CHAPTER II QUALITY ASSURANCE

| Section A. | Introduction |
|------------|--------------|
| | |

Section B. Quality Management

- 1. Quality Management Systems
- 2. Quality Management Plans
- 3. Quality Assurance Project Plans
- 4. Laboratory Quality Manual
- 5. Standard Operating Procedures
- 6. Procedural Change Authorization
- 7. Sample Handling and Custody
- 8. Document Control
- 9. Consistency of Documentation
- 10. Contingency Plans and Health and Safety Plans

Section C. Data Quality Objectives

- 1. Data Quality Objectives
- 2. Measurement Quality Objectives
- 3. Data Quality Indicators for Field-Processed Samples
- 4. Data Quality Indicators for Field Measurement
- 5. Data Quality Indicators for Laboratory Analyses

Section D. Field Quality Control

- 1. Annual Calibration
- 2. Routine Calibration
- 3. Calibration Samples
- 4. Field Measurement DQIs
- 5. Field Replicate Samples
- 6. Field Blanks
- 7. Sampling Equipment Blanks
- 8. Decontamination
- 9. Preservation, Shipping and Handling

Section E. External Performance Assessments

- 1. Split Sample Program
- 2. Performance Evaluation Samples
- 3. Audits of Data Quality
- 4. On-Site Audits

Section F. Comparability Studies

Section G. References

Appendix II-A: QA Project Plans and Laboratory Quality Manuals

SECTION A

INTRODUCTION

Title I, §117 of the Clean Water Act requires that the Chesapeake Bay Program Office (CBPO) support the Chesapeake Executive Council by implementing and coordinating science, research, modeling, support services, monitoring and data collection. The CBPO has maintained and supported a research-quality monitoring program for Chesapeake Bay tidal waters since the late 1980s when standardized sampling, analytical and data management procedures were developed and coordinated with the then Maryland Office of Environmental Programs and the Virginia State Water Control Board. In the 1990s, standardized River Input monitoring was initiated to measure the nutrient and sediment loadings from the of the watershed's nine largest rivers. Chesapeake Bay Nontidal water-quality monitoring was later expanded upstream into rivers and streams across the Bay watershed, with all six participating jurisdictions using comparable protocols.

The tidal and nontidal monitoring programs provide an uninterrupted record of high-quality data that is used to calculate status and trends of water quality constituents over time. Trend analyses in particular require very reproducible data that are collected at the lowest possible limits of detection. Changes in methods, laboratories, instruments, sampling sites, etc., may affect trends analyses so changes are carefully evaluated and approved to preserve the comparability of the data records over time.

Each participant in the Chesapeake Bay Tidal and Nontidal monitoring shall develop and implement a quality assurance (QA) program that is in accordance with the procedures and recommendations of this document. The purpose of this chapter is to establish data quality objectives and quality assurance protocols for incorporation into each organization's QA System, Quality Assurance Project Plans (QAPP) and Standard Operating Procedures (SOP).

Section B of this chapter describes the requirements and recommendations for a participant's quality management system and QA documentation. Section C covers the data quality objectives for field and laboratory operations. Section D is a summary of field quality control practices, with more detailed requirements for Tidal and Nontidal field procedures provided in Chapters IV and V respectively.

Section E describes routine inter-laboratory comparison studies, performance testing and external audits. Section F provides guidance for laboratories to conduct side-by-side comparisons prior to making procedural modifications.

SECTION B

QUALITY MANAGEMENT

1. Quality Management Systems

- 1.1. Organizations receiving EPA funds for monitoring are required to establish and document a formal quality management system (QMS) to ensure the generation of reliable and defensible data. A QMS is comprised of the organizational structure, objectives, policies, principles, responsibilities, and steps for ensuring quality and accountability in its work processes, products and services. A QMS includes:
 - 1.1.1. Field operations and support functions used to assure consistency and data integrity: training, procurement, information management, records, management reviews of operations and data quality, evaluation criteria and follow up response.
 - 1.1.2. Protocols for identifying out-of-control sampling, field measurements, and analytical conditions; processes for implementing and documenting the necessary corrective actions; decision rules and mechanisms for communicating the outcome.
- 1.2. The Chesapeake Bay Program recommends that participating laboratories develop and maintain a quality system that is equivalent to the National Environmental Laboratory Accreditation Institute (TNI) standards; however, laboratory accreditation is not required.
- 1.3. Laboratory quality management systems should be fully documented in a Laboratory Quality Manual.

2. Quality Management Plans

- 2.1. State agencies receiving EPA funds to conduct monitoring activities are required to document their quality management system in a Quality Management Plan (QMP).
- 2.2. EPA must review and approve the QMP prior to the initiation of environmental data collection and/or compilation activities. The document must be prepared in accordance with EPA QA/R-2: EPA Requirements for Quality Management Plans, which is available at www.epa.gov/quality1/qa_docs.html.
- 2.3. The QMP must be approved internally by the state QA Manager and the organization's senior management, and then be submitted to the EPA Project Officer at least 45 days prior to the initiation of data collection or data compilation. The U.S. EPA Region 3 Quality Assurance Manager approves the QMP.
- 2.4. An approved QMP is valid for up to five years unless there is a major program reorganization that affects quality assurance functions and structures in the organization.

3. Quality Assurance Project Plans

- 3.1. The Chesapeake Bay Quality Assurance Program requires the development and implementation of a Quality Assurance Project Plan (QAPP) for each of its monitoring activities. The QAPP must cover specific activities to be performed and procedures to be used by the Participant.
- 3.2. The purpose of the QAPP is to: 1) ensure that the level of needed data quality will be determined and stated before the data collection efforts begin and 2) ensure that all monitoring data generated and processed will reflect the quality and integrity established by the QAPP.
- 3.3. The QAPP is composed of standard elements that cover all aspects and activities of the monitoring, from planning, through implementation, to assessment. The document <u>EPA Requirements for QA Project Plans (QA/R-5)</u> fully describes the necessary elements which are outlined in Appendix II-A.
- 3.4. Review and Approval of QAPPs
 - 3.4.1 The EPA CBPO Project Officer and QA Coordinator will review and approve the QAPP at least to the "Conditionally Approved" level (meaning all technical issues having been resolved to the satisfaction of the CBPO) prior to data collection. The QAPP shall be reviewed and approved in the context of the Program's Data Quality Objectives (DQOs).
 - 3.4.2 The CBP QA Coordinator shall review and evaluate the implementation of the plans during the operational phases of sampling and analyses. The CBP QA Coordinator shall also assess the actual performance of the planned activity and subsequent results according to the criteria described in the QAPPs.

