# Quality Manual For
# The Analytical Service Center
# Virginia Institute of Marine Science

Rt 1208 Greate Road
Gloucester Point, VA 23062
(804-684-7000)

# College of William and Mary

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Date: 5/12/2021

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| Ownership: | Commonwealth of Virginia |

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|---|---|---|
| President | Dr. Katherine Rowe | President of the College of William and Mary |
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| Chief Operations Officer (VIMS) | Mr. Joseph Martinez | |
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QUALITY MANUAL FOR

Analytical Service Center
Virginia Institute of Marine Science
College of William and Mary

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## I. Quality Policy Statement

The laboratory management is committed to providing the necessary resources and to defining acceptable laboratory practices in the quality documentation to ensure compliance with the Virginia laboratory certification requirements. Management's policy is to ensure the information in quality documentation is communicated to, implemented and understood by all the laboratory staff performing work in the laboratory.

The quality manual documents the policies and references the procedures to ensure test data generated for submittal to the Virginia Department of Environmental Quality (VA DEQ) and other state and federal agencies are scientifically acceptable as defined by the method performance criteria.

The objectives of the Analytical Service Center (ASC) are to produce data of known and documented quality in order to meet QA/QC objectives and demonstrate conformance to the laboratory accreditation requirements. The objectives are measured with internal audits and evaluated as part of the management review.

The goal of the Analytical Service Center is to produce data that is in compliance with the Virginia laboratory certification requirements for noncommercial laboratories (1 VAC 30-45). When the data quality objectives (DQOs) for specific projects exceed the routine QA/QC objectives, the quality assurance project plans will address procedure changes, or new procedures required to meet the DQOs or will identify those instances where the DQOs cannot be met.

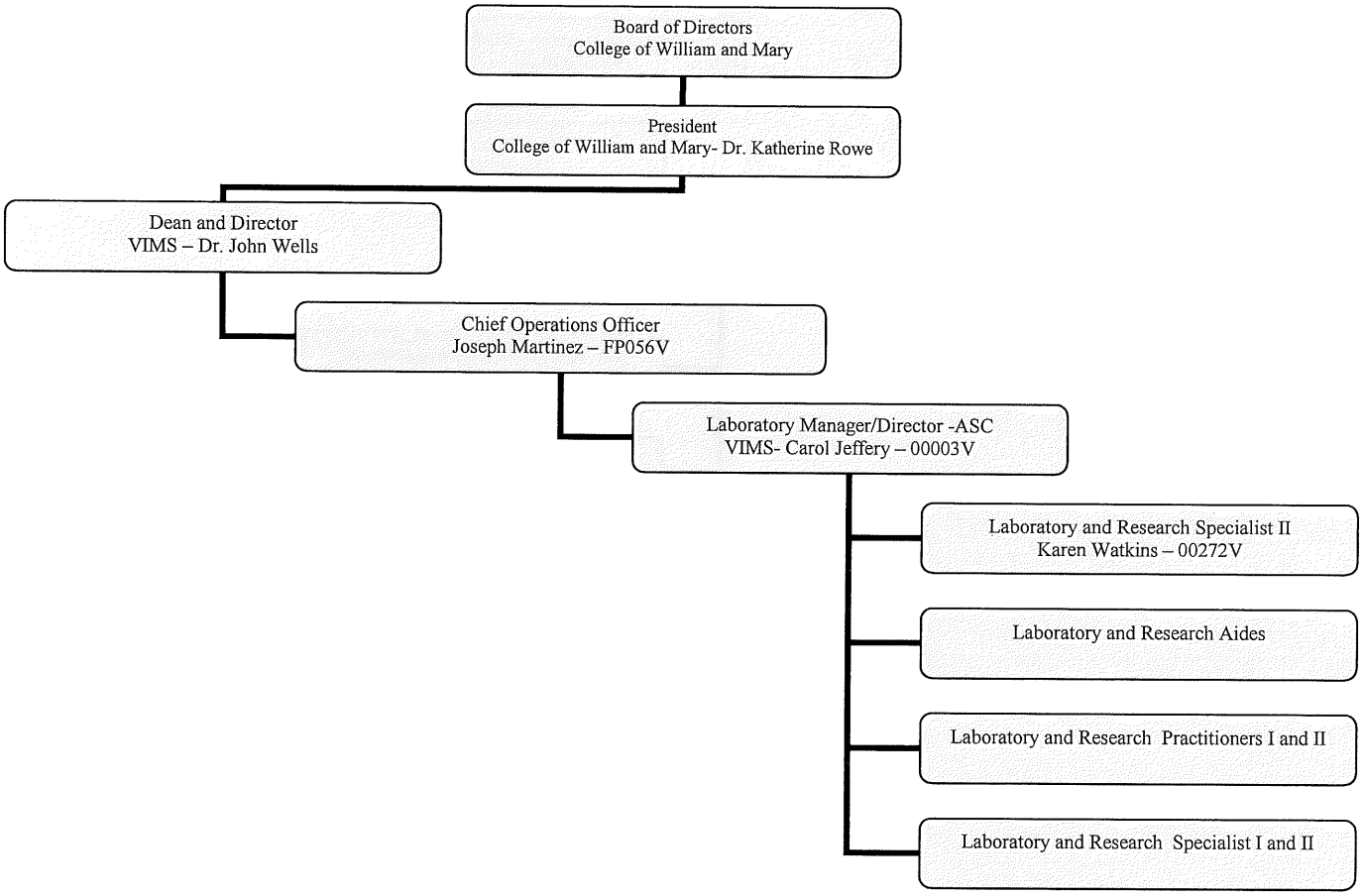
The Quality Manual (QM) is intended for the laboratory operations of the Analytical Service Center at the Virginia Institute of Marine Science. This laboratory provides analysis of samples as requested by state and federal agencies; as well as, institute supported research projects.

The objective of the QM is to provide a set of standards for the ASC to abide by for processing of samples so as to achieve high quality results. These guidelines will establish protocol by which each sample is maintained throughout the process of sampling, transportation, logging, documentation, analysis and final report generation. The QM outlines equipment maintenance and calibration schedules, equipment maintenance logs and calibration documentation. The QM includes a training guide for new technical personnel or those training to perform further analytical tests for which they have not yet been validated to run. Proficiency of individual's training is documented and maintained. The QM maintains procedures for diagnosing internal problems and outlines a series of corrective action steps to be taken. The laboratory internal quality system documented in the QM and each particular test standard operating procedure is reviewed periodically in

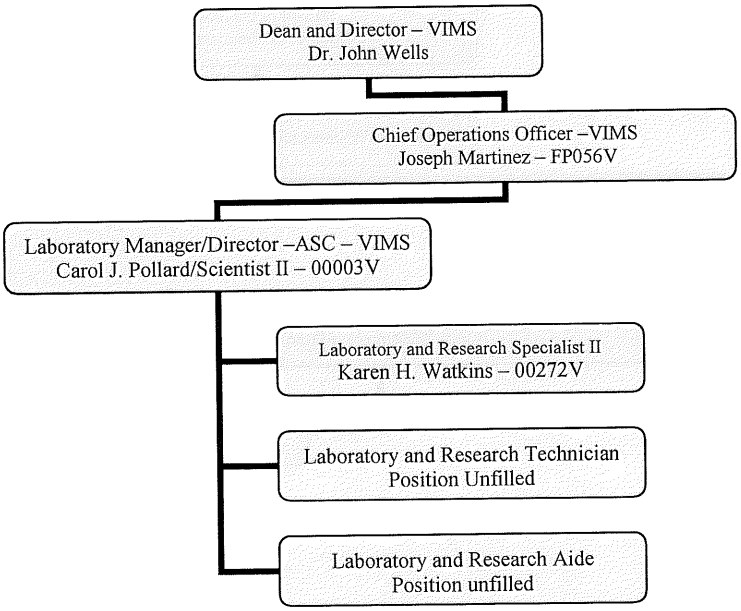
order to maintain consistency of testing, precision and accuracy of data results, sample control and all internal documentation.

# II. Organization, Management Structure and Personnel

## 2.1 College of William and Mary



## 2.2 Current Personnel - Virginia Institute of Marine Science



2.3 Personnel documentation is managed by the human resources department of the College of William and Mary. Individual personnel files are also maintained in the laboratory manager's office at the Analytical Service Center and include yearly evaluations, training forms and any other documentation of personnel performance generated by the laboratory manager.

2.4 Job descriptions are also maintained in the laboratory personnel file and are compiled by the Commonwealth of Virginia, Department of Human Resource Management (DHRM) for the state of Virginia and may be accessed via the internet at: http://www.dhrm.state.va.us

### 2.4.1 Scientist I

General Description: The Scientist I role provides career tracks for scientists that perform work in a laboratory, in the field, and/or for scientific research. Employees' responsibilities range from entry-level performing standardized scientific tests and research functions using established protocols, to performing independent analysis/studies and serving as technical advisors or lead workers. Employees conduct research, field and/or technical investigations and surveys, laboratory and/or statistical analyses and data interpretation.

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| **COMPLEXITY** | • Applies knowledge of scientific laboratory, field or research projects or programs for assigned scientific discipline.<br>• Applies knowledge of scientific principles and procedures and federal, state and local laws pertaining to the assigned discipline.<br>• Follows prescribed protocols or methods for standardized tests, but may exercise independent decision making as more difficult tests are assigned.<br>• May perform independent and complex studies, such as the analysis and interpretation of atypical test results.<br>• Assignments include: diagnosis of diseases and identification of disease agents; quantitative and qualitative analyses on complex chemical compounds; conducting and reporting results of analyses of geological, biological and environmental specimens or wildlife management research; collecting or preparing samples, researching; operating and maintaining instruments; writing reports (some for publication), making presentations, and/or providing expert testimony.<br>• Frequent contact with federal, state and local governments, laboratory colleagues, private organizations and the general public to report and interpret results, provide consultation, technical assistance and make presentations. |
| **RESULTS** | • Accurate scientific testing, research, or consultation to facilitate informed decision making minimizes adverse impact to health, safety, economic development, and the environment.<br>• Effective dissemination of information. |
| **ACCOUNTABILITY** | • Works independently under established protocols.<br>• May seek advice from supervisor or other resources on complex issues.<br>• May lead team or special project.<br>• May be responsible for day-to-day operation of program(s).<br>• May provide technical guidance to laboratory or field personnel. |

### 2.4.2 Scientist II (Technical- Lead)

General Description: The Scientist II role provides career tracks for scientists that perform a preponderance of advanced work and serve as an expert in a laboratory, in the field, and/or for research; or, for scientist supervisors. The first career track in this role is for employees performing complex scientific research projects or program oversight having a broad scope of responsibility. The second career track is for scientists that continue to deliver scientific services while assuming supervision of professional scientific staff and performing administrative responsibilities.

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| **COMPLEXITY** | • Applies knowledge of complex scientific laboratory, field or research projects or programs having a broad scope of responsibility in the assigned scientific discipline.<br>• Applies knowledge of scientific principles and procedures and federal, state and local laws pertaining to the assigned discipline.<br>• Assignments range from technical expert to state expert in a specialized scientific area.<br>• May originate new scientific concepts or approaches and may present or publish findings.<br>• May be responsible for auditing laboratories for quality assurance.<br>• Responsibilities include: independent examination, analysis and oversight of a variety of scientific samples; determining or developing testing or research methods and procedures; designing, conducting and evaluating complex analyses, scientific surveys and investigations, laboratory experiments and research; leading the development of research projects and databases; identifying and protecting endangered plant and animal species; writing articles or reports (some for publication), making presentations, and/or providing expert witness testimony; auditing and conducting inspections of laboratories; operating and maintaining complex instrumentation; leading critical investigations; serving as a technical expert and/or providing technical training to other professionals; guiding and reviewing the work of other scientists.<br>• Frequent contacts with federal, state and local governments, academic and scientific staff, private organizations, and the general public to report and interpret complex results, resolve customer concerns, respond to inquires, provide expert consultation and technical assistance, and make presentations. |
| **RESULTS** | • Accurate scientific testing, research, consultation and appropriate test and methodology design to facilitate informed decision making minimize adverse impact to health, safety, economic development, and the environment.<br>• Effective dissemination of information.<br>• Employees may conduct research and develop recommendations that lead to cost-effective and innovative and improved |

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| | technology, technical operations and services. |
| **ACCOUNTABILITY** | • Independently performs scientific analyses and research.<br>• Serves as lead workers and experts who are responsible for the review of work products and the accuracy of results.<br>• Provides technical guidance and serves as a technical expert. |

### 2.4.3 Scientist II (Supervisor)

General Description: The Scientist II role provides career tracks for scientists that perform a preponderance of advanced work and serve as an expert in a laboratory, in the field, and/or for research; or, for scientist supervisors. The first career track in this role is for employees performing complex scientific research projects or program oversight having a broad scope of responsibility. The second career track is for scientists that continue to deliver scientific services while assuming supervision of professional scientific staff and performing administrative responsibilities.

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| **COMPLEXITY** | • Applies knowledge of complex scientific laboratory, field or research projects or programs for assigned discipline.<br>• Applies knowledge of scientific principles and procedures and federal, state and local, laws pertaining to the assigned discipline.<br>• Applies knowledge of the principles and practices of supervision and administration.<br>• Frequent contacts with federal, state and local governments, private industry; hospitals, laboratories, physicians and clinics, academic and scientific staff, and the general public to report and interpret complex results, resolve customer concerns, respond to inquires, provide expert consultation and technical assistance, and make presentations exchange or provide research information. |
| **RESULTS** | • Accurate scientific testing, research, and supervision, and appropriate test and methodology design, minimize adverse impact to health, safety, economic development, and the environment.<br>• Supervisory actions and decisions impact the work team's ability to achieve program goals and objectives.<br>• Impacts public confidence, consumer satisfaction and overall program success. |
| **ACCOUNTABILITY** | • Supervises staffing, budget and administers projects.<br>• Provides guidance and training to other scientists.<br>• Serves as lead workers and experts who may be responsible for the review of work products and the accuracy of results.<br>• Responsible for the review of work products and the accuracy of results. |

### 2.4.4 Laboratory and Research Manager

General Description: The Laboratory and Research Manager role provides career tracks for managers of laboratories in a teaching, research, clinical, service or regulatory setting. Employees are responsible for making administrative decisions related to all laboratory operations and exercise broad-based administrative responsibility for all laboratory functions and personnel.

