, sample preparation, sample cleanup, sample analysis, and routine performance verification procedures. For all current SOPs see the Master File in Gillmor 327 or controlled copies in Gillmor Science Building Rooms 304 and 319 (hereafter referred to as Gillmor 304 and Gillmor 319).

6.2. SOPs for each analytical method are based on published procedures from regulatory agencies (e.g., US EPA). The published procedure will be referenced in the appropriate section of the SOP. There are no variances from the published procedure for any analytical method with an SOP. Note: See project study plans for additional information.

6.3. Additional performance verification of a method that is not a part of daily routine testing outlined in the SOP (e.g., Method Detection Limit (MDL), Demonstration of Performance, precision, accuracy, etc.) will be covered in the Quality Assurance Standard Operating Procedure (QA-SOP). For all current SOPs see the Master File in Gillmor 327 or controlled copies in Gillmor 304 and Gillmor 319.

SECTION 7.0: CALIBRATION PROCEDURES

7.1. Calibration type and frequency are specified within each method's Standard Operating Procedure (SOP).

7.2. All calculations for calibration are performed electronically by the software of the instrumentation (see Section 10) and verified by the analyst in accordance with the SOP.

SECTION 8.0: PREVENTATIVE MAINTENANCE AND DOCUMENTATION

8.1. All instrument manuals are located in a designated area near the specific instrument or in Gillmor 327.

8.2. Preventative maintenance and documentation procedures are specified within each method's Standard Operating Procedure (SOP).

8.3. If possible, at least one spare or "back-up" part for routinely replaced components will be kept in the laboratory. It is the responsibility of the analyst to inform the Laboratory Manager when the "back-up" has been used.

8.4. Any maintenance contracts for equipment covered by the QAP will be stored in the designated file in Gillmor 327.

8.5. Any new equipment warranty documents covered by the QAP will be stored in the designated file in Gillmor 327.

8.6. All balances are serviced once a year by an approved service agency (Table 8.1). Balance test confirmation paperwork is stored in Gillmor 327.

Approved Agencies	Address	Telephone No.
Mettler Toledo	1900 Polaris Parkway Columbus, OH 43240	1-800-638-8537

Table 8.1
Balance Service Agency

SECTION 9.0: INTERNAL QUALITY CONTROL

9.1. Internal quality control checks, their frequency, and the criteria for acceptability are specified within the Quality Assurance Standard Operating Procedure (QA-SOP).

9.2. Proficiency Testing (PT) for each method will be completed at least semi-annually. Samples containing required analytes are provided by an externally accredited source (see Section 15.0). Analytical results are submitted and accuracy is determined. Results of the PT studies are stored in the appropriate file in Gillmor 327.

9.3. Independent quality control standards will be prepared under the supervision of a Quality Assurance Coordinator (QAC) or purchased from an external supplier and analyzed on at least a quarterly basis. Results will be stored in the appropriate file in Gillmor 327 or on separate electronic "server-six$" and reported to the Laboratory Manager and/or electronically by the QAC.

9.4. Quality control performance parameters (e.g., Method Detection Limits (MDLs)) must be performed at least once a year and are specified in the QA-SOP (hereafter called the Demonstration of Performance). When completed, the SOP and any other documentation specified by the SOP will be updated with the most recent data (hereafter called Validation Data).

SECTION 10.0: DATA REDUCTION, REVIEW, AND REPORTING

10.1. Details regarding data calculation and reduction are listed within the method's Standard Operating Procedure (SOP). All calculations are performed electronically by the software of the computer that operates the instrument. Sample information is then transferred from the instrument computer to the laboratory's main database, the VAX system.

10.2. Data is retained by the laboratory in the form of a paper copy and an electronic version stored on the VAX system. The paper copy of the data is the primary method of data retention and stored in the designated file in Gillmor 327. The electronic data stored in the VAX system is backed-up at least bi-weekly to a disk format and stored in Campus Center 125.