4. Laboratory Quality System and Quality Manual

- 4.1. The purpose of the laboratory quality management system is to:
 - 4.1.1. Establish and maintain data integrity, validity, and usability standards.
 - 4.1.2. Ensure that sampling and analytical systems are maintained in an acceptable state of stability and reproducibility.
 - 4.1.3. Detect problems through data assessment and establish corrective action procedures to ensure that the sampling, analytical, and measurement processes are reliable.
 - 4.1.4. Document all aspects of the sampling, analytical, and measurement processes in order to provide data that are technically sound and legally defensible.
- 4.2. The laboratory quality management system should be documented in a Laboratory Quality Manual (QM). All policies and procedures governing the laboratory's quality system shall be documented in the QM, and all laboratory personnel shall follow the policies and procedures established by the quality manual.
- 4.3. The QM should present in specific terms, the policies, organization, objectives, and specific QA and QC activities designed to achieve the data quality requirements recommended in this document. Where applicable, Standard Operating Procedures (SOPs) pertaining to each element should be included or incorporated by reference as part of the QM. The QM should be available during on-site laboratory evaluations.
- 4.4. See Appendix II-A of this chapter for an outline of key elements in the laboratory quality manual.

5. Standard Operating Procedures

- 5.1. A SOP is a written document which provides directions for the step-by-step execution of an operation, test, or action which is commonly accepted as the method for performing certain routine or repetitive tasks. These tasks include operations such as sampling, sample tracking, analysis, glassware preparation, instrument calibrations, preventive and corrective maintenance, and data reduction and analysis. SOPs should be expressed in terms of fixed protocols which must be followed. Where options exist, these should be clearly described, and criteria for the selection of alternatives must be included. SOPs should be written such that the actual practices are recorded.
- 5.2. SOPs should be clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts. All SOPs should reflect activities as they are currently performed in the field and laboratory. In addition, all SOPs should be:
 - 5.2.1. Consistent with the field and laboratory methods contained in this document and/or established by Chesapeake Bay Program Workgroups.
 - 5.2.2. Consistent with applicable federal and state regulations and guidelines.
 - 5.2.3. Adequate to establish traceability of standards, instrumentation, samples, and monitoring
 - 5.2.4. Simple, so that any user with appropriate general education, experience, and training can duplicate the task as historically performed.
 - 5.2.5. Consistent with a) sound scientific and engineering principles, b) instrument manufacturers' instruction manuals and c) good laboratory practices.
 - 5.2.6. Complete enough so the user or auditor follows the directions in a logical step-wise manner through the sampling, analysis, and data handling processes.

5.3. Benefits of SOPs

- 5.3.1. Adherence to SOPs minimizes measurement bias and increases reliability.
- 5.3.2. SOPs provide a record of the performance of all tasks at any fixed point in time.
- 5.3.3. SOPs increase the opportunity for thorough review of procedures with appropriate signoff by management.
- 5.3.4. SOPs serve as training documents for new employees, resulting in consistent performance of tasks.
- 5.3.5. SOPs provide a historical record of changes made to the method over time.
- 5.4. The degree of adherence to the approved SOPs should be determined during systems audits. It is recommended that all SOPs be reviewed at least once a year, revised and approved by his/her supervisor, and submitted for review of changes to the CBP QA and Monitoring Coordinators.

- 5.5. Laboratory method SOPs should follow a standard format such as the example below.
 - 5.5.1. Title Page (method name, number, version, effective date, document control number)
 - 5.5.2. Log of Changes to Method (i.e., revision history)
 - 5.5.3. Scope and Application (matrices, analytical range, etc.)
 - 5.5.4. Summary of Test Method
 - 5.5.5. Definitions
 - 5.5.6. Interferences
 - 5.5.7. Safety
 - 5.5.8. Equipment and Supplies
 - 5.5.9. Reagents and Standards
 - 5.5.10. Sample Preservation and Storage
 - Quality Control (EPA, 2012)
 - Demonstration of Capability
 - 3. Method Detection Limit
 - 4. Laboratory Reagent Blank
 - 5. Laboratory Control Sample
 - 6. Matrix Spike and Matrix Duplicate
 - 7. Control Charts (or other trend analysis of QC results)
 - 8. Corrective Action (root cause analysis)
 - 9. QC Acceptance Criteria
 - 5.5.11. Calibration and Standardization
 - 5.5.12. Sample Preparation and Analysis
 - 5.5.13. Analytical Procedure
 - 5.5.14. Calculations (automated and manual) and Reporting
 - 5.5.15. Method Performance Summary
 - 5.5.16. References
 - 5.5.17. Tables, Diagrams, etc.

5.6. Procedural Change Authorization

- 5.6.1. The CBP Quality Assurance Coordinator must be notified of the intent to make any substantial or long-term change to a procedure or method, either in the field or laboratory. These changes include items such as instrument type and sampling stations.
- 5.6.2. The effects of any change in analytical instruments, reagents, calibration, digestion procedure, etc., should be quantified, documented and submitted to the CBP QA Coordinator prior to implementing. Section II.F of this chapter provides detailed instructions for performing method validation and comparability studies.
- 5.6.3. All modifications should be documented using the Chesapeake Bay Monitoring Program Procedure Modification Tracking Form (PMTF) (Figure II-1). The completed PMTF should be submitted to the State agency Monitoring Coordinator, CBP Quality Assurance Coordinator and CBP Water Quality Database Manager.
- 5.6.4. Minor changes in field or laboratory procedures, including detection limit changes, should be documented in the CIMS data submission tables.
- 5.6.5. Minor events and problems encountered during in Chesapeake Bay mainstem cruises may be reported in the CBP Monitoring Cruise Report and submitted to the State agency, who will

Comment [m1]: Is this possible?

Chapter II
Quality Assurance
DRAFT Revision 1
August 2012
ents, all remarks relating

then forward the information to the CBPO. For smaller sampling events, all remarks relating to field work may be reported in the CIMS WQ_Cruise and WQ_Event tables.

5.6.6. Modifications due to emergencies during a sampling cruise are authorized by the Chief Scientist with priorities for safety and completion of the cruise. The change should be documented within 30 days after the cruise, in either the PMTF or the Monitoring Cruise Report, depending on size or potential impact of the deviation on the data.

This form is used to request approval for modifications and to document approved modifications made to Chesapeake Bay Program Office procedures or methods. It is not a substitute for timely contact with the CBPO Quality Assurance Officer or his/her designee, who may be reached at 1.800.968.7229. A detailed method description including the proposed modification must be attached to this form prior to submitted to CRPO.

| at 1-800-968-7229. A detailed method descri | ription including the proposed mod | ification must be attached to this for | m prior to sub mittal to CBPO. |
|---|------------------------------------|---|--------------------------------|
| DATE SUBMITTED | | DATE APPROVED | |
| REQUESTOR NAME | | ORGANIZATION | |
| NEWLY PROPOSED [] MODIFICATION | FIELD-APPROVED [MODIFICATION |] APPROVED BY: DATE: | |
| TYPE OF PROCEDURE / METHOD | SAMPLING [] | ANALYTICAL [] | REPORTING [] |
| | FIELD [] MEASUREMENT | OTHER [] SPECIFY: | |
| DURATION | PERMANENT [] TEMPORARY [] | EFFECTIVE DATE: START DATE: END DATE: | |
| PROCEDURE/METHOD DESCRIPTION | | | |
| MODIFICATION DESCRIPTION | | | |
| JUSTIFICATION FOR MODIFICATION | | | |
| ANALYTICAL PARAMETERS THAT MAY BE AFFECTED BY THIS CHANGE | | | |
| AFFECTED QA PLAN(S) (TITLE, REVISION, & DATE) | | | |
| AFFECTED CRUISE(S) | | | |
| PMTF COMPLETED BY | NAME: | | DATE: |
| STATE APPROVAL: | NAME | | TITLE |
| | SIGNATURE | | DATE |
| CBPO APPROVAL: | NAME | | TTTLE |
| | SIGNATURE | | DATE |