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| **COMPLEXITY** | • Applies knowledge of the principles and methods of laboratory administration and management.<br>• Applies knowledge of the theory and application of lab methods and instrumentation for the assigned discipline(s).<br>• Ability to plan, lead and evaluate the work of professional staff.<br>• Frequent contacts, with regulatory boards or governmental entities regarding standards, quality assurance, and lab procedures.<br>• Frequent contact with internal departments regarding administrative matters and with vendors concerning equipment and supplies.<br>• Frequent contacts with researchers, staff and students to provide information, consultation or direction concerning laboratory operations.<br>• Plans, manages and evaluates the work of professional staff; develop goals, objectives and timetables; develop and monitor budgets; determine and implement technical methodologies, ensure that quality control standards and safety procedures are in compliance with government regulations and laws.<br>• May teach and/or serve as technical experts. |
| **RESULTS** | • Proper management of laboratory operations ensures the scientific accuracy, timeliness and quality of services and impacts public health, safety, and the environment. |
| **ACCOUNTABILITY** | • Responsible for the provision of quality and timely laboratory services.<br>• Manages work of subordinate supervisors and professional and technical staff engaged in laboratory operations and research.<br>• Establishes and monitors quality control procedures.<br>• May ensure compliance with established standards and guidelines as set forth by accredited programs. |

## 2.4.5 Laboratory and Research Specialist II (Supervisor)

General Description: The Laboratory and Research specialist III role provides career tracks for both employees who are laboratory specialists and research specialists performing advanced to expert level responsibilities and for supervisors in a laboratory, field setting, animal care facility or for scientific research. The first track is for positions conducting complex scientific procedures or research for a laboratory or program manager, principal investigator or project director. The second track is for laboratory and research specialists who continue to deliver scientific services while assuming additional supervisory and administrative responsibilities.

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| **COMPLEXITY** | • Applies knowledge of supervisory principles and practices.<br>• Applies knowledge of assigned technical or research area.<br>• Participates or leads in the design, modification and performance of laboratory or research projects.<br>• Frequent contact with principal investigators, research faculty, laboratory personnel and students to discuss policies, procedures and methodology; coordinate research projects; report and interpret results; and provide consultation, and technical assistance.<br>• Responsibilities include: writing grant proposals and identifying funding sources; designing and directing complex research projects in consultation with a principal investigator; modifying and developing laboratory and field procedures, quality control processes and determining work methods; reviewing the scientific work results; directing animal care according to applicable federal laws and regulations; hiring and training personnel or students; approving procurement, budgeting, other administrative duties; and teaching a laboratory section of college level students. |
| **RESULTS** | • Accurate scientific testing, quality control, research, or consultation and supervision minimizes errors in laboratory findings or research, and impacts the success of grant proposals and scientific findings.<br>• Work impacts the health, safety and well being of the general public, the environment, industry, research, and animal health and welfare. |
| **ACCOUNTABILITY** | • Independently supervises laboratory research operation or animal care facility to include business functions and accountability for services provided to administrators, faculty, and other users.<br>• Responsible for program and staff supervision.<br>• Leads teams or special projects.<br>• May be required to ensure compliance with established standards and guidelines as set forth by accredited programs. |

## 2.4.6 Laboratory and Research Specialist II (Advanced Expert)

General Description: The Laboratory and Research specialist II role provides career tracks for both employees who are laboratory specialists and research specialists performing advanced to expert level responsibilities and for supervisors in a laboratory, field setting, animal care facility or for scientific research. The first track is for positions conducting complex scientific procedures or research for a laboratory or program manager, principal investigator or project director. The second track is for laboratory and research specialists who continue to deliver scientific services while assuming additional supervisory and administrative responsibilities.

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| **COMPLEXITY** | • Applies knowledge of assigned technical or research area.<br>• Participates or leads in the design, modification and performance of laboratory or research projects.<br>• Frequent contact with private business, other state agencies or government entities, principal investigators, faculty, laboratory staff, students and researchers, and/or the general public to report and interpret results, provide consultation and technical assistance, and discuss research projects.<br>• Responsibilities include: participating in or leading work in the design, modification, evaluation and performance of laboratory, field surveys or research procedures; writing or modifying computer programs to analyze data and generate reports; researching literature related to project/procedures; conducting experiments; writing segments of reports and manuscripts; coordinating grants and budgets; assisting lower level staff; troubleshooting instrument problems and performing preventive maintenance on equipment. |
| **RESULTS** | • Accurate scientific testing, research, and consultation minimizes errors in laboratory findings, scientific research and fieldwork.<br>• Proper application of procedures impacts outcomes of tests and research, and the safety, health, and well being of the general public, research staff, and laboratory animals through identification of scientific findings, and contagious and benign disease sources. |
| **ACCOUNTABILITY** | • Independently performs and provides consultation on specialized laboratory/research procedures and projects.<br>• Independent decision-making on appropriate methods, design and data interpretation.<br>• Some positions ensure compliance with established standards and guidelines as set forth by accredited programs.<br>• Independently monitors, evaluates and analyzes quality control results and determines corrective action as needed. |

### 2.4.7 Laboratory and Research Specialist I

General Description: The Laboratory and Research Specialist I role provides career tracks for autopsy technicians, laboratory specialists, research specialists, assistants to chemists, microbiologists and other scientists who support in the performance of various technical, scientific, analytical or animal care activities for clinical, research, regulatory or laboratory programs, or in a veterinary hospital or animal care facility. Laboratory and research support responsibilities range from journey-level to advanced-level.

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| **COMPLEXITY** | • Applies knowledge of scientific/technical principles, practices, and regulatory requirements of functional areas.<br>• Performs a variety of procedures supporting clinical, research, field research, service or regulatory and/or diagnostic laboratory programs.<br>• Duties may include performing standardized or specialized scientific or clinical procedures, performing preliminary procedures to prepare, expedite, and facilitate further scientific examination and training, assisting in veterinary surgical procedures, analysis and compilation of data, communicating findings/research results, animal care, repairing and maintaining equipment and ordering supplies.<br>• May have contact with, private business, other state agencies or government entities, faculty, students and researchers, and/or the general public to communicate results and explain laboratory procedures or regulatory requirements. |
| **RESULTS** | • Proper application of procedures impacts outcomes of tests and research, legal evidence, and the safety, health, and economic well being of the general public, research staff, environment and/or animals through identification of scientific and legal findings, contagious and benign disease sources.<br>• Performance of duties may impact the level of public confidence and consumer satisfaction.<br>• May be required to adhere to established standards and guidelines as set forth by accredited programs. |
| **ACCOUNTABILITY** | • Independently performs standardized or specialized procedures and seeks advice on more complex or non-routine issues.<br>• Decision-making has moderate to significant impact on program's success.<br>• Exercises judgment and decision making to determine appropriate procedures; compiles data, documents and communicates findings.<br>• May lead, train or supervise students, staff or coordinate program activities. |

### 2.4.8 Laboratory and Research Technician

General Description:The Laboratory and Research Technician role provides career tracks for laboratory technicians, geological technicians, and laboratory animal caretakers that perform a variety of laboratory and/or research tasks in support of research/teaching, clinical services, geological services, field research or a regulatory laboratory. Employees are responsible for a variety of standard procedures that range from routine to specialized in the areas of cleaning and decontamination; performing standard/routine laboratory testing; sectioning and preparing rock and mineral samples for various mineralogical and laboratory analyses; preparing samples; recording data, and operating and maintaining tools and equipment.

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| **COMPLEXITY** | • Applies knowledge of sanitation techniques and laboratory safety.<br>• Applies knowledge of research and testing procedures and techniques, and of animal handling, care and welfare.<br>• Performs a variety of procedures supporting laboratory, research, clinical or autopsy and necropsy services, or geological services.<br>• May provide limited surgical assistance.<br>• Follows established directions and procedures.<br>• Frequent contacts with co-workers, supervisors, students, faculty, and research staff to discuss study techniques or results or handling and care of animals. |
| **RESULTS** | • Proper laboratory, procedures impact outcomes of tests and research, and impact the safety, health, and well-being of laboratory animals, staff and the general public through the identification of scientific and legal findings or contagious disease sources.<br>• Proper sanitation procedures ensure appropriate laboratory testing and research conditions.<br>• Responsible for equipment in support of research, laboratory, or clinical programs or teaching services.<br>• May be required to adhere to established standards and guidelines as set forth by accredited programs. |
| **ACCOUNTABILITY** | • Responsible for generally well defined procedures supporting laboratory, testing, research or clinical programs.<br>• Decision-making and judgment is typically based on clearly defined procedures, although skilled positions may exercise independent judgment.<br>• Refers non-routine issues to supervision.<br>• May lead other staff, activities, or provide guidance and leadership to students and interns.<br>• Development of competencies may lead to broader, more responsible assignments. |

### 2.4.9 Laboratory and Research Aide

General Description: The Laboratory and Research Aide role provides career tracks for laboratory aides and laboratory animal caretakers who follow a highly structured schedule in performing simple, repetitive tasks under the immediate supervision of higher-level laboratory, or research personnel. Typical duties include washing and sterilizing glassware and equipment; receiving, distributing and preparing packages, samples and supplies; preparing sample test kits, and preparing media.

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| **COMPLEXITY** | • Applies knowledge of basic sanitation techniques, laboratory safety procedures, research procedures, animal handling/care and hygiene, and use of laboratory equipment.<br>• Performs routine work with limited variation.<br>• Follows established directions and procedures.<br>• Limited contacts outside of co-workers and supervision.<br>• May perform a variety of tasks related to animal care such as feeding and caring for animals. |
| **RESULTS** | • Proper sanitation procedures ensure appropriate laboratory testing and research conditions.<br>• Provides responsible care of animals.<br>• Responsible for equipment in support of research, laboratory, or clinical programs or teaching services.<br>• May be required to adhere to established standards and guidelines as set forth by accredited programs. |
| **ACCOUNTABILITY** | • Decision-making and judgment is limited to assigned functional area and based on clearly defined procedures and guidelines or under direct supervision. |

## 2.5 Management Responsibilities and Personnel Qualifications

Responsible Official: Joseph Martinez
Quality Assurance Officer: Carol J Pollard
Laboratory Manager/Director: Carol J Pollard

a. The Laboratory Manager is responsible for:
   - Ensuring the supervision of all personnel employed by the laboratory.
   - Ensuring the quality of data produced by the laboratory
   - Oversight of training and personnel.
   - Updating laboratory procedures, operations of instruments, and laboratory support equipment.
   - Appointing personnel in the absence of laboratory staff.
   - Reviewing and approving any changes to the quality manual and associated quality documentation.
   - Signing final reports of laboratory data to be distributed to the client

b. The Quality Assurance Officer is responsible for:
   - Implementing and overseeing the quality system
   - Reviewing and approving any changes to the quality manual and associated quality documentation

c. The Qualified Laboratory and Research Specialist is responsible for:
   - Adhering to quality assurance plan
   - Performing technical laboratory tests and procedures
   - Reporting deviations from the quality assurance plan and taking necessary action to bring the quality management system back into compliance.

## 2.6 Biographical Sketches

Biographical sketches of the employees involved in reporting analytical data are updated regularly and maintained in the Quality Manual.

2.6.1 Carol J. Pollard – Laboratory Director/ Scientist II (Supervisor)

FORMAL EDUCATION: M.S. Marine Science, College of William and Mary, 2002
B.S. Environmental Science, Florida Institute of Technology, 1982

EXPERIENCE: 1995-present – Laboratory Director, Analytical Service Center, College of William and Mary

DUTIES: Responsibilities include establishing the laboratory goals, developing and monitoring the budget, coordinating personnel and maintaining safety and quality control programs. Also, assists scientists and students in experimental design and research projects. Conducts research in analytical method development. Also, coordinates with governmental authorities, particularly with Virginia department of environmental quality and the environmental protection agency.

EXPERIENCE: 1990-1995 – Laboratory Director/Contract Specialist. Microbac Laboratories, Inc.

DUTIES: Director of an environmental laboratory composed of staffing 30 professional and technical personnel. Responsibilities included managing all aspects of the laboratory from production, quality control, customer service and acquisition of laboratory certifications, to sales and marketing. Designed and implemented operating procedures for seven departments. As director, responsibilities included preparing Quality Assurance Manual, Safety Manual, and Analytical Procedures Manual; also introduced new analytical techniques in order to diversify services and to meet regulatory testing requirements utilizing a variety of analytical instrumentation and procedures. Trained technical staff and mentored students. Coordinated with government staff (VADEQ and EPA). As contract specialist, was responsible for acquiring multiple new contracts totaling more than $7million in annual revenue. Responsible for technical proposals and cost proposals for 22 sister laboratories as well as the Newport News facility, and pursued new business opportunities.

EXPERIENCE: 1987 – 1990, Inorganic Chemistry Department Manager/ Senior Organic Analyst, James R. Reed and Associates.

DUTIES: Oversee operations and production of the inorganic chemistry laboratory. Responsibilities included hiring and training of personnel, evaluations, budgeting, scheduling, purchasing and maintenance of the chemical inventory; also client point of contact for pricing, turnaround times and technical information. Instrumentation maintained included atomic absorption spectrophotometry (flame and furnace), autoanalyzers for nutrient and contaminant analysis, and spectrophotometers. Monitored preparation and analysis of environmental samples including water, wastewater, soil, sludges and hazardous wastes using EPA approved methodology; also drafted procedures of analysis for complex organic constituents to meet regulatory requirements. Responsible for maintenance of analytical instrumentation including gas chromatographs equipped with purge and trap units for volatile analysis, ECD and FID detectors for pesticide, herbicide and PCB's. Responsibilities included chemical and supply inventory.

EXPERIENCE: 1983-1987 Analytical Chemist, Bionetics Laboratory, Inc. and Interscience Research.

DUTIES: Responsible for the analysis of water, wastewater, soil, sludge and wastes utilizing wet chemistry techniques following EPA approved methodology; also analysis of compressed air for breathing quality for certification of cleanliness using gas chromatography techniques. Responsible for analysis of bulk materials and air samples for asbestos using phase contrast and polarized light microscopy; also responsible for quality control and statistical analysis to define acceptable results. Duties included data reporting and logging and tracking of samples for major contracts.

2.6.2 Karen Watkins – Laboratory and Research Specialist I

FORMAL EDUCATION: A.A. Business Administration, Rappahannock Community College, 2002

EXPERIENCE: 2008-present – Laboratory and Research Specialist I, Virginia Institute of Marine Science

DUTIES: Analyzing samples for nutrient levels including ammonia, nitrate, nitrite, and phosphorus on water, sediment, and particulate samples. Maintain and operate the Skalar autoanalyzer as well as the Lachat autoanalyzer. Perform in house analysis of Total Suspended Solids,

grain size particle analysis and Rapid Sand Analysis. Login samples for tracking documentation. Write revised Standard Operating Procedures (SOP's) for Analytical Service Center; and assisting students with analytical methods.

