SFWMD

SFWMD COMPREHENSIVE QUALITY ASSURANCE PLAN

1998

WRE INVENTORY #370

SOUTH FLORIDA WATER MANAGEMENT DISTRICT

Comprehensive Quality Assurance Plan #870166G
for
South Florida Water Management District
1480-9 Skees Road, West Palm Beach, FL 33411
(561) 686-8800

870166-22
994-McGinnes
SSL

Prepared by
South Florida Water Management District
1480-9 Skees Road, West Palm Beach, FL 33411
(561) 686-8800

Leslie A. Wedderburn, Department Director
Department of Water Resources Evaluation
Date 7/7/98

Maxine Cheesman, Division Director
Water Quality Monitoring Division
Date 7/7/98

Paul R. McGinnes, Division QA Officer
Water Quality Monitoring Division
Date 7/7/98

Linda Lindstrom, Division Director
Resource Assessment Division
Date 7/8/98

Tim Bechtel, Division QA Officer
Resource Assessment Division
Date 7/1/98

Alan Hall, Department Director
Ecosystem Restoration Department
Date 7/6/98

Nick Aumen, Research Program Director
Ecosystem Restoration Department — Date 7/7/98

Alan Steinman, Division Director
Okeechobee Systems Research — Date 7-7-98

Chuck Hanlon, Division QA Officer
Okeechobee Systems Research Division — Date 7.8.98

Patricia Strayer, Division Director
Kissimmee River Basin Division — Date 7/9/98

Brad Jones, Division QA Officer
Kissimmee River Basin Division — Date 7/8/98

Thomas Fontaine, Division Director
Everglades Systems Research Division — Date 7/7/98

Steve Kelly, Division QA Officer
Everglades Systems Research Division — Date 7/7/98

Kathy Pietro, Division QA Officer
Ecologically Engineered Systems Research Division — Date 7/8/98

Terri Bates, Department Director
Regulation Department — Date 7-8-98

Paul Whalen, Division Director
Everglades Regulation Division

7-8-98
Date

William Donovan, Division QA Officer
Everglades Regulation Division

7/8/98
Date

John Morgan, Director
Okeechobee Service Center

7/8/98
Date

Annoesjka Essex, Section QA Officer
Okeechobee Service Center

7/9/98
Date

DEP QA Officer

11-30-98
Date

THIS PAGE DELIBERATELY LEFT BLANK

TABLE OF CONTENTS
QA Plan Elements

FIGURES

TABLES

TABLES (cont.)

APPENDICES

THIS PAGE DELIBERATELY LEFT BLANK

3.0 STATEMENT OF POLICY

This document is a comprehensive quality assurance plan (CQAP) defining sampling and analytical protocols for the South Florida Water Management District (SFWMD or the District). These protocols encompass activities performed for surface water, estuarine systems, ground water, atmospheric deposition, biological tissue and sediment monitoring programs. The Water Quality Monitoring Division Laboratory of the SFWMD is certified by the Florida Department of Health and Rehabilitative Services as an environmental laboratory capable of performing metals, nutrients, microbiology and General Parameter Categories 1 & 2.

This CQAP is intended to be used as a reference, training guide, and statement of acceptable procedures to be used by SFWMD personnel collecting and analyzing samples and evaluating the quality and defensibility of the results obtained. It documents the minimum standards to be complied with for these activities and provides a reference for evaluating the procedures used during the time this CQAP is in effect.

The SFWMD is committed to the use of good QA/QC management practices to produce data of verifiable quality.

THIS PAGE DELIBERATELY LEFT BLANK

4.0 Organization and Responsibility

4.1 Capabilities

The South Florida Water Management District conducts field sampling for surface water, ground water, atmospheric deposition, sediments, tissues and soils. The laboratory is capable of performing analyses for inorganic anions, metals, physical properties, organics (TOC), microbiology and other tests such as chlorophyll, periphyton, and macrobenthic invertebrates.

4.2 Key Personnel

The following are key personnel associated with the collection and analysis of samples.

Department Director, Department of Water Resources Evaluation: Responsible for the allocation of resources throughout the department to meet the needs of the SFWMD for sampling and analytical services and championing the quality assurance program.

Department Directors, Department of Ecosystem Restoration and Department of Regulation: Responsible for the allocation of resources throughout the department to meet the needs of the SFWMD for sampling and supporting the quality assurance program within their respective departments.

Division Directors, Resource Assessment, Okeechobee Systems Research, Kissimmee River Restoration, Everglades Systems Research, Ecologically Engineered Systems Research, Everglades Regulation, Okeechobee Service Center and Field Engineering: Responsible for the allocation of resources, training of personnel, collection of samples and supporting the quality assurance program within their respective division to meet the needs of the SFWMD for sampling services.

Division Director, Water Quality Monitoring (WQM) Division: Responsible for the allocation of resources, training of personnel, collection and analysis of samples, oversees the QA program for the District and reporting of results to meet the needs of the SFWMD for analytical services.

Supervising Professional, WQM Division Quality Assurance Officer: Responsible for assuring that the laboratory and field personnel adhere to the approved methods of sample collection, analysis, maintenance of the Comprehensive Quality Assurance Plan, method validation studies, issuance of new methods, the administration of the internal and external laboratory audits, field audits and the review of legislation pertaining to laboratory quality assurance.

Supervising Professional, Chemist: Responsible for the supervision of the laboratory, review of quality control results, review of data, release of samples, training of personnel and adherence to required quality control procedures.

Senior Chemists: Responsible for the direct supervision of the assigned shift or group, review of quality control results, review of data, release of samples, training of personnel and adherence to required quality control procedures.

Senior Environmental Scientist-Special Projects, WQM: Responsible for overseeing organics and special projects monitoring for the division and the allocation of personnel for ground water sampling.

Staff Environmental Scientists, WQM: Duties include project management, report generation, data review, and collecting samples.

Resource Assessment, Okeechobee Systems Research, Kissimmee Systems Research, Everglades Systems Research, Ecologically Engineered Systems Research, Everglades Regulation, and Okeechobee Service Center Quality Assurance Officers: Responsible for coordination of all project quality assurance plans and quality assurance (QA) reports for the Division, review of quality control results for the projects, conducting field audits and training of division personnel in quality control procedures. The role of QA officer is separate and distinct from all other responsibilities for any specific project.

Supervising Professional, Environmentalists and Hydrogeologists: Responsible for the supervision of the project managers and field operations supervisors, training of personnel, coordination with divisional QA officers for QA/QC issues, development of sampling networks, review of quality control data and analytical results, and development of research projects.

Supervising Professional for Okeechobee and West Palm Beach Units, WQM: Responsible for supervision of the Water Quality Monitoring Division's Okeechobee and West Palm Beach Sample Collection Units, training of personnel in sampling and quality control procedures, design of sampling networks and research projects, review of quality control data and analytical results, and coordination with divisional QA officer.

Senior Technician Supervisors-Scientific, WQM: Responsible for the allocation of personnel and equipment for surface water sampling, training of personnel in sampling and quality control procedures, review of quality control data, review of analytical results for specified projects and sample collection.

Staff Programmer Analyst, WQM: Responsible for the maintenance and integrity of the laboratory information management system, programming computers, testing software packages, and operation of the LIMS.

4.3 Organization Charts

The following charts show the organization of the South Florida Water Management District.

Figure 4.1 is the overall organization of the SFWMD showing the Governing Board, Executive and Deputy Directors and the major departments and offices.

Figure 4.2 is the organization of the Department of Water Resources Evaluation showing the reporting relationships to the Department Director.

Figure 4.3 shows the organization of the Water Quality Monitoring Division through the supervisory levels.

Figure 4.4 shows the organization of the Resource Assessment Division through the supervisory levels.

Figure 4.5 shows the organization of the Department of Ecosystem Restoration through the supervisory levels for divisions conducting sample collection.

Figure 4.6 shows the organization of the Department of Regulation through the supervisory levels for divisions conducting sample collection.

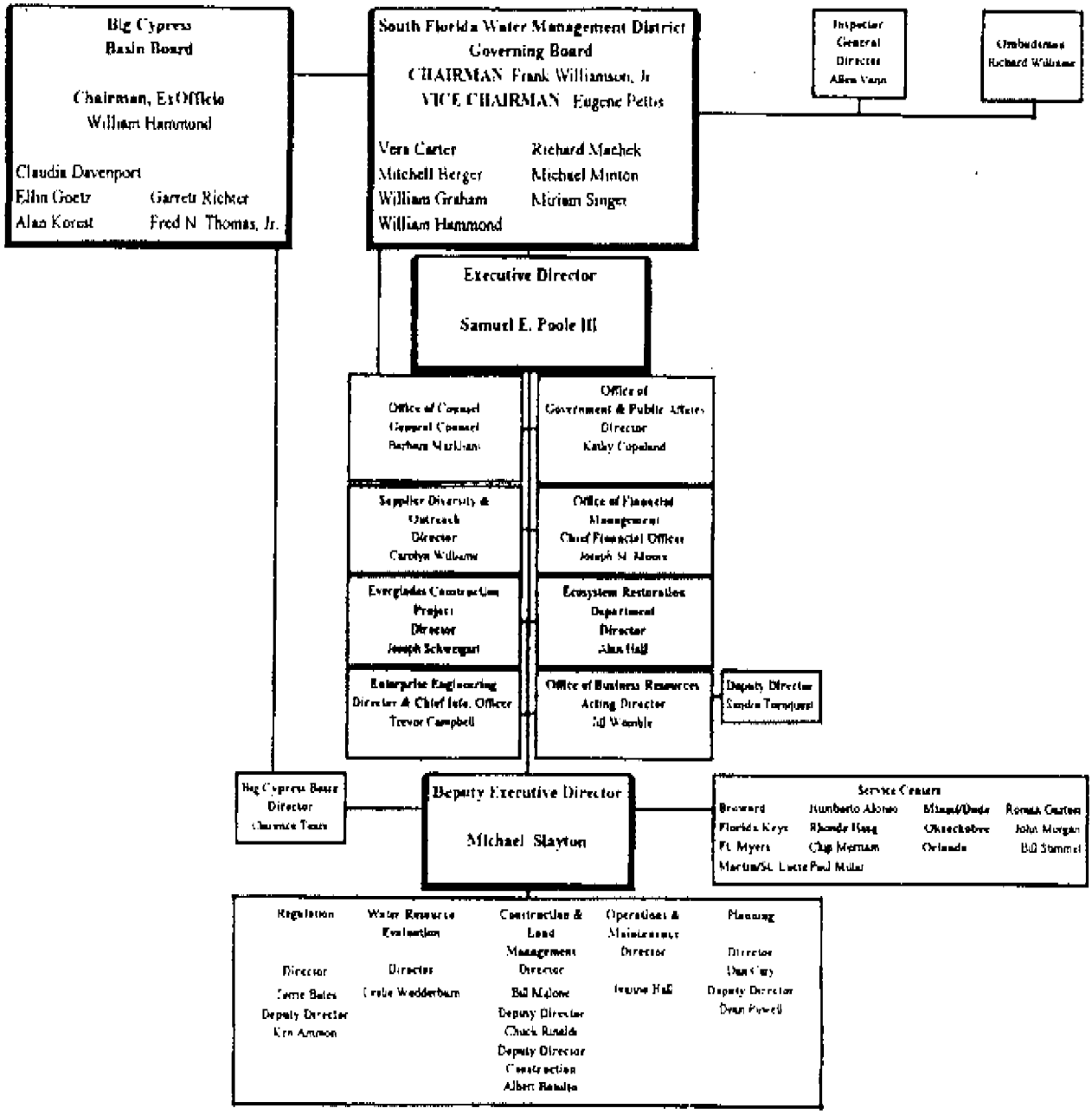


Figure 4.1 – South Florida Water Management District General Organization

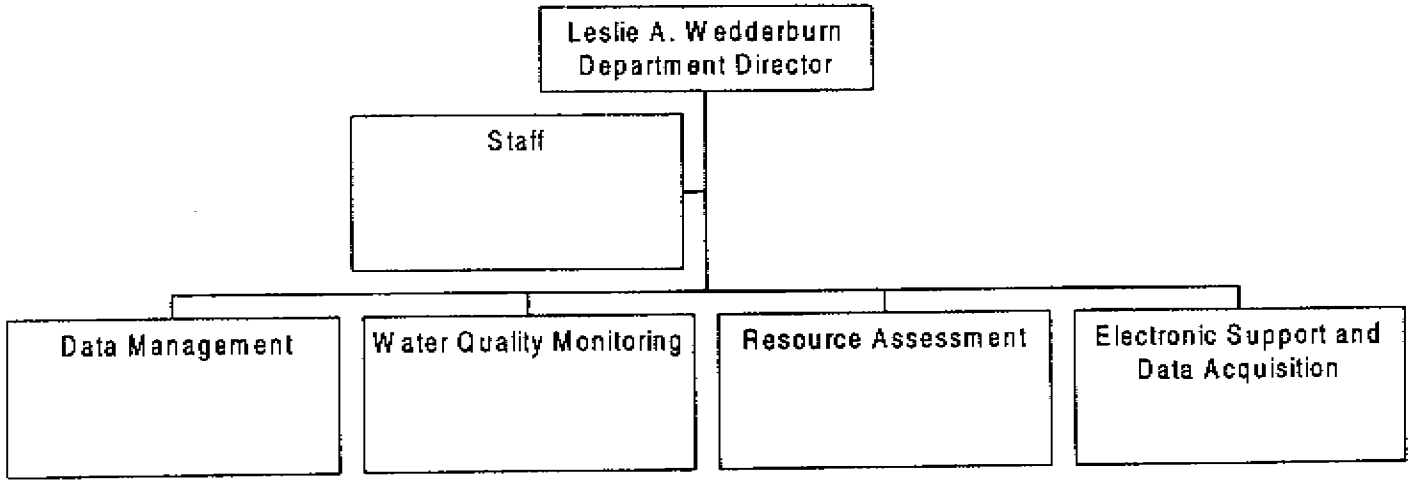


Figure 4.2 Department of Water Resources Evaluation

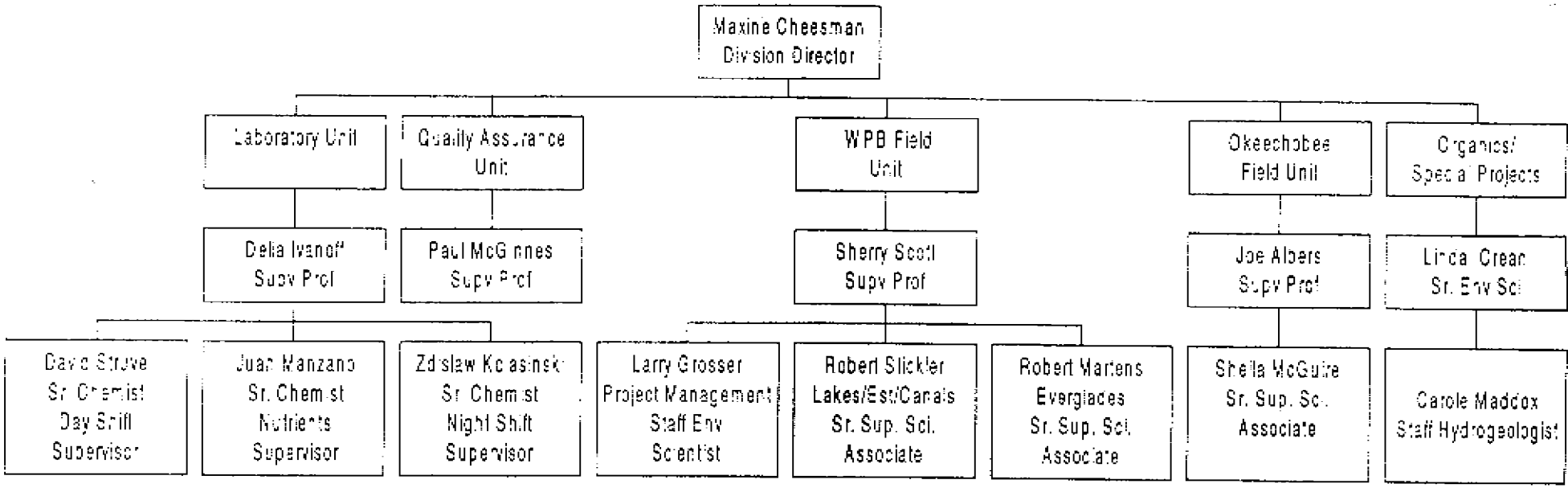


Figure 4.3 – Water Quality Monitoring Division Chart

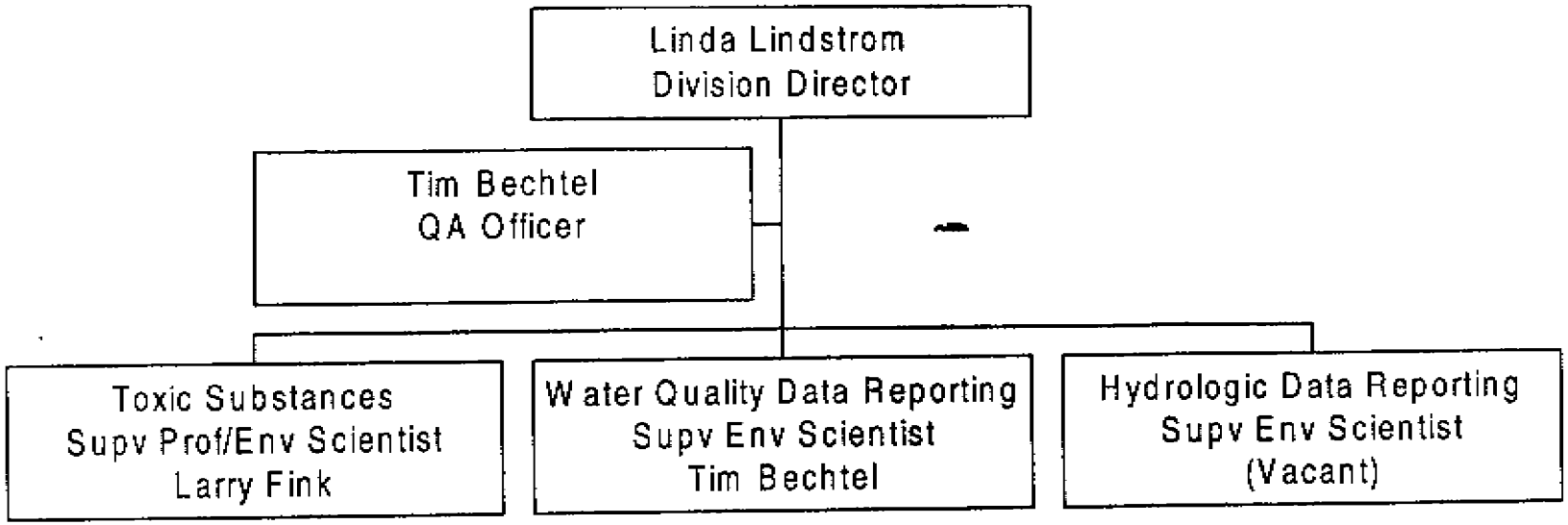


Figure 4.4 - Resource Assessment Division

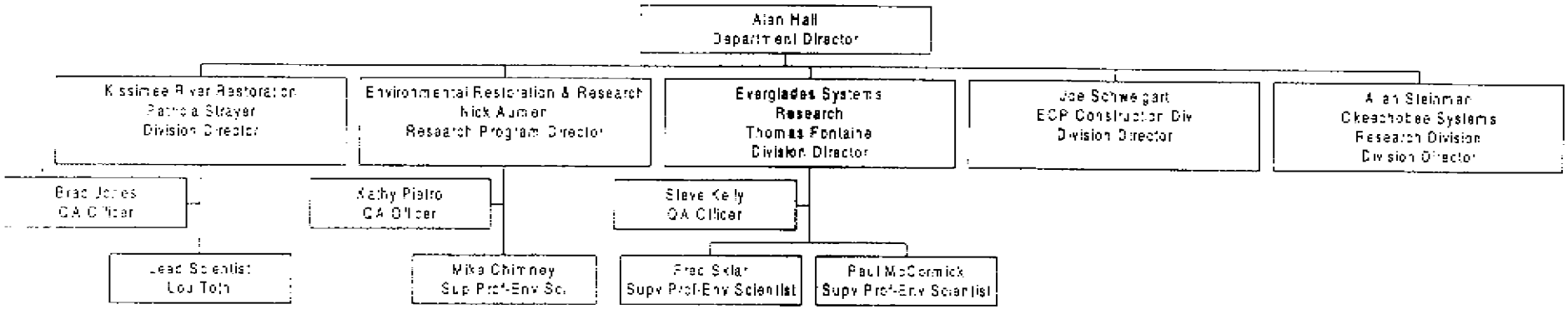


Figure 4.5 – Department of Ecosystem Restoration

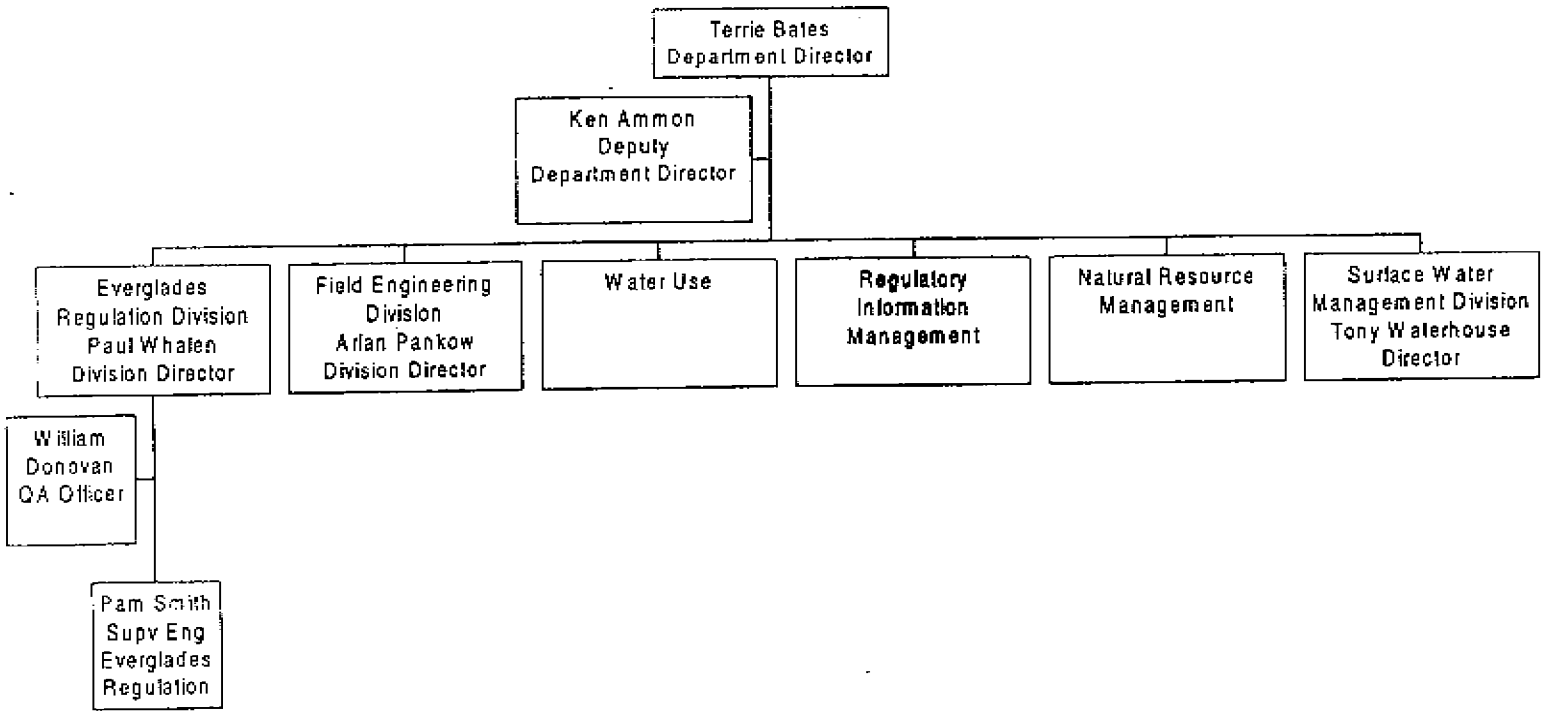


Figure 4.6 – Department of Regulation

THIS PAGE DELIBERATELY LEFT BLANK

5.0 QA TARGETS FOR PRECISION, ACCURACY AND METHOD DETECTION LIMITS

Tables 5.1 and 5.2 present the laboratory quality assurance objectives and Table 5.3 presents the field quality assurance objectives used by SFWMD. The data are generated from historical data collected in the laboratory and field or are target values for the specific parameters.

Table 5.1- QUALITY ASSURANCE OBJECTIVES

Minerals

Component	Matrix(es)	Analytical Method #'s	Precision (% RSD)	Accuracy (% Recovery)	MDL
Specific Conductance	Surface H_2O Ground H_2O	SM2510B	0 - 2.7	NA	0.4 umhos/cm
Residue, Filterable (TDS)	Surface H_2O Ground H_2O	SM2540C	0 - 8.1	NA	13 mg/L
Hardness	Surface H_2O Ground H_2O	SM2340B	NA	NA	5 mg/L
Calcium, Dissolved	Surface H_2O Ground H_2O	SM3120B SM3111B	0 - 2.7	92 – 109	0.2 mg/L
Magnesium, Dissolved	Surface H_2O Ground H_2O	SM3120B SM3111B	0 - 3.2	90 – 110	0.1 mg/L
Sodium, dissolved	Surface H_2O Ground H_2O	SM3120B SM3111B	0 - 2.8	92 – 109	0.2 mg/L
Potassium, Dissolved	Surface H_2O Ground H_2O	SM3120B SM3111B	0 - 3.8	90 – 110	0.1 mg/L
Alkalinity	Surface H_2O Ground H_2O	SM2320B	0 - 7.5	NA	NA
Chloride*	Surface H_2O Ground H_2O	SM4500-Cl B	0 - 1.7	96 – 106	0.50 mg/L
Fluoride*	Surface H_2O Ground H_2O	SM4500-F C	0 - 6.3	90 – 110	0.01 mg/L
Sulfate*	Surface H_2O Ground H_2O	SM4500-SO_4^{2-} B	0 - 1.8	95 – 105	1.0 mg/L

* These methods are not used for NPDES compliance

Table 5.1 (cont.) - QUALITY ASSURANCE OBJECTIVES

Nutrients

Component	Matrix(es)	Analytical Method #	Precision (% RSD)	Accuracy (% Recovery)	MDL (mg/L)
Ammonia Nitrogen	Surface H_2O Ground H_2O	SM4500-NH_3 H	0 -8.9	93 – 113	0.009
Un-Ionized Ammonia	Surface H_2O Ground H_2O	DEP SOP 10/3/83	NA	NA	NA
Nitrate	Surface H_2O Ground H_2O	SM4500-NO_3^- F	NA	NA	0.004
Nitrate + Nitrite	Surface H_2O Ground H_2O	SM4500-NO_3^- F	0 - 2.7	90 – 106	0.004
Nitrite	Surface H_2O Ground H_2O	SM4500-NO_2^- F	0 - 1.7	95 – 106	0.004
Orthophosphate	Surface H_2O Ground H_2O	SM4500-P F	0 - 2.5	92 – 106	0.004
Nitrogen, Total Kjeldahl	Surface H_2O Ground H_2O	EPA 351.2	0 - 7	90 – 110	0.5
Nitrogen, Organic	Surface H_2O Ground H_2O	SM4500-NH_3 H, EPA 351.2	NA	NA	0.5
Total Phosphorus	Surface H_2O Ground H_2O	SM4500-P F	0 - 4.1	90 – 110	0.004

Table 5.1 (cont.) - QUALITY ASSURANCE OBJECTIVES

Demands

Component	Matrix(es)	Analytical Method #	Precision (% RSD)	Accuracy (% Recovery)	MDL (mg/L)
Chemical Oxygen Demand	Surface H_2O Ground H_2O	EPA 410.4	0 – 10	80 - 120	3.0
Organic carbon, Total	Surface H_2O Ground H_2O	EPA 415.1	0 - 4.4	90 - 117	1.0
Organic carbon, Dissolved	Surface H_2O Ground H_2O	EPA 415.1	0 -3.6	91 - 114	1.0
Biochemical Oxygen Demand	Surface H_2O Ground H_2O	SM5210B	0 – 20	NA	2.0
Carbonaceous Biochemical Oxygen Demand	Surface H_2O Ground H_2O	SM5210B	0 – 20	NA	2.0

Table 5.1 (cont.) - QUALITY ASSURANCE OBJECTIVES

Trace Metals

Component	Matrix(es)	Analytical Method #('s)	Digestion Method #	Precision (% RSD)	Accuracy (% Recovery)	MDL (ug/L)
Aluminum total and dissolved	Surface H_2O Ground H_2O	EPA 202.2 SM3120B	SW-3015	0 – 5 0 - 10	80 – 120 80 – 120	4.0 4.0
Antimony, total and dissolved	Surface H_2O Ground H_2O	EPA 204.2 SM3120 B	SW-3015	0 – 7.2 0 – 10	90 – 110 90 – 110	2.2 2.2
Arsenic, total and dissolved	Surface H_2O Ground H_2O	EPA 206.2 SM3120B	SW-3015	0 – 9 0 – 10	85 – 115 85 – 115	1.5 1.5
Barium, total and dissolved	Surface H_2O Ground H_2O	EPA 208.2 SM3120B	SW-3015	0 – 5.4 0 – 7.4	85 - 115 85 – 115	0.2 0.2
Beryllium, total and dissolved	Surface H_2O Ground H_2O	EPA 210.2 SM3120B	SW-3015	0 – 6 0 – 10	90 – 110 90 – 110	0.1 0.1
Cadmium, total and dissolved	Surface H_2O Ground H_2O	EPA 213.2 SM3120B	SW-3015	0 - 2.8 0 – 10	90 – 110 90 – 110	0.3 0.3
Chromium, total and dissolved	Surface H_2O Ground H_2O	EPA 218.2 SM3120B	SW-3015	0 – 3.8 0 – 10	90.2 – 106 90 – 110	0.7 0.7
Chromium (VI), total and dissolved	Surface H_2O Ground H_2O	SM3500-Cr D	SW-3015	0 – 6.5	90 – 110	3.0
Copper, total and dissolved	Surface H_2O Ground H_2O	EPA 220.2 SM3120B	SW-3015	0 – 4.4 0 – 10	91– 108 90 – 110	1.2 1.2
Iron, total and dissolved	Surface H_2O Ground H_2O	SM3111B SM3120B	SW-3015	0 – 2.7 0 – 6.4	85 – 115 94 –109	3.0 3.0

Table 5.1 (cont.) - QUALITY ASSURANCE OBJECTIVES

Trace Metals

Component	Matrix(es)	Analytical Method #(s)	Digestion Method #	Precision (% RSD)	Accuracy (% Recovery)	MDL (ug/L)
Lead, total and dissolved	Surface H_2O Ground H_2O	EPA 239.2 SM3120B	SW-3015	0 – 7.5 0 - 10	85 – 115 85 – 115	0.8 0.8
Manganese, total and dissolved	Surface H_2O Ground H_2O	EPA 243.2 SM3120B	SW-3015	0 – 2.3 0 – 6.2	90 – 110 90 – 110	0.2
Mercury, total	Surface H_2O Ground H_2O	EPA 245.1 SFWMD 2140 1	SM3112B	0 – 9.4	90 – 110	0.2
Mercury, trace level	Surface H_2O Ground H_2O	EPA 1631 SFWMD 2140 2	EPA 1631	0 - 20	80 – 120	0.0005
Nickel, total and dissolved	Surface H_2O Ground H_2O	EPA 249.2 SM3120B	SW-3015	0 - 5.4 0 - 10	90 – 110 90 – 110	0.5 0.5
Selenium, total and dissolved	Surface H_2O Ground H_2O	EPA 270.2 SM3120B	SW-3015	0 - 5.6 0 - 10	90 – 110 90 – 110	1.0 1.0
Silica, dissolved	Surface H_2O Ground H_2O	SM4500-Si D Mod flex		0 – 4.1	92 – 109	1.0
Silver, total and dissolved	Surface H_2O Ground H_2O	EPA 272.2 SM3120B	SW-3015	0 - 3.5 0 - 10	85 – 115 85 – 115	0.5 0.5
Strontium, total and dissolved	Surface H_2O Ground H_2O	SM3111B SM3120B	SW-3015	0 - 5 0 - 10	80 – 120 80 – 120	0.3 0.3
Thallium, total and dissolved	Surface H_2O Ground H_2O	EPA 279.2 SM3120B	SW-3015	0 – 7.5 0 - 10	85 – 115 85 – 115	0.5 0.5
Zinc, total and dissolved	Surface H_2O Ground H_2O	SM3120B SM3111B	SW-3015	0 - 2.7 0 - 10	85 – 115 85 – 115	4.0 4.0

Table 5.1 (cont.) - QUALITY ASSURANCE OBJECTIVES

Miscellaneous Analytes

Component	Matrix(es)	Analytical Method #	Precision (% RSD)	Accuracy (% Recovery)	MDL
pH	Surface H_2O Ground H_2O	SM4500H^+ B	0 - 5.0	NA	NA
Residue, Non-filterable (TSS)	Surface H_2O Ground H_2O	EPA 160.2	0 - 18.3	NA	3 mg/L
Residue, Non-filterable Low Level (LTSS)	Surface H_2O Ground H_2O	EPA 160.2	0 - 10	NA	0.3 mg/L
Residue, Volatile (VSS)	Surface H_2O Ground H_2O	EPA 160.4	0 - 10	NA	3 mg/L
Chlorine Residual, total	Surface H_2O Ground H_2O	EPA 330.4	0 - 10	NA	0.02 mg/L
Inorganic carbon, Total	Surface H_2O Ground H_2O	SFWMD 3150.2	0 - 10	80 - 120	1.0 mg/L
Inorganic carbon, Dissolved	Surface H_2O Ground H_2O	SFWMD 3150.2	0 – 10	80 - 120	1.0 mg/L
Turbidity	Surface H_2O Ground H_2O	SM2130B	0 - 2	NA	0.1 NTU
Color	Surface H_2O Ground H_2O	SM2120B Modified	0 - 0.3	NA	1 Pt-Co unit
Alkaline Phosphatase	Surface H_2O Ground H_2O	SFWMD 3160.1	0 - 7.2	NA	1 nM/min-mL
Chlorophyll	Surface H_2O	SM10200H	0 - 15	NA	1 mg/m^3
Macrobenthic Invertebrates	Surface H_2O Sediments	SM10500	NA	NA	NA

Table 5.2 - MICROBIOLOGY PARAMETERS QUALITY ASSURANCE OBJECTIVES

Component	Matrix(es)	Analytical Method #('s)	Precision (% RSD)	Accuracy (% Recovery)[1]	MDL[2]
Total Coliform	Surface H_2O Ground H_2O	SM9222B SM9221B	0 - 10 0 - 20	NA	NA
Fecal Coliform	Surface H_2O Ground H_2O	SM9222D SM9221E	0 - 15 0 - 20	NA	NA
Heterotrophic Plate Count	Surface H_2O Ground H_2O	SM9215B	0 - 40	NA	NA
Fecal Streptococci	Surface H_2O Ground H_2O	SM9230C	0 - 37	NA	NA

[1] There are currently no established accuracy data for relating analytical results to field populations
[2] Since the ratio of culturable to unculturable cells depends on the sample, a generic MDL can not be determined.

Table 5.3 - FIELD QUALITY ASSURANCE OBJECTIVES

Component	Matrix(es)	Analytical Method #	Precision (% RSD)	Accuracy (% Recovery)	MDL
pH	Surface H_2O Ground H_2O	SM4500H+B	0 - 5	NA	NA
Oxygen, dissolved	Surface H_2O Ground H_2O	SM4500-O G	0 - 20	NA	0.1 mg/L
Specific conductance	Surface H_2O Ground H_2O	SM2510B	0 - 5	NA	50 uS/cm
Temperature	Surface H_2O Ground H_2O	SM2550B	0 - 3	NA	NA
Salinity	Surface H_2O Ground H_2O	SM2520B	0 - 5	NA	0.1 ppt
Turbidity	Surface H_2O Ground H_2O	SM2130B	0 - 20	NA	NA

6.0 SAMPLING PROCEDURES

6.1 Sampling Capabilities

The sampling capabilities of SFWMD are shown in Table 6.1. The EPA Region IV Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual (1996) is the reference used for the development of sampling procedures. This reference is available to all field personnel and is referred to in this document as EPA SOP & QAM.

Table 6.1 Sampling Capabilities by Major Category

Matrix	Parameters
Surface Water	Cations Inorganic Anions Metals Physical Properties Organics Extractable Organics Volatile Organics Microbiology Other (Macrobenthic Invertebrates/Chlorophyll)
Ground Water	Cations Inorganic Anions Metals Physical Properties Organics Extractable Organics Volatile Organics Microbiology
Soil/sediments	Cations Inorganic Anions Metals Physical Properties Organics Extractable Organics Volatile Organics
Atmospheric Deposition	Cations Inorganic Anions Metals Physical Properties
Biological Tissues	Cations Inorganic Anions Metals Physical Properties Organics

Samples are collected from the least to the most contaminated areas whenever possible, depending on time limitations and distance between sites. Fortunately, the majority of the District sampling sites are ambient water sources with little variation in concentration levels within a sampling trip. For ground water sampling for any site being sampled for organic contaminants or where the presence of fuel is suspected, any well suspected of having free product is not sampled. A new pair of disposable latex/PVC gloves is used at each sampling point for all types of sampling. The preferred order of sample collection is: 1) VOC, 2) POX, 3) TOX 4) TOC, 5) extractable organics, 6) total metals, 7) dissolved metals, 8) microbiological, 8) inorganics, 9) turbidity and 10) macrobenthic invertebrates.

6.2 Field Equipment

6.2.1 Surface Water Sampling Equipment

Table 6.2 lists the equipment used for grab sampling of surface water.

Table 6.2 Grab Surface Water Quality Sampling Equipment

Equipment Description	Type of Material	Use	Notes
2.2 liter vertical sampling bottle	acrylic	collection	1
2.2 liter horizontal sampling bottle	PVC	collection	1
3.2 liter horizontal sampling bottle	PVC	collection	1
Plastic bucket	Polyethylene	collection	1
Sample bottles	Polyethylene	collection	1
Filter holder	acrylic/poly-propylene	filtration	1
Filter units, high capacity (0.45 micron)	nylon	filtration	1
0.45 micron disposable filter units filter with pre-filter	polycarbonate	filtration	1
Pre-filter	glass fiber	filtration	1
0.45 micron filter	polycarbonate	filtration	1

Table 6.2 Grab Surface Water Quality Sampling Equipment (cont.)

Equipment Description	Type of Material	Use	Notes
60/150 ml syringe	plastic	filtration	1
Peristaltic Pump	Not Applicable	collection	1
Pump tubing	C-FLEX	collection	1
Sampling boom	PVC pipe with LDPE sample bottle attached	collection	1,2
Bailer	Teflon	collection	3
Lanyard(to support bailer)	Teflon coated Stainless steel	collection	
Gloves(short)	Latex	collection	4
Subsurface sampler	Stainless Steel	collection	3
Hamilton S1000Trachael Syringe	acrylic[1]; teflon[3]	collection	

[1]Not suitable for the collection of organics, extractable organics and VOCs [2]Used to hold tubing at desired depth while collecting sample with the pump. [3] Used to collect VOCs & extractable organics. [4] Does not contact sample

Autosamplers

The following types of autosamplers and equipment are used:

American Sigma Model 700/800/900 Autosampler	Polyethylene	Sample collection	1,4
American Sigma Model 6201 Autosampler	Polyethylene	Sample collection	1,3
Sample bottle 5 gal.	Polypropylene	Sample collection	2
Sample bottles 1 liter	Polypropylene	Sample collection	2
Pump tubing/Intake tubing	Silicon/PVC	Sample collection	2

Notes:[1] Does not contact sample [2] For Inorganic Anions, Cations, and physical properties(Hardness & Solids) with long holding times only [3] Cooled by a refrigeration unit [4] Autosampler bottles usually pre-preserved, unit not cooled; only used for TP when deployed more than 24 hours

Field Instrumentation

1. Hydrolab Scout II
2. Hydrolab IV
3. Hydrolab Surveyor II
4. Hydrolab Surveyor III
5. Hydrolab Surveyor 4
6. Solomat WP803
7. YSI Model 6000UPG Multiprobe - Temperature, pH, Spec. Conductance, DO, Turbidity, EH
8. YSI Model 6920 Multiprobe - Temperature, pH, Spec. Conductance, DO, Turbidity, Depth
9. YSI Model 600XL Multiprobe - Temperature, pH, Spec. Conductance, DO, EH
10. YSI Model 600R Multiprobe - Temperature, pH, Spec. Conductance, DO
11. Turbidimeter
12. Secchi Depth Disc
13. Licor spherical equipment sensors

Navigational Aids

1. USGS Quadrangle maps- for site location
2. Project location maps- for site location
3. WMD low band radio for communication
4. Mark Hurd Aerial Photographs-for site location
5. Global Positioning Systems for site location

6.2.2 Ground Water Sampling Equipment

Table 6.3 lists the equipment used for ground water quality sampling.

Table 6.3. Ground Water Sampling Equipment

Equipment Description	Type of Material	Use	Parameters
Purging Equipment			
1. Centrifugal Pump	Iron/Steel/Rubber	Purge Only	Inorganic/organic
Suction Hose	Flex PVC		
Drop Pipe	Teflon/Stainless Steel		
Check Valve	Teflon		
2. Submersible Pump	Stainless Steel/Teflon	Purge Only	Inorganic/organic
Suction Hose	Polypropylene		
Drop Pipe	Teflon/Stainless Steel		Organic only*
3. Geotech or Masterflex Peristaltic Pump	Aluminum Housing with Stainless Steel Rollers & Plastic Head	Purge	Inorganic/organic
Suction Hose	Teflon		Organic
	C-Flex		Inorganic
4. 2 or 3 Gallon Buckets	Polyethylene	Purge Only	Inorganic/organic
5. Electronic Water Level Indicator	Teflon/Stainless Steel	Prior to Purge	Inorganic/organic
6. Water Level Tape	Steel	Prior to Purge	Inorganic/organic
Sampling Equipment			
1. Bailer System w/top, bottom, filter adapter, control-flow bottom	Teflon	Sample	Inorganic/organic
Lanyard	Teflon Coated Stainless Steel		

* When sampling for organic contaminants, all parts of the apparatus contacting the sample are either teflon, teflon coated, or stainless steel.

Table 6.3. Ground Water Sampling Equipment (cont.)

Equipment Description	Type of Material	Use	Parameters
2. Peristaltic Pump	Aluminum Housing with Stainless Steel Rollers & Plastic Head	Sample	Inorganic/organic
Suction Hose	Teflon	Sample	Organic (except VOCs)
	C-Flex	Sample	Inorganic
Sample Bottles	Glass	Sample	Organic
Bottle Tops with Inflow/Outflow Ports	Teflon	Sample	Organic
3. Gloves	Latex	Sample	Inorganic/Organic
Field Filtration Equipment			
1. Disposable Filters (QED FF-8200 or similar)	Filter: Acrylic Copolymer with Polypropylene Body, 0.45 micron (1.0 micron used for permit compliance metals only)	Filtration	Inorganic
2. Handheld Vacuum Pump	PVC/Tygon Tubing	Filtration	Inorganic(except metals)

6.2.3 Soil/Sediment Equipment

The following equipment is used to collect soil/sediment samples.

Table 6.4 Soil/Sediment Sampling Equipment

Equipment Description	Type of Material	Use
Petite Ponar™	Stainless Steel	Sampling
Scoop/spoon	Stainless Steel	Sampling/Compositing (except VOCs)
Bowl/Tray	Stainless Steel	Compositing/Homogenizing (except VOCs)
Core	Stainless Steel	Sampling
Core	Aluminum	Sampling
Core	PVC	Sampling (except Organics)
Core attachment	PVC	Sampling (except Organics)

Table 6.4 Soil/Sediment Sampling Equipment (cont.)

Equipment Description	Type of Material	Use
Core stoppers	Rubber	Sampling (not for Organics)
Gloves(short & long)	Latex	Sampling

6.2.4 Biological Tissues Sampling Equipment

The following equipment is used to collect biological tissue samples.

Table 6.5 Biological Tissues Sampling Equipment

Equipment Description	Type of Material	Use
Shears	Stainless Steel	Sampling (above ground biota)
Knife	Stainless Steel	Sampling (above ground biota)
Core	PVC	Sampling (below ground biota)
Opaque Bags	Plastic	Sample storage (biota)
Soil Sieve,(5mm mesh size)	Stainless Steel	Sample processing
Gloves(short)	Latex	Sample Collection/Processing

6.2.5 Atmospheric Deposition Equipment

The following equipment is used to collect atmospheric deposition samples.

Table 6.6 Atmospheric Deposition Sampling and Processing Equipment

Equipment Description	Type of Material	Use	Notes
Aerochem Metric Wet/Dry Precipitation Collector	Aluminum	Sample Collection	2
Aerochem wetfall bucket	PVC	Sample collection	2
Snap Lids	High Density Polyethylene	Cover Collection Buckets	2
Plastic bucket	Polyethylene	Sample processing	2
Rubber spatula/tweezers/scoop	Plastic	Sample processing	2

Table 6.6 Atmospheric Deposition Sampling and Processing Equipment (cont.)