6. Sample Handling and Custody

- 6.1. Procedures should be established that ensure that samples are properly collected, preserved, transported, stored and analyzed within the required holding times.
- 6.2. The laboratory must establish and operate a system for assuring positive identification of samples and documentation of all samples received. To ensure sample integrity, procedures for sample identification, sample receiving, and custody should be developed, instituted and documented.
- 6.3. Sample Identification
 - 6.3.1. To assure traceability of samples while in possession, there should be a specified method for maintaining identification of samples in the field and throughout the laboratory.
 - 6.3.2. Each sample and sample preparation container should be labeled with a unique identifier that is cross-referenced with the corresponding documentation.
- 6.4. Sample receiving, storage and disposal requirements are described in Chapter VI, Sections C.2 & C.3

7. Document Control

7.1. The goal of the document control program is to assure that all documents and electronically stored information from a specified cruise are accountable, secure, and completely retrievable. Document control is recommended for each activity to include electronic as well as hardcopy documentation. Accountable documents should include but not be limited to field and laboratory logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all field and laboratory records are assembled and stored for delivery to the CBPO or are available upon request from the CBPO prior to the delivery schedule.

7.2. Preprinted Forms and Logbooks

- 7.2.1. All documents produced which are directly related to the sampling, preparation, and analysis of CBPO samples should be maintained for inspection by the CBPO. All observations and results recorded by field and laboratory staff but not on preprinted forms should be entered into permanent logbooks. When all data from a cruise are compiled, all original field and laboratory forms and copies of all cruise-related logbook entries should be included in the documentation package.
- 7.2.2. Pre-printed field and laboratory forms should contain the name of the field crew/laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.
- 7.2.3. Logbook entries should be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.
- 7.2.4. Logbook entries should be in chronological order.
- 7.2.5. Pages in both bound and unbound logbooks should be sequentially numbered.

- 7.2.6. Data sheets or logs should be maintained to enable a reconstruction of the sample collection or analysis in question.
- 7.2.7. Corrections to supporting documents and raw data should be made by drawing a single line through the error and entering the correct information. Corrections and additions to supporting documents and raw data should be dated and initialed. No information should be obliterated or rendered unreadable. <u>All notations should be recorded in ink.</u> Unused portions of documents shall be crossed out.

7.3. Storage of Files

7.3.1. Field and laboratory documents will be maintained in a secure location for a period of five years from the date of sample delivery.

8. Consistency of Documentation

- 8.1. A document control officer responsible for the organization and assembly of the data package should be assigned.
- 8.2. All copies of field and laboratory documents should be complete and legible.
- 8.3. Before releasing test results, the document control officer should assemble and cross-check the information on sample tags, custody records, laboratory bench sheets, personnel and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or sample delivery group is consistent throughout the data submittal package.
- 8.4. All documents relevant to each cruise, including logbook pages, bench sheets, screening records, re-preparation records, records of failed or attempted tests, and custody records should be inventoried.

9. Contingency and Health and Safety Plans

- 9.1. The Participant should develop and implement the following additional plans:
- 9.2. A contingency plan covering the availability and/or plan for a backup vessel.
- 9.3. A contingency plan for key field instrumentation failure.
- 9.4. A Health and Safety Plan in accordance with all applicable State and Federal regulations.

SECTION C

DATA QUALITY OBJECTIVES & INDICATORS

1. Introduction

Data Quality Objectives (DQOs) are qualitative and quantitative statements that specify the quality of data required supporting specific CBPO decisions. DQOs specify the level of uncertainty that a decision maker is willing to accept in results derived from monitoring data. The level of uncertainty is largely a function of sampling frequency and spatial density.

Sampling frequency and fixed station locations for the Chesapeake Bay Tidal Monitoring Program were established by comparing the power and robustness of various fixed station sampling designs (Alden, 1994). It was determined that 14 monitoring events, or "cruises" were sufficient for calculating long-term annual trends with acceptable confidence. As funding permits, additional cruises are added to capture major climatic and biological events. Approximately 100 mid-channel sampling locations or "stations" represent the different regions of the estuary and were selected to represent the Chesapeake Bay segmentation/ characterization scheme, which is based on circulation patterns, salinity and geomorphology.

Sampling frequency and fixed station locations for the Chesapeake Bay Nontidal Monitoring Program are described in Chapter V, Nontidal Water Quality Monitoring.

2 <u>Data Quality Indicators Objectives</u>

- 2.1 <u>Data Quality Indicators are quantifiable attributes applicable to an entire data set. The principal indicators of data quality are precision, bias, accuracy, representativeness, comparability, completeness, and sensitivity.</u>
- 2.2 For both field and lab measurements, Measurement performance, criteria (or acceptance limits) for sampling and analytical methods are are expressed established for each Data Quality Indicator (DQI). These limits also are known as in terms of Data-Measurement Quality Objectives (DQOsMQOs). DQOs define the acceptance thresholds for analytical data and are applied to various data quality indicators (DQIs).
 - The principal indicators of data quality are precision, bias, accuracy, representativeness, comparability, completeness, and sensitivity.
- 2.3 The DQOs-acceptance limits given in this section are associated with the methods contained in this document. Laboratories are expected to develop internal acceptance criteria based on recent internal performance data, which often results in more stringent acceptance thresholds than those given here. The objectives defined here are established through an iterative process, these values may be adjusted by the CBP QAO as a result of evaluations of performance data generated during this program.
- 2.4 DQOs for sampling activities are expressed in terms of comparability, representativeness, precision, accuracy, and completeness using the following criteria.