EXPERIENCE: 2007-2008 – Laboratory and Research Aide, Virginia Institute of Marine Science

DUTIES: Analyzing particulate samples for chlorophyll and TSS/TFS. Analyze sediment samples for moisture content and grain size. Prepare filters for sample preparation. Acid wash sample bottles and glassware for general use and perform routine maintenance of the laboratory.

EXPERIENCE: 1990-1993 Microbac Laboratories- Laboratory and Production Manager.

DUTIES: Oversee operations and production of the inorganic chemistry and microbiology laboratories. Assist with the Microbiology certification to include writing SOPs and inspections. Point of contact for multiple clients. Instrumentation maintained included atomic absorption spectrophotometry (flame and furnace), auto analyzers for nutrient and contaminant analysis, and spectrophotometers.

EXPERIENCE: 1988-1990 – Laboratory Technician, NASA Langley

DUTIES: Preparing and analyzing samples for air exhaust components of prescribed biomass burns. Utilize gas chromatography for analysis of CO2 and NO2.

EXPERIENCE: 1987-1988 Microbiology Technician, James R. Reed and Associates.

DUTIES: Perform analysis on drinking and waste water to include Total and Fecal Coliform, total plate count and biological oxygen demand.

EXPERIENCE: 1982 – 1987 Laboratory Technician, Bionetics Analytical Laboratory

DUTIES: Performing Analysis on drinking water and wastewater. Analysis included chemical oxygen demand, biological oxygen demand, total suspended solids. Microbiological techniques included total and fecal coliform counts. Logged in incoming samples.

# III. Ethics Policy and Data Integrity

The laboratory has developed an ethics policy and established procedures to educate and train personnel in their ethical and legal responsibilities. The laboratory performs routine data review to ensure the records are complete and that they demonstrate ethical conduct. Data integrity procedures are part of this quality manual.

The ethics agreement defines the employees' ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions.

## 3.1. Ethics Policy

Ethics is a set of moral principles, a code for right and wrong which conforms to accepted professional practices. All employees at all times shall conduct themselves in an honest and ethical manner. An employee must report any suspected unethical behavior or fraudulent activities to a supervisor. An employee has the right to remain anonymous.

"Laboratory fraud is defined as the deliberate falsification of analytical and quality assurance results, where failed method and contractual requirements are made to appear acceptable during reporting."[a] It is the intention of this laboratory to enact practices in which to minimize fraud and to detect fraud which may be occurring. This may be achieved by enhancing oversight activities, identifying "best practices" and adherence to the quality control steps outlined for specific analytes in standard operating procedures which are maintained and kept up to date, clearly identifying QA/QC requirements to personnel, internal audits and performance evaluations by split sample analyses and proficiency testing.

Data validation and periodic reviews of analytical runs are a way of comparing data with established quality criteria to ensure that the data generated and reported are adequate for their intended use. During the data review and validation process the reviewer will assess the standard calibration, acceptability of the quality control samples including standard reference materials, spikes, duplicates, drifts and blanks, calculations, and notations by the analyst. The analyst will be informed of any questionable practices or data which may result from the reviewer findings. The analyst will be asked to address the questions and provide the reviewer with needed information to finalize the assessment and verify that the data reported is of the acceptable quality as established by the QA/QC guidelines and data quality objectives.

3.2. Examples of unethical behavior include, but are not limited to the following:

- Improper manipulation of data or software
- Improper handling of data errors, non-compliant data, or QC outliers
- Lack of reporting unethical behavior by others
- Artificially fabricating results
- Misrepresenting data such as peak integration, calibration, tuning, or system suitability
- Improper clock setting to meet holding times
- Intentional deletion of non-compliant data

3.3. Ethics training will be conducted during the initial orientation and training of newly hired personnel. The training includes: A copy of this policy, a powerpoint presentation , a copy of "*On Being a Scientist: Responsible Conduct in Research*" provided by the Committee on Science, Engineering, and Public Policy, National Academy of Sciences and introduction to internal documents to include :

a. *Best practices for the Detection and Deterrence of Laboratory Fraud* , March 1997 Version 1, written by the California Military Environmental Coordination Committee
b. *Laboratory Fraud: Deterrence and Detection*, June 25, 1999 from the Office of The Inspector General
c. *Guidance on Environmental Data Verification and Data Validation* , November, 2002. EPQ QA/G-8

3.4. A newly hired employee will complete the Ethics Agreement and it will be maintained in the personnel file in the laboratory manager's office. Training and Ethics Agreement will be renewed yearly.

3.5. Ethics Agreement

**Ethics Agreement**
**Document Control # 00045**

Analytical Service Center
Virginia Institute of Marine Science
Rt 1208 Greate Road
Gloucester Point, VA 23062

**ETHICS AND DATA INTEGRITY AGREEMENT**

I. I, _____, state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at The Analytical Service Center , Virginia Institute of Marine Science.

II. I agree that in the performance of my duties at the Analytical Service Center, Virginia Institute of Marine Science, that

a. I shall not intentionally report data values that are not the actual values obtained;
b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and
c. I shall not reprpesent another individual's work as my own.

III. I agree to inform the Analytical Service Center, Virginia Institute of Marine Science, of any accidental reporting of non-authentic data by myself in a timely manner.

IV. I agree to inform The Analytical Service Center, Virginia Institute of Marine Science of any accidental or intentional reporting of non-authentic data by other employees.

_____

*(Signature)*

_____

*(Date)*

# IV. Document Control

The purpose of the document control system is to ensure that only the most recent revisions of SOPs, worksheets, forms, logs, etc. are available to the appropriate personnel, are timely, and receive the required approvals. All internal regulatory documentation, standard operating procedures, work instructions, service manuals, and product instructions are under document control. The Laboratory Manager/Quality Assurance Officer is responsible for the document control system and keeps a master list of the location of all documents and their current revision. The Laboratory Manager/Quality Assurance Officer approves all newly released documents and revised documents. Worksheets, forms, and logbooks are designed to include all information pertinent to the analysis or task performed. Each worksheet, form, and logbook includes a unique identifier. Worksheets and forms have a revision number and effective date. Appendix A lists documents in use at the ASC.

## 4.1 SOPs, Worksheets, Forms and Logs

4.1.1 When a Standard Operating Procedure (SOP) is revised, either on a routine basis or for a specific reason, such as change in method, the method is approved, a document # assigned and a copy is placed in the SOP manual as well as at the bench. All old copies are retired into the file cabinet labeled "Retired Documents" in the laboratory manager's office. Electronic files are moved into Z:\retired documents file. New revision electronic files are saved with the revision number.

4.1.2 When an SOP is no longer in use, at the laboratory manager's approval, all known copies are removed from the work area. One copy is labeled "retired" and the date and initials of the analyst retiring the method are documented on the front. The one copy is filed in the file cabinet labeled "Retired Documents" in the laboratory manager's office. Electronic files are moved into Z:\retired documents file. Extra copies are either shredded or put into the recycle bin.

4.1.3 When a log is retired, a hard copy of the log is made and filed in the file cabinet labeled "Retired Documents" in the laboratory manager's office. Electronic files are moved into Z:\retired documents file.

## 4.2 Master List of Document Control Numbers

4.2.1 A master list of all document control numbers is maintained at Z:\ascdocs\internal\logs\document control numbers.doc

## 4.3 Aspen Worksheets, Raw Data and Final Reports

Aspen is the laboratory information system (LIMS) currently operating the ASC Using Aspen, laboratory worksheets are generated by the analysts for daily run of analytes. Aspen is also used for logging in samples, generating identification login #, and is used for reporting of results, final data reports, billing and archiving.

4.3.1 When an Aspen Worksheet is completed, it is attached to all the raw data and filed in the laboratory manager's office in the appropriate file folder for approval. After approval it is filed in rm 104 in the Water Quality building in the appropriate file cabinet. All Aspen data sheets are maintained on the Z: drive in appropriate method file

4.3.2 Final reports after billing are filed in the laboratory manager's office in the file cabinet labeled "final data reports" All final reports are stored electronically on Z:\ascdocs\data*"year"*\reports*"lognumber"*

## 4.4 Chain of Custody

Chain of custody forms (Document Control Number: 00002) are supplied to clients who request them. Otherwise paperwork must accompany samples stating from whom they came, address and contact information, sample identification, collection date and analyses to be preformed. Samples are logged into Aspen LIMS and chain of custodies are filed with login information in the laboratory manager's office in the current year binder. Old chain of custody forms are filed in rm 105 of the Water Quality building according to year and are maintained for a minimum of 7 years.

## 4.5 Purchase Orders

Purchase orders are initiated by the laboratory manager. Copies of the originals and/or eVa generated POs are kept on the shelf in the laboratory located in the Seawater Research Laboratory (SRL) building rm 116. When supplies arrive they are checked against the original order, signed off on with initials and date received.

## 4.6 Billing

Billing is initiated by the laboratory manager. After report generation, the report and bills generated by Aspen are put into the "to be billed" file in the laboratory manager's office. Periodically the laboratory manager fills out a JV form (Document Control Number: 00037) to be submitted to Sponsored Research in Waterman's Hall. A hard copy is generated and an electronic file sent to Sponsored Research. The hard copy is maintained with the bills in the laboratory

manager's office on the shelf. Bills and JVs are maintained for a minimum of 3 years in hard copy and 7 years electronically.

## 4.7 Training Documents

Training documents are maintained in the laboratory manager's office in the personnel file.

## 4.8 Hazardous Waste Forms

Blank hazardous waste forms are kept on the Z: drive at Z:\ascdocs\internal\bulkwaste.doc (Document Control Number: 00038) or Z:\ascdocs\internal\hazardous waste.doc (Document Control Number: 00039). Completed documents are emailed to the safety office and maintained at Z:\ascdocs\internal\completed forms*"date"*hazardouswaste.doc. Hard copies are not maintained in the office but are maintained in VIMS safety office.

## 4.9 Archiving Raw Data and Forms

Raw data and handwritten forms are maintained in the Laboratory Manager's office for a minimum of seven years. Files transferred to a more permanent storage area are maintained according to year. Anyone accessing these files must seek permission from the laboratory manager and the manager will fill out the "archive data access log" which is document control number 00067.

## 4.10 Protecting Shared Worksheets

Shared excel worksheets which have imbedded calculations are protected in order to not allow access to sensitive areas of the worksheet that could be changed, manipulated, or deleted. In order to protect the worksheet in excel, under the heading "Review", click on the tab "Protect Worksheet". Highlight cells which need to be protected from manipulation including calculations. Then click "protect worksheet". When technicians pull up that worksheet to enter data, only those cells that are unprotected are allowed to enter information.

# V. Subcontracting Sample Analysis and Review of New Work

## 5.1 Subcontracting of Sample Analysis

It is the policy of the ASC that no subcontracting will take place.

## 5.2 Review of New Work

All new work is initiated by the Laboratory Manager who delegates responsibilities for the new work according to available resources. Staff will meet prior to initiation of new work in order to determine if appropriate facilities and resources are available. The plan for any new testing shall be reviewed and approved by the Laboratory Manager before commencing such work. After agreement is reached, facilities and resources are organized to efficiently perform the work. For any new testing requirements, the designated employee shall write a standard operating procedure and demonstrate capability to perform those tests prior to reporting results. The SOP(s) shall be under document control, and a Demonstration of Capability Statement(s) shall be on file.

## VI. Purchasing

Purchasing procedures follow the procurement requirements defined by the State of Virginia and the College of William and Mary. The technical specifications for equipment and supplies for the laboratory are defined in the laboratory SOPs. The laboratory technician documents the materials or equipment to be ordered. Orders under $5000.00 are reviewed and approved by the Laboratory Manager. In order to facilitate purchasing, a completed purchase order must be given to an authorized purchasing officer of the Virginia Institute of Marine Science. All orders are placed through the State of Virginia purchasing software at www.eva.state.va.us.

If the purchase exceeds $5000.00, bid options or sole source paperwork must be submitted to the College of William and Mary Office of Procurement. Only when a vendor has been selected by the Office of Procurement or a sole source plan accepted may the order be placed by an authorized purchasing officer of the Virginia Institute of Marine Science.

Procurement procedures must be conducted in a fair and impartial manner and fully conform to state law, the vendor's manual and the Purchasing Manual for Institutions of Higher Education. The DGS/DPS July 2006 Virginia Public Procurement Act (VPPA) mandates that competition in procurement is utilized to the maximum feasible extent.

The VIMS Procurement office is physically staffed weekly by a senior procurement officer on a rotating basis. For additional and more detailed information, please refer to the Pocket Procurement Guide (pdf).

## VII. Complaints

All complaints about the laboratory's activities are documented in a complaint file maintained in the laboratory. The file contains the date and name of the person receiving the complaint, a description of the complaint, source of the complaint, the resolution, and any written material accompanying the complaint. A corrective action form is used to document the complaint (see Section IX).

The Laboratory Manage/Quality Assurance Officer investigates complaints and promptly investigates all areas of activity and responsibility involved. The written results of the investigation including actions taken by the laboratory are reviewed, signed and dated by the Laboratory Manager/Quality Assurance Officer.

## VIII. Departure from Policies or Standard Operating Procedures

The Laboratory Manager has responsibility for ensuring adherence to the laboratory's policies and procedures. Arrangements for known and controlled departures from documented policies and procedures are allowed. Planned departures do not require audits; however, the departure will be fully documented and include the reason for the departure, the affected SOP(s), the intended results of the departure and the actual results. The procedure used to document any specific departure from policies or standard operating procedures and the data affected is the same as the corrective action procedure. (See Section IX)

## IX. Corrective Action

Corrective actions are the result of concerns regarding work performed by the laboratory, detected problems, or nonconformance and may be from clients, laboratory personnel, assessors or any person or organization with concerns. Records of the concern, nonconformance or complaint and subsequent actions are maintained.

The laboratory takes corrective action whenever unacceptable conditions exist or departures from policies and procedures occur. The following indicators are used to determine unacceptable conditions:

- QC samples outside of the established acceptance criteria
- Calibrations outside acceptable criteria
- Equipment failure
- PT studies outside acceptable limits
- Non-conformance identified during internal reviews
- Non-conformance identified during on-site assessments
- Non-conformance or problems identified after receiving a question or complaint

Once an unacceptable condition is identified, the laboratory investigates the problem and outlines a corrective action plan.

Corrective action for analytical discrepancies may include, but is not limited to, one or all of the following:

- Re-analysis of samples
- Re-calculation of results
- Re-calibration of instrument
- Preparation of new standards
- Re-analysis of blanks
- Dilution of samples
- Additional analyst training
- Replace equipment or supplies
- Re-sampling
- Recalled analysis results or amended reports

The need for corrective action will be initiated by the data generator. This may be at the time of QC data assessment and plotting of control samples. The data generator upon discovering information which exceeds the predetermined (historic) or listed acceptance limits will perform the following:

- Note the problem exists on the data run sheet
- Check methodology- review for performance failure, interference or matrix problems, and calculations.
- Check calculations and data transcription errors.
- Check reagents and standard for preparation errors.
- Check the instrument- consult instrument log and maintenance reports
- Review operations manual for the instrument and seek technical help from technical help services.

