QUALITY MANUAL

for

State of Florida

Department of Environmental Protection

Bureau of Laboratories

FDOH Certification Numbers E31640 and E31780

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Quality Manual for

State of Florida

Department of Environmental Protection

Central Laboratory

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Central Laboratory

Laboratory Annex

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ACRONYMS

ADaPT	Automated Data Processing Tool
AM	Agent Manager
AMU	Atomic Mass Unit
BETX	Benzene, Ethyl benzene, Toluene and Xylenes
BFB	Bromofluorobenzene used for mass spectral tuning
BOD	Biochemical Oxygen Demand
BNA	Base/Neutral, Acid Extractables
CA	Chemical Agent
CAHO	Chemical Agent Hygiene Officer
CAO	Chemical Agent Operator
CAS Number	Chemical Abstract Services Number
CBOD	Carbonaceous Biochemical Oxygen Demand
CCC	Continuing Calibration Compounds
CCCS	Continuing Calibration Check Standard
CCV	Continuing Calibration Verification
CFR	Code of Federal Regulations
CHO	Chemical Hygiene Officer
CHP	Chemical Hygiene Plan
CI	Chemical Ionization
CLP	Contract Laboratory Program
cm	centimeter
COD	Chemical Oxygen Demand
COR	Contract Officer's Representative
CV	Coefficient of Variation
CVAAS	Cold Vapor Atomic Absorption Spectrometry
CVAFS	Cold Vapor Atomic Fluorescence Spectrometry
DF	Dilution Factor
DFTPP	Decafluorotriphenyl Phosphine
DOT	Department of Transportation
DSHP	Dilute Solution Hygiene Plan
ECBC	U.S. Army Edgewood Chemical and Biological Center and Engineering Center
ECD	Electron Capture Detector
EI	Electron Ionization
EICP	Extracted Ion Current Profile (plot of ion abundance vs. time)
EPA	Federal Environmental Protection Agency
ERLN	Environmental Response Laboratory Network
FAC	Florida Administrative Code
FDEP	Florida Department of Environmental Protection

FDOH	Florida Department of Health
GC	Gas Chromatograph
GC/AFD	Gas Chromatograph/Atomic Fluorescence Detection
GC/MS	Gas Chromatograph/Mass Spectrometry
GLP	Good Laboratory Practice
HPLC	High Pressure Liquid Chromatography
Hz	Hertz
I.D.	Internal Diameter
ICAL	Initial Calibration
ICCS	Initial Calibration Check Standard
ID	Identification
IR	Infrared
IS	Internal Standard
ISE	Ion Selective Electrode
IUPAC	International Union of Pure and Applied Chemistry
L	Liter
LC	Liquid Chromatography
LCS	Laboratory Control Sample
LFB	Laboratory Fortified Blank
LIMS	Laboratory Information Management System
LM	Laboratory Manager
LOD	Limit of Detection
LOQ	Limit of Quantitation
m	Meter
MDL	Method Detection Limit
MS	Mass Spectrometry
MSD	Matrix Spike Duplicate or Mass Selective Device
MW	Molecular Weight
NELAP	National Environmental Laboratory Accreditation Program
NIOSH	National Institute of Occupational Safety and Health
NIST	National Institute for Standards and Technology
NPD	Nitrogen Phosphorus Detector
NPDES	National Pollution Discharge Elimination System
NTU	Nephelometric Turbidity Units
ºC	Degrees Celsius
P&T	Purge and Trap
PDF	Portable Document Format
PE	Performance Evaluation
PM 2.5	Particulate Matter ≤ 2.5 Microns

ppb	Parts per billion
PPE	Personnel Protective Equipment
ppm	Parts per million
ppt	Parts per trillion
PQL	Practical Quantitation Limit
PT	Proficiency Testing
PTFE	Polytetrafluoroethylene
QA/QC	Quality Assurance/Quality Control
QAP	Quality Assurance Plan
QATs	Quality Assurance Targets
QCCS	Quality Control Check Standard
QM	Quality Manual
RCRA	Resource Conservation and Recovery Act
RF	Radio Frequency; Response Factor
RL	Reporting Limit
RPD	Relative Percent Difference
RRF	Relative Response Factor
RRT	Relative Retention Time
RSD	Relative Standard Difference
RT	Retention Time
S/N	Signal-to-Noise Ratio
SARA	Superfund Amendments and Reauthorization Act
SD	Standard Deviation
SIM	Selective Ion Monitoring
SOP	Standard Operating Procedure
SPCC	System Performance Check Compounds
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Total Ion Current; Tentatively Identified Compound
TNI	The NELAC Institute
UDA	Ultra Dilute Agent
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
ZHE	Zero Headspace Extraction

1.0 INTRODUCTION, SCOPE AND APPLICABILITY

1.1 INTRODUCTION

The FDEP Bureau of Laboratories' mission is to aid in the protection of Florida's environment by providing legally defensible and scientifically credible analytical and technical support to the Department. Information generated by the Bureau of Laboratories is fundamental to the Department in carrying out its mission to preserve, protect, conserve, and restore the air, water, and natural resources of the state. The Bureau of Laboratories' management is committed to generating data of the highest quality necessary for fulfilling the mission of the laboratory.

1.2 SCOPE

The FDEP Central Laboratory is a full service environmental laboratory which provides chemical and biological analytical support to the following:

a. departmental programs

b. district operations

c. water management districts

d. environmental operations of other state agencies and commissions

e. local, state, and federal law enforcement agencies

The laboratory also provides chemical and biological analytical consulting services to the above listed programs.

1.3 APPLICABILITY

This document serves as the Quality Manual (QM) for the Chemistry and Biology sections of the Bureau of Laboratories.

2.0 REFERENCES

See Appendix C

3.0 TERMS AND DEFINITIONS

The relevant definitions from The NELAC Institute Standard, Volume 1, Module 2, Section 3.0 are the preferred references. See TNI Standard. Definitions related to this document that are used differently or do not exist in the above references are defined in the text.

4.0 MANAGEMENT REQUIREMENTS

4.1 ORGANIZATION

4.1.1 The Bureau of Laboratories is part of the Division of Environmental Assessment and Restoration of the Florida Department of Environmental Protection. The Central Laboratory is located at 2600 Blair Stone Road, Laboratory Annex, Tallahassee, FL 32399-2400. The telephone number is (850) 245-8085. The Florida Department of Health (FDOH) Certification Number is E31780.

4.1.2 The Quality Manual (QM) in conjunction with the Standard Operating Procedures (SOPs) provides guidance for the laboratory operations and serves as the document that defines the criteria necessary to meet the standards of TNI. The QM details the activities and evaluation criteria necessary to ensure that analytical data meet the requirements of TNI. The QM also documents procedures intended to ensure that all data are of high and known quality to meet the scientific objectives of the Department.

4.1.3 The management system described in this document is applicable to all chemistry and biology tests performed by the facility described in 4.1.1 unless otherwise agreed to meet the objectives of the project.

4.1.4 The responsibilities of key personnel are outlined in Section 4.1.5 of the QM. These responsibilities will be performed by the key personnel identified or duly delegated representatives.

4.1.5 The FDEP Central Laboratory:

(a) Shall conduct an annual management review according to Bureau Standard Operating Procedure SOP LB-010, *Quality System Management Review*, to ensure the maintenance of data integrity, quality and efficiency.

(b) Require that all laboratory employees are responsible and conduct themselves in a manner that does not impact the competence and operational integrity of the laboratory as outlined in SOP LB-012, *Code of Ethics*.

(c) Is subject to Chapter 119 of Florida Statutes; therefore all records and documents generated by the FDEP Central Laboratory, except those associated with active criminal investigations, are public records and may be subject to disclosure according to the guidelines and exceptions published in said Chapter. The Laboratory cannot guarantee the confidentiality of reports transmitted electronically or by facsimile.

(d) Has a data integrity training program (See section 5.2.7).

(e) Has a defined organizational structure including quality management, support services and technical operations, see Figure 4.1 (Chemistry Organization Chart), Figure 4.2 (Biology Organization Chart) and Figure 4.3 (LAB Support Organization Chart).

(f) Maintains job descriptions for all employees. See Figures 4.1, 4.2 and 4.3 for the responsibilities of key personnel.

(g) Annually reviews and updates (if necessary) all SOPs . The protocol for updating and reviewing SOPs is described in SOP LB-001, *Protocol for Preparing Standard Operating Procedures*, and in SOP LB-010, *Quality System Management Review*. Any updates to SOPs must have the approval of the supervisor responsible for the procedure contained in said SOP and must conform to the policies of the laboratory. It is the responsibility of laboratory supervisors to ensure proper documentation demonstrating that their employees have read, understood and are using the latest versions of SOPs and that this is documented in the LIMS training module. The latest official versions of SOPs are only located on the FDEP Intranet site and are accessible to all analysts and technicians throughout the laboratory. Laboratory analysts are required to successfully analyze initial and on-going demonstrations of capability according to Appendix B, section 6.0.

(h) Has technical management as identified in section 4.1.7.2.

(i) Has a full time designated Quality Assurance Officer. See section 4.1.7.1 for a description of responsibilities.

(j) Has alternate supervisors who, in the absence of key management personnel, are assigned to assume their responsibilities. In the absence of the Laboratory Director the Environmental Administrators will assume his duties.

(k) Has ethics training described in section 5.2.7 emphasizing the importance of the activities of each employee and the ramifications of them not performing their responsibilities according to laboratory procedures and policies.

4.1.6 A Quality System Management Review takes place annually to ensure that management holds regular staff meetings to discuss quality issues, workload and staffing issues and other items of importance to the laboratory. Necessary changes to the quality system as a consequence of performance on proficiency samples, round robins, split samples or audits are discussed for incorporation and implementation. See SOP LB-010, *Quality System Management Review*, for further information.

4.1.7 Additional requirements for laboratories:

4.1.7.1 The Quality Assurance Officer or his designee:

(a) serves as the primary contact for oversight and review of quality control data

(b) conducts all Quality Assurance responsibilities independent of other laboratory operations

(c) conducts oversight of QA data without outside influence

(d) has experience in QA/QC procedures and the laboratory Quality System

(e) has experience with the analytical methods

(f) conducts annual internal audits

(g) monitors QA/QC activities of the laboratory and notifies management of deficiencies

(h) monitors and ensures corrective actions are effective

4.1.7.2 The technical managers exercise day to day supervision and are responsible for the operation of both laboratories, except for the Bureau Chief and the Laboratory Directors who are responsible for the FDEP Central Laboratory.

4.2 MANAGEMENT SYSTEM

4.2.1 Description

The Central Laboratory is part of the Bureau of Laboratories in the Division of Environmental Assessment and Restoration. The laboratory is comprised of Chemistry and Biology laboratories and a Laboratory Support Section. Each section is managed by a Program Administrator who reports directly to the Bureau Chief.

CHEMISTRY

The Chemistry Section is divided into two subsections: organic analysis and inorganic analysis. The Section is headed by a Program Administrator, who is responsible for both the technical and administrative direction of the Section. The Program Administrator and the Quality Assurance Officer are committed to the QA program described in this plan. See Figure 4.1 for the organizational structure.

The analytical subsections are headed by Environmental Administrators who are responsible for ensuring their staff are cognizant of the objectives and requirements of the QM and SOPs and that data submitted to the QA Officer or Section Administrator meet these requirements. Each Environmental Administrator is supported by supervisors (either Environmental Managers or Chemist Administrators) responsible for all bench analysts and technicians in their group. The inorganics subsection is divided into three work groups; nutrients, general chemistry and metals. The organics subsection consists of the volatiles, semi-volatile and pesticide work groups.

BIOLOGY

The Biology Section is divided into three subsections: bench biology, applied biology and taxonomy. The Section is headed by a Program Administrator, who is responsible for both the technical and administrative direction of the Section. The Program Administrator and the Quality Assurance Officer are committed to the QA program described in this plan. See Figure 4.2 for the organizational structure.

The biology subsections are headed by Environmental Managers who are responsible for ensuring their staff are cognizant of the objectives and requirements of the QM and SOPs and that data submitted to the QA Officer or Section Administrator meet these requirements.

Each Environmental Manager is supported by Environmental Supervisors responsible for all bench analysts and technicians in their group. The bench biology subsection is divided into two work groups; toxicology/chlorophyll/sediment, and microbiology/BOD. The taxonomy subsection consists of the taxonomic preparation, algal ID, and invertebrate ID work groups.

Organization and Management of Ultra-Dilute Chemical Agent Facility

The Florida Department of Environmental Protection has been selected to participate in a national network of laboratories known as the Environmental Response Laboratory Network (ERLN). This network is one of several laboratory networks established through Presidential directives to ensure that critical services are available to support response and recovery operations following an emergency of national significance or terrorist attack. The Environmental Response Laboratory Network will be administered by the U.S. Environmental Protection Agency with support from the U.S. Department of Homeland Security.

The roles and responsibilities of the management and staff involved with work performed for the ERLN are provided in Appendix A.

Laboratory Support Section

The Laboratory Support Section (LSS) is composed of three groups; data support, laboratory quality assurance and sample support. Each of these groups is supervised by an Environmental Administrator or Environmental Manager that reports directly to the Program Administrator. See Figure 4.3 for the organizational structure.

4.2.2 Quality Policy Statement

The FDEP Bureau of Laboratories mission is to aid in the protection of Florida's environment by providing legally defensible and scientifically credible analytical and technical support to the Department. The Bureau's management is committed to generating data of the highest quality necessary for fulfilling the mission of the laboratory.

4.2.3 Management Commitment

The Bureau of Laboratories management is committed to generating data of the highest quality necessary for fulfilling the mission of the laboratory and satisfying customer expectations.

Bureau of Laboratories

G. William Coppenger, Ph.D.: Bureau Chief
Directs all administrative and technical activities of the Bureau of Laboratories.

Chemistry Laboratory

Timothy W. Fitzpatrick, M.S.: Program Administrator
Directs activities of the Chemistry Section and serves as Chief Chemist for the Department. Certifies analytical reports for release to clients. Serves as the Laboratory Director for the ERLN Ultra-Dilute Chemical Agent Laboratory.

Liang-Tsair Lin, Ph.D.: Environmental Administrator
Supervises the organic chemistry subsection of the laboratory. Responsible for results generated from analyses of water, soil/sediment, tissue and waste samples submitted for pesticide, volatile and semi-volatile organic analyses. Ensures compliance with quality control and laboratory quality assurance objectives. Certifies analytical reports for release to clients. Serves as the Assistant Laboratory Director for the ERLN Ultra-Dilute Chemical Agent laboratory.

Colin Wright, Ph.D.: Environmental Administrator
Supervises the inorganic chemistry subsection of the laboratory. Responsible for results generated from analyses of water, soil, tissue and waste samples submitted for inorganic analyses including metals, nutrient, general chemistry and major anion content and other related analyses. Ensures compliance with quality control objectives and laboratory quality assurance in the inorganic subsection. Certifies analytical reports for release to clients.

Dr. rer. nat. Bettina Steinbock, Environmental Manager
Supervises the nutrients and general chemistry work groups. Responsible for results generated from analyses of water, soil, tissue and waste samples submitted for inorganic tests including nutrient, general chemistry and major anion content and other related analyses. Ensures compliance with quality control objectives and laboratory quality assurance in the nutrients and general chemistry group. Certifies analytical reports for release to clients.

Sathavaram Reddy, Ph.D.: Chemist Administrator
Supervises the HPLC, semi-volatile and organic sample preparation work groups, and analyzes samples for the presence of semi-volatile organic pollutants and pesticides. Analyses are performed using HPLC and GC techniques. Responsible for: developing daily work plan for routine analytical work to ensure that sample holding time requirements and turn-around commitments are met; resolving analytical and instrumental problems; maintaining protocols to meet QA/QC objectives of the laboratory; supervising analysts and technicians in their duties; ensuring subordinates are following proper laboratory safety and waste management procedures; and implementing new or modified analytical procedures and instruments.

Michael Thompson, B.A.: Environmental Manager
Supervises the metals analysis work group. Responsible for results generated from metals analyses of water, soil, tissue and waste samples submitted to the laboratory. Ensures

compliance with quality control objectives and laboratory quality assurances in the metals group.