- 2.4.1 Following the sampling procedures and sample locations recommended in this document may ensure sampling comparability and representativeness of data generated to meet the CBP needs.
- 2.4.2 Overall precision of sampling and analytical procedures is assessed through replicate field samples and may be expressed as relative percent difference (RPD). *In-situ* measurements are not replicated. Sampling precision can be estimated by comparing overall precision to the analytical precision.
- 2.4.3 Positive bias from contamination is checked through the analysis of field blanks. Field spiked samples are not required for CBP nutrient monitoring.
- 2.4.4 Completeness of sampling is calculated based on the ratio of samples collected to samples that were planned, and is expressed as percent completeness.
- 2.5 DQOs for in-situ measurements are expressed in terms of comparability, representativeness, completeness, and minimum reporting levels using the criteria listed in Table II.2.
 - 2.5.1 Comparability and representativeness of field measurement data are ensured through adherence to the CBP methodologies and quality assurance protocols.
 - 2.5.2 Completeness of field measurement data is calculated based on the ratio of measurements made to measurements planned, and is expressed as percent completeness.
 - 2.5.3 Reporting levels for field instruments are based on
- 2.6 DQOs for laboratory analytical data are expressed in terms of comparability, representativeness, precision, accuracy, bias, completeness, and method detection limits (MDLs), using the following criteria.
 - 2.6.1 Accuracy, expressed as percent recovery, is based on the analysis of spiked samples and certified reference materials.
 - 2.6.2 Completeness of analytical data is calculated based on the ratio of samples that are analyzed to the number of samples collected, and is expressed as percent completeness.
 - 2.6.3 Method detection limits (MDLs) and practical quantitation limits (PQLs) should be determined for all parameters using the procedures in Chapter VI. Sections C.8 & C.9, which are based on 40 CFR Part 136, Appendix B.
 - 2.6.4 Real-time quality control charts for precision and accuracy should be developed and maintained for each parameter and appropriate concentration ranges, using the most recent 12 months of data. More points may be used if deemed necessary.
- 3 Data Quality <u>Indicators for Field Samples</u>

- 3.1 <u>DQIs Objectives</u> for <u>field</u> precision, bias and completeness are provided in Table II.1.
- 3.2 The overall sampling and analytical precision of each parameter is estimated from field split samples (sample types FS1 and FS2) that are processed exactly the same as regular samples. Dissolved parameters are expected to have higher precision due to the homogeneous nature of the sample.
- 3.3 Positive bias in sampling and analytical processes is estimated from field blanks (FB). Field blanks are processed in the field exactly the same as regular samples. A sample of unprocessed field blank water is called a source water blank (SWB).

Table II.1 Measurement Quality Objectives for Overall Precision, Bias and Completeness

| PARAMETERS | REFER- ENCE | PRECISION (FS1 & FS2) | BIAS (Field Blanks) | COMPLETE- NESS |
|---------------------------|----------------|--------------------------------|--|-------------------|
| Tidal Water Ovality | TS7 A 4 | Particulate: ≤ <u>30</u> % RPD | ≤ PQL <u>and</u> | 95% |
| Tidal Water Quality | IV.A.4 | Dissolved: ≤15% RPD | ≤ Source Water | |
| | | Particulate: ≤ <u>30</u> % RPD | | |
| Nontidal Water Quality | V C 4 | Dissolved: ≤20% RPD | ≤ PQL <u>and</u> ≤ Source Water 90% | |
| | | Whole Water: ≤20% RPD | | |

¹ Particulate parameters include TSS, Particulate Nitrogen, Particulate Carbon and Particulate Phosphorus.

 $^{^{2}}$ Whole water parameters include Total Nitrogen, Total Phosphorus and Total Organic Carbon.

4. <u>Data Quality Indicators for Field Measurements</u>

4.1 DQIs for field instrument post-calibration tolerance, completeness, precision and minimum reporting limits are provided in Table II.2.

Table II.2 Measurement Quality Objectives for Field Measurements

| PARAMETER | METHOD REFERENCE | POST- CALIBRATION TOLERANCE | COMPLETE- NESS | PRECISION / REPORTING LIMIT |
|-------------------------|---------------------|-----------------------------------|-------------------|-----------------------------------|
| рН | IV.B.3 | ± 0.2 units | 95% | 0.1 pH unit |
| Dissolved Oxygen | IV.B.3 | ± 0.3 mg DO/L | 95% | 0.1– 0.2 mg DO/L |
| Secchi Depth | IV.B.5 | NA | 95% | 0.05 - 0.1 meter |
| Specific Conductance | IV.B.3 | ± 5% of calibration standards. | 95% | 1 umho/cm |
| Salinity | IV.B.3 | NA | 95% | 0.1 psu |
| Light Attenuation | IV.B.6 | NA | 95% | 0.05% @ 100% light |
| Water Temperature | IV.B.3 | NA | 95% | 0.1°C |
| Depth | IV.B.1.3 | NA | 95% | 0.5 meter |

5. <u>Data Quality Indicators for Laboratory Analyses</u>

- 5.1 Precision, bias, and MDL objectives for laboratory water quality analyses are provided below in Table II.3.
- 5.2 Laboratory precision objectives are based on laboratory replicates (sample types LS1 & LS2).

Table II.3. Laboratory Measurement Quality Objectives – for Tidal and Nontidal WQ Parameters

| PARAMETER | REFER - ENCE | LAB PRECISION (LS1 & LS2) | METHOD BLANK | ACCURACY | Tidal MDL PQL (mg/L) | Nontidal MDL PQL (mg/L) |
|---|-----------------|---------------------------------|-----------------|--|----------------------------|-------------------------------|
| Total Dissolved Phosphorus | IV.D.2 | | | 90 - 110 % Spike Recovery | 0.001 | |
| Dissolved Ortho-Phosphate | IV.D.3 | | | 90 - 110 % Spike Recovery | 0.0006 | |
| Particulate Phosphorus (PP & PIP) | IV.D.4 | | | 90 - 110 % Spiked extract Recovery | 0.0012 | |
| Nitrite | IV.D.5 | | | 80 - 120% Spike Recovery | 0.0002 | |
| Nitrite + Nitrate | IV.D.6 | | | 90 - 110 % Spike Recovery | 0.0002 | |
| Ammonia | IV.D.7 | | | 80 - 120% Spike Recovery | 0.004 | |
| Total Dissolved Nitrogen | IV.D.8 | | | 90 - 110 % Spike Recovery | 0.026 | |
| Particulate Nitrogen | IV.D.9 | | | 80 - 120% SRM Recovery | 0.019 | |
| Particulate Carbon | IV.D.10 | | | 80 - 120% SRM Recovery | 0.097 | |
| Dissolved Organic Carbon | IV.D.11 | | | 85 - 115% Spike Recovery | 0.50 | |
| Chlorophyll-a | IV.D.13 | | | N/A | 0.2 μg/L | |
| Pheophytin | IV.D.13 | | | N/A | 0.2 μg/L | |
| Total Suspended Solids | IV.D.14 | | | N/A | 2.0 | |
| Silicates | IV.D.16 | | | 90 - 110% Spike Recovery | 0.013 | |

Chapter II Quality Assurance **DRAFT** Revision 1 <u>August 2012</u>

SECTION D

FIELD QUALITY CONTROL

1. Annual Calibration

- 1.1 An annual calibration is an extensive and thorough calibration using standards or instruments traceable to certified (e.g. National Institute of Standards and Technologies) instruments or standards. Annual calibrations of *in-situ* instruments may be performed by a manufacturer or specialized service contractor.
- 1.2 Annual calibrations will be performed on each field instrument, with the exception of LiCor® meters, where calibration is recommended annually, and required every two years.