10.3. Quality Control Data is collected for each method according to the Quality Assurance-Standard Operating Procedure (QA-SOP) and is reviewed by a Quality Assurance Coordinator (QAC) on a 3 or 4 week basis.

10.3.1 Primary responsibility for Quality Control Data resides with the analyst and is collected on a weekly basis. Data are added to Control Charts and examined to determine if the given system is out of control.

10.3.2 Review of the Quality Control Data by a QAC for the previous weeks' data must be completed with 2 weeks of the weeks collected. The QAC will report the findings of the review, including any deficiencies, to the Laboratory Manager (See Section 13.0).

10.3.3 Control Charts are stored in the appropriate file in Gillmor 327 or on separate electronic "server-six$" that is maintained by the Heidelberg University Computer Network Information Technologies department.

10.3.4 If any deficiencies are found by the QAC in the Quality Control Data (i.e., a system that is out of statistical control, etc.), the deficiencies will be brought to the attention of the Laboratory Manager and any applicable analyst. Corrective action measures will be taken until the deficiencies are corrected (see Section 11.0).

10.4. Sample data for each method is reviewed by the QAC on a quarterly basis. Data are extracted from the River file on the VAX, reviewed for completeness and errors, and corrected as needed. All corrections are documented in a correction log on server.

10.4.1. Primary responsibility for experimental data review resides with the QAC.

10.4.1.1. Using plotting software (e.g., Excel or Datadesk), individual and/or multiple parameters for each location are visually displayed so that disparate data points or "outliers" can be identified.

10.4.1.2. Questionable data are investigated by researching original data input to discover the possible source of the error or omission. Some accurate data that do not conform to expected patterns may appear to be "outliers".

10.4.1.3. If the error can be readily explained and documentation is available with the correct entry, the correction (if necessary) is made in the VAX database and the corrective action is documented.

10.4.1.4. If the error cannot be readily explained and/or documentation is unavailable, the questionable data are brought to the attention of the Laboratory Manager. A meeting between the laboratory manager and QAC will be conducted to determine the course of action. In most cases, the data will be retained in the VAX, but may be replaced with "-9" in any working databases to indicate missing data, and the corrective action will be documented electronically as "comments" in the data file.

10.4.1.5. Copies of corrected VAX data sets are added to existing working database servers for use by research scientists and educators. Working databases are updated at least quarterly by the QAC.

10.4.2. It is the responsibility of a QAC to update the appropriate files with the new experimental data (see Section 9.0).

10.4.3. It is the responsibility of the designee in the appropriate Study Plan to report data generated within the specifications of the QAP to the appropriate agency (e.g., QDC to the Ohio EPA).

SECTION 11.0: CORRECTIVE ACTION PROCEDURES AND CONTINGENCY PLAN

11.1. Corrective action procedures for quality control failures can be found within each method's Standard Operating Procedure (SOP).

11.2. If the NCWQR laboratory is unable to perform quality analyses for any reason, samples will be forwarded to an appropriate contract laboratory for analysis.

SECTION 12.0: AUDITS, ACCREDITATIONS, AND CERTIFICATION

12.1 All current accreditations and certifications and their durations are represented in Table 12.1.

Summary
Ohio Environmental Protection Agency -- Department of Environmental Services Audit and Successful Completion December 2007

Table 12.1
NCWQR Accreditations and Certifications

12.2 Internal auditing of the Quality Assurance Plan (QAP) is undertaken during the yearly review of the QAP, SOPs, and QA-SOP (See Section 14.5) by at least one Quality Assurance Coordinator (QAC) and any other assigned members of the NCWQR.

12.3 A QAC may audit the NCWQR at any time to ensure the proper implementation of the QAP. If any deficiencies are found, they will be corrected in a timely fashion and noted in the monthly report from the QAC to the Management (see Section 13.0).

12.4 As indicated in Section 9.0, PT samples are purchased from approved vendors (see Section 15.3) and tested as samples according to the appropriate SOP.