The Laboratory Manager will be notified and discuss with the data generator the steps that have been taken, and findings. The outcome will depend on the ability to account for errors. Otherwise reruns will be performed and QC checks implemented throughout the analytical run. The Laboratory Manager will then release the rerun data if QC checks are determined to fall within acceptable range. Otherwise further investigation will be necessary before any results are released.

Records of corrective actions will be documented on the Corrective Action Form (Laboratory Control Number: 00043), Fig. 9.1 and maintained by the Laboratory Manager. Procedure changes affecting the EPA Chesapeake Bay Program will be documented on the Laboratory Action Form (Laboratory Control Number: 00004), Fig 15.1 and maintained by the Laboratory Manager. Procedure changes affecting the EPA Chesapeake Bay Program will be submitted to the program on the Chesapeake Bay Monitoring Program Procedure Modification Tracking Form.

Figure 9.1

CORRECTIVE ACTION FORM
Document Control Number: 00044

**ANALYSIS:** _____ **DATE:** _____

**EVENT NAME / CATEGORY** _____ **CA #** _____

**PERSON COMPLETING FORM (NAME, TITLE):** _____

***RECORD INFORMATION BELOW OR ATTACH ADDITIONAL SHEETS. PROVIDE DOCUMENTATION WHENEVER POSSIBLE.***

**EVENT DESCRIPTION:**

**EVENT RESPONSE / INVESTIGATION STEPS:**

**ROOT CAUSE DETERMINATION:**

## CORRECTIVE ACTION (CA) FORM (cont'd)

**ACTION(S) TAKEN TO RESOLVE ISSUE AND PREVENT RECURRENCE:**

| | | |
|---|---|---|
| **Submitted By:** | | |

| | | |
|---|---|---|
| **Approved By:** | Laboratory Director or Technical Manager | Date: |
| **Reviewer Comments or Additional Actions Recommended:** | | |

Corrective Action Closed By Laboratory Manager: Signature: _____

Date:_____

# X. Records Management

The laboratory has implemented a record management system that allows the historical reconstruction of all laboratory activities. The laboratory keeps a record of each environmental analysis for at least three years as required by environmental regulation. Hard copies of the raw data and final reports, as well as, electronic copies are available for the last 7 years.

The laboratory maintains the following records either in the laboratory manager's office or on the hard drive dedicated to the ASC (Z:) by the ITNS department at VIMS:

- Personnel records and training records
- Final reports
- Chain of custodies
- Benchsheet forms
- Raw data
- Maintenance and instrument logs
- Chemical reagent log
- Chemical inventory
- Oven temperature logs
- Freezer temperature logs
- Water purity logs
- Refrigerator temperature logs
- Balance calibrations
- Thermometer logs and certificates of calibration
- Certificates of analysis for reference materials
- Proficiency testing reports
- Chesapeake Bay Split data reports
- Blind audit reports
- Internal audit reports
- External audit reports
- Archived data log

## 10.1 Chain of Custody, Raw Data, Electronic Files, Final Report and Database Maintenance

10.1.1 After initial log in, into the Aspen LIMS system as described on pg. 25 (Section 4.3), the chain of custody is signed, dated and the log identification numbers notated. The chain of custody is filed in a three ring binder designated for that fiscal year. The binder for the current calendar year is located in the Laboratory Manager's office. Previous year's chain of custody forms are filed in the file cabinets located in the Laboratory Manager's office.

10.1.2 After an analytical run has been approved, raw data is transferred to the appropriate raw data file located in the laboratory manager's office by the analyst. After final report generation, the raw data is filed by date and analysis in the laboratory manager's office and maintained for 3 years in the office. These files may be transferred to in a storage facility for a total of 7 years.

10.1.3 Electronic files are maintained on the laboratory server maintained by VIMS ITNS department. The ASC is given a drive to store all electronic data which is backed up nightly by the ITNS department.

10.1.4 Final reports are printed to hard and filed in the laboratory manager's office.

10.1.5 The Aspen LIMS, which maintains all the information for sample tracking from the logging in of samples, data analysis, and quality control data is also maintained by the VIMS ITNS department on a designated hard drive for the ASC.

10.1.6 The ASC's hard drive is maintained by the ITNS department at VIMS located in Waterman's Hall and is backed up nightly.

10.2 Archiving electronic and hard copy data

10.2.1 At the beginning of the new calendar year, all information in Aspen LIMS associated with completed sample batches are archived. Under the heading "Archived Samples" select "Transfer to Archive". A pathway must be created to Z:NAL\Aspen 7.5\archives\(date). Select all the data to be transferred. This information is now all removed to the archive which may be accessed through the "Archived Samples" pathway.

10.2.2 Hard copy data transferred to a storage area is considered archived and may be accessed when necessary. That access will be documented in the archived data access log maintained on Z:NAL\ascdocs\internal\logs\archived data access log.

## XI. Internal Quality System Audits

The Laboratory Manager/Quality Assurance Officer arranges for an internal quality system review annually. The audit is carried out by trained personnel who are independent (if possible) of the activity being audited. The review assesses the requirements of the quality assurance manual against laboratory operations, and laboratory operations against the laboratory's quality assurance manual and SOPs.

The results of the audits are documented in writing. Where audit findings cast doubt on the validity or correctness of the data, the laboratory will take immediate corrective action. Any corrective actions are documented. The Laboratory Manager ensures that the corrective actions are discharged within the agreed upon time frame. Any client whose work was possibly adversely affected shall be notified in writing.

## XII. Management Review

The laboratory management reviews the laboratory quality system and its testing and calibration activities annually to introduce any necessary changes or improvements. The review takes into account the outcome of recent internal audits, assessments by external bodies (DCLS or DEQ), the results of inter-laboratory comparisons, the results of proficiency tests, any changes in the volume and type of work undertaken, feedback from authorities or others, and corrective actions. The findings and any corrective actions from this review are documented.

## XIII. Personnel Training

Before conducting any analysis, each prospective analyst (or trainee) receives training by another analyst or supervisor who has completed training (the trainer). An analyst in training is supervised by an experienced trainer who is capable of training the trainee with a high degree of competency.

In addition to in-house training, additional training may be provided to the analyst in the form of educational courses, professional seminars, and continuing proficiency testing.

Analyst training and performance is considered complete after the analyst has produced a successful initial demonstration of capability for the method for which he/she will be responsible. In addition, acceptable results from a series of proficiency testing samples or internal blind quality control samples are documented for the trainee. The approach toward training at the Analytical Service Center consists of a four step plan. The first phase of training is one of observation. The trainee is introduced to the method reference and the standard operating procedure (SOP) and is given time to observe a qualified analyst perform the process (step 1). The trainee will then practice the procedure using separate aliquots of samples previously analyzed by a qualified analyst. The trainee's results will be compared to the analyst's results (step 2). After the trainee has performed the procedure satisfactorily under the trainer's supervision, the trainee is given four (4) blind audit samples to perform independent of the trainer (step 3). All data produced at this time will be reviewed by the laboratory manager. The mean recovery and standard deviations will be calculated and assessed against documented acceptance criteria. This is to insure compliance with all QA/QC practices. The laboratory manager will review accuracy and precision results with the trainee. Once the laboratory manager has signed off on all training forms, the trainee then qualifies to perform the procedure on real world samples and report data according to QA/QC protocol, (step 4).

Documentation for training is found in the analyst's personnel file in the manager's office. A three-part form will be maintained on each analyst for training. Part I is the checklist common to all personnel at the ACS. Part II is the testing procedure's checklist when a trainee becomes qualified to run a particular procedure independently. Part II requires both technical approval and quality control approval. Part III is a chart of all procedures the analyst has qualified for. The laboratory manager is responsible for maintaining the training documents. Analysts must complete the 4 part training program in order to become proficient in analyzing samples and reporting data.

13.1 Description of Analyst Testing Program at the ASC:

13.1.1 The trainee shall obtain a copy of the applicable test procedure (SOP) from the ASC Procedures Manual and report form. The trainee will study the test procedure and report forms to become

familiar with the equipment, terminology, test procedure, calculations, and test reports.

13.1.2 A qualified trainer will demonstrate the test procedure for the trainee.

13.1.3 The trainee will perform the test procedure under the guidance of the trainer until proficiency is obtained by successful analysis on replicate samples of pre-analyzed samples. The trainee will then independently analyze four (4) blind audit samples. The mean recovery and standard deviations will be calculated and assessed against documented acceptance criteria. The trainer will complete Part II of the training form and will be submitted to the laboratory manager for approval.

13.1.4 The trainee will independently analyze four (4) blind audit samples. Upon successful completion of the audit samples, the trainer will complete Part II of the training form and will be submitted to the laboratory manager.

13.1.5 The laboratory manager shall review the trainee documentation demonstrating that the trainee has successfully completed the hands-on training and demonstrates the ability to perform the test procedure appropriately. Part III of the trainee's record will be completed.

## 13.2 Training Forms

### 13.2.1 PART I – Training Form

**General Laboratory Checklist**

Name of trainee: _____

Trainer: _____

Date: _____

| | | |
|---|---|---|
| Laboratory Tour | ********** | |
| Safety | VIMS Safety Program completion | |
| | Fire Extinguishers | |
| | Eye Washes | |
| | Safety Shower | |
| | Absorbing pillows | |
| | Breathing protection | |
| | First Aid Kit | |
| | Flammable liquids cabinet | |
| | Acid storage | |
| | Hazardous waste labeling and storage | |
| Personal Safety Equipment | Safety Handbook | |
| | Lab Coats/aprons | |
| | Safety glasses | |
| | Dress Code | |
| Technical Systems | D.I. and R.O. water systems | |
| | pH/conductivity meters | |
| | Glassware preparation | |
| | Analytical Balance | |
| | Reagent Preparation | |
| Data Handling | Sample Log-in/C-O-C | |
| | Sample storage | |
| | Bench sheets | |
| | Lab equipment notebooks | |
| | Data approval | |
| Quality Assurance | Quality Manual | |
| | Specific test requirements | |
| | Authority | |

### 13.2.2 PART II – Training Forms

**Training Forms for Specific Procedures (ASC Procedures Manual)**

WATER

1) Filtering
2) Total Suspended and Volatile Solids
3) Chlorophyll A/ Pheophytin
4) Inorganic Nutrients (Ortho-phosphorus)
5) Inorganic Nutrients (Nitrite)
6) Inorganic Nutrients ( Nitrate/Nitrite)
7) Inorganic Nutrients (Ammonia)
8) Inorganic Nutrients (Silica)
9) Organic Nutrients – digestion procedure
10) Total and Inorganic Particulate Phosphorus
11) Organic Nutrients – TDN/TDP
12) Particulate Carbon and Nitrogen

SEDIMENT

1) Grain Size (Gravel, Sand, Silt, Clay wet pipette method)
2) Percent Water/moisture ASTM D 2216-98
3) Determining the amount of Material Smaller than 75 um ASTM D 1140-98
4) Wet Preparation of Soil Samples for Particle Size Analysis and Soil Constants ASTM D 2217-85
5) Dry Preparation of Soil samples for Particle Size Analysis and Soil Constants ASTM D 421-85
6) Standard Method for Particle Size Analysis of Soils ASTM D 422-63
7) Organic Matter/Volatile Solids
8) Carbon and Nitrogen in Sediment

INSTRUMENTS

1) Skalar Autoanalyzer
2) Exeter CHN Analyzer
3) Turner Designs Fluorometer
4) Sieve Shaker

CALIBRATIONS

1) Drying Oven — QM/In-house G-1
2) Thermometers — QM/In-house G-2

13.2.3 PART II – Example of Training Checklist

**Individual Procedure Checklist for Analyst Qualification Demonstration of Capability**

PROCEDURE: ASTM D 1140-97

ANALYST: _____

TRAINER: _____

DATE: _____

| | |
|---|---|
| | ➢ Read and understand SOP and reference material for method ASTM D 1140-97 |
| | ➢ Read and understand SOP and reference material for water content in soil method ASTM D 2216-98 |
| | ➢ Pass training requirements for ASTM D 2216-98 (water content in soil) |
| | ➢ Demonstrate use of balance, including internal calibration and tare function |
| | ➢ Prepare deflocculating reagent |
| | ➢ Visual inspection of No. 200 and No. 40 sieves |
| | ➢ Record oven temperature (100 +/- 5 $^0$ C) |
| | ➢ Correctly record tare weights and final weights on bench sheet |
| | ➢ Access computer calculation program and determine results |
| | ➢ Transfer results to appropriate excel folder on the server (Z:) |
| | ➢ Blind audit or proficiency test performed and passed |

Approval: _____ Date: _____
Trainer

Approval: _____ Date: _____
Laboratory Manager

## 13.2.4 PART III- Training Forms

**Analyst Training and Evaluation**

Analyst: _____

| Test Method | Check One | | Evaluated By | Date | Comments/Results |
|---|---|---|---|---|---|
| | Initial Training | 6 Month Eval. | | | |
| | | | | | |
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13.3 Method for Reviewing Test Technician's Competency at the ASC:

The laboratory manager is responsible for evaluating their analyst's competency six months after initial demonstration of capability by requiring each analyst to demonstrate the procedure for which he/she has been trained to perform. Copies of the results and evaluation records will be maintained in the personnel file located in the manager's office (first drawer in the file cabinet).

13.3.1 For each test, the manager will record the test demonstrated, the date of the demonstration and the results of the evaluation. In addition, the manager will sign each entry on the evaluation record.

13.3.2 If an unsatisfactory result is recorded for a specific test, the manager will review all observed deviations from the standard operating protocol with the analyst, observe the analyst re-demonstrate the test procedures and record the results as indicated above.

## XIV. Facilities and Environmental Conditions

The VIMS ASC is located in labs 115 and 116 and in the Seawater Research Laboratory (SRL) building. The SRL building has a swipe card as needed entry system and the doors remained lock except to those who have pre-authorization for entry. Testing occurs only within the laboratory space. The building is climate controlled and monitored 24 hours a day. Back-up generators are in place for refrigerators and freezers which need to have a maintained temperature and for instrumentation which requires around the clock electrical feed. DI water is supplied throughout the building which is circulated through reverse osmosis, followed by charcoal and four bed ion exchange resin tanks. At one tap in rm 116, a high ultra-pure system is available providing ASTM Type I grade water.