2. Routine Calibration

- 2.1 Field staff shall calibrate in-situ instruments before and after each sampling event, deployment, or multiple-day cruise to ensure that the field instrument response is comparable to the response that existed at the annual calibration.
- 2.2 Instrument calibrations are performed according to manufacturers' specifications.
 - 2.2.1 Routine calibration is required for *in-situ* dissolved oxygen (DO), pH, and conductivity measurements (but not for depth, PAR and temperature).
 - 2.2.2 For dissolved oxygen, a calibration check is recommended at the beginning of each sampling day. If daily DO checks deviate by ≥ 0.30 mg DO/L from the expected value, the sensor must be recalibrated before using. If a calibration check (daily or post-calibration) is ≥ 0.50 mg DO/L, censor all data corresponding back to the last calibration check using the CIMS WQ Problem Code "V" (Sample results rejected due to QC criteria).
 - 2.2.3 Calibration of Dataflow and extended *in-situ* deployments are performed for DO, pH, conductivity, chlorophyll and turbidity measurements. (See Participants QAPP for most approved procedures.)
- 2.3 SOPs for calibration should describe the preparation and use of the standard reference solution(s). If commercially prepared standards are used, cite the commercial source(s) in the field SOP and mark the date received on the bottle or calibration log.
- 2.4 When the calibration check indicates that a significant change occurred during a cruise, the instrument should be serviced and re-calibrated as described in the annual calibration.
- 2.5 If a daily or post-calibration check does not meet tolerances, qualify all data corresponding to the last calibration performed.

3. <u>Calibration Samples</u>

Calibration samples are water samples or independent *in-situ* measurements used to develop a statistical relationship between an *in-situ* measurement and the parameter of concern. One example is the collection of grab chlorophyll *a* samples for converting *in-situ* fluorescence measurements into chlorophyll *a* estimates. A second example is the collection of PAR measurements for converting *in-situ* turbidity measurements into corresponding light attenuation coefficients (Kd).

4. <u>Data Quality Indicators for Field Measurements</u>

Data quality objectives for field measurement post-calibration tolerance, completeness, precision and minimum reporting limit are provided in Table II.2.

Table II.2 Field Measurement Quality Objectives

| PARAMETER | METHOD REFERENCE | POST- CALIBRATION TOLERANCE | COMPLETE- NESS | PRECISION / REPORTING LIMIT |
|-------------------------|---------------------|-----------------------------------|-------------------|-----------------------------------|
| рН | IV.B.3 | ± 0.2 units | 95% | 0.1 pH unit |
| Dissolved Oxygen | IV.B.3 | 0.3 mg DO/L | 95% | 0.1– 0.2 mg DO/L |
| Secchi Depth | IV.B.5 | NA | 95% | 0.1 meter |
| Specific Conductance | IV.B.3 | ± 5% of std. | 95% | 1 umho/cm |
| Salinity | IV.B.3 | NA | 95% | 0.1 psu |
| Light Attenuation | IV.B.6 | NA | 95% | 0.05% @ 100% light |
| Water Temperature | IV.B.3 | NA | 95% | 0.1°C |
| Depth | IV.B.1.3 | NA | 95% | 0.5 meter |

5. Field Replicate Samples

- 5.1. Field Split (FS1 & FS2): Two representative portions are taken from one homogeneous sample and processed identically. The data from the field split samples are an indicator of reproducibility (precision) in the sample preparation and analysis steps.
- 5.2. Field Duplicate, Co-located (S1 & S2): A field duplicate is a sample taken at the same sample location and depth as a CBP sample. The sample and duplicate shall be taken in quick succession of each other. The data from field duplicates may be used to estimate overall precision or to deduce sampling precision.
- 5.3. The recommended frequency for collecting Field Split or Field Duplicate samples is according to the CBP monitoring program:

- 5.3.1. Mainstem Monitoring: Collect a field split or a field duplicate once for every 20 samples.
- 5.3.2. Tidal Tributary Monitoring: Each sampling group should collect field splits once per month, from both surface and bottom depths.
- 5.3.3. Non-tidal Monitoring: At least one field split sample per month or once for every 20 samples. Field split samples are collected from the churn splitter.

6. Field Blanks

6.1. A field blank is an equivalent aliquot of reagent water that is carried through the entire analytical procedure. The purpose of a field blank is to determine the levels of contamination associated with the processing and analysis of samples.

Blank values are NOT to be subtracted from sample results.

- 6.2. Field-filtered blanks are required for CBP monitoring programs that specify field filtration. After routine samples are processed, filter and preserve reagent grade water exactly the same as the samples. Both dissolved and particulate fractions shall be processed and submitted for analysis.
- 6.3. The frequency for preparing field-filtered blanks is according to the CBP monitoring program:
 - 6.3.1. Mainstem Monitoring: Prepare one field-filtered blank each day.
 - 6.3.2. <u>Tidal Tributary Monitoring</u>: Prepare at least one field-filtered blank per month.
 - 6.3.3. Non-tidal Monitoring: Prepare at least one field-filtered blank per month. Fill a churn splitter with reagent water prior to filtration.
- 6.4. If the concentration of a field blank exceeds the PQL, reporting level or the lowest analytical standard in the calibration curve, field and/or laboratory contamination should be suspected and corrective action initiated.
- 6.5. Corrective action includes an investigation of possible contamination sources (e.g., instrument calibration check, field blank water, sample containers, etc.) and procedural modifications if necessary.

7. Sampling Equipment Blanks

- 7.1. Equipment blanks indicate the effectiveness of the sampling equipment cleaning procedure. The equipment blank may be processed in the field office after the equipment has been cleaned.
- 7.2. An equipment blank is required once per year or whenever new equipment is used for the first time.
- 7.3. The equipment blanks consist of reagent-grade, deionized water that has been passed sequentially through each component of the sample processing and collection equipment, e.g., submersible pump and hose, Rosette bottles, sampling containers, churn splitter, filtration unit, etc.

Comment [m2]: USGS recommends

II-18

- 7.4. An analysis of the unfiltered reagent grade water used to prepare the blanks maybe helpful in interpreting the results if contamination is found.
- 7.5. If the concentration of the equipment blank exceeds the lowest analytical standard in the calibration curve, prepare blanks of just the sampling equipment to isolate the cause of the contamination.

8. Decontamination

Table II-D. Summary of Field QC Sample Requirements

| Field QC Sample | Frequency | Control Limit |
|------------------------|--|---------------------------------------|
| Field Rep (FS1/FS2) | Mainstem: 1 rep. per 20 samples Tidal Tributary: 1 station per month, at all depths Non-tidal: ≥ 1 per month (FS1/FS2) | 10 - 30% RPD, Parameter specific |
| Field Filtration Blank | Mainstem: Daily Tidal Tributary: 1 station per month, at all depths, by each region or sampling team. Non-tidal: ≥ 1 per month | < Lowest analytical standard (PQL) |
| Field Equipment Blank | Annually | < Lowest analytical standard (PQL) |

Formatted Table

SECTION E

EXTERNAL PERFORMANCE ASSESSMENTS

- 1. <u>Split Sample Programs Interlaboratory Comparisons</u>
- 1.0 Background and Objectives

All laboratories participating in Chesapeake Bay Water Quality monitoring programs are required to participate in the Chesapeake Bay Coordinated Split Sample Program (CSSP). The CSSP was established in June 1989 by recommendation of AMQAW, to the Monitoring Subcommittee. The major objective of this program is to establish a measure of data comparability among Participants in the monitoring programs.