Adrian Niculescu, M.S.: Chemist Administrator
Supervises the GC pesticide work group, which includes the sample preparation and analysis of target pesticides and PCBs. Analyses are performed using GC/ECD and GC/NPD techniques. Responsible for: results generated from pesticides and PCB analyses of water, soil, tissue and waste samples. Resolves analytical and instrumental problems, ensures compliance with QA/QC laboratory protocols and develops and implements new or modified analytical procedures.

Kerry Tate, Ph.D.: Environmental Manager
Supervises the volatile organics (VOC), methyl mercury and the PM 2.5 work groups. Analyses are performed using GC/MS, GC/AFD and gravimetric techniques, respectively. Responsible for analytical work generated from the analysis of environmental samples submitted to the laboratory for measurement of volatile organic pollutants, methyl mercury and atmospheric particles. Ensures compliance with quality control objectives, laboratory quality assurances, laboratory safety and waste management procedures in the VOC and PM2.5 work groups. Certifies analytical reports for release to clients. Serves as the Laboratory Manager, Agent Manager, and Co-Chemical Agent Hygiene Officer for the ERLN Ultra-Dilute Chemical Agent Laboratory.

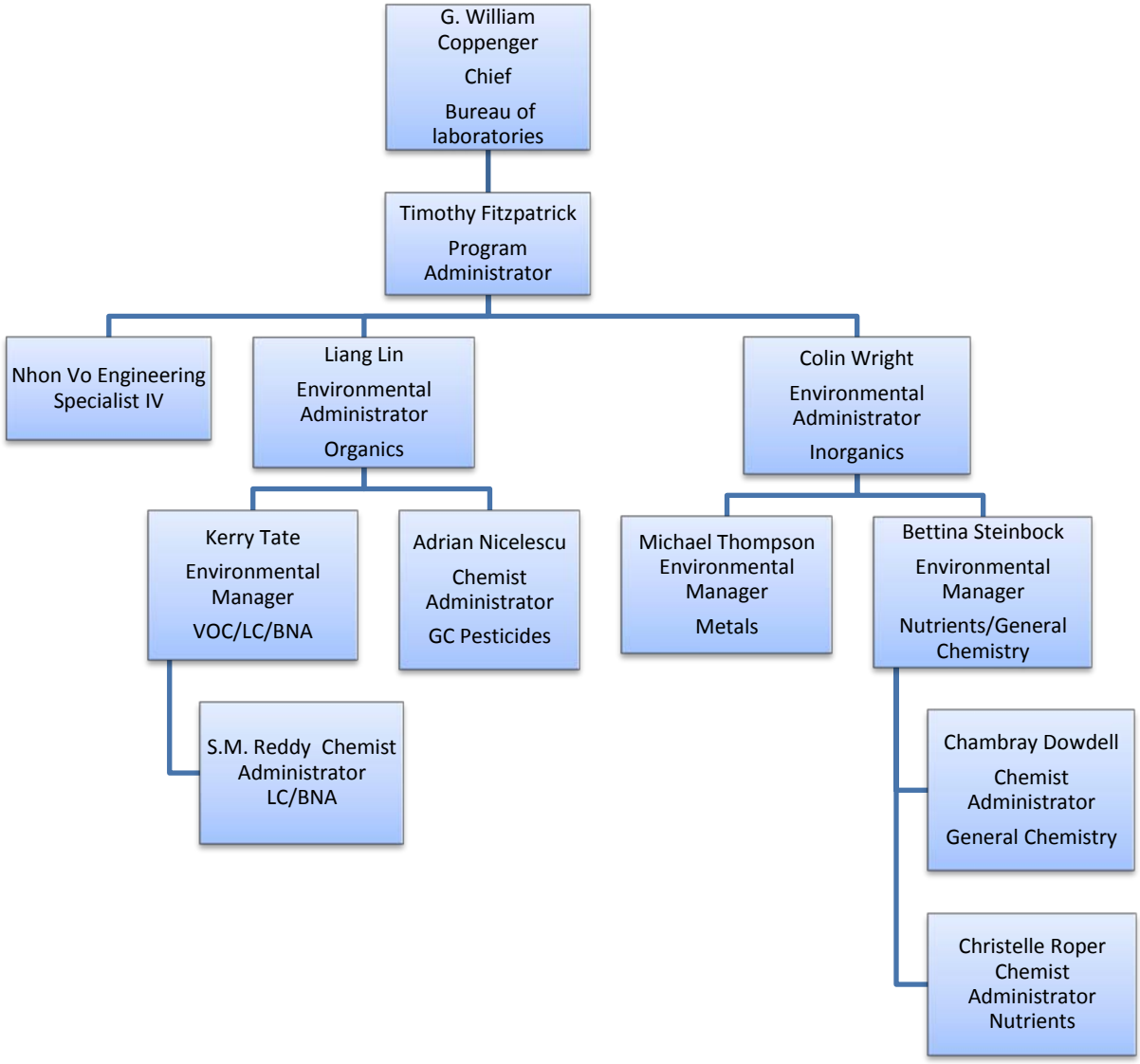
Chambray Dowdell, B.S.: Chemist Administrator
Supervises the general chemistry and major ion analysis work group. Responsible for results generated from general chemistry and major ion analyses of water, soil, tissue and waste samples submitted to the laboratory. Ensures compliance with quality control objectives and laboratory quality assurances in the general chemistry and major ions work group.

Nhon Vo: Engineering Specialist IV
Plans, designs and coordinates building renovations and construction for the FDEP Bureau of Laboratories. Coordinates and maintains the building mechanical and electrical systems overseeing upgrades and repair. Serves as the Property Inventory Officer, the Division Health and Safety Officer, the Hazardous Waste Coordinator, the Radiation Safety Officer and the Co-Chemical Agent Hygiene Officer for the ERLN Ultra-Dilute Chemical Agent Laboratory.

Figure 4.1 shows an organizational chart of the management staff of the chemistry laboratory.

Figure 4.1

Chemistry Section Organizational Chart



Biology Laboratory

David D. Whiting, M.S.: Biology Administrator
Section Administrator for the Biology Section. Responsible for overseeing all issues related to personnel, budget, infrastructure, workload, and analytical services. Coordinates document review and technical services for other agency programs. Directly manages the supervisors for all of the groups in the Biology Section. Represents the Biology Section in meetings with clients and management.

Elizabeth Miller, B.S.: Environmental Manager
Manager of the Bench Biology Workgroup. Assists with issues related to personnel, budget, infrastructure, workload, and analytical services. Responsible for overseeing administrative, technical, and analytical issues for the Bench Biology Workgroup. Represents the Biology Section in meetings with clients and management. Certifies analytical reports for release to clients.

Loretta Wolfe, M.S.: Environmental Manager
Manager of the Applied Biology Workgroup. Assists with issues related to personnel, budget, infrastructure, workload, and analytical services. Responsible for overseeing administrative, technical and analytical issues for the Applied Biology Workgroup. Reviews documents submitted to the Department for technical merit. Represents the Biology Section in meetings with clients and management. Certifies analytical reports for release to clients.

Cheryl Swanson, M.S.: Environmental Manager
Manager of the Taxonomy Workgroup. Assists with issues related to personnel, budget, infrastructure, workload, and analytical services. Responsible for overseeing administrative, technical and analytical issues for the Taxonomy Workgroup. Assists with field sampling. Reviews documents submitted to the Department for technical merit. Represents Biology Section in meetings with clients and management. Certifies analytical reports for release to clients.

J. Marshall Faircloth, B.S.: Environmental Supervisor II
Production coordinator for the Bioassay, Algal Growth Potential, Chlorophyll, and Sediment tests. Responsible for coordinating staff in the scheduling, preparation, and analysis of samples for the group, including associated quality control and data reporting. Performs on-site performance audit inspections of laboratories performing NPDES compliance monitoring tests. Serves as the liaison to the FDEP Ground Water Protection section in the evaluation of pesticides. Reviews documents submitted to the Department for technical merit.

Daisys Tamayo, B.S.: Environmental Supervisor II
Production coordinator for the Microbiology and BOD portion of the Bench Biology Workgroup. Responsible for coordinating staff in the scheduling, preparation and analysis of samples for the group, including associated quality control and data reporting. Assists with field sampling.

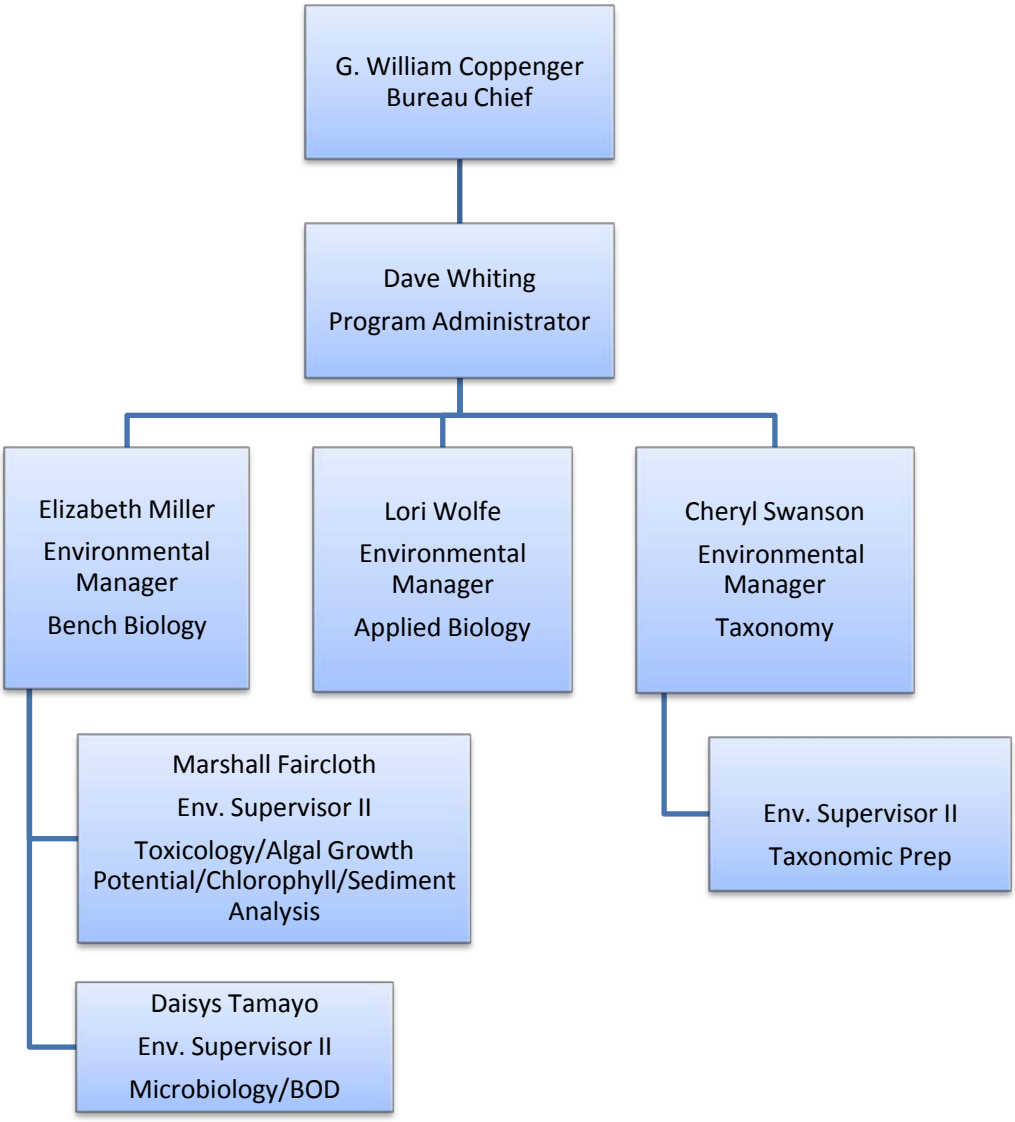
Jonathan Ray, B.S.: Environmental Supervisor II
Supervisor of the Taxonomic Preparation Workgroup. Responsible for coordinating staff in sample preparation and processing. Assists with field sampling, workload tracking, and tracking taxonomy related quality control. Assists in preparation and review of reports for

Fifth Year Biological Assessment of point source facilities. Represents Biology Section in meetings with clients and management.

Figure 4.2 shows an organizational chart of the management staff of the biology laboratory.

Figure 4.2

Biology Section Organizational Chart



Laboratory Support Section

Kathleen Lurding, M.Sc., Program Administrator
Directs the Bureau's laboratory support section. Supervises professional, technical and clerical staff as required. Oversees the design, construction, testing and implementation of data acquisition and management applications. Oversees user training. Consults with Bureau staff on hardware and software purchases. Provides technical assistance to FDEP staff and external stakeholders on issues of data management and assessment.

John Watts, B.Sc., Environmental Manager
Organizes and assists with prioritization of workload activities to support the analysis and interpretation of scientific data used to support FDEP and external programs. Plans, coordinates, and directs staff and/or contractors in the development or acquisition of computer software for use by the Bureau of Laboratories. Acts as the Web Administrator for the Bureau of Laboratories, overseeing the maintenance of information available to the Department and the public sector and the development of web-based tools for retrieving or storing information. Oversees computer software and hardware purchases, according to procedures outlined in the Governor's One Florida initiative. Supervises staff that performs the following functions:

- provides hardware and software technical support for the Bureau, including administration of servers and backup of data from analytical instruments and personal computers;
- supports the electronic acquisition, quality assessment, storage, and archive of chemical and biological analytical data according to approved QA/QC protocols;
- manages the computer hardware and software inventories for the Bureau.

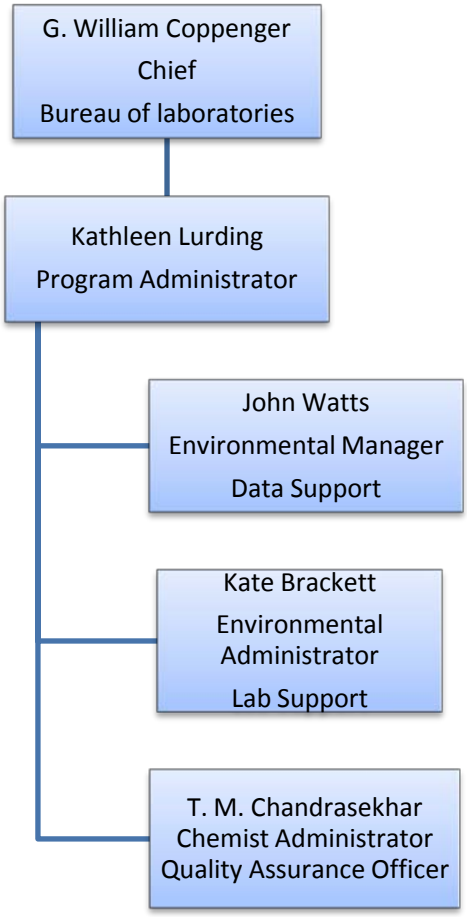
Kate Brackett, B.A.: Environmental Administrator
Serves as the Project Manager for the Bureau of Laboratories. Is the customer contact point in scheduling sample analyses and in responding to customer inquiries. Manages overflow laboratory and external contracts. Supervises operation of the laboratory receiving work group. Certifies analytical reports for release to clients.

T. M. Chandrasekhar, Ph.D.: Quality Assurance Officer/Chemist Administrator
Serves as the laboratory's Quality Assurance Officer. Initiates and oversees all internal QA/QC audits. Manages the laboratory's blind proficiency testing program and maintenance of the laboratory's SOPs. Evaluates quality control results, corrective actions and establishes policy for laboratory quality management. Reviews, certifies and signs analytical reports.

Figure 4.3 on the next page shows an organizational chart of the management staff of the laboratory support section.

Figure 4.3

Laboratory Support Section Organizational Chart



4.2.4 Meeting Customer Requirements

Samples must be scheduled with the laboratory prior to acceptance of the samples by the laboratory. The decision to formally accept samples is based on the client's expectations for analytical methods, turnaround times, laboratory capacity, sensitivity and confidentiality (for criminal events). All scheduled work is reviewed by Chemistry and Biology Section managers prior to approval for receipt by the laboratory. Samples are accepted for analysis by logging them into the LIMS and assigning tests.