12.4.1 Samples may be diluted given the possible concentrations to ensure they are within the routine working range of the instrument.

12.4.2 After completion of the analysis, results are reported to the PT agency by a QAC.

12.4.3 Acceptable performance is determined by comparing the NCWQR laboratory result to the Acceptance Range for the analyte.

12.4.3.1 For PT samples from the Water Supply Program (WS), the acceptance range is calculated based upon the United States Environmental Protection Agency Nation Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). The acceptance limits are defined as ± two standard deviations from the estimated mean. See Table 12.2 for an example.

Analyte	Reported Value	EPA Mean	EPA Standard Deviation	Acceptance Range	Evaluation
Chloride	94	92.6	4.14	83.2-102	Acceptable

Table 12.2
WS PT Sample Acceptance Example

12.4.3.2 For PT samples from the Water Pollution Program (WP), the acceptance range is calculated based upon the United States Environmental Protection Agency Nation Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). The acceptance limits are defined as ± three standard deviations from the estimated mean. See Table 12.3 for an example.

Analyte	Reported Value	EPA Mean	EPA Standard Deviation	Acceptance Range	Evaluation
Total Phosphorus (TP)	6.42	6.09	0.367	4.99-7.19	Acceptable

Table 12.3
WP PT Sample Acceptance Example

12.4.4 Any PT results determined to be outside the acceptance range will result in an investigation by the QAC. The investigation will involve, but is not limited to the following action items. The findings of the investigation will be noted in the appropriate file in Gillmor 327.

12.4.4.1 The QAC will request a re-test of the remaining PT sample. Should the PT sample return results within the acceptance range, a note will be placed in the PT file and analysis of the analyte will resume. Should the PT Sample still return results outside of the acceptance range, analysis will halt until a reason for failure can be identified.

12.4.4.2 The QAC will investigate the raw data produced by the instrument.

12.4.5 PT samples can be maintained and used as QC samples as outlined in Section 9.3. The acceptance criteria will be the same as Section 12.4.3.

SECTION 13.0: REPORTS TO MANAGEMENT

13.1. Monthly Reports will be generated to summarize QC data. These reports may include, but are not limited to, QC Data such as statistical process control chart results for blanks, sample duplicates, "spike" recovery and check standard analysis from each relevant SOP. The original print copy will be stored in the designated file in Gillmor 327. Additional copies will be made available to the members of the NCWQR Management Team upon request.

13.1.1. Quarterly results from the Performance Evaluation will be incorporated into the Monthly Reports as they become available.

13.1.2. Validation Study data will be incorporated into the Monthly Reports as they are completed.

13.2. Staff meetings will be held with the following frequency:

13.2.1. A Quarterly meeting will be held with the Laboratory Manager and designated analytical staff members to review QC Data, Performance Evaluations, Control Charts, etc.

13.2.2. An annual meeting will be held with all NCWQR employees to review summaries of quality control data and to discuss other quality concerns. This meeting may be used as training for any revisions to the QAP. An annual report will be prepared by a QAC and stored in the designated file in Gillmor 327.

SECTION 14.0: DOCUMENT RETENTION AND CONTROL

14.1. Original signed documents (e.g, QAP, SOPs, etc.) will be stored in the designated file cabinet in Gillmor 327 (hereafter referred to as the Master File). The signature with the most recent date will be considered the effective date of the document. A signed document with a greater version number will void any signed document with a lesser version number.

14.1.1. All Standard Operating Procedures (SOPs) and Quality Assurance Standard Operating Procedures (QA-SOPs) must be signed by the individual who prepared the document (Prepared by), a second member for the NCWQR (Reviewed by) and a QAC or the Director (Approved by).

14.1.2. The QAP must be signed by the individual who prepared the document (Prepared by), a QAC if not the preparer (Reviewed by) and the Director (Approved by).

14.1.3. It is the responsibility of a QAC to update the Master File and all controlled copies with the most recent copy of the document. In addition, a QAC will inform all members of the NCWQR that an updated version has gone into effect. This communication may be in the form of an e-mail.