Refrigerators, freezers and ovens are maintained, monitored and documented on any day that laboratory activity is taking place. Several balances are available for use including analytical balances and a microbalance. All balances are kept clean and free of debris in a well ventilated area when necessary. Balances are calibrated on the day they are used. Annual calibrations are performed by an outside source for precision and accuracy. Safety equipment including eye washes, fire extinguishers and ventilation hoods are maintained and monitored according to procedures established by VIMS safety office.

The laboratory is kept clean. Only cleaners which are ammonia and phosphate free are allowed in the laboratory work space. Attention is given to good housekeeping at all times. The laboratory is designed, and activities are conducted, so sample contamination is avoided. The laboratory has adequate lights and ventilation.

## XV. Test Methods and Validation

The laboratory maintains an in-house method, also referred to as the standard operating procedure (SOP) for each certified analyte or test method. Methods will consist of a standard format which would allow for duplication of the procedure in a step-by-step fashion to the commonly accepted methodology and to complete the task according to outlined acceptable standards. SOPs may contain copies of published or referenced materials pertinent to the procedure. SOPs should be documented in a clear, concise format to allow for duplication of results by qualified analysts.

15.1 The SOP format should include the following:

15.1.1 Title, ASC Method number and Document Control number
15.1.2 Scope and Application
15.1.3 Summary of Method
15.1.4 Method Detection Limit (MDL)
15.1.5 Definition and Abbreviations
15.1.6 Interferences
15.1.7 Safety
15.1.8 Apparatus and Equipment
15.1.9 Reagents and Standards
15.1.10 Sample Collection, Preservation, Shipping and Storage
15.1.11 Quality Control
15.1.12 Instrument Setup, Calibration and Standardization
15.1.13 Procedure
15.1.14 Calculations
15.1.15 Data Assessment and Corrective Action
15.1.16 Method Performance
15.1.17 Pollution Prevention
15.1.18 Waste Management
15.1.19 References

15.2 Procedural changes to Standard Operating Procedures are documented on the Laboratory Action Form (Fig. 15.1) and requires acceptance by the laboratory manager prior to implementation.

15.3 A list of the analytical test parameters, method references and target MDL are listed in Table 15.1.

Figure 15.1

Document Control No. 00004 Laboratory Action Form
LAF #_____

| | | | |
|---|---|---|---|
| DATE SUBMITTED | | DATE APPROVED | |
| REQUESTOR NAME | | APPROVED BY: | |
| TYPE OF PROCEDURE / METHOD | SAMPLING [ ]<br>QA MANUAL REVISION [ ] | ANALYTICAL [ ]<br>SOP REVISION [ ] | REPORTING [ ]<br>OTHER [ ] |
| DURATION | PERMANENT [ ]<br>TEMPORARY [ ] | EFFECTIVE DATE:<br>START DATE:<br>END DATE: | |
| PROCEDURE/METHOD DESCRIPTION | | | |
| MODIFICATION DESCRIPTION | | | |
| JUSTIFICATION FOR MODIFICATION | | | |
| Notes: | | | |

## TABLE 15.1a ANALYTICAL METHODS – VELAP Certified

| ANALYTICAL TEST | ANALYTICAL METHOD | REFERENCE METHOD | LAB PRECISION * | ACCURACY * | TARGET MDL * |
|---|---|---|---|---|---|
| Ammonia | Colorimetric - autoanalyzer | SM 4500-$NH_3$- H (2011) | ≤ 20% RPD | 80-120% spike recovery | 0.005 mg/L |
| Nitrate + Nitrite | Colorimetric - autoanalyzer | SM 4500-$NO^{3-}$ F (2011) | ≤ 20% RPD | 80-120% spike recovery | 0.005 mg/L |
| Nitrite | Colorimetric - autoanalyzer | SM 4500-$NO^{3-}$ F (2011) | ≤ 20% RPD | 80-120% spike recovery | 0.002 mg/L |
| Ortho-phosphate | Colorimetric - autoanalyzer | SM 4500-P F (2011) | ≤ 20% RPD | 80-120% spike recovery | 0.004 mg/L |
| Silica | Colorimetric - autoanalyzer | SM 4500-$SiO_2$ F (2011) | ≤ 20% RPD | 80-120% spike recovery | 0.10 mg/L |
| Total Dissolved Nitrogen | Persulfate Digestion Colorimetric - autoanalyzer | SM 4500-N C (2011) | ≤ 20% RPD | 80-120% spike recovery | 0.03 mg/L |
| Total Dissolved Phosphorus | Persulfate Digestion Colorimetric - autoanalyzer | SM 4500-P B 5(2011) | ≤ 20% RPD | 80-120% spike recovery | 0.006 mg/L |
| Chlorophyll a | Fluorometric | EPA 445.0 Rev 1.2 | ≤ 30% RPD | N/A | 1.0 ug/L ** |
| Pheophytin | Fluorometric | EPA 445.0 Rev 1.2 | ≤ 30% RPD | N/A | 1.0 ug/L ** |
| Total Suspended Solids – Residue Non-filterable | Gravimetric | SM 2540 D (2011) | ≤ 30% RPD | N/A | 3.0 mg/L ** |
| Volatile Suspended Solids | Gravimetric | SM 2540 D (2011) | ≤ 30% RPD | N/A | 3.0 ** |
| Enterococcus | MPN | SM 9230 B 22nd ed (2007)/Enterolert | N/A | N/A | 10 MPN |

## TABLE 15.1b ANALYTICAL METHODS –NON - VELAP Certified

| ANALYTICAL TEST | ANALYTICAL METHOD | REFERENCE METHOD | LAB PRECISION * | ACCURACY * | TARGET MDL * |
|---|---|---|---|---|---|
| Particulate Phosphorus | Acid Digestion Colorimetric - autoanalyzer | SM 4500-P F (1997) | ≤ 30% RPD | 80-120% spike recovery | 0.003 mg/L ** |
| Particulate Carbon | High temperature combustion | Exeter Analytical Manual | ≤ 30% RPD | 80-120% SRM recovery | 0.30 mg/L ** |
| Particulate Nitrogen | High temperature combustion | Exeter Analytical Manual | ≤ 30% RPD | 80-120% SRM recovery | 0.020 mg/L ** |
| Grain size | Wet pipette method | | N/A | N/A | 1.0 % |
| Total Organic Carbon (Solids) | High temperature combustion | Exeter Analytical Manual | ≤ 30% RPD | 80-120% SRM recovery | 0.40 % |
| Total Nitrogen (Solids) | High temperature combustion | Exeter Analytical Manual | ≤ 30% RPD | 80-120% SRM recovery | 0.03% |
| Percent Moisture | Gravimetric | ASTM D 2216-98 | ≤ 30% RPD | N/A | 0.1 % |
| Organic Matter | High temperature combustion Gravimetric | ASTM D 2974 | ≤ 30% RPD | N/A | 0.1 % |

* From "Recommended Guidelines for the Sampling and Analyses in the Chesapeake Bay Monitoring Program" Revision in progress of EPA903-R-96-006 (January, 2017)
** Based on volume filtered

## 15.4 Conducting Demonstration of Method Performance

15.4.1 Prior to implementation of a method, the laboratory prepares an initial demonstration of method performance in accordance with the method reference. This may include, but not limited to, calibration acceptance, SRM acceptance, blind audit or Proficiency Testing, split testing, repetition of analytical runs to a minimum of two acceptable runs. The MDL must be determined in accordance with acceptable methodology for the specific analyte. An evaluation of precision and accuracy must be performed, if applicable.

15.4.2 Initial demonstration of method performance must be repeated each time significant changes are made in instrumentation, personnel, or the method. For personnel, initial demonstration is documented in Demonstration of Capability records (Section XIII of the Quality Manual).

## 15.5 Analytical Quality Control Procedures

### 15.5.1 Standard Calibration Curves

15.5.1.1 Analyses requiring calibration curves shall have an initial calibration curve that includes a minimum of three points and a blank for linear analysis (i.e. spectrophotometric, fluorometric) and a blank. Non-linear curves may require more than one curve. A low standard at the PQL is required for many analytes. This requirement will be stated in the SOP.

15.5.1.2 A correlation coefficient of no less than 0.995 will be acceptable.

15.5.1.3 A point on the calibration curve will be run at the end of the batch to verify that the calibration curve has not deviated more than 10% of the initial value.

15.5.1.4 All analytical data reported will fall between two points on the calibration curve unless the accuracy of the results will be degraded due to dilution specifically in the instances of saline or colored matrices. Permission to report data outside the calibration curve must be received by the manager prior to reporting the data and will be flagged accordingly on the final report.

15.5.1.5 Many analytes require the back calculation of the calibration curve. If this is required, it is stated in the SOP and the back calculation will be determined for each calibration standard. The back calculated and true value will agree within 10%, unless

different criteria are specified in the individual method. At the lower limit of the operational range, acceptance criteria may be wider.

15.5.2 Reference Materials:

15.5.2.1 Standard reference materials (SRMs) shall be analyzed at the frequency defined within each method. Normally an SRM is run with every analytical run for nutrients. AN SRM is also run with every sediment total organic carbon run. Other SRMs are run periodically if quantity or availability is limited.

15.5.2.2 Synthetic Seawater is prepared according to the method listed in A Manual of Chemical and Biological Methods for Seawater Analysis, Parson, Timothy R; Yoshiaki Maita, and Carol Lalli.

15.5.3 Matrix Spike:

15.5.3.1 In order to evaluate the effects of the sample matrix on the analytical methods, a known quantity of analyte is spiked on an aliquot of sample. This should be done on 10 - 20% of the samples analyzed (if appropriate for the method).

15.5.3.2 The analyte concentration should be high enough to be seen over the original concentration of the sample and should not be less than four times the calculated MDL.

15.5.3.3 Matrix spikes shall not be performed on laboratory or field blanks or field QC.

15.5.3.4 The percent recovery of analyte from the matrix spike sample is calculated using the following equation:

$$\% \text{ Recovery} = \frac{(SSR)}{(SR+SA)} \times 100$$

where,

SSR = Spike sample result
SR = Sample result
SA = Spike added

15.5.3.5 If the recovery of the analyte falls outside the range designated in Table 15.1 in the Quality Manual, repeat analysis of sample and spike after checking for obvious sources of error. If the

recovery of the replicated spike of the same sample again falls outside the designated range, the recovery problem encountered with the spiked sample is judged to be matrix related, not system related and no further corrective action is required.

15.5.4 Laboratory Duplicates:

15.5.4.1 Laboratory duplicate analyses provide a measure of laboratory precision. Duplicates are prepared by taking two aliquots for analysis from a well homogenized sample. More replicates can be analyzed and reported.

15.5.4.2 A laboratory duplicate must be analyzed once for every 10 - 20 samples.

15.5.4.3 The precision is measured by calculating the difference between the two sample results. Control charts are based on 20 duplicates.

Upper control limit (UCL) = 3.27 x M
Upper warning limit (UWL) = 2.51 x M

Where M = mean of differences

15.5.4.4 If the precision does not fall within the control limits listed in Table 15.1 of the Quality Manual for any calculated data point, the sample should be reanalyzed and proper corrective action should be taken if not resolved. This could be a simple notation if the duplicates are at or near the Practical Quantitative Limit (PQL) or the lowest standard or could require recalibration and reanalysis of the samples.

15.5.5 Continuing Calibration Verification (CCV):

15.5.5.1 A calibration standard, check standard or LCS will be analyzed at an interval of every 10-20 samples during each analytical run and at the end of the analyses. The purpose of the CCV is to ensure that the calibration of the instrument remains valid.

15.5.5.2 The CCV standard must be within 90-110% of the known analyte concentration.

15.5.6 Method Blank:

15.5.6.1 A method blank shall be analyzed at the beginning and end of each analytical run and at an interval of 10-20 samples. The method blank is carried through all steps of sample preparation and analysis, along with samples.

15.5.6.2 Results of the method blank are used to check for possible contamination of samples in the preparation and analysis of samples.

15.5.6.3 If the method blank results is higher than the lowest standard, analyze another aliquot of the reagent blank. If it remains above the lowest standard then corrective action should be taken.

15.5.6.4 Corrective action to be taken might entail reanalysis of the analytical run and preparation of the samples. If unable to run samples, the data may be reported at the discretion of the laboratory manager with a data qualifier or the data may be deemed unreportable.

15.5.7 Minimum Detection Limit (MDL) /Limit of Detection (LOD) or Limit of Quantitation (LOQ):

15.5.7.1 LODs will be determined by the protocol in the mandated test method or applicable regulation. For chemistry testing, where applicable, procedure EPA 821-R-16-006, December 2016 Revision 2 will be used and verified on an annual basis.

15.5.7.2 MDLs for each analyte not applicable to the above LOD determination, are determined using the "Federal Register" (Appendix B ) 131:4375. This evaluation is made yearly from a sample obtained from the Chesapeake Bay at low concentrations, or alternatively, a standard made up in synthetic seawater may be acceptable. The standard should be made at a concentration of 1-5 times the expected MDL.

15.5.7.2.1 The sample is filtered in-house as required into separate pre-cleaned containers for specific parameters.

15.5.7.2.2 Filter particulates onto appropriate filters.

15.5.7.2.3 Analyze all replicates. The first 7 replicates of each will be used for the MDL determination.

15.5.7.2.4 To determine the MDL for an analyte, first calculate the standard deviation from the seven results. The

standard deviation is then multiplied by the "student t" value (3.143 or rounded to 3 for seven replicates ).

15.5.7.7 The concentration of the analyte used for the determination or verification of the LOD/MDL should be 5-10 times the expected MDL. Higher concentrations may yield unusuable determinations.

15.5.8 Practical Quantification Limit/Method Reporting Limit:

15.5.8.1 The practical quantitation limit (PQL/MRL) is approximately 3.18 (MDL).

15.5.8.2 The lowest calibration standard concentration must be at or below the PQL/MRL.

15.5.8.3 Some methods require a MRL QC sample spiked at 1-2 times the MRL to be run initially and at least quarterly. The MRL QC sample will be subjected to all sample preparation steps. The MRL QC sample is used for verification of the MRL and must recover +/- 20% of the value. Methods with this requirement must state the requirement within the SOP.

15.5.9 QA/QC Management: Determination of Accuracy and Precision

15.5.9.1 Accuracy is defined as the degree of agreement between the measured and the true value of standard reference materials (srm) and may be determined based on certified srm data sheets. SRMs are available for water and sediment samples. An SRM should be analyzed with every run unless an exception is outlined in the Standard Operating Procedure.