See SOP LB-018, *Standard Operating Procedure for Using the LIMS Scheduler*. The LIMS software system for scheduling samples assesses the capacity and previously scheduled workload of the laboratory and warns the user whenever laboratory capacity for any work group may be exceeded. The laboratory may elect to accept samples in excess of stated capacity if there is a reasonable expectation that the work can be completed within holding times.

See SOP LB-028, *Standard Operating Procedure for Tracking Priority Projects,* for details on how priority projects are handled and clients are kept informed of progress.

The customer is notified of any non-conformances that may affect the integrity of the data. The samples are analyzed unless the customer requests otherwise or the nature of the non-conformance makes analysis impractical. Data from compromised samples are flagged with the appropriate qualifier(s) or comments and a non-conformance report is issued with the report. The Laboratory non-conformance system procedures are described in SOP LB-002, *Non-Conformance Reporting System*.

Significant deviations from standard policies or practices of the laboratory are reported to the client and documented with the analytical reports. Any samples that are prepared or analyzed beyond accepted holding times have a statement automatically stamped with the data alerting the client to the fact that tests were conducted after the sample had expired. Similarly, the failure of any quality control checks is commented with the data, directing the client to the Quality Control Report for details of failures. Data qualifiers are used to alert clients of quality control problems and holding time exceedances.

Accepted samples that were improperly preserved are documented in the LIMS and analytical reports as sample level comments and/or in a LIMS non-conformance report. All other significant observations that do not conform to accepted practices or policies are documented and reported along with analytical results. Those documents may be as letters, interoffice memoranda or appendices to analytical reports. Sample integrity such as improper temperature and pH preservation, the presence of bubbles in VOC vials, insufficient volume, leaking or broken bottles, etc., are entered into a non-conformance report in the LIMS as well as documented on a log-in check form with the log-in technician's initials.

4.2.5 Technical and Supporting Procedures

The laboratory SOPs are divided into three groups, those for the chemistry laboratory, the biology laboratory, and the Bureau. The only location at which current revisions of all SOPs (PDF files) should be accessed is at http://www.dep.state.fl.us/labs/library/lab_sops.htm Printed copies are not considered by the laboratory to be official SOPs. For the purpose of reviewing and revising SOP's annually, copies of current revisions (Word) can be saved to the reviewer's computer from \\Tlhlab3\sops. The versions stored on TLHlab3 are locked and maintained for archival purposes only. The format of the SOPs is detailed in SOP LB-001, *Protocol for Preparing Standard Operating Procedures (SOPs).*

4.2.6 The responsibilities of all supervisors within the Bureau of Laboratories can be found in the appropriate job descriptions detailed in section 4.2.3. Personnel authorized to certify reports are stipulated in these job descriptions.

4.2.7 All laboratory personnel are required to annually review and update (if necessary) all SOPs that pertain to the work they perform within the laboratory (SOP LB-010). See section 4.1.5 (g) for further information on notifying personnel of system changes.

4.2.8 Additional Management System Requirements

4.2.8.1 See section 5.2.7 for a description of the Laboratory Data Integrity System.

4.2.8.2 The Quality Assurance Officer is responsible for keeping the Laboratory QM up to date. The QM is reviewed and revised at least annually and posted to the Bureau of Laboratories' web site at: http://www.floridadep.org/labs/library/progplan.htm. Revisions are approved through the laboratory supervisors and forwarded to the Quality Assurance Officer for incorporation into the QM. The posted version is the latest official version of the QM.

4.2.8.3 This QM along with the laboratory SOPs detail the FDEP Bureau of Laboratories' Quality System for the laboratory. This document contains all of the mandatory information required by Section 4.2.8.3 of the TNI standards Volume 1, *Management and Technical Requirements for Laboratories Performing Environmental Analysis*, Module 2: Quality System Requirements.

4.2.8.4 This QM contains or references all of the topics in Section 4.2.8.4 of the TNI standards Volume 1, *Management and Technical Requirements for Laboratories Performing Environmental Analysis*, Module 2: Quality System Requirements..

4.2.8.5 SOPs addressing all activities of the laboratory including all test methods and supporting activities may be found at: http://www.floridadep.org/labs/library/lab_sops.htm. All of the required topics in 4.2.8.5 (f) of the TNI standards are in or referenced in the test method SOPs.

4.3 DOCUMENT CONTROL

4.3.1 FDEP SOP LB-001, *Protocol for Preparing Standard Operating procedures (SOPs)*, explains how SOPs are created, revised, and approved. This SOP details where the current SOPs are maintained and archived. The electronic system for maintaining the SOPs has the capability of creating reports to track revision effective dates.

Record archiving procedures are found in SOP LB-013, *Standard Operating Procedure for Archiving/Retrieving Documents and Using the Document Control System*. Archived records include raw laboratory data, laboratory notebooks, final reports, administrative files, personnel records, purchase requisitions and purchase orders. The electronic document control system allows entering new archive records, identifying archives and locations and a check out system.

The QM is updated at a minimum on an annual basis. The official version of the QM is available for viewing by all employees at http://www.floridadep.org/labs/library/progplan.htm. All revisions of the QM are archived and easily accessible on a network drive at TLHlab3/SOPs/QA Manual.

4.3.2 Document Approval and Issue

4.3.2.1 The procedure by which an SOP is implemented or revised is as follows:

- Technician, chemist/biologist, and/or analytical group supervisor involved in carrying-out the procedure prepares a draft SOP detailing methodology.

- The draft SOP is reviewed by the Environmental Manager (EM) or Environmental Administrator (EA) in charge and submitted to the QA Officer as a final SOP.

- The QA Officer (or delegate) posts the final SOP to the Bureau of Laboratories' Internet site in 'portable document format' (PDF). The name of the SOP authorizer (supervisor submitting the final version), the effective date and the revision date are recorded using the SOP Update Utility module of the Laboratory Information Management System. The SOP expiration date will be set one year from the date the version was revised.

- To revise an existing SOP the revised SOP is submitted to the EM in charge of the pertinent work group. The EM reviews the SOP and submits it to the QA Officer, indicating whether the change is a minor or major revision. Minor revisions are those which do not involve modifications to the method, while major revisions indicate the method has been altered. A change in procedure that produces results that are incomparable with those reported before the method was modified would also justify a major revision. All SOPs must contain an Appendix of Significant Changes made during the revision period. The Appendix must include the date of any significant edits and should indicate the pertinent SOP sections that have been edited. The final SOP should be submitted to the QA Officer (or delegate) as a Word document with track changes turned on to indicate the content altered during the revision.

Note: In cases where a specific SOP version has been certified and subsequently undergoes a major revision, additional steps are required. The supervisor submitting the revision must also submit a method validation package and the appropriate paperwork for requesting NELAP certification of the new SOP version. ***Supervisors are responsible for ensuring that analysts are trained on new or revised SOPs and that former printed versions of SOPs are replaced with new versions. The only official versions of SOPs are those that reside on the laboratory's internet (or intranet) site for SOPs. Printed copies are not considered by the laboratory to be official SOPs.***

4.3.2.2 Revisions to the QM are submitted by the technician, chemist, biologist, and/or analytical group supervisor involved in carrying-out the procedure. The proposed revisions are reviewed by the Environmental Manager/Environmental Administrator and, if acceptable, forwarded to the QA Officer. The QA Officer evaluates the changes for compliance with laboratory policies and procedures and quality assurance issues and then is responsible for posting the revised manual.

4.4 REVIEW OF REQUESTS, TENDERS AND CONTRACTS

4.4.1 Contract/order review is an integral part of the FDEP Bureau of Laboratories. All contracts/orders are reviewed and accepted only if the requirements are clear and understood, and the laboratory has the capability and capacity to meet full customer expectations. The criteria used to review and accept projects are described in SOP LB-033, *Procedure for Receiving and Accepting Laboratory Projects*. Upon receiving a request from a client the FDEP Project Manager obtains specific project information. The information includes but is not limited to the following:

- The project description (and purpose if needed)
- The analyses requested
- The sample matrices
- The number/frequency of the samples
- Completion expectations

The FDEP Project Manager disseminates the information to the Laboratory Administrators for evaluation. If the FDEP Laboratory is able to accept the work then the FDEP Project Manager provides the following information in writing (or by e-mail) to the client:

- A list of the analytical methods the FDEP Lab will use.
- Information on whether the FDEP Lab has FDOH certification for the methods
- The detection limits and quantitation limits for the methods
- The costs of the analyses (if applicable)
- Additional information upon request, such as non-NELAP proficiency studies and quality assurance/control information

Once the client and the Lab mutually agree upon the project then the FDEP Project Manager obtains the account information.

4.4.2 Records of reviews, including any significant changes, are maintained.

4.4.3 Communications are maintained with the client from request/quote through commencement of work. This includes informing the client of any deviation from the contract or agreement.

4.5 SUBCONTRACTING OF ENVIRONMENTAL TESTS

Samples that need to be sent out to an overflow contract laboratory will show transfer to an overflow lab in the LIMS custody record. There is a task assignment number (e.g. SA034) documented on a task assignment form, Figure 4.4. The task assignment form includes a FDEP staff signature authorizing the work, a list of sample ID's and requested analyses with turnaround time, the date/time the samples were sent out and the identity of the custodian responsible. Samples, along with copies of the field information and the task assignment

form are delivered to the contract lab. The delivery person and the recipient at the contract lab must sign the task assignment form indicating the transfer date and time. The task assignment form becomes part of the custody record.

See SOP LB-008, *Standard Operating Procedure for Outsourcing Analyses through a Contract Laboratory*, for further details.

4.6 PURCHASING SERVICES AND SUPPLIES

4.6.1 A list of state approved suppliers for the purchase of supplies and services is maintained by the Florida Department of Management Services (DMS) at http://dms.myflorida.com/business_operations/state_purchasing/vendor_information
The laboratory maintains an inventory list of chemicals and supplies commonly used for analyses performed in the laboratory. See SOP LB-030, *Standard Operating Procedure for Purchasing and Receiving Laboratory Chemicals and Supplies*. This SOP includes procedures ensuring the necessary quality of the purchase, checking that the proper quality was received and how the supplies are stored if needed to maintain the quality.

4.6.2 Purchased services, supplies and consumable materials are not used until an inspection is performed to ensure compliance with purchasing specifications.

4.6.3 Purchasing records will contain descriptions of the quality required for testing.

4.6.4 If a chemical or consumable ordered by the lab is found to be lacking in the minimum quality required for its intended use, then the lab will pursue the purchase of the desired item from an alternative supplier that meets the required specifications and is listed in the Florida DMS database of state approved vendors. The list is maintained at:
http://dms.myflorida.com/business_operations/state_purchasing/vendor_information

4.7 SERVICES TO THE CLIENT

4.7.1 The FDEP Bureau of Laboratories does its utmost to meet its clients' needs, including:

- Affording customers access to the laboratory to witness testing when requested.
- Preparing, packaging and dispatching test items and reports as required by our customers for verification purposes.
- Advising, guiding and communicating with our customers on technical matters, providing opinions and interpretations for testing performed or to be performed.
- Communicating to our customers any major deviations in testing being performed. See SOP LB-002, *Non-conformance Reporting System*.
- Communicating to customers any delays that may result in the customers not receiving their test results in a timely manner.

- Notifying customers of any event that casts doubt onto the validity of results supplied to them.

4.7.2 The laboratory solicits customer feedback from its customers using a comprehensive written survey once a year. Feedback from our clients by other channels such as phone and email will be encouraged throughout the year to improve our operations. Feedback from lab customers will be maintained by the QA officer.

4.8 COMPLAINTS

The Bureau of Laboratories is committed to resolving complaints and implementing suggestions for improvement. All informal complaints, suggestions or requests for information are directed to the appropriate supervisor for resolution. If immediate resolution cannot be attained, the matter is submitted to the section administrator who may investigate and direct the resolution. Formal written complaints are logged with the section and, after investigation and resolution, are responded to in writing. Copies of responses are organized and filed by the Quality Assurance officer for reference.

4.9 CONTROL OF NON-CONFORMING ENVIRONMENTAL TEST WORK

(See SOP LB-002, *Non-conformance Reporting System*).

4.9.1 Significant deviations from the laboratories' policies and procedures, as outlined in the Quality Manual and SOPs, shall not be approved without appropriate documentation. Significant deviations from standard policies or practices of the laboratory are reported to the client and documented with the analytical reports. Any samples that are prepared or analyzed beyond accepted holding times have a statement automatically stamped with the data alerting the client to the fact that tests were conducted after the samples had expired. Similarly, the failure of any quality control checks is commented with the data, directing the client to the Quality Control Report for details of the failures.

4.9.2 Where non-conformances are indicative of systematic errors, the corrective action procedures described in section 4.11 will be instituted.

4.10 IMPROVEMENT

The Bureau of laboratories is committed to continually improving the quality management system by:

- Multi-tier and electronic data review process (see SOP LB-025, *Event Level Authorization Checklist* and SOP LB-026, *Job Level Authorization Checklist)*
- Internal and external audits
- Systematically evaluating quality data and updating allowable acceptance criteria
- Participating in round robins and laboratory intercomparisons
- Performing quality system management reviews (see SOP LB-010)

4.11 CORRECTIVE ACTION

4.11.1 General

Corrective actions for the chemistry section are presented by analyte group in Table 4.1. Included in the table are:

- QC Checks and Acceptance Criteria
- Corrective Actions
- Responsible Parties
- Notification Protocols

Corrective actions and notification protocols for the biology section are listed in Table 4.2. Corrective actions are initiated based on either the internal QC checks listed in Table 4.1 and Table 4.2, data validation by a reviewing authority or performance audits. Outside sources such as performance evaluation studies, split samples as well as recommendations by the FDEP QA Officer will initiate corrective actions. See FDEP SOP LB-002, *Non-Conformance Reporting System*, for additional information on initiating corrective actions.

4.11.2 Cause Analysis

All non-conformances will be evaluated to determine the root cause. Many factors are taken into consideration in this evaluation and the cause may not be directly attributable to laboratory operations.

4.11.3 Selection and Implementation of Corrective Actions

The details of identifying corrective actions and remedies taken are detailed in SOP LB-002. The likely cause of a given problem must first be identified and then corrective actions put into place to alleviate the problem. The extent of the corrective actions required will be evaluated against the seriousness of the non-conformance. All corrective actions will be administered within a reasonable time frame.

4.11.4 Monitoring

All corrective actions will be documented and monitored to ensure compliance with the laboratory's policy and procedures.

4.11.5 Additional Audits

The efficacy of any corrective actions will be evaluated during routine internal and performance audits. Additional audits will be scheduled to address non-conformances that will not allow the laboratory to meet their established operating protocols.

4.11.6 All actions taken under this section will be documented.

4.12 PREVENTIVE ACTION

4.12.1 Preventive action is a proactive process, used to identify process and quality system improvement opportunities (see for example, SOP LB-010, *Quality System Management Review*). Routine lab and field procedures are listed in Table 4.3, *Preventive Maintenance – Chemistry* and Table 4.4, *Preventive Maintenance – Biology*, and in the individual operational SOPs (http://www.floridadep.org/labs/library/lab_sops.htm). They list the types of analytical equipment utilized to perform routine analyses and the frequency of preventive maintenance tasks performed to ensure data quality.

4.12.2 All maintenance or repair to equipment is documented in a laboratory notebook. Documentation includes a description of the problem(s), work performed, date and analyst's initials. See section 5.5.7 for details. The management system will also be evaluated to institute process revisions to create a continuous improvement process. See Section 4.1.6 for an explanation of the Management Review system.

4.13 CONTROL OF RECORDS

4.13.1 General

4.13.1.1 For a description of laboratory *Records Maintenance and Storage* see SOP LB-006. Records associated with QA activities including external and internal audits, certification records, and PT studies are filed in a dedicated quality assurance filing cabinet.

4.13.1.2 All records shall be legible, easily accessible and stored in a manner that will minimize loss, damage or deterioration. The Department will continue to maintain client access to electronic and paper records for a period of not less than 5 years after the completion of the laboratory project (see SOP LB-006 and SOP LB-013 for details).