There are two CSSP sample types; the first is a saline water matrix, collected from the mainstem of the Bay. The second is a fresh water tributary sample, collected from the Potomac River. CSSP samples are collected four times a year for each type, and transported to the laboratories for processing the following morning.

1.1 <u>Summary of Criteria</u>

- 1.1.1 The Participant will participate in the applicable component(s) of the CSSP.
- 1.1.2 The SOPs that are developed and used should be in accordance with the Chesapeake Bay Coordinated Split Sample Program Implementation Guidelines, Revision 4 (December 2010), plus any revisions specified by the CBP QAO.
- 1.1.3 For each of the CSSP stations and on a quarterly basis, the Participant will receive and analyze three or four sub-samples. Treating each sub-sample as a discrete sample, participating laboratories are generally required to perform only those analyses which they routinely perform in support of basin-wide data collection programs. One of the three sub-samples should be used to generate laboratory duplicates and a laboratory spike. These QC samples should be analyzed concurrently with the associated CSSP sub-samples.
- 1.1.4 The routine submission of split sample data is the responsibility of each laboratory and its in-house data management organization.
- 1.1.5 To supplement the analyses of the CSSP sub-samples and the respective QC sample, a certified standard reference material (SRM) for each parameter should be analyzed where available. The analysis of standard reference materials provides a strong measure of comparability between all laboratories and within one laboratory's analytical system over time. It is a critical element of any diagnostic efforts associated with the CSSP.

2. <u>Performance Testing</u>

- 2.1 The University of Maryland Chesapeake Biological Laboratory prepares blind audit samples for all water quality parameters. The blind audit samples are distributed semiannually to participating laboratories.
- 2.2 Laboratories also participate in the USGS Standard Reference Sample study for nutrients. Lab managers are advised to analyze both high and low concentrations unless one concentration far exceeds their normal ranges of operation.

3. Audits of Data Quality

- 3.1 State agency staff or contractors shall review field blank and field duplicate data to assess the quality of sampling activities.
- 2.2 Analytical and measurement data should be reviewed in order to assess the quality of measurement and analytical activities, respectively.
- 2.3 The CBP-provided software will electronically verify the data quality for every submittal or the data review criteria will be implemented.
- 2.4 The Participant will prepare and submit a summary with each data set. The summary must include an explanation for each data point that did not meet the QC criteria established for each method, and deviations that occurred during the generation of the data.
- 2.5 The Participant will be informed if any of the submitted data do not fall within the prescribed QC limits. Any errors found will be corrected by the Participant at no additional cost to the CBPO.
- 2.6 The CBP Grant Project Officer has the ultimate responsibility to accept or reject each data submittal.

3. On-Site Audits

- 3.1 The CBP QAO or representative will conduct periodic on-site evaluations of field and laboratory activities. The frequency of these on-site audits may be increased depending on the Participant's performance. On-site evaluations are carried out to monitor their ability to collect and analyze samples according to the DQOs established by the CBP Monitoring Program.
- 3.2 The CBP QA Coordinator and a State representative will inspect the Participant's field and laboratory facilities to verify the adequacy and maintenance of instrumentation, the continuity of personnel meeting experience and/or education requirements, and the acceptable performance of analytical and QC procedures. The Participant should expect that items to be monitored will include but not be limited to the following:
 - Size and appearance of the facility.
 - Quantity, condition, availability, and scheduled maintenance and performance of instrumentation.
 - Availability, appropriateness, and use of field and laboratory SOPs.

- Field and laboratory staff qualifications, experience, and personnel training programs.
- Reagents and sample storage facilities.
- Reagent and test solutions preparation logbooks and raw data.
- Field and laboratory bench sheet and logbook maintenance and review.
- Review of the sample analysis/data package inspection procedures.
- 3.3 Prior to an on-site evaluation, various documents pertaining to performance of the Participant is integrated in a profile package for discussion during the evaluation. Items that may be included are previous on-site reports, laboratory evaluation sample scores, review of data, QA materials, and data trend reports.
- 3.4 The CBP QAO or representative will discuss his/her findings with the Participant in the presence of a representative from the State agency. During the debriefing, the auditor will present his/her findings and recommendations for corrective actions to field and laboratory personnel.
- 3.5 Following an on-site evaluation, audit reports which discuss deficiencies found during the on-site evaluation will be forwarded to the Participant. The Participant must respond to the audit report within 30 days of the report and, concurrently, the report must be sent to the CBP QAO and the State representative.
 - 3.5.1 If the Participant fails to take appropriate corrective action to resolve the deficiencies discussed in the on-site reports, any further sampling or analytical activities will not be conducted

SECTION F COMPARABILITY STUDIES

1. Background

Chesapeake Bay Program (CBP) data are used to calculate long-term trends in contaminants, which require very precise, unbiased data that are comparable over long periods of time. Seemingly insignificant changes in procedures may cause step-trends over time. To prevent this, the CBP requires that the effects of any change in instruments, reagents, calibration, digestion procedures, etc., be quantified, documented and submitted to the CBP QA Coordinator prior to implementing.

Although EPA allows certain changes in methods without official approval under 40 CFR Part 136.6, the Chesapeake Bay Program requires that some of the modifications allowed under §136.6 be submitted to and approved by the CBP QA Officer prior to implementation (see Section F.2.7 below).

- 2. Demonstrating Equivalency of Method Modifications
 - 2.1. Method modifications requirements of EPA §136.6:
 - 2.1.1. The underlying chemistry and determinative technique are essentially the same;
 - 2.1.2. The modified method produces equivalent performance for the analyte(s) of interest and
 - 2.1.3. The equivalent performance is documented.
 - 2.2. Side-by-side comparability studies must be performed using paired samples that are analyzed by each method or technique.
 - 2.2.1. The different methods should be conducted on the same day to minimize changes between the sample pairs.
 - 2.1.4.2.2.2. Side-by-side analyses should be conducted on different days, weeks or even months to incorporate the day-to-day variability of each method.
 - 2.2.2.3. When validating new procedures, laboratories must adhere to the standardized QC procedures detailed in the CBP method and incorporate these criteria into the method.
 - 2.3.2.4. Laboratories must use a reference matrix (usually, reagent water) and field samples for the validation study. If a laboratory intends to apply the method to more than one matrix type, the laboratory must validate the method on field samples of each matrix type. Fresh water and saline waters are considered different matrices.
 - 2.4.2.5. The new method must meet or exceed the performance measures of the original method. These measures include MDL, spike, duplicate and blank results; calibration checks, standard reference material, calibration correlation coefficients, etc.
 - 2.5.2.6. Modifications to procedures for method-defined analytes such as TSS, TOC and chlorophyll are not allowed, nor changes that would result in measurement of a different form or species

Comment[m3]: Can these be split
from the same digested sample or
extract?

of an analyte. Modifications that require validation and CBP approval include: _Changes in sample preparations such as digestions, distillations, and extractions. New instrumentation or a change from manual discrete instrumentation to automated. 2.6.2.2.7.2. Changes in reagents, reaction times and temperatures. _ Minor modifications that do not require a validation study or CBP approval include: 2.7.1.2.8.1. Changes in the calibration range. Adjusting sample sizes or diluting samples to optimize method performance. Changes in pH adjustment reagents or buffer reagents provided that the changes do not produce interferences. Changes in equipment operating parameters such as minor changes in the monitoring wavelength of a colorimeter or modifying the temperature program for a specific GC column. Replacement of instrument components purchased from, or recommended by the original manufacturer.