Equipment Description	Type of Material	Use	Notes
Gloves(short)	Latex	Sample collection/processing	1
Portable Scale (Mettler SB12001)	Metal/Plastic	Weigh Wet Samples	

Notes:[1] Equipment will not contact sample. [2] For inorganic ions, physical properties, Aluminum and Iron collection/processing only.

6.2.6 Low Level Mercury Sampling Equipment

The following equipment is used to collect low level mercury samples.

Table 6.7 Low Level Mercury Sampling Equipment

Equipment Description	Type of Material	Use
gloves (long & short)	plastic	sample collection
gloves.(short)	latex	sample collection
bags (small & large)	plastic	wrapping bottles/equipment
bottles	Teflon	sample collection
Masterflex pump	Not Applicable	sample collection
pump tubing	Teflon	sample collection
pump tubing	C-FLEX	sample collection
battery (12 volt)	Not Applicable	pump operation
filter units, high capacity, 0.45 micron QED FF8200 or similar	nylon	filtration
screen (100 micron)	nitex	pre-screening
filter holder	Teflon	hold screen
sampling boom	PVC pipe with LDPE sample bottle	hold tubing at desired depth while collecting sample

6.2.7 Macrobenthic Invertebrate Sampling Equipment

The following equipment is used to collect macrobenthic samples. The equipment used will depend on the habitats available in the area.

Table 6.8 Macrobenthic Invertebrate Sampling Equipment

Equipment Description	Type of Material	Use
Hester-Dendy artificial substrates	Tempered hardboard	Sample collection
dip nets	Nylon	Sample collection
Petite Ponar type Sampler	Steel	Sample collection
Ekman type grab sampler	Steel	Sample collection
sieve (U.S. Standard No. 30)	Steel	Processing
glass jars	Glass	Sample Storage
sorting trays	Stainless Steel	Processing

6.2.8 Miscellaneous Equipment

Sample bottles

60 ml polyethylene sample bottles - for inorganics only
125 ml polyethylene sample bottles - for inorganics only
250 ml polyethylene sample bottles - for inorganics only
500 ml polyethylene sample bottles - for inorganics only
1000 ml amber polyethylene sample bottles - for inorganics and chlorophyll only
125 ml teflon or amber glass sample bottles (mercury only)
250 ml polyethylene sample bottles (HNO_3 cleaned, trace metals only)

Note: Sample containers for VOCs, organics, extractable organics, and ultra trace mercury are provided by contract laboratories.

Microbiology sample containers

125 ml pre-sterilized Whirlpaks™

Sample preservation supplies

1. 50% H_2SO_4 in plastic dropping bottle
2. 50% HNO_3 in plastic dropping bottle
3. 50% NaOH, and Zinc Acetate for Sulfide
4. pH strips, 0 - 3 range & 11 - 14 range
5. Safety goggles
6. Acid Spill kit
7. Base Spill Kit
8. 10% buffered formalin
9. 70-80% ethyl alcohol

Note: Preservatives for VOCs, organics, extractable organics & ultra trace mercury are provided by contract laboratories.

Hydrolab field calibration kit

1. Certified pH buffers - 4, 7, and 10
2. Certified conductivity standards - range appropriate for trip sampling sites
3. Ring stand
4. Screwdrivers - straight edge and Phillips
5. Calibration cup with both hard and soft end caps
6. DO membranes and O-rings
7. DO and pH electrolyte solutions
8. Turbidity Standards
9. pH cell caps
10. Technical manual and tools

Coolers with wet ice; sufficient size and quantity to contain all anticipated samples

QA/QC supplies

1. Analyte free water - for field blanks and rinsing equipment
2. Trip Blanks (VOC collection trips only)

Miscellaneous supplies

1. Polyethylene bucket - for inorganic sample processing
2. Disposable latex/PVC gloves, powder free (PVC not for organics or VOCs)
3. Polyethylene trays - for providing clean working areas (does not contact sample)
4. Sample tags with rubber bands or sample labels
5. Waterproof pens
6. Clipboard
7. Field notebook (bound, waterproof)
8. Chemistry field data log sheets
9. First aid kit
10. Watch (with second hand or timer)
11. Personal protective equipment
12. Gloves (latex and/or PVC)

6.3 Decontamination Procedures

All sampling equipment is transported to the field pre-cleaned and ready to use. Cleaning procedures are described in Section 6.3.1 through 6.3.10 and are as required by EPA SOP & QAM, Appendix B. All sample collection equipment and unpreserved containers are rinsed three times with sample water before the sample is collected with the following exceptions: VOCs, bacteriological samples (Whirlpaks™), Total Recoverable Petroleum Hydrocarbons (TRPHs), and Oil and Grease.

Analyte free water is obtained from laboratory or field prep areas which have water purification systems. Analyte free water is generated to provide a source of water in which all interferences and analytes are below detection limits. Field personnel use analyte free water to prepare field blanks, equipment blanks, and for the final decontamination rinse of field equipment. The reliability and purity of the analyte free water is monitored through the results obtained from the equipment and field blanks. The District does not provide a decontamination service to its clients.

6.3.1 Laboratory Cleaning

In the laboratory the sampling equipment is cleaned using the following procedure:

1. Wash all surfaces thoroughly with tap water and phosphate free soap(such as Liquinox). Use a brush to contact all surfaces and remove stubborn debris. Heavily contaminated equipment is disposed of properly.
2. Rinse thoroughly with tap water.
3. Rinse with 10% hydrochloric acid (10% nitric acid for trace metal equipment only).
4. Rinse thoroughly with analyte free water.
5. Rinse thoroughly with pesticide grade isopropyl alcohol (equipment used for organic sampling only).
6. Rinse thoroughly with analyte free water.
7. Allow to air dry completely.
8. Cleaned bailers are wrapped in aluminum foil with the shiny side out (if applicable) or untreated butcher paper, for storage and transportation.

Equipment is properly disposed of if decontamination is not effective. The cleaning procedures used for the field equipment are documented in the equipment logbook and include which equipment was cleaned, the procedure used, and the date and initials of the person performing the cleaning.

6.3.2 Field cleaning for Inorganic Surface Water Sampling

Sampling equipment for nutrients, major ions and physical parameters that is reused from site to site is rinsed twice with analyte free water and then three times with sample at each sample site before the sample is collected. Before the next sampling event, all equipment is cleaned as stated in section 6.3.1. The District is currently in the process of validating this cleaning procedure for trace metals.

6.3.3 Organic Surface Water Sampling

A subsurface vertical or horizontal sampling bottle is used to collect organics in surface water. Samples are collected by immersing the sampling bottle upstream of the person collecting the sample into the surface water body at a depth of 0.5 meters until it is full. The surface sampler does not come into contact with the sample. A pre-cleaned teflon bailer may also be used in the absence of strong currents or where a discrete sample at a specific depth is not required. The bailer is cleaned according to section 6.3.1, and is used only once in the field. The bailer is rinsed three times with sample water before collecting the sample.

6.3.4 Ground Water Equipment

All ground water sampling equipment is transported into the field pre-cleaned and ready to use. Laboratory cleaning procedures for the field equipment are described in sections 6.3.1. The sampling equipment is used only once in the field and transported back to the lab for cleaning. The identification numbers of the sampling equipment used at each well are recorded in the field notes. All ground water sample collection equipment and sample containers are rinsed three times with sample water before the sample is collected, with the following exceptions: VOCs, and any sample bottles containing pre-measured preservative. After use, the sampling equipment is rinsed with analyte-free water and returned to the sample preparation area for thorough in-house cleaning.

6.3.4.1 Teflon Drop Pipe Decontamination

Teflon drop pipes are decontaminated in-house according to section 6.3.1.

6.3.4.2. PVC and Polypropylene Hose

When purging a well with a centrifugal pump prior to organic contaminant sampling, one end of a PVC hose is attached to the pump while the other end is connected to a Teflon drop pipe with a teflon check valve on the bottom end. Only the Teflon drop pipe touches the water in the well. The PVC hose is decontaminated before use in each well by rinsing with dilute Liquinox. The hose is then rinsed with copious amounts of DI water and wiped dry with clean lab-grade paper towels. The PVC hose is not allowed to come into contact with the ground water.

When purging a well with a centrifugal pump prior to sampling for inorganics, a length of polypropylene hose equipped with a check valve on the end is attached to the pump head and lowered into the water column. The polypropylene hose is decontaminated before use by rinsing with dilute Liquinox, rinsed with DI water, and wiped dry with clean lab-grade paper towels.

After purging a well by one of these methods, the hose is slowly removed from the well casing while the pump is still running to reduce the possibility of water draining back into the well from the inside of the hose.

6.3.4.3 Filtration Units

Filtration units (QED FF 8200 or equivalent) are disposable and individually packed in plastic bags to prevent contamination prior to use.

6.3.4.4 Teflon Lanyards

Teflon coated, stainless steel lanyards are decontaminated in-house according to section 6.3.1. All sampling equipment is rinsed three times with sample water before sample collection.

6.3.4.5 Submersible Pumps

Submersible pumps are used for purging only and are decontaminated in-house according to section 6.3.1, with the exception of the acid rinse due to the stainless steel construction (the solvent rinse is optional if organics are not sampled). The interior is cleaned between sites by flushing thoroughly with DI water.

6.3.5 Sediment/Soils Equipment

The field sampling equipment is cleaned prior to being taken to the field by the following procedure: the dredge, scoop, bowl, and corer (stainless steel) are washed with Lab grade detergent, rinsed three times with tap water, rinsed with analyte free water, rinsed twice with pesticide grade isopropyl alcohol, and rinsed three times with analyte free water. The isopropyl alcohol is containerized for proper disposal. PVC corers are washed with Lab grade detergent, rinsed with tap water, dipped in 10% HCl, rinsed with tap water, then rinsed with DI water and allowed to dry. The corers are bagged in polypropylene bags until taken into the field.

6.3.6 Sample Bottles and Filtering Equipment

In the laboratory, the reusable sample bottles (except for trace metals) and reusable filter holders are washed with lab grade detergent, rinsed with tap water, 10% hydrochloric acid, tap water, analyte free water, and finally air dried. Dry bottles are stored with the caps on and filter holders are stored in a closed container. Disposable bottles, certified pre-cleaned from the vendor, are also used.

Polyethylene trace metal bottles are washed with lab grade detergent, rinsed with hot tap water, soaked in 20% nitric acid overnight, rinsed with analyte free water, and air dried. At the time of sampling, the bottle is rinsed three times with sample water

The Teflon sampling bottles and glass volumetric flasks for total mercury are cleaned by rinsing three times with DI water, filling the bottles with DI water, adding approximately 1ml of digestion reagent (Bromine Monochloride), and allowing bottles to soak for a minimum of 18 hours. Before being used, containers are neutralized by rinsing with approximately 0.2 mls of 30% w/v hydroxylamine hydrochloride per 100 mls of DI water. Containers are then rinsed 5 - 7 times with D I water. Teflon bottles are then shaken dry and capped until sampling. Volumetric flasks are partially filled with D I water for solutions preparation.

Reusable filter holders are soaked in a weak lab grade detergent solution before washing to soften any residues which may be in the filter holders from the previous sampling process according to Section 6.3.1. Reusable filter holders are then washed following the same procedure as inorganic sample bottles. Disposable filter holders are purchased pre-cleaned and are not cleaned before use. Filtering syringes are washed with lab grade detergent, then rinsed with tap water followed by analyte free water and allowed to air dry.

Clean bottles/equipment are obtained from the lab by field personnel prior to each trip. Bottles for the collection of samples sent to contract laboratories are provided by the laboratory performing the analyses. These bottles have been cleaned by the contract laboratory according to that laboratory's procedures prior to shipment to the District. The contract laboratories must have approved cleaning procedures in their Comprehensive Quality Assurance Plans.

6.3.7 Autosamplers

All autosampler bottles are cleaned in the laboratory using the standard bottle cleaning procedure described in Section 6.3.1 as per EPA SOP & QAM guidelines, Appendix B, Sections B.5 and B.6. Tubing is not cleaned but replaced. The sampler and sampler tubing is dedicated to each site. The sampler is programmed to rinse the tubing twice with sample before collection and then purge the tubing after the sample is taken. The pump and intake tubing for autosamplers is replaced at least quarterly or when first deployed, except for the autosamplers deployed at pump stations where the intake tubing is permanently in place. The entire sampler is cleaned when it is removed from the site for repair or transferred to another site. In an emergency, pump tubing may be cleaned by soaking in hot water and Liquinox and scrubbing the outside and ends with a small bottle brush. The tubing is then rinsed with tap water, followed by DI water.

6.3.8 Atmospheric Deposition Equipment

Atmospheric deposition buckets are cleaned according to section 6.3.1, Once dry, the buckets are capped and taken to the site. Each lot of cleaned buckets is tested for proper decontamination by pouring one liter of DI water into the bucket, swirling, allowing to steep overnight, and analyzing for the parameters of

interest.

6.3.9 Ultra Trace Mercury Equipment

All ultra trace mercury sample collection equipment (including sample bottles) is pre-cleaned before use by the contract laboratory using methods as specified in the contract laboratory's approved CompQAP. The filtration units are disposable. The SFWMD does not clean any ultra trace mercury equipment. Sample bottles are rinsed three times in the field with sample before the sample is collected. When using the peristaltic pump, a minimum of three sample hose volumes (200 ml) of sample water are flushed through the tubing or filter before the sample is collected. The contract laboratories ship all equipment, including filters and sample trains for the peristaltic pump, clean and in bags, and supply ultra pure water for blanks, rinsing, and field use. All equipment is taken into the field in plastic bags.

6.3.10 Analyte Free Water Containers

The analyte free water containers are cleaned in the laboratory using the standard bottle cleaning procedures described in Section 6.3.1 as per EPA SOP & QAM guidelines, Appendix B.

6.4 Sampling Procedures

Water sampling locations in the SFWMD are frequently established at water control structures where known flow rates can be combined with chemical concentrations to determine loadings. However, in lakes, rivers, marshes, estuarine systems, storm water runoff, and agricultural point sources, sampling sites are chosen based on other criteria as described in the appropriate Quality Assurance Project Plans. In any case, the sample must be collected in such a manner as to ensure that it is representative of the water body being studied. A new pair of disposable latex/PVC gloves are used at each sampling point for all types of sampling. All grab samples of surface water are collected at a depth of 0.5 meters, or at mid depth where the water is less than 1 meter deep. Except otherwise noted, all samples are preserved according to table 6.9, 6.10, 6.11, 6.12, or 6.13.

6.4.1 Surface Water (except organics and autosamplers)

The surface water sampling procedures (except organics and autosamplers) are given in the flow chart in Figure 6-1. The following special considerations are observed when applicable:

1. If a boat is used, the sample is taken from the bow, and/or upwind and upstream from the motor.
2. When wading, the sample is collected upstream from the collector.
3. Care is taken not to disturb the sediment in the immediate sampling area.
4. Pre-preserved containers are not used as collection containers.
5. Intermediate containers are inverted, immersed to the appropriate depth, and turned upright pointed in the direction of flow, if applicable.
6. Samples are preserved according to Table 6.9, 6.10, 6.11, 6.12, or 6.13.

6.4.2 Surface Water - Organics

Surface water samples for organic analysis are collected directly into the sample bottles using a subsurface grab sampler equipped with a pre-cleaned glass bottle provided by the contract laboratory. All unpreserved

containers (except for VOCs) are rinsed three times before sample collection. The sample containers are immersed, inverted to 0.5 meter below the surface, and, pointing in the direction of flow, turned upright until full.

For Volatile Organic Compound samples, the water is poured slowly down the edge of the 40 ml Teflon-lined septum glass vial from the pre-cleaned glass bottle to minimize aeration. The vial is filled to the point of creating a convex meniscus. The cap and septum is placed, teflon side down, on the meniscus and sealed. The vial is inverted and lightly tapped on the lid to dislodge any entrapped air bubbles. The absence of air bubbles indicates a proper sample collection. If air bubbles are present, the bottle is opened, additional sample is added and the vial is resealed. Additional sample is added a maximum of three times. If a seal cannot be obtained, the vial, sample, and septum are discarded, a new vial is used and the sampling procedure is repeated. A teflon bailer may also be used in the absence of strong currents or where a discrete sample at a specific depth is not required. Each sample is identified by the project code and sequential sample number. During sample collection, the date, time, location, water movement, weather and site conditions are recorded. The preservation technique and holding times are shown in Table 6.10. Following collection of the sample, the bottles are sealed, tagged or labeled, and placed in wet ice.

The samples are sent to the certified contract laboratory having a DEP approved Comp QAP with appropriate sample identification and chain of custody form. The samples are packed in coolers with bubble-wrap or other appropriate packing material to avoid breakage. Samples are kept at 4°C with wet ice. Sample containers for VOC analysis are placed into separate bubble-pack bags for each station. At least one trip blank must be included in each separate cooler containing VOC samples. Coolers are taped shut with shipping tape, labeled appropriately and shipped to the laboratory using common carrier overnight delivery.

6.4.3 Surface Water – Autosamplers

Water quality autosamplers are used for the collection of daily composite or discrete samples. The choice of whether to use a discrete or composite autosampler depends on the requirements of the project, the facilities available at the sampling site, and the parameters for which samples are to be collected.

Autosamplers are dedicated to a specific location as long as they are working properly. If an autosampler fails to perform, the sampler head including the pump hose, electronics, and flow sensor are replaced as required. Sampler intake tubing is dedicated to a collection site and is replaced at a minimum of every quarter or when the autosampler is first deployed. The tubing may be replaced sooner if algal growth is observed in and around the inflow tubing.

6.4.3.1 Discrete Autosamplers

The discrete automatic sampler is programmed to rinse the sample collection tubing twice prior to sample collection and to purge the tubing following collection. The samplers are programmed depending on the project requirements. For example, the programming may include collection of daily composites at a rate of 80 ml of sample at 144 minute intervals and addition of the sample to the correct individual sample bottle for a total of 10 samples per 1 liter discrete sample bottle. When samples are analyzed for total Kjeldahl nitrogen, total phosphorus, ammonia and nitrate plus nitrite only, an appropriate quantity of 50% sulfuric acid is pre-added to the discrete autosampler bottles before sample collection. The amount of acid is selected to maintain pH <2 after sample collection. Sampler intakes at water control structures are usually located 1 to 2 feet below the historic low mean water level and 1 foot off the wing wall. Placement of the sample intake depends of the goals and specifications of the project, or may be determined by the type of

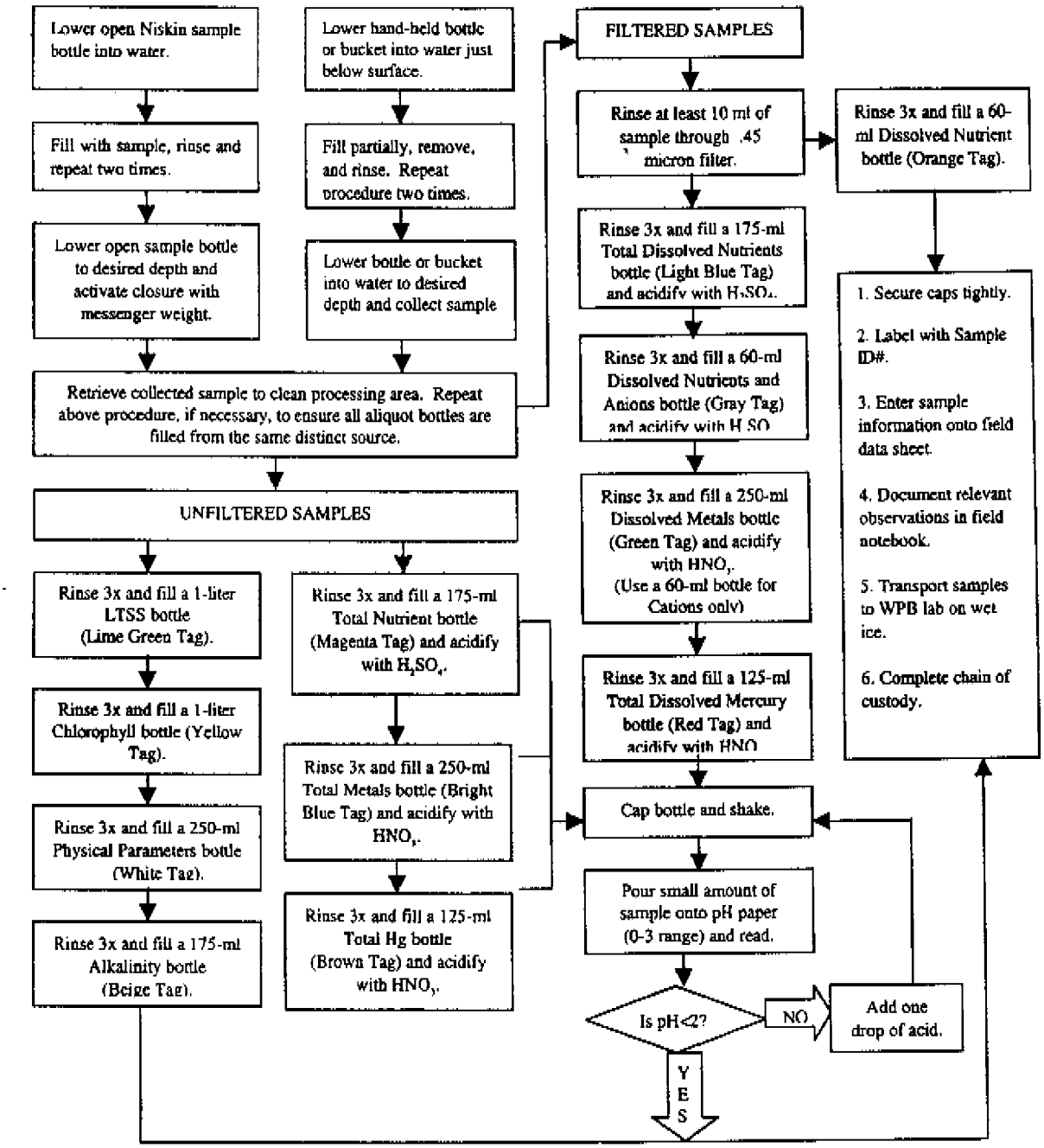
structure.

After sample collection the bottles are capped, thoroughly mixed by inverting five times, labeled, placed on ice immediately and transported to the field laboratory and stored at 4°C. Within 24 hours, aliquots of the samples are transferred to laboratory sample bottles, tagged, and stored at 4°C until transported to the analytical laboratory. Samples are submitted to the analytical laboratory within two days of the day the sampler was serviced. Routinely, samples are poured into sample bottles at the collection site, preservation is checked and samples are immediately placed on wet ice for transport to the analytical laboratory. These procedures are only followed for long holding time parameters.

6.4.3.2 Composite Autosamplers

Most of the composite autosamplers used by the District are installed at pump stations where electrical power is readily available. Autosamplers located at pump stations generally work in synchrony with each of the pumps in the pumping battery. The autosampler is activated once any of the pumps becomes operational, and is usually programmed to collect an aliquot at pre-determined intervals for each operating pump. The aliquot is dispensed into a refrigerated five gallon polyethylene bottle. Generally, the refrigerated autosamplers are deployed for up to one week and samples picked up within 24 hours after sample collection has been completed. Deployment times may vary depending on project requirements as specified in the project specific Quality Assurance Plan. The refrigerated composite sample is homogenized by capping and shaking the bottle. An aliquot is transferred unfiltered to a 125ml sample bottle for TKN and TPO_4 and another aliquot is filtered into a 60 ml sample bottle, for NO_x and NH_4 testing. Both aliquots are preserved upon collection in the field as indicated in Table 6.9. The District is currently conducting a validation study to determine if a difference exists between pre- and post-preserved samples with and without refrigeration. If the study indicates that a significant difference exists between the two preservation protocols, all affected data will be flagged.

Figure 6.1 Inorganic Surface Water Sampling Procedure



6.4.4 Sediment/Soil

6.4.4.1 General Considerations; Duplicate Analyses

The District collects both grab and core samples for sediment analysis of inorganic and organic analytes. The type of sampling employed depends on the requirements of the project for which the samples are being collected. Field equipment is cleaned prior to the field trip and after each sample according to the procedures described in Section 6.3.5. Whenever possible, sufficient equipment should be pre-cleaned in the laboratory for each trip so that field cleaning will be unnecessary.

Duplicates are collected to measure the variability inherent in the sampling process. Duplicates for sediments are collected from the same sample. Since a true split sediment sample is almost impossible under field conditions, split soil samples are considered duplicates. If analyses for pesticides, extractable organic compounds, or VOCs are to be performed, the sample containers must be glass with teflon lined lids. VOC bottles must have a teflon-lined septum. For other classes of analytes, glass or plastic jars, or other approved containers supplied by sub-contracting laboratories, may be used as sample containers. The containers are completely filled with sample by gently packing the sample into the container with a spoon or scoop to minimize air bubbles trapped in the container in order to minimize sample oxidation that could influence certain test results.

6.4.4.2 Grab Samples

Sediment samples for organics are collected by hand grab using a stainless steel scoop/spoon, stainless steel petite Ponar™ dredge, or stainless steel core. The stainless steel scoop/spoon is utilized only in quiescent shallow waters. The petite Ponar™ dredge is effective over the wide range of circumstances encountered during the collection of sediment samples. The petite Ponar™ dredge can be used on soft or hard bottoms under a variety of flow and depth conditions. Sediment samples from canals are collected mid-stream using a small boat. The dredge is lowered by rope until contact with the bottom is established, then the dredge is slowly retrieved and emptied into a stainless steel bowl/tray. The stainless steel corer is used when collecting shallow sediment samples. Each sample (except samples for VOC analysis) is thoroughly mixed in the stainless steel bowl with a pre-cleaned stainless steel spoon. The sediment in the bowl should be scraped from the walls and bottom, rolled to the middle and initially mixed. The mass is quartered, moved to the opposite sides of the bowl, and each quarter individually mixed. Each quarter is then rolled to the center of the bowl and the entire mass is mixed again. This process is continued until the mass is as homogeneous as possible. A pre-cleaned stainless steel spool or small scoop is used to transfer the required amount of final homogenized material from near the center of the mass into the appropriate sample bottle. This is done quickly to prevent oxidation of metal ions or volatilization of organic compounds. The bottle is filled to its rim.

6.4.4.3 Core Sampling

Sediment cores are collected using PVC corers if the sample will not be analyzed for organics. Intact sediment cores are obtained by driving a PVC coring tube to a depth of approximately 50 cm into the soil. Under shallow conditions (water depth < 30 cm) this is achieved using a block of wood to protect the neck of the core tube and then striking the block several times with a hammer such that the core penetrates the soil with minimal compaction. If the water is greater than 30 cm, a PVC coupling should be attached to the coring device. This attachment is comprised of a ball valve and a length of PVC pipe. This is fitted to the neck of the PVC coring tube and a closed ended piece of metal pipe with a diameter larger than the upright

PVC pipe is used to pound the core into the sediment. During the insertion, the ball valve should be open. Prior to pulling the core out of the sediment, the ball valve should be closed. The sediment core, with the overlying water, is then labeled, capped at both ends using rubber stoppers, stored out of direct sunlight, and transported to the laboratory. If the sediment surface within the core is at a significantly different depth than the adjacent soil, compaction has occurred, and the core should be discarded.

6.4.5 Ground Water

Ground water wells are purged from shallowest to deepest well, if more than one well is located at a site, and then samples are collected from the least to most contaminated wells whenever possible. The order of collection is: 1) VOCs, 2) Extractable Organics, 3) Total Metals, 4) Dissolved Metals, 5) Inorganics and 6) Radionucleides. The SFWMD does not use temporary well points or dedicated equipment for ground water monitoring. Samples are not collected from wells which contain free product.

A protective covering of visqueen plastic is placed on the ground around the well at all sampling sites to reduce the potential for contamination. A fresh pair of disposable non-powdered latex gloves are worn at each sample site while purging and another fresh pair worn for collecting samples. All samples are collected using a Teflon bailer which is suspended by a Teflon coated stainless steel lanyard and carefully lowered into the well. Prior to the collection of the sample, the bailer is rinsed three times with sample. The lanyard and bailer are not allowed to touch the ground during sampling or purging.

The bailer, filled with sample, is retrieved from the well and placed on a bailer stand. VOC samples are collected first via a controlled-flow bailer bottom and are poured slowly down the side of the sample vial to minimize sample aeration. The vial is filled to the point of creating a convex meniscus. The cap is secured with the Teflon side of the septum contacting the sample and no headspace in the sample container. The vial is inverted and gently tapped to locate bubbles. If bubbles are present, the sample in the vial is discarded and sample is recollected and checked for bubbles. Additional sample is added a maximum of three times. If an acceptable sample cannot be collected, the sample, vial and septum are discarded, a new vial and septum are used, and the collection procedure begins again.

To filter a sample, a one piece, molded, in-line disposable filter is attached to the bottom of the bailer with a Teflon adapter. The top of the bailer is attached to a vacuum pump by a Teflon attachment so the bailer can be pressurized to force water through the filter. The filters are 0.45 micron unless a dissolved metals sample is collected for permit compliance, in which case the filter pore size is required to be 1.0 micron. The first 100 ml of sample to pass through the filter is discarded as rinse water, and the last 100 ml of sample water in the bailer is not used since it has been in contact with the air at the top of the bailer. Duplicates are collected by sampling from consecutive bailers.

If the analyses are to be performed by a contract laboratory, the bottles are cleaned by the contract lab according to that laboratory's procedures prior to shipment to SFWMD. The contract laboratory must have approved cleaning procedures in their Comprehensive Quality Assurance Plan. Certified disposable VOC vials are purchased from approved suppliers.

Splits are not routinely collected in the groundwater program. When splits are required to measure the performance between two or more laboratories, splits are collected from the same bailer. For large volume samples that may require more than one bailer load, the first half volume of the first bailer load is poured into the first set of containers and the second half in the second set of containers. Then the first half-volume of the second bailer load is poured into the second set of containers and the second half in the first set of

containers, etc., until both sets are full.

The sample bottles are labeled with the date, sample number, and project name before sampling at a site begins; sample time is added to the labels as samples are collected. After collection, the samples are preserved, according to tables 6.9, 6.10, 6.11, 6.12, and 6.13, and immediately placed on ice in a closed container. Exposure of organic samples to sunlight is kept to a minimum. When VOCs are collected, trip blanks are transported with the sample bottles to make certain that the samples have not become contaminated. These trip blanks accompany the samples from the time the empty sample bottles are shipped from the contract laboratory until the samples are analyzed. Trip blanks account for at least 5% of the samples that are analyzed. At least one trip blank must be included in each separate cooler.

6.4.5.1 Wells with In Place Plumbing

These wells are purged for a minimum of 15 minutes, until three bore volumes have been removed, or until the well has chemically stabilized, whichever is greater. The sample is taken from the faucet closest to the source and before any screens, aerators, or filters. The flow rate is reduced as necessary to avoid any undue disturbance. Unfiltered samples are collected directly into the sample containers from the spigot. Filtered samples are collected by filling a bailer from the spigot. The bailer is rinsed three times with sample, then filled and pumped through a filter attached to the bottom of the bailer with a Teflon adapter. Sample bottles are rinsed once with sample (unless sample bottles are pre-preserved, or collection is for VOCs).

6.4.5.2 Flowing Artesian Wells

These wells are purged until three bore volumes have been evacuated, or until the well has chemically stabilized, whichever is greater. The flow of water from the well is adjusted to minimize the aeration and disturbance of samples. Unfiltered and filtered samples are collected directly from the discharging water into a bailer.

6.4.5.3. Monitoring Wells

The depth to water in each well relative to a measuring point is measured twice using an electronic water level indicator. Both values, which must be accurate to within 1/10" of a foot, are recorded on the field log sheet. More stringent measurements may be required for specific project programs. These values will later be used to calculate water elevation relative to mean sea level. The same measuring point is used every time the well is sampled. When the wells are surveyed, the survey point is used for the measuring point. Water level measuring devices are rinsed with dilute lab grade detergent and DI water, and wiped dry with a clean lab-grade paper towel before measuring each well.

Prior to collection of samples, a centrifugal pump, a submersible pump or a peristaltic pump is used to purge each well. The choice of an appropriate purge pump is determined by a combination of factors: the total volume of water necessary to be purged, the diameter of the casing of the well, and the depth-to-water are a few of those factors. Centrifugal and submersible pumps (or the associated generator) are gas powered. Extreme care must be used when handling and placing these units to minimize on-site contamination. Place them downwind, away from any sampling activities.

The centrifugal pump is connected to a flexible PVC suction hose, which is cleaned prior to use by the method documented in section 6.3.4.2. Neither the pump, nor the PVC hose comes in contact with the water in the well. The other end of the hose is connected to a rigid three-foot length of Teflon drop pipe, which

is equipped with a Teflon check valve at its bottom end to prevent the back flow of purged water into the well. The drop pipe and check valve are cleaned prior to use by the decontamination procedures outlined for other sampling equipment in section 6.3.1.

The submersible pump is decontaminated as specified in section 6.3.4.5. It is connected either directly to a polypropylene hose (inorganics), or to a three-foot Teflon drop pipe (organics) before being connected to the polypropylene hose. The hose is cleaned by the method detailed in section 6.3.4.2; the drop pipe is cleaned by the decontamination procedures outlined in section 6.3. The submersible pump is equipped with an internal check valve to prevent the back flow of purged water into the well. The submersible pump with the 150' electrical lead is capable of purging water from as far down as 135 feet (if a well were to be drawn down that far). When purging a well, the drop pipe or the head of the submersible is lowered to the top of the water column so that the purging process removes all of the standing water. If the water level is drawn down during the purging process and air begins to enter the drop pipe or the head of the submersible, it is lowered to the new water level. If the water level is drawn down too severely, the purge rate is decreased.

The battery powered peristaltic pump is used occasionally to purge low volume wells. If the well will be sampled afterward for inorganic constituents, C-flex tubing is lowered into the water column as well as placed through the pump head. The C-flex is cleaned by the method detailed in section 6.3.4.2. If the well will be sampled after purging for organic compounds, Teflon tubing is lowered into the water column and connected to the C-flex tubing, which runs through the pump head. The Teflon tubing is cleaned per the decontamination procedures outlined in section 6.3.1.

Three standing water volumes, minimum, are removed from the well. The volume of water to be purged is calculated using the following formula:

Minimum Purge Volume in Gallons $= (D^2) * (DW-DTW) * 0.1224$

D = Casing Diameter in Inches
DW = Depth of Well in Feet
DTW = Depth to Water in Feet

The constant 0.1224 is a units conversion factor.

The volume of water to be removed from the well must be calculated to provide sufficient purging. The flow rate is estimated by measuring the amount of time required filling a bucket of known volume. The required purge volume is then divided by the estimated flow rate to find the estimated time necessary to purge the well. Flow is measured several times during purging to be certain that it does not change.

Although a minimum of three water column volumes must be purged, the well is not considered to be ready for sampling until the well has chemically stabilized. Temperature, pH and specific conductivity are monitored and readings are recorded on the field log sheet at time intervals equal to one-half of a bore hole volume. A minimum of seven readings are normally recorded – an initial reading, and six more readings recorded at each one-half bore hole volume – until the well has stabilized. Chemical stability readings are made in a flow through chamber to minimize atmospheric contact with the sample. The well is considered to be chemically stable when the last three consecutive readings of temperature, pH, and specific conductivity are within 5% or 0.1 unit for pH readings. The purge volume is also noted on the field log sheet. Only the final readings taken after the well has stabilized are input into the sample results database. The drop pipe is slowly raised out of the well while the purge pump is still running to make certain that all

of the water above the drop pipe inlet is purged. This procedure also minimizes the possibility of the back flow of water from the drop pipe or suction hose.

Wells with low hydraulic conductivity sediments that can be purged dry at one liter per minute are not sampled because the water quality is not deemed to be representative of the aquifer water quality. The SFWMD does not sample wells for which recovery cannot be accomplished in four hours or less.

All ground water samples are collected using a Teflon bailer system or a peristaltic pump. When handling bailers or other sampling equipment, clean powder-free Latex gloves are worn. The Teflon bailer is connected to a lanyard with Teflon coated stainless steel line and carefully lowered into the column of well water. The bailer is filled with well water, removed from the well and discarded three times before beginning sample collection.

A minimum of 500mls of purged well water should be flushed through the lines of the peristaltic pump before inorganic sampling actually begins. Organic sampling with the peristaltic pump would require that the glass sampling bottle, which comes with the unit, be rinsed three times and the rinse water discarded before filling any sample bottles. This procedure is done as a final rinse of the pre-cleaned sampling equipment or tubing. The lanyard, bailer and tubing are not allowed to touch the ground. After sampling, the equipment is rinsed with DI water and returned to the lab for cleaning.

6.4.5.4 Porewater Wells and Peepers

Porewater is collected using porewater wells and peepers (Appendix G) for certain mandated projects and other research projects. Once collected, samples are processed and handled according to the QAPP or research SOP for each project.

6.4.6 Biological Tissue

6.4.6.1 Above Ground Macrophyte Biomass

Above ground vegetation is clipped at the sediment-water interface with a pair of sheers or knife, and separated into subsamples by species. Each biomass subsample is placed into an individual large opaque plastic bag, labeled, and transported to the laboratory for processing.

6.4.6.2 Below Ground Macrophyte Biomass

After the above ground vegetation has been removed from the sampling quadrant, a soil core is collected from the quadrant using a soil corer. Each soil core is placed into an individual large opaque plastic bag, labeled, and transported to the laboratory for processing.

6.4.7 Hazardous Wastes/Drums

The SFWMD does not sample hazardous waste or drums.

6.4.8 Waste Water

The SFWMD does not sample waste water.

6.4.9 Microbiological

Microbiological samples are collected and preserved on ice, with extra care in order to prevent sample contamination. The samples are not composited. The personnel collecting the samples do not touch the rims or top of the Whirlpak™ sample containers. A Whirlpak™ must be discarded if it is suspected that the top portion has been touched. Microbiological sampling is closely coordinated with the laboratory performing the analyses to ensure that samples are analyzed within holding times.

6.4.10 Oil and Grease

Oil and Grease samples are collected directly into the sample container without rinsing with sample. Sample containers are not pre-acidified. Sample bottles for Oil and Grease are provided by the contract laboratory.

6.4.11 Low Level Trace Mercury

The SFWMD follows the clean hands (CH), dirty hands (DH) technique as specified in *EPA Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels*, when collecting low level total and methyl mercury samples in surface water. CH, using non-powdered plastic or latex gloves, touches only clean surfaces (such as new gloves, new plastic bags, or surfaces that have been cleaned in an ultra-clean facility. DH, using wrist length latex or plastic gloves, touches the cooler and the sample bottle covered with two plastic bags. DH opens the outer plastic bag, CH reaches in, pulls up bottle covered with inner plastic bag, and removes the bottle from the inner bag.

CH reaches into the water and collects 100 ml of surface water by immersing the sample bottle about 10 cm under the surface. The bottle is rinsed by shaking the contents (cap on) and dumping the contents away and downstream from the sampling area. This rinse is repeated two more times. The bottle is filled a final time with more than 100 ml and the cap secured tightly by hand. CH then puts the bottle in the inside bag, seals it, and places the inside bag into the outside bag. DH seals the outside bag and places it back inside the container. DH records the sample bottle number, time, site and other information on the field data log sheets, and later transfers the information to the field notebook. Relevant site conditions are recorded into the field notebook.

Samples may also be collected using a pump. The CH/DH procedure is used. DH removes the sampling train (filter holder with nitex screen, Teflon tubing and C-FLEX tubing-all connected) from the cooler and opens the outside bag. While CH holds the tubing, DH secures it to the sampling boom with tie wraps. DH opens the pump head, places the pump tubing inside, and closes the pump head. DH opens the ring stand clamp, CH places the pump tubing inside and DH closes the clamp. CH then changes gloves prior to the sampling event.

To begin sample collection, DH positions the end of the sampling train about 10 cm below the water surface and about one meter from shore. DH starts the pump and begins flushing. DH removes a sample bottle from the cooler and opens the outside bag. CH opens the inside bag and removes the sample bottle. CH opens the sample bottle, empties the contents away from the sampling area and fills the bottle with approximately 50 ml of sample water. The bottle is rinsed by shaking the contents (cap on) and dumping the contents away from the sampling area. This rinse is repeated two more times. The bottle is filled a final time with more than 100 ml and the cap secured tightly by hand. CH then puts the bottle in the inside bag, seals it, and puts the inside bag down inside the outside bag. DH seals the outside bag and places it back inside the cooler.

To prepare a filtered surface water sample, DH stops the pump, removes a filter cartridge from a cooler, opens the bag, CH removes the filter and connects it to the end of the sampling train. DH starts the pump and the filtered sample is collected as above. All equipment is rinsed with sample water at the next site before collecting a sample. To control mercury contamination, CH secures a clean plastic bag to each end of the sampling train for transport between sites. The equipment and supplies are stored inside coolers and bags in a place relatively free of mercury contamination. Dust and human breath (from dental amalgams) contain large amounts of mercury and should be avoided during sample collection. Samples should not be collected in the rain.

6.4.12 Benthic Macroinvertebrates

The SFWMD follows macroinvertebrate sampling methods as detailed in the Florida Department of Environmental Protection (FDEP) Biology Section Standard Operating Procedures (Appendix E) and in EPA/600/0-90/000, *Macroinvertebrate Field and Laboratory Methods for Evaluating the Biological Integrity of Surface Waters*. The field methods used to study benthic macroinvertebrate communities relate to qualitative, semi-quantitative and quantitative sampling, and are dependent on the data quality objectives of the study. Generally, there are four categories of benthic macroinvertebrate samples collected: cores, grabs, artificial substrates, and miscellaneous. Field equipment is cleaned prior to the field trip and after each sample according to the procedures described in Section 6.3.5.

a. Corers

Coring devices may be used at various depths in any substrate that is sufficiently compacted so that an undisturbed sample is retained; however they are best suited for sampling homogeneous soft sediments, such as silt, clay or sand. Macrofauna are sampled using single and multiple-head corers, tubular inverting samplers, open-ended stovepipe devices, hand corers and/or box corers. Devices are lowered slowly into the substrate to ensure good penetration and to prevent organisms from escaping. Visual inspection of each sample is necessary to ensure an adequate amount of sample is obtained.

b. Grab samplers

Grab samplers are designed to penetrate the substrate by gravity and have spring or gravity activated closing mechanisms. They are used to sample a unit area of the habitat. The habitat and substrate type sampled, depth of penetration, angle and completeness of jaw closure, loss of sample during retrieval, disturbance at the water-sediment interface, and effect of high flow velocities all affect the quantity and species of macroinvertebrates collected by a particular grab. Petite Ponar™ or Ekman grab samplers are typically used; however the type and size of the selected device depends on the substrate composition, water depth, and hoisting gear available. The sampler is lowered slowly to avoid a disturbance of the surface sediment and to ensure the device bites vertically. Upon tripping of the closing mechanism, the sediment is contained and the device is retrieved. The sample is then placed into a suitable container for transporting to the laboratory or is placed directly into a sieving device and processed.

c. Artificial substrate samplers

Artificial substrate samplers, such as the multiplate (modified Hester-Dendy) sampler, consist of natural or artificial materials of various composition and configuration. They are placed in the water body for a predetermined period of exposure and depth for the colonization of indigenous macroinvertebrates. This type of sampling is used to augment bottom substrate sampling because many of the physical variables encountered in bottom sampling are minimized, e.g. light attenuation, temperature changes, and substrate variation. The samplers are deployed in the euphotic zone of the water column to promote maximum colonization. They are exposed for a preset period of time and retrieved vertically from the water. The

samplers are placed in a preservative container and transported to the laboratory for processing.

d. Miscellaneous qualitative devices

Many devices such as dip nets, bare hands, tongs, and forceps may be used to collect benthic macroinvertebrates. Dip nets are used by sweeping the net through the water or by holding the net stationary against the bottom and disturbing the substrate, causing the benthos to be swept into the net.

Samples collected by any of the aforementioned devices contain varying amounts of fine materials which can be removed by sieving immediately in the field, or in the laboratory. If laboratory sorting can be performed within 24 hours, place samples immediately on ice. If sorting will be delayed, preserve with 10% formalin. After organisms have been removed from detritus, they should be placed into 70% ethanol.

6.4.13 Atmospheric Deposition

Atmospheric deposition samples are collected according to the National Atmospheric Deposition Program (NADP) guidelines. Samples are preserved according to table 6.9.

The District uses Aerochem Metrics Wet/Dry Precipitation collectors. These automated collectors are aluminum structures with two 3.5 gallon PVC buckets which serve as receptacles for wet and dry deposition, although dry deposition samples are no longer collected and processed by the District. An aluminum lid covers the wet side bucket under dry conditions. When the humidity sensor detects rain, the lid rotates over the dry side bucket, uncovering the wet side bucket. The wet buckets are collected on Tuesdays only, according to NADP guidelines. The buckets are capped (while using PVC/Latex gloves) and taken to the laboratory for processing within 24 hours. Clean buckets are placed into the Aerochem collectors. Before clean buckets are placed into the collectors, one bucket from each decontamination lot is checked by pouring one liter of DI water into the bucket, allowing the water to sit overnight (capped) in the laboratory, and processing the water as an equipment blank.