4.13.1.3 See section 4.1.5 (c) concerning access to laboratory records and documents.

4.13.1.4 Electronic records are copied (incremental backups) onto backup tapes each business night. Full backups are conducted during weekends and the files archived to DVD as needed.

The laboratory maintains a document control system for tracking the archive process for paper records and laboratory logbooks. The document control system is an electronic database that resides on the laboratory's server and is available through the Department Intranet. Paper records are boxed, labeled and submitted to the State Records Center for storage. The State Records Center assigns the boxes a tracking number that is entered into the document control system, along with a description of the box contents, the date range of records in the box, the archival date and the name of the submitter. When records are needed for retrieval, the document control system database is searched for the pertinent box identification number and that number is submitted to the State Records Center for retrieval.

The Office of Technology and Information Services (OTIS) maintains the Oracle database where LIMS records are stored. A complete backup of the oracle tables is exported to a password-protected server administered by OTIS. This backup file is copied onto backup tape each business night. Copies of pertinent raw and processed data may be maintained in electronic and paper format. Additionally, the laboratory maintains paper copies of all final analytical reports and client custody records. These paper records are maintained in the laboratory and archived offsite as needed in the State Records Center. All paper records are maintained in a temperature and humidity controlled environment and are protected by fire suppression equipment.

4.13.2 Technical Records

4.13.2.1 Most of the data generated by the laboratory during the analytical testing process is in the form of electronic records. Those data consist of raw data files generated by analytical instrumentation, chromatography acquisition software, etc. as well as processed and final database records residing in the Laboratory Information Management System (LIMS).

All raw data files, including processed chromatography data and formatted, processed instrument files are stored on servers maintained by OTIS. Those files are organized by file type and date of generation and are copied onto backup tapes each business night. The files are archived to optical compact disk (DVD) as needed. Software and hardware systems will be maintained to ensure that raw data are available for a period of not less than five (5) years after completion of the laboratory project. Records maintained shall allow the re-creation of the calibration and test procedures and personnel responsible for the different aspects of the test procedure. See Bureau of Laboratories SOP LB-006, *Standard Operating Procedure for Records Maintenance and Storage*, for complete details.

14.13.2.2 The nature and intent of all documentation will be clearly established and all records will be captured at the time of generation.

14.13.2.3 Entry errors on paper records will not be obliterated or erased. Corrections will be made by marking a line through the error so that it is legible. The marked error shall be initialed and dated and a reason for the correction will be annotated when the cause is not obvious or due to simple transcription errors. Access to electronic records is restricted and where possible an electronic audit trail is maintained for write access only.

4.13.3 Additional Records

a) Documentation of Sample History

Sample Custody

The custody of a sample is defined as one of the following:

- It is in the sampler's or transferee's actual possession;

- It is in the sampler's or transferee's view, after being in his/her physical possession;
- It was in the sampler's or transferee's physical possession and then he/she secured it or placed in a designated secure area to prevent tampering.

Routine Custody

For most projects the FDEP Laboratory provides sampling containers to field personnel. The sample chain of custody begins prior to shipping empty containers to the field personnel. Prior to shipping, the lot numbers of containers are documented on the Sampling Kit Packing List, e.g., Figure 4.5, and in the LIMS. The Packing List is signed by the lab technician and shipped with the containers. Samples are collected by field personnel utilizing procedures identified within their field QA Plans. If sample bottles are provided by the Central Laboratory, custody begins at the point of sample kit preparation. After collection, the samples are shipped to the Central Laboratory by common carrier or are hand-delivered by the field staff. See SOP LB-015, *Standard Operating Procedure for the Packing and Shipping of Sampling Kits, and* SOP HG-015, *Preparation of Sampling Kits for the Preparation of Trace Level Mercury Water Samples.*

Legal chain of Custody

For legal chain of custody samples, a chain of custody form is used in addition to, or instead of, a sample submittal form although the submittal form can also provide legal chain of custody information. See examples of the chain of custody record in Figure 4.6. For any given sampling event, custody Records are kept with the other field paperwork in the event folder at all times until the analysis is complete. A copy is sent with the final report to the customer while the original is kept in the section file with the report. At the customer's request, a copy of the internal custody records (from the LIMS and laboratory log books) can also be sent. See SOP LB-023, *Standard Operating Procedure for Documenting Evidentiary Chain of Custody within the DEP Laboratory*, for complete details.

Sample Custody Policy

See section 5.8 under Handling Samples and Test Items and SOP LB-016, *Laboratory Support Section Receiving Room: Standard Operating Procedure for the Receipt and Log-in of Samples by the DEP Central Laboratory*

Once a sample is logged in, a custody log is automatically created for that sample in the LIMS. This log tracks the sample's movement throughout the lab, from sample receipt to sample disposal. See an example of the laboratory custody log in Figure 4.7. When a sample is removed from the sample storage area, the custody is transferred from the storage area to the user. This transaction is recorded in the custody log by date, time and user. Each LIMS user is issued a unique ID card with a bar code with their User ID. Bar code scanners are located throughout the laboratory. When users wish to check out or transfer samples, they scan their ID card, which

transfers their unique user ID to the custody record. As each sample has a bar code label, the user can easily check out samples by using the bar code scanner. In the event the network or LIMS is down, the user can also manually record their user ID and use a logbook for recording sample custody.

When users are finished with samples, they must return them to their designated storage locations for check-in and secure storage. After samples have been analyzed and final analysis reports are issued to the customer, samples are either disposed of properly (see Table 4.5, *Waste Disposal Procedures*), returned to the client, or (in the case of legal samples) stored until the client approves disposal or transfer of the samples. For routine sample disposal the technician scans the samples in the LIMS. The sample custody record notes the date, time and identity of the person disposing of the sample. While records of sample login, sample check-in/check-out, sample transfers between analysts and sample disposal are recorded electronically in the LIMS custody log, movements of other samples (extracts, digestates, etc.) are documented in various digestion, extraction and analysis logbooks. All logbook records are dated and initialed by the person(s) who carries out the task. Any errors in the documents are struck through with a single line and marked with the person's initials. See examples of biology logbooks in Figure 4.8 and chemistry logbooks in Figure 4.9 .

Inter-laboratory Custody

See section 4.5 for a description of custody for samples sent to a sub-contract laboratory.

Laboratory Information Management System (See SOP LB-009, *Standard Operating Procedure for Validation of LIMS Software Development or Modifications that Affect the Calculation/Assessment of Analytical Results)*

The laboratory uses a custom Oracle based LIMS using client-server technology. The main server is maintained by the Office of Technology and Information Services. The database applications and client hardware are maintained by the Laboratory Support Section.

To gain access to the LIMS, users must provide valid network and LIMS usernames and passwords. They are then presented with a menu containing only those functions required to perform their job duties, based on the security access level assigned to their unique username. For example, only the Program Administrator, Environmental Administrators, Environmental Managers and Chemist Administrators can change any part of an authorized job or change the sample information and its data. Any changes made are documented by the person's name and dated automatically in the LIMS records. When all the jobs within a LIMS event are authorized for release, the event analysis report is reviewed and certified in LIMS (see SOP LB-025). The review process is described in section 5.10.2.

The LIMS software is modified on a continuing basis by the Bureau's Support Section. Revisions of the code are documented with each application. Requests for code changes, additional functionality or interface enhancements are logged in to a web-based Help Desk application. They are prioritized according to the potential for interference with data integrity or workload efficiency. Resolutions to each help desk case are documented and stored in the Help Desk data tables. See SOP LB-009, *Standard Operating Procedure for Validation of LIMS Software Development or Modifications that Affect the Calculation/Assessment of Analytical Results.*

b) Laboratory records are maintained to ensure their availability for a minimum of five years after completion of the laboratory project.

c) Records shall be made available to the accreditation body.

d) FDEP SOP LB-006, *Standard Operating procedures for Records Maintenance and Storage*, clarifies that software and hardware systems will be maintained to ensure that raw data are available for a period of not less than five (5) years after completion of the laboratory project.

e) Procedures for accessing archived records are in SOP LB-013, *Standard Operating Procedure for Archiving/Retrieving Documents and Using the Document Control System*.

f) Copies of pertinent raw and processed data may be maintained in electronic and paper format. Additionally, the laboratory maintains paper copies of all final analytical reports and client custody records. Records retained include:

- A test method description or reference Sample ID
- Instrument identification and a reference to operating conditions
- A reference to associated calculations manual or verification of reported results and QC assessment
- Review process of analytical data
- Method performance and quality control expectations
- Software security, documentation and assessment
- Analysts signatures, initials or electronic identification
- Documentation to support all aspects of sample handling to include preparation, cleanup, incubation periods, weights, and instrument readouts
- Test results and record of responsible parties for laboratory records
- Documentation supporting reagent and standard history
- Calibration and calibration acceptance criteria
- Proficiency Test Results
- Record of demonstration of capability for all analysts

g) All hand written records shall be in permanent ink and any errors in the documents are struck through with a single line and marked with the person's initials. If the reason for the correction is not obvious an explanatory comment shall be provided.

h) In the event that the Bureau of Laboratories ceases operation, all records will be turned over to the Department's Chief Information Officer and all clients will be notified. The Department will continue to maintain client access to electronic and paper records for a period of at least five (5) years after the completion of the laboratory project.

4.14 INTERNAL AUDITS

Internal system audits of the laboratory systems are conducted as described below. In addition, internal performance audits are initiated to help resolve problems and confirm the efficacy of the testing system. System audits of overflow laboratories will be conducted following the pattern of the internal system audits.

4.14.1 The laboratory Quality Assurance Officer conducts internal system audits on select laboratory systems. These internal audit procedures follow these general guidelines:

- Selected systems will be audited annually.
- The QA Officer will conduct the audits.
- The audit will consist of the random selection of a previously reported sample project, tracking of these samples through the system, evaluation of sample results, and a follow-up laboratory audit.
- System components to be audited will include, but are not limited to:

    (i) All documentation associated with sample and data handling, to include linkage mechanism employed between all records for tracking documentation for any sample datum.

    (ii) Use of established, approved procedures as outlined in this QM

    (iii) Proper execution of established procedures.

    (iv) Sample and data handling activities include:

        [a] All sample log-in, trafficking, and log-out

        [b] Sample preparations

        [c] Method calibrations

        [d] Sample analyses

        [e] Data reduction, validation and reporting

        [f] Preventive maintenance and repair procedures

        [g] Standard and reagent preparations and storage

        [h] Sample and waste disposal

[i] Container and labware decontamination

[j] QC management practices and assessment of analytical precision, accuracy and sensitivity.

4.14.2 See examples of typical audit checklists, Figure 4.10. A report will be completed identifying deficiencies and corrective actions to be taken.

4.14.3 Deficiency lists and associated corrective action orders will be formally promulgated to responsible staff. Corrective actions will be taken in a timely manner and all customers shall be notified in writing if the laboratory results were impacted.

4.14.4 Subsequent checks will verify the implementation and effectiveness of the corrective actions. Details are provided in section 4.11.

4.14.5 Additional items:

- The laboratory will notify clients immediately upon the identification of the need for corrective actions that affected the generated data.
- Management shall ensure that effective corrective actions have been instituted within the agreed upon time frame.
- Internal audits as described in this section shall be conducted annually.

4.15 MANAGEMENT REVIEWS

4.15.1 Management reviews shall be conducted according to FDEP SOP LB-010 , *Quality System Management Review* . The Quality Assurance Officer shall prepare a report summarizing the completion of items 4.14.1 through 4.14.5 to include the completion date and responsible personnel for each of the activities. This report will include a summary of corrective or preventive actions, assessments by external bodies, the results of inter-laboratory comparisons or proficiency tests, QC activities, staffing issues or any relevant client feedback or complaints.

4.15.2 Performance reports are generated monthly, semi-annually and annually to track workload performance, efficiency and spending. The reports are maintained by the laboratory and disseminated to laboratory supervisors.

4.15.3 The Quality Assurance Officer shall prepare a Quality System Management Review report on an annual basis.

4.16 DATA INTEGRITY INVESTIGATIONS

See Section 5.2.7 for a description of the data integrity program. All investigations will be conducted in a confidential manner. Investigations will be documented and affected clients will be notified.

5.0 TECHNICAL REQUIREMENTS

5.1 GENERAL

5.1.1 It is recognized that many factors affect the correctness and reliability of the tests that the laboratory performs, including:

- human factors
- accommodation and environmental conditions
- test methods and validation
- equipment
- measurement traceability
- sampling
- handling of test items

5.1.2 The laboratory shall take into account these factors in developing test procedures, personnel training and equipment selections.

5.2 PERSONNEL

5.2.1 All laboratory personnel are required to maintain SOPs that pertain to the work they perform within the laboratory and laboratory analysts must perform initial and continuing demonstrations of proficiency according to Section 4.1.5 (g).

Qualifications for personnel performing specific tasks are based on education, experience and training procedures. All personnel must meet established minimum requirements to perform their assigned tasks for performing tests, evaluating results, certifying results. Personnel will not perform the tests unsupervised without passing minimum training requirements. Position descriptions (PDs) are maintained for each position and contain the minimum qualifications required for employment (see Section 5.2.4).

Managers responsible for interpreting results are knowledgeable of the test procedures, the intent of their use and typical interferences, requirements for their use and the significance of deviations from accepted protocols.

5.2.2 Personnel training shall be conducted according to DEP SOP LB-011, *Laboratory Training System and Records Management*. All analysts and technicians engaged in analytical work are required to complete training in the methodological requirements for assigned analyses on an annual basis.

5.2.3 All tests performed by the FDEP Bureau of Laboratories are conducted by personnel employed by the Florida Department of Environmental Protection. Procedures for subcontracting environmental tests are provided in Section 4.5.

5.2.4 Job descriptions are maintained for each position within the Bureau of Laboratories. Summaries of the responsibilities of the laboratory supervisors/managers may be found in Section 4.1.5(f) of this manual.

The job/position descriptions include:

- Planning and performing tests.
- Evaluation of test results.
- Reporting opinions and interpretations.
- Method modification and development and validation of new methods.
- Qualifications and training programs.
- Expertise and experience required.
- Managerial duties.

5.2.5 FDEP management ensures the competency of all who operate equipment, perform tests, evaluate results and sign test reports. Adequate supervision is provided for staff undergoing training. Personnel performing specific tasks are qualified on the basis of education, training, experience and/or demonstrated skills, as required. See position descriptions, personnel files, SOP LB-011, *Laboratory Training System and Records Management*, and the management descriptions in Section 4.1.5 (f).

5.2.6 Additional Personnel Requirements

Technical Manager Qualifications

Technical managers at the FDEP laboratory will meet all the requirements of the State of Florida Department of Management Services broadband classifications and NELAC standards for education and experience.

5.2.7 Data Integrity Training

All employees of the FDEP Central Laboratory are held to high professional ethical standards in the performance of their duties. All employees are required to read, understand and sign an 'Ethics Statement' attesting to their commitment to honesty and integrity in performance of their duties. In addition, all employees are required to attend an annual ethics training class. Improper, unethical or illegal actions will be dealt with according to the published Administrative Directives of the Florida Department of Environmental Protection found at: http://www.dep.state.fl.us/admin/depdirs/pdf/202.pdf.

a) See SOP LB-012, *Code of Ethics*, for additional information.

b) The annual training includes protocols for reporting ethics issues, providing examples of ethical violations and reviewing the consequences of unethical behavior and resources where additional information can be referenced. The training is updated each year to address current issues. All attendees of the training course are required to sign an attendance sheet verifying that they participated in the course.

5.3 ACCOMODATION AND ENVIRONMENTAL CONDITIONS

5.3.1 Environmental controls in the laboratory are appropriate for the tests being performed. Environmental conditions that can affect test results are listed in the relevant

SOPs. For each area that requires a controlled environment, the conditions are documented. Environmental factors such as light, temperature, ventilation and space are considered to allow tests to be performed safely and effectively.