15.5.9.2 Spiking a known amount of analyte into the sample itself will also allow us to measure accuracy. A small amount of high concentration standard is introduced into a sample aliquot. The spike is then carried through the entire procedure and % recovery is determined. Low spike recovery may indicate a methods problem or error or may also indicate matrix problems.

$$\% \text{ Recovery} = \frac{SR}{STV} \times 100$$

SR = Spike recovered
STV = Spike true value (sample + addition)

15.5.9.3 Ten percent (10%) of sample analyzed should be spiked for a accuracy on samples which can be spiked.

15.5.9.4 Analytical precision is the degree to which an analytical result can be reproduced. This can be determined by running 10 -20 % of samples in duplicate. Sediment samples are to be run in duplicate for 10% of the samples since we may not have the ability to spike a sediment.

15.5.9.5 Reproducibility (precision) can be calculated duplicates:

$$RPD = \frac{/R/}{Y} \quad x100$$

RPD = Relative percent difference
/R/ = Absolute difference of the two values
Y = Average of the replicate

15.5.10 Control Charting

15.5.10.1 Control limits are determined statistically. The standard deviation is calculated for $\geq 30$ points or as the data package allows.

15.5.10.2 Acceptable limits are equal to the calculated mean of those points $\pm 2$ standard deviations.

15.5.10.3 Warning limits are equal to the calculated mean $\pm 3$ standard deviations.

15.5.10.4 A process is out of control if 3 or more data points are outside the warning limit.

15.5.10.5 A warning of possible systematic error is indicated if 7 successive data points fall away from the mean on the same side of the center line, if 7 or more data points fall outside of either warning limit, or if a discernible trend develops.

15.6 Microbiological Quality Control Procedures

15.6.1 Sterility Checks – Sterility checks on sample containers shall be performed on at least one container for each lot purchased using a nonselective growth media such as tryptic soy broth. A sterility blank shall be performed on each batch of dilution water prepared in the laboratory or on each lot of pre-prepared, ready-to-use dilution water purchased with nonselective growth media such as tryptic soy broth. A log will be kept demonstrating these checks.

15.6.2 Volume checks – Non-class A glassware, disposal pipettes and any marked container shall be calibrated once per lot prior to use. This is to

include disposable pipettes and purchased pre-prepared sodium thiosulfate bottles. A log will be kept demonstrating these checks.

15.6.3 Incubator temperatures – Temperatures of incubators shall be documented twice daily, at least four hours apart, on each day of use.

15.6.4 Dilution water blanks - A dilution water blank when purchased from IDEXX will be analyzed with each run.

15.6.5 QC checks - A purchased QC will be run with each new Enterolert lot purchased from IDEXX for Enterocoocus.

# XVI. Equipment, Reagents, Supplies and Reference Materials

All equipment, reagents, supplies and reference materials necessary for analyses are kept on hand for the specific analysis for which the Analytical Service Center performs. For calibrations of analytical instrumentation, the laboratory uses standards that assure the measurements made are traceable to national standards of measurement, such as NIST traceable standards (when available) or certified reference materials. Calibration procedures are established for all applicable tests. These procedures are detailed in the SOP for the analysis.

## 16.1 Available Laboratory Equipment and Instrumentation

The ASC maintains up-to-date laboratory technology and instrumental capabilities. The ASC is adequately stocked with gravity ovens, combustion ovens, autoclave, centrifuge, top loading balances, analytical balances, Millipore ultra-pure filtration units, wide range of sieves, hoods, stirrers, hot plates, refrigerators, freezers, glassware and disposable items.

All equipment is properly maintained. Procedures for maintenance of equipment is documented in the SOP or in the Quality Manual. Any defective equipment is removed from service and labeled until repaired or disposed of. Equipment is not put back into service until the laboratory demonstrates that it is functioning correctly. Calibration records are maintained for all measuring equipment (see laboratory bench sheets and logs).

Laboratory support equipment is calibrated, or verified, or both, before being put into service and on a continuing basis. The procedure for calibration and verification of equipment are found in the Quality Manual (G-1 and G-2).

The following is a list of major instrumentation owned by the ASC:

### 16.1.1 Exeter Analytical CE-440 Carbon/Nitrogen (CHN) Analyzer

High temperature combustion at 980° C, silver/tungsten catalyzed
Detector = Independent sequential conductivity bridges.
Autosampler = Integral, sequential.
Dedicated computer control and data acquisition.
Analytes: PC, TC, TOC, PN, TN.

### 16.1.2 Skalar SANplus Continuous Flow Analyzer SA1050 Autosampler

SA503-365.1 -- OP, TDP, PP, PIP, TP, TIP
SA467-353.2 -- NO2
SA461-353.2 -- NO2+3. TDN, NO2

SA156-350.1 -- NH3
Detectors = SA6270/6255 Matrix photometers.
Flow Cell = SA6275(50 mm) with SA6256 detection head/flow cell.
Autosampler = SA1050 random access.
FLOW ACCESS data reduction package

16.1.3 Turner Designs Trilogy Laboratory Benchtop Fluorometer

Capable of discrete sample measurement of various fluorescent materials including chlorophyll a, chlorophyll pigments and by-products (phaeophytin), turbidity and various other analytes.

16.1.4 Latchat Quickchem 8500 Series 2 Flow Injection Autoanalyzer

ASX-500 Series XYZ Autosampler
RP-100 Reagent Pump
Omnion 3.0 Software
Datasystem with software

16.1.5 Thermo Spectronic UV1- Double Beam UV/VIS Scanning Spectrophotometer

Measurement range 190 - 950 nm, slit width = 2 nm.
Variable pathlength

16.1.6 Retsch AS 200 SIEVE SHAKER Serial # 124210603K

16.1.7 Sartorius MICRO BALANCE – Model # MSE6-6500

16.1.8 Mettler AE 110 TOP LOADING BALANCE – Serial # L44926

16.1.9 Idexx Quantitray Sealer

16.1.10 Access to RSA ANALYZER - Computer controlled, top-loading balance

16.1.11 Fisher Scientific Isotemp 750F OVEN . Serial # 812N0279

16.1.12 Lindberg/Blue GRAVITY OVEN. Serial # 0250-501567-PO

16.1.13 IEC Model HN-SII Centrifuge

16.1.14 Brinkman 2540E Autoclave

16.2 Instrument Maintenance

The purpose of this procedure is to document the recommended maintenance schedules for the main instruments operated in the Analytical Service Center when they are in use.

16.2.1 Skalar San Sun Plus II/Latchat Quickchem 8500 Continuous Flow Analyzer- Routine

16.2.1.1 Inspect Fittings for tightness and closure

16.2.1.2 Inspect tubing for cleanliness and integrity

16.2.1.3 Inspect mechanical parts for proper fit and operation

16.2.1.4 Clean system following each analysis by:

Pump DDI water through all lines for 10 min.
Pump 1% Hypochlorite solution through reagent lines for at least 15 minutes.
Pump DDI water for at least 15 mins. to rinse.

16.2.1.5 Rinse autosampler racks with tap water.

16.2.2 Skalar San Sun Plus II/Latchat Quickchem 8500 Continuous Flow Analyzer- Monthly

16.2.2.1 Move pump tubing to second bridge position, if possible

16.2.2.2 Grease Platens

16.2.2.3 Verify Reactor temperatures

16.2.3 Skalar San Sun Plus II/Latchat Quickchem 8500 Continuous Flow Analyzer- Quarterly

16.2.3.1 Replace Pump tubing, including autosampler pump

16.2.3.2 Archive files in Z: directory

16.2.3.3 Check air manifold for moisture build-up, blow out if necessary.

16.2.4 Skalar San Sun Plus II/Latchat Quickchem 8500 Continuous Flow Analyzer- Annually

16.2.4.1 Replace all transfer lines, reagent lines

16.2.4.2 Replace all AIR tubing

16.2.5 Spectrophotometers/Fluorometers- Routine

16.2.5.1 Inspect for cleanliness, remove dust and debris.

16.2.6 Spectrophotometers/Fluorometers- Monthly

16.2.6.1 Check calibration using a known calibration standard.

16.2.7 Analytical Balance: A Service Contract with Precision Weighing, Inc. covers yearly calibration of all usable balances. In between yearly calibrations, there are NIST traceable metric weights for in house checking on a daily basis. Daily calibration using two weights bracketing the usage range of the balance is documented.

16.2.8 Exeter Analytical CE-440 Carbon/Nitrogen Analyzer – Routine

16.2.8.1 Verify temperature settings

16.2.8.2 Verify bridge voltages and adjust C and N to read $\leq$ 2500 uV

16.2.8.3 Run a series of blanks to stabilize C and N blank values

16.2.8.4 Run a conditioner (calibration standard)

16.2.8.5 Clean waste receptacle

16.2.8.6 Check injector drive

16.2.8.7 If the instrument has been turned off, upon startup run calibration for K determinations

16.2.8.8 Check oxygen and helium levels

16.2.9 Exeter Analytical CE-440 Carbon/Nitrogen Analyzer – Monthly

16.2.9.1 Check tightness of fittings with o-rings

16.2.10 Exeter Analytical CE-440 Carbon/Nitrogen Analyzer – Quarterly or as reduction tube or Helium tank is changed

16.3.9.1 Change o-rings, if brittle

16.3.9.2 Change water trap and carbon dioxide trap and helium trap

16.3.9.3 Check count on combustion tube. Change combustion tube @ or close to 2500 drops

16.3.9.4 Check oxygen tank

16.2.11 Exeter Analytical CE-440 Carbon/Nitrogen Analyzer – Yearly

16.2.11.1 Check valve operation and change internal parts if necessary

16.2.12 Ovens

16.2.11.1 Temperatures are recorded on a daily basis when the oven is in use.

16.2.11.2 Calibrate yearly using in-house procedure G-1

16.2.13 Refrigerators and Freezers

16.2.13.1 Temperatures are recorded daily when in use

16.2.14 Thermometers

16.2.14.1 Calibrate or replace thermometers yearly. Only calibrated thermometers will be placed into service. Those thermometers with an expired calibration will be removed from service, recalibrated or replaced with thermometers which have been recently calibrated. A log of thermometers will be kept at Z:NAL\ascdocs\internl\logs\thermometer logs

16.2.14.2 Calibrate or replace NIST thermometer every five years

16.2.15 Autoclave

16.2.15.1 Record temperature, pressure and cycle time daily when in use

16.2.16 Muffle furnace

16.2.16.1 The muffle furnace thermocouple will be calibrated yearly using a calibrated thermocouple. A log of muffle furnace calibrations will be kept on Z: NAL\ascdocs\internal\logs

16.2.17 Mechanical Pipettors

16.2.17.1 Mechanical pipettors are calibrated annually by an external calibration service.

16.2.17.2 Mechanical pipettors will be calibrated quarterly internally. Accuracy must be within +/- 2.5%.

TABLE 16.1 Maintenance Schedule- Continuous Flow Analyzers

Skalar SanSun Plus II/Latchat Quickchem 8500

| | Daily | Bi-Weekly | Monthly | Quarterly | Yearly |
|---|---|---|---|---|---|
| 1. Inspect fittings | X | | | | |
| 2. Inspect tubing/replace tubing | X | | | | |
| 3. Inspect mechanical parts | X | | | | |
| 4. Check Calibration | X | | | | |
| 5. Clean with appropriate reagent/DDI | X | | | | |
| 6. Move pump tubing to second bridge | | X | | | |
| 7. Verify reactor temperatures | | X | | | |
| 8. Clean Platen with water and lint-free cloth/grease | | X | | | |
| 9. Flush debubbler/replace tubing | | | X | | |
| 10. Replace pump tubing, including autosampler tubing | | | X | | |
| 11. Archive files | | | X | | |
| 12. Check air manifold | | | X | | |
| 13. Inspect source lamp/replace if needed | | | | X | |
| 14. Clean optical filters with optical paper | | | | X | |
| 15. Replace internal tubing in flow cell | | | | | X |
| 16. Replace all transfer lines, reagent lines | | | | | X |
| 17. Replace all air tubing | | | | | X |

TABLE 16.2 Maintenance Schedule- Spectrophotometer/Fluorometer

| | Daily | Weekly | Monthly | Quarterly | Yearly |
|---|---|---|---|---|---|
| 1. Inspect for cleanliness | X | | | | |
| 2. Inspect mechanical parts | X | | | | |
| 3. Run calibration | X | | | | |
| 4. Rinse cells after use | X | | | | |
| 5. Keep covered when not in use | X | | | | |
| 6. Calibrate with external standard | | | X | | |

TABLE 16.3 Maintenance Schedule – Balances

| | Daily | Weekly | Monthly | Quarterly | Yearly |
|---|---|---|---|---|---|
| Clean after every use | X | | | | |
| Cover / close after every use | X | | | | |
| External Calibration | | | | | X |
| | | | | | |

TABLE 16.4 Maintenance Schedule – Exeter Analytical CE-440 CHN Analyzer

| | Daily | Weekly | Monthly | Quarterly | Yearly |
|---|---|---|---|---|---|
| Verify temperature settings | X | | | | |
| Verify bridge voltages | X | | | | |
| Run series of blanks | X | | | | |
| Run conditioner | X | | | | |
| Clean waste receptacle | X | | | | |
| Check injector drive | X | | | | |
| Determine K values, if necessary | X | | | | |
| Check oxygen and helium levels | X | | | | |
| Check tightness of fittings | | | X | | |
| Change o-rings if necessary | | | | X | |
| Check water trap | | | | X | |
| Check helium trap | | | | X | |
| Check carbon dioxide trap | | | | X | |
| Check combustion tube count | | | | X | |
| Check oxygen tank | | | | X | |
| Check valves - operation | | | | | X |

TABLE 16.5 Maintenance Schedule – Ovens

| | Daily | Weekly | Monthly | 4 months | Yearly |
|---|---|---|---|---|---|
| Record temperature reading | X | | | | |
| Calibration/Verification per procedure G-1 | | | | | X |

TABLE 16.5 Maintenance Schedule – Refrigerators/Freezers

| | Daily | Weekly | Monthly | 4 months | Yearly |
|---|---|---|---|---|---|
| Record Temperature readings | X | | | | |

TABLE 16.6 Maintenance Schedule – Thermometers

| | Daily | Weekly | Monthly | 4 months | Yearly | 5 Years |
|---|---|---|---|---|---|---|
| Calibrate or replace calibrated thermometers | | | | | X | |
| Replace NIST thermometer or calibrated | | | | | | X |
| Muffle Furnace Thermocouple | | | | | X | |

TABLE 16.7 Maintenance Schedule – Autoclave

| | Daily | Weekly | Monthly | 4 months | Yearly |
|---|---|---|---|---|---|
| Record Temperature readings | X | | | | |

TABLE 16.8 Maintenance Schedule – Mechanical Pipettor

| | Daily | Weekly | Monthly | Quarterly | Yearly |
|---|---|---|---|---|---|
| Record Temperature readings | | | | X | X |

16.3 Instrument Calibration and verification

16.3.1 General Policies:

16.3.1.1 Required equipment shall be calibrated at specified intervals following the general procedures indicated below.

16.3.1.2 Newly acquired equipment without manufacturer's certification and equipment that has not been calibrated or verified because it has been removed from service shall be calibrated or verified before being placed in service.