All wet side buckets are weighed using a top loading balance and subtracting the clean bucket tare weight (determined before deployment in the field). Any non-representative foreign matter such as frogs, insects, lizards, or vegetative material are removed with a clean scoop or tweezers. Any removed contaminants are documented in the field notebook. The sample is then processed as a routine surface water sample, including filtration and preservation as required. Quality control samples (equipment blanks, splits and field blanks) are submitted according to routine QA procedures.

Dry side buckets are inspected for contamination as above, and notes on the presence and nature of any contamination are recorded into a field notebook.

6.4.14 Marsh Sampling

Some field trips require transport by helicopter. Most of a helicopter trip can be prepared early, thereby making the actual sampling event run smoothly.

Samples for inorganic analyses are collected in polyethylene sample bottles for each site and each QC sample. Samples can also be collected directly into an intermediate container or using a peristaltic pump.

6.4.14.1 Surface Water Collection, Grab Samples

1. After helicopter lands, wade out away from helicopter disturbance area, preferably downstream if flow is visible and/or downwind.
2. Be careful to avoid weeds and the creation of turbidity.
3. Measure and record the total depth of the water using a long, rigid, graduated pole.
4. Don clean pair of PVC sampling gloves.
5. If water depth > 20 cm, immerse intermediate sample container(s) in an undisturbed area upstream from the sampling personnel, rinse three times, and fill to the brim.
6. Cap and label the container(s) and place on wet ice.
7. Measure and record Depth, Temperature, pH, Conductivity, DO and Total Depth, at middle depth using a multi-parameter field instrument.

6.4.14.2 Surface Water Collection, Peristaltic Pump

6.4.14.2.a Platform sampling:

1. After the helicopter lands at the South end of the platform, walk to the North end of the platform, which is in the direction of the water flow.
2. Set up peristaltic pump and place a screen on the intake end of the tubing (to block large chunks of algae and plant from flowing into the sample). Place the end of the tube into the water column (about 10-15 cm beneath surface) being sure not to disturb bottom sediments. Turn the pump on and let the water run for about 10-15 seconds to rinse the tube.
3. Put on plastic gloves and begin to fill unfiltered sample bottles, being sure to rinse the bottle 3 times before collecting sample. Also, make sure look in the bottle to see if the sample is clean and particulate-free. For filtered samples, place in-line filter on the outflow end of tube and let the water flow through filter for a few seconds. Rinse bottles 3 times and fill.
4. After all the bottles are full, acidify appropriate samples and place in bag.
5. Record water depth measurement and Hydrolab readings (once again being sure not to disturb bottom sediments with probe).
6. Gather equipment, walk back to helicopter, and place samples in a cooler.

6.4.14.2.b Pontoon Sampling

1. The pilot lands the helicopter partially on the vegetation so that it will stay in one place to minimize disturbance to the water column.
2. One person steps out onto the pontoon and attaches the end of the tube (which has a screen) to a long pole that extends about 10 to 15 feet from the helicopter. The person on the pontoon extends the pole out into the open water and finds a spot that appears undisturbed. The end of the tube is submerged in the water column about 10-15 cm below the surface of the water.
3. When an appropriate sampling spot has been found the person in the helicopter turns on the peristaltic pump and begins to sample, making sure the sample is clean and particulate-free. If the sample is not clean, the pole is moved to another spot until a clean sample can be obtained
4. The procedure for filling sample bottles is the same as above.
5. When bottles are filled, the person on the pontoon records a water depth measurement and a Hydrolab reading from the pontoon.

6.4.14.3 Sediment Sampling

Should the trip require sediment collection, samples are processed on site into appropriate containers as per techniques referenced in Section 6.4.4.

6.4.14.4 Sample Processing

Within four hours of sample collection, aliquots are processed from the large intermediate containers into more appropriate containers for lab analyses. Samples are processed and placed into appropriate sample containers according to the procedure illustrated in Figure 6.1.

6.4.15 Duplicates/Split Samples

Duplicates are collected to measure the variability inherent in the sampling process. Duplicates are obtained by repeating the entire acquisition technique used to obtain the first sample immediately following the initial sample collection, sample collection being defined as the sampling event from initial sample acquisition through filling and labeling the bottles. Duplicates for water are collected by sampling from successively collected volumes. Duplicates for soils are collected from the same sample source and same composited soil sample (i.e., soil obtained from the same soil sampling device). Split samples are collected to measure the variability between laboratories, and are taken from the same parent sample (a true split of soil, sediment or sludge is almost impossible to accomplish under field conditions; a split soil sample should be considered a duplicate and not a split sample). Split samples (for water) are taken from consecutive sample volumes from the same sampling device (i.e. from same bailer, sample container, or Niskin bottle), or by mixing in a large intermediate vessel.

Note: for large volume samples that may require more than one bailer load, the first half volume of the first bailer load is poured into the first set of containers (the second half into the second set of containers), then first half volume of second bailer load is poured into the second set of containers (second half into first set of containers) etc. until both sets are full.

6.5 Documentation

The following is a list of the field records that are maintained.

1. Field Trip Checklist (surface water/groundwater)
2. Chemistry Field Data Log
3. Bound field notebook (project specific)

6.6 Preservation

Holding times and preservation techniques for each parameter are given in Tables 6.9, 6.10, 6.11, 6.12, and 6.13.

With the exception of marsh sampling as discussed in Section 6.4.14, samples are preserved in the field at the time of sample collection either by using preservatives provided by the SFWMD laboratory or the contract laboratory. If sample containers are received with preservative from the contract laboratory (VOCs), additional preservative is requested so it is available if needed. ACS reagent grade or better preservatives are used.

For unfiltered samples, the appropriate preservative is added to the sample bottle after the bottle is filled. The bottle is capped and shaken after which a small amount of the sample is poured onto a narrow range pH (0 - 3 pH units) test strip to ensure pH<2. If pH is not <2, additional acid is added drop-wise, the bottle is capped and shaken, and the pH is tested again. This procedure is followed until pH<2. The pH is checked on all samples requiring preservative and this amount of acid is added to the equipment blank and field blank. The amount of acid added is recorded in the field notebook. For filtered samples, the acid is added after filtration following the procedure outlined for unfiltered samples. Fresh preservatives are obtained from stocks biweekly or as needed for all sampling trips. Preservatives are taken into the field in polyethylene dropper bottles.

6.7 Sample Dispatch

Samples to be analyzed by the SFWMD laboratory are submitted to the laboratory by field personnel. The majority of samples are submitted the same day they are collected but all samples are submitted as soon as possible after collection in order to meet recommended holding times.

Samples to be analyzed by contract laboratories are shipped to the laboratory by common carrier overnight delivery the same or next day they are collected. All samples are carefully packed with appropriate material to prevent breakage, and sample chain of custody sheets are included with the samples. Insulated coolers are used for sample shipment and are sealed with shipping tape to avoid tampering. If samples must be kept at 4°C, wet ice is used during shipping.

6.8 Field Waste Disposal

All field generated wastes and purge waters are disposed of properly in a manner that will not contaminate the sampling site. Highly contaminated wastes are segregated and containerized for proper disposal, including the use of the services of a commercial disposal company if required. The SFWMD does not sample hazardous waste sites so the only field generated wastes are acids and isopropyl alcohol. The isopropyl alcohol is containerized for proper disposal. Acids are disposed of on site in a location that will not contaminate the sampling area; no concentrated acids are taken into the field during SFWMD sampling trips. The calibration standards for field parameters are flushed into the sanitary sewer.

Table 6.9 Holding Time and Preservation for Water Inorganics[1]

Parameter	Holding Time	Preservation	Container & Size
Alkaline Phosphatase	24 hours	Cool, 4°C	Plastic, 125 ml
Alkalinity	14 days	Cool, 4°C	Plastic, 125 ml
Ammonia	28 days	Cool, 4°C, pH<2(H_2SO_4)	Plastic, 60 ml
Biochemical Oxygen Demand	48 hours	Cool, 4°C	Plastic, 500 ml
Carbonaceous Biochemical Oxygen Demand	48 hours	Cool, 4°C	Plastic, 500 ml
Chemical Oxygen Demand	28 days	Cool, 4°C, pH<2(H_2SO_4)	Plastic, 125 ml
Chloride	28 days	None required	Plastic, 60 ml
Chlorine, Total Residual	Analyze immediately	None required	Plastic/glass, 250 ml
Chromium VI	24 hours	Cool, 4°C	Plastic, 250 ml
Color	48 hours	Cool, 4°C	Plastic, 60 ml
Fluoride	28 days	None required	Plastic, 500 ml
Inorganic Carbon	14 days	Cool, 4°C	Plastic, 125 ml[2]
pH	Analyze immediately	None required	Plastic, 125 ml
Kjeldahl nitrogen	28 days	Cool, 4°C, pH<2(H_2SO_4)	Plastic, 125 ml
Mercury	28 days	pH<2(HCL)	Teflon, 125 ml
Metals	6 months	pH<2(HNO_3)	Plastic, 250 ml
Nitrate + nitrite	28 days	Cool, 4°C, pH<2(H_2SO_4)	Plastic, 60 ml
Nitrite	48 hours	Cool, 4°C	Plastic, 60 ml
Oil and Grease	28 days	Cool, 4°C, pH<2(H_2SO_4)	Glass, 1 Liter
Organic Carbon	28 days	Cool, 4°C, pH<2(H_2SO_4)	Plastic, 125 ml
Orthophosphate	48 hours	Filter immediately, cool, 4°C	Plastic, 60 ml
Total phosphorus	28 days	Cool, 4°C, pH<2(H_2SO_4)	Plastic, 125 ml
Residue, filterable, nonfilterable, volatile	7 days	Cool, 4°C	Plastic, 500 ml[3]
Silica	28 days	Cool, 4°C	Plastic, 60 ml
Specific conductance	28 days	Cool, 4°C	Plastic, 500 ml[3]
Sulfide	7 days	Cool, 4°C, 20 drops Zinc Acetate+ NaOH to pH >9	Plastic, 250 ml
Sulfate	28 days	Cool, 4°C	Plastic, 60 ml
Turbidity	48 hours	Cool, 4°C	Plastic, 500 ml[3]

(1) From 40 CFR, Part 136, Table II (7-1-90)
(2) Not listed in 40 CFR, Part 136, Table II (7-1-90)
(3) 250 ml plastic bottle may be used if BOD is not requested

Table 6.10 Holding Time and Preservation for Water Organics

Parameter	Holding Time	Preservation[1]	Container & Size
Volatile (Purgeable) Organics	14 days (preserved with 1:1 HCl), 7 days (unpreserved)	Cool, 4°C	Glass, 40 ml, Teflon lined septum
Base neutral acid extractable compounds	7 days until extraction, 40 days after extraction	Cool, 4°C	Amber Glass, 1 L, Teflon lined cap
Organochlorine Pesticides and PCB's	7 days until extraction, 40 days after extraction	Cool, 4°C	Amber Glass, 1 L, Teflon lined cap

(1) If Residual chlorine is present, Sodium Thiosulfate is added to the sample vial first. The vial is then filled to almost full volume with sample, acid is added, and finally the vial is filled as per procedure.
Note: It is not recommended to mix the two preservatives (and sample) together in an intermediate vessel.

Table 6.11 Holding Times and Preservation for Sediments, Soils and Tissues[1]

Parameter	Holding Time	Preservation	Container & Size
Volatile organics	14 days	Cool, 4°C	Glass, 4 oz. wide mouth with Teflon/silicone septum
Semivolatile organics	14 days until extraction, 40 days after extraction	Cool, 4°C	Glass, 8 oz. wide mouth with Teflon/silicone septum (50 grams)
Total metals	6 months	Cool, 4°C	Glass or plastic, 8 oz. wide mouth (200 grams)
Mercury	28 days	Cool, 4°C	Glass or plastic, 8 oz. wide mouth (200 grams)
Nutrients & Inorganics[2]	Not Specified	Cool, 4°C	Glass, 500 ml or plastic, 8 oz. wide mouth with Teflon lined closure

(1) From Table 5, Chapter 62-160, F. A. C.
(2) From USEPA Standard Operating Procedures and Quality Assurance Manual (Appendix A).

Table 6.12 Holding Times and Preservation for Microbiologicals[4]

Parameter	Holding Time	Preservation[2]	Container & Size[3]
Microbiologicals[1]	6 hours	Cool, $4C^0$ /$Na_2S_2O_3$	Glass or Plastic > 125 ml

(1) Parameters included are: Fecal Coliform, Total Coliform and Fecal Streptococci.
(2) Addition of sodium thiosulfate is only required if the sample has a detectable amount of residual chlorine, as indicated by a field test using EPA Method 330.4 or 330.2 or equivalent.
(3) Pre-sterilized Whirlpak bags (or equivalent) are typically used for sampling.
(4) From Table 8, chapter 62-160, F.A.C.

Table 6.13 Holding Times and Preservation for Biologicals

Parameter	Holding Time	Preservation	Container & Size
Chlorophyll	(I) 24 hours to filter (ii) 21 days after filtration	(I) Cool, 4°C, dark, filter with $MgCO_3$, (ii) Filter frozen (until testing)	Amber Plastic, 1 L
Macrobenthic Invertebrates	Preserved in the field with formalin, then ethanol in the laboratory or placed on ice and preserved with formalin within 8 hours, then preserved with ethanol	10% buffered formalin, then 70% ethanol	Glass or Plastic

6.9 Field Reagent and Standard Storage

Table 6-14 lists the storage procedures for reagents, standards and solvents.

Table 6-14. Reagent, Solvent and Standard Storage

Chemical	Method of Storage
Sulfuric Acid	Stored in original containers in vented acid storage cabinet. Note: Each acid is stored in a separate cabinet. Acid is transported in the field in polyethylene dropper bottles.
Nitric Acid	See above.
Phosphoric Acid	See above.
Isopropyl Alcohol	Stored in original containers in vented solvent storage cabinet. Taken into the field in glass containers carried in a safety carrier.

Table 6-14. Reagent, Solvent and Standard Storage (cont.)

Chemical	Method of Storage
pH Standards	Stored in cabinet designated for standard and reagent storage. Cabinet is in air conditioned laboratory. Standards taken into the field in polyethylene bottles.
Conductivity Standards	Stored in cabinet designated for standard and reagent storage. Cabinet is in air conditioned laboratory. Standards taken into the field in polyethylene bottles.
Formalin (10%)	Stored in cabinet designated for standard and reagent storage. Cabinet is in air conditioned laboratory. Taken into the field in polyethylene bottles.
Ethyl Alcohol	Stored in original containers in vented solvent storage cabinet. Taken into the field in approved non-combustible containers.
Sodium Hydroxide	Stored in polyethylene dropper bottles as provided by contract labs. Transported to the field in polyethylene bottles. Stored in cabinet designed for standard and reagent storage.
Zinc Acetate	Stored in polyethylene dropper bottles as provided by contract labs. Transported to the field in polyethylene bottles. Stored in cabinet designed for standard and reagent storage.
Hydrochloric Acid	Stored in original container. Used for field decontamination and to preserve mercury samples in the lab.

7.0 SAMPLE CUSTODY

A verifiable trail of documentation for each sample must be maintained from the time of sample collection through the analytical laboratory to the final reporting or archiving of data.

The purpose of sample custody is to provide a clear description of sample and container traceability from sample collection to final sample disposition and to identify those persons responsible for collection and analysis.

7.1 Documentation

Custody starts with the person who prepares for the field trip. This person, who may be a Staff Scientific Associate, Senior Scientific Associate, Specialist Scientific Associate or Senior Supervising Scientific Associate, obtains the appropriate size pre-cleaned bottles from the bottle storage area. This person signs the trip checklist (Figure 7-1) when all items necessary have been prepared and loaded into the vehicle.

The field sheets become a part of the project records maintained by the project manager with copies retained by the laboratory. Entries on all records, laboratory and field, are made in waterproof ink with errors deleted by crossing one line through them. All field documentation is signed or initialed by the field personnel.

Samples of the documentation are given in Figures 7-1 and 7-4.

Figures 7.2 and 7.3 show the sample tags or labels which include the preservation instructions, bottle size, and the parameters to be tested. Information on the tag or label includes project code, the date and time the sample was taken, and the sample number. The sample number is a unique sequential number for that project. Sample seals are not used by SFWMD.

Figure 7-4 shows the Chemistry Field Data Log used to record the field information. The Chemistry Field Data Log includes the site name or station code, the date and time of sample collection, the signature of the person relinquishing the samples, the field ID number, the number of samples collected, the intended analyses and preservation requirements, a comment section, and a place for the person who receives and logs-in the sample to sign, date, and record the corresponding laboratory sample numbers. Common carrier is not used for samples analyzed by the District laboratory.

The data from the Chemistry Field Data Log is manually entered into the Laboratory Information Management System (LIMS) through the Sample Log-In screens shown in Figure 7-5 and 7-6. This data entry process automatically generates a unique sequential number consisting of the login group hyphenated with the number of the sample for that group. A login group is defined as a group of samples collected for a given project on a given day. This sample number is used to track the progress of the sample through the laboratory.

Figure 7.1

FIELD TRIP PREPARATION AND CHECK-OFF LIST

PROGRAM:_____ **DATE:** _____ **PROGRAM CODE:**_____
(Routine / Quarterly / Bi-Annual)

_____* Cellular Phone
* Sign Out Board
_____* Maps
* Vehicle Packet/Credit Cards
* Sign Out Vehicle
_____* Gas Vehicle
* Maintenance Check
_____* Coolers/Ice

_____* Pre-Cleaned Buckets
_____* Filtration Unit
_____* Processing Tray
_____* Syringes
* Pre-cleaned Sampling Bottle

_____* Calibrated Hydrolab
* Field Notebook
_____* Labels or Tags/Rubber Bands
_____* Field Data Logs
* Waterproof Pens

_____* Acids H2SO4 / HNO3
_____* pH Test Strips
_____* Goggles, Gloves
_____* 5 Gallons DI Water

* **Bottles: 250ml-___, 125ml-___, 60ml- , 250ml/TM-_____, 125ml/Hg-___, 500ml- , 1 liter- ,**

_____* Bottles: One Liter Bottle for Chlorophyll

_____* Stations: Routine / As Specified:_____

_____* Keys:_____

PERSONAL ITEMS

* Watch _____* Sunglasses _____* Drinking Water _____* Hat
* Sunscreen _____* Rain gear _____* Food _____* Mosquito Repellent

POST TRIP PROCEDURES

_____*Check that coolers contain ice and check mark the Asamples in ice" box on the log in sheet
_____* Sort Samples in Sequence and by Label/Tag Color
_____* Place Samples in Refrigerator marked "incoming samples"
* Sign Chain of Custody Sheet/Get Time Stamp
_____* Place Chain of Custody Sheet on the "to be logged in" board
_____* Return Clean Bottles to Lab
_____* Separate Filter Holders and place in Soak Solution
_____* Place Dirty Bottles in Washing area
_____* Replace Water in Hydrolab Stand (Tap Water Only!)
* Clean Out Vehicle
_____* Fill Out Trip Ticket/Return Keys
* Return Field Notebooks to Office
_____* Report Equipment(Vehicle, Hydrolab, etc.) Problems to Supervisor

Figure 7.2
Sample Submission Diagram for Unfiltered Water

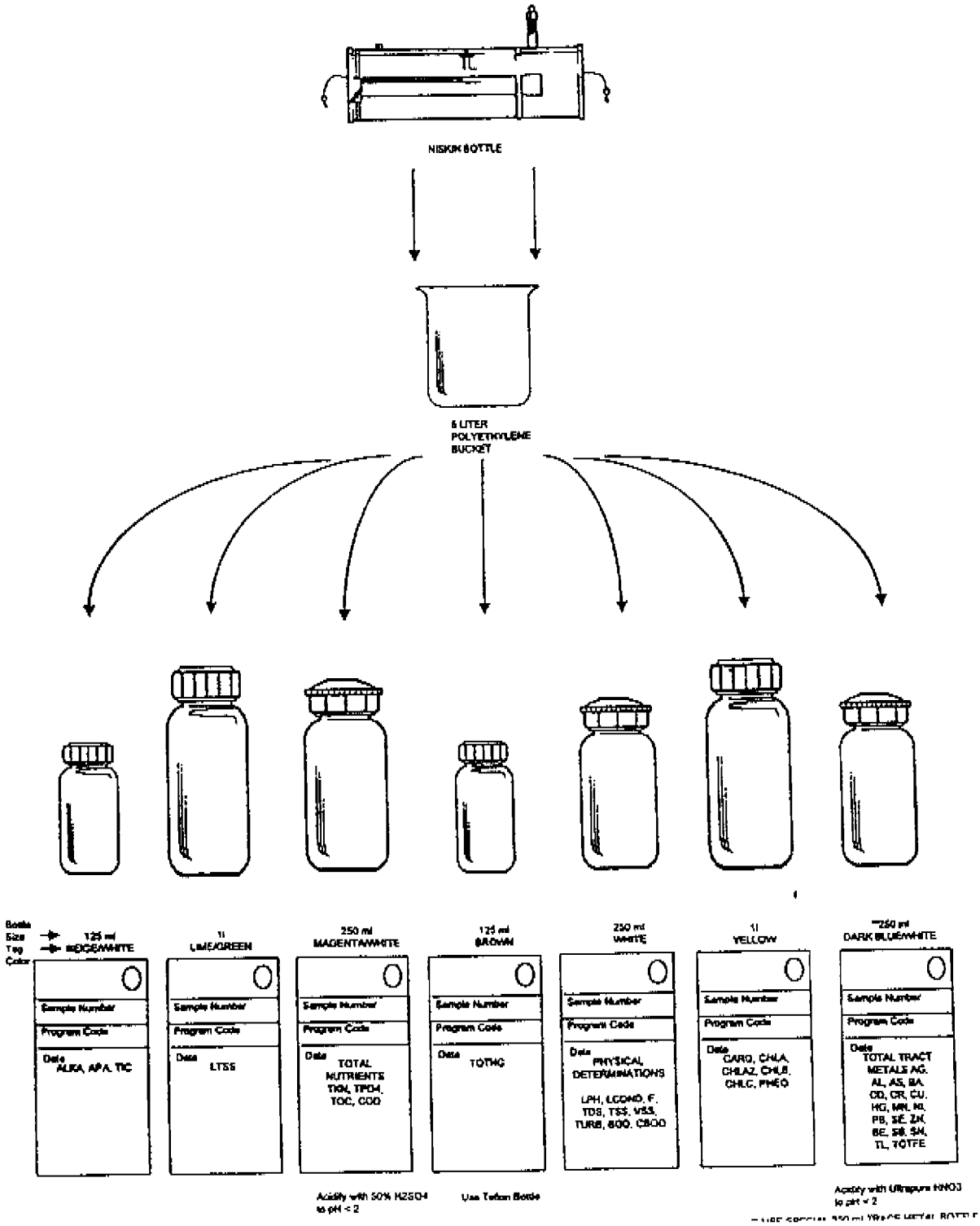


Figure 7.3
Sample Submission Diagram for Filtered Water

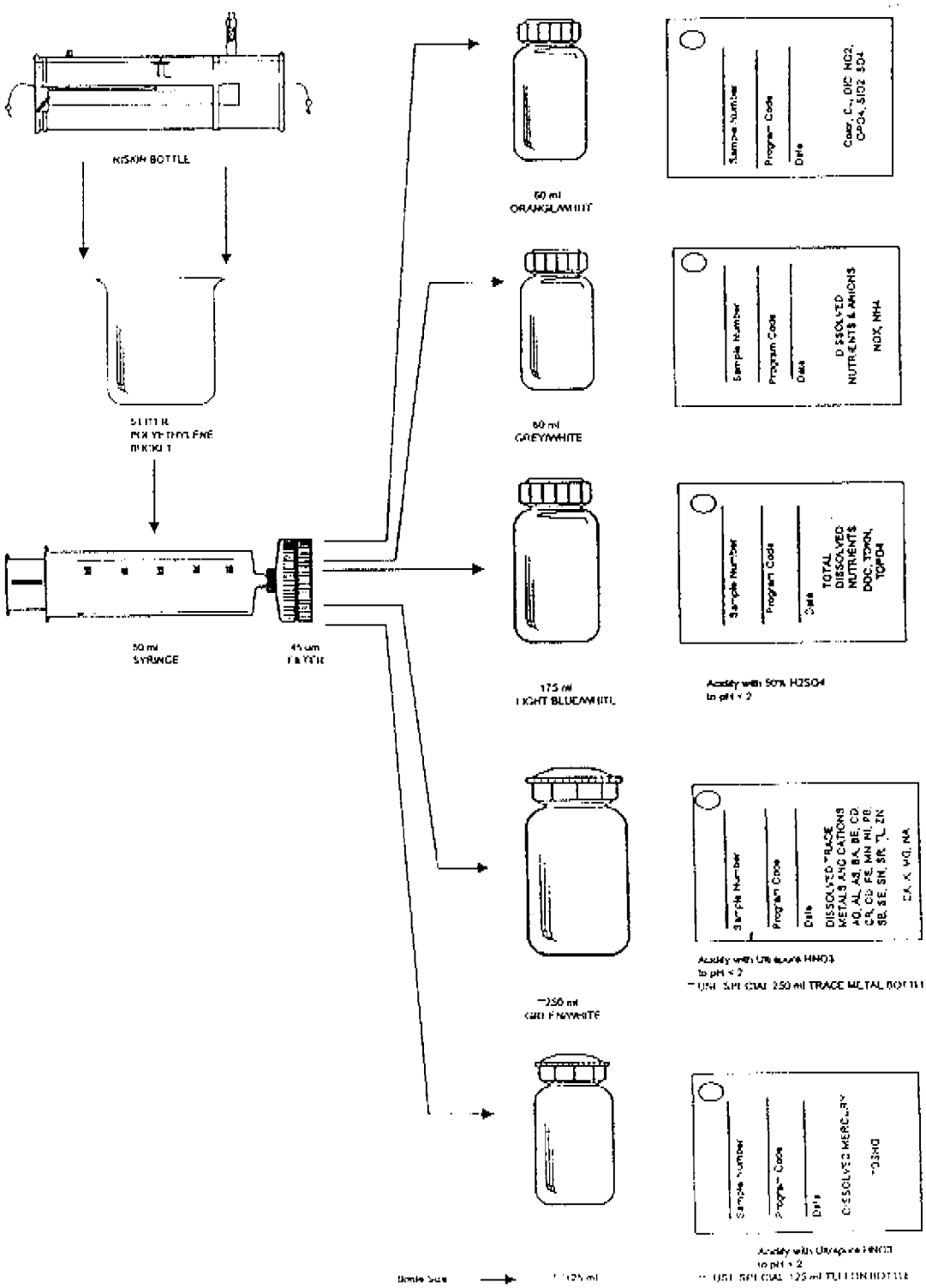


Figure 7.4

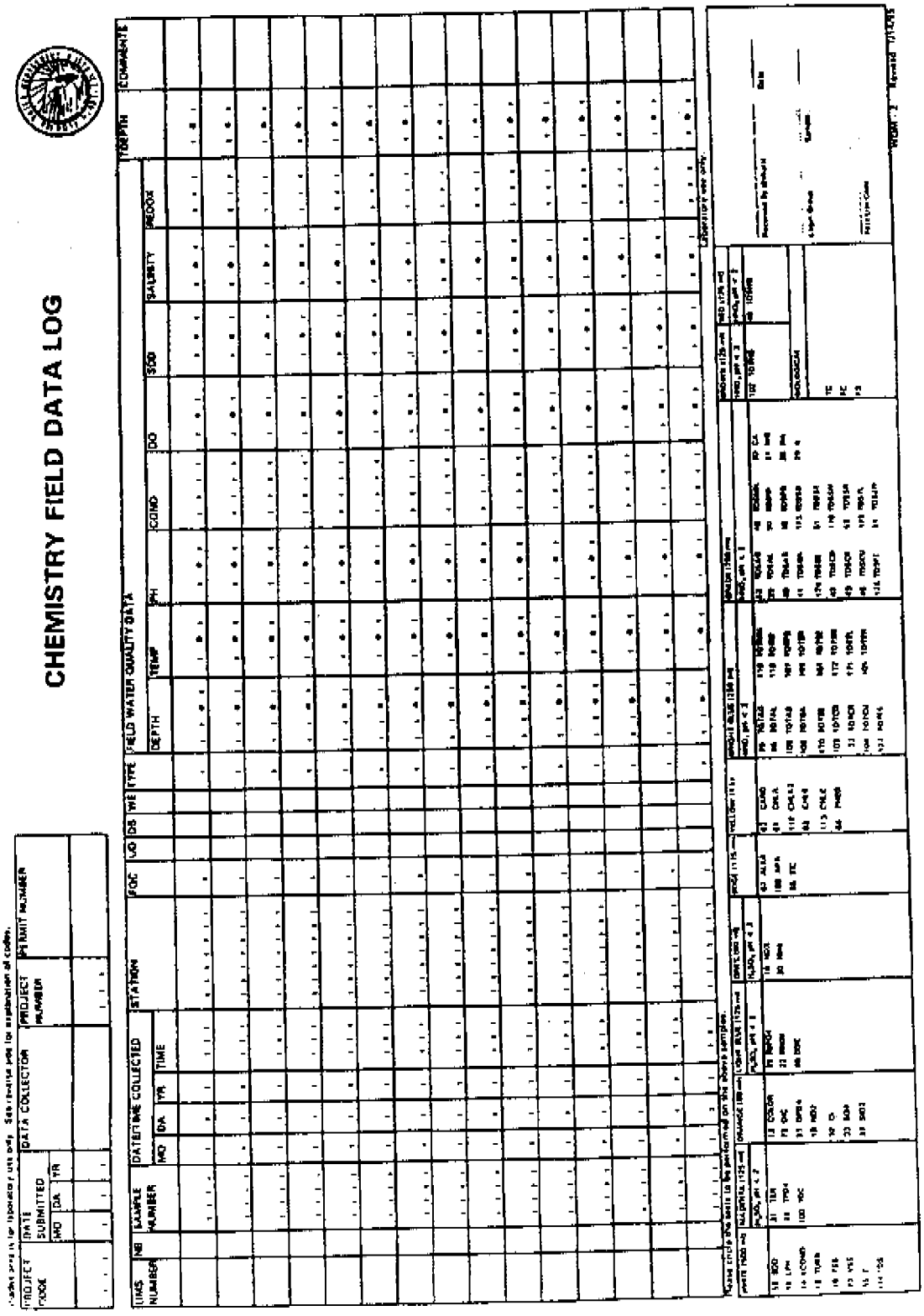


Figure 7.5
Sample Log-In to LIMS

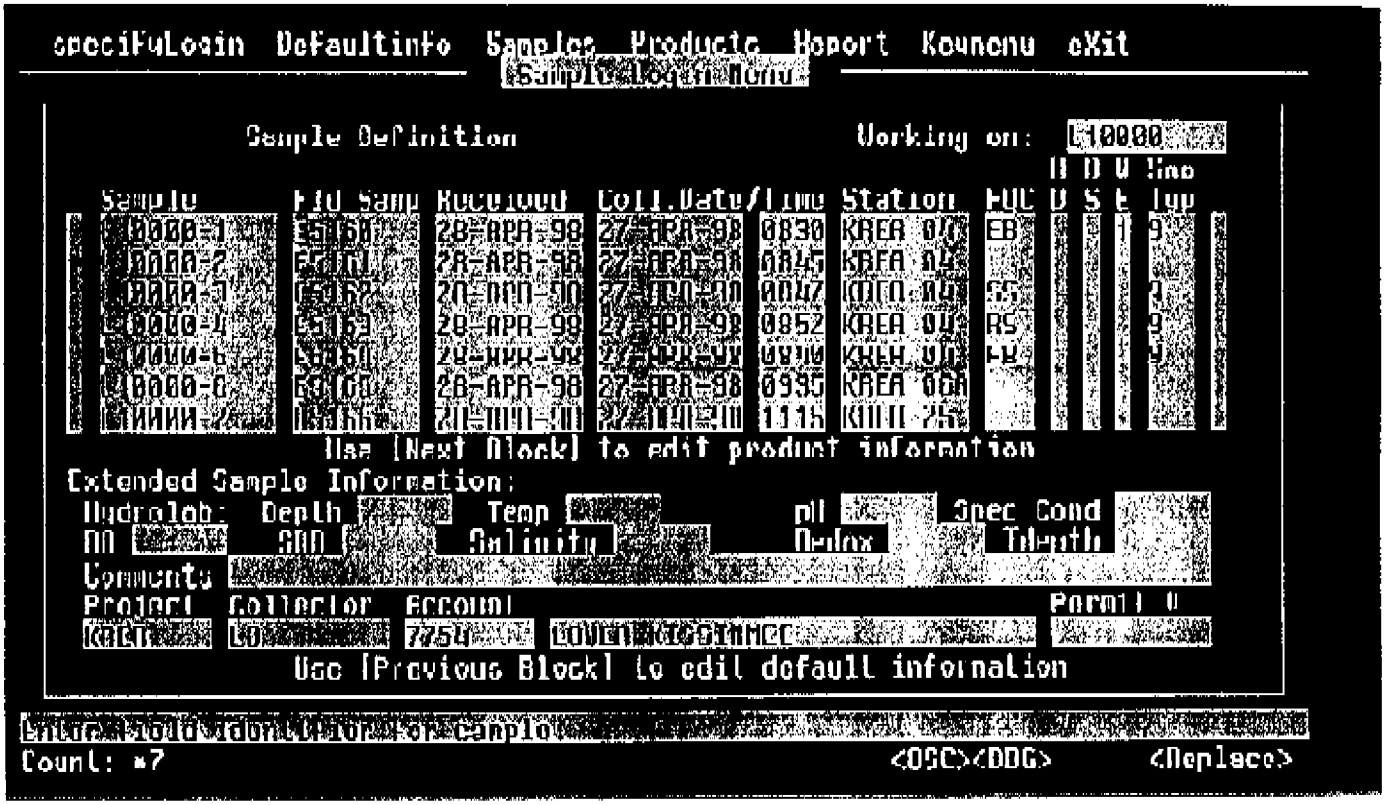
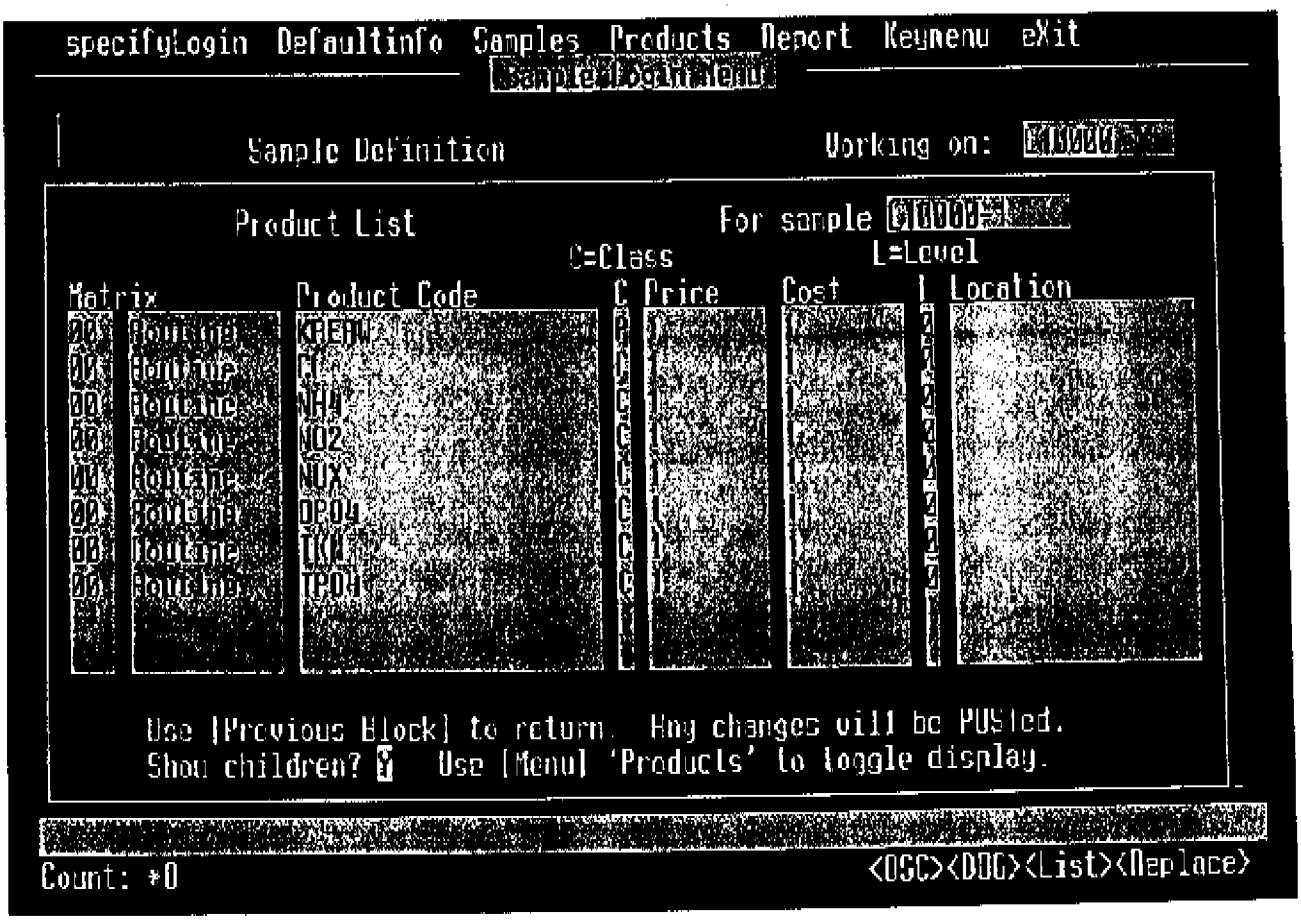


Figure 7.6
Sample Product List



The sample preparation (digestion) records are maintained with the analytical run. The information required is the laboratory sample number, standards, QC samples, dilution factors, person preparing the samples, and the date of preparation. The analysis logs for each type of instrument are shown in Figures 7-7 to 7-13. The microbiology logs are shown in Figures 7-14 to 7-17.

If samples are collected and sent to a contract laboratory by common carrier for analysis, the custody forms supplied by the contract laboratory are used. Only laboratories that have DEP approved comprehensive quality assurance plans are used as contract laboratories. The pre-cleaned sample containers received from the contract laboratory are delivered to the project manager who is responsible for their secured storage.

7.2 Field Custody Protocols

The samples are labeled at the time of collection using waterproof tags or labels which have been filled out with waterproof pens. The tags are attached with rubber bands to the neck of the sample bottle or labels are affixed directly to the sample bottles. Some bottles may come with pre-attached labels already affixed to the bottles which may also be used.

Each project has a unique one to four character project code. The sample field numbers are generated by a combination of this project code and up to a five digit sequential sample number that unequivocally links the collected sample to the time and date of collection, and sampling point. The field sample numbers are associated with the station (site) code on the Chemistry Field Data Log and in the field notebook. The field sample number can be used to trace a sample through the sample tag, the Chemistry Field Data Log, the field notebook, the laboratory information management system (LIMS), and final data archival.

The field records are maintained and stored by the project manager, who is responsible for maintaining all records of the project for the period of time specified by the Florida standards for record management. The field records which are identified by the project code may include the field notebooks and the Chemistry Field Data Logs.

All physical parameter measurements obtained in the field are written on the Chemistry Field Data Log shown in Figure 7-4 at the time of sample collection. These measurements include temperature, pH, specific conductivity, dissolved oxygen, oxidation-reduction potential, Secchi disc depth, total column depth, turbidity and salinity. Other information that must be noted on this form includes project code, collector ID, sample number, date and time of collection, station or site ID, parameter analysis, types of QC samples collected (when and where collected), depth from which sample was collected, flow conditions, requested parameters, and chain of custody documentation. Beginning and ending times of any composite sampling is noted. Optional information on this form includes upstream/downstream notation, discharge information, weather, sample type, and a comments section.