3. Validation of Method Modifications

3.1. Method Compilation

Prior to conducting a validation study, the laboratory should document (or reference) the exact procedures that will be used for the new method. The new method should be followed as written. If changes are necessary during the course of validation, then the date and rationale for the changes should be noted. All measures of performance must be repeated following a change in procedure.

3.2. Method Detection Limit Study

The lab must use the procedures specified in the modified method to perform a method detection limit (MDL) study in accordance with the procedure given at 40 CFR Part 136, Appendix B. Each laboratory must perform its MDL study on an instrument that is calibrated to encompass the projected PQL.

3.3. Calibration

Following completion of the MDL study, re-calibrate the instrument to include a standard less than or equal to the PQL concentration. The laboratory must demonstrate that the linearity criterion and the MDL of the modified method are as good as, or better than those of the original method.

3.4. Initial Precision and Recovery

3.4.1. After successfully calibrating the instrument, perform an initial precision and recovery (IPR) analyses using the procedures specified in the EPA reference method. The IPR consists of analyses

of four replicates of reagent water spiked with the analytes of interest.

- 3.4.2. For each analyte, the precision of analysis of the replicates, as determined by the standard deviation or relative standard deviation (RSD) of the measurements, should be less than the standard deviation or RSD specified in quality control (QC) acceptance criteria in the method. Similarly, for each analyte, the average percent recovery of the measurements should fall within the range of percent recovery specified in the method. If either the precision or recovery test is failed, the test is repeated until the laboratory is able to meet precision and recovery requirements.
- 3.4.3. Include a minimum of one blank in the initial demonstration, and the concentration of the analyte(s) in the blank should be less than the level(s) specified in the method. Repeat the initial demonstration with the modified method as an integral part of the method, until the QC acceptance criteria in the method for precision and recovery and for the blank are met. Otherwise, the modification will not be permitted. Maintain records that document that the initial demonstration was performed on the modified method and those requirements for precision and recovery and the blank were met.

3.5. Field Sample Validation

- 3.5.1. After successful completion of IPR analyses, the method modification is to be validated on the matrix type(s) chosen for the validation study.
- 3.5.2. Validation must include the analysis of paired samples using the current method and the modified method. This will permit an estimation of bias between the methods.
- 3.5.3. The numbers of analyses should be 100 samples per matrix, and cover the typical ranges of seasonal concentration and spatial differences.
- 3.5.4. Ideally, the two methods will be conducted on the same day to minimize differences between the sample pairs.
- 3.5.3.3.5.5. Analytical batches should be run on different days, weeks or even months to incorporate the day-to-day variability of each method.

3.6. Ongoing Precision and Recovery

The laboratory must demonstrate that it can meet the precision and recovery QC acceptance criteria of the original method. Each batch of samples which includes field samples, but not the IPR samples, must include ongoing precision and recovery QC samples.

3.7. Calibration Verification

The laboratory must verify calibration as described in the method. The field samples discussed in Section 3.5 above must be analyzed in a separate batch of determinations from the initial calibration sequence, so that calibration verification is performed. Calibration verification sample results of the modified method must meet the acceptance criteria of the original method. Ideally, 30 calibration verification sample pairs would provide more confidence in the statistical analysis. Recommend at least 5 CCV samples of several (3 to 5) different concentrations.

3.8. Contamination Level in Blanks

The laboratory must prepare and analyze at least one method blank with each sample batch during which the matrix samples are prepared and analyzed. The actual number of blank samples analyzed by each laboratory must meet or exceed the frequency specified in the method. The laboratory modified method must demonstrate that it can meet the QC acceptance criterion for blanks that is specified in the method.

4. Statistical Analysis

- 4.1. A *paired t-test* is best for the comparison of two different methods on samples of different concentrations, especially if the differences between pairs are normally distributed. A two-sided test with a p-value of 0.01 is recommended.
- 4.2. A Wilcoxin Signed-Rank test is necessary when the differences between the sample pairs are not normally distributed.
- 4.3. An *Analysis of Variance* (SAS® PROC GLM, a General Linear Model) is recommended if one wants to compare the effects of interferences on the different methods. Samples with the potential to interfere must be identified to conduct this analysis.

Note: A regression analysis that compares one method directly against another is NOT recommended since neither method can be assumed to have no error. Such an analysis would result in a "regression dilution effect". However, some labs have used linear regression when the paired t-test showed a significant difference and a correction factor was needed for an unavoidable method change.

5. <u>Documentation</u>

5.1. See Table II.4

Table II.4 Documentation of Method Equivalency

| | Table II.4 Documentation | New Method | Current Method | EPA Method |
|-----|--|------------|----------------|------------|
| 1. | Title and Description List & attach SOPs for new and current. | Tew Medica | | |
| 2. | Procedural differences | | | |
| 3. | Concentrations of calibration standards | | | |
| 4. | Initial Precision & Recovery | | | |
| 5. | Calibration Verification -Initial Cal Verification Result -Ongoing Cal Verification Res | | | |
| 6. | Method Detection Limit | | | |
| 7. | Correlation coefficient of calibration curve | | | |
| 8. | Sample matrix and concentration range for each (fresh and saline waters are separate matrices) | | | |
| 9. | Paired t-test results (per each matrix) A two-sided t- test with p-value of 0.01 | | | |
| 10. | Wilcoxin Signed-Rank test (if paired differences are not normally distributed) | | | |
| 11. | Other Statistics | | | |
| 12. | Certified reference material results with certified values | | | |
| | PT sample and results (USGS, ERA, CBP blind audit, etc.) | | | |
| _ | Method blank results | | | |
| 15. | Instrument blank (if comparing instruments) | | | |
| | Spiked sample results (Sample conc. and % recovery of each spike) | | | |
| | Duplicate sample results Rep 1, Rep 2 values and RPDs) | | | |
| | 9. Raw Data sample pairs (Submit excel file or equivalent) | | | |
| 20 | 0. Analyte carry-over | | | |

SECTION H

REFERENCES

Alden et al. 1994. An Assessment of the Power and Robustness of the Chesapeake Bay Program Water Quality Monitoring Program: Phase II - Refinement Evaluation. Chesapeake Bay Program Office, Annapolis, MD. http://archive.chesapeakebay.net/pubs/calendar/MONSC_06-05-09_Handout_4_10309.pdf