5.3.2 Environmental conditions are maintained to meet test procedure requirements and are controlled so as not to invalidate test results or increase measurement uncertainty. If it is determined that test results are being adversely impacted by the test conditions, the tests will be terminated, corrective actions instituted and client notification of the impacted data will take place.

5.3.3 The physical location of activities will be such that potential contamination will be minimized.

5.3.4 Access to all laboratories is restricted to authorized personnel and approved visitors. Visitors are supervised at all times.

5.3.5 All laboratory areas will be maintained in a clean and orderly manner.

5.4 ENVIRONMENTAL TEST METHODS AND METHOD VALIDATION

Table 5.1 contains a listing of all chemistry analytes, preparative and analytical methods, matrices, accuracy and precision targets derived from Laboratory Control Samples or method requirements, and MDL's/PQL's. Modifications to methods in Table 5.1 are summarized in Table 5.10 and in the method SOPs.

Table 5.2 contains the listing of all biology parameters and their associated matrices, methods and QA targets. Modifications to methods in Table 5.2 are summarized in Table 5.11 and in the method SOPs.

MDLs are set such that the risk of reporting a false positive is less than 1%. MDLs are determined using the method specified in the Federal Register, 40 CFR Part 136 Appendix B, using laboratory control samples prepared near the estimated detection limit as surrogates to estimate methodological noise for censored methods (e.g., chromatographic methods which censor analytical noise) or, for uncensored methods, using actual method blanks to directly measure methodological noise. Where the possibility exists for significant systematic bias from sample preparation and handling or from the analytical determinative step (typically inorganic analyses), bias is taken into account when calculating detection limits. Published MDLs may be set higher than experimentally determined MDLs to (1) avoid observed positive interferences from matrix effects or common reagent contaminants or (2) for reporting convenience (i.e., to group common compounds with similar but slightly different experimentally determined MDLs). MDLs are determined in a suitable analyte-free matrix when possible. For certain analytes and matrices, no suitable, analyte-free matrix may be available. In those cases, MDLs are determined in the absence of any matrix, but in the presence of all preparatory reagents carried through the full preparatory and determinative steps. LOD verification procedures may be found in SOP LB-031, *Limit of Detection Verification*.

Practical Quantitation Limits (PQLs) are set at 3 to 5 times the reported MDL unless otherwise noted. Because PQL level checks are required, the practicality of the preparation of standards using commercial analytical mixes may dictate to some extent the reported PQL. Generally the PQL is not set at less than 3 times the MDL. However, in some instances, systematic bias (e.g., analyte background in reagents, etc.) necessitates that the reported MDL be elevated to levels that are readily quantifiable. In those instances, the PQL may be set at a level less than 3 times the reported MDL.

Except where specified in individual methods, the QA targets for all inorganic analyses are within the range of 80 - 120 % for accuracy and < 20 RPD for precision, unless laboratory-generated data indicate that tighter control limits can be routinely maintained. This convention was adopted due to the fact that targets set according to historical data are usually less stringent. The organic QA targets are likewise statutory in nature. Warning and control limits for organic analyses are initially set for groups of compounds based on preliminary method validation data. When additional data is available, the QA targets may be reconsidered. All QA targets are routinely re-evaluated at least annually (and updated, if necessary) against laboratory generated data to insure targets continue to reflect realistic, methodologically achievable goals.

The sources for these methods may be found in:

EPA

- *Methods for Chemical Analysis of Water and Wastes;* USEPA Office of Research and Development, Cincinnati, OH, 3/83; EPA 600/4-79-020.
- *Methods for the Determination of Metals in Environmental Samples*, USEPA Office of Research and Development, Washington DC, 6/91, EPA/600/4-91/010.
- *Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW-846*; USEPA Office of Solid Waste and Emergency Response, Washington, D.C.
- *Method for the Determination of Organic Compounds in Drinking Water, Supplement I*, EPA 500/4-90/020, July 1990.
- *Code of Federal Regulations, Title 40, Part 136*; U.S. Government Printing Office, Washington, D.C., July 1993.
- *Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater*; USEPA Office of Water, Washington, D.C., 8/93.
- EPA Quality Assurance Guidance Document 2.12: *Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods.* Volume II, Part II, U.S. Environmental Protection Agency, November 1998.

APHA-AWWA-WPCF

- Standard Methods for the Examination of Water and Wastewater, 19th Edition, 1995 (designated as SM in Table 5.1).
- Standard Methods for the Examination of Water and Wastewater, online Edition (designated as SM in Table 5.1 and Table 5.2).

HRS

- Standard Operating Procedures, Laboratory Services, Florida Department of Health and Rehabilitative Services (designated HRS SOP).

FDEP

- Method for Determination of Petroleum Range Organics Rev. 1, 11/1/1995, Florida Department of Environmental Protection (designated FL-PRO).

Massachusetts DEP

- Method for Determination of Extractable Petroleum Hydrocarbons, 01/1998, Massachusetts Department of Environmental Protection (designated MA DEP EPH).

SOPs address all applicable aspects of the testing procedures to include sample handling, transport, storage, preparation, calibration, test procedures, statistical techniques for evaluating data and measurement uncertainty.

5.4.2 Selection of Methods

The laboratory shall employ published analytical methods or methods that have been recognized to meet the needs of the client and are appropriate for the tests being conducted. Guidance will be provided by the laboratory when there is a question about the test method to be used. The laboratory will notify the client when an inappropriate method is requested.

5.4.3 When a new test method must be developed, validation procedures as developed under the QA Rule, 62-160, FAC, of the Department of Environmental Protection will be followed before it is put into use.

5.4.4 See TNI Standard Volume 1, Modules 4, 5 and 7 for the use of non-standard methods in Chemistry, Microbiology and Toxicology respectively.

5.4.5 See TNI Standard Volume 1, Modules 4, 5 and 7 for the validation of new methods in Chemistry, Microbiology and Toxicology respectively.

5.4.6 For quantitative laboratory measurements, statistical quality control measures are used within the Bureau of Laboratories to estimate the uncertainty associated with

analytical methods. See SOP LB-003, *Estimation of Measurement Uncertainty*, for a complete discussion.

5.4.7 Control of Data

5.4.7.1 Calculations and data transfers are evaluated by a multi-level system detailed in SOP LB-025, *Event Level Authorization Checklist* and SOP LB-026, *Job Level Authorization Checklist* .

5.4.7.2 Tracking changes to the Laboratory Information Management System (LIMS) and software used for data calculations and assessments are detailed in FDEP SOP LB-009, *Standard Operating Procedure for Validation of LIMS Software Development or Modifications that Affect the Calculation/Assessment of Analytical Results* , and SOP LB-006, *Standard Operating Procedure for Records Maintenance and Storage*. Permissions are established for all electronic records to restrict unauthorized access.

5.5 CALIBRATION REQUIREMENTS

5.5.1 Table 5.3 contains a list of instrumentation utilized by the chemistry laboratory for the determination of the test parameters in Table 5.1. All necessary equipment to perform the tests and associated calibrations for the methods in Table 5.1 are under the control of the FDEP Bureau of Laboratories, and described in the lab's SOPs.

Table 5.4 contains a list of instrumentation utilized by the biology laboratory for the determination of test parameters in Table 5.2. Calibration requirements for laboratory and field instruments in Biology are listed in Table 5.5.

5.5.2 All equipment and associated software used by the laboratory shall meet the accuracy requirements and specifications of the test methods before being placed in service.

5.5.3 Supervisors shall ensure that all personnel have received adequate training on the use of laboratory equipment and that manufacturer's instruction manuals are easily accessible.

5.5.4 All equipment and instruments including its software, where applicable, are uniquely identified.

5.5.5 Records are maintained for all equipment and associated software to include:

- Identity of the piece of equipment and associated software
- Unique identifier
- Checks that equipment meet specifications
- Location of the equipment
- Manufacturer's instructions
- Calibration records
- Maintenance logs

5.5.6 All maintenance or repair to equipment is documented in laboratory notebooks. Documentation includes a description of the problem(s), work performed, date and analyst's initials. Sample analyses' plans for instrument failure or maintenance are handled in this order: use backup instrument, delay the analysis if holding time can be met, postpone the sampling event or send samples to the overflow laboratory. Table 4.3 and Table 4.4 present the frequency of preventive maintenance tasks performed to ensure data quality in the chemistry and biology laboratories respectively.

5.5.7 Any equipment found to be unserviceable is tagged with an "Out of Service" tag if it is shared by multiple work groups or will be out of service for an extended period. If the unserviceable equipment is used only by a single work unit and the condition is deemed temporary (a service call has been made and/or the equipment is awaiting repairs by qualified technicians), then the supervisor may elect to notify staff directly rather than to tag the equipment.

5.5.8 All support equipment requiring calibration will be labeled or otherwise documented to indicate that calibration has been performed and when it is due.

5.5.9 The calibration of all instruments will be verified following instrument maintenance.

5.5.10 See the calibration procedures in the associated test SOPs that detail the type of checks and the frequency to verify continued calibration.

5.5.11 Any allowable correction factors, e.g. thermometer calibrations, which require the readout to be adjusted, will be clearly labeled and positioned for easy access by the analyst.

5.5.12 Procedures to gain access and the tracking of changes to the LIMS are addressed in section 4.13.3 (a) under the Laboratory Information Management System. Only authorized personnel are allowed access to the laboratory to avoid tampering with the instrumentation.

5.5.13 Additional Requirements and Clarifications

5.5.13.1 Support Equipment

- All support equipment is maintained in proper working order.
- Maintenance or repair to equipment is documented in a laboratory notebook. Documentation includes a description of the problem(s), work performed, date and analyst's initials.
- Calibration procedures for support equipment are found in the Preventive Maintenance tables (Table 4.3 and Table 4.4), SOP CM-011, *Gravimetric Analysis: Analytical Weight and Balance Calibration,* and the test SOPs.
- Calibration checks will be conducted against nationally recognized standards.
- Calibrations or verifications will be within the specifications or the equipment shall be removed from service until appropriate repairs can be conducted.
- Correction factors to be applied associated with the results of any thermometers will be clearly labeled and positioned for easy access by the analysts

- See the Preventive Maintenance Tables Table 4.3 and Table 4.4 for support equipment checks
- Auto-Pipets are calibrated monthly according to SOP NU-039, *Calibration of Auto-Pipettes*

5.6 MEASUREMENT TRACEABILITY

5.6.1 This section of the ISO standard is not applicable to environmental measurements.

5.6.2 This section of the ISO standard is not applicable to environmental measurements.

5.6.3 Reference Standards and Reference Materials

5.6.3.1 Reference Standards

A summary of standard sources, preparation frequency, storage and traceability is given in Table 5.6.

5.6.3.2 Where possible, Reference Materials will be traceable to NIST or EPA .

5.6.3.3 Intermediate checks

Continuing calibration checks will be conducted according to the technical SOPs, http://www.dep.state.fl.us/labs/library/lab_sops.htm.

5.6.3.4 Standards are stored as specified in Table 5.6.

5.6.4 Additional Requirements and Clarifications

5.6.4.1 The laboratory is a participant in TNI's Performance Evaluation Study, the RCRA Laboratory Evaluation Program, and the USGS Standard Reference Sample Project. The QA Officer manages the performance testing program.

5.6.4.2 All reagents and solvents must be dated upon receipt and again at opening. In addition to this, a computerized inventory program has been implemented to keep track of information on all reagents, solvents and chemicals. Table 5.7 lists location and storage of reagents used by the chemistry and biology laboratories. The preparation of laboratory standards from primary stock standards, neat standards or reagents are documented in laboratory notebooks or the Standard Preparation Tracker module of the LIMS.

Additional documentation can be found in the relevant test SOPs and (for example) the following SOPs: SOP HG-012, *Mercury Group Standards Preparation Procedures*, SOP LC-039, *HPLC Standard Preparation*, SOP NU-041, *Reagent Preparation Procedures for general chemistry and nutrients*, SOP NU-042, *Standard Preparation Procedures for general chemistry and nutrients* and SOP SV-009, *BNA Standard Preparation.*

5.7 COLLECTION OF SAMPLES

The FDEP Chemistry Laboratory does not provide field sampling services. The laboratory does provide sample containers (sampling kits) to its customers on request. However, the FDEP Biology Laboratory provides field sampling services. Table 5.8 lists the sampling capabilities and associated sampling equipment of the Biology Laboratory.

5.7.1 The Biology Section follows the protocols described in the Department's Statewide SOP for Field Activities. All FDEP Field SOPs referenced in this document are available on the Department's Internet site, http://www.dep.state.fl.us/labs/bars/sas/qa/sops.htm. They include:

FS 1000 *General Sampling Procedures*
FS 2000 *General Aqueous Sampling*
FS 2100 *Surface Water Sampling*
FS 2200 *Groundwater Sampling*
FS 2300 *Drinking Water Sampling*
FS 2400 *Wastewater Sampling*
FS 3000 *Soil Sampling*
FS 4000 *Sediment Sampling*
FS 5000 *Waste Sampling*
FS 6000 General Biological *Tissue Sampling*
FS 7000 General *Biological Community Sampling*
FS 8100 *Contaminated Surface Sampling*
FS 8200 *Clean Sampling for Ultra-Trace Metals in Surface Waters*

Sampling details such as the choice of container materials, sample volumes, preservation techniques, and holding times for analytes are described in DEP SOP FS 1000, *General Sampling*.

5.7.2 Deviations from documented sampling procedure are documented in the field as comments on the submittal form which is part of the chain of custody for the samples. Figure 4.6 shows examples of the submittal form used during the collection of chemical and biological samples. Depending on the nature of the deviation, the deviation is recorded as a non-conformance and a non-conformance report is included with the sample report. The sample data will be suitably qualified by the laboratory to accurately reflect the nature of the deviation.

5.7.3 Documentation procedures for recording relevant data during sampling are described in field SOP FD 1000, *Documentation Procedures*. This SOP, along with other field SOPs is located at http://www.dep.state.fl.us/labs/bars/sas/qa/sops.htm.

5.7.4 All submittal forms include the date and time of sample collection. Deviations from documented sampling procedure are documented at collection as described in section 5.7.2.

5.8 HANDLING SAMPLES AND TEST ITEMS

5.8.1 See Section 4.13.3 (a) and SOP LB-015, *Laboratory Support Section: Standard Operating Procedure for the Packing and Shipping of Sampling Kits.*

5.8.2 See Section 4.13.3 (a) and SOP LB-016, *Laboratory Support Section Receiving Room: Standard Operating Procedure for the Receipt and Log-in of Samples by the DEP Central Laboratory* for identifying samples upon receipt and movement through the laboratory.

5.8.3 See Section 4.2.4 for procedures associated with any non-conformances that may affect the integrity of the data. See SOP LB-016, *Laboratory Support Section Receiving Room: Standard Operating Procedure for the Receipt and Log-in of Samples by the DEP Central Laboratory,* Section 3, for sample receiving and documentation of non-conformances associated with sample receipt.

5.8.4 Sample location can be tracked from most workstations in the laboratory and can be changed if the sample is relocated. Samples are stored in a number of different areas, separate from all standards and reagents:

(a) air conditioned room: metal samples and others not requiring refrigeration.

(b) walk-in refrigerator 5: organic samples (e.g. BNA, pesticides)

(c) walk-in refrigerator 4: nutrients

(d) double wide refrigerator 6: nutrients

(e) incoming sample refrigerator: temporary storage for temperature sensitive biology samples awaiting login

(g) double door refrigerator 1: soils/sediments

(h) triple door refrigerator 7: nutrients and VOCs

(i) triple door refrigerator 8: soils/sediments

(j) walk-in bio-freezer: biological tissue and other samples requiring freezing

(k) walk-in bio-refrigerator: biology samples that require refrigeration

(l) flammables storage refrigerator 9

(m) mercury clean lab

(n) Biology

(o) three double-door refrigerators, ERLN01, ERLN02 and ERLN03 for emergency sampling events

5.8.5 Additional Requirements – Documentation

For both chemistry and biology, samples of a similar nature submitted together from one collector are aggregated into a single event. Event identification numbers are unique and have the format Customer Name -YYYY-MM-DD-xx where YYYY-MM-DD is the date the event

is created and xx is an accession number that is reset to -01 each day and incremented as events are logged in. Within each event, samples are grouped by analyses into jobs. All final review and reporting of data to submitters is event specific.