Figure 7.7 Digestion Log

FORM 0217
Rev 10/87

P-E 3600 I.D./W.T. FILE

I.D. / W.T. FILE: _____ SAMPLE WEEK: _____

DATE: _____ DISC. #: _____

ANALYST: _____

PAGE: _____

PARAMETER				
ABSORBANCE				
DATA FILE				

	LIMS NUMBER		PROJECT CODE	SAMPLE NUMBER	DILUTIONS/RERUNS			
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

Figure 7.8

ATOMIC ABSORPTION ANALYSIS LOG

Parameter _____ Number of Samples _____ Date _____

High Standard _____ Operator _____

Detection Limit _____ Method file name _____ Flame Furnace (circle 1)

CALIBRATION INFORMATION

	CONCENTRATION	ABSORBANCE
S1		
S2		
S3		
S4		
S5		

FILE NAMES

ID/WT filename _____

Data filename _____

Name of file transferred to the LIMS _____

Samples _____

Comments/Problems _____

QUALITY CONTROL

QC1 _____ QC2 _____

Accepted Values: _____ _____

Repeat: X = _____

Std. Dev. = _____

Coef. Var. = _____

Stad. Add. % Recovery:

1. _____
2. _____
3. _____
4. _____

FURNACE PARAMETERS

Step	1	2	3	4	5	6
Temp °C						
Ramp (s)						
Hold (s)						
Read						
Rec						
Baseline						
Int. Flow ml/min						

Graphite Tube: _____

Sample Volume: _____

Modifier Vol. _____

Replicates _____

Purge Gas _____

FLAME PARAMETERS

_____ : _____ (gas)

Burner _____

Modifier _____

Figure 7.9

ANALYSIS LOG

Technician _____ Date _____ Std. Cal./Abs./Ret. Time _____

Parameter _____ Method # _____ Instrument # _____

Tray Protocol File Name _____ WG: _____

Comments

_____
_____
_____
_____
_____
_____

Sample Numbers

_____
_____
_____
_____
_____
_____

CALIBRATION:

Channel 1:

_____
_____
_____
_____
_____
_____

Channel 2:

_____
_____
_____
_____
_____
_____

Standards

_____
_____
_____
_____
_____
_____

QUALITY CONTROL:

QC1: _____/_____
QC2: _____/_____
: _____/_____
: _____/_____
STAD: _____
_____
_____

CORRELATION: _____
TEMPERATURE: _____
ELECTRODE EFF: _____
ELECTRODE SLOPE: _____
_____
_____
_____

LIMS#: _____
GAIN: _____

RPT: _____
_____
_____
_____
MEAN: _____
CV: _____
SD: _____

WQM-6 Revised 5-1-96

Figure 7.10

Water Quality Monitoring Division
Calibration Log for Physical Parameters

ANALYST: _____ DATE: _____

PARAMETER: _____ _____

STANDARDS:

Known Value	Instrument Reading
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

REP No. Quality Control Samples:

( ) QC1 True Value	QC2 True Value
( ) _____	( ) _____
( ) _____	( ) _____
( ) _____	( ) _____
( ) _____	( ) _____
( ) _____	( ) _____
( ) _____	( ) _____

REPEAT SAMPLE READINGS

( ) _____	( ) _____	( ) _____	( ) _____
( ) _____	( ) _____	( ) _____	( ) _____
( ) _____	( ) _____	( ) _____	( ) _____
( ) _____	( ) _____	( ) _____	( ) _____

SAMPLES ANALYZED: _____ _____ _____ _____

_____ _____ _____

Figure 7.11

DEPARTMENT OF WATER RESOURCES EVALUATION

Ion Chromatography Log

Analyst : _____ Date: _____

Parameter: _____ Method: _____

Workgroup: _____

Samples Analyzed: _____ _____ _____

_____ _____ _____

_____ _____ _____

_____ _____ _____

Comments: _____

_____

Calibration: Cl SO4

Retention Time: _____ _____

Standards:	Peak Area	Peak Area	
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

OC1 _____ _____

OC2 _____ _____

C V. _____ _____

STAD _____ _____

GAIN _____ _____

Figure 7.12

Department of Water Resource Evaluation
Water Quality Monitoring Division

Carbon Analyzer Log

Analyst:_____ **Date:**_____

Workgroup:_____ **Method: EPA 415.1**

TOC/DOC

Samples Analyzed:

_____
_____
_____
_____

Comments:

_____
_____
_____

Standards:	**Concentration**	**Response**
	100 ppm	_____
	50 ppm	_____
	20 ppm	_____
	0 ppm	_____

Quality Control:

QC1:_____
QC2:_____

SPIKE#1:_____
SPIKE#2:_____
SPIKE#3:_____

REPEAT#1:_____
REPEAT#2:_____
REPEAT#3:_____
REPEAT#4:_____

MEAN: _____
STD DEV: _____
%CV: _____

Figure 7.13

ICP ANALYSIS LOG
standard method 3120b

Analyst:	Date:	Metal:

Method File Name:
LIMS File Name:

Sample List or See Attached

Calibration Curve Correlation Coefficient (r):_____

	Concentration	Emission Counts
Blank	0.0	
Standard 1		
Standard 2		
Standard 3		
Standard 4		
Standard 5		

QC 1_____ Accepted Value _____
QC 2_____ Accepted Value _____

Repeat: Mean: _____ Standard Deviation: _____ %CV: _____
STAD % Recovery 1. _____ 2. _____ 3. _____
4. _____ 5. _____ 6. _____

ICP Parameters:

RF Power: 1400 Neb Flow: 0.7 Aux Flow: 0.4 Pump Rate: 2.0

Comments/Maintenance/Problems

Figure 7.14

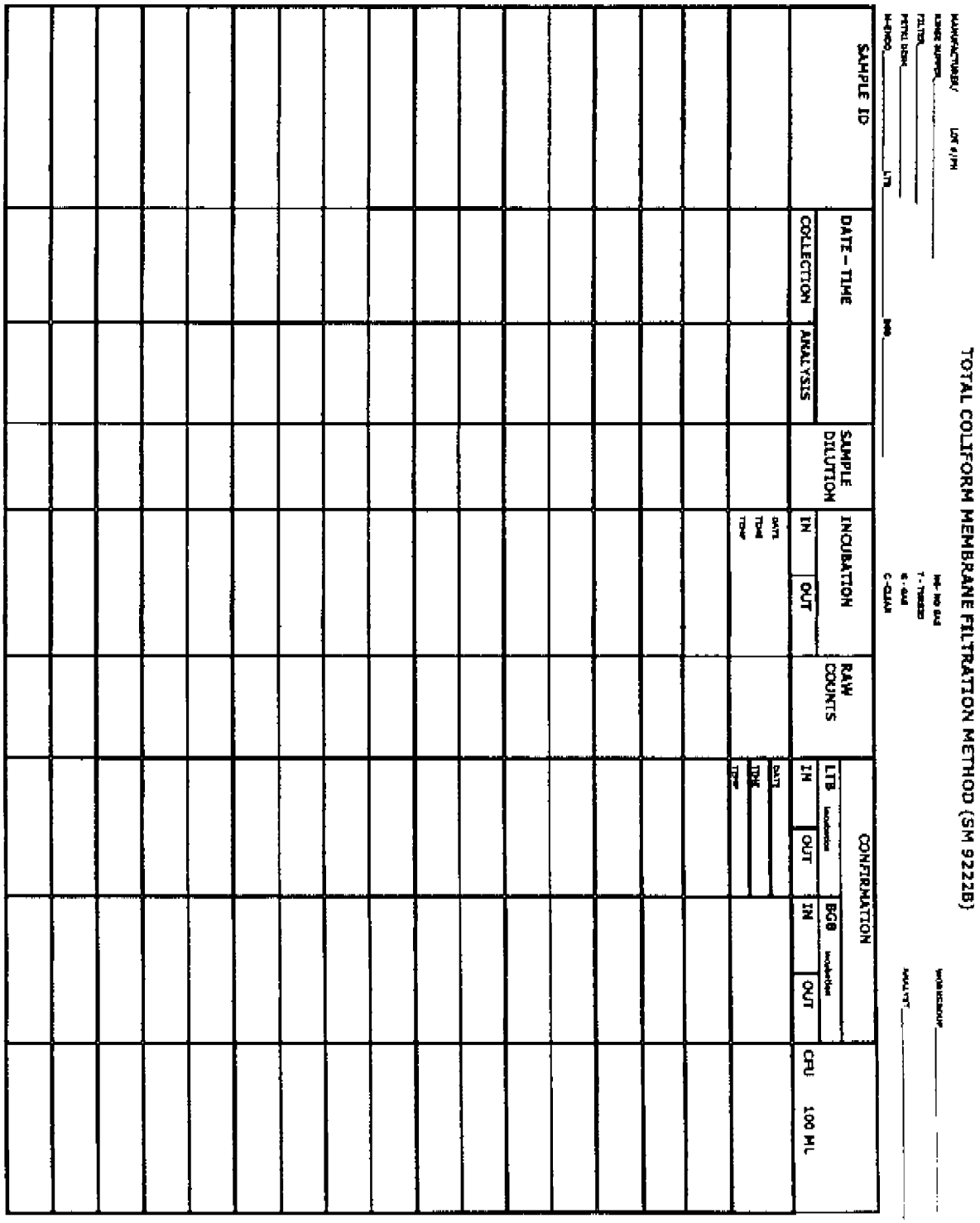

TOTAL COLIFORM MEMBRANE FILTRATION METHOD (SM 9222B)
SAMPLE ID
DATE – TIME
COLLECTION
ANALYSIS
SAMPLE DILUTION
INCUBATION
IN
OUT
RAW COUNTS
CONFIRMATION
LTB
EC
BGB
CFU 100 ML

Figure 7.15

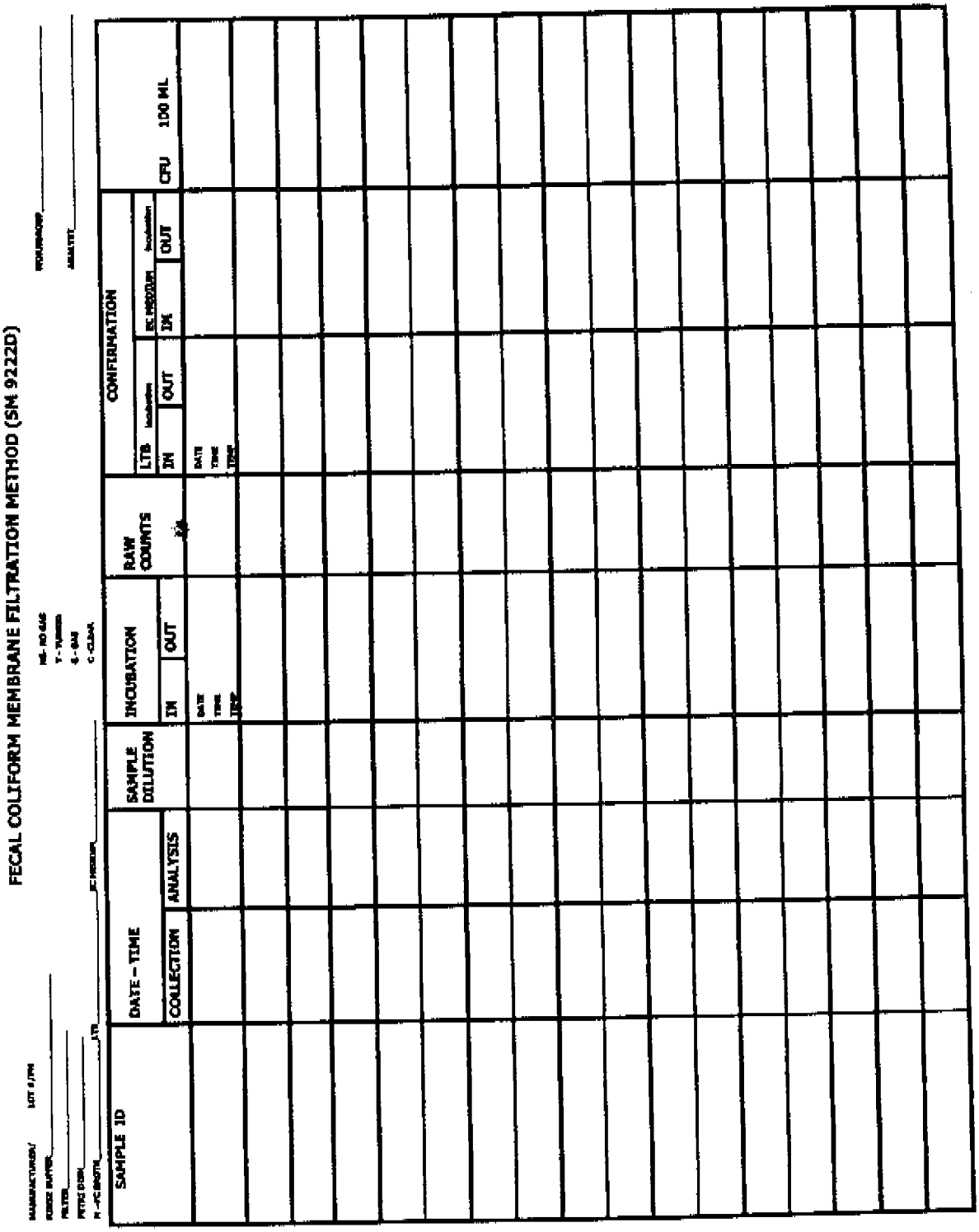
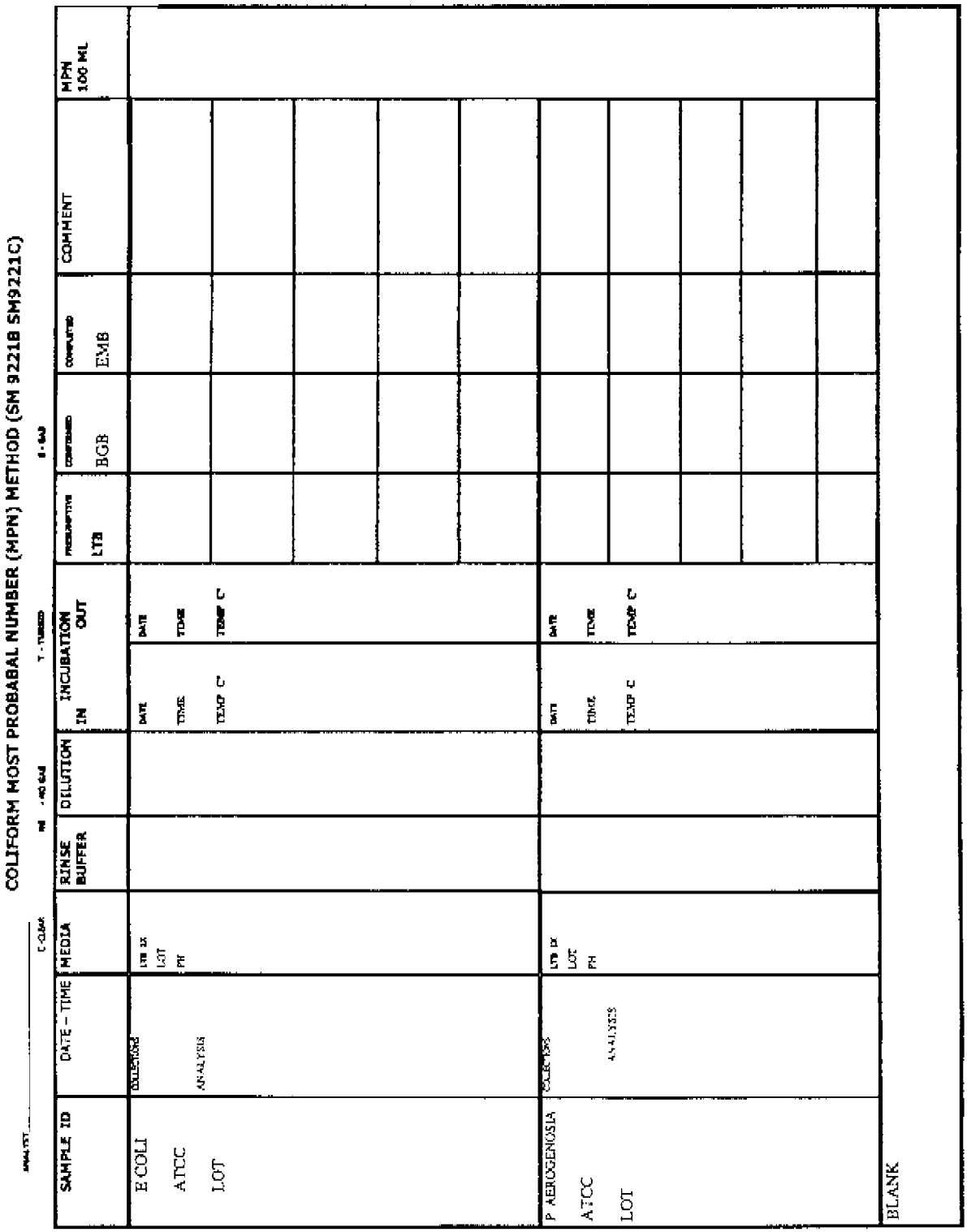

FECAL COLIFORM MEMBRANE FILTRATION METHOD (SM 9222D)
SAMPLE ID
DATE – TIME
COLLECTION
ANALYSIS
SAMPLE DILUTION
INCUBATION
IN
OUT
RAW COUNTS
CONFIRMATION
LTB
IN
OUT
EC MEDIUM
Incubation
IN
OUT
CFU
100 ML

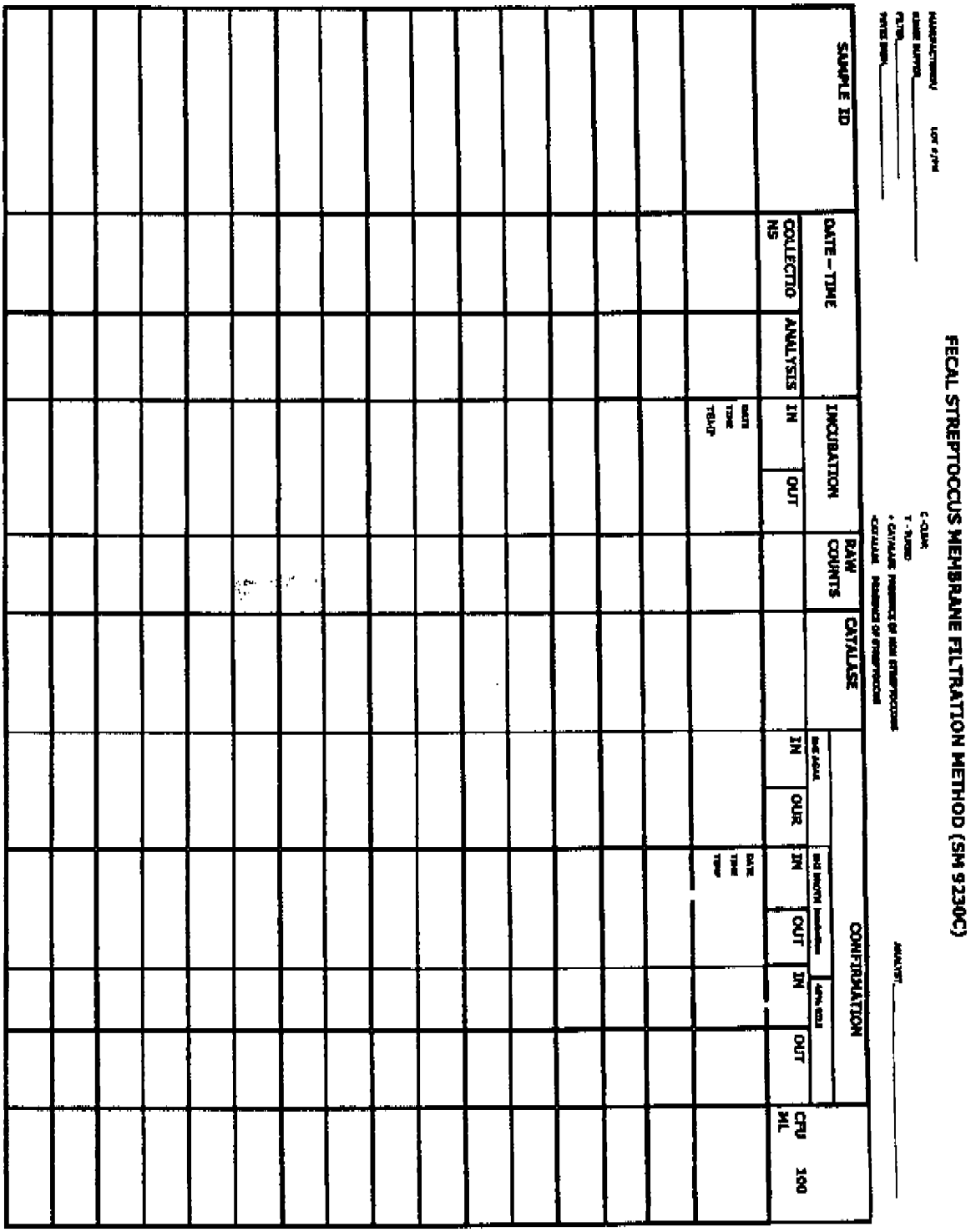
Figure 7.16



COLIFORM MOST PROBABAL NUMBER (MPN) METHOD (SM 9221B SM9221C)

SAMPLE ID	DATE – TIME	MEDIA	RINSE BUFFER	DILUTION	INCUBATION IN	INCUBATION OUT	PRESUMPTIVE LTB	CONFIRMED BGB	COMPLETED EMB	COMMENT	MPN 100 ML
E COLI ATCC LOT	COLLECTED ANALYSIS	LTB ID LOT PH			DATE TIME TEMP C°	DATE TIME TEMP C°					
P AEROGENOSA ATCC LOT	COLLECTED ANALYSIS	LTB ID LOT PH			DATE TIME TEMP C	DATE TIME TEMP C°					
BLANK											

Figure 7.17


FECAL STREPTOCOCCUS MEMBRANE FILTRATION METHOD (SM 9230C)
SAMPLE ID
DATE – TIME
COLLECTIO NS
ANALYSIS
INCUBATION
IN
OUT
RAW COUNTS
CATALASE
IN
OUR
CONFIRMATION
IN
OUT
IN
OUT
CFU
ML
100
ANALYST

Relevant field observations are noted in a bound waterproof notebook at the time of sample collection. These include sample number, station or site name, date and time, weather, flow conditions, water color, water smell, water clarity, weed conditions, number of drops and type of acid added to each bottle, persons other than sampling personnel at the site, type of purging and sampling equipment used with corresponding ID# (if available), field decontamination performed and if applicable, wave height, bottom conditions, algae description, use of boats and/or other fuel powered equipment, calibration information and standards used for calibration.

For monitoring wells, the following information is recorded in addition to any applicable information from the above list: depth to water, calculation for purge volume, determination of volume purged, method of purge, purging rate, date and time the well was purged, and readings taken until the well stabilized.

For wells with in place plumbing or artesian wells, the following information is recorded: plumbing or tap material, flow rate at which the well was purged, time the well was allowed to purge, and the flow rate when the sample was collected.

A monitor well database is kept for all of the wells that are sampled by the SFWMD. This includes the following information: well casing material, well diameter, type of casing, screen diameter, screen type, total depth of the well, casing depth, method of well installation, date of well installation, driller's name, latitude, longitude, measuring point elevation, and land surface elevation. In addition, new wells installed by the SFWMD also have the drilling mud type and name recorded. For sediments, the depth at which the sample is taken is recorded. Drilling/boring information is not used for sampling.

7.2.1 Sample Transport

Following collection of the sample, the bottles are sealed, tagged, and returned to the laboratory for shipment to the contract laboratory along with appropriate sample identification (i.e., sample name and number), and chain of custody form. Samples are packed in coolers with bubble-wrap or other appropriate packing material to avoid breakage. Samples for VOC analysis are placed into a separate bubble-pack bag for each station. Chain of custody forms are enclosed in a plastic bag for protection from water damage. Samples are kept at 4°C with wet ice. Coolers are taped shut using packing tape and taken to the SFWMD shipping area for pickup by common carrier. Coolers are shipped to the laboratory overnight, and the shipping receipts are retained. Chain of custody forms are returned to SFWMD with the analytical results.

7.2.2 Sample Transmittal

The Chemistry Field Data Log and examples of the sample tags are shown in Figures 7.2 through 7.4. The Chemistry Field Data Log includes the field ID number, date and time of sample collection, station (site) code, intended analyses (designated by circling desired parameters in figure 7.4), method of preservation, whether or not the samples are still in wet ice, and limited comments about the sample or sample container.

7.3 Laboratory Operations

The samples are brought to the laboratory by the field sampling personnel and those samples to be analyzed by the SFWMD laboratory are placed in the designated refrigerator. The person bringing the samples to the laboratory signs and stamps the Chemistry Field Data Log with the time clock. The Chemistry Field Data Logs are given to the person responsible for logging-in the sample. The information from the Chemistry Field Data Log is entered into the LIMS and a laboratory sample number is generated by LIMS consisting of a login group hyphenated with the number of the sample within the group. For example, L1001-1 is the first sample of the first login group. The person logging-in the samples records the LIMS numbers on the Chemistry Field Data Log, initials and dates the form at the time the samples are logged (in the "received by" section). The LIMS numbers, date logged, and initials of the logger are also maintained in a bound laboratory notebook. A computer-generated list, verified by the data entry technician, is obtained with the respective field and LIMS numbers for each sample.

Labels for each aliquot are generated by LIMS and are manually attached to the sample field tag or directly to the sample container. As each sample is labeled, the technician checks the proper match of field and LIMS numbers, and examines the sample bottle for leakage, cracks, and any other obvious faults. Five percent of the samples (at least one from each batch) are checked for proper preservation. The results of this check are documented in a bound notebook.

7.3.1 Sample Rejection

Samples are rejected if the bottle is leaking or cracked, if there is no Chemistry Field Data Log submitted with the samples, if the Chemistry Field Data Log is incomplete, if the samples were not brought in wet ice, or if holding times or preservation protocols have been violated.

7.3.2 Sample Security, Accessibility, and Storage

The samples are accessible to the laboratory and field staff during working hours. The doors to the exterior of the building are locked when the last member of the staff leaves for the day. The door to the sample receiving area is opened at 6:00 A.M. The only persons authorized to be in the laboratory are the laboratory staff and the sampling personnel delivering samples. All visitors must be escorted by a member of the division staff.

Samples are stored in refrigerators designated for sample storage only. No VOC's are stored in the laboratory refrigerators and standards are stored in refrigerators specifically designated for standard storage.

Sample digestates are stored in the digestion laboratory until they are analyzed. Analysis takes place within holding times.

7.3.3 Sample Distribution and Tracking

The analysts query the LIMS database daily for samples requiring their assigned analyses and generate work groups for these samples. Analysts are required to analyze reworks and the oldest

samples on each day's first run. The supervisor receives a daily backlog report listing all samples showing incomplete analyses.

The analysts are responsible for removing the required samples from the refrigerators, analyzing them, and returning them to the refrigerators immediately following analysis. Bound notebooks are used to track digested samples. The person doing the digestion lists the samples, standards and QC samples digested in the batch.

Following completion of the analyses and review of the results, the laboratory supervisor or analyst removes the samples which are ready for disposal from the refrigerators. The status of the sample is changed from NEED to DONE in the disposal department in LIMS. Samples are disposed of by diluting and dumping into the sanitary sewer. None of the samples that the SFWMD analyzes are considered hazardous waste samples. Digestates containing hazardous materials, (i.e. the mercury in Kjeldahl nitrogen digestates), are treated as hazardous waste and are collected and stored for disposal by a commercial hazardous waste company.

7.3.4 Interlab Custody

The SFWMD has only one laboratory and does not transfer samples to any other laboratory. However, in the event of catastrophic failure, samples will be transported to a contract lab by common carrier accompanied by the log in sheets and the contract lab's chain of custody.

7.4 Electronic Data Records

A LIMS database is used by SFWMD for sample tracking, data storage, and data reduction.

7.4.1 Security System

The LIMS database has several levels of security. The Staff Programmer Analyst responsible for its operation and maintenance has the highest level of security and can access all information and programs in LIMS. The Division Director, Laboratory QA Officer, Supervising Professionals and Senior Chemists can authorize modification of all sample and results information. The analysts can enter analytical results but cannot modify the sample information or results. The project managers can only read and print results. User names and passwords are assigned to each person. Back up of both the file system and database is performed daily so that in the event of a catastrophe, only one day of data would be lost. One week's worth of system files is stored in the vault.

7.4.2 Forms

Project Managers review weekly data results and identify tests for rework or field sample data for modification. These copies are stored by date produced and are kept according to the record management standards.

7.4.3 Electronic Data Transfer

All requests for copies of the data, electronic or hard copy, are made through the appropriate database personnel in RAD. All data at SFWMD is public information. The project manager is responsible for requesting the data and submitting it to the requestor. For all data transmitted electronically, (e.g. by modem, or diskettes), hard copies are sent via mail following data transmittal. In addition, the District makes data available through an on-line database code named REMO that can be accessed directly by computer modem.

7.4.4 Documentation and Verification

All LIMS documentation is maintained by the Staff Programmer Analyst. Laboratory analysts are responsible for maintaining the documentation records and the maintenance logs. The documentation for all instrument software is located in the laboratory at the instrument. Software problems are included in the maintenance log for the instrument. Software revisions are installed and the records maintained by the Staff Programmer Analyst.

Software is verified by comparing the results generated by the new software to the results from the old software for at least six analytical runs. The evaluation of the results must yield no significant statistical difference. The QC results are also used to show that the software is performing correctly.

Data entry is verified by comparing the results obtained by the instrument software to the results entered into the database. Manual data entry is verified by comparing the results in the database to the results on the data entry forms.

All original hard copies of analytical runs are kept in the QC unit filing area. Any division staff requesting these documents must sign them out and return the documents promptly to the same location.

THIS PAGE DELIBERATELY LEFT BLANK

8.0 Analytical Procedures

The analytical procedures used by SFWMD are listed in Section 5.0.

8.1 Field Screening Methods

A phosphorus screening method is used in the field to determine samples which may be exceeding established limits. The screening data is used only to determine which samples should be sent to the laboratory for testing (see appendix C). The criteria used to determine which samples are to be sent to the overflow lab are project specific.

8.2 Laboratory Glassware Cleaning and Storage Procedures

For physical parameters and nutrients, the laboratory glassware is cleaned by washing with a Lab grade detergent solution, rinsing with D.I. water, 10% hydrochloric, and finally D.I. water. Once air or oven dried, glassware is stored capped, in cabinets, in the appropriate analytical or digestion area.

Digestion tubes, beakers and other pieces of glassware are washed in a labware washer which has been programmed to follow the above procedure except for the acid rinse. The program is stopped after the first tap water rinse, the glassware is removed from the washer and rinsed with 10% hydrochloric acid and then returned to the washer for the final tap and analyte free water rinses. The glassware is then allowed to either air dry or is placed in a glassware dryer. Once dry, the glassware is stored in clean drawers in the appropriate analytical or digestion area. TKN digestion tubes are not dipped in acid.

A complete supply of glassware is dedicated for use in the metals laboratory. Pipettes are soaked in 1% lab grade detergent (Liquinox™ or equivalent), and cleaned in a pipette washer using three volumes of D. I. water followed by soaking in 10% HNO_3 for 48 hours minimum , and washing with three volumes of analyte free water. Volumetric flasks and glassware dedicated to major cation analyses are rinsed three times with analyte free water after each use, air dried, and stored in cabinets in the appropriate analytical or digestion area. Glassware dedicated to trace metals is rinsed with analyte free water after each use, soaked in 20% HNO_3 bath, rinsed with analyte free water, allowed to air dry, and stored separately from other glassware in the metals analysis area. Polyethylene, glass, or Teflon trace metal bottles are soaked in 20% nitric acid for a minimum of 24 hours, analyte free water rinsed, and stored in cabinets in the metals area filled with analyte free water which has been acidified to approximately 1% with double distilled nitric acid.

8.3 Laboratory Method Modifications

The color procedure has been modified for use in the laboratory. The samples submitted to this laboratory are from natural surface and ground water sources within the boundaries of the SFWMD. The color in the samples is due primarily to vegetative decay and not from industrial sources. Measurement of the color at 465 nm gives results comparable to those measured visually by technicians. Use of the spectrophotometer eliminates the natural variation in color perception found

in the human eye allowing the lab to consistently report results regardless of which technician performs the analysis. The procedure used is given in Appendix A.

The adopted silica method is a modified method tested by Alpkem for use in Rapid Flow Analyzers. The adopted method reduces saltwater interferences and is found in Appendix B.

Alkaline phosphatase activity is measured using the Methylumbelliferyl Phosphate (MUP) assay (Petterson and Jansson, 1978). The procedure is included as Appendix D.

Total and dissolved inorganic carbon (TIC/DIC) is determined using a high temperature combustion infrared detection system following the EPA Method 415.1 for total organic carbon. The procedure is included as Appendix F.

8.4 Laboratory Reagent Storage

The storage of the laboratory reagents and chemicals is given in Table 8-1.

Table 8-1. Reagent and Chemical Storage

Chemical	Method of Storage
Mineral acids	Stored in original containers in vented cabinet designed for acid storage. Note: each type of acid is stored in a separate cabinet.
Liquid bases	Stored in original containers in a vented cabinet designed for corrosive storage.
Organic solvents	Stored in original containers in a vented cabinet designed for flammable storage in the outside storage area.
Compressed gases	Stored in original containers in the compressed gas storage area in the outside storage area.
Dry chemicals	Stored in original containers segregated by reactivity in the dry chemical storage area.

As each chemical is received, it is dated and initialed by the person unpacking it. When a new container is opened for use, it is dated and initialed by the person who opened it.

8.5 Waste Disposal

The laboratory has a designated hazardous waste storage area outside the laboratory. The process wastes containing mercury and phenol are collected for disposal by a hazardous waste company. As each waste is generated, the volume is entered on a monthly hazardous waste report. When the

volume collected reaches a specific level set by Risk Management, the waste is removed by a commercial waste hauler and disposed of according state and federal guidelines.

Any small amounts of reagents are transferred to the hazardous waste storage area as they expire or are no longer needed. The waste disposal company then picks them up for proper disposal.

Concentrated acids and bases are neutralized or diluted in the laboratory then poured down a drain with copious amounts of water; diluted acids or acid solutions are poured directly down a drain into the lab drainage system with copious amounts of water where the waste passes through a neutralization tank on the way to the sanitary sewer system. Samples are disposed of by washing them into the sanitary sewer system with copious amounts of water. Nitric acid is disposed of according to local, state and federal regulations.

THIS PAGE DELIBERATELY LEFT BLANK

9.0 Calibration Procedures and Frequency

Table 9.4 indicates the calibration procedures and frequency used for the laboratory instrumentation; Table 9.5 lists the calibration procedures and frequency used for field instruments.

9.1 Instrumentation Lists

The following is a list of the laboratory instrumentation.

Manufacturer	Model and Description
Hach	Model 2100 AN Turbidimeter Model 18900 Ratio Turbidimeter Model 45600 COD Reactor
Fisher Scientific	Model 50 Ion Analyzer pH Electrodes Model AB 15 pH Meter Model 21K/R Marathon Centrifuge Model 307A BOD Incubator Model SPT - IH Stereoscope
Orion	Combination Fluoride Electrode Model 162 Conductivity Meter Model 960 Autochemistry System pH Electrode with ATC
Mettler	P160 Top Loading Balance AE163 Analytical Balance (2)AE100 Analytical Balance
Bausch & Lomb	Spectronic 501 Visible Spectrophotometer
Dionex	4000i Ion Chromatograph DX500 Ion Chromatograph
Alpkem	(3)RFA300 Rapid Flow Analyzer with PC workstation (3)RFA500 Rapid Flow Analyzer with PC workstation
Perkin Elmer	1100B Flame Atomic Absorption Spectrophotometer Z5100 Furnace Atomic Absorption Spectrophotometer Optima 3000XL ICP Spectrometer Lambda 6 UV-VIS Spectrophotometer
Shimadzu	Model 5050A Total Organic Carbon Analyzer

9.1 Instrumentation Lists (cont.)

Manufacturer	Model and Description
Rosemount Dohrmann	DC-190 Total Organic Carbon Analyzer
Millipore	Cytofluor 2350 Fluorescence Measurement System
Barnstead	Model 2250 Autoclave
Reichert	Quebec Dark field Colony Counter
Precision	Model 6M Incubator Model 251 Coliform Water Bath
YSI	Model 59 DO Meter Model 5905 DO Probe
Thermolyne	(4) Model 9000 Oven Furnatrol II Muffle Furnace Type 37900 Culture Incubator
CEM	MDS-2100 Microwave Digester
Tecator	(2) Digestion System 40, 1016 Digester
Ultra Lum	Ultra Violet Digestion Cabinet
Lachat	Quickchem 8000 Mercury Analyzer Quickchem 8000 Automated Ion Analyzer (2) BD-46 Digestion Blocks
Powers Scientific	(2) Refrigerator
Amerikooler	Refrigerator
Jordan	Refrigerator
Market Forge	Autoclave

9.1 Instrumentation Lists (cont.)

The following is a list of the field instrumentation.

Manufacturer	Model and Description
Hydrolab	Model 4031 pH, Conductivity, ORP, and Temperature Meter Model 4041 pH, Conductivity, DO, and Temperature Meter Surveyor II pH, Conductivity, DO, Temperature, Salinity, Surveyor III pH, Conductivity, DO, Temp., Salinity ORP, Turbidity and Depth Meter Surveyor 4 pH, Conductivity, DO, and Temperature Meter
Solomat	WP803
YSI	Model 58 Dissolved Oxygen Meter Model 6000UPG Multiprobe pH, Conductivity, DO, Temperature,
EH	Turbidity Model 600XL Multiprobe pH, Conductivity, DO, Temperature, EH
HF Scientific	Model DRT-15CE Turbidity Secchi Depth Disc
Licor	Spherical Quantum sensors

9.2 Standard Receipt and Traceability

Standards are received by the Senior Chemists, Senior Laboratory Analysts or Staff Laboratory Analysts, initialed, dated, and stored in the designated area for the particular standard.

The preparation dates of in-house primary stock solutions are recorded in a log book along with the following information regarding purchased stock solutions: analyte, concentration, supplier, date opened, expiration date and date of disposal. Only one bottle of each purchased analyte stock solution may be in use at one time. Purchased stock solutions are replaced according to expiration date or sooner if the stock is depleted. Manufacturer's certificates of analysis and/or records of traceability for purchased stock solutions are filed in a notebook according to analyte or analytical category. Records of the preparation are kept in a logbook. Working calibration standards are prepared fresh daily. The standard sources and preparation are given in Tables 9-1and 9-2.

The calibration procedures for laboratory instruments are given in Table 9-3, for laboratory equipment in Table 9.4, and for field instruments in Table 9-5. QC check standards from a different source than the calibration standards are used to check the initial calibration for both laboratory and field instruments.

Calibration information for the field is recorded in the Field notebook or Calibration Book. Date and time of calibration, technician, standards used, standard results and temperature, and instrument used are recorded.

Calibration information for the laboratory is recorded in the individual instrument log and includes the analyst, date of analysis, standard values, millivolt or absorbance values for the standards, correlation coefficient, results of continuing and initial calibration standards, precision and accuracy results, and samples analyzed.

Table 9.1: Standard Sources and Preparation

Instrument Group	Standard Sources	How Received	Source Storage	Preparation from Source	Lab Stock Storage	Preparation Frequency
Atomic Absorption/ Emission	Spex/JT Baker/ NIST/EM Scientific	Solutions of 1000 mg/L and 10,000 mg/L	Room temperature	Primary stocks (>1 mg/L) prepared from source Working stocks	0.2% HNO3 at room temp. NA	Weekly or as needed Daily/Weekly
Continuous Flow & Ion Chromatograph	Commercial lab supplier	Dry, ACS reagent grade	Room temperature	Primary stocks, 1000 mg/L prepared from source Working stocks	Refrigerator NA	Monthly Daily/Weekly
Organic Carbon/ Inorganic Carbon	Commercial lab supplier	Dry, ACS analytical grade	Room temperature	Primary stocks, 1000 mg/L prepared from source Working stocks	Refrigerator NA	Monthly Daily/Weekly
Alkaline Phosphatase	Sigma Chemicals	Dry, Enzymatic grade	Room temperature	Primary stocks, 10 micro Molar in Tris Buffer Working stocks	Room temperature NA	Monthly Daily/Weekly
pH Standards	Commercial lab supplier	pH 4,7,10 solutions	Room temperature	NA	NA	Replace on expiration
Conductivity Standards	Commercial lab supplier	200, 720, 1413, 2000 uS	Room temperature	NA	NA	Replace on expiration
Turbidity	Hach	Sealed Gel Standards	Room temperature	NA	NA	Annual replacement
Color	Commercial lab supplier	500 Pt-Co units	Room temperature	Working stocks	NA	Weekly
Analytical balances	Commercial lab supplier	Class S weights	Dessicator, room temperature	NA	NA	NA
Mercury	Commercial lab supplier	10 mg/ml NIST/NBS traceable standard	Room temperature	Primary stocks 100mg/l & 1000ug/l prepared from source Working Stocks	Room temperature Room temperature	Weekly or as needed Daily

Table 9.2: Solutions Requiring Standardization

Test	Standard sources	How Received	Source Storage	Preparation from Source	Standardization procedure and Criteria	Standardization Frequency	Preparation Frequency
Alkalinity Titration	Commercial Lab Supplier	0.02 N Sulfuric Acid	Acid Storage Cabinet	Used as is from supplier (certified)	Normality checked with 0.05 N sodium carbonate; must be within 5% of expected value	Weekly and each time a new lot is used	Used as is from Supplier

Table 9.3: Laboratory Equipment Calibration

Equipment	Calibration	Acceptance Criteria	Frequency
Analytical Balance	Calibrated with the 100g weight, then checked with entire set of Class S weights	All weights within 2 % of known value	Daily
	Maintenance contract exists for calibration	NA	Semi-Annually
Autoclave	Check timer with stopwatch	Timer set for 15 min. maintains 121°C for at least 15 min. The entire cycle is completed within 45 min.	Quarterly
	Autoclave Tape/Strips	Indicates acceptable sterilization obtained	Monthly
	Spore check	No visible spores	
Incubator	Temperature recorded from a calibrated thermometer. Adjustments made as needed	+/- 1 °C	Twice daily
Water Bath	Temperature recorded from a calibrated thermometer. Adjustments made as needed	+/- 0.2 °C	Twice daily
Mercury Thermometer	Checked with a NIST(NBS) certified thermometer	+/- 0.5 °C	Semi-Annually
Digital Thermometer	Checked with a NIST(NBS) certified thermometer	+/- 0.1 °C	Quarterly

Table 9.4: Laboratory Instrument Calibration

Instrument	# Standards Initial Calib.	Accept/Reject Criteria - Initial Calibration	Frequency	# Standards Continuing Calibration	Accept/Reject Criteria - Cont. Calibration	Frequency
Atomic Absorption/ Emission	3-5	Corr. Coefficient >0.995	Daily prior to use or failure of cont. calibration	1-5	Concentration within 5% of known value (mid-range)	Every 20 samples
Continuous Flow	5 – 7	Linear Regression Corr. Coefficient >0.998	Daily prior to use or failure of cont. calibration	1-7	Concentration within 5% of known value (mid-range)	Every 20 samples
Silica only	5 - 7	Quadratic Regression Corr.	(same)	1 - 7	(same)	
Ion Chromatograph	5	Corr. Coefficient > 0.998	Daily prior to use or failure of cont. calibration	1	Concentration within 5% of known value (mid-range)	Every 20 samples
Carbon Analyzer	3	Concentration within 5% of known value	Daily prior to use or failure of cont. calibration	1-3	Concentration within 5% of known value (mid range)	Every 20 samples
pH Meter	3	pH 7 = 0+/-5 mV; pH 4 = 177+/-10 mV; Eff = 1.00+/-0.05	Daily prior to use or failure of cont. calibration	1-3	Concentration within 5% of known value (mid-range)	Every 20 samples
Conductivity Meter	3	Concentration within 5% of known value	Daily prior to use or failure of cont. calibration	1-3	Concentration within 5% of known value (mid-range)	Every 20 samples
Turbidimeter	3	Concentration within 5% of known value[1]	Daily prior to use or failure of cont. calibration	1-3	Concentration within 5% of known value (mid-range)	Every 20 samples
Visible Spectrophotometer	5	Linear Regression Corr. Coefficient >0.998	Daily prior to use or failure of cont. calibration	1	Concentration within 5% of known value (mid-range)	Every 20 samples
Fluorometer	5	Linear Regression Corr. Coefficient >0.998	Daily prior to use or failure of cont. calibration	1-3	Concentration within 5% of known value (mid- range)	Every 20 samples

(1) Gel standards are checked monthly with Formazin control solutions of the same concentration, (i.e. 1.8, 18.0 and 180 NTU), to ensure integrity.
Note: Method Calibration requirements will be followed if more stringent than those listed in the QAP.

Table 9-5. Field Instrument Multi-parameter Instruments (Hydrolabs & YSI's)

Instrument Probe	# Standards Initial Calibration	Accept/Reject Criteria-Initial Calibration	Frequency	# Standards Continuing Calibration	Accept/Reject Criteria-Continuing Calibration	Frequency
PH	2 (1 pH 7 & 1 pH 4 or 10)[3]	Reading within 0.1 pH unit	Daily prior to use or failure of cont. calibration	1	Concentration within 5% of known value	At the end of the day or within 24 H of initial calibration
Conductivity	1 in the expected Range[4]	Concentration within 5% of known value	Daily prior to use or failure of cont. calibration	1	Concentration within 5% of known value	At the end of the day or within 24 H of initial calibration
Dissolved Oxygen	Winkler titration	Concentration within 5% of known value	Annually	Saturated air	Concentration within 5% of known value	At the end of the day or within 24 H of initial calibration
Temperature	1	Temperature within ±0.5 °C of known value Thermometer calibrated to NIST thermometer	Daily	1	Temperature ± 0.5°C of NIST thermometer	Quarterly[5]
Turbidity	1	Reading within 5% of known value	Daily	1	Concentration within 10% of known value	Daily prior to use
Automatic Samplers	Correct sample volume verified by using graduated cylinder	Volume within 5% of programmed volume	Daily prior to use	NA	NA	Daily or weekly prior to deployment of instrument.

(1) The Units all have automatic temperature compensation for pH, conductivity and DO measurements. Meters are checked with NIST traceable thermometers
(2) Buffer pH 4 or pH 10 solution used, respectively, with acidic or basic samples.
(3) The calibration check is conducted weekly for instruments deployed in the field
(4) YSI recommends calibration at 1000us/cm. A standard in the expected range of the samples is then checked for the 5% compliance
(5) The thermometer that is used in the daily check is either purchased as certified to an NIST thermometer or checked by comparing a low temperature reading (~10°C) and a high temperature reading(~40°C) quarterly. If the difference is more than 0.5°C, the thermometer is discarded. If the difference is constant at the two readings and <0.5°C, the thermometer is used with the application of a correction factor.

10.0 Preventative Maintenance

Preventative maintenance is a necessary part of a successful quality assurance program. Time must be allocated to clean and maintain all equipment used for the collection and analysis of a sample. Equipment which is not operating properly may give unreliable results.

10.1 Equipment Maintenance

Field maintenance procedures are outlined in Table 10-1; laboratory maintenance procedures are outlined in Table 10-2. Maintenance that cannot be performed by SFWMD personnel is done by the manufacturer or its designee, and several of the instruments are covered by manufacturer's service contracts.

10.2 Maintenance Documentation

The field equipment maintenance activities are documented in bound notebooks assigned to each instrument. Service reports for repairs that cannot be done by SFWMD personnel are kept on file by the Technician Supervisor.

The laboratory equipment maintenance activities, as listed in Table 10-2, are documented in a separate bound notebook for each instrument. Service reports for repairs that cannot be done by SFWMD personnel are kept on file in a notebook located at the instrument.

10.3 Contingency Plans

The SFWMD has replacements for most critical instruments which can be used in the event of a breakdown. Instrument service contracts are maintained on all major pieces of equipment and response time is typically 48 hours. Overflow laboratories are contracted to provide analytical services in the event of a catastrophic failure.