U.S. Environmental Protection Agency. 2012. 40 CFR Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures. Federal Register/Volume 77 / No. 97/ May 18, 2012/ Rules and Regulations

U.S. Environmental Protection Agency. 1983. Chesapeake Bay: A Framework for Action, Appendix F – A Monitoring and Research Strategy to Meet Management Objectives. Chesapeake Bay Liaison Office, Annapolis, MD. http://www.chesapeakebay.net/content/publications/cbp_12405.pdf

 $\label{lem:u.s.} U.S.\ Environmental\ Protection\ Agency.\ 2001a.\ \textit{EPA\ Requirements\ for\ Quality\ Management\ Plans\ (QA/R-2),} \\ EPA240/B-01/002,\ March\ 2001.\ \ \underline{\text{http://www.epa.gov/quality1/qs-docs/r2-final.pdf}}$

U.S. Environmental Protection Agency. 2001b. *EPA Requirement for Quality Assurance Project Plans (QA/R-5)*, EPA240/B-01/003, March 2001. http://www.epa.gov/quality1/qs-docs/r5-final.pdf

U.S. Environmental Protection Agency. 2010. Quality Management Plan for the Chesapeake Bay Program. Chesapeake Bay Program Office, Annapolis, MD. http://archive.chesapeakebay.net/pubs/quality_assurance/CBPO_QMP_2010_final.pdf

Chesapeake Bay Program. 2010. Chesapeake Bay Coordinated Split Sample Program Implementation Guidelines, Revision 4, December 2010. Chesapeake Bay Program Office, Annapolis, MD. http://archive.chesapeakebay.net/pubs/quality_assurance/CSSP_Guidelines_12-17-10.pdf

U.S.EPA (2007) Solutions to Analytical Chemistry Problems with Clean Water Act Methods, March 2007 http://www.epa.gov/waterscience/methods/files/pumpkin.pdf

APPENDIX II

QA PROJECT PLANS and LABORATORY QUALITY MANUAL

1. Quality Assurance Project Plan

The QA Project Plan shall be composed of standard elements that cover the entire project from planning, through implementation, to assessment. The document EPA Requirements for QA Project Plans (QA/R-5) fully describes the necessary elements which are summarized below.

A. Project Management

The elements in this group address the basic area of project management, including the project history and objectives, roles and responsibilities of the participants. These elements ensure that the project has a defined goal, that the participants understand the goal and the approach to be used and that the planning outputs have been documented.

B. Data Generation and Acquisition

The elements in this group address all aspects of project design and implementation. Implementation of these elements ensure that appropriate methods for sampling, measurement and analysis, data collection or generation, data handling, and QC activities are employed and are properly documented.

C. Assessment and Oversight

The elements in this group address the activities for assessing the effectiveness of the implementation of the project and associated QA and QC activities. The purpose of assessment is to ensure that the QA Project Plan is implemented as prescribed.

D. Data Validation and Usability

The elements in this group address the QA activities which

A1 Title and Approval Sheet

A2 Table of Contents

A3 Distribution List

A4 Project/Task Organization

A5 Problem Definition/Background

A6 Project/Task Description

A7 Quality Objectives and Criteria

A8 Special Training/Certification

A9 Documents and Records

B1 Sampling Design (Experimental Design)

B2 Sampling Methods

B3 Sample Handling and Custody

B4 Analytical Methods

B5 Quality Control

B6 Equipment Testing & Maintenance

B7 Instrument & Equipment Calibration

B8 Inspection & Acceptance of Supplies

B9 Non-direct Measurements

B10 Data Management

C1 Assessments & Response Actions, i.e., corrective action

C2 Reports to Management

D1 Data Review, Verification & Validation

D2 Verification & Validation Methods

D3 Reconciliation with User Requirements

occur after the data collection phase of the project is completed. Implementation of these elements ensures that the data conform to the specified criteria, thus achieving the project objectives.

2. Laboratory Quality Manual (Example Format)

- 1.0 Quality Assurance Policies
 - 1.1 Quality Assurance Policy Statement
 - 1.2 Proficiency Test Program
 - 1.3 Review of Requests for the Acceptance of New Work
 - 1.4 Document Control System (for bench sheets, log books, SOPs, etc.)
- 2.0 Organization and Responsibilities
 - 2.1 Organizational Chart
 - 2.2 Management
 - 2.3 Laboratory Director, Associate Laboratory Director
 - 2.4 Technical Staff (Include IT, Analysts, etc.)
 - 2.5 Information Management System
 - 2.6 Training
 - 2.7 Laboratory Capabilities
- 3.0 Quality Assurance Indicators
 - 3.1 Determining Control Limits for:
 - o Precision & Accuracy
 - o Representativeness
 - o Completeness
 - Comparability
 - 3.2 Procedure for Method Detection Limit Studies
- 4.0 Sample Handling
 - 4.1 Sample Tracking
 - 4.2 Sample Acceptance Policy
 - 4.3 Sample Receipt Protocols
 - 4.4 Sample Storage Conditions
 - 4.5 Chain of Custody
 - 4.6 Sample Disposal
- 5.0 Calibration Procedures and Frequency
 - 5.1 Traceability of Calibration
 - 5.2 Instrument Calibration (initial and continuing)
- 6.0 Test Methods and Standard Operating Procedures
 - 6.1 Reference Method (authoritative source)
 - 6.2 Demonstration of Method Capability
 - 6.3 Method Detection Limit
 - 6.4 Changes and Modifications

- 7.0 Quality Control Checks
 - 7.1 Internal Quality Control Samples
 - 7.2 Instrument-Specific Quality Control Checks
 - 7.3 Standard Reference Materials
- 8.0 Data Reduction, Review, Reporting and Records
 - 8.1 Data Reduction and Review
 - 8.2 Secondary Data Review
 - 8.3 Report Format and Contents
 - 8.4 Records Management and Control
- 9.0 Performance and System Audits and Frequency
 - 9.1 Internal Laboratory Audits
 - 9.2 Managerial Review
 - 9.3 Third Party Audits
- 10.0 Facilities, Equipment, and Preventative Maintenance
 - 10.1 Facilities and Equipment
 - 10.2 Computers and Electronic Data Security Requirements
 - 10.3 Preventative Maintenance
 - 10.4 Inspection/Acceptance Requirements for Supplies and Consumables
- 11.0 Corrective Action System
- 12.0 Subcontracting and Support Services And Supplies
 - 12.1 Subcontracting Laboratory Services
 - 12.2 Outside Support Services and Supplies
 - 12.3 Customer Complaint Resolution
- 13.0 References
 - Appendix A: Certification Statement
 - Appendix B: Initial Demonstration of Capability
 - Appendix C: Certification Statement for Method Validation
 - Appendix D: List of Instrumentation
 - Appendix E: Nutrient and Sediment Laboratory QC Criteria