Additionally, each sample is assigned a unique sample identification number of the format LLL-YYYY-MM-DD-xx-yy where LLL-YYYY-MM-DD-xx is the job identification number and yy is an accession number between -01 and -99. LLL represents the lab location (TLH is for Tallahassee). YYYY-MM-DD represents the day the samples are logged into the LIMS. The LIMS also maintains an additional internal accession number (with bar code) for each sample. At log-in, a bar coded label corresponding to the sample's unique laboratory identification number is printed and placed on each sample bottle.

All log-in information is cross-checked by a second log-in technician, after which, event login is authorized in the LIMS. The event folder is placed in a central location to await the final report.

Biology

The sample station, replicate number (if any), date, and analysis type form a unique identification for each sample. Each sample container is labeled with this information plus preservation type (if any) using waterproof markers. Field and laboratory sheets use the same nomenclature. Time of sampling is marked on the submittal form to document sequence of sampling. Laboratory bench sheets are filled out using pens with waterproof black ink. Data submittal forms are filled out using pens with waterproof black ink if conditions allow, or dark, soft-lead pencils if conditions are wet.

Algal Growth Potential/Limiting Nutrient Bioassays, , Physical-chemical parameters, Phytoplankton, Toxicity Bioassays

Sample containers are identified with the site name, station number, replicate, date sampled, and time collected. The sample submittal form (Figure 4.6) is completed, including time of sampling as well as any physical-chemical parameters. Habitat assessments (FDEP SOP forms FD 9000-04 through FD 9000-06) and Physical-Chemical Characterization Field Data Sheet (FDEP SOP form FD 9000-03) may be completed.

Chlorophyll-a, Microbiology, BOD, Grain-size analyses

Refer to SOP BB030, *Sample Custody, Preparation Labels, and Worksheet Instructions for Bench Biology Samples*.

Benthic macroinvertebrates

Sample containers are identified with the site name, station number, replicate, and date collected. This information is written directly on the Whirlpak bags used for holding Hester-Dendy samplers. Physical-chemical parameters, associated site information, and substrate sampling information (for Stream Condition Index samples) are recorded on the Physical-Chemical Characterization Field Data Sheet (FDEP SOP form FD 9000-03) and Habitat Assessment data sheets (FDEP SOP forms FD 9000-04 and FD 9000-05).

Periphyton

Sample containers are identified with the site name, station number, and date collected. Physical-chemical parameters, associated site information, and substrate sampling information are recorded on the Physical-Chemical Characterization Field Data Sheet (FDEP SOP form FD 9000-03) and Habitat Assessment data sheets (FDEP SOP forms FD 9000-04 and FD 9000-05).

5.8.6 Sample Acceptance Policy

Samples arriving at the FDEP Central Laboratory are evaluated at the time of receipt. The samples must meet certain requirements in order to be processed in the laboratory. If some of the requirements are not met, the customer is notified of the discrepancy by the receiving staff and a Non-conformance Report (NCR) is created. For Biological samples, receiving room staff contact Biology Section staff with notification of the types of samples received and that they need to be picked up.

The Receiving staff member inspects the samples for damage and sustained holding conditions and, if anything is improper, notes it on the Log-in Checklist. For short hold biology samples, the samples are picked up by biology staff and checked out using the LIMS system to establish custody. See SOP BB-030 for details. Samples must meet the following requirements or they will be subject to rejection:

- The labels and writing on the containers must be waterproof so that the containers can be correctly identified upon receipt at the lab.
- There must be a unique identifier on each container (field ID/test ID combination).
- The information on the submittal form must coincide with that on the containers. Examples of the submittal form supplied by the Central Laboratory is given in Figure 4.6. The minimum information required includes:

    a) a unique sample location/field ID combination

    b) the date and time of sample collection

    c) the collector's name

    d) a LIMs Request ID (contains customer/project information)

    e) a sample matrix

    f) the analyses requested

- The containers must be preserved appropriately.
- The samples must be collected in the appropriate type of container.
- There must be sufficient volume for analysis.
- The samples must be shipped/delivered to the lab in order for the laboratory to have sufficient time to meet analytical holding times.

If the information provided is insufficient to correctly process the samples an effort is made to reach the collector by phone. If the information cannot be obtained in a timely manner, the samples are subject to rejection.

5.8.7 Sample Receipt Protocols

5.8.7.1 At the time of receipt, log-in technicians check the temperature of the coolers. The log-in technicians verify the submittal form information against the sample bottles; any discrepancies are resolved. The pH of preserved samples is checked by technicians prior to sample login. The sample is checked for sufficient volume. Sample integrity such as improper temperature and pH preservation, insufficient volume, leaking or broken bottles etc., are entered into a non-conformance report in the LIMS as well as documented on a Log-in Check form with the log-in technician's initials. The VOC vials are checked for the presence of bubbles by analysts in the VOC laboratory. Trace level mercury and methylmercury water samples have special handling and custody procedures. Details of these procedures are described in SOP LB-016 and SOP HG-001.

5.8.7.2 See section 4.2.4 for procedures associated with non-conformances that may affect data quality.

5.8.7.3 The samples are logged into the LIMS with all the information listed above from the submittal form plus the following:

- STORET station number or NPDES number (if available)
- field parameter values (if available)
- work module number (if available)
- mode of sample delivery (e.g. common carrier, Federal Express, etc.) and delivery date
- field comments (if available)
- lab comments (if available)
- sample storage location in the laboratory
- name of the log-in person is stored in LIMS automatically, with the time and date of sample log-in.
- bottle lot number (manufacturer's lot number on bottle)

Samples are assigned to analysts or technicians by generation of assignment worksheets or backlog reports as appropriate. As the samples move through the laboratory, they and the individual analyses assume the following status designations automatically or when manually flagged by authorized personnel:

- **W**- awaiting preparation (i.e., digestion or extraction) = appears automatically at log-in for samples with analyses that require sample preparation
- **V**- available for analysis = appears automatically at log-in for samples that do not require sample preparation or appears when the completion of sample preparation is flagged by the technician
- **C**- analysis and data entry complete = changes from 'V' automatically when data are entered into the LIMS
- **A**- results authorized for release = changes from 'C' when data are authorized
- **X**- canceled

Sample holding times relating to sample preparation or analysis are entered automatically at sample login. The deadline is calculated and updated each day by the computer and printed out on the worksheet or backlog report. Supervisors have the responsibility to ensure that all analyses under their supervision are prepared and analyzed within the holding times. Table 5.12 describes the list of acceptable sample containers, preservation techniques and holding times for samples received by the laboratory. Chemists, biologists and technicians who schedule their work priority from backlog reports have the responsibility to complete their individual work within the holding time. An example of a backlog report is given in Figure 5.1.

5.8.7.4 Copies of the submittal form and any field sheets are scanned into a computer and the file is named using the event ID. The originals of the submittal form, log-in check forms and other related documents are filed together in an event folder labeled with the event name.

5.8.7.5 Sample custody procedures are described in section 4.13.3. Figure 4.6 shows examples of submittal forms used as transmittal forms for routine sample custody and documentation of custody. The information on the submittal form must coincide with that on the sample containers and include elements listed in section 5.8.6.

5.8.8 Legal chain of custody procedures are described in section 4.13.3.

5.8.9 Additional Requirements – Sample storage and disposal

Samples, sample fractions, extracts and digestates are stored according to the storage instructions in the preservation tables FS 1000-4 through FS 1000-11 from DEP SOP FS 1000, *General Sampling Procedures* (http://www.dep.state.fl.us/labs/bars/sas/qa/sops.htm). The manner of storage will prevent cross contamination and will isolate the samples from standards, reagents and food.

Completed samples with jobs authorized (checked from computer) are disposed of. Samples flagged as hazardous in the LIMS are disposed of according to Table 4.5. Nonhazardous aqueous samples are poured down the sink drain while flushing with tap water. Non-hazardous solid samples are disposed of in the garbage. Disposal of all samples is documented on the LIMS chain of custody record with the technician's name and date.

5.9 QUALITY ASSURANCE FOR ENVIRONMENTAL TESTING

5.9.1 The laboratory has an established quality control program for monitoring the performance of test methods conducted under this manual. The types of quality control (QC) checks and the frequency at which they are performed are listed in Table 5.9 and in the method or test SOPs. A batch of samples consists of 20 or fewer samples that are prepared and/or analyzed in a single run. Saline matrices are batched separately where the test is impacted by high conductivity samples.

The QC objectives will be considered when selecting methods and in evaluating the capability of the laboratory to handle sample loads that meet the QC objectives.

a) Calibration standards are checked against certified reference materials or other independently prepared standards where available.

b) The laboratory is a participant in performance testing studies. See section 5.6.4.1.

c) Replicate analyses are used to evaluate precision. Precision is expressed by the relative percent difference (RPD) to compare duplicate samples/ spikes A and B and is based on the formula:

$$\text{RPD (\%)} = \frac{|A-B|}{(A+B)} \times 200$$

Precision may be determined from duplicate authentic samples, from duplicate LCSs or from matrix spike duplicates. Where RPDs are calculated based on matrix spike duplicates, A and B represent the raw results of the spiked sample (spike plus the background).

d) The accuracy of the test method is assessed in terms of percent recovery for Laboratory Control Samples (fortified blanks) and matrix spikes to evaluate matrix impact.

Percent Recoveries for an LCS is calculated as:

$$\text{\% Recovery} = \frac{SC}{EV} \times 100$$

or for Matrix Spikes as

$$\text{\% Recovery} = \frac{SC - UC}{EV} \times 100$$

where:

SC = Concentration in the spiked sample
UC = Concentration in the unspiked sample (in the case of results below the MDL for the unspiked sample zero is used as the concentration)
EV = Expected value

5.9.2 Quality assurance targets (QATs) for each QC check are defined in terms of relative precision (P) or accuracy (A). Analyte concentrations associated with each QC check are defined as high, mid, or low, depending on what range of the calibration curve the check concentration falls.

A statistical program, written in Borland Delphi and integrated with the LIMS, handles the calculations of mean and standard deviation and also calculates warning and control limits for QC elements. QC data are uploaded from electronic QC analysis tools to the LIMS for storage. The statistical program looks at historical data and at a specified frequency, re-calculates accuracy and precision acceptance limits for a specific sample matrix, instrument

type and QC element. Warning and control limits are calculated when at least seven or more data points are available.

They may be updated when:

(a) a minimum of at least 7 new data points are available;
(b) significant changes are made to the instrument or analytical method;
(c) they have not been updated in the last twelve months.

The user selects at least 7 new, or the most recently generated data points (accuracy, precision or MDL values). The program calculates warning limits for these elements based on approximately two standard deviations from the mean and control limits based on approximately three standard deviations from the mean. In general, the laboratory utilizes method or laboratory defined warning and control limits for reporting data (i.e., statutory control limits). Those statutory limits may be modified utilizing statistical information collected over time. The precision and recovery data are used for the diagnosis of analytical problems. For laboratory parameters, calculated statistical control limits are used as criteria to accept or reject data only if they are more stringent than the criteria listed in Table 5.1.

The formulae used for the calculation of standard deviation, mean, upper and lower control and warning limits are shown below. (Reference chapter 6 of "*Handbook for Analytical Quality Control in Water and Wastewater Laboratories*" - *EPA 600/4-79-019, March 1979*).

(a) Standard deviations are calculated based on the formula:

$$Sp = \sqrt{[\sum_{i-1}^{n} P_i^2 - (\sum_{i-1}^{n} P_i)^2 / n] / n - 1}$$

Where Sp = standard deviation of the population
n = total number of points in the population
Pi = the value for each point

(b) The mean is calculated as the average of all points:

$$\overline{P} = \frac{\sum_{i=1}^{n} Pi}{n}$$

(c) For recovery, the upper and lower control limits are based on a 99% confidence level.

$$UCL = P + t_{0.99}Sp$$
$$LCL = P - t_{0.99}Sp$$

(d) The upper and lower warning limits for recovery are based on a 95% confidence level.

$$UCL = P + t_{0.95}Sp$$
$$LCL = P - t_{0.95}Sp$$

where $t_{0.99}$ and $t_{0.95}$ are Student's t factors for 99% and 95% confidence, respectively.

Because levels of statistical confidence vary with sample size, a fixed level of statistical confidence is employed that approximates 2 and 3 standard deviations. Those control limits are based on requirements specified in various EPA methods and in EPA's 'Handbook for Analytical Quality Control in Water and Wastewater Laboratories'. The statistical program utilizes a Student's t table, setting warning limits at 95% confidence and control limits at 99% confidence. Those Student's t factors correspond approximately to 2 and 3 standard deviations for 7 collected datum points (~1.9 Sp and ~3.1 Sp, respectively). The advantage of using Student's t factors is that control limits are based on known confidence limits regardless of the number of datum points in the population.

5.9.3 Essential Quality Control Procedures

(a) Standard Quality Controls

See Table 5.9 for standard quality controls to include the following essential controls:

i) Positive and negative controls (Laboratory Control Samples, LCS, and method Blanks)

ii) Controls to evaluate the variability, repeatability of the test (Replicates/Duplicates)

iii) Test method accuracy (calibrations, continuing calibrations, certified reference materials, PT studies and matrix spikes)

iv) Measures to evaluate test method capability

- Detection Limit Studies
- Determination of Quantitation Limits
- Range of applicability

Method Detection Limits (MDL and Practical Quantitation Limits (PQL))

The Method Detection Limit (MDL) and Practical Quantitation Limit (PQL) are defined and used for the same objectives in all analyses. However, because of differences in the nature of various analyses, the calculation procedures vary; nevertheless, the underlying principles and concepts are applied uniformly. Described below are the most representative procedures used in this laboratory. (See Appendix C for specific references.) Details of calculations for each analytical method may be found in the laboratory's SOPs.

- The MDL is defined as the minimum concentration of an analyte that can be measured by the method with 99% confidence of its presence in the sample

matrix. For measurements that always produce an analytical signal in method blanks (e.g., inorganic parameters) , MDLs are usually determined from prepared samples of analyte free matrix containing all preparation reagents. The MDL is established at a level that excludes 99% of the analytical noise, consistent with the intent of 40 CFR part 136 Appendix B. The applicable Student's T value (corresponding to the appropriate degrees of freedom; $\alpha = 0.01$) is multiplied by the measured standard deviation of the population of method blank results. That factor is added to the average method blank value. If the average blank result is less than zero, either zero is used as the average or, if analytical interferences are suspected in playing a role in the measurements, the absolute value of the average method blank value may be used as the average. The latter option has the potential of elevating the MDL to accommodate unknown interferences and should be used with discretion. This protocol, generally consistent with the intent and practice of 40 CFR part 136 Appendix B, avoids reporting method detection limits that are unrealistically low and below or within the population of method blank results.

For measurements that do not produce an analytical signal in method blanks (e.g., organic parameters and some inorganic parameters) MDLs are established by analyzing 7 to 20 replicate reagent spikes fortified at 1 to 5 times the estimated MDL, per 40 CFR part 136 Appendix B. The MDL is set to the students T value (corresponding to the appropriate degrees of freedom; $\alpha = 0.01$) times the resulting standard deviation of the measured concentrations of the replicates.

Note: Tables 5.1 and 5.2 are updated annually so the MDLs used by the laboratory may sometimes vary with those listed in both tables.

- The PQL is the lowest level of concentration that can be reliably achieved within specified limit of precision and accuracy during routine laboratory operating conditions. This laboratory sets the PQLs at 3 to 5 times the MDL depending on the method of analysis and the analyte, unless otherwise specified.

- A System Performance Check is defined as the procedure in which a standard consisting of one or more analytes is introduced into the analytical system to verify its performance (responses, peak shapes, retention times or spectra) meets the minimum acceptable criteria. See SOP LB-031, Limit of Detection Verification.

  Additional details about the lab's procedures for evaluating test method capability can be found in SOP LB-007, *Procedure and Policy for Method and Instrument Validation.*

v) Data Reduction

A LIMS module performs sample and QC calculations (e.g., accuracy, precision and % RSD). This module called the "QC Manager" is also used to upload sample and QC results into the LIMS data tables. Sample and QC result calculations are reduced as follows:

- Results from analyzed sample extracts or digestates are processed by the analytical instruments' PC-based data systems or by the laboratory Chromatography software, based on the method protocols in Table 5.1 and Table 5.2. These raw sample results are electronically downloaded from the analytical instrument to the QC Manager. For those instruments not interfaced to a PC, results from the instruments are manually entered into the QC Manager.

- Sample results and QC results are linked together by batch numbers which are generated by the QC Manager, so sample prep and analysis batches are always identified with their associated QC. The analyst enters pertinent sample prep/analysis data (amount sample digested or extracted, final digestate or extract volume, dilution factors, spiking level/solution used, etc.) and then signals the QC Manager to begin calculations. Examples of typical water and sediment calculations performed follow:

  For water samples: $C\ (\mu g/L) = D\ (\mu g/mL) \times V_f\ (mL) / V_i\ (L)$

  For soil/sediment samples: $C\ (\mu g/kg) = D\ (\mu g/mL) \times V_f\ (mL) / [M_s\ (kg) \times k]$

  where:

  C = Concentration of analyte in sample

  D = Concentration in extract or digestate

  V_f = Volume of extract or digestate

  V_i = Initial volume digested or extracted in L.

  M_s = Mass of sample digested or extracted

  k = Dry weight correction factor

- The resulting sample and associated QC results are reviewed by the analyst, and if deemed acceptable, are then uploaded to the LIMS. Current acceptance criteria (warning and control limits) for each QC element are stored within the QC Manager. If QC results are outside of the current control limits, data are flagged as unacceptable, and the associated sample batch may then be re-submitted for re-digestion/re-extraction and/or re-analysis. Additional information may be found in the individual test SOPs.

vi) All reagents and solvents are dated upon receipt and again at opening. In addition to this, a computerized inventory program has been implemented to keep track of information on all reagents, solvents and chemicals. See SOP LB-030 for additional purchasing procedures. All chemical reagents and standards are checked before use to ensure performance within the appropriate laboratory and test method specifications. Table 5.7 summarizes reagent storage.

vii) Selectivity shall be evaluated by employing method requirements and practices established by the laboratory detailed in the test SOPs to confirm responses to the

analyte. Checks used will include dual column confirmation, inter-element interference checks, retention time windows, mass spectral tuning, method blanks. See Table 5.9 for a summary of the QC checks.

viii) All test conditions will be monitored and documented where required by the method to ensure constant, consistent and documentable conditions.

(b) The laboratory utilizes method or laboratory defined warning and control limits for reporting data. Those limits may be modified utilizing statistical information collected over time. The precision and recovery data are used for the diagnosis of analytical problems. For laboratory parameters, calculated statistical control limits are used as criteria to accept or reject data only if they are more stringent than the criteria. See section 5.3.1 for additional information. QA targets and their use are provided in Tables 5.1 and 5.2.

Each analyst and/or technician is responsible for determining that the results of each analytical measurement have all associated QC measurements and that the acceptance criteria are met and documented according to protocol. The analyst and/or technician is responsible for checking calculations, completing sample preparation, calibration, analysis and instrument logs, and completing all internal custody documentation. All written records and logs must be made using indelible ink and must include the analyst's signature or initials. Any corrections to written records must be made using a single strikeout of the original entry. The corrected entry must be dated and initialed by the individual making the correction. No correction fluid or obliterations may be made to the written records.

Each supervisor is responsible for reviewing this work for completion and correctness prior to authorizing the individual results for release.

The data verification procedures consist of all the QC validations and calculations checks discussed above. In addition, soundness of all data is evaluated by the nature of the sample, the inter-relationship among the parameters and the historical values if available, etc. Any discrepancy or inconsistency will initiate a recheck of data or reanalysis of the sample(s).

5.10 REPORTING RESULTS

5.10.1 Test results are reported accurately, clearly, unambiguously and objectively and contain any method required information, reporting requirements of the TNI standards and requirements of the State of Florida QA Rule, Chapter 62-160, FAC. Reports shall supply the customer all the necessary information for the interpretation of the results.

5.10.2 Test reports shall contain all of the information required in 5.10.2 of the TNI standard.

Each supervisor or workgroup designee is responsible for authorizing the individual analysis results and samples for release. When all the samples within a job are authorized, the managers or supervisors are responsible for reviewing and authorizing the job for release. After all the jobs in an event are authorized, the LIMS automatically generates a list of

reports that are ready to be reviewed and certified. The Program Administrator (or his/her designee) reviews the report in LIMS and evaluates the data using a quality assessment tool, Automated Data Processing Tool (ADaPT). See SOP LB-025, *Event Level Authorization Checklist,* for complete details.

After the review is completed, the report is certified in LIMS. Except for criminal cases, after certification, one copy of the event analysis report is printed, with electronic signature, and a pdf file of the signed report is automatically created and transferred to an ftp directory accessed by the client along with a pdf of the sample submittal form , any associated paperwork received from a subcontract laboratory, and an ADaPT data file. One copy of the final report is retained with the original submittal form in the laboratory. An electronic copy along with copies of the submittal form is provided to the submitting agency. All final sample results with associated QC data are archived together in the LIMS committed database and can be retrieved in the future if necessary.

For criminal case reports the generated printout does not have an electronic signature. The reviewing manager must sign and date two hard copies. One copy is retained by the FDEP Bureau of Laboratories and the second copy is mailed to the client. If any analyses or preparations exceed holding time before completion, the results are automatically qualified. See SOP LB-027, *Standard Operating Procedure for Reporting Qualified Data,* for the laboratory data qualification policies and application of Table 1 of the FDEP QA Rule, Chapter 62-160, FAC.

Results associated with quality control data that exceed acceptance criteria will be qualified with a "J". An appropriate comment will be used to qualify results whenever:

1) batch or sample specific quality control results for an analyte cannot be realistically assessed (e.g., due to excessive analyte levels in a matrix spike);

2) quality control data indicate the uncertainty associated with the measurement(s) is outside acceptable limits;

3) sample matrix presents an unusual challenge to a method or instrument. The decision to qualify a result on these factors is at the discretion of the authorizing supervisor and must comply with FDEP SOP LB-027, *Standard Operating Procedure for Reporting Qualified Data*.

Laboratory reports shall not be reproduced except in full without written permission of the lab. The report will include the page number and total number of pages.

5.10.3 Test Reports

5.10.3.1 Content in addition to the requirements of 5.10.2

a) Comments will include deviations from the test method or any conditions affecting the reported results.

b) Any non-conformances to the procedures in this manual or to the test method will be identified in a report comment or a non-conformance report. See FDEP SOP LB-002, *Non-conformance Reporting System*.

c) See SOP LB-003, *Estimation of Measurement Uncertainty*, for reporting of measurement uncertainty. All reports indicate that uncertainty associated with the analytical results can be estimated from the reported quality assurance results and from published test performance acceptance criteria.

d) See section 5.10.5 below for opinions and interpretations.

e) Additional information required by specific methods and clients will either be provided in the report as deemed necessary or directly to the client.

5.10.3.2 An electronic copy of the report, along with copies of the submittal form is provided to the customer or submitting agency. Sampling records and comments are limited to the submittal forms provided by the sampling party or agency to the laboratory.

5.10.4 Calibration Certificates as addressed in ISO 17025 are not applicable to environmental testing.

5.10.5 Laboratory test data will be qualified according to FDEP SOP LB-027, *Standard Operating Procedure for Reporting Qualified Data*. Any opinions and interpretations of the data will be conducted and documented where appropriate directly with the client.

5.10.6 Analyses performed by a subcontract laboratory will be clearly identified on the test report. See section 4.5 for additional information.

5.10.7 Electronic Transmission of Results

Although the laboratory may transmit data in various electronic formats to clients upon request, the laboratory considers that only the report with a signature represents the official analysis report.

5.10.8 Report contents are uniform and designed to clearly and unambiguously present the required test information to the client.

5.10.9 Amendments to Test Reports and Calibration Certificates

Required amendments to test reports will consist of a recreation of the entire report. The amended report will be identified as such and the original report will be referenced.

5.10.10 Exceptions

All reports are created following the same procedures. Abbreviated reports are not created for any sample analyzed by the laboratory.

5.10.11 Additional Requirements

a) A preparation and analysis log is included with each test indicating the prep and analysis date of each sample. If the activity (preparation or sampling) has a holding time of 72 hours or less the time of the activity will be included.

b) Unless otherwise noted, analytical values for soil and sediment samples are reported on a dry weight basis, and analytical values for waste and tissue samples are reported on a wet weight value. This information is provided in the remarks section of the analytical report.

c) All test components that are not accredited are identified on the test report.

d) Numeric results outside of the calibration range where possible are diluted and re-analyzed. In situations where this is not possible the reported results will be qualified according to established laboratory data qualification protocols. See FDEP SOP LB-027, *Standard Operating Procedure for Reporting Qualified Data*.

APPENDIX A

ROLES AND RESPONSIBILITIES FOR THE CHEMICAL AGENT LABORATORY

The implementation of safety, security, and chemical hygiene procedures is the responsibility of all facility staff. The following subsections describe specific safety and chemical hygiene responsibilities for the Florida Department of Environmental Protection Bureau of Laboratories' Chemical Agent Laboratory. It is the responsibility of all laboratory staff and their managers to know and follow the provisions of this plan. Responsibilities are listed by title.

1.1 Laboratory Director

The Laboratory Director is responsible for ensuring that this administrative practice is followed by all users of ultra-dilute chemical warfare agent (CWA) solutions and that resources and support are provided for the implementation of this plan and the requirements outlined therein. The following tasks are the responsibility of the Laboratory Director:

- Responsible for the health and safety of personnel.
- Responsible to ensure all recognized hazards are promptly addressed.
- Interact with laboratory management and personnel to ensure that DSHP procedures are understood and followed and assistance or resources are provided as needed.
- Serve as a back up to the Agent Manager (AM) by holding a back-up key (primary lock and key entry system) to the UDA standard storage refrigerator in case of the AM's absence.

1.2 Laboratory Manager

The Laboratory Manager (LM) is responsible for the daily operation of the CWA laboratory and daily execution of the Dilute Solution Hygiene Plan as it relates to the laboratory's activities. The LM is responsible for the health and safety of the Chemical Agent Operators (CAOs) during all UDA procedures. The LM also shares responsibilities for development, implementation, review, and support of the DSHP with the Chemical Agent Hygiene Officer (CAHO). The following tasks are the responsibility of the Laboratory Manager:

- Ensure that the Ultra-Dilute Agent laboratory has required safety supplies and equipment necessary to handle or store UDA materials safely.
- Ensure that UDA personnel are familiar with the Dilute Solution Hygiene Plan (DSHP) and routinely follow the requirements and procedures.
- Ensure that safety equipment is checked and ready for use.
- Request and coordinate delivery of UDA reference material from the U.S. Army Edgewood Chemical and Biological Center and Engineering Center (ECBC) with Contracting Officer's Representative (COR) or ECBC Chemical UDA Accountability Officer.

- Document all requests, coordination, and communication concerning delivery of Ultra-Dilute Agents in the Accountability Assessment log.
- Appoint a qualified CAO to perform the dry runs with dilute agents to test Standard Operating Procedures (SOPs) and approve staff readiness.
- Routinely observe CAOs performing UDA SOPs and provide recommendations in conjunction with the CAHO to improve UDA laboratory safety.
- Determine staffing, UDA laboratory access and/or training level needs as necessary and ensure identified training levels are met by the individual.
- Conduct regular UDA inspections of housekeeping, personal protective equipment (PPE), and emergency equipment.
- Ensure that current copies of the CHP, DSHP, procedures, and other pertinent documents are readily accessible for use in the Ultra-Dilute Agent laboratory.
- Ensure that security requirements are met as specified in the Security Plan.
- Perform quarterly accountability audits with the Agent Manager as a formal witness.
- Serve as back-up for the Agent Manager for receipt of Standard Reference Materials (SRMs).
- Serve as a back-up Agent Manager for other functions.

1.3 Chemical Agent Hygiene Officer

The Chemical Agent Hygiene Officer (CAHO) is a UDA laboratory specific position and is an extension of the facility Chemical Hygiene Officer (CHO). It may or may not be the actual facility CHO. The CAHO position is held by an individual who has the knowledge and competence to develop and implement this plan, as qualified by appropriate levels of education, training, and experience. The CAHO also must demonstrate the ability to use appropriate equipment and testing procedures to anticipate, identify, and evaluate health, safety, and environmental hazards, as well as the ability to suggest means for reducing those risks. The following tasks are the responsibility of the Chemical Agent Hygiene Officer:

- Development, implementation, review, and technical support of the DSHP in conjunction with the Laboratory Manager.
- Provide technical, environmental, health and safety guidance to the LM in development and implementation of programs and procedures related to the UDA laboratories.
- Advise and assist in the improvement of Ultra-Dilute Agent laboratory safety procedures, and review and update the DSHP on an annual basis as necessary.
- Responsible for the DSHP with full authority to prepare and enforce safety policies.
- Investigate reports or situations of non-conformance with DSHP requirements.

- Prepare and/or approve UDA training materials for required visitor, Chemical Agent Operator (CAO), responder and remediation training levels.
- Provide and/or approve training level certifications for visitors, staff, managers, and others as appropriate for the assigned duties and level of potential exposure.
- Audit UDA laboratory functions for compliance with the DSHP, Occupational Safety and Health (OSHA) regulations, Resource Conservation and Recovery Act (RCRA) regulations and other requirements for laboratory procedures.
- Determine the level and type of personnel protective equipment (PPE) required for the various UDA procedures, conditions, audits, dry runs, and emergency response for decontamination procedures.

1.4 Agent Manager

The Agent Manager (AM) is responsible for assuring the accurate accountability of the UDA agent reference materials, from receipt and storage of the primary materials from ECBC through usage and disposal. The following tasks are the responsibility of the Agent Manager:

- Serve as the primary contact with the UDA regulatory authorities and is the team leader for compliance surveys, audits and inspections by EPA, ECBC or other bodies.
- Assure accurate accountability of UDA agent reference materials (primary material as received from ECBC) from receipt through disposal, and is responsible for maintaining the security and integrity of stored and in-use primary UDA solutions at all times.
- Sign courier forms for receipt of CWA material, complete the ECBC chain of custody form, and accompany the UDA solutions until they are secure in the laboratory.
- Perform checks for each working day of accountability records and housekeeping in the UDA laboratories. (Note: A “working day” is defined as any day where UDA materials are removed from secured storage.)
- Prepare monthly agent inventory reports and perform and document quarterly accountability audits.
- Serve as backup for coordinator of UDA primary solution delivery in case the Laboratory Manager is not available.
- Hold the keys (primary lock and key entry requirement) to the UDA standard storage refrigerator.

1.5 Chemical Agent Operators

A Chemical Agent Operator (CAO) is anyone that works directly with dilute agent solutions or unknown samples potentially containing chemical warfare agents. Chemical Agent Operators

must be certified on the SOP under which they will be using CWA dilute solutions. The following tasks are the responsibility of the CAO:

- Perform all UDA operations using only the current and approved SOP for which operator certification has been completed.
- Conduct all operations strictly in accordance with the provisions of the DSHP, SOPs, and all applicable regulations, manuals, and directives. Any improvements or alternations must be formally approved by laboratory management before changes to the original SOP operations or procedures can be implemented.
- Observe receipt of UDA materials at the loading dock, transport of the material to the UDA laboratory, and completion of the initial entry in the UDA Accountability log.
- Document the preparation, transfer, and disposal of dilute agent standards of the Chemical Agent in the LIMS Standard Preparation Module.
- Ensure all chemical solutions are properly labeled and stored; this also includes good housekeeping practices within the work area. Responsible for maintaining the security and integrity of the UDA solutions at all times.
- Responsible for tracking the current location of all CWA dilute standards they are using. This includes documentation of transfer to other laboratories for analysis or use.
- Responsible for maintaining security for stored and in-use CWA Dilute standards at all times. Hold the numerical combination to the key pad entry system (secondary lock and key entry requirement) on the UDA standard storage refrigerator.
- Receive chemical agent orientation and safety training, and comply with the accountability procedures in addition to responsibilities under the primary CHP.
- Responsible for informing their supervisor of any factors that may compromise the safe operation of the UDA program.
- Report any accidents, incidents, hazardous conditions, or unusual circumstances to the immediate supervisor, LM, AM, or CAHO/CHO. Any event that could potentially cause or resulted in exposure or injury within the designated UDA laboratory areas must be reported.
- Use Personnel Protection Equipment required by the SOPs in the prescribed manner.