Table 10-1. Field Equipment Maintenance Schedule

Instrument	Specific Activity	Frequency
Hydrolabs (all models)	DO probe membrane and electrolyte changed	Quarterly/AN
	Conductivity sensors are sanded with emery cloth	Quarterly
	pH and reference electrodes cleaned with methanol	Quarterly/AN
	pH reference electrode refilled with 3M KCl	Quarterly/AN
	All outside surfaces cleaned and rinsed with analyte free water	Daily
Y S I Multiprobe Instruments	PH/Redox combination probe cleaned with Isopropanol, cotton swab and rinsed w. D.I. water	Monthly/AN
	D. O. membrane and electrolyte solution changed	Monthly/AN
	D. O. sensors sanded w/ provided sanding disk	As Needed
	Conductivity probe cleaned w/ dilute Liquinox & soft brush, rinsed thoroughly w/ D. I. Water	Monthly/AN
Pumps (Gorman Rupp 2IIP Centrifugal)	Check oil and add if needed	Before use
	Drain pump of water	After use
	Wipe clean of mud and grease	After use
	Change oil & filter	Quarterly
	Change spark plugs & adjust carburetor	Quarterly
Autosamplers	Check battery charge & replace as needed	Before use
	Check programming	Before use
	Check pumping volume with a graduated cylinder	Before use
	Check indicating desiccant & change as needed	Before use
	Change pump tubing	Quarterly
	Calibrate Autosampler	Quarterly
	Check distributor arm tubing replacement to make sure it delivers sample directly to bottle	Quarterly
	Liquid sensor cleaned	Quarterly
	Intake tubing strainers cleaned	Quarterly

Aerochem Collectors	Check temperature of sensor plate by touching Remove & cap collection buckets Apply a few drops of water to sensor plate to check lid operation Check for snug fitting lid over collection bucket Check temperature of sensor plate after operation to see if warm to the touch Wipe top and bottom of lid & air dry Install clean collection buckets	Before use Weekly Before use Before use Before use Before use Weekly

Table 10-2. Laboratory Equipment Maintenance Schedule

Instrument	Specific Activity	Frequency
Atomic Absorption	Check gases	Daily
	Service maintenance	Semiannually
	Flame: Nebulizer cleaned ultrasonically in Liquinox™ solution, rinsed with tap water, dipped in 10% HNO_3, rinsed with DI water.	Biweekly
	Burner head soaked in Liquinox™ solution.	After use
	Furnace: Windows inspected and cleaned with isopropyl alcohol.	Daily
	Tubes and platforms inspected and changed	AN
	Furnace decontamination as recommended by Perkin Elmer	Weekly/AN
Atomic Emission / ICP	Check pump & system tubing	Daily
	Inspect Torch & RF coil for deposits or moisture and clean if necessary	Daily
	Inspect filters	Monthly

Instrument	Specific Activity	Frequency
Atomic Emission (cont.)	Clean nebulizer	Biweekly
	Flush torch with 5% HNO_3 then DI	After use
	Pump air through spray chamber	After use
Continuous Flow -Alpkem RFA300 -Alpkem RFA500	Inspect all tubing and fittings	Daily
	Wash manifold/flow cell	Daily
	Inspect filters	Weekly
	Replace pump tubes	Biweekly
	Clean rollers & grease	Monthly
	Service maintenance	Semiannually
Ion Chromatograph	Check tubing and fittings for leaks	Daily
	Clean columns and change bed supports	Monthly
	Preventative maintenance by manufacturer	Semiannual
Carbon Analyzer	Check/replace O-rings	Weekly/AN
	Change acid	When 2/3 empty
	Replace copper & glass wool	When discolored
	Replace injection port septa	Weekly/AN
	Inspect/replace combustion tube and catalyst	Biweekly
pH Meter	Rinse electrode with DI water	Before & after use
	Add reference solution	Daily/AN
Conductivity Meter	Rinse electrode with DI water	Before & after use
Dissolved Oxygen	Rinse probe with DI water	Before & after

Instrument	Specific Activity	Frequency
Meter	Probe membrane and electrolyte changed	use Quarterly/AN
Turbidimeter	Clean cuvettes	Daily
	Adjust calibration	AN
Visible Spectrophotometer	Clean flowcell / cuvette	Daily
	Change pump tubes	Semiannual
Fluorometer	Calibration Service & inspection	Annually
Analytical Balances	Clean weighing compartment	After each use
	Clean interior/exterior	Monthly
	Calibration check against class "s" weights	Monthly/ AN
	Calibration service & inspection	Semiannually
Ovens	Check temperature	Daily
	Calibrate thermometer to NIST thermometer	Semiannual
Refrigerators	Check temperature	Daily
	Calibrate thermometer to NIST thermometer	Semiannually
Digestion blocks/ COD Reactor	Check temperature	Weekly
	Clean blocks	Monthly
	Calibrate thermometer to NIST thermometer	Semiannually
Centrifuge	Clean holder	After use
	Clean walls	After use
Autoclave	Check water level	Before use
	Clean interior and replace water	Before use

Instrument	Specific Activity	Frequency
	Check pressure during operation	With each use
Colony Counter	Adjust focus and brightness	With each use
BOD Incubator	Check temperature	Twice daily
Incubator	Check temperature	Twice daily
Water Bath	Check temperature	Twice daily
	Change water	Monthly
Stereoscope	Replace bulb	AN
	Wipe lens	AN
Lachat QuikChem® Mercury Analyzer	Clean surfaces	Daily
	Clean rods/moving parts	Monthly
	Flush pump tubes	Daily
	Replace pump tubes	Bi-Weekly/AN
	Clean detectors	Daily
	Check discoloration of Perma Pure Dryer	Semiannually
	Replace inner membrane of Perma Pure Dryer	Annually
Ultra-Lum UV Digester	Wipe down interior and exterior	Daily/AN
MDS – 2100 Digester	Check door, seals and cleaning	Monthly
	Wipe microwave cavity	Monthly
	Clean cavity exhaust outlet	Weekly

AN - As Needed

11.0 Quality Control Checks, Routines to Assess Precision and Accuracy, and Calculation of Method Detection Limits

The determination of the quality of an analysis is dependent on the use of quality control check samples in the field and laboratory. The definitions of each type of check sample can be found in DER QA-001/90, Appendix C.

11.1 Field Quality Control Checks

The field quality control checks are given in Table 11.1. The DEP QC procedures confirm the precision of the sampling techniques, that the equipment is clean and addresses the effects of the sample handling and transport. All blanks are preserved and transported in the same manner as the samples.

The field quality control check samples consist of the following:

A. Field Blank - a deionized water sample poured directly into the sample container on site, preserved and kept open until sample collection is completed for the routine sample at that site.

B. Replicate Sample – Two or more samples collected at the same time from the same source.

C. Split Sample – Two or more samples that are taken from the same sample collection event, in essence splitting one sample into two or more portions; all bottles are filled from the same sample collection device or sample composite. Split samples are usually collected for inter-laboratory comparison with one part of each portion of the split sent to each laboratory in the comparison study. Splits may also be collected and submitted to one laboratory to assess analytical precision.

D. Equipment Blank - after field cleaning of sampling equipment, the final deionized water rinse is collected and analyzed as an equipment blank. Equipment blanks are collected before sample collection begins, and at a rate of one every twenty samples thereafter, although they may be collected more often according to the requirements of the sampling event. Equipment Blanks are prepared by pouring one liter of DI water into the sample collection container and through each piece of sampling equipment. For trips requiring more than one liter of water, the volume required to fill the sample bottles may be used. This volume will vary by project and must be documented in the field logbook. The Equipment Blank is filtered, preserved and handled as a routine sample.

E. Trip Blank - Analyte free water blank (VOCs only) prepared before sample containers are transported to the field. Trip blanks remain unopened and are handled in the same manner as the samples.

Table 11.1 Field Quality Control Checks

Type	# Samples per Trip	Frequency (All Parameter Groups)
Equipment Blank, Pre-cleaned Equipment.	>20	1 blank prior to sampling on-site and 1 blank for every 20 additional samples (or portion of 20 samples)
	1-20	1 prior to sampling, on-site
Equipment Blank, Field Cleaned Equipment.	>20	1 blank for every 20 samples taken from field cleaned equipment
	5-20	1 blank on field cleaned equipment
	<5	1 on either pre-cleaned or field cleaned equipment
Trip Blank (VOC only)	1 or more	1 for each volatile organic method per cooler used to transport samples
Field Duplicate/Replicate	1 – 10	1 field duplicate/replicate + 1 split
Split Samples	11-20	2 field duplicates/replicates + 2 splits
	21-30	3 field duplicates/replicates + 3 splits
	>30	1 for every 10 samples
Field Blank	1 – 20	1 field blank
	21-40	2 field blanks
	>40	1 for every 20 samples
Field Measurements QC Check Standards. (multi-parameter instruments only)	1 or more	1 at the end of the day or within 24 hours of initial calibration
Field Measurements (Single parameter instruments)	1 or more	Every 4 hours and at the end of the day

The field quality control check samples described in A and C are included for each group of samples collected for each event at a rate of 5%. These samples are submitted to the laboratory with the routine samples for that event. Equipment blanks (D) are prepared on-site before sampling begins and at a rate of 5%.

Replicate samples are submitted at a rate of 10%. Trip blanks (E) are submitted with volatile organics (one in each cooler) and are provided by contract labs or prepared when sample containers are prepared.

11.2 Laboratory Quality Control Checks

The laboratory quality control checks are listed in Table 11.2. These requirements are minimum standards for the operation of the laboratory; additional QC checks may be performed to further assess the operation of individual procedures, or if requested by a supervisor, the QC unit, or the DEP or DHRS.

11.3 Species Identification

The SFWMD maintains the following in-house specimen collections for species identification:

1. Herbarium
2. Estuarine larval fish, zooplankton and benthic invertebrates
3. Phytoplankton (photographic reference record from the Caloosahatchee River)

Plant species and freshwater fish identification are done in-house. Verification of identifications by outside experts is done on an as needed basis.

Estuarine species identification is done in-house. A reference sample is made for each species collected. The reference samples and 5-10% of the samples collected are set aside for identification by an outside expert.

Species identification for freshwater invertebrates is done in-house using an in-house type specimen collection. The reference samples and 5-10% of the samples collected are set aside for identification by an outside expert. Ten percent of all sorting and identification is confirmed by a different SFWMD staff member. Counts should agree within 10%.

Table 11.2. Laboratory Quality Control Checks

Type	**Frequency (All parameter groups)**
Method Reagent Blank	1 per sample set (batch)
Matrix Spikes (spike added prior to sample preparation)	1, and 1 additional for every 20 samples if more than 20 samples are analyzed; if more than one matrix, 1 from each matrix.
Quality Control Check Samples	Blind Performance Evaluation Samples- analyzed in duplicate at least semiannually*
Quality Control Check Standards	Analyzed at the beginning of each analytical run to verify standard curve. One QC is also analyzed at the end of the analytical run.
Duplicate Samples	1, and 1 additional for every 20 samples if more than 20 samples are analyzed; if more than one matrix, 1 from each matrix.
Continuing Calibration Standard	At a rate of 1 for every 20 samples in an analytical set (at least one in each batch is at a concentration of 1-2 times the PQL).

* If blind QC data is not acceptable, results are reported to DEP in the QA Report.

The following QC checks are done for each microbiology test.

Table 11.3 Microbiology QC Checks

QC Check	Frequency	Purpose	Acceptance Criteria
Autoclave tape and indicators	with each sterilization batch	assure complete sterilization	Tape writing visible and dark
Spore check	Monthly	assure complete sterilization	No viable cultures
Incubator temperature check	twice/day	maintain proper temperature	35.0 +/- 0.5 ° C
Water bath Temperature check	twice/day	maintain proper temperature	44.5 +/- 0.2 ° C
Thermometer calibration with NIST thermometer	semi-annually	assure accurate temperature readings	< 0.2+/- ° C correction required

11.4 Laboratory Quality Control Checks (Microbiology)

11.4.1 Laboratory Quality Control Checks for all Microbiology Tests

QC checks for microbiology include: (see also Table 11.3)

Annual Water Quality Test (Suitability Test) - Standard Methods 9020
Inhibitory Residue Test for each new lot of detergent (on file) - Standard Methods 9020
Monthly Heterotrophic Plate Count - Standard Methods 9215B
Annual Metals Test - Standard Methods 9020
Monthly Chlorine Residual - Standard Methods 9020
Monthly Conductivity - Standard Methods 9020

All materials such as filters, plates, Whirlpaks™ and media are checked for sterility using a non-selective broth followed by incubation for 24 hours at 35.0 +/- 0.5°C. Bound log books are used to record phosphate rinse buffer and media preparation and sterility checks, autoclave cycles and checks, temperature checks, equipment maintenance, and sample analysis.

Each filtration series or sample set is run with a control blank at the beginning, end, and with every ten samples. Analysis duplicates are run every ten samples. Each new lot of dehydrated or commercially prepared media is checked before use with positive and negative culture controls. Ten percent of all positive samples are counted by different technicians, and duplicate counts must agree within 5%.

11.4.2 Laboratory Quality Control Checks for Membrane Filtration Procedures

A control blank is run at the beginning and end of each filtration series. In addition, a carry over blank is run every ten samples. The log sheets used for the membrane filtration procedures are found in Figures 7.14, 7.15, 7.16, and 7.17.

11.4.3 Laboratory Quality Control Checks for Most Probable Number Procedure (MPN)

With each MPN analysis, ten ml of sterile phosphate rinse buffer is used as a blank control with LTB media. The completed test is performed on ten percent of all coliform positive samples.

11.5 Routine Methods Used to Assess Precision and Accuracy

11.5.1 Definitions

Accuracy can be defined as the agreement between the actual obtained result and the expected result. QC check samples having a known or "true" value are used to test for the accuracy of a measurement system.

Precision can be defined as the agreement or closeness of two or more results and is an indication that the measurement system is operating consistently and is a quantifiable indication of variations introduced by the analytical system over a given time period.

11.5.2 Reportable Data for Field QC Samples

The formulas used to calculate the precision and accuracy of the QC checks are:

Percent Relative Standard Deviation for precision of routine, split and replicate samples.

$$\%RSD = \frac{S}{X} * 100$$ where S = Standard Deviation, and X = mean

Relative Percent Differences for precision of duplicates.

$$RPD = \frac{abs[A - B]}{A + B} * 200$$ where A and B are the analytical values for the two duplicate samples

The quality control data is kept in table format with updates calculated at least annually. The laboratory staff is given the control limits for ready reference as samples are analyzed. The formulas for calculating control limits are based on the standard deviation of at least 6 measurements and preferably 20 measurements for each type of sample. The standard deviation is calculated according to the following formula:

$$s = \left[\frac{\Sigma(X_i - X)^2}{(n-1)} \right] 1/2$$

where X is the mean, X_i is an individual value, and n is the number of values.

$$\text{Mean} = X = \frac{\Sigma X_i}{n}$$

The limits for field parameters are 2s for warning limits and 3s for control limits.

11.5.3 Reportable Data for Laboratory QC Samples

The precision and accuracy of each parameter are measured on a daily basis. The field blanks, splits,

and replicates are analyzed as routine samples.

Accuracy may be quantified by comparing results obtained for QC check samples to their true values and calculating a percent recovery using the following equation:

$$\% \text{ Recovery} = \left[\frac{\text{Experimental}}{\text{Known}}\right] * 100$$

The values obtained for a matrix spike are used to calculate percent recovery using the following equation:

$$\text{Percent Recovery} = \%R = \left(\frac{[\text{matrix spike}] - [\text{sample}]}{[\text{spike}]}\right) X\ 100$$

Percent recovery values may be used as an indication of bias. The target control limits for accuracy are calculated for each parameter or are a default value of " 10% of the known true value.

To determine the precision analyses performed in the SFWMD laboratory, one sample is chosen at random from each group of 20 samples as the repeat (or replicate) sample for each parameter. Each replicate sample is then analyzed twice during the analytical run, and the precision of the analysis is calculated from the precision of the replicate determinations analyzed during the run using the following equation:

$$RPD = \frac{abs[A - B]}{A + B} * 200$$

where A and B are the analytical values for the two duplicate samples

On a daily basis, the results obtained for each of the quality control checks used are compared to the acceptable limits for precision and accuracy. Target limits are set based on prior performance or at a default value of " 10% of the known true values.

The procedures used to determine precision and accuracy are given in Table 11.4.

11.5.4 Method Detection and Practical Quantitation Limits

The method detection limits (MDLs) are determined by the procedures in 40 CFR Part 136, Appendix B. They are updated annually and when necessary due to equipment or procedural changes. See tables 5.1, 5.2 & 5.3 for current MDLs.

The practical quantitation limit is 12 times the pooled standard deviations derived from the procedures to determine the method detection limit, or roughly 4 times the MDL, and is updated annually.

Table 11.4 Procedures Used to Determine Precision and Accuracy

Method	Purpose	Concentration Level	Method References
Matrix Spike	Accuracy	Low Level Mid Level High Level	Nutrients, Trace Metals, Anions, Cations
Duplicates	Precision	Low Level Mid Level High Level	All parameters
Replicate for analytical run	Precision	Mid Level High Level	All parameters
QC Check Samples (PE)	Accuracy	Low Level Mid Level High Level	All parameters
QC Check Standards (QC)	Accuracy	Low Level Mid Level High Level	All parameters for which a sample is available
Method Reagent Blank	Accuracy	Low Level	Total Nutrients and Total Metals
Mid-Range Check Standard	Precision and Accuracy	Mid Level	Nutrients, Trace Metals, Anions and Cations

12.0 Data Reduction, Validation and Reporting

12.1 Data Reduction

12.1.1 Field Reportable Data

All field measurement data are directly read from the instruments. These measurements include pH, specific conductance, dissolved oxygen, temperature, ORP, salinity, turbidity, and depth. The data is automatically temperature compensated for pH, specific conductance, and dissolved oxygen. The cell constant for specific conductance is determined by the manufacturer. The field technician does not perform any calculations on field data.

The technician responsible for data entry inputs all field data from the Chemistry Field Data Log into the computer.

12.1.2 Laboratory Reportable Data

The laboratory measurements which are read directly from the instruments, requiring no calculations, are pH, specific conductance, turbidity, color and fluoride. The pH and conductivity meters have automatic temperature compensation. The conductivity cells are purchased with cell constants provided by the manufacturer and are checked monthly. No calculations are performed using a single point reference or internal standard. Table 12.1 shows the formulas used to calculate specific parameters.

The analyst (Assistant Laboratory Technician, Laboratory Technician, Senior Laboratory Technician, or Chemist) is responsible for running the computer programs which provide the results in the appropriate concentrations and entering those results into the database.

The documentation for the results include the strip chart and chromatogram recordings and/or raw data files for computerized calculations. The strip charts and chromatograms are identified with the date, the computer file name if applicable, and the initials of the analyst. The computer files are named using the instrument code, date, and sequential file number for the day.

12.2 Data Validation

12.2.1 Laboratory Data Integrity

The Analysts/Supervisors are responsible for checking the raw data entries and calculations for correctness. The Laboratory Quality Assurance Officer is responsible for checking sample preparation logs, and instrument/analytical logs for adherence to QC protocols and sample identification. The QA Officer is also responsible for checking the calibration integrity by comparing the present instrument responses to the historical values, plotting the instrument responses, and providing the results to the Laboratory Director and Laboratory supervisors.

12.2.2 Field Data Integrity

The Quality Assurance Officer for each division is responsible for checking calibration integrity by checking the calibration logs and comparing present values to historical values and the sample custody integrity by checking the paperwork to ascertain that only trained personnel collected samples and that they were preserved and transported correctly. The Project Manager is responsible for checking raw data entries and calculations by reviewing the records for accuracy and use of proper formulas.

12.3 Specific Data Validation Procedures

12.3.1 Laboratory Data Validation

The analyst (Asst. Lab Technician, Lab Technician, Senior Lab Technician, or Chemist) is responsible for the first step in the validation process. It is his/her responsibility to follow the procedures correctly, perform the quality control checks, and report any discrepancies to his/her supervisor and/or the Laboratory Quality Assurance Officer. The Senior Chemists are responsible for investigating the discrepancy and determining the cause. The Senior Chemists are also responsible for the review of all data to identify obvious anomalies. The Laboratory Quality Assurance Officer is responsible for reviewing the quality control results for each run, and insuring that all QC criteria are met. The Quality Assurance Officer must update the acceptable quality control limits for all parameters quarterly.

Laboratory quality control checks are used to validate the laboratory results. Each of the QC check samples will be discussed.

Method Reagent Blank - if the result is greater than the detection limit, the run is stopped and the blank is prepared again. All samples since the last acceptable blank are reanalyzed.

Matrix Spike Sample - if the result is outside the current acceptable limits, the sample will be prepared again. If the value is outside the range again, a matrix problem is suspected and it is noted in the database that the sample exhibits matrix interference.

Quality Control Check Standards - if the result is outside the current acceptable limits, the run is stopped and the instrument re-calibrated. If necessary, new calibration standards are prepared and the instrument is checked for leaks, cracks in tubing, correct reaction temperature, correct wavelength or filter, and correct calculation procedure in the computer.

Blind Quality Control Check Samples - if the results for these blind samples are incorrect, the entire procedure is checked for errors. The analytical results are reported in the quality control report.

Replicate or Duplicate Samples - used to assure that analytical precision is maintained throughout the analytical run. At least one replicate is run for each analytical run. If more than 20 samples are analyzed in an individual analytical run, one replicate, chosen at random from each group of 20 samples, is run for each group of 20 or fraction thereof. Field duplicates and splits are treated as

individual samples and are not considered analytical duplicates.

Continuing calibration standard - used to confirm that the calibration curve remains constant throughout the analytical run. Continuing calibration standards are run at the rate of at least one standard for each 20 samples or fraction thereof. The value obtained for the calibration standard, either measured as a concentration or instrument response, must remain within 5% of the initial value throughout the run.

12.3.2 Field Data Validation

The field sampling personnel (Scientific Technician, Senior Scientific Technician, Technician Supervisor, or Staff Environmental Scientist) are responsible for following the sampling procedures, reviewing the Chemistry Field Data Logs, and filling out all forms correctly and completely. The Technician is responsible for reviewing field data submitted to the laboratory for data entry for accuracy, initial data review following analysis, and review of the field quality control results for adherence to established standards. The Staff Environmental Scientist or Senior Environmental Scientist is responsible for reviewing all data for his/her project(s) to assure that the data quality objectives for the project(s) are being met.

The following field quality control standards are used to validate the field collected data and the sample collection process:

Quality Control Check Standards - used to check the calibration of the instruments on a continuing basis. Results must be within the established acceptable limits.

Equipment Blanks - used to measure the effectiveness of sample equipment decontamination.

Field Blanks - used to measure the amount of environmental contamination in the samples.

Field Duplicates - used to measure the precision of the sample collection process.

Field Splits – used to measure the precision of the analytical process following the act of sample collection; generally used to evaluate inter-laboratory performance between two or more laboratories.

Trip Blanks - (for VOC only) used to measure shipping/handling contaminants and problems.

12.3.3 Project Data Validation

The Project Manager is responsible for the final review of data. The Laboratory Quality Assurance Officer is responsible for review of the laboratory QC data and the field quality control data. The Project Manager is responsible for review of all supporting documentation and the review of the data for anomalous results.

12.4 Data Reporting

Following the data validation, the results are either manually entered or sent directly from the laboratory instrument to the LIMS data base by the Analysts (Analysts, Senior Lab Analyst, Staff Lab Analyst or Chemist). The Senior Chemists check entered data by means of automated computer programs to ensure detection of aberrant data (e.g., $NO_3^- > NO_x^-$) to avoid its inclusion into final reports. All reports are generated electronically from this data base by the Water Quality Monitoring Division Programmer Analyst. Hard copy reports are routinely generated for all electronically transmitted data.

All requests for data reports must be made through the Data Reporting Unit in WRE. An Example of a Report from LIMS is given in Figure 12-1.

12.5 Data Storage

The records that will be retained are the strip charts, chromatograms, data files, Chemistry Field Data Logs, manual data entry records, daily QC reports, instrumentation logs and LIMS back up tapes.

The hard copy information is retained in the laboratory for no more than one year. After that it is sent to warehouse records storage and will be microfilmed if storage for longer than three years is needed.

The magnetic tapes used for weekly back-up of the LIMS are maintained in two sets. One set is kept in an in-house vault for immediate access and one set is sent to secure storage outside the SFWMD for recovery in the event of a catastrophic event.

Archived records are indexed based on date for hard copy laboratory records, and by project for field notebooks and results in the water quality database. The Project Manager is responsible for the storage of all project data for at least three years.

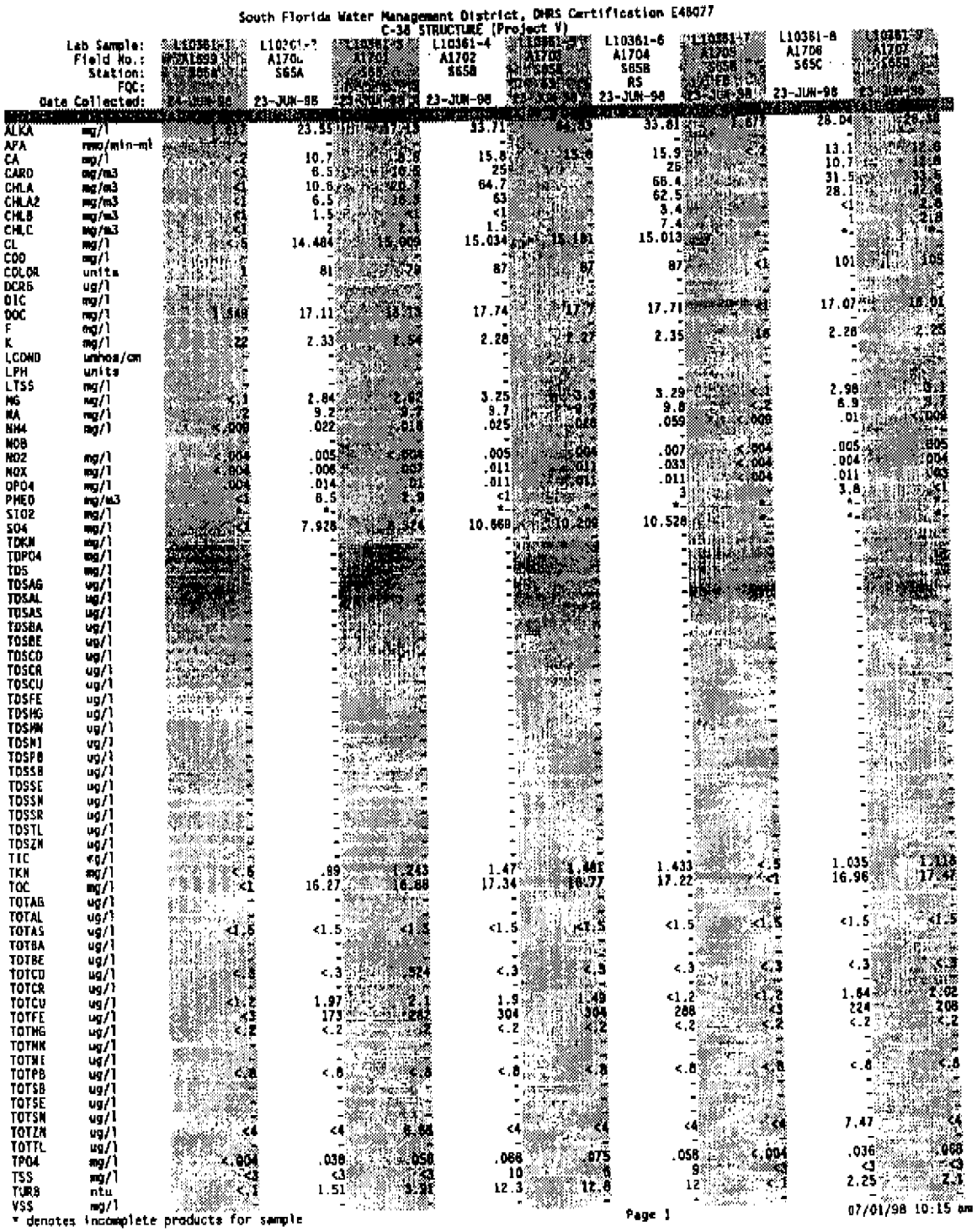
Table 12.1 Formulas Used for Calculations

Parameter	Formula
Alkaline Phosphatase	Linear regression
Alkalinity	Computer calculation: ml of titrant * N * 50,000/ml sample
Ammonia	Computer generated linear regression
Ammonia, unionized	Ammonia value X chart value (pH & Temp)
Biochemical Oxygen Demand/ Carbonaceous Biochemical Oxygen Demand	$(DO_{(initial)} - DO_{(5\ day)}$ - Seed Correction)/P P = decimal volumetric fraction of sample used Seed Correction = DO loss in seed control X f f = ratio of seed in sample to seed in control
Calcium, dissolved	Computer generated linear regression
Chemical Oxygen Demand	Computer generated linear regression
Chloride	Computer generated quadratic regression
Chlorine Residual, total	Calculation: 1 ml of FAS titrant = mg/L Cl
Inorganic Carbon, total and diss.	Computer, mean of 2 Repeats, multiple point calibration
Iron, total and dissolved	Computer generated linear regression
Nitrogen, organic	Computer calculation, TKN – Ammonia
Nitrogen, total dissolved	Computer calculation, TDKN + (Nitrate + Nitrite)
Nitrogen, total	Computer calculation, TKN + (Nitrate + Nitrite)
Nitrogen, total Kjeldahl	Computer generated linear regression
Magnesium, dissolved	Computer generated linear r regression
Nitrate + nitrite	Computer generated linear regression
Nitrite	Computer generated linear regression
Nitrate	Computer calculation, (Nitrate + nitrite)- Nitrite
Total phosphorus	Computer generated linear regression
Organic Carbon, total and dissolved	Total – Inorganic, mean of 2 Repeats, multiple point calibration
Orthophosphate	Computer generated linear regression
Potassium, dissolved	Computer generated linear regression
Residue, filterable	$\frac{\text{Final Weight-Tare, g}}{\text{Volume, ml}} * 10^6$
Residue, nonfilterable	$\frac{\text{Final Weight-Tare, g}}{\text{Volume, ml}} * 10^6$

Parameter	Formula
Residue, volatile	$\frac{\text{Nonfilterable Final Weight-Weight(550°C),g} * 10^6}{\text{Volume, ml}}$
Silica, dissolved	Computer generated quadratic regression
Sodium, dissolved	Computer generated linear regression
Strontium, dissolved	Computer generated linear regression
Sulfate	Computer generated quadratic regression
Trace metals	Computer generated linear regression
Hardness	Computer calculation: 2.497[Ca] + 4.118[Mg]
Chlorophyll	Chl. a = 11.85(OD664) - 1.54(OD647) - 0.08(OD630) Chl. b = 21.03(OD647) – 5.43 (OD664) -2.66(OD630) Chl. c = 24.52(OD630) - 7.6(OD647) - 1.67(OD664) Pheophytin = (26.7(1.7*OD665acidified)) - OD664 Carotenoids = 4.0(OD480) Chl. a corrected = 26.7(OD664) – OD665(acidified) Where: OD(wavelength) = absorbance (wavelength) - Absorbance at 750 nm in non-acidified sample and : results are multiplied by volume of extract and divided by the volume of sample filtered in m^3
Total Coliform, membrane filter	# colonies/volume of sample (ml) * 100
Total Coliform, MPN	MPN tables[1]
Fecal Coliform, membrane filter	# colonies/volume of sample (ml) * 100
Fecal Coliform, MPN	MPN tables[1]
Heterotrophic Plate count	# colonies/volume of sample tested (ml)
Fecal Streptococci	# colonies/volume of sample tested (ml)

(1) MPN tables found in SM 17th ed., Table 9221:V, p. 9-78.

Figure 12.1
Example of The Final Report



South Florida Water Management District, DBRS Certification E48077
C-38 STRUCTURE (Project V)

Lab Sample:	[illegible]	L10361-2	[illegible]	L10361-4	[illegible]	L10361-6	[illegible]	L10361-8	[illegible]
Field No.:	[illegible]	A1704	[illegible]	A1702	[illegible]	A1704	[illegible]	A1706	[illegible]
Station:	[illegible]	S65A	[illegible]	S65B	[illegible]	S65B RS	[illegible]	S65C	[illegible]
FQC:	[illegible]		[illegible]		[illegible]		[illegible]		[illegible]
Date Collected:	[illegible]	23-JUN-98	[illegible]	23-JUN-98	[illegible]	23-JUN-98	[illegible]	23-JUN-98	[illegible]

ALKA	mg/l	[illegible]	23.55	[illegible]	33.71	[illegible]	33.81	[illegible]	28.04
AFA	mmo/min-ml	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
CA	mg/l	[illegible]	10.7	[illegible]	15.8	[illegible]	15.9	[illegible]	13.1
CARD	mg/m3	[illegible]	6.5	[illegible]	25	[illegible]	26	[illegible]	10.7
CHLA	mg/m3	[illegible]	10.6	[illegible]	64.7	[illegible]	66.4	[illegible]	31.5
CHLA2	mg/m3	[illegible]	6.5	[illegible]	63	[illegible]	62.5	[illegible]	28.1
CHLB	mg/m3	[illegible]	1.5	[illegible]	<1	[illegible]	3.4	[illegible]	<1
CHLC	mg/m3	[illegible]	2	[illegible]	1.5	[illegible]	7.4	[illegible]	1
CL	mg/l	[illegible]	14.484	[illegible]	15.034	[illegible]	15.013	[illegible]	-
COD	mg/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
COLOR	units	[illegible]	81	[illegible]	87	[illegible]	87	[illegible]	101
DCRB	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
DIC	mg/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
DOC	mg/l	[illegible]	17.11	[illegible]	17.74	[illegible]	17.71	[illegible]	17.07
F	mg/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
K	mg/l	[illegible]	2.33	[illegible]	2.28	[illegible]	2.35	[illegible]	2.28
LCOND	umhos/cm	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
LPH	units	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
LTSS	mg/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
MG	mg/l	[illegible]	2.84	[illegible]	3.25	[illegible]	3.29	[illegible]	2.98
NA	mg/l	[illegible]	9.2	[illegible]	9.7	[illegible]	9.8	[illegible]	8.9
NH4	mg/l	[illegible]	.022	[illegible]	.025	[illegible]	.059	[illegible]	.01
NOB		[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
NO2	mg/l	[illegible]	.005	[illegible]	.005	[illegible]	.007	[illegible]	.005
NOX	mg/l	[illegible]	.006	[illegible]	.011	[illegible]	.033	[illegible]	.004
OPO4	mg/l	[illegible]	.014	[illegible]	.011	[illegible]	.011	[illegible]	.011
PHEO	mg/m3	[illegible]	6.5	[illegible]	<1	[illegible]	3	[illegible]	3.8
SIO2	mg/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
SO4	mg/l	[illegible]	7.928	[illegible]	10.668	[illegible]	10.528	[illegible]	-
TDKN	mg/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDPO4	mg/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDS	mg/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSAG	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSAL	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSAS	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSBA	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSBE	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSCD	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSCR	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSCU	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSFE	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSHG	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSMN	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSNI	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSPB	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSSB	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSSE	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSSN	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSSR	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSTL	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TDSZN	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TIC	mg/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TKN	mg/l	[illegible]	.89	[illegible]	1.47	[illegible]	1.433	[illegible]	1.035
TOC	mg/l	[illegible]	16.27	[illegible]	17.34	[illegible]	17.22	[illegible]	16.96
TOTAG	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TOTAL	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TOTAS	ug/l	[illegible]	<1.5	[illegible]	<1.5	[illegible]	<1.5	[illegible]	<1.5
TOTBA	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TOTBE	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TOTCD	ug/l	[illegible]	<.3	[illegible]	<.3	[illegible]	<.3	[illegible]	<.3
TOTCR	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TOTCU	ug/l	[illegible]	1.97	[illegible]	1.9	[illegible]	<1.2	[illegible]	1.64
TOTFE	ug/l	[illegible]	173	[illegible]	304	[illegible]	288	[illegible]	224
TOTHG	ug/l	[illegible]	<.2	[illegible]	<.2	[illegible]	<.2	[illegible]	<.2
TOTMN	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TOTNI	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TOTPB	ug/l	[illegible]	<.8	[illegible]	<.8	[illegible]	<.8	[illegible]	<.8
TOTSB	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TOTSE	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TOTSN	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TOTZN	ug/l	[illegible]	<4	[illegible]	<4	[illegible]	<4	[illegible]	7.47
TOTTL	ug/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-
TPO4	mg/l	[illegible]	.038	[illegible]	.066	[illegible]	.058	[illegible]	.036
TSS	mg/l	[illegible]	<3	[illegible]	10	[illegible]	9	[illegible]	<3
TURB	ntu	[illegible]	1.51	[illegible]	12.3	[illegible]	12	[illegible]	2.25
VSS	mg/l	[illegible]	-	[illegible]	-	[illegible]	-	[illegible]	-

* denotes incomplete products for sample

07/01/98 10:15 am

THIS PAGE DELIBERATELY LEFT BLANK

13.0 Corrective Action

Corrective action is required in those cases when the criteria levels for the quality control measures are not met. The specific corrective actions for each type of quality control measure are given in Tables 13.1, 13.2 and 13.3.

The analyst (Asst. Lab Technician, Lab Technician, Senior Lab Technician or Chemist) or field sample collection technician (Asst. Scientific Technician, Scientific Technician, Senior Scientific Technician) is responsible for assessing each QC measure and initiating corrective action according to Tables 13.1 and 13.2, respectively. The Supervising Professionals, Senior Chemists, Senior Supervising Technicians-Scientific, and the divisional Quality Assurance Officers are responsible for approving the corrective action taken or for initiating further steps to solve the problem.

Corrective action may be initiated by external sources or events, which may include performance evaluation results, performance audits, system audits, split sample results, and laboratory/field comparison studies. DEP recommended corrective action will be initiated as a result of systems or performance audits, split samples, or data validation review.

Problems requiring corrective action and corrective actions taken are documented in detail in one of the following: analysis logbooks, digestion logbooks, or instrument maintenance logs depending on the nature of the problem and how it was solved. The supervisor will report the problem to the appropriate Quality Assurance Officer who has the responsibility for determining if the solution is acceptable and, if not, what further steps should be taken.

Table 13.1 Corrective Actions for the Laboratory

QC Activity	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank; Method Reagent Blank	Instrument response < MDL response	Prepare new blank, if same response determine cause of contamination: reagents, environment, equipment failure, etc.
Initial Calibration Standards	Correlation coefficient >0.995 for AA/AE, >0.998 Visible spectrometer, fluorometer	Reanalyze standards; if same response, re-optimize instrument; if same response, prepare new standards
Check Standards (QC)	Accuracy within established limits determined by QC group	Reanalyze QC check standard; if same response, prepare new primary and calibration standards
Continuing Calibration Standards	± 5% of expected value	Reanalyze standard; if same response, recalibrate and reanalyze run from last continuing calibration standard
Replicate Sample	Precision within limit established by QC group	Determine cause: baseline drift, carryover, etc. Reanalyze all samples if correctable cause not found
Duplicate Sample	Precision within limit established by QC group	Reanalyze duplicates; reanalyze all samples between duplicates
Matrix Spikes	Accuracy within established limits determined by QC group	Remake spike and reanalyze; if acceptable, reanalyze affected portions of run; if still not acceptable, spike a different sample. If second sample spike is acceptable, analyze first sample by standard addition. If second sample is not acceptable, spike all samples in that LIMS group in order to check for matrix interference.
Microbiology Control Blanks	Control Blank < 1	Sample resubmitted

Table 13.2 Corrective Actions for the Field

QC Activity	Acceptance Criteria	Recommended Corrective Action
Initial Calibration Standards	Value within ± 5% of expected value or within established limits	Reanalyze standards, if same response, optimize instrument, if same response, use new standards.
QC Check Standards / Re-calibration standards	Value within 90 - 110 % known true value or within established limits	Reanalyze check standard; if same response, recalibrate instrument

Table 13.3 Corrective Actions resulting from Field Quality Control Checks

QC Activity	Acceptance Criteria	Recommended Corrective Action
Equipment/Trip/Field Blank	Value < twice MDL or less than established acceptance limits	Laboratory should reanalyze blanks: If same response, project manager should check recorded cleaning procedures and flag sample trip results for affected and related parameters as questionable or invalid data for all samples for all analytes with results where the result is less than 3 times the highest positive blank concentration. If second analysis acceptable, reanalyze affected samples in first run.
Duplicate/Split Samples	Precision within limits established by the QC group	Laboratory should reanalyze duplicates: If same response, mark sample trip results for affected and related parameters as questionable or invalid data as required. If reanalysis shows Field Collection to be acceptable, reanalyze all samples analyzed with the Field samples the first time.

THIS PAGE DELIBERATELY LEFT BLANK

14.0 Performance and Systems Audits

Audits are an essential part of the quality assurance program for both laboratory and field operations. Systems audits are conducted to measure compliance with the comprehensive and project quality assurance plans. Performance audits are conducted to evaluate the quality of the data outputs with respect to mandatory limits or the laboratory's own performance standards.

14.1 Systems Audits

A systems audit is used to evaluate the entire measurement system both in the field and laboratory. It is a detailed review of each component of the sample collection process from equipment cleaning, through submission of the samples to the laboratory, and the laboratory process from sample log-in to archival of the results. Each element must be evaluated for conformance to appropriate methodology, approved procedures and the appropriate Quality Assurance Plans. A list of any deficiencies discovered must be made and subsequently addressed to correct, improve, or modify the system as necessary.

14.1.1 Internal Systems Audits

Systems audits are performed using the forms shown in Figure 14.1. Audits are conducted semiannually by one of the following persons: the Division Quality Assurance Officer, Senior Technician, Senior Scientific Technician Supervisor, or Environmental Scientist.

14.1.2 External Systems Audits

There are no regularly scheduled external systems audits. The SFWMD will submit to audits conducted by the DEP Quality Assurance Section and HRS.

14.2 Performance Audits

Performance audits are used to evaluate the routine quality control program of the laboratory.

14.2.1 Internal Performance Audits

Internal performance audits are conducted semiannually by the Water Quality Monitoring Division Quality Assurance Officer. The audit may consist of any or all of the following: resubmission of previously analyzed samples under a different LIMS number, preparation of additional QC samples, samples split with another laboratory, and submission of spiked samples, all of which are blind to the analysts. For certain parameters or projects, performance audits may be conducted more frequently to comply with specific permit, QAPP, or regulatory guidelines.

An audit may be conducted at any time on a suspect parameter in addition to the semiannual audits for the entire laboratory. A performance audit on a parameter is mandatory when 10% of the analytical runs for that parameter fail one or more quality control criteria. The results of the

performance audits are included in the quarterly quality assurance reports issued to the Division Director by the WQMD Quality Assurance Officer.

14.2.2 External Performance Audits

The laboratory participates in three external performance audit programs. They are:

1. Florida Environmental Laboratory Certification Program administered by the Department of Health and Rehabilitative Services, semiannually
2. United States Geological Survey, Denver, annually
3. Florida Department of Environmental Protection Phosphorus Round Robin, semiannually

FIGURE 14.1 - SYSTEMS AUDIT CHECKLIST

Project Name:_____ Project No:_____

Project Manager:_____ Date:_____

Project Code (field and lab use):_____

Field Auditor:_____ Lab Auditor:_____

Signature:_____ Signature:_____

	Planning and Preparation:	YES	NO
1.	Was a QA Project Plan prepared for this project?	___	___
2.	Was a briefing held with project participants, both field and lab? Date:_____	___	___
3.	Were additional instructions given to participants (i.e., changes in project plan)?	___	___
4.	Was there a written list of sampling locations and descriptions?	___	___
5.	Was there a map of sampling locations available to field personnel?	___	___
6.	Was sampling scheduled with the field technician supervisor in advance (minimum one week)?	___	___
7.	Were analyses scheduled with the laboratory in advance (minimum one week)?	___	___

Comments :_____

	General Sampling Procedures:	YES	NO
1.	Were sampling locations properly selected?	____	____
2.	Were samples collected for all required analysis?	____	____
3.	Was sampling equipment protected from possible contamination prior to sample collection?	____	____
4.	Were clean disposable latex or vinyl gloves worn during sampling?	____	____
5.	Were gloves changed for each sample station?	____	____
6.	If equipment was cleaned in the field, were proper procedures used?	____	____
7.	What field instruments were used? _____		
8.	Were calibration procedures documented in the field notes?	____	____
9.	Was calibration check conducted within 24 hours or at the end of the sampling day for Hydrolabs and every 4 hours and at the end of the day for other field instruments?	____	____
10.	Were samples chemically preserved at time of collection?	____	____
11.	Was the preservative amount recorded in the field notes?	____	____
12.	Was the pH of preserved samples checked to insure proper preservation?	____	____
13.	Were the proper sample bottles used?	____	____
14.	Were samples iced at the time of collection?	____	____
15.	Were sample bottles rinsed with sample before filling?	____	____

	General Sampling Procedures: (continued)	YES	NO
16.	Were field conditions recorded in the field notes?	____	____
17.	Was the filtering equipment pre-rinsed with sample?	____	____
18.	Were samples processed on a clean tray/surface?	____	____
19.	Was waste material containerized and maintained separate from samples and equipment?	____	____

Comments:_____

_____

_____

_____

_____

	Surface Water Sampling:	YES	NO
1.	What procedures were used to collect the surface water samples?		

_____

_____

_____

_____

2.	Was a Niskin bottle used for sample collection?	____	____
3.	Was a pump used for sample collection?	____	____
4.	If pump was used, what type?_____		
5.	Was the pump properly cleaned before/between sites?	____	____
6.	Was the pump tubing properly cleaned before/ between sites?	____	____
7.	Was sample collected in polyethylene bucket?	____	____
8.	Did samplers wade in stream during sample collection?	____	____
9.	Were samples collected upstream from the sampler or vehicle?	____	____

	Surface Water Sampling (continued)	**YES**	**NO**
10.	Were autosamplers used?		
11.	Were samples composited?		
12.	How were samples composited?_		
13.	Were autosampler bottle(s) capped and shaken well before compositing or processing?		
14.	Was tubing changed within the last three months?		
15.	Were Preventative maintenance protocols followed and documented?		

Comments:

	Well Sampling:	**YES**	**NO**
1.	Was depth of well determined?		
2.	Was depth to water determined?		
3.	Was measuring tape properly decontaminated between wells?		
4.	How was the volume of water originally present in each well determined?		
5.	Was the volume determined correctly?		
6.	How was completeness of purging determined: Volume measure_ Time Flow rate Scond./pH/T		

Well Sampling (continued) **YES** **NO**

7. Was a sufficient volume purged? ____ ____

8. Was the well over-purged? ____ ____

9. Was the disposal of purge water handled properly? ____ ____

10. Was a dedicated (in-place) pump used? ____ ____

If no: Describe method of purging:_____

_____

_____

_____

11. How were the samples collected?
Bailer? ____ ____
Pump? ____ ____
Other? _____

_____

_____

_____

12. Construction material of bailer?_____

13. If a pump was used, describe how it was cleaned before and/or between wells:_____

_____

_____

_____

14. Were the samples properly transferred from bailer to sample bottles (i.e., was the purgeable sample agitated, etc.)? ____ ____

15. Was the rope or line allowed to touch the ground? ____ ____

16. Was a teflon coated stainless steel cord used? ____ ____

Comments:_____

_____

_____

_____

Sediment Sampling: YES NO

1. What procedures were used to collect the samples?

2. Were the samples well mixed prior to placing the sample in the sample container?

3. Were the samples composited?

Comments:

Atmospheric Deposition: YES NO

1. Were buckets continuously capped before deployment?
2. Were previously deployed buckets sealed immediately?
3. Were field conditions documented in the field log?
4. Were sample buckets secured and kept clean in vehicle?
5. Was preventative maintenance and cleaning performed as specified in the SOP/CompQAP?
6. Was balance calibration performed?
7. Was tare weight legible?
8. Was tare weight determined within the last quarter?
9. Was Bucket Equipment Blank performed?