14.1.4. All previous versions of original signed documents will be stored in the Master File for a minimum of three years after being voided. The date which the document is voided will be marked on the original signed document by a QAC.

14.1.5. Current copies of electronic documents will be stored in the WQL folder on "server-six" that is maintained by the Heidelberg University Computer Network Information Technologies department. Electronic copies and any printouts made from them are considered uncontrolled; however, they may be used as long as the document is effective.

14.2. Controlled copies of all effective SOPs and the QA-SOP can be found within designated binders in Gillmor 319 & 304. Any duplicates made from these copies are considered uncontrolled.

14.3. Revisions to the QAP may be initiated by any member of the NCWQR; however, all changes must be approved by the Director (see Section 14.1.2).

14.4. SOPs may be written or revised by any member of the NCWQR; however, all changes must be approved by a QAC (see Section 14.1.1).

14.5. The QAP and all SOPs must be reviewed at least once a year. A year is defined as the Water Year which runs from October 1st to September 30th.

14.5.1. If no changes are needed for the QAP, the QA-SOP or an SOP after the yearly review, the original copy in the Master File will be marked as reviewed by a QAC.

14.5.2. SOPs that have been updated with the most current Validation Data (see Section 9.0) will be considered reviewed for the year in which the Validation Study took place.

14.6. Logbooks will be used to document the performance of assigned equipment and preparation of solutions.

14.6.1. Logbooks will be in the form of a bound laboratory notebook.

14.6.2. Completed logbooks will be stored in the designated area in Gillmor 327 for at least 3 years.

14.7. Material Safety Data Sheets (MSDSs) for all chemicals used within the NCWQR will be stored in Gillmor 327. The MSDSs will be continually updated by a QAC.

SECTION 15.0: PROCUREMENT

15.1. The minimum grade of reagents or supplies required for analyses are listed in each method's SOP. A reagent or supply may only be substituted with an equivalent or higher grade.

15.2. Any vendor may be used to provide a chemical, reagent, or supply on the condition that the grade received is sufficient for the analysis. If a particular vendor's product does not give a suitable performance, a note will be placed in the method's SOP.

15.3. Proficiency Testing (PT) samples may be obtained from one of the following sources, Table 15.1. It is the responsibility of the Lead QAC, or designee, to order the PT samples and log them in as samples.

Approved Agencies	URL and email	Telephone No.
Phenova	www.phenova.com info@phenova.com	866-942-2978

Table 15.1
Proficiency Testing Sample Approved Supplier

15.4. All reagents used in testing covered by the QAP must meet the minimum quality standards stated in the method's SOP.

SECTION 16.0: CHANGE HISTORY

16.1 Version 1 -- Initial issuing of Quality Assurance Plan; Version Author, Aaron Roerdink

16.2 Version 2 – Update organizational chart, new director; Update Qualified Data Collector (QDC) Certifications; ; Version Author, Aaron Roerdink

16.3 Version 3 – Update organizational chart; remove Appendix A form 1; Add statement for analytical method variances, Section 6.2; Update instrument chart, new ion chromatograph; Add statement of electronic storage, Section 9.3; Update QC data handling, Section 10.3; Add statement of successful OEPA audit, Section 12.1; Add criteria for acceptance of inter/intra laboratory studies, Section 12.4; Add yellow sticker for approved reagents, Section 15.4; Version Author, Aaron Roerdink

16.4 Version 4 – Change references of “College” to “University”; update organizational chart; update PT approved suppliers; clarify location of independent quality control standard results; clarify the difference between Quality Control Data and Experimental Data in Section 10; update laboratory floor plan; Version Author, Aaron Roerdink

16.5 Version 5 – Update organizational chart; Version Author, Aaron Roerdink

16.6 Version 6 – Update organizational chart; add position descriptions, update PT approved suppliers; update laboratory floor plan; Version Author, Aaron Roerdink