1.6 Inventory Witness

A witness is required when auditing the CWA dilute solution inventories. The witness is selected by the Agent Manager from the list of personnel with training appropriate to enter the CWA Dilute Solution Laboratory. The witness must be independent of the dilute agent operations being audited. The physical inventory witness verifies the status of the chemical inventories by signing and dating the inventory logbook.

APPENDIX B

CHEMISTRY ESSENTIAL QUALITY CONTROLS

1.0 INTRODUCTION

This section details the Quality Controls used by the FDEP Bureau of Laboratories for chemical testing. Adherence to the Quality System detailed in the General Requirements Module will ensure that all the QC checks addressed in this appendix are being followed.

2.0 SCOPE

This section lists the essential quality control procedures performed by the FDEP Laboratories for all testing where applicable. Additional requirements detailed in the test applicable regulations, test or FDEP SOP will also be followed.

3.0 TERMS AND DEFINITIONS

The terms as defined in section 3 of the Quality Systems General Requirements will be applicable to this module.

4.0 METHOD SELECTION

The FDEP Laboratory will only use standard methodologies acceptable to our clients and in compliance with regulatory requirements. Supporting information may be found in section 5.4.1 of the general requirements module.

Test method quality controls, QC outlined in the test SOPs and requirements of this module will be followed for all tests where applicable. If no QC exists in a method employed by the laboratory checks will be instituted from a similar method where available.

5.0 METHOD VALIDATION

5.1 Validation of Methods

a) The laboratory shall validate standard methods by performing LOD and LOQ determinations, evaluating precision and bias and employing and achieving method criteria for checks such as mass spectral tuning and retention time windows. See SOP LB-007, *Procedure and Policy for Method and Instrument Validation.*

b) New methods, non-standard methods and laboratory designed methods and method modifications shall be validated to confirm that the methods are fit for the intended use. The validation procedures shall be conducted according to SOP LB-007 and the requirements of the FDEP QA Rule, Chapter 62-160, FAC.

5.2 Limit of Detection and Quantitation

Procedures shall be documented for each quality system matrix.

5.2.1 All sample processing and analysis steps will be included in the test determination and will be documented. Test methods utilized by the laboratory will provide a LOD that meets the objectives of the analytical project.

(a) The LOD shall be determined for each matrix/technology/analyte by the protocol stipulated in the test method or appropriate regulation. In the absence of this information it will be performed as detailed in Section 5.4 and 5.9.3 of the general requirements module.

(b) The LOD verification will be conducted according to Section 5.9.3 c and SOP LB-031.

(c) A limit of detection study will not be conducted if spiking solutions or QC samples are not available.

(d) The LOD shall be determined in a matrix free of interferences, where available.

(e) The LOD will be performed each time there is a change in how the method is performed or when an instrumentation change impacts the sensitivity of the method.

(f) The LOD shall be verified annually for each matrix, technology and analyte.

5.2.2 Limit of Quantitation

The established Limit of Quantitation (LOQ) shall be the same as or above the LOD. See sections 5.4 and 5.9.3 for the method of establishing the LOQ or PQL. Since FDEP regulations require reporting down to the LOD, annual LOQ verifications are not required.

5.3 Evaluation of Precision and Bias

(a) Precision and bias is evaluated according to Section 5.9.2 in the General Requirements Module. Initial precision targets are established from the demonstration of capability or method validation and limits may be updated as more data is generated.

(b) Procedures for assessing precision and accuracy for non-standard methods are described in in section 5.9.2 of the general requirements module and the QC from the SOPs associated with the individual tests. If there are variations on how the QC is assessed due to the unique nature of the tests they will be discussed in the appropriate SOP. Precision and bias will be evaluated against test method, client or contractual targets and laboratory established targets. Precision and bias are evaluated over varying analyte concentrations defined as high, mid, or low, depending on what portion of the calibration curve the check concentration falls. See Table 5.9 for the concentration ranges of each QC check. The assessment of precision and bias will be done independently for each quality system matrix and each analyte shall be assessed through the entire measurement system.

(c) The range of applicability will be determined as detailed in section 5.9.3.

(d) Method validation protocols as detailed in section 1.5 of this appendix will also be used for precision and Bias assessments.

5.4 Evaluation of Selectivity

All analytical method and checks identified in the associated test procedure SOPs will be used to evaluate selectivity. These checks will include, but are not limited to, mass spectral tuning, retention time windows, second column confirmation, interference checks and method blanks.

6.0 DEMONSTRATION OF CAPABILITY

6.1 General

Demonstrations of Capability (DOC) are documented electronically as detailed in FDEP Bureau of Laboratories SOP LB-011. All supporting data related to the demonstration of capability will be retained and easily accessible.

6.2 Initial DOC

Initial demonstrations of capability are performed for all analytes and methods prior to use of the method and if there are any changes in instrument type, personnel, test method or anytime the test method has not been performed by the laboratory or analyst in a 12 month period.

6.2.1 Records of the initial demonstration of capability shall include at a minimum the required information in this section of the TNI standard.

6.2.2 Procedures for conducting the Initial Demonstration of Capability

(a) The Initial Demonstration of capability shall be performed as stipulated in Volume 1, Module 4 of the TNI standard.

(b) The test shall be repeated for either the failed analyte(s) or all of the parameters of interest when there is a failure of one or more of the established test acceptance criteria.

(c) Repeated failures will trigger corrective actions to remedy problems with the measurement system.

(d) An initial demonstration of capability will be performed whenever an analyte is added to an existing accredited test method.

6.3 On-going DOC

6.3.1 On-going demonstrations of capability are conducted annually (at least once per calendar year) by laboratory analysts by successfully analyzing either:

(a) a blind sample;

(b) a blind performance evaluation sample;

(c) four consecutive lab QC samples (LCS);

(d) an authentic sample that has been analyzed by another trained analyst; or

(e) another acceptable, documented method of demonstrating capability.

6.3.2 Documentation for only one test method is maintained for similar test methods using the same technology. EPA test method 1311 (TCLP) and 1312 (SPLP) are considered similar methods differing only in the leaching solution. For some methods, it is not feasible or practical to include all analytes in the blind performance samples, laboratory control samples or authentic samples. If an analyst is demonstrating on-going capability using one of those samples and an analyte was not added or present in the sample, the analyte must still be reported by the analyst. Acceptability of results for analytes not added or present in ongoing capability demonstration samples shall be based on the supervisor's judgment (either using non-detection as a criterion or, if the amount is judged to be a co-contaminant, based on comparability of results produced by other experienced analysts).

7.0 TECHNICAL REQUIREMENTS

7.1. Initial Calibration

7.1.1 Calibration and standardization procedures, frequencies and documentation protocols for the instrumentation are found in the technical SOPs, http://www.dep.state.fl.us/labs/library/lab_sops.htm

It is the laboratory's policy that method calibration requirements will be followed if more stringent than those described in this manual.

Protocol for Determining the Test Method Range of Applicability:

During the development of new test methods and during initial demonstrations of capability (method validation studies), an evaluation will be made of the dynamic range over which the method is applicable. That evaluation will take into consideration the type of calibration protocol (linear, nonlinear), the change in sensitivity over the tested calibration region, the detection limit of the method and the limit of quantitation. Once a valid range of applicability is established, calibration standards will be used to bracket the range over which quantitation will occur. Results reported from data that were generated outside the determined range of applicability will be flagged as estimates (unless the sample was diluted prior to analysis in order to bring concentrations within the established test method range of applicability).

During the establishment of the test method range of applicability, calibration standards will be prepared and analyzed over the estimated or published range of applicability. If a linear calibration protocol is to be used, either a) the correlation coefficient of the calibration values plotted against their respective response factors must be greater or equal than 0.995, b) the relative response factors (response factor/calibration value) over the range of calibration must have a relative standard deviation of less than or equal to 10% or c) conditions for linearity specified in the applied, published method must be met. If the above conditions are not met, either the linear dynamic range must be decreased until those conditions are met or a non-linear calibration protocol must be used. Whenever a non-linear calibration protocol is utilized, a minimum of 5 calibration points must be defined for a second order fit; a third order fit requires a minimum of 6 calibration points. When using non-linear calibration procedures, loss in sensitivity (Δ response/Δ concentration) can occur

at high concentrations. To ensure that signals are not quantified in regions of poor sensitivity, control standards must be analyzed at the highest point of the nonlinear calibration curve during method validation and must meet the reference method acceptance criteria for calibration. The lower limit of the test method range of applicability is normally established at the method detection limit. The initial demonstration of capability includes establishment of the method detection limit and lower limit of quantitation.

7.2 Continuing calibration

See the SOPs referenced in section 1.7.1 that include both initial and continuing calibration.

7.3 Quality Control

Quality control checks shall be detailed in the test SOPs and QC SOPs associated with the test type. The QC types for the tests and frequencies are summarized in Table 5.9. The QC types addressed are:

7.3.1 Negative Controls

a) Method blanks are analyzed with the same procedure and test conditions as the test samples and are used to assess possible contamination during the sample preparation and processing steps. Corrective actions associated with a contaminated blank will include reprocessing the associated batch samples or qualifying all of the associated prep batch samples according to the procedures given in SOP LB-027, *Standard Operating Procedure for Reporting Qualified Data*.

b) Method blanks are performed at a minimum of one per prep batch and consist of a quality system matrix that is similar to the associated samples that is known to be free of the analytes of interest. In instances when no readily available and economical analyte free matrix can be identified at the levels of detection required to satisfy client objectives laboratory grade water will be used.

c) Method blanks are not applicable to certain tests. See Table 5.9.

7.3.2 Positive Controls

7.3.2.1 The Laboratory Control Sample is taken through the entire preparation and analysis procedure and and the results are compared against established acceptance criteria. Results outside of the acceptance criteria are re-analyzed or qualified according to SOP.

7.3.2.2 Laboratory Control Samples are performed at a minimum of one per preparation batch . Laboratory Control Samples are not applicable to analytes for which no spiking solutions are typically available. See Table 5.9.

7.3.2.3 The LCS is prepared by spiking a known concentration of analyte into a quality system matrix known to be free of the analyte of interest or it may be a media containing a verified concentration of the analyte. The analytes to be spiked will be those specified by the test method or in the absence of this information in the method:

a) The analytes selected will represent the chemistries and elution patterns of the reported components.

b) For multi-component tests the number of analytes spiked will conform to the TNI standard and the laboratory will ensure that all targeted compounds are spiked over a two year period.

7.3.3 Sample Specific Controls

These controls document the effect of the matrix on the method performance and are not a measure of laboratory performance. The results of these control samples are evaluated and documented.

7.3.3.1 Matrix Spike; Matrix Spike Duplicates

a) See Table 5.9 for the frequencies of these checks. Corrective actions for results outside of routine performance specifications include qualifying the impacted sample. See SOP LB-027, *Standard Operating Procedure for Reporting Qualified Data*.

b) The procedure for determining the spiked analytes is the same as for the LCS given in section 1.7.3.2.3 above.

7.3.3.2 Matrix Duplicates

These are sample duplicates that are taken through the entire analytical process – except for in-bottle digestions (e.g. some low-level mercury analyses) where a sample is split into matrix duplicates after digestion. See the frequencies in Table 5.9. These checks are only performed when there is a good chance that the target analyte is present. The RPD of the duplicates is calculated and compared to expected routine performance specifications.

7.3.3.3 Surrogate Spikes

Surrogates are added prior to extraction and are used for all appropriate tests. The surrogates used represent the chemistries of the targeted compounds of the method. Results are compared to method requirements and historical laboratory performance. Corrective actions include qualifying the individual samples when surrogate recoveries are outside of the established range.

7.3.4 Protocols for data reduction are in Section 5.9.3 (a) (v) of the general requirements module and the individual test and supporting SOPs. All data reduction procedures shall be documented.

7.3.5 Reagent Quality, Water Quality and Checks

(a) Reagent grade chemicals shall be used for all tests where the test method does not specify the reagent purity. Reagent purity requirements within the test method shall be followed. All reagents and solvents are dated upon receipt and again at opening. In addition to this, a computerized inventory program has been implemented to keep track of information on all reagents, solvents and chemicals.

(b) Water sources are monitored through the use of method blanks. Corrective actions are immediately taken when blank contamination is attributable to the water source.

(c) Titrant concentrations are verified and documented according to procedures identified in the test method SOPs.

7.3.6 Selectivity is evaluated by following all required checks within the test method and the FDEP test SOP.

7.4 Data Acceptance/Rejection Criteria

7.4.1 Negative Controls – Each method blank is evaluated to determine the impact on the associated sample batch. See the test method SOPs and SOP LB-027, *Standard Operating Procedure for Reporting Qualified Data* for corrective actions and documentation associated with method blank contamination.

7.4.2 Positive Controls – Laboratory Control Samples

a) The results of the LCS is calculated according to Section 5.9 of the general requirements module and compared against established acceptance criteria. The result of the calculation is documented.

b) The protocol for allowable marginal exceedances will be according to this section of the TNI standard. Further details are provided in SOP LB-027, *Standard Operating Procedure for Reporting Qualified Data* .

7.4.3 Sample Specific controls

a) Matrix Spike; matrix Spike Duplicate

Percent recovery from matrix spikes and relative percent difference from the duplicate matrix spikes will be calculated as detailed in Section 5.9 of the general requirements module. The results of these calculations will be documented and compared against established acceptance criteria.

b) Matrix duplicates

Precision is evaluated using the calculation for RPD in Section 5.9 of the general requirements. Results are documented and compared against established acceptance criteria.

c) Surrogate Spikes

The recoveries of surrogates are calculated according to the formula given in Section 5.9 of the general requirements module. Results will be documented and acceptance criteria will be established based on the test method or a documented internal procedure. Results will be evaluated for the effect on individual samples.

7.5 Sample Handling

a) Samples requiring thermal preservation will be monitored to meet the preservation requirements referenced in Section 5.8.7. Samples that are delivered to the laboratory on the same day of sample collection and have not had adequate time to achieve the required temperature shall be considered acceptable if they are received on ice. This will be documented as part of the sample receipt procedure.

b) See section 5.8.6, for the laboratory sample acceptance policy and FDEP SOP LB-016, *Laboratory Support Section Receiving Room: Standard Operating Procedure for the Receipt and Log-in of Samples by the DEP Central Laboratory.*

APPENDIX C

REFERENCES

1. "Definition and Procedure for the Determination of the Method Detection Limit-Revision 1.11", 40 CFR Part 135, Appendix B.
2. "Nomenclature, symbols, units and their usage in spectrochemical analysis II", Spectrochim. Acta B, 33B, 242 (1978).
3. "Limit of Detection: A Closer Look at the IUPAC Definition", Analytical Chemistry 55, 712A-718 A (June 1978).
4. Handbook for Analytical Quality Control in Water and Wastewater. EPA 600/4-79-019. March 1979.
5. Methods for Chemical Analysis of Water and Wastes, USEPA Office of Research and Development, Rev. 3/83. Cincinnati, OH, 3/83; EPA 600/4-79-020.
6. Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW-846; USEPA Office of Solid Waste and emergency Response, Washington, D.C.
7. Method for the Determination of Organic Compounds in Drinking Water, Supplement I, EPA 600/4-90/020, July 1990.
8. Standard Methods for the Examination of Water and Wastewater (designated SM), American Public Health Association, Washington, DC, 1995.
9. Standard Operating Procedures, Laboratory Services, Florida Department of Health and Rehabilitative Services (designated HRS SOP).
10. Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, USEPA Office of Water, Washington, D.C., 8/93.
11. Code of Federal Regulations, Title 40, Part 136; U.S. Government Printing Office, Washington, D.C., July 1993.