Comments:

Other Sampling:

1. What other types of samples were collected during this investigation?_____
_____
_____

2. What procedures were used for the collection of these samples?_____
_____
_____
_____

Comments:_____
_____
_____
_____

	Field Quality Control:	**YES**	**NO**
1.	Were QC samples specified in a QA Project Plan?	____	____
2.	Were the QC samples collected in accordance with the QA project plan?	____	____
3.	Did sampling personnel utilize any trip blanks?	____	____
4.	Did sampling personnel utilize any field blanks?	____	____
5.	Were any equipment blanks collected?	____	____
6.	Were any duplicate samples collected?	____	____
7.	Check method used to collect split sample Filled one large container and then transferred portions _____ Sequentially filled bottles _____		
8.	Were chain of custody records completed for all samples?	____	____

	Field Quality Control (continued):	**YES**	**NO**
9.	Were all samples identified with appropriate tags?	____	____
10.	Were sample I.D. tags filled out properly?	____	____
11.	Did information on sample I.D. tags and Chemistry Field Data Log match?	____	____
12.	Were samples kept in a secure place after collection?	____	____
13.	Was Chemistry field Data Log signed by sampling personnel?	____	____
14.	Were amendments to the project plan documented (on the project plan itself, in a project logbook, elsewhere)?	____	____

Comments:_____
_____
_____
_____
_____

	General Laboratory Procedures:	**YES**	**NO**
1.	Have unique sequential laboratory numbers been assigned to each sample?	____	____
2.	Has the data from the Chemistry Field Data Log been input to the computer directly?	____	____
3.	Have samples been stored in an appropriate secure area?	____	____
4.	Has sample custody been maintained by the laboratory?	____	____
5.	Has the proper bar code label been attached to each sample I.D. tag?	____	____
6.	Were the samples properly divided into aliquots?	____	____

Comments:_____

	Analytical Methods:	YES	NO
1.	Have approved analytical methods or procedures been followed?	____	____
2.	Does the project plan include copies of any non-standard methods without appropriate quality assurance results for validation of the method?	____	____
3.	Does use of the analytical methods specified result in data of adequate detection limit, accuracy, and precision to meet the requirements of the project?	____	____

Comments:_____

	Laboratory Quality Control:	YES	NO
1.	Have approved sample holding times been observed?	____	____
2.	Have replicate analyses been performed on at least one sample?	____	____
3.	Have spike analysis been performed on at least one sample?	____	____
4.	Have the quality control reporting forms been properly filled out?	____	____
5.	Are current instrument calibration curves used for all methods?	____	____
6.	Did the spiking procedures follow acceptable protocols for quantity and concentration?	____	____

	Laboratory Quality Control (continued):	**YES**	**NO**
7.	Are quality control charts used to track QC precision and accuracy?	____	____
8.	Are QC charts kept up to date?	____	____
9.	Is the precision of the data presented within acceptable limits?	____	____
10.	Is the accuracy of the data presented within acceptable limits?	____	____
11.	Are recent (one year or less) performance audit results available?	____	____
12.	Has the laboratory followed the preventative maintenance procedures outlined in the QA plan?	____	____

Comments: _____

_____

_____

_____

_____

	Data Validation and Reporting:	**YES**	**NO**
1.	Were all the steps in the data validation procedure outlined in the QA plan followed?	____	
2.	Was the data reported in the proper format with the proper units?	____	____
3.	Was the laboratory I.D. number included on each page of the data?	____	____

Comments: _____

_____

_____

_____

_____

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
1100.1	COLOR	1.2	SM 2120B	EFFECTIVE
PAGE 1 OF 6				DATE 07/22/97

APPROVALS/DATE			
DIVISION DIRECTOR 8/26/97	LAB SUPERVISOR 8-11-97	QUALITY ASSURANCE 8-22-97	METHODS 7/24/97

SECTION 1.0 - TABLE OF CONTENTS

SECTION 2.0 - METHOD DESCRIPTION/HISTORY

2.1 Color in water may result from the presence of metallic ions (iron and manganese), organic acids, humus and peat materials, plankton, weeds, and industrial waste.

2.2 In our laboratory, color analysis is performed on samples filtered through 0.45um membranes. Filtration removes large particulates and turbidity that may interfere with spectrophotometric measurements. It should be noted that the filtration procedure may remove some of the color from the sample.

2.3 The color of the sample is determined by spectroscopic comparison to platinum-cobalt color solutions at 465 nm in a 1 cm quartz flowcell. One unit of color corresponds to 1 mg/L of platinum in the form of the chloroplatinate ion.

2.4 This method version 1.2 was adopted on July 22, 1997 and is an internally developed (SFWMD) method and is based on Standard Methods SM 2120B. This revision reflects changes in the entry of standard values into LIMS and calibration check frequency.

SECTION 3.0 - SAFETY PRACTICES

3.1 Wear safety glasses and a full-length, long-sleeved laboratory coat.

3.2 Latex or polyethylene gloves (non-powdered) may be worn when handling the samples.

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
1100.1	COLOR	1.2	SM 2120B	EFFECTIVE
PAGE 2 OF 6				DATE 07/22/97

APPROVALS/DATE			
DIVISION DIRECTOR 8/24/97	LAB SUPERVISOR 8-11-97	QUALITY ASSURANCE 8-22-97	METHODS 7/24/97

ECTION 3.0 - SAFETY PRACTICES (CONT'D)

3.3 All personnel conducting this method should be familiar with the SFWMD Chemical Hygiene Plan and should have reviewed any pertinent Material Safety Data Sheets. Note: pay special attention to the MSDS for Platinum Cobalt Solution, as it is a suspected carcinogen (Note: Prolonged exposure to Platinum-Cobalt solutions causes degradation of tooth enamel).

3.4 No hazardous wastes are generated by this procedure; liquid waste may be disposed of in the sink.

SECTION 4.0 - LIST OF EQUIPMENT/INSTRUMENTATION

4.1 Visible Spectrometer, Model Spectronics 501

4.2 Sample introduction system (flowcell, transfer tubing, and pump)

4.3 Beaker 1 L, polypropylene, for waste collection (Fisher #02-586-6H)

SECTION 5.0 - REAGENTS

5.1 Cell cleaning solution (5% Potassium Hydroxide in Methanol).

SECTION 6.0 - STANDARDS

6.1 Platinum Cobalt Color Stock Solution/Standard 500 c.u. - STD1 (500 mg/L) - (Fisher Scientific #SO-P-120).

6.2 Platinum Cobalt Color Standard 300 c.u. - STD2 (300 mg/L) - measure 30 mL of stock solution with a class A volumetric pipet and dilute with D.I. water to 50 mL in a class A volumetric flask.

6.3 Platinum Cobalt Color Standard 100 c.u. - STD3 (100 mg/L) - measure 10 mL of stock solution with a class A volumetric pipet and dilute with D.I. water to 50 ml in a class A volumetric flask.

6.4 Platinum Cobalt Color Standard 50 c.u. - STD4 (50 mg/L) - measure 5 mL of stock solution with a class A volumetric pipet and dilute with D.I. water to 50 mL in a class A volumetric flask.

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
1100.1	COLOR	1.2	SM 2120B	EFFECTIVE
PAGE 3 OF 6				DATE 07/22/97

APPROVALS/DATE			
DIVISION DIRECTOR	LAB SUPERVISOR	QUALITY ASSURANCE	METHODS
8/26/97	8-11-97	8-22-97	7/24/97

SECTION 7.0 - QUALITY CONTROL

7.1 The Platinum Cobalt Color standard recoveries are checked (see acceptance criterion in Section 8.0) by the analyst before conducting sample analyses. The results are recorded on the physical parameters logsheet and the values are also entered into the LIMS with QC designations STD1, STD2, STD3, etc.. The 100 mg/L standard (STD3) should be reran every 20 samples to check continuing calibration.

7.2 The sample holding time is 48 hours. Notify the supervisor or team leader if any samples are out of the holding time.

7.3 QC1 and QC2 are prepared on request by the QA unit. QC1 and QC2 are analyzed at beginning of each run and are repeated after each 20 samples. Select a true matrix sample for repeat analysis. If there is limited volume to use the same sample throughout the run, two or more samples maybe used for the repeat and reported separately.

7.4 A repeat analysis (sample selected at random) should be conducted for every 20 samples analyzed. These results are recorded on the QC sheet and physical parameters log. The mean and coefficient of variation of the replicate set is determined (using the formula below) and noted on the QC sheet and physical parameters logsheet.

$$\%RSD = \frac{STD.\ DEV.}{MEAN\ CONC.} * 100$$

7.5 All quality control data must be within the current established limits before entering sample data into the LIMS system.

7.6 Samples must be at room temperature and should be shaken gently prior to analyses; excessive shaking will entrain air and result in erroneous readings.

SECTION 8.0 - STEP-BY-STEP PROCEDURE

8.1 Create a workgroup for the analyses to be conducted. Refer to the ACS LIMS users Guide for instructions on "Creation of a Workgroup". **Remove the samples to be tested from the refrigerator and allow them to warm to room temperature (this may require several hours). Failure to do so will result in erratic readings.**

... and allow it to stabilize for at least 30

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
1100.1	COLOR	1.2	SM 2120B	EFFECTIVE
PAGE 4 OF 6				DATE 07/22/97

APPROVALS/DATE			
DIVISION DIRECTOR 8/26/97	LAB SUPERVISOR DPS 8-11-97	QUALITY ASSURANCE 8-12-97	METHODS 7/24/97

SECTION 8.0 - STEP-BY-STEP PROCEDURE (CONT'D)

8.3 Log on to the LIMS terminal (enter username and password) adjacent to the spectrometer and type "lims" at the UNIX prompt. Select 'Workstat', 'Dataentry', and 'Color'. Enter the workgroup number and the system will prompt you for the result of the first sample.

8.4 Add 4 drops of cell cleaning solution (5% Potassium Hydroxide in Methanol) into 20 mL of fresh D.I. water in a 25 mL glass graduated cylinder. This mixture should be used to clean-up the cell before conducting any further work.

8.5 Latch the platens on the pump, insert the inlet tubing into the cleaning solution prepared in 8.4 and turn on the pump and allow all of the solution to pass into the flow cell.

8.6 Enter the Absorbance mode on the spectrometer by pressing the <%T/A/C> button on the spectrometer.

8.7 Rinse a clean 50 mL beaker with D.I. water and fill it with fresh D.I. water. Place the inlet tube into the beaker of distilled water and allow the water to pass into the flow cell until the reading on the spectrometer stabilizes. When stable, zero the spectrometer by pressing the <SECOND FUNCTION> button and the <100 %T/ Zero A> button.

8.8 Next place the inlet tube into the 500 c.u. color standard (STD1) until the absorbance reading stabilizes and check the absorbance. If the reading is in the acceptable range (0.130 - 0.145) continue to the next step. If the reading is not acceptable repeat steps 8.4 to 8.8. If an acceptable reading cannot be obtained, contact your shift supervisor or the QA officer for assistance.

8.9 Enter the concentration mode by pressing the <%T/A/C> button. Remove the inlet tube from the STD4 and clean the inlet tube with a Kimwipe. Place the inlet tube into the beaker of D.I. water and zero the instrument as in step 8.7. Place the inlet tubing back into the beaker of 500 c.u. standard and allow the reading to stabilize. Calibrate the instrument by pressing "5", "0", "0", and <ENTER>.

8.10 Clean the inlet tube with a Kimwipe prior to its insertion into the next solution. Pump the 300 c.u. standard (STD2) into the flow cell allow the reading to stabilize and record the reading on the physical parameters log sheet. The reading should be 300 ± 5 c.u.

8.11 Repeat step 8.10 for the remaining standards. The 100 c.u. (STD3) and 50 c.u. (STD1) standards should read within ± 3 c.u.

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
1100.1	COLOR	1.2	SM 2120B	EFFECTIVE
PAGE 5 OF 6				DATE 07/22/97

APPROVALS/DATE

DIVISION DIRECTOR	LAB SUPERVISOR	QUALITY ASSURANCE	METHODS
8/26/97	DDL 8-11-97	8-22-97	7/24/97

SECTION 8.0 - STEP-BY-STEP PROCEDURE (CONT'D)

8.12 If the standards are within limits, insert the inlet tube into the QC1 and QC2 solutions respectively and record the result; they should read within the current limits for the QC solution.

8.13 The inlet tube should be flushed with D.I. water and the exterior cleaned with a Kimwipe after removing it from any standard, QC, or sample solution to prevent cross contamination.

8.14 If the QC samples are within limits, proceed to analyze the samples by placing the inlet tube into the appropriate sample. When the reading has stabilized press the "send" button once on the spectrometer to send the result to the LIMS system. The system will then prompt you, one by one, for each sample in the workgroup. The computer then shows you the value that was entered and asks you to confirm the result entered or to re-enter the value. Once you have accepted the entered result, the computer will advance to the next sample. Hitting "Enter" with no test result will cause the program to skip that sample and go on to the next sample. The 100 mg/L standard (STD 3) and a repeat sample should be rerun every 20 samples to check continuing calibration. Analyze QC2 at the end of the run and report the value on the instrument log and into LIMS.

8.15 Verify the correctness/acceptability of the results (lab and field QC criteria). Note any discrepancies on the log sheet.

SECTION 9.0 - DATA HANDLING

9.1 The detailed data handling procedures are covered in section 8.0, however, steps 9.2 through 9.8 summarize the data handling process.

9.2 Sign onto the workstation (enter username and password) at the UNIX prompt type "lims" and create a workgroup for the test COLOR and exit the SEEDPAK1 Main Menu (see Creating a Workgroup and Printing a Workgroup in the ACS LIMS Users Guide).

9.3 Log on to the LIMS terminal (enter username and password) adjacent to the spectrometer and type "lims" at the UNIX prompt. Select 'Workstat', 'Dataentry', and 'Color'. Enter the workgroup number and the system will prompt you for the result of the first sample.

9.4 The system will then prompt you, one by one, for each sample in the workgroup. Hit the send button on the spectrometer and the system will send the result. The computer then shows you the value that was entered and asks you to confirm the result entered or to re-enter the value.

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
1100.1	COLOR	1.2	SM 2120B	EFFECTIVE
PAGE 6 OF 6				DATE 07/22/97

APPROVALS/DATE			
DIVISION DIRECTOR 8/26/97	LAB SUPERVISOR DBL 8-11-97	QUALITY ASSURANCE 8-22-97	METHODS 7/24/97

SECTION 9.0 – DATA HANDLING (CONT'D)

9.5 Once you have accepted the entered result, the computer will advance to the next sample. Hitting "Enter" with no test result will cause the program to skip that sample and go on to the next sample. To get back to a sample that you skipped, hit Select 'Workstat', 'Dataentry', and 'Color'. Once you enter the workgroup number again, you will be prompted with the samples that are incomplete.

9.6 To end the program, hit "Enter" until you see "Done..." and are back at the LIMS menu.

9.7 Pick up the data entry report for the samples you have just entered from the system printer.

SECTION 10.0 - INSTRUMENT CLEANUP/SHUTDOWN/TROUBLESHOOTING

10.1 The spectrometer should be wiped clean with a damp paper towel after each use.

10.2 The flow cell should be cleaned after each use with the cell cleaning solution by pumping it into the cell and allowing it to stand for a few minutes. Pump D.I. water through the cell after the cleaning solution for a few minutes to completely remove it from the flow cell.

10.3 Leave the cell filled with D.I. water when not in use.

10.4 Release the clamps from the pump tubes when not in use.

10.5 Consult your supervisor before making any major changes, adjustments, and/or repairs to the instrumentation.

SECTION 11.0 - REFERENCES

11.1 Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992.

11.2 ACS LIMS Users Guide, version 1.0, 1992.

11.3 SFWMD Comprehensive Quality Assurance Manual, current version.

11.4 Spectronics 501 Operators Manual

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3120.1	SILICA (SiO_2)	1.1	SM4500SiD (Modified)	EFFECTIVE
PAGE 1 OF 10				DATE 12/01/96

APPROVALS/DATE			
DIVISION DIRECTOR 8/27/97	LAB SUPERVISOR DPRanoff 8-27-97	QUALITY ASSURANCE 8-27-97	METHODS 8/27/97

SECTION 1.0 - TABLE OF CONTENTS

SECTION 2.0 - METHOD DESCRIPTION/HISTORY

2.1 Silicon ranks next to oxygen in abundance in the earth's crust. Degradation of silica-containing rocks or sand results in the presence of silica in natural waters as suspended particles, in a colloidal or polymeric state, and as silicic acids or silicate ions. The silica content of natural water most commonly is in the 1 to 30 mg/L range, although concentrations as high as 100 mg/L are not unusual and concentrations exceeding 1000 mg/L are found in some brackish waters and brines.

2.2 The reaction of silicate with molybdate forms B-molybdosilicic acid at a pH of 1.0-1.8. The B-molybdosilicic acid is reduced by tin (II) to form molybdenum blue, which is measured at 820 nm.

2.3 Interference from orthophosphate and tannin is eliminated by the use of tartaric acid. Color absorbing at the analytical wavelength will interfere. Samples for analysis are filtered in the field through a 0.45 um filter.

2.4 The analyses are conducted in a highly automated instrument called a Rapid Flow Analyzer (RFA). This instrument is equipped with an autosampler for sample introduction, a peristaltic pump, a mixing manifold, and a photometer for colorimetric measurement. The analog output of the photometer is relayed to a personal computer equipped with ALPKEM data collection software. The same software is used to calculate sample concentrations.

2.5 This modified method version is based on APHA Standard Method 4500SiD. This revision, version 1.1, was adopted on December 1, 1996 and reflects changes due to modifications of the PPE codes and changes in [illegible] This method should not be modified except with the express

METHOD NAME	REVISION	REFERENCE	STATUS
SILICA (SiO_2)	1.1	SM4500SiD (Modified)	EFFECTIVE
OF 10			DATE 12/01/96

APPROVALS/DATE

DIVISION DIRECTOR	LAB SUPERVISOR	QUALITY ASSURANCE	METHODS
8/22/97	DBI 5-7-97	8-22-97	9/5/97

SECTION 3.0 - SAFETY PRACTICES

3.1 Wear safety glasses and a full-length, long-sleeved laboratory coat.

3.2 Latex or polyethylene gloves (non-powdered) may be worn when handling the samples.

3.3 All personnel conducting this method should be familiar with the SFWMD Chemical Hygiene Plan and should have reviewed any pertinent Material Safety Data Sheets.

3.4 The disposal of samples can be done in the sink, flushing with ample amounts of tap water.

3.5 Preparation of reagents containing hydrochloric acid, chloroform and stannous chloride solutions should be conducted in a fume hood. The reagents should be prepared by slow addition of concentrated hydrochloric acid to D.I. water. Use an acid resistant bottle carrier when carrying glass containers of concentrated hydrochloric acid and chloroform.

3.6 Before starting any run, all lines connecting the instrument to the reagents should be checked and tightened if necessary. In case of a leak onto an electrical system, the power should be disconnected before conducting any repairs.

3.7 The electrical power should be disconnected before conducting any repairs inside the instrument on controllers, electrical wiring or any other components near sources of electricity.

3.8 In case of spills of concentrated hydrochloric acid the spill should be first treated with an appropriate spill kit and the contaminated absorbent should be collected and placed into adequate storage containers for disposal.

3.9 Follow the Personal Safety Protection Codes below during analysis:

A = Lab coat, glasses
B = Lab coat, acid resistant gloves, glasses
C = Lab coat, glasses, apron, gloves
1X = Rubber acid carrier
2X = Lab coat, acid resistant gloves, goggles, face shield, apron
3X = Lab coat, apron, acid resistant gloves, face shield+goggles+respirator or full-face respirator
G = Flush sink drain with ample amount of tap water

3.10 In case of spills be sure to utilize the appropriate material kit to absorb the spill, if you are not sure of the appropriate material or method for cleaning a spill contact your supervisor. **Notify a supervisor immediately in case of any large spill of hazardous materials.**

	METHOD NAME	REVISION	REFERENCE	STATUS
3 OF 10	SILICA (SiO_2)	1.1	SM4500SiD (Modified)	EFFECTIVE
				DATE 12/01/96

APPROVALS/DATE			
DIVISION DIRECTOR	LAB SUPERVISOR	QUALITY ASSURANCE	METHODS
8/26/97	5-7-97	8-12-97	5/5/97

SECTION 4.0 - LIST OF EQUIPMENT/INSTRUMENTATION

4.1 ALPKEM™ Rapid Flow Analyzer (RFA), Model 300 with XYZ autosampler

4.2 Personal Computer equipped with A/D converter, printer and ALPKEM data reduction software.

4.3 Class A volumetric glassware (pipets and volumetric flasks)

4.4 Clean Nalgene plastic containers.

SECTION 5.0 - REAGENTS

5.1 Sodium lauryl sulfate solution - Dissolve 5.0 grams of dodecyl sodium sulfate in about 90 mL of deionized water in a 250 mL Erlenmeyer flask. It may be necessary to warm the mixture to obtain a homogeneous solution. Transfer the solution to a 100 mL volumetric flask and dilute to mark with deionized water. Transfer to a small plastic dropping bottle for daily use. **(Safety PP = A)**

5.2 Tartaric acid 10% w/v - Dissolve 100 grams of tartaric acid in approximately 800 mL of deionized water in a 1 liter volumetric flask. Cap and shake to dissolve the salt; dilute the solution to 1 liter with D.I. water. Transfer the solution to a liter plastic container. Add 2 drops of chloroform and shake well. Store the reagent at 2-6 °C. **(Safety PP = A)**

5.3 Hydrochloric acid 1.2 N - In a fume hood, cautiously add (with stirring) 100 mL of concentrated (12N) hydrochloric acid to approximately 800 mL of deionized water contained in a 1 liter volumetric flask. When the solution has returned to room temperature, dilute to 1 liter with D.I. water and mix well. Store the reagent in a 1 liter plastic container. **(Safety PP = 1X, 2X)**

5.4 Stock stannous chloride - In a fume hood, cautiously add (with stirring) 10 mL of concentrated (12N) hydrochloric acid to 10 mL of deionized water in a 50 ml pyrex beaker. Dissolve 10 grams of stannous chloride in the acidic solution. Heating may be required to obtain a homogeneous solution. Store the stock solution in a tightly closed plastic container and refrigerate at 2-6 ° C. **(Safety PP = 1X, 2X)**

5.5 Working stannous chloride reagent - In a 60 mL plastic container, mix together 50 mL of 1.2 N hydrochloric acid and 0.5 mL stock stannous chloride. This reagent should be prepared fresh daily. **(Safety PP =A)**

5.6 Ammonium molybdate reagent - Dissolve 1.080 grams of ammonium molybdate in approximately 80 mL of deionized water in a 100 mL volumetric flask. Add 0.3 mL (9 drops) of 50% sulfuric acid. Add 1 mL of

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3120.1	SILICA (SiO_2)	1.1	SM4500SiD (Modified)	EFFECTIVE
PAGE 4 OF 10				DATE 12/01/96

APPROVALS/DATE			
DIVISION DIRECTOR 8/26/97	LAB SUPERVISOR DBI 5-7-97	QUALITY ASSURANCE 8-22-97	METHODS 5/5/97

SECTION 6.0 - STANDARDS

NOTE: Immediately after mixing, the standards should be poured into labelled, clean 175 mL plastic containers.

6.1 Silica stock standard - NIST standard solution or traceable stock, 10 mg/mL as Si (21.3 mg/mL SiO_2).

6.2 **Solution A (1065 mg/L or 1.065 mg/mL)** - In a 100 mL class A volumetric flask, pipet 5 mL of stock solution and dilute to the mark with deionized water. Cap the flask and mix well.

6.3 **Standard 1 (21.3 mg/L)** - In a 100 mL class A volumetric flask, pipet 2 mL of solution A and dilute to the mark with deionized water. Cap the flask and mix well.

6.4 **Standard 2 (10.65 mg/L)** - In a 100 mL class A volumetric flask, pipet 1 mL of solution A and dilute to the mark with deionized water. Cap the flask and mix well.

6.5 **Standard 3 (5.33 mg/L)** - In a 200 mL class A volumetric flask, pipet 1 mL of solution A and dilute to the mark with deionized water. Cap the flask and mix well.

6.6 **Standard 4 (2.67 mg/L)** - In a 100 mL class A volumetric flask, pipet 50 mL of standard 3 and dilute to the mark with deionized water. Cap, mix well and transfer to a clean 175 mL plastic bottle.

6.7 **Standard 5 (0.0 mg/L)** - Deionized water only.

SECTION 7.0 - QUALITY CONTROL

7.1 QC1 and QC2 are prepared fresh monthly or as needed by the QA unit. QC1 and QC2 are analyzed at beginning of each set of analyses. QC2 is repeated at the end of the analytical run.

7.2 Spikes are prepared from samples selected at random (1 for every 20 samples analyzed), and are made by adding 0.1 mL of the solution A (with a 1.0 mL Tensette) to a 10 mL volumetric flask and diluting to the mark with the sample. Calculate the spike recovery using the formula below.

$$\%RECOVERY = \frac{((SPIKE\ RESULT) - (0.99 * SAMPLE\ RESULT))}{(SPIKE\ CONCENTRATION)} * 100$$

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3120.1	SILICA (SiO_2)	1.1	SM4500SiD (Modified)	EFFECTIVE
PAGE 5 OF 10				DATE 12/01/96

APPROVALS/DATE			
DIVISION DIRECTOR 8/26/97	LAB SUPERVISOR DBI 5-7-97	QUALITY ASSURANCE 8-22-97	METHODS 5/5/97

SECTION 7.0 - QUALITY CONTROL (CONT'D)

7.3 A repeat analysis (of a matrix sample chosen at random) must be run for every 20 samples analyzed. Enter each repeat values, the calculated Mean, Standard Deviation, and Coefficient of Variation on the QC sheet. Use the formula below to calculate the percent relative standard deviation.

$$\%RSD = \frac{STD.\ DEV.}{MEAN\ CONC.} * 100$$

7.4 All quality control data must be within the current established limits, and the run must be checked and initialed by the supervisor or the QA officer before sending the sample data into the LIMS system.

SECTION 8.0 - STEP-BY-STEP PROCEDURE

8.1 Sign on to LIMS and create a workgroup (refer to section 9.0 - Data Handling).

8.2 Turn 314 regulated power ON. Turn on the power to the computer.

8.3 Turn on light source.

8.4 Place all reagent lines in DI H_2O containing 5 drops of sodium lauryl sulfate.

8.5 Latch platens and turn on 302 pump module.

8.6 After flow has stabilized, verify a smooth and consistent bubble pattern throughout the manifold.

8.7 Set photometer parameters as defined by flow diagram.

8.8 On photometer, set center knob to "sample" position.

8.9 Slowly turn the sample fine adjust knob to set the LCD display to 5.00 volts.

8.10 On the photometer, set the center knob to "reference" position.

8.11 Slowly turn the reference fine adjust knob to set the LCD display to 5.00 volts.

8.12 Place center knob in "absorbance" position. Using the reference fine adjust knob, set the LCD display to 0.20 (± 0.01).

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3120.1	SILICA (SiO_2)	1.1	SM4500SiD (Modified)	EFFECTIVE
PAGE 6 OF 10				DATE 12/01/96

APPROVALS/DATE			
DIVISION DIRECTOR	LAB SUPERVISOR	QUALITY ASSURANCE	METHODS
8/26/97	5-7-97	8-22-97	5/5/97

SECTION 8.0 - STEP-BY-STEP PROCEDURE (CONT'D)

8.13 Place reagent lines in their respective containers. Allow 2 to 3 minutes before placing stannous chloride line in the reagent bottle.

8.14 After 15 minutes, reagent flow should be stabilized. Repeat steps 8.8 through 8.12.

8.15 While reagents are stabilizing, prepare the sample table.

8.17 Build a sample table by selecting **F4** and entering standard, sample, and quality control identifiers with dilution factors (if applicable).

8.18 When sample table is finished, press **ALT S** to save the table. Sample tables are named using a 3 letter parameter code, a 3 digit Julian date, and a letter corresponding to the order of analysis; example: **SIO136A** - 1st run; **SIO136B** - 2nd run.

8.19 Press **ALT P** to print a hard copy of the table file.

8.20 Press **ESC** to return to main menu.

8.21 Press **F5** to get to data collection mode. Use the "space" bar to turn on Channel 3. Press F3 to monitor baseline.

8.22 While baseline is being monitored, begin placing the samples into the autosampler according to the sample table.

8.23 Manually activate the sampler and set the stop count to the cup number corresponding to the end of the first set of standards.

8.24 Observe that the standards appear linear and that peaks do not have spikes or any unusual shape to them. Press reset on sampler.

8.25 If standards appear normal, press escape on computer. Set the total number of samples on the autosampler. To begin data collection, press **ALT and 1** (simultaneously), next, press start on the autosampler.

8.26 Finish placing all the samples into the autosampler according to the sample table.

8.27 When the run is complete, press escape to return to the blue data collection screen. Press Alt and 1 to stop the analytical run.

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3120.1	SILICA (SiO_2)	1.1	SM4500SiD (Modified)	EFFECTIVE
PAGE 7 OF 10				DATE 12/01/96

APPROVAL/DATE			
DIVISION DIRECTOR 8/26/97	LAB SUPERVISOR DBI 8-27-97	QUALITY ASSURANCE 8-22-97	METHODS 5/5/97

SECTION 9.0 - DATA HANDLING

9.1 Sign onto the workstation.

9.2 Go into the ACS LIMS via the command **LIMS** and create a workgroup for SIO2 and exit the SEEDPAK1 Main Menu (see Creating a Workgroup and Printing a Workgroup in the ACS LIMS Users Guide).

9.3 Make sure the sample table to be calculated is the current one loaded. Otherwise, go to F4 and recall the right sample table file (ALT 1, L, Filename)

9.4 At the main menu on the computer, press **F8** (calculation sub-menu).

9.5 Press F8 again; select: from raw data. Press "**ENTER**".

9.6 At filename prompt, enter the filename to be calculated example: **filename.D13**. Be sure to include the extension **.D13** in order to select the correct data file.

9.7 Review peak finding parameters and press "enter" at execute Y prompt (see 9.19.2).

9.8 Observe that peak markers appear on each peak. Press **F9** to continue.

9.9 After observing standard curve, press F9 until calculation values appear on screen.

9.10 Check each quality control value to be sure it falls within acceptable limits.

9.11 If run is acceptable, press F2 (LPT1), then press Y to print a hard copy of the file.

9.12 When hard copy is finished printing, press F2 again. Backspace over LPT1. Type in filename followed by **.IN** extension. Example: **SIO136A.IN**. Press N at **Y/N** prompt.

9.13 Press F9 to save and exit.

9.14 To transfer a file to LIMS, hit ESC to escape to main menu screen. Press **F10**, then press S (shell).

9.15 At the C:> prompt, type **CD Data**.

9.16 At the **C:\Data** > prompt, type: **S2SEND** Filename.IN 2

9.17 After file is sent, pick up report at the printer.

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3120.1	SILICA (SiO_2)	1.1	SM4500SiD (Modified)	EFFECTIVE
PAGE 8 OF 10				DATE 12/01/96

APPROVALS/DATE			
DIVISION DIRECTOR 8/x/97	LAB SUPERVISOR DPT 5-7-97	QUALITY ASSURANCE 5-22-97	METHODS 5/5/97

SECTION 9.0 - DATA HANDLING (CONT'D)

9.18 Fill out all log books and QC sheets daily for each analytical run.

9.19 Software Settings

9.19.1 Channel Setup (Main Menu: F3 - METHOD TABLE)

CHANNEL # = [3]
CHANNEL NAME = [SIO2]
START IGNORE TIME = [0]
INITIAL BASELINE LEAD TIME = [60]
FINAL BASELINE LAG TIME = [60]
CORRECTIONS CODE = [N]
CYCLE TIME = [70]
COLLECTION RATE = [2] POINTS / SEC
CHANNEL OFF-SCALE WARNING = [OFF]
OFF-SCALE WARNING LIMIT = [OFF]
CHANNEL ZERO SCALE WARNING = [OFF]
INVERT RAW DATA? Y/N [N]

9.19.2 Calculations Setup Parameters (Calculations Submenu: F5 - SETUP)

CHANNEL #:	3
Plot curve Y/N	N
Auto/Interactive	INTER
Decimal places 0-7	3
First sample #:	1
Peak Height/Area	HEIGHT
Threshold 1-300	15
Ascending Slope 0-100	1
Apex 1-100	10
Descending Slope 0-100	1
Plateau Points 0-100	1
Integration Points 1-100	3
Execute Y/N	Y

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3120.1	SILICA (SiO_2)	1.1	SM4500SiD (Modified)	EFFECTIVE
PAGE 9 OF 10				DATE 12/01/96

APPROVALS/DATE			
DIVISION DIRECTOR 8/26/97	LAB SUPERVISOR 5-7-97	QUALITY ASSURANCE 6-22-97	METHODS 5/5/97

SECTION 9.0 - DATA HANDLING (CONT'D)

9.19.3 Standards Table (Main Menu: F7 - STANDARDS TABLE)

Calibration Code:	1 (Linear Least-Squares)
Units:	mg/L
Name:	SIO2
S1	21.3
S2	10.65
S3	5.33
S4	2.67
S5	0
S6..S20	0

SECTION 10.0 - INSTRUMENT CLEANUP/SHUTDOWN/TROUBLESHOOTING

10.1 Remove stannous chloride reagent line 2 to 3 minutes before other reagent lines. Pre-rinse all lines in clean DI water.

10.2 Place all lines in clean DI water and flush instrument for 20 - 30 minutes.

10.3 Turn pump module off and unlatch platens.

10.4 Turn off light source and power module.

10.5 Dispose of all sample cups, clean the work area, and rinse and store all glassware.

10.6 If troubleshooting is necessary, refer to the RFA manual - troubleshooting section. Document all troubleshooting and maintenance in the instrument maintenance notebook.

10.7 Consult the Shift Supervisor Officer before making any major changes, adjustments or repairs to the instrument.

SECTION 11.0 - REFERENCES

11.1 EPA Methods for Analysis of Water and Wastes, EPA-600/4-79-020, March 1979.

11.2 Standard Method for the Examination of Water and Wastewater, 17th Edition

METHOD #.	METHOD NAME	REVISION	REFERENCE	STATUS
3120.1	SILICA (SiO_2)	1.1	SM4500SiD (Modified)	EFFECTIVE
PAGE 10 OF 10				DATE: 12/01/96

APPROVALS/DATE			
DIVISION DIRECTOR	LAB SUPERVISOR	QUALITY ASSURANCE	METHODS
8/14/97	DBT 5-7-97	8-22-97	5/5/97

SECTION 11.0 - REFERENCES (CONT'D)

11.3 ACS LIMS Users Guide, version 1.0, 1992

11.4 SFWMD Comprehensive Quality Assurance Manual, current version.

11.5 ALPKEM RFA 300 series Operator's Manual

11.6 ALPKEM Softpac Software Manual

Soluble Reactive Phosphate

Reference: Stannous Chloride - APHA Standard Methods. 15th ed.. p. 417. Method 424E (1980).

Equipment: Spectrophotometer. Hach 2000

Reagents: The reagents for use in this procedure are purchased as part of a test kit. K-8513, Phosphate (M-Blue). 0-4 ppm. from Chemetrics. Inc.. Route 28. Calverton. Virginia. 22016-0214. The ammonium molybdate (R-8513) is supplied in evacuated 13mm diameter glass ampoules. The stannous chloride (A-8500) is supplied in a plastic dropper bottle. Each kit contains enough reagent for testing 30 samples.

Standards: Stock solution = 1000 mg/l PO4-P = 4.394 grams potassium phosphate, monobasic (KH2PO4) dissolved in 1 liter deionized (18 megohm) water. This solution is prepared monthly in West Palm Beach by the Lab QA Officer.

Working standard solutions are prepared from the stock and preserved with 10 drops of 50% sulfuric acid before dilution to 1000 ml.

4.0 mg/L = 40 ml stock diluted to 1000 ml with di H_2O
2.0 mg/L = 20 ml stock diluted to 1000 ml with di H_2O
1.5 mg/L = 15 ml stock diluted to 1000 ml with di H_2O
1.0 mg/L = 10 ml stock diluted to 1000 ml with di H_2O
0.5 mg/L = 5 ml stock diluted to 1000 ml with di H_2O
Blank = 1000 ml di H_2O

Standard Additions (Spikes): 5.0 ml of the 2.0 mg/L working standard is added to 20 ml of sample. Standard addition concentration is equal to 0.80 times the samples concentration plus 0.5 mg/L.

QC Check Solutions (Known): Prepared monthly by the Laboratory Quality Assurance Officer in West Palm Beach.

Procedure: Sample and standard treatment: 25 mls of sample or standard is poured into a beaker. Two drops of stannous chloride (A-8500) is added and mixed well. The tip of the evacuated ampoule containing the ammmonium molybdate solution is broken off under the surface of the sample. The ampoules then fill automatically. The ampoule is inverted several times to mix the sample and solution and the color is allowed to develop for at least 10 minutes but less than 30 minutes. The absorbance is measured by inserting the ampoule into the spectrophotometer.

Calibration: The spectrophotometer is calibrated by treating the working standards and deionized water blank by the procedure described above. The absorbance is measured at 690 nm and a calibration curve is generated.

Quality Control Procedures

1. Calibration standards are run every 20 samples.
2. A standard addition is run every 10 samples.
3. A QC check samples is run every 10 samples.
4. A repeat (duplicate) is run every 10 samples.

The results from all quality control samples must fall within the current acceptable limit ranges

General Description This method measures reactive (ortho) phosphate on an unfiltered sample Suspended sediment is not found to produce significant interference due to its settling out during the 10 - 30 minute color development time. Similarly, at this wavelength, sample color does not interfere. The sample values range between the laboratory values for total phosphorus and orthophosphate. The sensitivity of this procedure is 0.02 mg/L.

METHOD	METHOD NAME	REVISION	REFERENCE	STATUS
3160.1	ACID AND ALKALINE PHOSPHATASE ACTIVITY (APA)	00	Pettersson and Jansson, 1978	EFFECTIVE
PAGE 1 OF 8				DATE 10-01-94

APPROVALS/DATE			
DIVISION DIRECTOR 11/12/94	LAB SUPERVISOR 11-1-94	QUALITY ASSURANCE 11-1-94	METHODS DPDuarte

SECTION 1.0 - TABLE OF CONTENTS

SECTION 2.0 - METHOD DESCRIPTION/HISTORY

2.1 Phosphatases can be associated with algal and bacterial cell wall and released into water by disintegrating algal cells. These enzymes catalyzes the hydrolysis of phosphomonoesters to orthophosphate and an alcohol:

$$R\text{-}PO_4 + H_2O \longrightarrow ROH + H_2PO_4$$

2.2 Phosphatases are classified as either acid or alkaline, depending on the pH of the environment in which they exist. The determination of acid phosphatase activity (APA) and alkaline phosphatase activity is conducted at this native pH by adjusting the pH of the buffer solution.

2.3 The substrate used in this assay is methylumbelliferyl phosphate (MUP), which has a low background fluorescence, thus allowing assay of wide variety of concentration with very high sensitivity. The amount of substrate added is determined by preparing increasing amount of substrate solution. V_{max} is calculated as the optimum amount of substrate for enzymatic hydrolysis.

2.4 Basically, MUP is prepared in a pH adjusted buffer and added into the sample. The phosphatase enzyme that maybe present in the sample will hydrolyze MUP into methylumbelliferone and phosphate. Methylumbelliferone fluoresces at a specific wavelength when excited with UV light and can be quantified by a spectrophotometer or a fluorometer. A computer aided Cytofluor, a fluorescence plate scanner, is used in our laboratory to perform the analysis.

METHOD	METHOD NAME	REVISION	REFERENCE	STATUS
3160.1	ACID AND ALKALINE PHOSPHATASE ACTIVITY (APA)	00	Pettersson and Jansson, 1978	EFFECTIVE
PAGE 2 OF 8				DATE 10-01-94

APPROVALS/DATE			
DIVISION DIRECTOR 11/2/94	LAB SUPERVISOR 10-11-94	QUALITY ASSURANCE 11-1-94	METHODS DBDuvall

SECTION 3.0 - SAFETY PRACTICES

3.1 All personnel conducting this method should be familiar with the SFWMD Chemical Hygiene Plan and should have reviewed any pertinent Material Safety Data Sheets.

3.2 No hazardous wastes are generated by this procedure; liquid waste may be disposed of in the sink.

3.3 Follow the Personal Safety Protection Codes below during analysis:

A = Lab coat, glasses, gloves
B = Lab coat, apron, acid resistant gloves, face shield+goggles+respirator or full-face respirator
C = Lab coat, acid resistant gloves, goggles, face shield, apron
D = Lab coat, acid resistant gloves, glasses
E = Lab coat, glasses, apron, gloves
F = Rubber acid carrier
G = Flush sink drain with ample amount of tap water

SECTION 4.0 - LIST OF EQUIPMENT/INSTRUMENTATION

4.1 Millipore CytoFluor 2350 Multiwell Fluorescence Plate Reader

4.2 IBM-compatible PC, with Windows 3.1 and Excel 3.0, and a printer

4.3 Cytofluor computer interface

4.4 Multiwell Low fluorescence plates (24 wells), opaqued

4.5 Multiwell pipetter, 0-150 µL capacity

4.6 Eppendorf Micropipettor, adjustable 250 µL-1000 µL

4.7 Analytical balance, 0.1 mg sensitivity

4.8 Volumetric flasks, 10 and 1000 mL capacity

4.9 Graduated pipets, 1, 2, 5, 10 mL capacity

4.10 Freezer

4.11 Incubator, ambient to 40°C

SECTION 5.0 - REAGENTS

5.1 Tris stock buffer: Prepare a 0.1M solution of Tris (base) buffer. Add 12.11 g Tris to 1000 mL volumetric flask and add enough Millipore water to bring it into solution. Add 0.2037 g of anhydrous $MgSO_4$, to get a final concentration of 0.01M. (Safety PP = A)

5.2 Working buffer A: Measure 100 mL of Tris stock buffer into 1L volumetric flask. Bring to volume using sterile Millipore water. Adjust the pH to **pH 8.00 for alkaline phosphatase**, by slowly adding 1N HCl while stirring. The final concentration of this solution is 0.01MTris/0.001M $MgSO_4$. (Safety PP = A)

5.3 Working buffer B: Measure 100 mL of Tris stock buffer into 1L volumetric flask. Bring to volume using sterile Millipore water. Adjust the **pH 6.5** to for **acid phosphatase**, by slowly adding 1N HCl while stirring. **The final concentration of this solution is 0.01MTris/0.001M $MgSO_4$. (Safety PP = A)**

5.4 Substrate : Methylumbelliferyl phosphate (MUP) (FW=256.2). Weigh 0.128 g of MUP and bring to 250 mL volume using the TRIS stock buffer. **(Safety PP = A)**

SECTION 6.0 - STANDARDS

6.1 Stock standard (1000µM MU): Dry approximately 1g of methylumbelliferone (MU) overnight at 105 °C. Weigh out 0.1982 g of oven-dried MU into a 1L volumetric flask and dilute to volume with working Tris buffer A or B. Keep at **room temperature**, in a dark container.

6.2 Secondary stock standard (10 µM MU): Pipet 100 µL of stock MU solution into a 10 mL volumetric flask and add working Tris buffer A or B to volume.

6.3 Working Standards

Std.	Methylumbelliferone conc.,		Vol. of Stock MU, mL	Final Volume, mL (Add Tris Buffer C to volume)
	µM	nM		
S1	0.1	100	0.1	10
S2	0.3	300	0.3	10
S3	0.5	500	0.5	10
S4	1.0	1000	1.0	10
S5	2.0	2000	2.0	10

METHOD	METHOD NAME	REVISION	REFERENCE	STATUS
3160.1	ACID AND ALKALINE PHOSPHATASE ACTIVITY (APA)	00	Pettersson and Jansson, 1978	EFFECTIVE
PAGE 4 OF 8				DATE 10-01-94

APPROVALS/DATE

DIVISION DIRECTOR	LAB SUPERVISOR	QUALITY ASSURANCE	METHODS
11/12/94	10-4-94	1-11-94	RBDuarte

SECTION 7.0 - QUALITY CONTROL

7.1 Run a duplicate set of each working standard for each analysis. Record the fluorescence value on the instrument log.

7.2 Run a QC solution after a set of working standard and at the end of each analysis.

7.3 A repeat analysis should be conducted for every 20 samples analyzed. These results are recorded on QC sheet and instrument log. The mean and coefficient of variation of the replicate set is determined and noted on the QC sheet and physical parameters log.

7.4 All quality control data must be within the current established limits before entering sample data into the LIMS system. Consult the supervisor or QA officer if unable to obtain acceptable QC result.

7.5 Samples should be mixed thoroughly each time when taking an aliquot.

SECTION 8.0 - STEP-BY-STEP PROCEDURE

8.1 Create a workgroup for APA (Product). Refer to the ACS LIMS users Guide for instructions on "Creation of a Workgroup" and to Section 9.2.1 and Section 9.2.1. Remove the samples to be tested from the refrigerator and allow them to warm to room temperature (this may require several hours).

8.2 Turn on the Cytofluour and allow it to warm up for at least 15 minutes. Select the CYTOCALC Program (double click with the mouse).

8.3 Determine the pH of the water samples by referring to the hydrolab data or by laboratory measurement with a pH meter. Prepare a fresh set of working Tris buffer A or B, based on the pH range of the samples to analyze, and by following the procedure in Section 5.0. (Therefore, TRIS buffer A should be prepared and used if sample pH is greater than 7, and TRIS buffer B should be used when the sample pH is less than 7).