16.3.1.3 When any equipment is overloaded, mishandled, giving results that are suspect, or is not meeting specification tolerances, the manager shall remove it from service and clearly mark it by a label. The equipment shall be returned to service only after appropriate repairs are made and calibration and verification shows the equipment to function satisfactorily or to meet specification tolerances.

16.3.2 General Procedures:

16.3.2.1 The manager shall maintain a file for each piece of

equipment requiring calibration or verification. The file for each piece of equipment shall contain detailed records of calibration or verification work performed in chronological order and shall be kept in the manager's office.

16.3.3 Calibration and verification: equipment and calibration verification schedule

(Date)
EQUIPMENT CALIBRATION AND VERIFICATION INFORMATION

| Item(s) | Calib/Verif Interval | Location of Records | Calib/Verif Procedure Ref. |
|---|---|---|---|
| Balances | 12 Mo. | WQ Bldg. Rm 105 | ASTM D4753 |
| Balances | Daily | RM 116 - SRL | |
| Ovens | 12 Mo. | WQ Bldg Rm 105 | In-House Proc. G-1 |
| Pipettors | 12 Mo. | WQ Bldg. Rm 105 | External / Internal Calibration Service |
| Thermometers | 12 Mo. | WQ Bldg Rm 05 | In-House Proc. G-2 |

16.3.4 Verification Record- Example (Ovens)

(Date)

EQUIPMENT VERIFICATION RECORD

Inspected By:_____ Date:_____

Apparatus: Lindberg-Blue Gravity Oven Verification Procedure # ___G-1
Serial # 0250-501567- PO

Verification Frequency : 12 months Last verification Date:_____

Next Due Date:_____

Verification equipment used: _____
_____

1) Temperature setting : _____

Result for Position #1_____ within acceptance?_____
(after 1 hour)
Result for Position #2_____ within acceptance?_____
(after additional 2 hours)
Result for Position #3_____ within acceptance?_____
(after additional 2 hours)
Result for Position #4_____ within acceptance?_____
(after additional 2 hours)

2) Temperature setting : _____ (if required)

Result for Position #1_____ within acceptance?_____
(after 1 hour)
Result for Position #2_____ within acceptance?_____
(after additional 2 hours)
Result for Position #3_____ within acceptance?_____
(after additional 2 hours)
Result for Position #4_____ within acceptance?_____
(after additional 2 hours)

## 16.3.5 Calibration/Verification Procedure: G-1

Equipment Checked: DRYING OVEN

Purpose:
This method provides instructions for verifying the dial settings on general-purpose drying ovens.

Inspection Equipment Required:

1. A calibrated thermometer graduated in 1.0°C increments having a range which includes the temperature range to be checked.
2. A brass thermometer well or sand well to retain heat while the oven door is open.This is essential for a constant temperature reading.
3. A clothes pin or holder to secure the thermometer in such a manner as to enable the operator to read the scale easily from outside or inside the oven.

Tolerance:
Drying ovens shall be capable of maintaining a constant temperature range listed in the appropriate test methods.

Procedure:

1. Place the thermometer inside the brass well with the clothes pin attached to the thermometer. Position the thermometer on the shelf where the samples are normally dried.
2. Take the first reading at least 1 hour after closing the oven (oven should remain undisturbed).
3. Take as many readings as necessary to determine if the temperature range is within the specified tolerance (three consecutive readings, taken no less than 2 hr. apart, within the tolerance allowed are adequate.)
4. Adjust the temperature of the oven if an observed temperature reading is outside the tolerance specified (allow at least 2 hr. for the temperature to stabilize between each adjustment). Return to step 3.

## 16.3.5 Verification Record- Example (Thermometers)

(Date)

EQUIPMENT VERIFICATION RECORD

Inspected By:_____ Date:_____

Apparatus: Thermometer
Serial #

Verification Procedure # ___G-2

Verification Frequency : 12 months

Last verification Date:_____

Next Due Date:_____

Verification equipment used: _____
_____

1) Temperature setting : _____

| NIST Thermometer # | Temperature Recorder | Lab Thermometer # | Temperature recorded | Correction |
|---|---|---|---|---|
| | | | | |

## 16.3.6 Calibration/Verification Procedure: G-2

Equipment Checked: THERMOMETERS

Purpose:
This method provides instructions for verifying the thermometer readings and accuracy

Inspection Equipment Required:

1. NIST calibrated thermometer
2. Sand in flask or water bath

Tolerance:
Thermometers shall be capable of reading at 0.5 °C intervals. Correction factors will be determined for each calibrated thermometer.

Procedure:

1. Once a year, all of the thermometers are checked at operating temperatures against one of the NIST traceable thermometers.
2. To point-check the accuracy of Thermometers, place a flask of sand into the oven, or water into refrigerator at the temperature required for the method of interest.
3. Place the NIST traceable thermometer into the flask.
4. Conduct a comparison check of the thermometer to the NIST thermometer.
5. The thermometers must be suspended in the sand or water in the flask, not resting on the bottom of the flask.
6. Alternatively, a water bath or ice bath is used, where applicable, to check the temperature of the thermometers by simultaneous immersion of the NIST traceable thermometer and the thermometer to be calibrated into the water bath or ice bath at the temperature of interest.
7. Any difference in temperature readings between the NIST traceable thermometer and the laboratory thermometer is recorded on the Point-Check Calibration of Laboratory Thermometers Record Form. A label displaying the date of calibration and correction factor (even if it is zero), is placed around the top of the corresponding thermometer.

16.4 Volumetric Glassware:

Use these methods to insure that measurements are correct.

16.4.1 The meniscus bottom should be tangent to the calibration mark on volumetric flasks, pipettes, and graduated cylinders.

16.4.2 Glassware is calibrated at 20°C when purchased. If the temperature of the fluid being measured is quite cold or hot, it will affect your volumetric measurements. Wait until the temperature is approximately room temperature before adjusting measurement.

16.4.3 The letters TD and TC on volumetric glassware mean "to deliver" or "to contain" respectively.

16.4.4 When emptying a volumetric pipette, hold it in a vertical position and let the liquid flow unrestricted. Keep the tip of the pipette touching the wall of the flask for a couple of seconds after the flow has stopped. Do not blow out the remaining liquid in the pipette.

16.4.5 All glassware used in the laboratory is designated Class "A". These include pipettes, burettes, graduated cylinders and volumetric flasks shall be used unless otherwise specified in the procedure.

16.4.6 All glassware is properly cleaned as specified in the SOP.

16.5 Reagents and Supplies:

16.5.1 Traceability of chemicals, reagents and standards

16.5.1.1 Ordered chemicals are logged into the chemical inventory log upon receipt at Z:NAL\ascdocs\internal\logs\chemical inventory. The chemical is given a unique laboratory log number. The number is displayed on the bottle. The chemical is dated and initialed upon arrival and dated and initialed again when the container is first opened. The person receiving the chemical or supply should initial the P.O. , a copy of which is maintained in the laboratory.

16.5.1.2 When reagents are prepared, a log of the reagent, preparation date, expiration and chemical log number of each chemical used (generated when the chemical is received and entered into the chemical inventory) is documented. Each reagent is given a

unique reagent log number and documented at Z:NAL\ascdocs\internal\logs\reagent log\ "analysis".

16.5.1.3 When standards are prepared (stock, intermediate, and daily standards), documentation of the preparation date, expiration and chemical log number of each chemical used (generated when the chemical is received and entered into the chemical inventory) is documented. Each standard is given a unique standard log number and documented at Z:NAL\ascdocs\internal\logs\stock standards\ or Z:NAL\ascdocs\internal\logs\SKALAR\nutrient standard prep log.

16.5.2 Analytical reagent grade materials are used in the laboratory, if available.

16.5.3 All stock and standard solution containers in which the solutions are stored will have the content, preparation date, initials and concentration labeled on the container. When stock and standard solution and reagents are prepared, log the reagent in the Chemical Reagent Log noting Log # of each chemical used from the Chemical Log. A separate log for calibration stock and intermediate standards are maintained.

16.5.4 All containers in use will have name of contents on them.

16.5.5 When making a new stock solution, always dry the chemical at 105°C for 24 hours before weighing on the analytical balance.

16.5.6 When using water for dilution, always use fresh, deionized ASTM Type I water unless the specific procedure states otherwise.

16.5.6 Check warning labels on chemical containers and/or MSDS information. Use proper safety precautions when handling any chemical.

16.5.7 The purity and related quality of a reagent in a procedure has a direct and significant bearing on the integrity of the test. In the case that high quality reagent is needed, it will be noted in the Standard Operating Procedure. Expiration dates of chemicals must be noted to insure accurate analyses.

16.5.8 All solvents used in the laboratory are stored in flame proof cabinets which are exhausted outside.

16.5.9 Acids are stored in designated storage areas and marked as so.

16.5.10 Dry chemicals are stored in the designated storage area.

16.5.11 Inventories are prepared and submitted to the Safety Office on a yearly basis.

16.5.12 The laboratory does not use prepared reagents, standards, or purchased chemicals outside the expiration dates.

16.5.13 Rinse all empty acid containers with tap water and dispose of properly.

16.5.13 Consumables and other supplies are to be dated and initialed upon arrival and receipt into the laboratory and dated and initialed again when the container is first opened. The person receiving the supply should initial the P.O. which is maintained in the laboratory.

16.4 Reference Materials

To ensure accurate and precise measurements, the laboratory uses reference materials that are traceable to a national standard of measurement where commercially available, such as NIST, NELAC, or are traceable to certified reference materials. The laboratory retains the Certificates of Reference Materials to demonstrate traceability.

## XVII. Sample Handling

All samples accepted into the laboratory are given a unique identifying number and logged into the laboratory's LIMS system. This unique number identifies each sample from collection to disposal. Sample batches are labeled with a number beginning with the last two digits of the year followed by the batch number and finally the individual sample number (ex. 16-001-001) the number is reset within the LIMS system at the beginning of each calendar year

### 17. 1 Chain of custody

Each sample batch is accompanied by a chain of custody (COC) or is emailed by the client requesting analysis to the laboratory manager (Ex. Figure 17.1). The COC will contain, at a minimum, the following information:

1) Name of customer
2) Contact information for the customer
3) Date of sample collection
4) Analysis required
5) Account # to be charged (if applicable)
6) Login # s assigned to the batch
7) Date of receipt
8) Initials of receiver
9) Temperature of a sample at time of receipt if shipped
10) Comments on condition of the sample, if applicable

### 17.2 Sample Acceptance Policy

Upon receipt of the samples to the laboratory, the laboratory will verify the integrity of the sample by checking for the following items and notating them on the COC form.

1) Leakage or breakage.
2) Completeness of sample COC.
3) Correctness of sample identification and comparison to the COC provided to ensure samples received are those listed on the COC. A minimum of date of sampling, sample location identification and the name of the customer is required to identify each sample properly.
4) Use of appropriate sample containers and adequate volume, for example, sterile bottles for microbiological analysis, acid-washed, laboratory supplied bottles for nutrients. Chlorophylls should be packaged in foil covered containers to prevent exposure to fluorescent light.
5) Ensure that volumes filtered are documented for particulate samples.
6) Temperature check of a sample in the batch when samples are unfrozen when received, otherwise, frozen samples are notated as such on the COC.

FIGURE 17.1

VIMS - Analytical Service Center
Virginia Institute of Marine Science/College of William and Mary
Rt 1208 Greate Road Gloucester Point, VA 23062
Contact: Carol Pollard – Laboratory Manager tel. (804)684-7213 email: pollard@vims.edu

Document Control Number : 00002

**Chain Of Custody**

SOURCE/STUDYNAME:_____ DATE OF SAMPLING_____

COLLECTED BY:_____ ACCOUNT NUMBER:_____

PROJECT _____ Comments_____

| | SAMPLE NAME | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | ASC Filtered? | | | | | | | | | | | |
| Water - DISSOLVED | $NH_4$ | | | | | | | | | | | |
| | $NO_{2+3}$ | | | | | | | | | | | |
| | $NO_2$ | | | | | | | | | | | |
| | $PO_4$ | | | | | | | | | | | |
| | SI | | | | | | | | | | | |
| | TDN/TDP | | | | | | | | | | | |
| | CHLORO/PHEO | | | | | | | | | | | |
| | BISI | | | | | | | | | | | |
| Water - PARTICULATES | PP | | | | | | | | | | | |
| | PIP | | | | | | | | | | | |
| | PC/PN | | | | | | | | | | | |
| | TSS | | | | | | | | | | | |
| | FIX SOL | | | | | | | | | | | |
| | %Moist/water | | | | | | | | | | | |
| | Organic matter | | | | | | | | | | | |
| | Wet Pipette GS | | | | | | | | | | | |
| | RSA | | | | | | | | | | | |
| | TOC/TN | | | | | | | | | | | |

Received By:_____ Date:_____

Relinquished By:_____ Date:_____

FURTHER INFORMATION:

17.3 Sample Login

When sample batches are received and the sample batch has been accepted, the samples are individually logged into the ASPEN LIMS system. Upon the generation of a new batch number in the system, the person logging in the sample batch will be directed to identify the client, the analysis requested, the appropriate account number to charge, the date (and time) of sampling, if required, date of receipt, identification of the person accepting the sample, and the individual sample identification. Samples can then be added to the batch as necessary.

17.4 Sample Storage, Preservation and Holding Times

17.4.1 The laboratory will store samples, sub-samples, and/or preparation products, such as extracts or digestates, according to the specified conditions in the approved method. All samples, sub-samples, and/or preparation products will be stored properly to ensure there is no contact with potential sources of contamination, no propensity for deterioration or damage. Refrigerators and freezers for sample storage are maintained at 4°C (± 2.0 °C) and -20°C (± 2.0 °C), respectively and are checked on a daily basis for proper operation.

17.4.2 The method of preservation for filtered water (ex. dissolved nutrients) is freezing at -20°C (± 2.0 °C) and analyzed within 28 days. Dissolved silica sample should not be frozen but maintained in a refrigerator at 4°C (± 2.0 °C) until analyzed. Silica samples should be analyzed within 28 days. If silica sample is frozen, thaw the sample for 24 hours prior to analysis. For particulate filters (ex. PC/PN, TSS/TFS, PIP, PP) the filters should be put into labeled petri dishes and frozen at -20°C (± 2.0 °C) with analysis within 28 days. Chlorophyll a filters should be immediately wrapped in foil, labeled and frozen at -20°C (± 2.0 °C) with analysis within 28 days. Sediment samples for any analysis should be placed in the refrigerator and maintained at 4°C (± 2.0 °C) until analyzed.

17.5 Sample Disposal

Following analysis, the remaining water sample is stored in the refrigerator maintained at 4°C (± 2.0 °C). Soil samples are stored in a designated area in the laboratory upon completion of the analysis. After the final report is approved and the report is received by the client, the samples will stay in the refrigerator or designated storage area for 30 days. Samples after that time period will then be disposed of according to federal and state requirements. Any sample deemed hazardous will be labeled as such and submitted with proper paperwork to the Safety Office for disposal through a hazardous waste disposal company.

## XVIII. Assuring the Quality of Test

The laboratory demonstrates the quality of analytical results through the implementation of the quality control plan.

18.1 Data Audits

These are independent checks on the data performed by the Laboratory. The checks are done by having laboratory duplicate, matrix spikes, and Standard Reference Material. Data is maintained with the raw data and electronic files on the Z: drive.

18.2 Performance Audits

18.2.1 Outside performance samples are analyzed annually for evaluation of performance. In addition, the facilities will be visited by an outside Quality Assurance Officer as an on-site audit periodically.

18.2.2 Blind Audits are prepared by CBL in Maryland and distributed twice a year for inter-laboratory comparisons. Results for the last three years are on the Chesapeake Bay Program(CBP) website (http://www.epa.gov) and are readily available.

18.2.3 Split samples from the CBP are delivered quarterly for inter-laboratory comparisons. Results for the last three years are on the Chesapeake Bay Program(CBP) website (http://www.epa.gov) and are readily available.

18.2.4 Proficiency nutrient audit samples are supplied by the USGS-Office of Water Quality proficiency program at least yearly.

18.2.5 Nutrient Audit samples are supplied by the National Estuarine Research Reserve Program annually.

18.2.6 Proficiency samples are purchased bi-annually form a certified company. If at any time, an analyte is determined to be unacceptable, the lab will participate in two more consecutive studies in order to assure proficiency in the test. Unacceptable runs will be studied to determine possible causes for the unacceptable result. This may result in a corrective action before the next proficiency sample is ordered.

18.3 Corrective Action System

18.3.1 Throughout the Quality Manual and the Laboratory Procedure Manual, there are listed the steps to take when data is out of control or equipment and instruments break down. The system is dependent on having control limits set and having backup equipment and parts available. The laboratory requests an appropriate volume of sample should an analytical run result in unacceptable quality control sample results. The

sample ensures the ability for the laboratory to perform a rerun of the samples.

18.3.2 The need for corrective action will be initiated by the data generator. This may be at the time of QC data assessment and calculating of control sample recoveries. The data generator upon discovering information which exceeds the listed acceptance limits will perform the following:

18.3.2.1 Note the problem exists on the data run sheet

18.3.2.2 Check methodology- review for performance failure, interference or matrix problems, and calculations.

18.3.2.3 Check calculations and data transcription errors.

18.3.2.4 Check reagents and standard for preparation errors.

18.3.2.5 Check the instrument set up and consult instrument log and maintenance reports

18.3.2.6 Review operations manual for the instrument and seek technical help from technical help services.

18.3.3 The laboratory manager will be notified and discuss with the data generator the steps that have been taken, findings, and the outcome will depend on the ability to account for errors. Otherwise reruns will be performed and QC checks implemented throughout the analytical run.

18.3.4 The Laboratory will then release the rerun data if QC checks are determined to fall within acceptable range. Otherwise further investigation will be necessary before any results are released.

18.3.5 Records of corrective actions are maintained by the laboratory manager.

18.4 System Audits

18.4.1 System Audits are carried out by the Project Quality Assurance Officer or equivalently qualified designated person. Ideally, on a yearly basis, the laboratory operations and data management would be audited. The NELAC or VELAP standards would serve as the guide for this audit. The report is distributed to the laboratory manager and his/her supervisor and maintained for three years.

18.5.1 The College of William and Mary will periodically provide an outside auditor to audit the procedural systems and documentation. The auditor will file a final report to those participating in the audit as well as the Dean of the Virginia Institute of Marine Science.

# XIX. Reporting Results

## 19.1 Record Control and Data Maintenance

19.1.1 After initial log in, into the ASPEN LIMS system as described in section 17.3, the chain of custody is filed in a three ring binder designated for that fiscal year in the laboratory manager's office. At the beginning of each calendar year, the previous year's COCs are filed in the filing cabinet in the manager's office.

19.1.2 Using the ASPEN LIMS program, the analyst will generate an analytical worksheet by test group. These worksheets provide the login # and sample information for the analyst's analytical batch run. The LIMS system prioritizes samples by holding time, but the analyst selects the individual samples to run in a batch taking into account holding times and matrix matching. A VIMS benchsheet can be printed at this time.

19.1.3 The analytical worksheet lists the samples for the analyst to pull for analysis and the benchsheet is taken to the lab bench for analysis. Comments, volumes, weights, and pertinent information to the individual tests can be entered directly onto the benchsheet.

19.1.4 Using the ASPEN LIMS system for some analysis (ex. TSS, chlorophyll, grain size, moisture), an electronic worksheet for the recording of the data can be generated from the appropriate template. The template contains pre-determined calculations and can be populated with the login # of samples from the benchsheet. The analyst works from this electronic worksheet, entering data, which is then stored in the data file on the Z: drive under the appropriate year and test group.

19.1.5 Nutrient analysis worksheet is completed by hand and attached to the benchsheet. It identifies the parameters to be run simultaneously, the calibration standards and quality control standards to be run in the analytical run. Other notes concerning the samples are documented on this form.

19.1.6 When the data batch analysis is complete, the raw data and electronic worksheets are saved in the final format on the Z: drive under the appropriate year and test group, printed to hard copy, and attached to the original benchsheet.

19.1.7 Data is then verified by the analyst. The analyst must review the following items:

- Calibration of the instrument verifying it meets calibration criteria
- Quality control data confirming it meets acceptance criteria
- Calculations
- Completing and accuracy of documentation

19.1.8 Using ASPEN LIMS, data is transferred via the existing analytical worksheets tab directly from the electronic worksheet. Quality control data is entered manually if needed. The QC is confirmed and approved by the analyst. The worksheet is approved by analyst. A hard copy of the analytical worksheet, benchsheet, electronic worksheet and computer printout of data is placed in the completed data folder in the laboratory manager's office.

19.2 Final Reporting

19.2.1 The laboratory manager utilizes the ASPEN LIMS system to view completed batches. For each batch of samples completed, data is processed into an interim data report. From the interim data report, the manager manually formats the report putting columns in the correct order, reporting the correct figures in the MDL column. This interim report is saved in the report file under each login # generated for that year.

19.2.2 The data from the interim report is copied into the final report template. The manager finalizes the report ensuring the data formats are correct as well as entering any client specific information including the project name, client, and date of analysis, if required.

19.2.3 Data qualifiers are added to the report to qualify any below detection limit data points, or highlight data points that have a notation such as an asterisk or qualifier number or letter. Data qualifiers are added to data that does not meet analytical or internal QC criteria.

19.2.4 The final report is saved in the report file with the interim report. The report is printed and emailed to the customer. The printed copy is put in the "to be billed" file in the laboratory managers office.

19.3 Billing

Using Aspen LIMS, a billing sheet is produced for each batch of samples. If the client is external to the institute, an invoice is produced and emailed to Accounts Payable located in Waterman's Hall. If the client is an internal VIMS client, a Journal Voucher (JV) is filled out with appropriate index number and the JV and the internal billing documents are sent to accounts payable.

## XX. Glossary

Most of the definitions presented here are selected definitions from 1 VAC 30-45, *Certification for Noncommercial Environmental Laboratories*, the provisions of the Virginia Administrative Code under which noncommercial environmental laboratories are certified in Virginia. Other definitions are included and their source is noted in parentheses at the end of the definition and the full source provided at the end of the glossary.

Acceptance criteria - specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Accuracy - the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations. Accuracy is an indicator of data quality.

Aliquot - means a portion of a sample taken for analysis.

Analyst or laboratory technician - the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Analyte - the substance or physical property to be determined in samples examined.

Analytical batch - a batch composed of prepared environmental samples (extracts, digestates or concentrates) that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Analytical method - a technical procedure for providing analysis of a sample, defined by a body such as the Environmental Protection Agency or the American Society for Testing and Materials, which may not include the sample preparation method.

ASC – Analytical Service Center (located at Virginia Institute of Marine Science, College of William and Mary)

Assessment - the evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and its systems or both to defined criteria.

Assessor - the person who performs on-site assessments of laboratories' capability and capacity for meeting the requirements under this chapter by examining the records and

other physical evidence for each one of the tests for which certification has been requested.

Audit - a systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.

Batch - environmental samples that are prepared together or analyzed together or both with the same process and personnel, using the same lot or lots of reagents.

Blank - a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. Blanks include the following types:

1. Field blank - a blank prepared in the field by filling a clean container with pure deionized water and appropriate preservative, if any, for the specific sampling activity being undertaken.

2. Method blank - a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Calibration - to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

Calibration curve - the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration standard - a substance or reference material used to calibrate an instrument.

Certified reference material - a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation that is issued by a certifying body.

Commercial environmental laboratory - an environmental laboratory where environmental analysis is performed for another person.

Corrective action - the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

Data Quality Objectives – objectives to be met pertaining to quality assurance and quality control standards as well and documentation, sample handling and data handling. These may be specific to projects or programs and must be documented prior to initiation of work.

Demonstration of capability - the procedure to establish the ability of the analyst to generate data of acceptable accuracy and precision.

Detection limit - the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence.

Document control - the act of ensuring that documents, and revisions to the documents, are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (AQSC)

Environmental laboratory or laboratory - a facility or a defined area within a facility where environmental analysis is performed. A structure built solely to shelter field personnel and equipment from inclement weather shall not be considered an environmental laboratory.

Facility - something that is built or installed to serve a particular function.

Finding - an assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition.

Holding time (or maximum allowable holding time) - the maximum time that a sample may be held prior to analysis and still be considered valid or not compromised.

Internal standard - a known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method. (NELAC)

International System of Units (SI) - means the coherent system of units adopted and recommended by the General Conference on Weights and Measures.

Laboratory control sample (LCS) - a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Laboratory duplicate - aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Laboratory manager - the person who has overall responsibility for the technical operation of the environmental laboratory and who exercises actual day-to-day supervision of laboratory operation for the appropriate fields of testing and reporting of results. The title of this person may include but is not limited to laboratory director, technical director, laboratory supervisor or laboratory manager.

Limit of detection (LOD) - an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent.

Limit of quantitation (LOQ) - the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

Matrix - the component or substrate that may contain the analyte of interest. A matrix can be a field of certification matrix or a quality system matrix.

Matrix spike (spiked sample) - a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spike duplicate (spiked sample duplicate) - a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method detection limit (MDL) - one way to establish a limit of detection, defined as the minimum concentration of a substance (an analyte) that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (NELAC)

National Environmental Laboratory Accreditation Conference (NELAC) - a voluntary organization of state and federal environmental officials and interest groups with the primary purpose to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP.

National Environmental Laboratory Accreditation Program (NELAP) - the overall National Environmental Laboratory Accreditation Program of which NELAC is a part.

National Institute of Standards and Technology (NIST) - an agency of the U.S. Department of Commerce's Technology Administration that is working with EPA, states, NELAC, and other public and commercial entities to establish a system under which

private sector companies and interested states can be certified by NIST to provide NIST-traceable proficiency testing (PT) samples.

Owner - any person who owns, operates, leases or controls an environmental laboratory.

Preservation - refrigeration and/or reagents added at the time of sample collection, or later, to maintain the chemical and/or biological integrity of the sample. (NELAC)

Positive control - measures taken to ensure that a test or its components are working properly and producing correct or expected results from positive test subjects.

Precision - the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is an indicator of data quality. Precision is expressed usually as standard deviation, variance or range, in either absolute or relative terms.

Proficiency test or testing (PT) - evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

Proficiency test (PT) sample - a sample, the composition of which is unknown to both the analyst and the laboratory provided to test whether the analyst or laboratory or both can produce analytical results within specified acceptance criteria.

Proficiency testing (PT) program - the aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.

Quality assurance - an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality assurance officer - the person who has responsibility for the quality system and its implementation. Where staffing is limited, the quality assurance officer may also be the laboratory manager.

Quality control - the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Quality control sample or QC sample - a sample used to assess the performance of all or a portion of the measurement system. QC samples may be certified reference materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking. (NELAC)

Quality manual (QM) - a document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality system - a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control.

Range - the difference between the minimum and maximum of a set of values.

"Reference material" means a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement test method, or for assigning values to materials.

Spike - a known mass of target analyte added to a blank sample or sub-sample, used to determine recovery efficiency or for other quality control purposes. (NELAC)

Standard operating procedure (SOP) - a written document that details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Standardized reference material (SRM) - a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method.

Statistical Minimum Significant Difference (SMSD) - the minimum difference between the control and a test concentration that is statistically significant; a measure of test sensitivity or power. The power of a test depends in part on the number of replicates per concentration, the significance level selected, e.g., 0.05, and the type of statistical analysis. If the variability remains constant, the sensitivity of the test increases as the number of replicates is increased. (NELAC)

Test - a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure.

Test method - an adoption of a scientific technique for performing a specific measurement as documented in a laboratory standard operating procedure or as published by a recognized authority.

Traceability - the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

Validation - the confirmation by examination and provision of objective evidence that the particular requirements of a specific intended use are fulfilled. (NELAC)

Verification - the confirmation by examination and provision of evidence that specified requirements have been met. (NELAC) NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

VA DEQ - Virginia Department of Environmental Quality

Working range - the difference between the limit of quantitation and the upper limit of measurement system calibration. (NELAC)

**Definition Sources**

American Society for Quality Control (ASQC), Definitions of Environmental Quality Assurance Terms, 1996

National Environmental Laboratory Accreditation Conference (NELAC), 2003 Standards.

SIGNATURE FOR REVIEW

| Date | Printed Name | Signature | Rev. no |
|---|---|---|---|
| 5/22/2020 | CAROL J JEFFERY | Carol J Jeffery | 9 |
| 6/2/20 | Karen Watkins | Karen Watkins | 9 |
| 5/30/2021 | Carol J Jeffery | Carol J. Jeffery | 10 |
| 6/10/2021 | Karen Watkins | Karen Watkins | 10 |
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