8.4 Prepare a fresh set of working standards, as described in Section 6.0.

8.5 The well plate template is displayed automatically when entering CYTOCALC program. If the 24-well plate template is not displayed, open the file 24well.CFL (file, open, 24 well.CFL), or the most recent run file with 24 well. Edit the plate protocol by entering the last four digits of the sample number. Use one plate for every 10 samples. It is important to have a duplicate of each standard for the CYTOCALC to function.

METHOD	METHOD NAME	REVISION	REFERENCE	STATUS
3160.1	ACID AND ALKALINE PHOSPHATASE ACTIVITY (APA)	00	Pettersson and Jansson, 1978	EFFECTIVE
PAGE 5 OF 8				DATE 10-01-94

APPROVALS/DATES			
DIVISION DIRECTOR 11/02/94	LAB SUPERVISOR 10-11-94	QUALITY ASSURANCE 1-11-94	METHODS

SECTION 8.0 (CON'T)

8.6 Alternatively, a new protocol can be created by following these steps:

a. On the well displayed on the screen, click the well you want to use.

b. On the upper right hand corner of the screen, click the well assignment you need (Bl=Blank, Un=Sample, St=Standard, Co=Control, Em=Empty)

c. On the edit bar located at the upper portion of the screen, edit the correct I.D. You will need to enter the concentration values of each standard and control.

8.7 Measure 2 mL of blank, standard and QC into each assigned well.

8.8 Pipet 1.8 mL of samples according to the tray protocol. Add 200 µL of MUP into each sample well. **DO NOT ADD MUP SUBSTRATE TO STANDARD, BLANK AND QC WELLS.**

8.9 Immediately place the well plate in the door transport, ensure proper seating of the plate and that the first well is on the top right position. Scan within 10 seconds (Click RUN, COLLECT DATA, then enter WORKGROUP number under file name). Record the exact time of the start of the incubation. The system will automatically scan for time 0 reading, then a message "50 % completed will be displayed" on the scan window. (This means that a second scan will resume after 1800 seconds or 30 minutes).

8.10 At the end of the second scan, the system will prompt a message, "100 % completed", and will give you an option to CLOSE DOOR or IGNORE DOOR. Choose IGNORE DOOR if another plate is to be scanned, and CLOSE DOOR if no more plate is to be scanned.

8.11 At this stage, a message may appear "Cytofluor setting has been changed". Hit OK to display the plate. Note that the fluorescence data will be displayed on the plate layout.

8.12 On the top right corner of the screen, depress the LINK button. Note that any associated well (standard, blanks, QC and sample well) is highlighted (black background). Double click each well that is not highlighted. IMPORTANT: Only highlighted well (blackened background) will be calculated with the standard curve.

8.13 After highlighting each well, depress the DATA button on the right hand corner of the screen. The system will prompt "ACCEPT CURRENT LINK?". If this is your final link, select YES to proceed. Otherwise, depress the plate button on the upper right hand corner of the screen and make any ID corrections. (At this stage, any outlier standard or sample can be hidden to exclude from calculations. Press the HI button).

SECTION 9.0 - DATA HANDLING

9.1 Cytofluour PC-Data Handling

8.14 (After LINK command from Steps 8.12 and 8.13) On the menu bar, select DATA, FORMAT. Specify SCAN 1, then press CALCULATE (Ensure that **Calculate and Statistics are marked with X on the screen).**

8.15 The computer will automatically go to Excel Program, and display the worksheet with the calculated values. Enter the complete LIMS ID for each sample and QC.

8.16 Select FILE, PRINT to print the time 0 worksheet. Select FILE, SAVE AS, and give file name (.csv) to save file

8.17 Select FILE, END DATA DISPLAY to return to Calculate window. Specify Scan 2. Repeat steps 8.14 to 8.16 to calculate, print and save results of SCAN 2. Save as Filename.csv.

9.2 APA Calculation (PC Windows-Excel)

9.2.1 To calculate final APA values in nM/min-mL, the equation below is used:

$$\text{APA (nM/min-mL)} = \frac{\text{MU conc. at time 0 (µM)} - \text{MU conc. after 30 minutes (µM)}}{(30 \text{ minutes} \times 1.8 \text{ mL})} \times 1000$$

This equation is entered into Excel worksheet, with filename APA.XLM. Calculation can be done in these worksheet by opening this file, then the time 0 and time 30 minutes files. Copy and paste the time 0 values into APA.XLM worksheet. Repeat to copy time 30 values into the calculation worksheet. Calculation will be automatic. Review the QC and blank values to ensure accurate calculations.

9.2.2 The calculated APA values can be sent to LIMS following Step 9.2.2.

METHOD	METHOD NAME	REVISION	REFERENCE	STATUS
3160.1	ACID AND ALKALINE PHOSPHATASE ACTIVITY (APA)	00	Pettersson and Jansson, 1978	EFFECTIVE
PAGE 7 OF 8				DATE 10-01-94

APPROVALS/DATE			
DIVISION DIRECTOR 11/12/94	LAB SUPERVISOR 10-11-94	QUALITY ASSURANCE 1-11-94	METHODS

SECTION 9.0 - DATA HANDLING (CON'T)

9.2 LIMS Database

9.2.1 Prior to running samples: Go into the ACS LIMS via the command sl and create a workgroup for the test APA and exit the SEEDPAK1 Main Menu (see Creating a Workgroup and Printing a Workgroup in the ACS LIMS Users Guide).

9.2.2 After running the samples: Examine the results for acceptability of QC samples and repeats. either by manual entry or by transfer from disk through the LIMS Database Analyst. To enter manually, go the WORKSTAT, MANUAL ENTRY, APA. Enter the workgroup number. This will prompt the sample numbers one by one. Enter the concentration results.

9.2.2 Pick up the LIMS data entry report for the samples you have just entered from the system printer and examine against the raw data report. Submit any necessary corrections to the DATA UNIT by filling a LIMS Database Correction form.

SECTION 10.0 - INSTRUMENT CLEANUP/SHUTDOWN/TROUBLESHOOTING

10.1 Wipe dry the surfaces of the plate incubator, the Cytofluor and the work areas. Use D.I. water to clean any spill on the instrument.

10.2 Rinse the plate thoroughly with dilute Liquinox™ and D. I. water. Place the plate upside down on a paper towel, to dry. Discard the plate if any visible crack or scratches is observed.

SECTION 11.0 - REFERENCES

11.1 Pettersson, K. and M. Jansson. 1978. Determination of phosphatase activity in lake water-a study of methods. Verh. Internat. Verein. Limnol. 20:1226-1230.

11.2 Prof. Robert G. Wetzel. 1994. Personnal Communication. Department of Biological Sciences, The University of Alabama, Tuscaloosa, Alabama 35487-0344, USA.

11.3 Cytofluor 2300 Manual. 1992. Millipore Corporation, Bedford, MA.

METHOD	METHOD NAME	REVISION	REFERENCE	STATUS
3160.1	ACID AND ALKALINE PHOSPHATASE ACTIVITY (APA)	00	Pettersson and Jansson, 1978	EFFECTIVE
PAGE 8 OF 8				DATE 10-01-94

APPROVALS/DATE

DIVISION DIRECTOR	LAB SUPERVISOR	QUALITY ASSURANCE	METHODS
11/2/94	10-11-94	11-94	D B Ivanoff

FIGURE 1.0 SAMPLE MULTIWELL PROTOCOL (24-well plate)

(A1) S1 (2000 nM)	(A2) S2 (1000 nM)	(A3) S3 (500 nM)	(A4) S4 (300 nM)	(A5) S5 (100 nM)	(A6) BLANK
(B1) S1 (2000 nM)	(B2) S2 (1000 nM)	(B3) S3 (500 nM)	(B4) S4 (300 nM)	(B5) S5 (100 nM)	(B6) BLANK
(C1) QC1	(C2) QC2	(C3) Sample	(C4) Sample	(C5) Sample	(C6) Sample
(D1) Sample	(D2) Sample	(D3) Sample	(D4) Sample	(D5) Sample	(D6) RPT

ER Biology Section

v. 1—7/6/93

SOP #BA-7
Benthic Macroinvertebrate Dip Net Sample Collection

(based on Plafkin, et al, 1989, *Rapid bioassessment protocols for use in streams and rivers: benthic macroinvertebrates and fish*, EPA/444/4-89-001)

STEPS	COMMENTS
aterials	
Field Physical/Chemical Characterization Data Sheet *Habitat Assessment Sheet* Dip Net with No. 30 mesh 4-liter wide-mouth plastic jugs 100% formalin	Prepare per SOP #BA-2.1
ethods	
Visually examine the area or reach to be sampled. You must either walk or boat throughout the aquatic system, paying close attention to its physical and habitat characteristics.	In fairly small (1st to 4th order) streams, the length of a discrete station should consist of a 100 m stretch of stream, and the width should be from bank to bank. In very large systems it may be necessary to establish more than one station to adequately characterize the biota.
Fill out *Field Physical/Chemical Characterization Data Sheet* and *Habitat Assessment Sheet*. The percent coverage of substrate type refers to how much of each habitat type is actually present at the sampling site.	See SOP's #BA-17 and BA-18 for instructions on filling out these forms. It is important to accurately determine the spatial extent of each substrate type (in a 3 dimensional context) for habitat scoring procedures.
Determine the number of sweeps to perform in each habitat type out of the 20 total sweeps per station. This requires a two step process. First, select the "major" or "most productive" habitats for the stream type. Use the following formula to calculate the number of sweeps in each habitat type:	Generally, the most (to least) productive habitat types are as follows: snags, aquatic vegetation, leaf packs, roots, undercut banks, rocky outcrops, muck, and sand. All but the last two can be considered "major" or "productive".
$\text{Number of sweeps per major Habitat} = \frac{20}{(\text{Number of Major Habitats}) + 1}$	Example: If 3 major habitat types are present, perform 5 sweeps in each of these habitats and divide the remaining 5 sweeps up among the other non-major habitats, so that a total of 20 sweeps are performed. If 4 major habitats are present, perform 4 sweeps in each of these, then 4 divided up among the remaining types. For 5 major habitats do 3 in each major habitat and divide the remaining 5 sweeps up among the other non-major types.
The result is rounded to the nearest integer. The remaining number of sweeps (to make a total of 20) is evenly divided among the minor habitats (such as sand, mud, or muck in most cases).	Proper interpretation of benthic collections requires that samples be collected from multiple habitats that are representative of the site. If possible, the same habitats should be sampled at reference and test sites the same number of times to isolate the effects of water quality on the benthic community.

DER Biology Section

v. 1—7/6/93

STEPS	COMMENTS
4. Perform 20 discrete 0.5 meter sweeps with the dip net. Sample the available substrates as determined by the above procedures.	When performing an upstream/downstream type of study, sample the downstream station first to prevent upstream invertebrates from drifting into a location they were not originally inhabiting.
a. In streams with sufficient water velocity, the most effective way to capture invertebrates is to place the bottom rim of the dip net downstream of the area to be sampled. Disturb, agitate, or dislodge organisms (with hands and/or feet) from substrates (snags, etc.) at a distance of 0.5 m upstream of the net.	Catch organisms by allowing them to flow into the net and also by sweeping the net towards disturbed material.
b. For areas without flow, disturb an area of substrate that is one dip net width wide and approximately 0.5 m long; and sweep the net over the area a few times to ensure the capture of organisms which were living there.	Several sweeps over the same 0.5 meter area are recommended to make sure all organisms are captured. This sampling effort in a discrete 0.5 meter spot is considered as 1 sweep.
c. **For heavily vegetated areas** (some streams, lake margins, or wetlands) jab the net into the base of the vegetation, digging down to the substrate, and dislodge organisms using a one-half meter sweeping motion with the net.	**Where a continuous half meter sweep is impossible, take 2 quarter meter sweeps in the same area to attain a full 0.5 meter sweep.**
d. Sample leaf packs (if present) by disturbing leaf pack areas with hands or feet before scooping one-half meter worth of material into the net.	
e. Sand, muck, mud, and silt (non-major habitats) can be sampled by taking 0.5 meter sweeps with the net while digging into the bottom approximately 1 cm.	If the net is pushed too deep in course sand, very little of the sand will be washed through the net resulting in a sample that contains few organisms and is hard to process.
5. Record the number of sweeps for each habitat on the *Field Physical / Chemical Characterization Data Sheet*.	Ideally, control and test sites will be sampled the same number of times in the same habitats.
6. Reduce the sample volume after each discrete sample by dislodging organisms from larger debris (but retaining invertebrates in the net or sieve) and discarding the debris. Save finer debris plus organism mixture in large wide mouth jugs. Try to reduce enough of the sample volume in the field so that no more than 2 gallons of material are collected. If this is not possible, put the material into additional jugs. Sample reduction is easier in the laboratory.	The relative proportions of the organisms collected must be maintained intact to calculate many community metrics. Some field picking of delicate organisms is acceptable as long as community composition is not altered. Indicate on the label how many jugs the entire sample is contained in, *e.g.*, "1 of 2", "2 of 2".

DER Biology Section

v. 1—7/6/93

STEPS	COMMENTS
7. If laboratory sorting can be performed within 24 hours, place samples immediately on ice. Cold temperatures should slow organisms enough to prevent predation (and subsequent alteration of community structure). If sorting will be delayed, preserve with 10% formalin (do this by adding one part of 100% formalin to the jug with 9 parts ambient water). After organisms have been removed from detritus, they should be placed into 70% ethanol.	If organisms are too active during sorting, pour some carbonated water, clove oil, or other relaxing agent into the sample. Samples that will not be sorted within 2 days should be preserved in formalin. Ethanol alone will not prevent the vegetative debris from decomposing, resulting in a sample that is very unpleasant to sort.

DER Biology Section

v. 1—7/6/93

SOP #BA-8
Benthic Macroinvertebrate Qualitative (Dip Net) Sample Handling

STEPS	COMMENTS
Materials	
1. Waterproof paper and permanent marker	For making labels
2. U.S. 30 mesh sieve	
3. U.S. 10 mesh sieve	
4. Ethanol filled squeeze bottle (80%)	
5. White enamel pan, marked with a grid of 5 cm squares	
6. List of random numbers	
7. 250-mL glass jar	
8. Dissecting microscope	
9. 100 x 15 mm petri dish	
10. Forceps	
11. Vials for picked organisms (1 or 2 dram)	
12. Laboratory counter	
13. *Benthic Macroinvertebrate Bench Sheet*	
Methods	
1. Check labels so you know which sample you are dealing with (control site, test site, etc.). Make a very clear label to go into the bottle of picked bugs with station identification, date sampled, replicate number, and your initials.	Make sure that you know how many containers in which the particular sample is stored (there may be several jugs). The entire sample must be included in this reduction and homogenization process.
2. Place a portion of the contents of the sample (fist sized) into a U.S. 10 mesh sieve with a U.S. 30 mesh sieve underneath.	
3. Rinse with tap water (a small hose attached to the faucet works best), spraying organisms and small detritus down into the U.S. 30 mesh sieve. Visually inspect large debris (leaves, plants, twigs) held in the U.S. 10 mesh sieve for animals before discarding. Wash fine debris (silt, mud) through the bottom (U.S. 30 mesh) sieve . Repeat procedures #2 and #3 until all the sample from all the jugs has been processed.	This inspection is best accomplished by placing the debris in a white pan and observing it with the Luxo® lighted magnifier. Organisms found (generally the ones too large to pass through the U.S. 10 mesh sieve) should be placed into the U.S. 30 mesh sieve with the rest of the unpicked sample.
4. Place sample in gridded pan. Each 5 cm grid should have a pre-assigned number. Liquid present in the sample should be sufficiently reduced to prevent material from shifting among grids during the sorting process.	There are 24 total 5 cm grids in a standard white enamel pan.

DER Biology Section

v. 1—7/6/93

SOP #BA-8.1
Preparation of 80% Ethanol

STEPS	COMMENTS
Materials	
1. 100% Ethanol (HPLC grade) 2. D.I. water 3. 4000-mL graduated cylinder 4. 4-L glass amber jug 5. Plastic funnel to fit into 4-L jug	
Methods	
1. Fill graduated cylinder with 3200 mL of 100 % ethanol.	This operation is easier with two people as the ethanol is purchased in 5-gallon cans. One person holds the cylinder while the other pours the ethanol.
2. Add D.I. water to the graduated cylinder until the total volume is 4000 mL.	
3. Using the funnel, transfer the dilute ethanol to the 4-L jug which should be properly labeled as 80% ethanol.	
4. Rinse graduated cylinder and funnel with D.I. water and return them to the shelf above the sink.	

DER Biology Section

v. 1—7/6/93

STEPS	COMMENTS
5. Thoroughly mix the sample so that a homogenous distribution of organisms is achieved in the detrital matrix.	
6. Select a grid using the random number table. Remove the contents of the entire grid and place in a glass jar.	Use a ruler to delineate the edges of a grid while removing the sample.
7. Take a small amount of this detritus plus organism mixture and place it in the bottom portion of a petri dish. Add enough liquid (usually ethanol) so that the material is wet enough to move around easily with forceps.	The top portion of the petri dish may later be placed over the sample to prevent desiccation if you must leave the sample overnight.
8. Place the petri dish under a dissecting scope set at low power (approximately 7x or 10x). In a deliberate, systematic manner scan back and forth or up and down picking each and every organism from the aliquot and placing it into an alcohol-filled vial (clearly identified as per step #1).	Using a pair of forceps in each hand enables you to better tease organisms out of fibrous detritus. Forceps should be sharp and properly aligned. Use the laboratory counter to keep a running total of the number of organisms picked.
9. After scanning dish in one pattern (*e.g.*, up and down), go back through using a different pattern (back and forth) to assure that all organisms have been removed from the aliquot.	Picking accuracy should be checked by a co-worker in 10 % of the samples.
10. Continue steps 6, 7, 8, and 9, until you obtain a minimum of 100 organisms. Once a grid is selected, its entire contents must be sorted.	If an obvious organism is observed but its grid number was not selected and no examples of that organism were present in grids which were selected, that organism may be noted as qualitatively observed. The organism should NOT be included in the analysis.
11. Record the information requested on the *Macroinvertebrate Lab Bench Sheet* which includes site, laboratory sample number, STORET station number, sample type, replicate number, and date collected. Include the initials of the persons who collected and sorted the sample. Record the number of grids selected (*e.g.*, "4 of 24") to enable conversion to total abundance present in the original sample.	Failure to record the number of grids selected (out of the total grids possible) seriously compromises the usefulness of the data.

DER Biology Section

v. 1—7/6/93

SOP #BA-9
Benthic Macroinvertebrate Grab Sample Collection
(Modified from *Standard Methods 10500B.3*)

STEPS	COMMENTS
Materials	
1. Ekman or Petite Ponar dredge	
2. U.S. 30 mesh box sieve	
3. White enamel pan	
4. Plastic squeeze bulb ("turkey baster")	
5. Small bucket	
6. Wide mouth plastic sample containers	
7. Tape and permanent markers	For making labels
Methods	
1. Use of the Ekman dredge is restricted to sampling soft substrates (silt, muck) in areas with little current. The Ponar dredge may be used for sampling under these conditions and also in areas with a harder substrate (rocks, shells, sand).	The number of replicates collected is dependent upon several factors including the area sampled by the device, the purpose of the study, and the degree of patchiness in the distribution of the organisms at the site. Routinely, take 3 dredges. All replicates are placed in separate sample containers (for statistical analyses). If it has been determined that you are sampling in an exceptionally depauperate area, additional replicates may be required (pilot study needed). In that case the number of replicates sampled at the group of stations you wish to compare should be equal.
2. When sampling from a boat, dredge samples are collected from the rear and downstream of the vessel to avoid contamination of other types of samples with disturbed sediments. Rinse the box sieve with ambient water and tie it to the side of the boat where samples will be collected.	The box sieve is constructed of fiberglass-coated wood and U.S. 30 mesh screen. When placed in the water, it will float at the surface. If a box sieve is unavailable, the dredged material may be washed in the dip net providing it is fitted with a U.S. #30 mesh sieve material. The disadvantage of using the dip net is that it requires 2 people (1 to hold the net, 1 to manipulate the dredge).
3. Ekman: Open the spring-loaded jaws and attach the chains to the pegs at the top of the sampler. Lower the dredge to the bottom, making sure it settles flat. Holding the line taught, send down the messenger to close the jaws of the Ekman dredge. Pull the sampler to the surface and place it immediately into the box sieve. Carefully open the jaws and disgorge the contents into the sieve, rinsing to assure complete sample purging.	The spring-loaded Ekman is dangerous. Hold the dredge firmly above the hinges, and be very careful that no body parts get pinched by the snapping jaws, which could produce serious injury. Check to make sure the jaws are fully closed and that no sample was lost while lifting the dredge.

DER Biology Section

v. 1—7/6/93

STEPS	COMMENTS
3. Ponar: Open the jaws and place the cross bar into the proper notch. Lower the dredge to the bottom, making sure it settles flat. When tension is removed from the line, the cross bar will drop, enabling the dredge to close as the line is pulled upward during retrieval of the dredge. Pull the Ponar to the surface and place it immediately into the box sieve. Carefully open the jaws and disgorge the contents into the sieve, rinsing to assure complete sample purging.	Check to make sure the jaws are fully closed and that no sample was lost while lifting the dredge.
4. Swirl the box sieve in the water with a back and forth motion to wash the fine sediments through. Concentrate the remaining sample into one corner of the sieve.	If a sediment type is especially clayey or mucky, it may be necessary to use a hand to break up clumps and agitate the sample to reduce it. Make sure you rinse any detritus from your hand back into the sieve.
5. Fill the small bucket with ambient water and use this water to fill the squeeze bulb. Using the squeeze bulb, rinse the sample from the sieve to the enamel pan.	Take care to rinse the entire contents of the sample into the pan. Some organisms may to stick to the screen.
6. Transfer the sample from the enamel pan into the pre-marked wide mouth jug (again, using the squeeze bulb), making sure the location, date, and replicate number is accurate.	
7. Preserve the sample with 10% formalin by adding a 10 to 1 ratio of water to 100% formalin. If laboratory processing is possible within 8 hours, the samples may be stored on ice, without addition of formalin.	Rose bengal dye may be added to the sample, as a picking aid, if desired.

R Biology Section

v. 1—7/6/93

SOP #BA-10
Benthic Macroinvertebrate Grab Sample Handling

(Modified from *Standard Methods 10500C*)

STEPS	COMMENTS
aterials	
Waterproof paper and permanent marker	For making labels
U.S. 30 mesh sieve	
Ethanol filled squeeze bottle (80%)	Prepare per SOP #BA-8.1
Glass jars	
Dissecting microscope	
100 x 15 mm petri dish	
Forceps	
Vials for picked organisms (1 or 2 dram)	
Benthic Macroinvertebrate Bench Sheet	
ethods	
Check labels so you know which sample you are dealing with (control site, test site, etc.). Make a very clear label to go into the bottle of picked bugs with station identification, date sampled, replicate number, and your initials.	It is critical that no errors be made during this step. If samples are mixed up, the entire study could be rendered invalid.
Pour the contents of the wide mouth jug over a U.S. 30 mesh sieve. Rinse the jug with tap water to make sure all organisms are put into the sieve.	
Rinse with tap water (a small hose attached to the faucet works best). Wash fine debris (silt, mud) through the sieve. Any large debris (leaves, shells, etc.) present should be brushed clean of organisms and discarded. Rinse the organism plus detritus mixture to one small area of the sieve.	
Using an ethanol-filled (80%) squeeze bottle, rinse the organism plus detritus matrix into the smallest practical container (usually a 100 mL to 250 mL glass jar). **Put the label inside the jar.**	Place jars on the sample shelf so that samples for a given study are organized together and clearly marked.
Record the information requested on the *Macroinvertebrate Lab Bench Sheet*, which includes site, laboratory sample number, STORET station number, sample type, replicate number, and date collected. Include the initials of the persons who collected and sorted the sample.	

DER Biology Section

v. 1—7/6/93

STEPS	COMMENTS
6. Take a small amount of detritus plus organism mixture and place it in the bottom half of a petri dish. Add enough liquid (usually ethanol) so that the material is wet enough to move around easily with forceps.	The top portion of the petri dish may later be placed over the sample to prevent desiccation if you must leave the sample overnight. Do not overload the petri dish with too much sample - this results in sloppy work.
7. Place the petri dish under a dissecting scope set at low power (approximately 7x or 10x). In a deliberate, systematic manner scan back and forth or up and down, picking each and every organism from the aliquot and placing it into an alcohol-filled vial (clearly marked as per step #1).	Using a pair of forceps in each hand enables you to better tease organisms out of fibrous detritus. Forceps should be sharp and properly aligned.
8. After scanning dish in one pattern (*e.g.*, up and down), go back through using a different pattern (back and forth) to assure that all organisms have been removed from the aliquot.	
9. Continue steps 6, 7, and 8 until the sample is finished.	Picking accuracy should be checked by a co-worker in 10% of the samples.

DER Biology Section

v. 1—7/6/93

SOP #BA-11
Benthic Macroinvertebrate Core Sample Collection

(Modified from *Standard Methods 10500B.3*)

STEPS	COMMENTS
Materials	
1. Coring Device	
2. U.S. 30 mesh box sieve	
3. White enamel pan	
4. Plastic squeeze bulb	
5. Small bucket	
6. Wide mouth plastic sample containers	
7. Tape and permanent markers	For making labels
Methods	
1. Use of coring devices is restricted to sampling fairly soft substrates (silt, muck, with only small amounts of sand or shell) usually in marine systems. The Biology Section uses two sizes of coring devices.	The number of replicates collected is dependent upon several factors, including the area sampled by the device, the purpose of the study, and the degree of patchiness in the distribution of the organisms at the site. Routinely, take enough cores so that an area equivalent to 3 Ponar dredges is collected (approximately 675 cm^2). With our large (4 inch diameter) coring device, collect 8 replicates to achieve this. All replicates are routinely placed in separate sample containers (for statistical analyses). Depending on the study objectives, replicates may also be composited, as long as the number of replicates is equal for each station and clearly recorded so that the number of organism per square meter can be calculated.
2. When sampling from a boat, use the 4 inch diameter coring device that is attached to a long pole, and has a valve near the top. Core samples are collected from the rear and downstream of the vessel to avoid contamination of other types of samples with disturbed sediments. Rinse the box sieve with ambient water and tie it to the side of the boat where samples will be collected.	The box sieve is constructed of fiberglass-coated wood and U.S. 30 mesh screen. When placed in the water, it will float at the surface. If a box sieve is unavailable, the dredged material may be washed in a dip net, providing it is fitted with a U.S. #30 mesh sieve material. The disadvantage of using the dip net is that it requires two people (one to hold the net, and one to manipulate the coring device).
3. Lower the coring device to the bottom with the valve open. After quickly pushing the device into the sediments, close the valve. The resulting vacuum will keep the material in the tube as it is raised up to the boat.	Many clean water organisms are somewhat motile and may elude capture if you are not quick during sampling.
4. When collecting samples in wadable waters, a smaller coring device (2 inch diameter) can be used. This corer utilizes a flapper-valve equipped stopper which is inserted into the top of the pipe. Vacuum inside the pipe holds the material until the stopper is removed.	This small corer should be used primarily for non-biological sediment sampling (grain size, metals, etc.), as it is thought to be too small to effectively capture many organisms (*e.g.*, crustaceans or tubicolous worms which are generally large in size) considered useful in impact determination.

DER Biology Section

v. 1—7/6/93

STEPS	COMMENTS
5. Pull the sampler to the surface, open the valve or remove the stopper, and place it immediately into the box sieve. Disgorge the contents into the sieve, rinsing to assure complete sample purging.	
6. Swirl the box sieve in the water with a back and forth motion to wash the fine sediments through. Concentrate the remaining sample into one corner of the sieve.	If a sediment type is especially clayey or mucky, it may be necessary to use a hand to break up clumps and agitate the sample to reduce it. Make sure you rinse any detritus from your hand back into the sieve.
7. Fill the small bucket with ambient water and use this water to fill the squeeze bulb. Using the squeeze bulb, rinse the sample from the sieve to the enamel pan.	Take care to rinse the entire contents of the sample into the pan. Some organisms may to stick to the screen.
8. Transfer the sample from the enamel pan into the pre-marked wide mouth jug (again, using the squeeze bulb), making sure the location, date, and replicate number is accurate.	
9. Preserve the sample with 10% formalin by adding a 10 to 1 ratio of water to 100% formalin. If laboratory processing is possible within 8 hours, the samples may be stored on ice, without addition of formalin.	Rose bengal dye (use a very small amount) may be added to the sample, as a picking aid, if desired.

DER Biology Section

v. 1—7/6/93

SOP #BA-12
Benthic Macroinvertebrate Core Sample Handling
(Modified from *Standard Methods 10500C*)

STEPS	COMMENTS
Materials	
1. Waterproof paper and permanent marker	For making labels
2. U.S. 30 mesh sieve	
3. Ethanol filled squeeze bottle (80%)	
4. Glass jars	
5. Dissecting microscope	
6. 100 x 15 mm petri dish	
7. Forceps	
8. Vials for picked organisms (1 or 2 dram)	
9. *Benthic Macroinvertebrate Bench Sheet*	
Methods	
1. Check labels so you know which sample you are dealing with (control site, test site, etc.). Make a very clear label to go into the bottle of picked bugs with station identification, date sampled, replicate number, and your initials.	It is critical that no errors be made during this step. If samples are mixed up, the entire study could be rendered invalid.
2. Pour the contents of the sample container (wide mouth jug or Whirl-pak bag) over a U.S. 30 mesh sieve. Rinse the container with tap water to make sure all organisms are put into the sieve.	
3. Rinse with tap water (a small hose attached to the faucet works best). Wash fine debris (silt, mud) through the sieve. Any large debris (leaves, twigs, etc.) present should be brushed clean of organisms and discarded. Rinse the organism plus detritus mixture to one small area of the sieve.	
4. Using an ethanol-filled (80%) squeeze bottle, rinse the organism plus detritus matrix into the smallest practical container (usually a 100 mL to 250 mL glass jar). **Put the label inside the jar.**	Place jars on the sample shelf so that samples for a given study are organized together and clearly marked.
5. Record the information requested on the *Macroinvertebrate Lab Bench Sheet*, which includes site, laboratory sample number, STORET station number, sample type, replicate number, and date collected. Include the initials of the persons who collected and sorted the sample.	

DER Biology Section

v. 1—7/6/93

STEPS	COMMENTS
6. Take a small amount of detritus plus organism mixture and place it in the bottom half of a petri dish. Add enough liquid (usually ethanol) so that the material is wet enough to move around easily with forceps.	The top portion of the petri dish may later be placed over the sample to prevent desiccation if you must leave the sample overnight. Do not overload the petri dish with too much sample. this results in sloppy work.
7. Place the petri dish under a dissecting scope set at low power (approximately 7x or 10x). In a deliberate, systematic manner, scan back and forth or up and down, picking each and every organism from the aliquot and placing it into an alcohol-filled vial (clearly marked as per step #1).	Using a pair of forceps in each hand enables you to better tease organisms out of fibrous detritus. Forceps should be sharp and properly aligned.
8. After scanning dish in one pattern (*e.g.*, up and down), go back through using a different pattern (back and forth) to assure that all organisms have been removed from the aliquot.	
9. Continue steps 6, 7, and 8 until the sample is finished.	Picking accuracy should be checked by a co-worker in 10% of the samples.

ER Biology Section

v. 1—7/6/93

SOP #BA-13
Benthic Macroinvertebrate Hester-Dendy Sample Collection

(Modified from *Standard Methods 10500B.5*)

STEPS	COMMENTS
Materials	
3 or 4 Hester-Dendy artificial substrates	
Customized Hester-Dendy block, with coupling nuts for attachment of HD samplers and eye bolts for attachment of cable	
Stainless steel cable	
Nico-press® tool with fasteners	
Whirl-pak® bags	
Permanent marker.	For labeling bags
Methods	
Attach three HD's to the HD block, and place the block at a depth of one meter (or the deepest spot available if shallower than one meter). Take care to place control and test site blocks in areas of similar flow and habitat type. Space for 4 H-D's has been provided on the block, for use in studies requiring additional replication.	Knowledge of the system's hydrologic regime is important to make sure samplers will not go dry during the 28 day incubation period. For example, if it is flood stage and you expect the water to drop 2 meters in the next few weeks, place sampler so that it will be 1 meter deep at the end of incubation. In shifting sand substrates place the block so that existing snags will deflect sand from being deposited on the samplers. This can be determined by close examination of the bottom topography.
Attach cable to a point on the bank sufficiently high to enable recovery even if the water level increases.	Wrap the cable around the base of a tree on the bank and use the Nico-press® tool and fasteners to secure the block. If vandalism is a potential problem, attempt to conceal the cable so that no one but you can find it. If the Nico-press® tool is unavailable, the fasteners may be crimped by hammering (two hammers are needed).
After a 28 day incubation period, recover the HD samplers. Approach the block carefully, without disturbance, from the downstream position. In a deliberate, gentle manner lift the block straight up from the bottom and immediately place on a flat surface.	Wade or use a boat. DO NOT pull the block up from the shore.
Quickly place the Whirl-pak bags over all the HD's, and unscrew them from the block. If an organism is observed crawling off a HD, capture it and put it in the appropriate Whirl-pak. Fill the Whirl-paks with ambient water (so that all the plates are wet), secure them (twirl three times and twist the ends), and place on ice.	Whirl-paks should be pre-labeled with the station, sample date, and replicate number, using the permanent marker. Samples should NEVER be preserved until after organisms are scraped from the Hester-Dendy plates. Preservatives will poison the plates, preventing them from being used again.

DER Biology Section

v. 1—7/6/93

SOP #BA-14
Benthic Macroinvertebrate Hester-Dendy Sample Handling

(Modified from *Standard Methods 10500C*)

STEPS	COMMENTS
Materials	
1. Waterproof paper and permanent marker	For making labels
2. U.S. 30 mesh sieve	
3. Wrench for diamantling HD	
4. Ethanol filled squeeze bottle (80%)	Prepare per SOP #BA-8.1
5. Glass jars	
6. Dissecting microscope	
7. 100 x 15 mm petri dish	
8. Forceps	
9. Vials for picked organisms (1 or 2 dram)	
10. *Benthic Macroinvertebrate Bench Sheet*	
Methods	
1. Check labels so you know which sample you are dealing with (control site, test site, etc.). Make a very clear label to go into the bottle of picked bugs with station identification, date sampled, replicate number, and your initials.	It is critical that no errors be made during this step. If samples are mixed up, the entire study could be rendered invalid.
2. Pour the contents of the Whirl-pak bag (an assembled Hester-Dendy and associated detritus) over a U.S. 30 mesh sieve. Using a wrench to remove the bottom nut, dismantle the Hester-Dendy.	Rinse the Whirl-pak bag with tap water to make sure all organisms are put into the sieve.
3. Rinse with tap water (a small hose attached to the faucet works best). Scrape and simultaneously rinse organisms off HD plates with fingers (or a soft brush), using care not to damage the organisms. Wash fine debris (silt, mud) through the sieve. Any large debris (leaves, twigs) present should be brushed clean of organisms and discarded. Rinse the organism plus detritus mixture to one small area of the sieve.	Save the HD plates and hardware, and place them into the drying oven.
4. Using an ethanol-filled (80%) squeeze bottle, rinse the organism plus detritus matrix into the smallest practical container (usually a 100 mL to 250 mL glass jar). Put the label inside the jar.	Place jars on the sample shelf so that samples for a given study are organized together and clearly marked.

ER Biology Section

v. 1—7/6/93

STEPS	COMMENTS
Record the information requested on the *Macroinvertebrate Lab Bench Sheet*, which includes site, laboratory sample number, STORET station number, sample type, replicate number, and date collected. Include the initials of the persons who collected and sorted the sample.	
Take a small amount of detritus plus organism mixture and place it in the bottom half of a petri dish. Add enough liquid (usually ethanol) so that the material is wet enough to move around easily with forceps.	The top portion of the petri dish may later be placed over the sample to prevent desiccation if you must leave the sample overnight. Do not overload the petri dish with too much sample, as this results in sloppy work.
Place the petri dish under a dissecting scope set at low power (approximately 7x or 10x). In a deliberate, systematic manner, scan back and forth or up and down, picking each and every organism from the aliquot and placing it into an alcohol-filled vial (clearly marked as per step #1).	Using a pair of forceps in each hand enables you to better tease organisms out of fibrous detritus. Forceps should be sharp and properly aligned.
After scanning dish in one pattern (*e.g.*, up and down), go back through using a different pattern (back and forth) to assure that all organisms have been removed from the aliquot.	
Continue steps 6, 7, and 8 until the sample is finished.	Picking accuracy should be checked by a co-worker in 10% of the samples.

Macrobenthic Invertebrate Keys

Merrit, R.W. and K. W. Cummins, eds., 1984. An Introduction to the Aquatic Insects of North America. Second Edition. Kendall/Hunt Publishing Company, Dubuque, IA. 722 pp.

Heard, W. H. 1979. Identification Manual of the Freshwater Clams of Florida. State of Florida Department of Environmental Regulation, Technical Series Vol 4, No. 2. 83 pp.

Brigham, A.R., M.V. Brigham, and A. Gnilka. 1982. Aquatic Insects and Oligochaetes of North and South Carolina. Midwest Aquatic Enterprises, Mahomet, IL.

Berner, L. and M.L. Pescador. 1988. The Mayflies of Florida (Revised edition). Univ. of Fla. Press, Gainesville, FL. 267 pp.

Thompson, F.G. 1984. The Freshwater Snails of Florida, A Manual for Identification. Univ. of Fla. Press, Gainesville, FL. 94 pp.

Needham, J.G. and M.J. Westfall, Jr. 1955. A Manual of the Dragonflies of North America (Anisoptera). University of California Press, Berkeley. 615 pp.

Wiggins, G.B. 1977. Larvae of the North American Caddisfly Genera (Trichoptera). University of Toronto Press, Toronto. 401 pp.

Young, F.N. 1954. The Water Beetles of Florida. Univ. of Fla. Press, Gainesville. 238 pp.

Epler, J.H. 1992. Identification Manual for the Larval Chironomidae (Diptera) of Florida. Final Report for Florida Department of Environmental Regulation, Contract Number SP251.

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3150.2	TOTAL INORGANIC CARBON (TIC) DISSOLVED INORGANIC CARBON (DIC)	2.0	EPA 415.1	EFFECTIVE
PAGE 1 OF 6				DATE: 04/27/98

APPROVALS/DATE			
DIVISION DIRECTOR	LAB SUPERVISOR	QUALITY ASSURANCE	METHODS
5/11/98	5/11/98	5/11/98	5/11/98

SECTION 1.0 - TABLE OF CONTENTS

SECTION 2.0 - METHOD DESCRIPTION/HISTORY

2.1 Inorganic carbon species in natural waters include gaseous and dissolved CO_2, carbonic acid (H_2CO_3), bicarbonate (HCO_3^-) and carbonate (CO_3^{-2}), occurring in either free forms or as conjugate base (example $CaCO_3$, Na_2CO_3, $MgCO_3$, $NaHCO_3$, etc). The nature and abundance of individual species depends on the source of water and the underlying soil/sediment material.

2.2 The carbonate species determine the capacity of water to neutralize acid or base (alkalinity or acidity, respectively). Carbon dioxide is a participant in the biological processes of respiration (CO_2 produced), and biosynthesis of autotrophs or photosynthetic organisms (CO_2 consumed).

2.3 Inorganic carbon is determined directly using a high temperature combustion-infrared detection system. Analysis is done on a non-acidified sample, **unfiltered for TIC** and **filtered through a 0.45 um filter for DIC**. In the absence of a set holding time for inorganic carbon, the **holding time** for alkalinity **(14 days)** is followed.

2.4 Calibration is done using standards ranging from 0-200 mg/L C (in the form of Na_2CO_3) and the concentration of the samples are expressed as mg/L C.

2.5 This method (Version 2.0) was revised 27-APR-98 to reflect changes due to new instrumentation and changes in the LIMS programming. The method is the same in principle as Version 1.0.

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3150.2	TOTAL INORGANIC CARBON (TIC) DISSOLVED INORGANIC CARBON (DIC)	2.0	EPA 415.1	EFFECTIVE
PAGE 2 OF 6				DATE 04/27/98

APPROVALS/DATE			
DIVISION DIRECTOR 5/12/98	LAB SUPERVISOR 5/4/98	QUALITY ASSURANCE 5/4/98	METHODS 5/4/98

SECTION 3.0 - SAFETY PRACTICES (CONT'D)

3.1 Wear safety glasses and a full-length, long-sleeved laboratory coat.

3.2 Use acid resistant gloves when handling concentrated phosphoric acid.

3.3 All personnel conducting this method should be familiar with the SFWMD Chemical Hygiene Plan and should have reviewed Material Safety Data Sheet for phosphoric acid.

3.4 The electrical power should be disconnected before conducting any repairs inside the instrument on controllers, electrical wiring or any other components near sources of electricity.

3.5 In case of spills of concentrated H_3PO_4, treat first with an appropriate spill kit and collect the contaminated absorbent and place into adequate storage containers for disposal.

3.6 When changing compressed air bottles, take extra precaution in transporting the bottle to and from the room. It is advisable to request delivery by the vendor to the desired location. After closing the main tank valve, release the pressure slowly in the old tank until the pressure gauge displays 0 psi. Disconnect the regulator from the bottle and cap the bottle tightly with the provided cap. If transporting is required, carefully load the bottle on the hand truck bottle carrier and,secure tightly with the strap. Use the same carrier to transport a new compressed air bottle. Secure the new bottle with the strap and connect the regulator. Use teflon tape to avoid any leak. After the regulator is secured and the main tank valve opened, use the SNOOP™ to check for any leaks.

SECTION 4.0 - LIST OF EQUIPMENT/INSTRUMENTATION

4.1 Shimadzu 5050A TIC Analyzer with autosampler

4.2 Volumetric Flasks (Class A): 1000 mL, 100 mL capacity

4.3 Volumetric pipets (Class A): 20, 10, and 2 mL

4.4 Amber Bottle, 1L

4.5 Tensette pipet, 0.1-1.0 mL capacity

4.6 8 mL Disposable Test Tubes

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3150.2	TOTAL INORGANIC CARBON (TIC) DISSOLVED INORGANIC CARBON (DIC)	2.0	EPA 415.1	EFFECTIVE
PAGE 3 OF 6				DATE 04/27/98

APPROVALS/DATE			
DIVISION DIRECTOR 5/12/98	LAB SUPERVISOR 5/11/98	QUALITY ASSURANCE	METHODS 5/11/98

SECTION 5.0 - REAGENTS

5.1 Phosphoric acid, concentrated, reagent grade

5.2 Carbon-free water (distilled water, preferably double distilled)

5.3 Working Phosphoric acid solution (20%): Add 75 mL of concentrated phosphoric acid to 175 mL of DI water in the HDPE reagent bottle. Transfer to the solution to the acid reservoir (located in side of the analyzer).

5.4 Sodium Carbonate, anhydrous.

5.5 Zero (synthetic) air (<1 ppm CO_2 or hydrocarbons)

SECTION 6.0 - STANDARDS

6.1 Stock Inorganic Carbon (IC) standard solution (1000 mg C/L) – Dissolve 1.7660 g of Sodium Carbonate in ~70 mL of DI water contained in a 200 mL class A volumetric flask. Swirl to dissolve the salt, and dilute to the mark with DI water. **(Do not add sulfuric acid to this reagent).**

6.2 Standard 1 (Blank) - Deionized water

6.3 Standard 2 (20 mg C/L) - Dilute 2 mL of stock IC solution and dilute to 100 mL with DI water using a Class A volumetric flask.

6.4 Standard 3 (100 mg C/L) - Dilute 10 mL of stock IC solution and dilute to 100 mL with DI water using a Class A volumetric flask.

6.3 Standard 4 (200 mg C/L) - Dilute 20 mL of stock IC solution and dilute to 100 mL with DI water using a Class A volumetric flask.

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3150.2	TOTAL INORGANIC CARBON (TIC) DISSOLVED INORGANIC CARBON (DIC)	2.0	EPA 415.1	EFFECTIVE
PAGE 4 OF 6				DATE 04/27/98

APPROVALS/DATE			
DIVISION DIRECTOR 5/12/98	LAB SUPERVISOR 5/11/98	QUALITY ASSURANCE 5/11/98	METHODS 5/11/98

SECTION 7.0 - QUALITY CONTROL

7.1 A check standard (Standard 3) is run every 20 samples to verify the calibration. The recovery of this standard must be within ±5% of the true value (47.5 – 52.5 mg C/L).

7.2 QC1 and QC2 will be prepared by the QA unit. QC1 and QC2 are analyzed at beginning and end of each set of analyses. Results must be within current QA acceptance limits. In case of unacceptable recoveries, see your supervisor or QA unit staff.

7.3 A repeat sample, selected at random should be analyzed for every 20 samples analyzed. These results are recorded on the QC sheet. The mean and coefficient of variation of the replicate set is determined and noted on the QC sheet. If recoveries are not within the current QA acceptance limits, see your supervisor or QA unit staff.

7.4 A spiked sample must be analyzed for every 20 samples. Add 0.5 mL of stock IC solution, using a tensette pipette, into a 10 mL volumetric flask. Bring to volume with the sample being spiked and mix well. This results in an addition of 50 mg/L C.

7.5 All quality control data must be within the current established limits before entering sample data into the LIMS system. Complete and submit the QC result form.

SECTION 8.0 STEP-BY-STEP PROCEDURE

8.1 Daily Start-up

8.1.1 Check the gas supply. Ensure that there is enough gas for a day's operation. Change the air tank when pressure goes below 100 psi.

8.1.2 Check the acid reservoir level (located on the inside of the analyzer). Make sure that the reservoir at least ½ full before beginning the analyses.

8.1.3 Check the humidifier level (located on the inside of the analyzer). Make sure the water level is between the marks on the container, if not add DI water.

8.1.4 If the instrument power is not on, refer to section 10.3 and follow the procedure to power up the instrument.

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3150.2	TOTAL INORGANIC CARBON (TIC) DISSOLVED INORGANIC CARBON (DIC)	2.0	EPA 415.1	EFFECTIVE
PAGE 5 OF 6				DATE 04/27/98

APPROVALS/DATE			
DIVISION DIRECTOR	LAB SUPERVISOR	QUALITY ASSURANCE	METHODS

SECTION 8.0 STEP-BY-STEP PROCEDURE (CON'T)

8.2 Placing samples into the autosampler.

8.2.1 Create a workgroup for the samples to be analyzed (see section 9.1) and complete a sample ID worksheet. The worksheet is preformatted with the proper location of standards, check standards and QC samples. Enter the sample ID, and type for each sample in the workgroup. This worksheet will be used as guide to place the samples in the autosampler.

8.2.2 Remove the samples from the refrigerator, and using the worksheet as a guide, pour the standards, QC solutions and samples into disposable test tubes and place them into the autosampler in the location indicated on the worksheet.

8.3 Starting the analytical run.

8.3.1 From the main menu on the analyzer select "Autosampler", The screen will display the initial and final sampler positions to be analyzed. Make sure that the initial sample is set to 1 and the final sample position is set to the number of the last tube in the autosampler tray.

8.3.2 Press the "Next" key twice and Press "Start/Stop" on the analyzer keypad to begin the analyses. A tray of 74 samples will take ~14 hours to complete. Because of the susceptibility of the sample to exchange CO_2 with the atmosphere, do not pour more than 20 samples at a time.

8.4 Completion of the run

8.4.1 When the run is completed, remove the printed results from the analyzer and write the individual results on the sample ID worksheet. Check to make sure that all of the QA/QC samples are within limits.

8.4.2 Complete an instrument log form for the run and place it into the instrument logbook.

8.4.3 Enter the results to LIMS (see Section 9.3).

SECTION 9.0 DATA HANDLING

9.1 Follow the instructions given in the LIMS manual to create a workgroup for TIC or DIC.

METHOD #	METHOD NAME	REVISION	REFERENCE	STATUS
3150.2	TOTAL INORGANIC CARBON (TIC) DISSOLVED INORGANIC CARBON (DIC)	2.0	EPA 415.1	EFFECTIVE
PAGE 6 OF 6				DATE 04/27/98

APPROVALS/DATE			
DIVISION DIRECTOR 5/12/98	LAB SUPERVISOR 5/11/98	QUALITY ASSURANCE	METHODS 5/11/98

SECTION 9.0 DATA HANDLING (CONT'D)

9.3 Log into the LIMS, and at ISOTOPE%, type **lims**. At the mainmenu select "**Workstat**", "**Dataentry**", and "**Manual**". Enter **TIC** or **DIC** under parameter, then enter the Workgroup #. Type the each sample concentration corresponding to the sample number from the sample ID worksheet and hit **ENTER**, after each entry. Press **ENTER** again after confirming that the correct result has been entered.

SECTION 10.0 - INSTRUMENT CLEANUP/SHUTDOWN/TROUBLESHOOTING

10.1 **Leave the furnace, gas and the instrument on**, unless the unit will not be used for a prolonged period.

10.2 Remove used test tubes, empty the contents on the sink, and dispose of glass test tubes in designated glass disposal receptacle. Wipe up any spill around the work area and on the instrument.

10.3 Instrument Startup

10.3.1 If the instrument has been powered off, make sure that the gas flow is turned on, and switch the power on by pressing the switch on the left side of the analyzer. It will take a few seconds for the initialization screen to appear.

10.3.2 When the initialization screen appears, press the "Initialize Autosampler" key to initialize the autosampler. This will take about one minute.

10.3.3 After the autosampler has initialized, the furnace will begin to heat. Again, check the gas flow and make sure it is set to 150 ml/min; adjust, if necessary, by turning the carrier gas knob inside the analyzer front panel. Allow the instrument to stabilize for 2-3 hours before beginning any analyses.

SECTION 11.0 - REFERENCES

11.1 U. S. Environmental Protection Agency. March 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020.

11.2 American Public Health Assoc., 1989. Standard Method for the Examination of Water and Wastewater, 17th Edition.

EVERGLADES SYSTEMS RESEARCH DIVISION STANDARD OPERATING PROCEDURES	Version: 3.0
	Revision: 1.0
Everglades Nutrient Removal Project - SOP #2	Date: 09/28/94
ENR Sediment/Porewater Monitoring Plan	FileName: ENRSED01.SOP

1.0 INTRODUCTION

1.0 PURPOSE AND SCOPE

The Everglades Nutrient Removal (ENR) Project (Figure 1) is a demonstration-scale constructed wetland (1500 ha) designed to reduce total phosphorus (TP) loads in surface water entering the Loxahatchee National Wildlife Refuge. The long-term TP removal mechanism is generally regarded to be through peat accumulation which will be documented using clay markers and sediment erosion tables. The immobilization of phosphorus within the sediments will be monitored by measuring the relative concentrations of different P forms over time. The field sampling program designed to collect these later data, the ENR Sediment/Porewater Monitoring Plan (SPMP), is described in this Standard Operating Procedure.

1.2 SAMPLING LOCATIONS

A total of 36 sediment/porewater sampling sites will be distributed throughout the ENR Project (Figure 1). Sites will be oriented along the water quality gradient that is anticipated to develop when the wetland is in operation as follows:

1. *Cell 1* - Based on its physical layout and close proximity to the inflow pump station, this Cell will be used to evaluate the peat accumulation and build-up of P in the sediments as a function of distance from the inflow. Core samples will be collected along two transects located 250, 500, 1000, 2000 and 4000 m downstream of the inflow culverts (i.e., a total of 10 sites). At each site, three cores will be collected. Each core will be cut into 0-5, 5-10, and 10-30 cm depth sections. Core sections from the same depth interval at each site will be composited for chemical analyses;

2. *Cells 2 and 4* - The complex shape of Cell 2 and the small size and open-water environment of Cell 4 do not lend themselves to the sampling design specified for Cell 1. Instead, two sites near the inflow and two sites near the outflow will be established for Cells 2 and 4 (i.e., a total of four sites). At each site three cores will be collected. Each core will be cut into 0-5, 5-10, and 10-30 cm depth sections. Core sections from the same depth interval at each site will be composited for chemical analyses;

3. *Cell 3* - The complex vegetation community established in Cell 3 allows us the opportunity to examine sediment biogeochemistry as affected by different macrophyte species. Three replicate cores samples will be collected at both the inflow and outflow from within the planting blocks for three species of vegetation (i.e., a

total of 18 sites). These samples will be sectioned at the same depth intervals as described for the other cells (i.e., 0-5, 5-10 and 10-30 cm), however, they will not be composited for chemical analyses. This will permit us to perform statistical comparisons of P retention and other sediment parameters among the different vegetation types.

1.3 SAMPLING FREQUENCY

The ENR Sediment/Porewater Monitoring Plan consists of three distinct sampling programs: porewater, P fractionation, and nutrient depth profiles. Sample collection for these programs will be conducted quarterly, annually, and semi-annually, respectively. Samples for P fractionation and nutrient depth profile analyses will be collected as detailed in Section 3.1 - Sediment Core Collection; porewater will be collected as described in Section 3.2 - Porewater Collection.

2.0 FIELD AND LABORATORY EQUIPMENT

2.1 ITEM CHECKLIST

2.1.1 Sediment Cores

PVC coring tube (3 in) with spares
PVC coring attachment
rubber stoppers
hammer
pipe pounder
wooden pounding block
knife
sample labels and rubber bands
compass
metric measuring tape
clipboard
pencils/pens
water-proof marking pens
deionized water
SPMP chain-of-custody and field data sheets
field notebook
ENR Sediment/Porewater Monitoring Plan SOP

2.1.2 Sediment Equilibrators

flushed peepers in storage cases
syringes
60 mL plastic bottles
labels and rubber bands
latex disposable gloves
deionized water
filter holders
0.45µm polycarbonate filters
1.5µm glass fiber filters
pounding block
mallet
light-tight ice chests
ice
water-proof marking pens
pencils/pens
clipboard
chemical preservatives (H_2SO_4 and HNO_3)
compact pH meter
pH meter calibration buffers (pH 4 and 7)
pH strips
SPMP chain-of-custody and field data sheets

field notebook
ENR Sediment/Porewater Monitoring Plan SOP

2.1.3 Porewater Wells

Erlenmeyer flasks
manual hand pump and spares
sample labels and rubber bands
syringes
60 mL bottles
0.45µm polycarbonate filters
1.5µm glass fiber filters
light-tight ice chests
ice
latex disposable gloves
preservation chemicals (H_2SO_4, HCl, HNO_3)
deionized water
spare parts for wells
clip board
pencils/pens
water-proof marking pens
SPMP chain-of-custody and field data sheets
field notebook
ENR Sediment/Porewater Monitoring Plan SOP

2.2 EQUIPMENT LOCATION

1. All field and laboratory equipment is stored in the ESRD equipment lockers located in the Division's offices in B-50 or in the B-113 storage area. Staff should be familiar with the location of all equipment and supplies required for performing this procedure.

2. Deionized water, labels, bottles, and SPMP chain-of-custody forms are obtained from WRED.

2.3 SPECIAL INSTRUCTIONS

1. WRED should be notified at least one week prior to initiating field sampling for sediments.

2. Sample bottles may be obtained from the WRED prep trailer. Preservatives are prepared by the WRED Laboratory QA unit.

3. ESRD equipment must be checked out in advance in the equipment log book. All equipment should be tested prior to usage. Report equipment damage or malfunction to the ESRD equipment coordinator.

2.4 EQUIPMENT CALIBRATION

1. Two pH buffers which bracket the analytical range of interest (e.g., 4 and 7) should be used to calibrate the pH meter following the manufacturer's instructions.

2. Meter calibration must be rechecked every four hours while in the field and at the end of the day before leaving the last sampling site.

3. All field calibration records must be kept in the field notebook.

3.0 FIELD AND LABORATORY PROCEDURES

3.1 SEDIMENT CORE COLLECTION

1. Intact sediment cores will be obtained by driving a PVC coring tube (Figure 2) to a depth of approximately 50 cm into the peat. Prior to driving the core tube into the sediment, a knife should be used to cut through any surface roots, plant material, or other detritus at the bottom of the tube. This will help minimize compaction of the core during sampling.

2. Under shallow conditions (i.e., water depth < 30 cm) this will be achieved using a block of wood to protect the neck of the core tube and then striking the block several times with a hammer such that the core penetrates the soil with minimal compaction.

3. If the water depth is greater than 30 cm, a PVC coupling should be attached to the coring device (Figure 2). This attachment is comprised of a ball valve and a length of PVC pipe. This is fitted to the neck of the PVC coring tube and a closed ended piece of metal pipe with a diameter larger than the upright PVC pipe is used to pound the core into the sediment. During the insertion, the ball valve should be open. Prior to pulling the core out of the sediment, the ball valve should be closed.

4. The sediment core, with the overlying water, will then be labeled, capped at both ends using rubber stoppers, stored out of direct sunlight, and transported to the laboratory for analysis within 12 hrs. Each sample label will contain the following information:
   a. Station designation,
   b. Sample replicate information if appropriate
   c. Date,
   d. Time, and
   e. Initials of field personnel collecting sample.

5. If the sediment surface within the core is at a significantly different depth than the adjacent soil this suggests that compaction has occurred. The core should then be discarded and another one collected.

6. Triplicate cores will be collected at six randomly selected sites in Cell 3 to determine within-site variability for three different species of vegetation.

7. Field personnel will complete all appropriate sections of the SPMP sample chain-of-custody form (see Appendix) immediately after collecting a sediment core sample at each station.

8. Both crew members will verify that all necessary samples have been collected and the information recorded on the sample label and chain-of-custody form is correct before leaving a station.

9. All samples should be delivered to WRED as soon as possible for shipment to the contract laboratory.

3.2 POREWATER COLLECTION

Porewater will typically be collected using porewater wells which will have screens installed to isolate one of the following depths: 0-5 cm, 5-10 cm, or 10-30 cm. Three wells, one for each depth increment, will be required at each site. The choice of this collection technique was based on relatively volume of sample needed for chemical analysis. Two other sample collection procedures, i.e., spinning down sectioned sediment cores and sediment equilibrators (for looking at fine chemical profiles within the soil) may possibly be used in conjunction with porewater wells to address specific research questions.

3.2.1 Centrifuge Extraction of Porewater

Intact cores will be cut into sections that correspond to 0-5 cm, 5-10 cm, or 10-30 cm depth increments and the porewater extracted from the solid material by spinning down each section in a centrifuge at 5000 rpm for 20 minutes.

3.2.2 Porewater Wells

1. Each porewater well is composed of a PVC pipe with a well screen (Figure 3). The size and position of the screen on each well will depend on the desired sampling depth (e.g., 0-5 cm, 5-10 cm, or 10-30 cm depth increments). The end of the well that enters the soil is capped with a pointed PVC fitting while the sampling end is closed off using a rubber stopper. A clear acrylic (or similar inert plastic) tube extends through the rubber stopper to the base of the well. The acrylic tube is closed to the atmosphere by the attachment of a small length of impermeable hose which is closed with a pinch-clamp. This hose is also used to attach the well to a manual vacuum pump.

2. Prior to sampling, each well is completely evacuated using a manual vacuum pump. This water is initially collected in the Erlenmeyer flask and then discarded. The well is then allowed to recharge for approximately 15 minutes (note: absolute time is dependent on volume of sample needed).

3. Another small portion of water (ca. 10 to 20 mL) is subsequently withdrawn from the well and used to rinse out the Erlenmeyer flask. This aliquot is then discarded. The final sample is collected into the flask and transferred to a plastic 1 L bottle. If the Erlenmeyer flasks are reused in the field they must be rinsed thoroughly with deionized water between sites. Samples should be filtered immediately after collection.

4. Each sample is processed immediately (i.e., filtered and preserved, if necessary), labeled, and stored on ice in a light-tight cooler. The sample preparation, filtering, and handling protocols used for this study will follow procedures listed in the SFWMD's Laboratory Comprehensive Quality Assurance Plan (Section ___).

Samples will be transported back to the laboratory within 12 hrs from the time of field collection. Each sample label will contain the following information:

a. Station designation,
b. Sample replicate information if appropriate
c. Date,
d. Time, and
e. Initials of field personnel collecting sample.

5. Field personnel will complete all appropriate sections of the SPMP chain-of-custody and field data form (see Appendix) immediately after collecting a porewater well sample at each station.

6. **Both crew members will verify that all necessary samples have been collected and the information recorded on the sample label and chain-of-custody form is correct before leaving a station.**

7. All samples should be delivered to WRED as soon as possible for shipment to the contract laboratory.

3.2.3 Sediment Equilibrators

A sediment equilibrator (or "peeper") is a device used to collect porewater from small depth increments within the sediment. Each equilibrator is composed of a base of Plexiglas, 77 cm long x 10 cm wide x 2 cm thick, with a number of "cells" (7 cm x 1 cm x 1.5 cm) milled into it (Figure 4). A 0.40 µm membrane filter is placed over each of the cells, which have been filled with deionized water. A coarse nylon mesh is used protect the membrane. A slotted cover sheet of Plexiglas is screwed to the base to secure the nylon mesh in place and seal the membrane over the cells. Each peeper is then placed in a Plexiglas storage container containing deionized water which is bubbled with Nitrogen gas to maintain an oxygen-free environment. The peepers are transported to the field in the storage container. At the sampling site, the peepers are inserted into the soil and left to equilibrate for a period of two weeks. After two weeks, the peepers are removed from the soil and a water quality sample is extracted from the cells using syringes. To minimize air intrusion into the cells, samples are collected immediately after the Peeper is removed from the soil. Those cells that were positioned within the most reduced soil conditions, i.e., the deeper cells, are sampled first. To calculate fluxes of materials between the water and sediments it is essential that some of the cells which extend into the overlying water are also sampled.

3.2.3.1 Sediment Equilibrator Preparation

1. Peepers are cleaned with Liquinox soap and rinsed with deionized water. The peepers should be kept in a horizontal position when preparing. **Note:** All preparation is done while wearing gloves to prevent contamination of the peeper.

2. Peeper cells are filled completely with deionized water.

3. 8 x 10 cm sheets of Nuclepore 0.4 µm polycarbonate filters (Costar-Nuclepore, catalog #113607) are cut lengthwise and three of these prepared sheets are laid over the deionized water-filled cells. Large air bubbles are removed by gently lifting the edges of the membrane and then replacing it on the water's surface. Each piece of cut membrane should overlap two cells.

   Note: The filter material is cut, three sheets at a time, using a blade paper cutter that has been thoroughly cleaned with Liquinox and rinsed in deionized water; keep the blue separating paper on the membrane when cutting.

4. Sheets of nylon "no-seeum" netting, cut to the dimensions of the peeper, are placed over the membranes covering the cells. Care is taken not to separate the overlapped membrane from the Plexiglas base in the process. The netting is cut using a blade paper cutter and scissors.

5. Areas where the membrane and netting cover the screw holes are punched through using a syringe needle.

6. The slatted top of the peeper is then carefully put into position on top of the base section. Screws are tightened using a power screwdriver to join the two sections together.

   Note: All peepers are numbered; the same numbered bottom section has to be used with the corresponding slatted top section because of the variability between peepers.

7. Examine the peepers after they have been assembled. If there has been a separation of the membrane from the Plexiglas base, if the nylon netting has ripped, or if large air bubbles exists in a cell, the peeper must be re-assembled.

8. Peepers that pass inspection are then placed in a Plexiglas storage container containing deionized water. A plastic tube slid through a rubber stopper is inserted into a hole in the container's lid. The top of the tube is attached to a tank containing Nitrogen gas. Prior to installation in the field, nitrogen gas is bubbled through the storage container to purge the deionized water of all oxygen. Containers should be purged for at least three hours and preferably overnight.

9. The peepers must remain in these oxygen deficient storage containers until immediately before insertion into the soil at the sampling site.

3.2.3.2 Sediment Equilibrator Deployment and Incubation

1. Transport the peeper storage containers to the field site. At each sample location a peeper is removed from the its container and gently driven into the sediment using a block of wood and a mallet. Peepers should be deployed such that at least four

cells are above the sediment-water interface. Peeper sites should be conspicuously marked with a flag to alert boat traffic to their presence .

2. Peepers are incubated in the field for a period of two weeks.

3.2.3.3 Equilibrator Retrieval and Sampling

1. Before retrieving a peeper, mark the soil/water interface level on the side of the peeper with a wax pencil.

2. Remove the peeper from the sediment and carry it in a horizontal position back to the boat for processing.

3. Using a wax pencil, mark off 2 cm intervals or "zones" along the length of each peeper. The zones start at the soil/water interface and extend 4 cm above the interface line and 10 cm below the interface line. Each sample will be a composite of the water collected from the two cells within a zone. Sample only those cells that have an intact membrane.

   Note: The sample label records the relative vertical position of the cells during peeper incubation, i.e, zones above the soil/water interface are designated as positive while zones below the interface are negative. For example, the 0 to 2 cm interval above the soil/water interface is labeled 0+2, the 2 to 4 cm interval above the interface is labeled +2+4, the 0 to 2 cm interval below the interface is labeled 0 -2, the 2 to 4 cm interval below the interface is labeled -2 -4, and so on.

4. Process cells in zones below the soil/water interface first. To collect a water sample pierce the membrane of a cell with the tip of a syringe needle. The needle will release any pressure that has built up inside the cell.

5. Slowly draw the water out of the cell with the syringe. Care must be taken from this point onward not to contaminate the sample with atmospheric oxygen.

6. Place the tip of the filled syringe on the bottom of a 60 mL plastic bottle. Slowly discharge the contents of the syringe into the bottle, keeping the needle tip submerged below the water at all times.

7. If pH is measured in the field, follow step 7a below. If pH is not measured in the field follow step 7b.

   a. Withdraw a small subsample of water (ca. __ mL) from each cell in a zone and measure its pH using a Horiba compact pH meter. Record the pH on the field data sheet (see Appendix)

   b. Withdraw a 2 mL subsample from the 60 mL bottle with a syringe and place the

tip of the syringe into a rubber stopper. This prevents degassing of the sample and thus ensures an accurate pH measurement back at the laboratory. Store these syringes on ice in a light-tight cooler and transport back to the laboratory for pH analysis within 12 hrs. Each syringe should be labelled as described in step 8 below. If the same syringe is used to collect more than one sample, the syringe must be (1) rinsed thoroughly with deionized water then (2) rinsed three times (volume permitting) with the next sample.

8. Approximately one half of the water remaining in the bottle is split and placed into another 60 mL plastic bottle. One sample bottle is given an orange label and is not acidified. The other sample bottle is given a gray tag and is acidified to a pH < 2 with diluted H_2SO_4

   Note: the acid is diluted to 50% strength using deionized water; acid is prepared by the WRED Laboratory QA unit and is not diluted in the field.

9. Label each sample with the following information:
   a. Station designation,
   b. Sample replicate information if appropriate
   c. Date,
   d. Time,
   e. Incubation zone, and
   f. Initials of field personnel collecting sample.

10. Samples are immediately placed on ice in a light-tight cooler and transported back to the laboratory within 12 hrs.

11. Field personnel will complete all appropriate sections of the WRED sample chain-of-custody form (see Appendix) immediately after collecting a peeper sample at each station.

12. **Both crew members will verify that all necessary samples have been collected and the information recorded on the sample label and chain-of-custody form is correct before leaving a station.**

13. All samples should be delivered to WRED as soon as possible for shipment to the contract laboratory.

3.2.3.4 Sediment Equilibrator Field Data Sheet

1. The station name is the name of the sample site designation combined with the peeper # at the site. For example, if three peepers are at site ENR401, they are referred to as ENR401-1, ENR401-2, ENR401-3.

2. On the field data sheet, it is critical to correctly record the depth of the sample, the

interval range in the comment section, and type of sample.

a. The depth of the sample is recorded as the highest number of the range of the interval. For example, the depth of interval 0 to 2 cm above the soil/water interface is 2, the depth of interval 2 to 4 cm above the interface is 4, the depth of the interval 0 to 2 cm below the interface is -2, the depth of the interval 2 to 4 cm below the interface is -4, and so forth.

b. The sample type code is especially important because it designates the sample as being surface water (type = 0) or interstitial water (type = 10). Surface water are the intervals above the soil/water interface; interstitial water are the intervals below the interface.

3. Refer to Section 7.1 and 7.2 of the SFWMD Laboratory Comprehensive Quality Assurance Plan for general field data sheet documentation (in addition to what is stated in this SOP) and field chain-of-custody protocols.

4.0 SAMPLE SUBMISSION PROCEDURES

Sediment and porewater samples will be collected by ESRD personnel and subsequently delivered to the contract laboratory for analysis. The sample transfer mechanism will be coordinated with WRED and the contract laboratory. The chain-of-custody sheets will accompany the samples at all times. These sheets will document the transfer of samples between departments within the District and between the District and the contract laboratory.

5.0 HEALTH AND SAFETY

ESRD personnel will observe all standard District health safety policies that pertain to conducting field studies while collecting and analyzing ENR sediment and porewater samples. These policies are detailed in the ENR Safety Manual. Copies of this manual are available from the Project Manager and are stored in the ESRD offices located in B-50. Health and safety items of particular importance for this project include the following:

1. The maximum speed limit is 35 mph on paved roads and 30 mph on unpaved roads and levees. The narrow roadways on the ENR levees would dictate that maximum speeds be kept under 30 mph, especially when towing a boat.

2. Field work shall generally be conducted by crews consisting of at least two people. The only exception to this rule will be road surveys (e.g., reading staff gauges, bird censuses, etc.) which are conducted from a vehicle.

3. No field work will be performed during a thunderstorm or impending lightning. If threatening weather develops, seek shelter in a building or vehicle.

4. Personnel will wear a life jacket at all times while working from or riding in a boat and wading in the marsh.

5. Personnel shall have access to a cellular phone, radio, or other forms of emergency communication at all times while working in the field.

6. Avoid contact with wildlife, especially snakes and alligators.

7. The District's drug-free and smoke-free workplace regulations in buildings and vehicles will be adhered to at all times. Do not smoke in the vicinity of boat gas tanks.

8. All personnel must have received Hazard Communication Training and have read and be familiar with Material Safety Data Sheets for H_2SO_4, HNO_3, HCl, pH buffer solutions, and pH electrolyte filling solution.

6.0 QUALITY ASSURANCE/QUALITY CONTROL

The Project Manager will issue all field and laboratory personnel a copy of the SPMP Standard Operating Procedures and will verify that each staff member has read the SOP. A Quality Control program will be established for the analyses of sediment and porewater. Results of the QC analyses will be reported along with the regular data by the contract laboratory to the Project Manager. Any discrepancies in field or laboratory procedures will be documented by completing a deviation report (see Appendix). Competed deviation reports will be submitted to the Project Manager for review and appropriate action. Sample chain-of-custody forms (see Appendix) will document the transfer of samples between departments within the District and between the District and the contract laboratory.

The Quality Control Program will consist of the following additional samples collected and analyzed for each sampling trip.

6.1 SEDIMENT CORE COLLECTION

1. Triplicate cores collected at six randomly selected sites and three vegetation types in Cell 3.

2. One known spike prepared and submitted to the contract laboratory by the WRED QA laboratory unit.

6.2 POREWATER COLLECTION

6.2.1 Porewater Wells

1. Equipment blank (EB) samples are generated by passing 1 L of deionized water through all field sampling equipment. One EB will be collected prior to going into the field and additional EBs collected (a) after every 20 field samples and (b) at the end of sampling if the total number of field samples is not an even multiple of 20, e.g., if 19 samples are collected then two EBs are required; if 22 samples are collected then three EBs are required.

2. Split samples (SS) are generated by dividing a single field sample into two separate samples. One SS will be collected (a) after every 20 field samples and (b) at the end of sampling if the total number of field samples is not an even multiple of 20, e.g., if 19 samples are collected then one SS is required; if 22 samples are collected then two SSs are required.

3. Field duplicate (FD) samples are generated by collecting an additional sample immediately after the routine field sample using the same procedures. One FD will be collected (a) after every 10 field samples and (b) at the end of sampling if the total number of field samples is not an even multiple of 10, e.g., if 9 samples are collected then one FD is required; if 12 samples are collected then two FDs are required.

2. Field blank (FB) samples are generated by filling a sample bottle with deionized water and allowing that bottle to remain open while collecting the routine field samples. One FB will be collected (a) after every 20 field samples and (b) at the end of sampling if the total number of field samples is not an even multiple of 20, e.g., if 19 samples are collected then one FB is required; if 22 samples are collected then two FBs are required.

6.2.2 Sediment Equilibrators

1. One EB will be collected prior to going into the field and additional EBs collected (a) after every 20 field samples and (b) at the end of sampling if the total number of field samples is not an even multiple of 20.

2. One FD will be collected (a) after every 10 field samples and (b) at the end of sampling if the total number of field samples is not an even multiple of 10.

3. One FB will be collected (a) after every 20 field samples and (b) at the end of sampling if the total number of field samples is not an even multiple of 20.

Table 1. Chemical constituents to be measured in ENR sediment and porewater samples.

POREWATER

alkalinity
alkaline phosphatase activity
chloride
conductivity
dissolved organic carbon
total inorganic carbon
total organic carbon
total dissolved phosphorus
ortho-phosphorus
Al - total
Ca - dissolved and total
Fe - dissolved and total
K - dissolved and total
Mg - dissolved and total
Mn - dissolved and total
Na - dissolved and total
NH_4
NO_2
NO_2+NO_3
P - hydrolyzable
pH
silica
sulfate
sulfide
total dissolved Kjeldahl nitrogen

SEDIMENT NUTRIENT PROFILES

%ash
%moisture
Al - exchangeable and total
alkalinity
bulk density
Ca - exchangeable and total
cation exchange capacity
chloride

Table 1. (continued) Chemical constituents to be measured in ENR sediment and porewater samples.

SEDIMENT NUTRIENT PROFILES (cont.)
conductivity
Fe - exchangeable and total
K - exchangeable and total
Mg - exchangeable and total
Mn - exchangeable and total
Na - exchangeable and total
pH
silica
sulphur
total carbon
total organic carbon
total nitrogen
total phosphorus
P FRACTIONATION

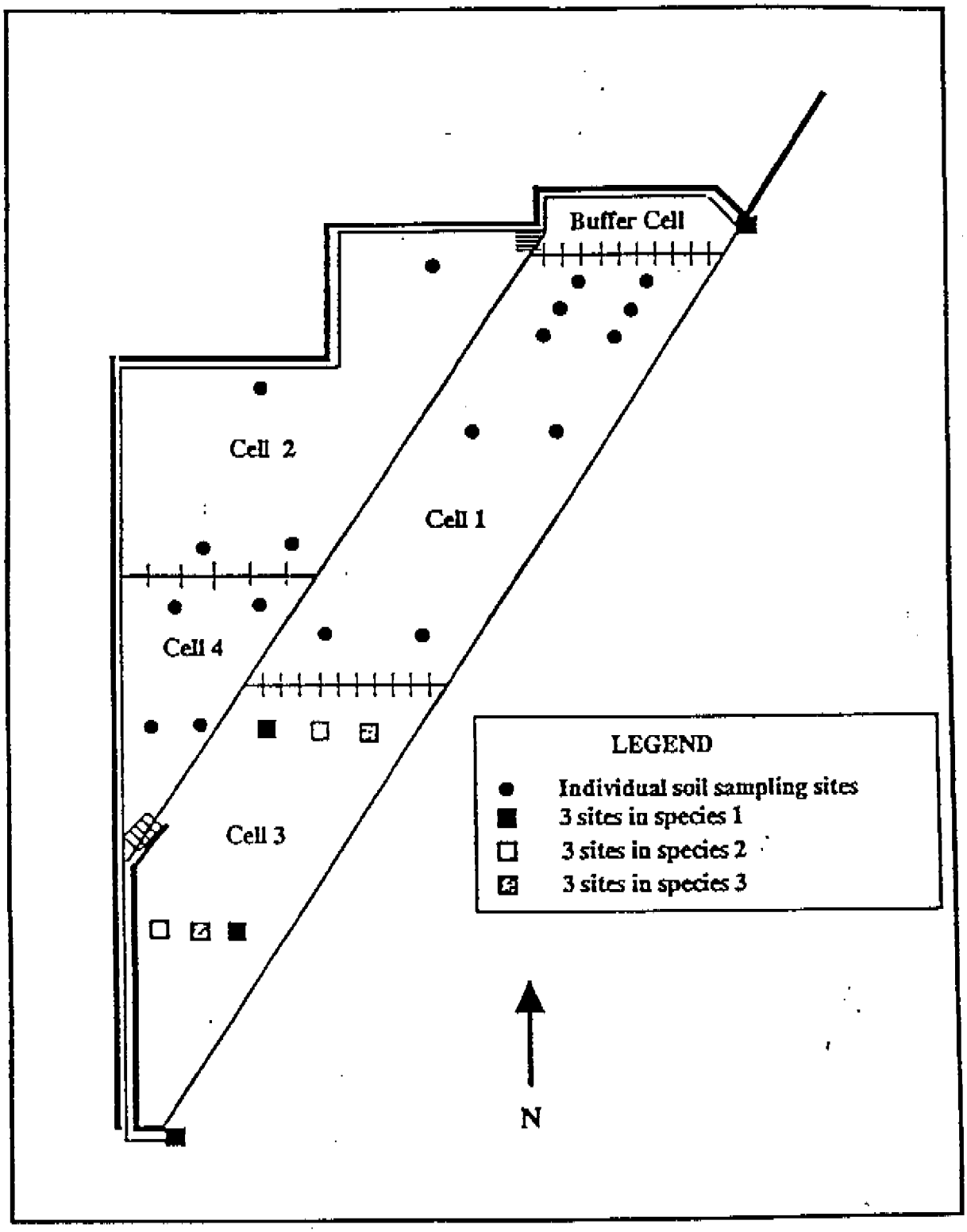


Figure 1. ENR site map showing locations of sampling stations.

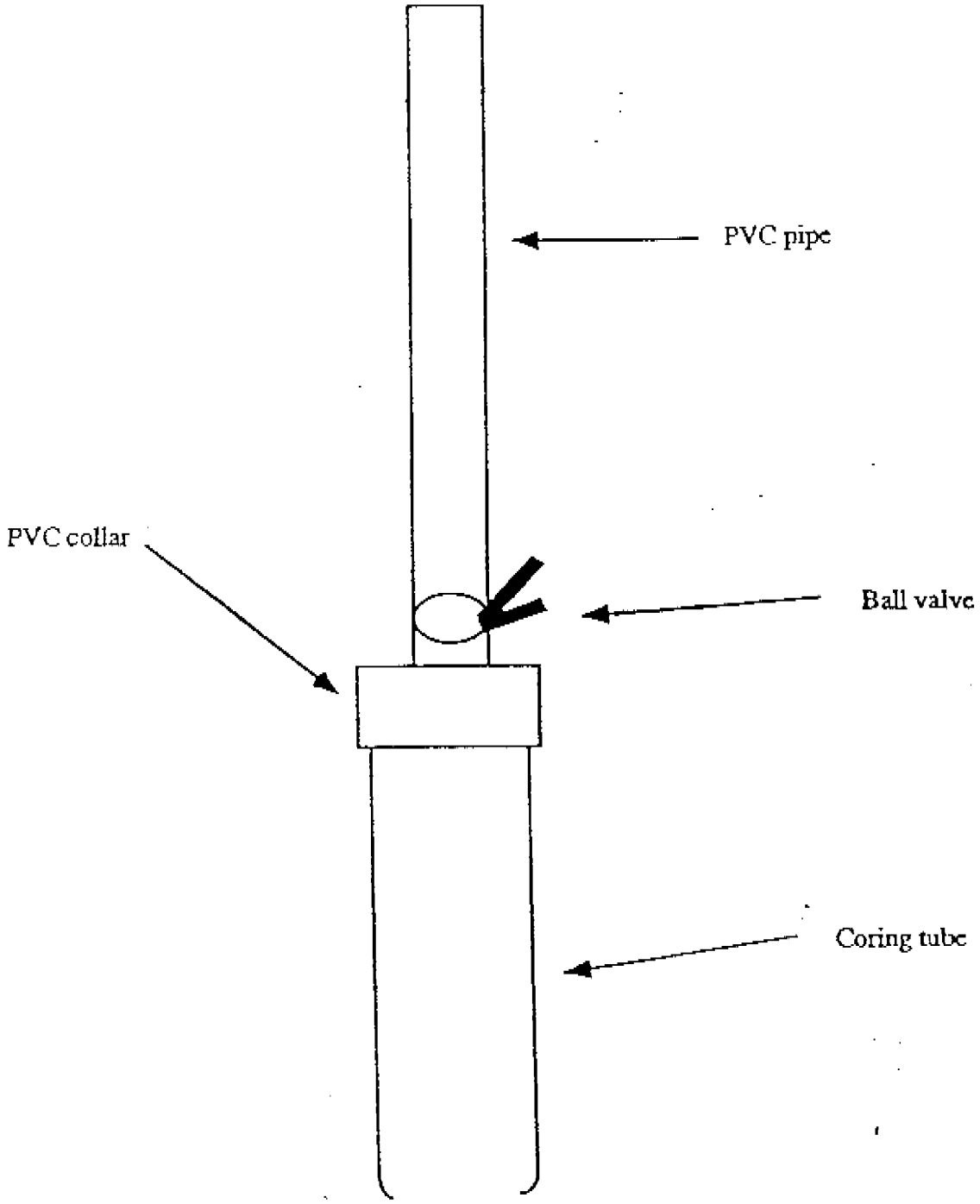


Figure 2. Schematic of PVC sediment core device used to collect core samples in the ENR.

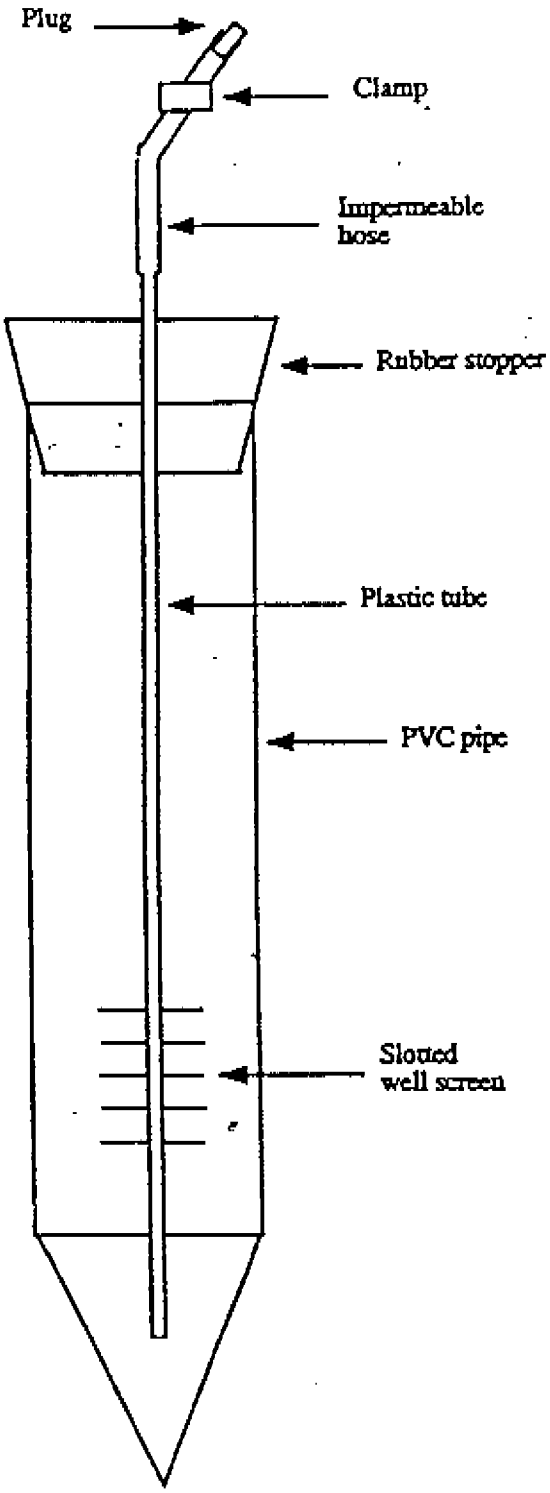


Figure 3. Schematic of PVC well used to collect porewater samples in the ENR.

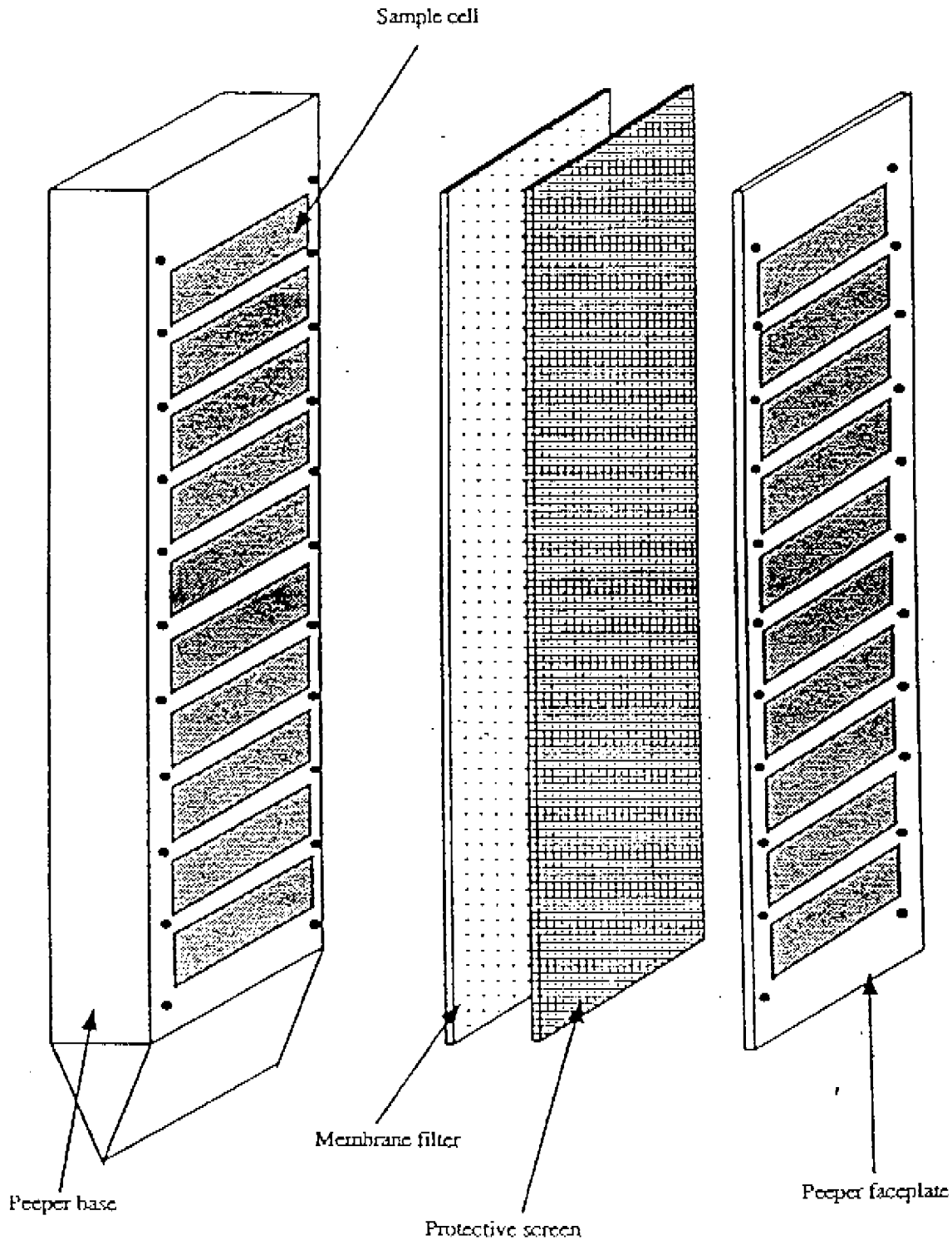


Figure 4. Schematic of plexiglas Sediment Equilibrator used to collect porewater samples in the ENR.

STANDARD OPERATING PROCEDURE DISTRIBUTION LIST

PERSONNEL	DATE
Scott Wiseman (10-14-94)	
Paul McCormick (10-14-94)	
Tim Bramlett (10-14-94)	
Zaki Mostafa (10-21-94)	
Shili Miao (10-14-94)	
Fred Stolar (10-14-94)	
Kathy Pistro (10-14-94)	
Chad Kennedy (10-14-94)	
Jim Laing (10-14-94)	
Robert Stafford (10-14-94)	
Pete Peeslik (10-14-94)	
Tim Beckstel (10-14-94)	
Larry Fink (10-14-94)	
Maria Lencaft-Manzano (10-14-94)	
Tom Kosky (10-14-94)	

APPENDICES

EVERGLADES SYSTEMS RESEARCH DIVISION
PROJECT DEVIATION REPORT

Project Name: _____

Sampling Program: _____

Report Submitted By: _____ Date: _____

Description of Deviation: _____

_____

_____

_____

_____

Probable Impact of Deviation on Data: _____

_____

_____

_____

Corrective Action to Prevent Deviation From Reoccurring in the Future: _____

_____

_____

_____

_____

_____

Project Scientist: _____ Date: _____

Project Manager: _____ Date: _____

CHAIN CUSTODY

Page ___ of ___

DB Environmental Laboratories, Inc.
414 Richard Rd., Suite 1
Rockledge, FL 32955
(407) 639-4396

Client: (Company & Individual) | Address (City, State, Zip) | Phone No. | Fax No. Contact Person/Phone No.

Original Report To: (Company & Individual) | Address (City, State, Zip) | Phone No. | Fax No. (Optional) Additional Reports Sent To:

Invoice To: (Company & Individual) | Invoice No.

Project Name/Number | Collected By: (Signature)

	Sample ID	Date/Time	Field No.	Lab No.	Water Matrix (checked): City	Ground	Waste	Surface	Soil	Other	Street Address, Specific Location, Sequence No.	Chlorine Concentration (mg/L)	Requested Analysis: PRES.										REMARKS
1																							
2																							
3																							
4																							
5																							
6																							
7																							
8																							
9																							
10																							
11																							
12																							
13																							
14																							
15																							
16																							

CUSTODY TRANSFER:

	Relinquished by:	Received by:	Date/Time:
1.			
2.			
3.			

Delivered Directly to Lab ☐ Shipped ☐

Method of Shipment: FedEx ☐ Ups ☐ Other_____

Received in cooler: yes ☐ no ☐ Temp °C_____

pH checked in lab: yes ☐ no ☐

Lab Recipient_____ Date_____ Time_____

LABORATORY REMARKS: