California Regional Water Quality Control Board
Colorado River Basin Region

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Secretary for Environmental Protection

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Arnold Schwarzenegger
Governor

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August 31, 2004

Amy King/Julia Saylor
Tetra Tech Inc.
1230 Columbia Street, Suite 520
San Diego, CA 92101

RE: QAPPs for Region 7

Hello Amy and Julia,

Enclosed you will find copies of the requested QAPPs for the Colorado River Basin Regional Water Quality Control Board. I apologize for the delay. Please contact me at (760) 776-8920 if you need more information.

Thank you for everything!

Sincerely,

Sheila Ault
Environmental Scientist
Colorado River Basin Regional Water Quality Control Board

Enclosure

Doug Wylie's... copy

QUALITY ASSURANCE PROJECT PLAN

FOR

SALTON SEA NUTRIENT WATER QUALITY MONITORING

Prepared by and for
California Regional Water Quality Control Board Staff
Colorado River Basin Region

APRIL 2002

Quality Assurance Project Plan
Salton Sea Nutrient Water Quality Monitoring

APPROVALS:

California Regional Water Quality Control Board

Jose Angel, P.E., Supervising WRC Engineer
Watershed Protection Division Chief

4/25/02
Date

Doug Wylie

Doug Wylie, P.E., Senior WRC Engineer
Project Manager

4-25-02
Date

FOR MCO

Maria de la Paz Carpio-Obeso, Ph.D.
Environmental Scientist
Quality Assurance Officer

4/25/02
Date

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1 PROJECT MANAGEMENT

1.1 INTRODUCTION

The Porter-Cologne Water Quality Control Act (Porter-Cologne) is the principal law governing water quality regulation in California. This statute established the State Water Resources Control Board (SWRCB) and nine Regional Water Quality Control Boards (RWQCBs), which are charged with implementing its provisions. Porter-Cologne establishes a comprehensive program for the protection of water quality and the beneficial uses of water.

Staff from the California Regional Water Quality Control Board, Colorado River Basin Region (Regional Board), will determine whether discharges in the vicinity of the Salton Sea are adversely impacting water quality of the lake. The Regional Board is the lead agency on this project. Specifically, this investigation focuses on Salton Sea surface water quality from the outflow of the Coachella Valley Water District and the Imperial Irrigation District drain systems, and the Alamo and New Rivers. This Quality Assurance Project Plan (QAPP) is subject to approval by Regional Board staff.

This QAPP follows the format that the U.S. Environmental Protection Agency (USEPA) established in its *Requirements for Quality Assurance Project Plans for Environmental Data Operations, EPA QA/R-5, March 2001*. This QAPP also complies with quality assurance/quality control (QA/QC) procedures of the SWRCB Quality Assurance Program Plan (State Water Resources Control Board 1994). This QAPP describes the quality assurance (QA) and quality control (QC) procedures associated with monitoring activities to characterize impacts of aforementioned discharges on the Salton Sea.

The Quality Assurance Officer is responsible for ensuring that QAPP commitments are implemented and followed to meet project objectives. The Quality Assurance Officer will be independent from the units generating data for this project. The Quality Assurance Officer may, upon mutual concurrence, request modification of this QAPP by the project manager. The QAPP modification process consists of incorporating necessary changes into the QAPP document, obtaining approval signatures, and distributing the revised document to project personnel.

1.2 PROJECT TASK ORGANIZATION

Specific project responsibilities of the Regional Board staff are outlined below. A project organization chart is in Appendix I.

Jose Angel, Project Supervisor, Supervising WRC Engineer, 760-776-8932

- Review and approve the QAPP and subsequent revisions.
- The primary decision maker, responsible for oversight of the project at Regional Board level.

Doug Wylie, Project Manager, Senior WRC Engineer, 760-346-6585

- Review and approve the QAPP and subsequent revisions.
- Ensure that the QAPP is implemented and followed to meet the project objectives.

- Review reports and ensure plans are implemented according to schedule.
- Conduct Health and Safety briefing for sampling team prior to each sampling event.
- Coordinate field and laboratory activities.
- Conduct project activities in accordance with the QAPP.
- Report to the Quality Assurance Officer and management regarding the project status. Prepare interim and final reports for the Quality Assurance Officer and management.

Maria de la Paz Carpio-Obeso, Quality Assurance Officer, Environmental Scientist, 760-674-0803

- Review and approve the QAPP and subsequent revisions.
- Ensure that the QAPP is implemented and followed to meet the project objectives.
- Review reports and ensure plans are implemented according to schedule.
- Responsible for operation of the Regional Board Laboratory.
- Responsible for coordinating lab quality assurance activities.

Jeff Allred, Field Lead Person, WRC Engineer, 760-776-8946

- Responsible for maintaining and calibrating instruments in the field.
- Responsible for coordinating filed activities and ensuring they are consistent with QAPP.
- Assist with monitoring activities as required.
- Prepare a narrative report on sampling event for the Project Manager.
- Responsible for delivery of samples to the laboratory.
- Responsible for decontamination of sampling equipment used in field.

Nadim Zeywar, Field Sampler, Environmental Scientist, 760-776-8971

- Assist with monitoring activities as required.
- Prepare the Quality Assurance Project Plan (QAPP) and revisions.
- Responsible for processing data, maintaining the project database, and validating the field data.

Jason Voskanian, Field Sampler, SETT, 760-776-8930

- Assist with sampling activities as required.
- Responsible for delivery of samples to the laboratory.

Phan Le, WRC Engineer, 760-346-7491

- Assist with sampling activities as required.
- Responsible for calibration of metering equipment prior to sampling event.
- Responsible for assisting Lab Director with water quality analysis.

Jon Rokke, Field Sampler, WRCE, 760-776-8959.

- Assist with sampling activities as required.
- Responsible for delivery of samples to the laboratory.

Kola Olatunbosun, Field Sampler, WRCE, 760-776-8986

- Assist with sampling activities as required.
- Responsible for delivery of samples to the laboratory.

Theresa Illare, Field Sampler, Environmental Scientist, 760-776-8971

- Assist with sampling activities as required.
- Responsible for delivery of samples to the laboratory.

Maribel Rodriguez, Field Sampler, SETT, 760-776-8971

- Assist with sampling activities as required.

Jose Cortez, Field Sampler, WRC Engineer, 760-674-8142

- Assist with sampling activities as required.

Logan Raub, Field Sampler, Environmental Scientist, 760-776-8966

- Assist with sampling activities as required.

Ivory Reyburn, Field Sampler, Environmental Scientist, 760-776-8933

- Assist with sampling activities as required.

1.3 PROBLEM DEFINITION/BACKGROUND

The Salton Sea Transboundary Watershed is located in southeastern California in the Colorado Desert region of the Sonoran Desert. This watershed drains approximately 8,360 square miles and contains five main surface water bodies: the Salton Sea, Alamo River, New River, Imperial Valley agricultural drains, and Coachella Valley storm water channel (Whitewater River channel).

Pursuant to Section 303(d) of the Clean Water Act (CWA), the Regional Board is developing a nutrient Total Maximum Daily Load (TMDL) for the Salton Sea. This TMDL is being developed because the Region's 303(d) list of impaired water bodies identifies the Salton Sea as water quality limited, in part, because nutrient (biostimulatory substance) concentrations violate the water quality objectives (WQOs) established by the Regional Board to protect the following Salton Sea beneficial uses: aquaculture; warm freshwater habitat; wildlife habitat; preservation of rare, threatened, or endangered species; water contact recreation; non-contact water recreation; and potential industrial service supply. Other violated WQOs include aesthetic qualities, dissolved oxygen, biostimulatory substances, and turbidity.

The Alamo and New Rivers transport agricultural discharge and municipal effluent from the Imperial Valley to the Salton Sea. In addition, the New River transports municipal and industrial effluent from Mexicali, Mexico. The Whitewater River transports agricultural discharges, and municipal and industrial effluent, from the Coachella Valley. In addition, agricultural drains discharging directly to the Sea are an important source of pollutants.

The Salton Sea is classified as a eutrophic lake – impaired by nutrients, which result in low dissolved oxygen, high ammonia and phosphorus levels, algal blooms, and foul odors. The Salton Sea is a federal (since 1924) and state (since 1968) designated repository for agricultural, surface, and subsurface drainage waters from the Imperial and Coachella Valleys. Over 70% of freshwater inflows to the Sea consist of agricultural drain water from Imperial Valley. Because the Sea has no outlet and an evaporation rate of 152 cm/year, salts concentrate in it and nutrients enhance the formation of eutrophic conditions.

Great concern has been expressed about the Salton Sea's increasing salinity, contamination from agricultural and urban sources, algal blooms, and disease outbreaks and large die-offs of fish and

waterbirds between 1992 and the present date. Concern has increased due to the importance of the Salton Sea ecosystem to the Pacific Flyway and endangered species.

Therefore, the Regional Board staff is developing a monitoring program to quantify the loads of nutrients to the Salton Sea. This estimation will be included into the TMDL for the Salton Sea.

1.4 PROJECT/TASK DESCRIPTION

Water samples will be analyzed by the laboratory for orthophosphates, total phosphorus, nitrate, nitrite, ammonia, total nitrogen, total organic carbon, calcium carbonate (hardness), alkalinity, sulfate, biological oxygen demand, chemical oxygen demand, chlorophyll A, total suspended solids (TSS), and turbidity. Field measurements will be made for temperature, pH, dissolved oxygen (DO), electrical conductivity (EC), redox potential, and water flow.

The project consists of monthly sampling events for a minimum of 24 months. During each of these sampling events, water samples will be collected from forty seven (47) monitoring stations, as described in Section 2.1, below. Data from the sampling events, in addition to previously collected data, will be assessed by the project's Quality Assurance Officer based on the results of quality control activities such as analysis of quality control samples and adherence to quality control procedures in the collection and storage of samples. The Project Manager will maintain organized records containing all original sample documentation, such as field notes, chain of custody forms, and laboratory analysis results.

1.5 DATA QUALITY OBJECTIVES AND CRITERIA

The mission of the Regional Board is to preserve and enhance the water quality in the Region for the benefit of present and future generations. With this concept in mind, the Regional Board will ensure that beneficial uses of water bodies within the Region are protected as required by State and Federal laws; this implies that regulatory actions will be taken if a pollution source is identified.

The Water Quality Control Plan for the Colorado River Basin Region (Basin Plan) established water quality objectives that will protect beneficial uses of its water bodies, in this case the Salton Sea. The quality of data obtained for this project should support a determination of whether contributing streams in the Salton Sea are degrading water quality in the lake (source analysis). To determine the extent to which discharges of nutrients from rivers and agricultural drains are impacting the Salton Sea, Regional Board staff will collect water samples and monitor water quality at the outflow of the main rivers and agricultural drains for key indicator parameters. Monitoring stations were chosen based on best professional judgment, locations of contributing streams, previous sampling results, direction of flow in the lake, inflows, and site safety and accessibility.

The Regional Board also will use the data for water quality control planning. In turn, this information may be used by the Regional Board for the recommendation/implementation of infrastructure projects that would result in the elimination of pollution caused by the contributing inflows.

As indicated in Section 1.4, the purpose of this project is to determine the amount and sources of nutrient loading to the Salton Sea. As a result, special attention will be placed on the interpretation of the laboratory nutrient analysis.

Because of these data quality needs, strict adherence to holding times, bottle and preservation requirements, collection techniques, and analytical methodology as stated in the published methods and within the contents of this document is necessary. Data quality objectives for all of the measured parameters are listed in Table No. 1 below. Data Quality Indicators are discussed in Section 1.4.1.

Table 1: QA Objectives for all Measured Parameters

Orthophosphate	Water	mg/L	20	80-120	95
Total Phosphorus	Water	mg/L	20	80-120	95
Ammonia	Water	mg/L	20	80-120	95
Nitrate	Water	mg/L	20	80-120	95
Nitrite	Water	mg/L	20	80-120	95
Total Kjeldahl Nitrogen	Water	mg/L	20	80-120	95
Total Organic Carbon	Water	mg/L	20	N/A	95
Hardness	Water	mg/L	20	N/A	95
Alkalinity	Water	mg/L	20	N/A	95
Sulfate	Water	mg/L	20	80-120	95
Total Suspended Solids	Water	mg/L	20	80-120	95
Biological Oxygen Demand	Water	mg/L	20	80-120	95
Chemical Oxygen Demand	Water	mg/L	20	80-120	95
Turbidity	Water	mg/L	20	80-120	95
Temperature	Water	°C	N/A	± 0.15	95
PH	Water	N/A	N/A	± 0.2	95
Dissolved Oxygen	Water	mg/L	N/A	0-200% air saturation: ± 2% of reading or 2% of air saturation[1] 200-500% air saturation: ± 6% of reading	95
Electrical Conductivity (EC)	Water	µmhos/cm	N/A	± 0.5% + 0.001 mS/cm	95
Redox Potential	Water	Eh system	N/A	± 20 mV	95
Chlorophyll A	Water	ug/L Chl.	N/A	N/A	95
Water flow	Water	Cfs	N/A	N/A	95

[1] Completeness criteria will not be applied to results from QC samples.

1.5.1 DATA QUALITY INDICATORS

Precision

The degree of refinement of a measurement will be assessed as the relative percent difference (RPD) for laboratory duplicate samples and field duplicates.

$$RPD = \frac{(C1 - C2) * 100}{\left(\frac{C1 + C2}{2}\right)}$$

RPD = relative percent difference
C_1 = larger of the reported value or measurement
C_2 = smaller of the reported value or measurement

Standard deviation will be used if precision is calculated from more than 3 replicates.

Accuracy

Degree of conformity of a measurement to the actual value or standard will be determined by using spiked samples for inorganics. Samples marked QA/QC will be submitted to the current laboratory contractor to evaluate any matrix effects. The samples will be analyzed for ammonia and nitrates, spiked, and reanalyzed. The percent recovery for the QA/QC samples will be calculated and used to assess matrix interference. The following will be used when a reference material is used:

$$\%R = 100 * \left(\frac{C_M}{C_{RM}}\right)$$

$\%R$ = Percent recovery
C_M = Measured concentration of reference material (RM)
C_{RM} = Actual concentration of reference material (RM)

Representativeness

Representativeness will be assured by using a statistically significant number of water samples.

Completeness

A minimum of ninety-five percent (95%) of the water samples collected are expected to yield valid usable data. This will result in the generation of sufficient data to meet the final test design criteria.

$$\%C = 100 * \left(\frac{V}{T}\right)$$

$\%C$ = Percent completeness
V = Total number of measurements or laboratory results judged valid
T = Total number of measurements or laboratory results

1.6 DOCUMENTATION AND RECORDS

In order to maintain a clear record of sample collection and custody, Regional Board field staff will keep field notes, sample collection records, copies of chain of custody forms, and quality control sample records for each sampling event.

Regional Board staff will maintain Project records in accordance with the QAPP. These records will consist of:

- field logs/notes, quality control logs, and calibration logs
- laboratory analytical reports
- preliminary data reports summarizing field activity and quality control for each sampling event
- miscellaneous correspondence
- final report

The field notes will be entered into bound field log notebooks with pre-numbered pages. Each page of the field logs and field data worksheets will be dated and signed by a member of the sampling team at each sampling station, at the time of sampling, and the following information will be entered into the field log book:

- Observations about the weather and sampling station
- The latitude and longitude of the sampling station, as determined using a global positioning system (GPS) receiver when necessary
- Identification codes (sample I.D.), specific sampling point locations, and sampling methods for all samples taken.
- The instream YSI readings for temperature, dissolved oxygen, pH, conductivity, and Redox Potential.
- Sample codes and time and location of preparation for all quality control samples prepared in the field
- Any deviations from QAPP procedures
- Any noteworthy observations
- Flow data

Quality control (QC) samples will be documented in a bound Quality Control Log with pre-numbered pages. The Quality Control Log will document the quality control samples submitted to laboratories and the results of the analysis of these samples. For each QC sample, the quality control log will contain the:

- sample identification code
- supplier of the QC sample
- value reported by the supplier
- date of preparation and submission
- name and signature of the person submitting the QC sample
- laboratory performing the analysis
- analysis method
- reported value from the laboratory

Calibration of the YSI 6600 multiprobe multi-parameter water quality sonde will be documented in a bound calibration log field notebook with pre-numbered pages. The calibration log will contain:

- date and time of calibration
- persons performing the calibration
- signature of one of the persons performing the calibration

- all standard solutions used in calibration, including the source and date of preparation of the standard solution
- initial reading of the YSI when tested against each standard solution, and the temperature of each standard solution at the time of calibration
- any deviations from the QAPP
- any difficulties or relevant notes about the calibration

Upon completion of the laboratory analysis of the samples from each sampling event, the laboratory will prepare and submit to the Project Manager a Laboratory Analytical Summary. The summary should consist of analytical results and chain of custody forms.

A Preliminary Data Report will be developed by the Project Manager, and filed with the Quality Assurance Officer within 10 days from the date the Project Manager receives all lab results for a sampling events. This report will summarize field activities and observations; it also will include field measurements and the results of laboratory analysis. This report also will include a quantitative analysis and discussion of the results of quality control activities, and what these results indicate about the quality of data generated in each sampling event. The report can also include recommendations to improve/modify the QAPP.

The field logs, quality control log, and calibration log along with all additional documentation consisting of any laboratory records and chain of custody forms, will be stored in an organized manner by the Laboratory Director, and will be stored in an accessible manner at the Laboratory.

Once all the sampling is completed, a narrative report will be prepared by the Project Manager for the Quality Assurance Officer and management. At a minimum, this report will discuss any problems encountered and their solutions. Additionally, it will discuss any deviations from this QAPP, if any, as well as the quality of all data.

Quality control records will be maintained documenting the preparation and use of quality control samples and equipment calibration. Chain of custody forms will contain sample identification codes, collection times and locations, and names and signatures of all persons in custody of the samples.

Laboratory records of sample analysis will be collected from each laboratory, showing the samples analyzed, the persons analyzing the samples, the time and date of analysis, and any deviation from standard operating procedures. In addition to maintaining the documentation and records listed above, Regional Board staff will enter all data and meta-data from these forms into a single database, which will be utilized for data validation and data assessment. Meta-data is all the relevant information related to the data itself. The maintenance of the database, as well as the storage of all documentation and records listed above, will be the responsibility of the Project Manager.

1.6.1 TRAINING AND CERTIFICATION REQUIREMENTS

Field samplers must have completed a 40-hour OSHA-approved HAZWOPER training course, and if necessary an 8-hour HAZWOPER yearly refresher course. The Project Manager will ensure that all field samplers have valid and current HAZWOPER training. There are no specialized training/certification requirements needed to perform the Project's activities.

2 DATA GENERATION/DATA ACQUISITION

2.1 SAMPLING PROCESS DESIGN

During the project, Regional Board staff will take samples and collect data from sixty (60) monitoring stations in the Salton Sea at the contributing inflows. The Laboratory will be required to conduct the analyses within the specified holding periods, and in a reasonable time. The Project Manager and QA Officer will evaluate the data generated during each sampling event to determine if any changes in the QAPP are necessary to better meet study objectives. The following paragraphs provide the rationale for the selection of the constituents and monitoring stations.

2.1.1 SAMPLING CONSTITUENTS

The fifteen monitoring stations will be sampled for the constituents listed in Table No. 2, below.

Table 2: Sampling Constituents

Orthophosphate	mg/L	USEPA 365.2
Total Phosphorus	mg/L	USEPA 365.2
Ammonia-N	mg/L	USEPA 350.1
Nitrate-N	mg/L	USEPA 300.0
Nitrite-N	mg/L	USEPA 353.2
Total Kjeldahl Nitrogen	mg/L	USEPA 351.3
Total Organic Carbon	mg/L	USEPA 415.1
Hardness (CaCO3)	mg/L	USEPA 130.2
Alkalinity	mg/L	USEPA 310.1
Sulfate	mg/L	USEPA 375.1
Biological Oxygen Demand ($20^{\circ}C$ BOD_5)	mg/L	USEPA 405.12
Chemical Oxygen Demand	mg/L	USEPA 410.2
Total Suspended Solids	mg/L	USEPA 160.2
Turbidity	NTU	USEPA 180.1
Temperature	°C	YSI Probe
pH	pH Units	YSI Probe
Dissolved Oxygen	mg/L	YSI Probe
Electrical Conductivity (EC)	µmhos/cm	YSI Probe
Chlorophyll A	ug/L	HPLC, Bidigare et al. 2002
Redox Potential	E_h	YSI Probe

The sampling constituents include both causal factor indicators (nutrients that stimulate plant growth) and biological response indicators (assessment of impacts on water quality). The complexity and site-specific nature of biostimulatory substances require an array of indicators to assess and estimate the load.

Phosphorus

Phosphorus concentration is considered an indicator because algal growth in the Salton Sea may be limited by the availability of that nutrient. It can be measured in several forms. Total phosphorus (TP) and orthophosphates are used largely for setting criteria for lake management. TP (organic and inorganic phosphorus) is important for TMDL load estimations and numeric targets. Orthophosphate is directly available for plant uptake.

Nitrogen

Nitrogen concentration can serve as a valuable indicator in nitrogen limited ecosystems. It can be measured in inorganic (ammonia, nitrate, nitrite) and organic (total nitrogen) forms. Inorganic forms are available for algae uptake. Total Kjeldahl nitrogen is often a good indicator of algal biomass in lakes.

Dissolved Oxygen Concentration

Dissolved Oxygen Concentration is an important indicator where aquatic life is a beneficial user. This parameter is used widely and is established in state water quality standards.

Total Organic Carbon

Total Organic Carbon may indicate the available energy source for the heterotrophic community and their response impact to algal growth. The measurement of total organic carbon also can indicate or be interfered by the amount of suspended sediments.

Total Suspended Solids

TSS has an impact on water transparency and its source may be both algae and sediments. Site-specific quantitative relationships can be developed to predict transparency. TSS reveals the fine suspended solids that frequently are transported with water flow. Phosphorus generally adsorbs to these fine sediment particles. This is one pathway of phosphorus to the Salton Sea.

Electrical Conductivity or Total Dissolved Solids

TDS or EC is a measurement of salinity.

Chlorophyll A

Chlorophyll A is a reliable indicator of algae biomass. This is the photosynthetic pigment of algae cells. Algae are generally either directly (algal blooms) or indirectly (low dissolved oxygen, low pH, high turbidity) responsible for several problems due to excessive nutrient concentration. Both seasonal mean and instantaneous maximum concentrations can be used to determine impairments.

Transparency

Secchi depth is widely used to estimate algal biomass and trophic state, although this estimation can be interfered from a variety of other sources like suspended sediments. Turbidity also is used to estimate both algal biomass and trophic state.

pH

Excess algae levels can be responsible for extreme diurnal fluctuations in water pH. Generally, aquatic organisms are most sensitive to extreme pH levels.

Hardness
Hardness can be represented as the sum of calcium and magnesium concentrations.

Alkalinity
Alkalinity describes the capacity of water to neutralize acid.

Nutrient ratios
Ratios of nutrient concentrations might indicate the relative intensity of algal growth in a calendar season (summer : winter), crop growth season (vegetable crops : perennial crops), response to a specific stream, etc. However, this ratio may be difficult to interpret due to unknown mixing times and unknown algae uptake rates.

Redox Potential
Redox Potential indicates the reducing conditions of the lake/river, dissolved oxygen, and bioavailability of other ions.

Biological Oxygen Demand
Biological Oxygen Demand is a nutrient overenrichment indicator. This parameter reveals the amount of bioavailable carbon (energy) for heterotrophic microorganisms. These organisms are active and important in nitrogen and phosphorus cycles. Therefore, this parameter may indicate microbial activity and nutrients available for algae uptake.

Chemical Oxygen Demand
Chemical Oxygen Demand is used to measure everything that can be oxidized in the water sample (organic and inorganic).

2.1.2 MONITORING STATIONS

Regional Board personnel conducted field inspections in the area of the lake to ascertain general characteristics (e.g., direction of flow, cross-sectional areas, flow) of tributaries, and to identify potential sampling points. Currently, almost all drains and rivers will be monitored. However, future monitoring stations will be chosen based on best professional judgment, locations of contributing streams, previous sampling results, direction of flow in the lake, inflows, and site accessibility. Visual inspections on flow direction and flow rate of the small drains will be conducted prior to sampling to indicate any backflow from the Salton Sea to the drains. Table No. 3 shows the monitoring stations.

Table 3: Description of Sampling Stations

Site No.	Site Label	Description
01	FD	F Channel, Riverside County
02	ED	E Channel, Riverside County
03	OG	Oasis-Grant, Riverside County
04	DD	D Channel, Riverside County
05	CD	C Channel, Riverside County
06	AV83	Avenue 83 Drain, Riverside County
07	AV79	Avenue 79 Drain, Riverside County
08	LO	Lincoln-Oasis Drain, Riverside County
09	AC	A Channel, Riverside County
10	AV76	Avenue 76 Drain, Riverside County
11	AV74	Avenue 74 Drain, Riverside County
12	CVSWC	Coachella Valley Storm Water Channel, Riverside Co.
13	JST	Johnson Street Drain, Riverside County
14	GRST1	Grant Street Drain, Riverside County
15	GRST2	Grant Street 0.5 Drain, Riverside County
16	HST1	Hayes Street Drain, Riverside County
17	HST2	Hayes Street 0.5 Drain, Riverside County
18	GAST1	Garfield Street Drain, Riverside County
19	GAST2	Garfield Street 0.5 Drain, Riverside County
20	AST1	Arthur Street Drain, Riverside County
21	AST2	Arthur Street 0.5 Drain, Riverside County
22	CSTE	Cleveland Street East Drain, Riverside County
23	CSTW	Cleveland Street West Drain, Riverside County

24	CAC	Caleb Channel, Riverside County
25	CST2	Cleveland Street 0.5 Drain, Riverside County
26	MST	McKinley Street Drain, Riverside County
27	SCR	Salt Creek, Riverside County
28	ND5	Niland 5 Drain, Imperial County
29	ND4	Niland 4 Drain, Imperial County
30	ND3	Niland 3 Drain, Imperial County
31	ND2	Niland 2 Drain, Imperial County
32	ND1	Niland 1 Drain, Imperial County
33	ZD	Z Drain, Imperial County
34	WD	W Drain, Imperial County
35	UD	U Drain, Imperial County
36	TD	T Drain, Imperial County
37	SD	S Drain, Imperial County
38	RD	R Drain, Imperial County
39	QD	Q Drain, Imperial County
40	PD	P Drain, Imperial County
41	OD	O Drain, Imperial County
42	ARO	Alamo River Outlet (Garst Road), Imperial County
43	VD3	Vail 3 Drain, Imperial County
44	PUMD	Pumice Drain, Imperial County
45	VD5	Vail 5 Drain, Imperial County
46	VD5A	Vail 5A Drain, Imperial County
47	VD6	Vail 6 Drain, Imperial County
48	VCD	Vail Cut-Off Drain, Imperial County
49	NRO	New River Outlet, Imperial County

50	TRD12	Trifolium 12 Drain, Imperial County
51	TRD13	Trifolium 13 Drain, Imperial County
52	TRD14A	Trifolium 14A Drain, Imperial County
53	TD1	Trifolium 1 Drain, Imperial County
54	TRSD	Trifolium Storm Drain, Imperial County
55	TRD18	Trifolium 18 Drain, Imperial County
56	POED	Poe Drain, Imperial County
57	TRD19	Trifolium 19 Drain, Imperial County
58	TRD20	Trifolium 20 Drain, Imperial County
59	TRD22	Trifolium 22 Drain, Imperial County
60	TRD23	Trifolium 23 Drain, Imperial County

To help determine the spatial distribution in the area of the monitoring station, the monitoring station consists of three (3) sampling points (S1, S2, and S3), distributed along the surface and perpendicular to the flow of the canal/drain being monitored. At the outlets of the New and Alamo Rivers, the sampling points are to be spaced at approximately equal intervals from each other and from the edge of the canal/drain (i.e., at a distance equal to w/4, where "w" is the top width of the cross-sectional area). Figure No. 1, below, illustrates this.

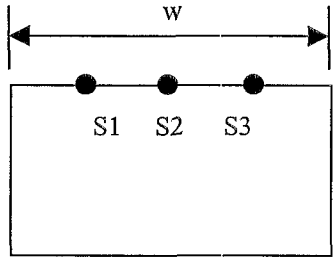

Figure 1: Sampling Points at Monitoring Stations

However, at the other monitoring locations, only one point will be used because of the narrowness of the drains.

2.2 SAMPLING METHODS REQUIREMENTS

In order to obtain comparable and accurate results, appropriate sampling protocols must be followed uniformly at each sampling station for each constituent during all sampling events. The following two sections describe sampling and handling procedures for each sample parameter.

2.2.1 BIOLOGICAL, CHEMICAL, AND PHYSICAL INDICATORS

To prevent sample contamination, one pre-cleaned sample bottle will be dedicated for each sample parameter or parameters at each sampling point. Grab samples will be taken at one (1) foot below water surface (bws) at each sampling point of each monitoring station using a 1000-ml polyethylene pre-cleaned bottle attached to the end of a "S wing Sampler®". Then, with the sampler downstream of the bottle, the bottle will be plunged downward approximately 1 foot into the water, and allowed to fill with the opening pointed slightly upward into the current. The bottle then will be raised out of the water, and the 1000-ml sample will be distributed immediately to the appropriate, uniquely-labeled sample bottles (470-mL). The bottles then will be immediately capped tightly and placed into ice chests. The bottles will be obtained from the laboratory contractor and will contain the required preservatives.

In order to ensure accurate results, acceptance requirements for all sample containers are as follows:

- Inorganic Sample Storage Containers are to be certified clean and pre-preserved
- Organic Sample Storage Containers are to be certified clean and pre-preserved

- The laboratory contractor must submit written documentation verifying sample container specifications

Table 4 shows required containers, preservatives, techniques, and holding times for all constituents.

Table 4: Required Containers, Preservatives, Techniques, and Holding Times

Orthophosphate	1-L low density polyethylene bottle with poly-lined, white poly cap	Cool below 4 °C	28 Days
Nitrite			
Nitrate			
Total Kjeldahl Nitrogen			
Total Suspended Solids			7 days
Turbidity			48 hours
Biological Oxygen Demand			48 hours
Chemical Oxygen Demand			
Sulfate			28 days
Alkalinity			
Total Phosphorus	1-L low density polyethylene bottle with poly-lined, white poly cap	Cool below 4 °C; Sulfuric Acid Preservative (pH <2)	28 days
Ammonia			
Total Organic Carbon	VOA vial	Cool below 4 °C	28 days
Chlorophyll A	1-L low density polyethylene bottle with poly-lined, white poly cap	Cool below 4 °C; Mg CO3	Analysis should be performed ASAP following sampling
Hardness (CaCO3)	1-L low density polyethylene bottle with poly-lined, white poly cap	Cool below 4 °C; Sulfuric Acid Preservative (pH <2)	6 months

2.2.2 FIELD MEASURED PARAMETERS

A YSI 6600 Multiprobe will be used to measure the levels of dissolved oxygen (DO), pH, temperature, electrical conductivity (EC), and redox potential at each sampling point. The probe will be deployed at one (1) feet bws. The readings from the YSI 6600 probe will be taken within, at most, a two-foot radius of the point of sample collection, simultaneously as the samples for lab analyses are collected. The sample ID numbers, YSI 6600 readings, field observations, and any deviation from standard operating procedures will be recorded in the field notebook immediately following collection of each sample. Also, stream flow will be obtained from USGS, IID, and CVWD measurements.

2.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Each sample container will be labeled with a unique sample identification code, as well as the date and location where the sample was taken. The sample ID code is of the format:

Project - Sampling Station – Location - Sample Type – Sample Parameter

The location refers to the cross-section, which is numbered from left to right when facing downstream. Sample type indicates a sample (0), duplicate (1), spike (2), MS/MSD (matrix spike/matrix spike duplicate) (3/4). Nitrate, nitrite, ammonia, total Kejldahl nitrogen, orthophosphates, total phosphorus, total organic carbon, calcium carbonate, sulfate, biological Oxygen demand, chemical oxygen demand, chlorophyll A, Turbidity, and total suspended solids parameters are indicated by NO3, NO2, NH3, TKN, o-PO4, Total PO3, TOC, CaCO3, SO4, BOD, COD, CHL A, TUR, and TSS respectively. Waterproof ink will be used to encode the self-adhesive sample labels. The labels will be affixed to the sample bottles according to manufacturer's specifications.

All samples for lab analyses will be placed in waterproof plastic "zip lock" bags and immediately be stored in an ice chest. Samples from each cross-section shall be stored in separate zip lock bags. The ice chest will contain sufficient ice to maintain the samples at a temperature below 4°C at all times until they are relinquished to the appropriate laboratory personnel. The samples will be delivered to the appropriate laboratory, taking into consideration the holding times listed in Table 4. A ten-day notice will be given to the analytical laboratory prior to the delivery of samples. Samples will be retained in the custody of Regional Board staff until they are delivered to laboratory personnel.

All samples will be delivered with chain of custody forms. A sample chain of custody form is included in Appendix II. Any violation of holding times or other sample handling and custody requirements will be reported to the Project Manager and the QA Officer, and recorded in the quality control records, and taken into consideration during data validation, as described in section 4.1.

2.4 ANALYTICAL METHODS REQUIREMENTS

The laboratory will analyze all samples except the field measurements. Spike samples for laboratory analyses will be obtained from Environmental Research Associates, Arvada, CO. All samples will be analyzed using USEPA approved methods. The process for selection of laboratories for this sampling event involved reviewing Standard Operating Procedures (SOPs) (Appendix III and Attachment B), QA documents, corrective action plans, detection limits, and laboratory location.

2.5 QUALITY CONTROL REQUIREMENTS

In order to assess whether data quality requirements are being met, a number of quality control checks will be implemented, as described below:

- Duplicate or co-located samples will be taken for all constituents at approximately 10% of the sampling points.

- Field blanks for inorganic parameters will be submitted to the current laboratory contractor at a rate of 1 blank/day/event.

- Spike samples, containing a known concentration of a specific chemical, will be obtained from Environmental Resource Associates and submitted to the current laboratory contractor along with the other samples. The 5-mL samples will be diluted with 1 liter of water to a known concentration. Two spike samples for every chemical parameter will be submitted every three sampling events. In addition, a dilution water blank will submitted for analysis with the spike samples to evaluate any bias. All laboratory results must be within 20% of the concentration value submitted by Environmental Research Associates.

- Matrix effects for inorganics will be evaluated by the collection of two double volume samples at two separate locations. These two samples will be submitted to the current laboratory contractor with the designation "QA/QC" samples. These known samples are called matrix spike (MS) and matrix spike duplicates (MSD). The current laboratory contractor will analyze, spike, and reanalyze the samples, and will calculate a percent recovery. This value will be used to ascertain any matrix effects.

- Temperature blanks will be included in each ice chest submitted to a lab. The temperature of these blanks will be analyzed using chain of custody to ensure that the samples have been maintained at the prescribed temperature (4°C).

All QC samples will be stored and labeled following the same methods. QC samples will be submitted to the labs as blind samples. Table No. 5, below, summarizes the QC samples to be utilized.

Table 5: Quality Control Sample Requirements

Duplicate Samples (10%/Event, 6 for each parameter or group of parameters)	60
Field Blanks (1/day/event)	4
Spike Samples (2 for each appropriate parameter per three sampling events)	14
Dilution Water Blank	1
Matrix Spike Samples (2 double volume samples at two separate locations per sampling event)	4

2.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Proper maintenance procedures will be followed and documented. The YSI 6600 Multiprobe will be tested, inspected, and serviced as necessary prior to each sampling event, pursuant to procedures recommended in the YSI, Inc., *6-Series Environmental Monitoring Systems Operations Manual*. Its batteries will be checked at the beginning of the first sampling event and replaced as

appropriate. The EC and pH probes will be tested using a 1,000 µmhos/cm EC solution and a pH solution (4, 7, 10) respectively, prepared by the Regional Board laboratory. The DO probe will be tested using saturated air. All probes will be visually inspected for damage at each sampling point prior to field measurements, and will be serviced as appropriate.

2.7 INSTRUMENT CALIBRATION AND FREQUENCY

The YSI 6600 will be calibrated prior to the beginning of each day's sampling pursuant to the calibration procedures recommended in the YSI, Inc., *6-Series Environmental Monitoring Systems Operations Manual* (1999). For subsequent sampling events, the YSI 6600 will be calibrated at the lab following the Manual prior to the beginning of sampling events. Results of calibration measurements will be documented in the field log notebook and presented to the QA Officer. The DO probe will be calibrated using tap water. A three-point calibration, using pH 4, pH 5 and pH 10 calibration standards, will be performed on the pH probe. Table 6, below shows the parameter specifications for the YSI 6600.

Table 6: Parameter Specifications for the YSI 6600

pH	0 to 14 units	± 0.2 units	0.01 units	3-pt, with pH buffered solutions (pH 4, 7 & 10)
Temperature	– 5 to 45 °C	± 0.15 °C	0.01 °C	* not required
Dissolved Oxygen	0 to 50 mg/L	0-20 mg/L, ± .2 mg/L 20-50 mg/L, ± 0.6	0.01 mg/L	% air saturation
Conductivity	0 to 100 mS/cm	± 0.5% of reading + 0.001 mS/cm	0.001 mS/cm	KCl

*As per the manufacturer's specifications. Temperature accuracy is verified every 6 months using a thermometer which is calibrated with a thermometer traceable to the National Institute of Standards and Technology.

2.8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES/CONSUMABLES

All supplies and consumables will be documented and inspected upon receipt and before sampling. Seals need to be intact. Sampling bottles should be clean and contain the proper preservatives. A log on the inspection/acceptance process will be kept. Records will include identification number, item description, date received, date accepted, expiration date, handling and storage conditions, and name of the inspector.

2.9 DATA ACQUISITION REQUIREMENTS (NON-DIRECT METHODS)

Several other institutions have analyzed the Salton Sea for many constituents. Previous data relative to this project will be used after checking its quality. Criteria for accepting the already-collected data include its representativeness of similar conditions, any documented bias, logical methods of evaluating the data and its applicability to this project, and data summarization. The QAPP Developer will supply previous data to the QA Officer as requested.

2.10 DATA MANAGEMENT

2.10.1 DATA STATISTICAL ANALYSIS

Data from the sampling methods will be analyzed statistically using the Spreadsheet Excel software. Data will be entered into Excel in columns, with one column for each method. Descriptive Statistics (e.g. mean, standard deviation, and coefficient of variation) will be computed for each column.

The mean is calculated as the sum of all observations divided by the total number of observations. The standard deviation is a measure of the spread of the data, and can be calculated as the square root of the sample variance. The variance measures the variability or spread of the observations about the sample mean and can be calculated as the sum of the squared differences from the sample mean divided by the number of observations less one. Coefficient of variation is calculated as the standard deviation divided by the mean. A coefficient of variation of more than one (1.0) will indicate that the data are not normally distributed.

3 ASSESSMENT AND OVERSIGHT

3.1 ASSESSMENT AND RESPONSE ACTIONS

Surveillance of the records and overall status of the project will be conducted by the QA Officer to ensure that all requirements of the QAPP are met. Surveillance will be conducted after each sampling event, after all laboratory results have been received for that sampling event.

A Technical Systems Audit also will be performed by the QA Officer. During this audit, the QA Officer will examine field activities and record-keeping procedures to assess conformance to the QAPP. This audit will take place any time, and at the discretion of the QA Officer. Any non-conformance with the QAPP will be corrected and documented as described in Section 4. Performance Evaluations of laboratories will be conducted through the use of quality control samples, namely split samples and matrix spike samples. A review of the laboratory's QA for this project also will be conducted.

At the completion of the project, but prior to producing the final report, an Audit of Data Quality will be performed to assess the handling of all data and to correct any errors found in the project database. A Data Quality Assessment also will be performed in which statistical tools will be used to determine whether the data met all assumptions that the Data Quality Objectives and data collection design were developed under, and if the total error in the data is tolerable. The total error present will be quantified to determine if the quality of the data is adequate to support a determination regarding the influence of contributing inflows on the water quality of the Salton Sea.

3.2 REPORTS TO MANAGEMENT

Upon completion of the project, the Project Manager will prepare a final Project report. This final report will include a summary of the activities performed, the resulting data, and the quality of the resulting data, and will identify any samples that indicate violations of Water Quality Standards. This final report will contain an assessment of whether or not contributing streams in the Salton Sea were polluting the lake during the time period of the study, and a statement of the confidence with which the assessment was made, based on the quality of the data. This report will be forwarded to management, as well as appropriate officials from Imperial County, Imperial Irrigation District, Coachella Valley Water District, Salton Sea Nutrients TMDL TAC Members, and Riverside County.

4 DATA VALIDATION AND USABILITY

4.1 DATA REVIEW, VERIFICATION, VALIDATION, AND RECONCILIATION

4.1.1 DATA REVIEW

Regional Board staff will be responsible for validating the project's data to ensure that QA guidelines have been followed. QA performed by the Regional Board will ensure that the data transfer process is error-free, that results reported are reasonable in relation to the distribution of previously reported results by Imperial County and the Regional Board, and that samples were analyzed in accordance with procedures in Table No. 2 and 4.

After each sampling event, the Project Manager will review the field notes and field data generated to assess adherence to the project sampling design in terms of the spatial distribution of sampling locations. Departures from the sampling design will be considered in the design of each subsequent phase of sampling. Deviations from the sampling design may change the data needed to characterize the system. Departures from the sampling design also may be due to unforeseen field conditions, which may require adjustment of the sampling design. Significant departures from the project sampling design and responses to those departures will be noted in the project database, as well as the Audit of Data Quality, and the final report. In the Data Quality Assessment, the Project Quality Assurance Manager and Project Manager will consider the effects of any departures from the sampling design on the overall completeness of the data generated, and thus the usability of the data set for drawing conclusions.

4.1.2 DATA VERIFICATION

Verification of adherence to the sample collection and equipment decontamination procedures contained in Attachment A of this report will be determined through the field records, Technical Systems Audit, and project surveillance identified above. All information will be considered in the final Audit of Data Quality. Some departures from the sample collection procedures are unacceptable, and will result in data that will not be considered valid for use in this study. Unacceptable departures from sample collection procedures include the use of contaminated sampling bottles, lack of critical sample collection information, or any other activity which would result in cross-contamination or incorrect identification of samples.

Departures from the sample handling and custody procedures contained in section 2.3 will be determined through the review of chain of custody forms and laboratory analysis forms. In order for data to be considered valid for meeting the data quality objectives of this study, all samples' chain of custody forms must be in the possession of the project manager, and strict adherence to holding times and temperatures must be followed. Data generated from samples that do not meet these requirements will not be considered valid for use in this study.

Verification of proper calibration of the YSI 6600 will be performed during the audit of data quality through a review of the quality control records. Calibration values also will be assessed to determine the potential error in field measurements. Measurements will not be made unless the

instrument is properly calibrated.

4.1.3 DATA VALIDATION

Validation of laboratory data will be performed in the Audit of Data Quality by assessing the results of QC sample analyses. Inorganic lab data will be validated for precision, accuracy, and completeness according to criteria in Table No. 1.

Data for inorganic QC samples falling outside the specified precision will be re-analyzed. The laboratory will be notified of this procedure. Should the analyses confirm the previous results, the sample collection and sample handling procedures will be labeled as "suspect" and, subsequently, re-evaluated. Any value that cannot be confirmed, based on the acceptable recovery for a split sample, will be rejected.

Lab data results for all other samples also will be range-checked for outliers by comparing lab results with Regional Board Trend Monitoring Program data. Values falling outside the expected ranges will be labeled as "suspect" and further investigated. Values, which are expected to be normally distributed, but labeled "suspect", will be further evaluated and rejected using Chauvenet's Criterion (i.e., if the value deviates from the mean value of the data set by more than $1.96\forall$, where $\forall$ is the standard deviation for the data set). Results that clearly depart from the established distribution will be identified, and Regional Board staff will discuss these results with data providers to ensure accuracy.

The data then will be entered into a database by Regional Board staff. It is conceivable that errors could occur in entering the data (e.g., transposing the decimal point for a particular result or keying in the wrong Sample ID). Therefore, once a data set has been entered into the database, all records will be checked by the Project Manager to ensure accuracy.

Regional Board staff will discuss missing data with the laboratories submitting the data. In some cases, missing data will be denoted as missing in reports. For all missing data, and any other data requiring special explanation, qualifiers will be included in the database and in data reports. Missing data will be designated as "NR," meaning *Not Reported*.

The Regional Board Quality Assurance Manager will be responsible for validation and final approval of all data for use in this study. The final project report will contain a discussion of relevant information obtained through the Audit of Data Quality about the quality, validity, completeness, and limitations of the data obtained in this study.

Data objectives for this project do not require a full, formal, and independent data validation. The data has no legal requirement for independent validation. Although the data is considered legally defensible as presented herein, all records will be available for independent evaluation should the need arise at a later date.

The current laboratory contractor will communicate with the Quality Assurance Officer about any problem and need for data reconciliation.

5 HEALTH AND SAFETY PLAN

5.1 CONTAMINATION CONTAINMENT ZONES

Upon arrival at a particular sampling station, an exclusion, decontamination, and support (clean) zone will be established and maintained at the sampling station throughout the duration of sampling at the particular station. The process of establishing and breaking down containment zones will be repeated at each sampling station. The exclusion zone consists of the shore of the lake and extends ten (10) feet inland. The decontamination zone will be set up adjacent to the exclusion zone, extending 10 feet inland of the exclusion zone boundary. The decontamination zone will be used for personnel and equipment decontamination and will include wash water, soap, paper towels, and trash bags. All contaminated solid waste material will be placed in trash bags for proper disposal. Only biodegradable antibacterial soap will be used at the site. Wash water runoff will be contained and disposed of properly. The clean area will be set inland of the decontamination zone.

5.2 PERSONAL PROTECTIVE EQUIPMENT

The general concerns at sampling sites are the potential exposure to pathogens and toxicants present in waters being sampled, the risk of being struck by an automobile when taking samples near the roadside or off of bridges, and the risks of sunburn, excessive heat exposure, and insect and possibly snake bites. In addition, the sampling crew should be aware of the risk of falling into a drain. No less than three experienced samplers will be out in the field at one time. (The sampling crew also will have a functional cellular phone in one of the vehicles).

- Any member of the sampling team has the authority to stop the sampling event when he/she determines that conditions at the site (e.g., rain, dust, local emergency, etc.) preclude safe sampling. A Hazard Evaluation Plan (HEP) will be done for each day of sampling

- The following precautions will be taken to reduce the risk of being around automobile traffic. At roads, bridge crossings, and wherever traffic reasonably is expected to be present, traffic cones will be set at approximately 30-foot intervals to form at least a 5-foot wide "safety corridor" between the traffic and sampling crew. At the beginning and end of the corridor, one State vehicle must be parked as part of the "safety corridor". The parked vehicle and traffic cones must be clearly visible to on-coming traffic from a distance of at least 120 feet. Samplers also will be required to wear orange vests.

- To reduce the risk of heat exposure and sunburn, samplers will wear sunscreen and the vehicle will always have plenty of cold drinking water supplied by the Project Manager. If any of the samplers begin to experience symptoms of heat exhaustion, such as cramps or dizziness, he or she will immediately be removed from the sun and given plenty of cool liquids. If these symptoms persist, he or she will be taken to the nearest hospital.

- Extra caution should be used when working near or around drains to reduce the risk of potentially falling in.

- To reduce the risk of insect bites, samplers will use insect repellent.

- To reduce the risk of snake bites, samplers will check for snakes prior to entering the area. If a snake bite occurs, ice will be placed on the bite. The sampler will be transported immediately to the nearest medical facility.

- The main health threat during sampling is exposure to pathogens and toxicants through incidental and accidental contact with Salton Sea water. The following personal protective equipment will be used for those directly handling samples at the Salton Sea:

    - Face Shield
    - Latex Examination Gloves (inner gloves)
    - Nitrile Gloves (outer gloves)
    - Tyvek Suit or isolation gown
    - Boot covers

5.3 PERSONNEL DECONTAMINATION PROCEDURES

The Support Zone must not be entered with contaminated PPE (Personal Protective Equipment). All team members coming out of the Contaminated Zone must proceed immediately to the Decontamination Zone and use the following decontamination procedures before proceeding to the Clean Zone:

1. Remove boot covers and place them in a plastic bag;

2. Wash outer rubber gloves with antibacterial soap prior to removal of any other PPE. Place outer gloves in the storage bin labeled "De contamination PPE No. 1";

3. Carefully remove Tyvek suit and place it in the storage bin labeled "De contaminated PPE No. 2" (making sure not to let skin contact the outside of the suit);

4. Remove face shield and place it in a plastic bag;

5. Remove latex gloves carefully to avoid contact with bare skin and dispose of them in the trash bag. Thoroughly wash hands with antibacterial soap; and

6. Properly dispose of wash water.

Note: everything that is touched (pens, pencils, rinse water bottles, probes, etc.) with dirty gloves could be contaminated. Avoid touching these items with bare skin.

5.3.1 EMERGENCY NUMBERS AND FACILITIES

Sampling personnel should call 911 in the event of an emergency. The hospital nearest the sampling location can be:

Name: Kennedy F. Memorial Hospital
Address: 47111 Monroe Av., Indio, CA
Phone: (760) 347-6191

Name: Pioneers Memorial Hospital
Address: 207 W. Legion Rd., Brawley, CA
Phone: (760) 347-6191

Name: El Centro Regional Medical Center
Address: 1415 Ross Av., El Centro, CA
Phone: (760) 339-7100

Other emergency numbers include:

Name: Imperial County Sheriff – Dispatch
Address: 328 Applestill Road
P.O. Box 1040
El Centro, CA 92244
Phone: (760) 339-6311

Name: Riverside County Sheriff – Dispatch
Phone: 911 (emergency calls only)
1-800-950-2444 (emergency and crime reporting dial)

In case of an emergency, sampling personnel also should contact the QA as soon as practical at (800) 796-7363, PIN 102-9073.

5.3.2 AFTER SAMPLING

Place samples into an ice chest filled with wet ice, and keep water drained from ice chests to avoid soaking container labels. Due to relatively short holding times, samples are to be delivered expeditiously to the current laboratory contractor. Contaminated equipment should be packed in designated containers for transport to the Regional Board office. Decontaminate and properly clean ALL items that were exposed in the field. Make copies of field notes and put originals in the appropriate binder.

5.4 DECONTAMINATION PROCEDURES

Please see Attachment A.

6 BIBLIOGRAPHY

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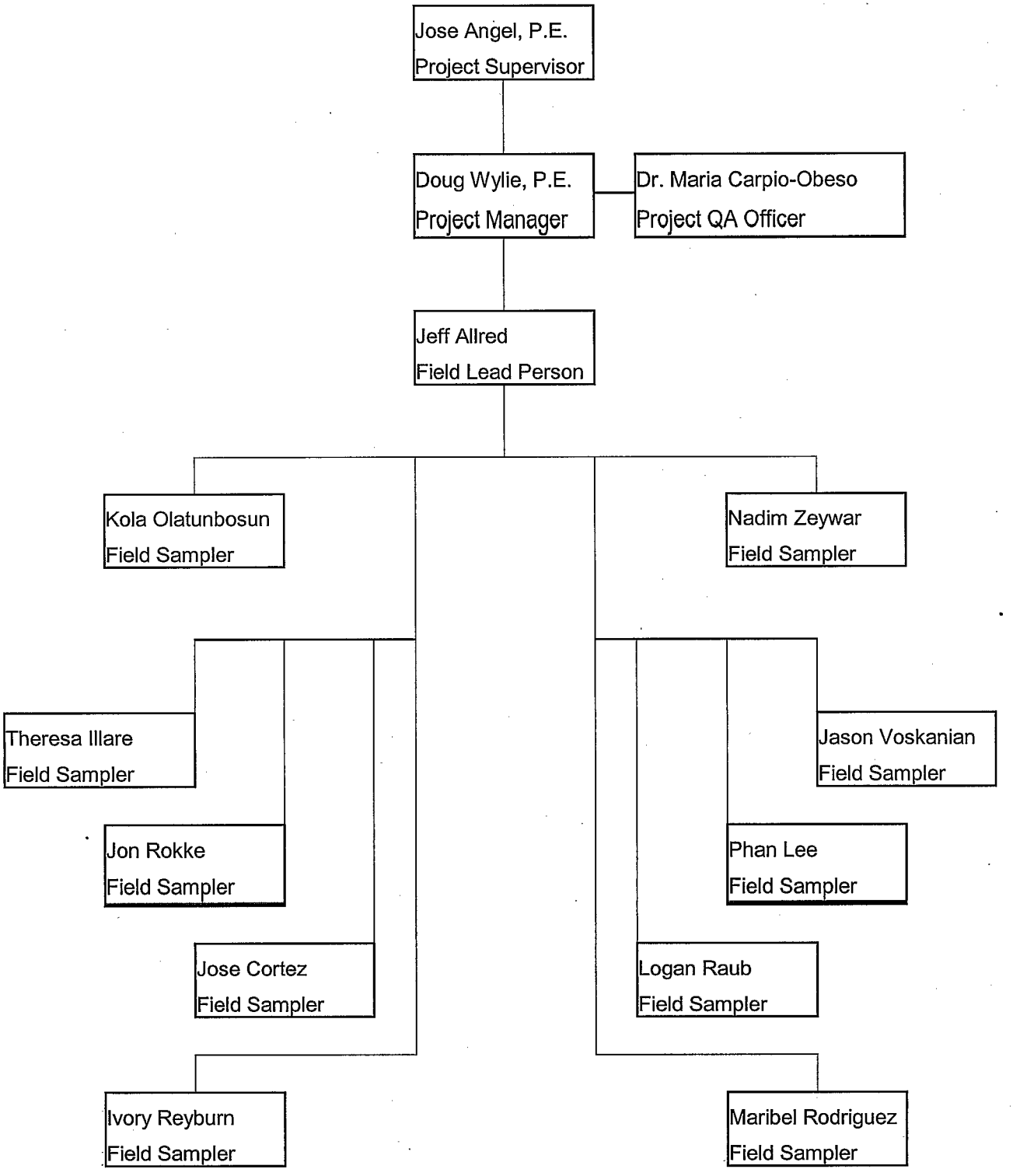
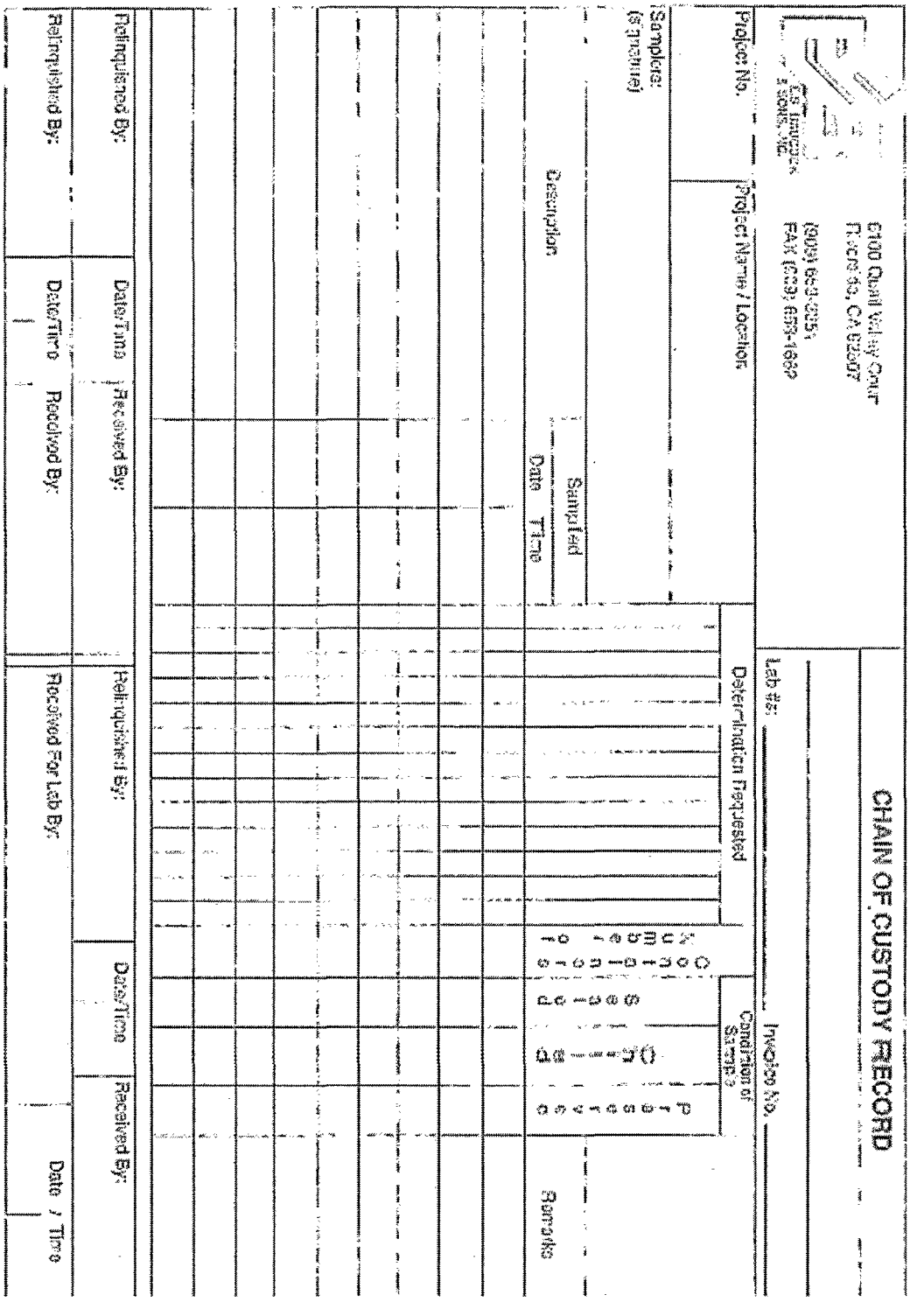
7 APPENDIX

APPENDIX I, PROJECT ORGANIZATION CHART



APPENDIX II: SAMPLE FORMS



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METHOD #: EPA 300.0, and 9056
SM 4110 B
CA DHS IC Rev 0

TITLE: The Determination of Inorganic Anions in Water by Ion Chromatography

ANALYTE:	CAS #
Chloride Cl	7782-50-5
Fluoride F	7782-41-4
Nitrate (NO_3)	
Nitrite (NO_2)	
Phosphate (PO_4)	
Sulfate (SO_4)	
Perchlorate (ClO_4)	
Para-Chlorobenzene Sulfonic Acid (PCBSA)	

INSTRUMENTATION:

IC: Dionex 500DX and 120DX (see sec. 6.2)
Data Handling: Pentium Processor with Peak-Net software on Windows NT platform.
Printer: HP Laser Jet 2100
Autosampler: Alcott Micromeritics 728

1.0 Scope and Application

1.1. This method covers the determination of the following inorganic anions.

		RL, mg/L
1.1.1.	Method A.	
1.1.1.1.	Fluoride	0.1
1.1.1.2.	Chloride	1
1.1.1.3.	Nitrate-N	0.2
1.1.1.4.	Nitrite-N	0.1
1.1.1.5.	Phosphate-P	0.05
1.1.1.6.	Sulfate	0.5
1.1.1.7.	PCBSA	10
1.1.2.	Method C	
1.1.2.1.	Perchlorate	0.004

Note: RL = Reporting Limit

1.2. The matrices applicable to each method are shown below:
1.2.1. Drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after extraction 2.3),
1.2.2. Drinking water and reagent waters.

1.2.3. Drinking water, groundwater and reagent waters.

1.3. The Single Laboratory Method Detection Limit (MDL, defined in section 13.1) for the above analytes is listed in Tables 1A through 1C. The MDL for a specific matrix may differ from those listed, depending upon the nature of the sample.

1.4. The working range for these analytes is as follows:

1.4.1.	Fluoride	0.1-5 mg/L
1.4.2.	Chloride	1-250 mg/L
1.4.3.	Nitrate-N	1-250 mg/L
1.4.4.	Nitrite-N	0.1-5.0 mg/L
1.4.5.	Phosphate-P	0.05 -5.0 mg/L
1.4.6.	Sulfate	1-400 mg/L
1.4.7.	Perchlorate	0.004-0.25 mg/L
1.4.8.	PCBSA	1-500 mg/L

1.5. This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatogram. Each analyst must demonstrate the ability to generate acceptable results with this method, using the procedure described in Section 10.2.

1.6. When this method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of fortified sample matrix covering the anions of interest. The fortification procedure is described in Section 11.9.

2. Summary of Method

2.1. An aliquot Sample (25 µL for Method A, and 740 µL for Method C)of sample is injected into an eluent stream and passed through a series of ion exchangers. The system is comprised of a guard column, separator column, and suppressor device. These separate the ions based on their affinity for a low capacity, strongly basic ion exchanger. They are then directed onto a strongly acidic cation exchanger where they are converted to their highly conductive acidic forms. The conductivity of these acid forms is measured. Identification is based on retention time. Quantitation is based on peak height or peak area.

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2.2. The main differences between Method A and C are the separator columns, guard columns and eluents. Sections 6 and 7 will elicit the differences.

2.3. In order to use this method for solids an extraction procedure must be performed (See Sec 11.10).

3. Definitions (see SOP Q15 for further definitions)

3.1. Stock standard solution - a concentrated solution containing a single certified standard that is a method analyte. Stock standard solutions are used to prepare calibration standards.

3.2. Calibration standards (CAL) - a solution of analytes prepared in the laboratory from stock standard solutions and diluted as needed and used to calibrate the instrument response with respect to analytic concentration.

3.3. Quality control sample (QCS) - a solution containing known concentrations of analytes, received quarterly from an outside vender (such as ERA). The analyzing laboratory uses this solution to demonstrate that it can obtain acceptable identifications and measurements with a method.

3.4. Performance evaluation sample (PE) - a solution of method analytes acquired from an outside source. A volume of the solution is added to a known volume of reagent water and analyzed with procedures used for samples. Analyte true values are unknown to the analyst.

3.5. Initial Calibration Check ICC (or Calibration Check standard) - a solution of analytes prepared in the laboratory by adding appropriate volumes of the stock standard solutions to reagent water used to evaluate the performance of the instrument system right after a calibration is performed. The low-level calibration standard is reinjected as well as the LCS to satisfy this requirement.

3.6. Laboratory duplicates (DUP) - two aliquots of the same sample that are treated exactly the same throughout laboratory analytical procedures. Analyses of laboratory duplicates indicate precision associated with laboratory procedures but not the sample collection, preservation, or storage procedures.

3.7. Laboratory fortified sample matrix (LFM) or Matrix Spike (MS) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM (or MS) is analyzed

exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM (or MS) corrected for background concentrations.

3.8. Laboratory Control Sample (LCS) referenced in the method for oxyhalides as the Continuing Calibration Check and in the method for perchlorates as the Laboratory Fortified Blank and Instrument Performance Check. An aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control.

3.9. Reporting Level Check A standard is run daily at the reporting limit to demonstrate that the laboratory is capable of making accurate and precise measurements at the required reporting detection limit (a). Once a year this standard is run seven times in a row as part of a detection limit study (b).

3.10. Method Blank (MB) An aliquot of D.I. water is analyzed at the beginning of a run and every ten samples.

4. Interferences

4.1. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems.

4.2. The water dip or negative peak that elutes near and can interfere with the fluoride peak can usually be eliminated by the addition of the equivalent of 1 mL of concentrated eluent (7.3 100X) to 100 mL of each standard and sample.

4.3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

4.4. Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems. Caution: filtration may remove perchlorate.

4.5. Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L this interference may not be significant, however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.6. The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

4.7. The quantitation of unretained peaks should be avoided, such as low molecular weight organic acids (formate, acetate, propionate, etc.) which are conductive and coelute with or near fluoride and would bias the fluoride quantitation in some drinking and most waste waters.

5. Safety

5.1. Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. No known carcinogenic materials are used in this method.

5.2. See SOP S01 , Concentrated Acids and Bases
SOP S02 – Compressed Gas Cylinder Handling
SOP S03 – Spill Control Policy

6. Apparatus and Materials

6.1. Balance - Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2. Ion chromatograph - Analytical system complete with ion chromatograph and all required accessories including syringes, analytical columns, compressed gasses and detectors.

6.2.1. Anion guard column: A protector of the separator column. If omitted from the system the retention times will be shorter. Usually packed with a substrate the same as that in the separator column.

6.2.2. Anion separator column:
6.2.2.1. Anion separator column (Method A):

6.2.2.1.1. AS-4A 4mm Dionex Column
6.2.2.1.2. AG4A 4mm Dionex Guard Column
6.2.2.2. Anion separator column (Method C):
6.2.2.2.1. AS-5 Dionex Column
6.2.2.2.2. AG-5 Dionex Guard Column
6.2.3. Anion suppressor column:
6.2.3.1. Anion suppressor column (Method A): Anion self-regenerating ASRS-11.
6.2.3.2. Anion suppressor column (Method C): Anion micromembrane suppressor AMMS-11
6.2.4. Detector – CD20 conductivity cell.

7. Reagents and Consumable Materials

7.1. Sample bottles: Glass or polyethylene of sufficient volume to allow replicate analyses of anions of interest.

7.2. Reagent water: Nanopure, free of the anions of interest. Water should contain particles no larger than 0.20 microns with a conductance of <0.1uS/cm.

7.3. Eluent solution:
7.3.1. Method A: Dissolve 0.571 g sodium bicarbonate ($NaHCO^3$) and 0.763 g of sodium carbonate (Na^2CO^3) in 1 liter of nanopure water (7.2) and dilute to 4 liters.
7.3.2. Method C: Add 19.2 mL of 50% NaOH and 0.4765 g of 4-cyanophenol to 1 liter of nanopure water (degassed by Nanopure process DHS-IC-Rev 0 7.2). Dilute to 2 liters.

7.4. Regeneration solution (MicroMembrane Suppressor) Concentrated Sulfuric Acid:
7.4.1 Method C: 3.9 mL per 4 liters nanopure water.

7.5. Stock standard solutions, 1000 mg/L (1 mg/mL); Stock standard solutions are purchased as certified solutions.

Note: Stability of standards: Stock standards (7.5) are stable for at least one month when stored at 4-C. The bottle expiration dates are used as a guideline. Dilute working standards should be prepared each time a calibration is performed. LCS solutions are prepared weekly except those that contain phosphate which are prepared fresh daily (c).

8. Sample Collection, Preservation and Storage

8.1. Samples should be collected in scrupulously clean glass or polyethylene bottles.

8.2. Sample preservation and holding times for the anions that can be determined by this method are as follows.

Analyte	Preservation	Holding Time
Chloride	None required	28 days
Fluoride	None required	28 days
Nitrate-N/Nitrite-N		
Unchlorinated	Cool to 4°C	48 hours
chlorinated	Cool to 4°C	14 days
combined	conc. H_2SO_4 pH < 2	28 days
o-Phosphate-P	Cool to 4°C	48 hours
Sulfate	Cool to 4°C	28 days
Perchlorate	Cool to 4°C	28 days
PCBSA	Cool to 4°C	28 days

8.3 The method of preservation and the holding time for samples analyzed by this method are determined by the anions of interest. In a given sample, the anion that requires the most preservation treatment and the shortest holding time will determine the preservation treatment. It is recommended that all samples be cooled to 4-C and held no longer than 28 days for Method A or Method C.

9. Calibration and Standardization
(See Standard Logs for recipes of all standards.)

9.1. Establish ion chromatographic operating parameters equivalent to those indicated in Table 1A or 1B.

9.2. For each analyte of interest, prepare calibration standards at a minimum of three concentration levels (five for method C) by adding accurately measured volumes of one or more stock standards (7.5) to a volumetric flask and diluting to volume with reagent water. The curve is forced through the 0 point. An acceptable curve has a $r^2 \geq 0.995$. A method blank is analyzed after the calibration to verify this point (d). If a sample analyte concentration exceeds the calibration range the sample may be diluted to fall within the range. If this is not possible then three new calibration

concentrations must be chosen, two of which must bracket the concentration of the sample analyte of interest. Each attenuation range of the instrument used to analyze a sample must be calibrated individually.

9.3. Using injections of 0.1 to 1.0 mL (determined by injection loop volume) of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte. During this procedure, retention times must be recorded.

9.4. The calibration curve must be verified on each working day, or whenever the anion eluent is changed, and after every 20 samples. If the response or retention time for any analyte varies from the expected values by more than the range indicated under CCV standards, the test must be repeated, using fresh calibration standards. If the results are still out of range, a new calibration curve must be prepared for that analyte.

ICV Standards

	Analyte	Conc.	Acceptance Range %
Method A:			
	Cl	150ppm	90-110
	NO_3	111ppm	90-110
	SO_4	250ppm	90-110
	F	2ppm	90-110
	NO_2	2ppm	90-110
	PO_4	2ppm	90-110
Method C:	ClO_4	25ppb	90-110

CCV Standards

	Analyte	Conc. Mid	Conc. High	Acceptance Range %
Method A:				
	Cl	10	150ppm	90-110
	NO_3	44.3	111ppm	90-110
	SO_4	30	250ppm	90-110
	F	0.5	2.0ppm	90-110
	NO_2	0.5	2.0ppm	90-110

	PO_4	0.5	2.0ppm	90-110
Method C:	ClO_4	125	250ppb	90-110

Calibration Standards

Method A:

Std #1				Std #4			
Cl	1ppm	F	0.05ppm	Cl	100ppm	F	1.0ppm
NO_3	1ppm	NO_2	0.05 ppm	NO_3	150ppm	NO_2	1.0ppm
SO_4	1ppm	PO_4	0.05 ppm	SO_4	150ppm	PO_4	1.0ppm

Std #2				Std #5			
Cl	10ppm	F	0.1ppm	Cl	200ppm	F	2.0ppm
NO_3	25ppm	NO_2	0.1 ppm	NO_3	200ppm	NO_2	2.0ppm
SO_4	30ppm	PO_4	0.1 ppm	SO_4	350ppm	PO_4	2.0ppm

Std #3				Std #6			
Cl	20ppm	F	0.5ppm	Cl	250ppm	F	5.0ppm
NO_3	50ppm	NO_2	0.5 ppm	NO_3	250ppm	NO_2	5.0ppm
SO_4	60ppm	PO_4	0.5 ppm	SO_4	400ppm	PO_4	5.0ppm

PCBSA Std #1 1ppm, Std #2 5ppm, Std #3 10ppm
Std #4 50ppm, Std #5 100ppm

Method C:

ClO_4: Std #1 4ppb, Std #2 10ppb, Std #3 50ppb
Std #4 100ppb, Std #5 250ppb

Lab Controls

Method A:

Acceptance range 90% - 110%

Cl	High	150 ppm	Low	10ppm
NO_3	High	111 ppm	Low	44.3ppm
SO_4	High	250 ppm	Low	30ppm
F	High	2.0 ppm	Low	0.5ppm
NO_2	High	2.0 ppm	Low	0.5ppm
PO_4	High	2.0 ppm	Low	0.5ppm

PCBSA 25ppm Acceptance range 60% - 120%

Method C:

Acceptance range 80% - 120% (e)
ClO4 25ppb

Matrix Spikes

Method A: No spikes analyzed (f). Duplicates are analyzed instead since these analytes are rarely none detected.

Method C:

Acceptance range: waters 80% - 120% max RPD 20
Soils 75% - 125% max RPD 35

ClO4 12.5ppb X any prep or dilution factor

10. Quality Control

10.1. Our laboratory has a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability (10.2) and the analysis of control samples as a continuing check on performance. The laboratory maintains performance records to define and document the quality of data that are generated.

10.1.1. In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in Section 10.2.

10.1.2. 5 to 10% of all samples are run in duplicate.

10.2. Before performing any analyses, the analyst demonstrates the ability to generate acceptable accuracy and precision with this method using a laboratory performance standard. Each analyst will analyze four replicates of a standard that is ten times their most recently proven MDL. Method C perchlorate requires four replicates at 25ppb. The acceptance criteria for this study is 90 – 110% recovery for water matrices and 80 – 120% recovery for solid matrices (g).

10.3. The laboratory develops and maintains accuracy statements of laboratory performance for each matrix being analyzed by the laboratory

10.4. Before processing any samples, the analyst demonstrates through the analysis of an aliquot of D.I. water (MB) that all glassware and reagent interferences are under control. Each time there is a change in reagents, the MB is monitored for the appearance of negative peaks as a safeguard against laboratory contamination (h).

10.5. When doubt exists over the identification of a peak in the chromatogram, confirmatory techniques such as sample dilution and fortification must be used.

10.6. Quality control check samples are analyzed concurrently with those performance evaluation sample studies required to maintain state certification.

10.7. For Method C (i), the linear calibration range is verified every 6 months or whenever a significant change in the instrument response is observed.

10.8. In order to verify that standards have been prepared correctly a LCS is performed using a standard of known concentration from an independent source. This laboratory control sample containing each analyte of concern is analyzed with each batch of samples processed. If more than 20 samples are run in a batch analyze one LCS for every 20 samples (10 for drinking water). Evaluate the accuracy by comparing to laboratory acceptance criteria. If acceptable data cannot be obtained, locate the problem and correct it. If during the course of a run a LCS is out of range, if possible it is rerun on the spot. If this is not possible the analyst may reevaluate the data based on peak height rather than peak area. If the data still does not fall within the acceptance criteria, the analyst may choose to use the six point calibration curve (for method A) to interpret the data rather than the three point lower level curve. If all the LCS' are in range under these conditions, the data is accepted. Otherwise a fresh calibration is performed and all samples are rerun starting from the last acceptable LCS.

11. Procedure

11.1. Set-up:

11.1.1. Prepare Eluant. Turn He valve to 5psi for method A and 30 psi for Method C. Check that the He line is connected to the eluant bottle. Set pump rate as per table 1.

11.1.2. On peaknet program – click on run icon. Under file click on load method. Method A – anion 300; Method C – a-clo4.met.
11.1.3. Wait for conductivity and pressure to stabilize.

11.2. Standardization and Calibration:
11.2.1. Using a clean syringe, fill one vial with the Method Blank.
11.2.1.1. Place vial in position #1 of autosampler.
11.2.1.2. Press <START> enter.
11.2.1.3. Init V <1> enter.
11.2.1.4. Rinse <0> enter.
11.2.1.5. Last V <1> enter.

11.2.2. Using a clean syringe, fill one vial with an initial calibration verification standard.
11.2.2.1. Place vial in position #2 of autosampler.
11.2.2.2. Press <START> enter.
11.2.2.3. Init V <1> enter.
11.2.2.4. Rinse <0> enter.
11.2.2.5. Last V <2> enter.
11.2.3. The initial calibration verification standard should read within the established control limits. If it does not, reinject it, if it still does not work, recalibrate.
11.2.3.1. Load calibration standards on the autosampler
11.2.3.2. Inject six calibration standards.
11.2.4. Check an initial calibration verification standard again.

11.3. Analysis:
11.3.1. Fill vials with sample, filtering (for Method A) through a 0.2 μm disc filter. For methods B or C, samples containing suspended material may be centrifuged or decanted.
11.3.2. Start the autosampler on vial 1 through 64.
11.3.2.1. Press <START> enter.
11.3.2.2. Init V <1> enter.
11.3.2.3. Rinse <0> enter.
11.3.2.4. Last V < # of last vial> enter.
Note: for method C, 2 vials per sample are loaded onto the autosampler.
11.3.3. Run continuing calibration verification standards every 10 samples. Run a lab control, method blank, and duplicate every 20 samples. Run matrix spikes every 20 samples for method B and C. Run a check standard at the end.
11.3.4. If a sample is above the high standard, dilute with D.I. water, according to the thickness and height of the peak. Make sure the peaknet software is calculating appropriately by observing peak heights and retention times.

11.4. Shutdown
11.4.1. Under Run – load stop method.
11.4.2. Turn pressure valve to 0 psi.

Note: Tables 1A and 1B summarize the recommended operating conditions for the ion chromatograph. Included in this table are estimated retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the requirements of Section 10.2 are met.

11.5. Check system calibration daily and, if required, recalibrate as described in Section 9.

11.6. Load and inject a fixed amount of well mixed sample. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.

11.7. The computer software comes with default retention time window widths. This is used to make identifications unless experience shows that the window requires adjustment (j). The experience of the analyst weighs heavily in the interpretation of chromatograms.

11.8. If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

11.9. If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

Note: Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases this peak migration may produce poor resolution or identification.

11.10. The following extraction should be used for solid materials. Add an amount of reagent water equal to ten times the weight of dry solid material taken as a sample. This mixture is agitated for sixty minutes by shaking intermittently. Filter the resulting slurry before injecting using a 0.45 micron membrane type filter. With the exception of method C, this can be the type that attaches directly to the end of the syringe. Two samples per batch are spiked prior to extraction. These spikes are used to demonstrate that good recovery and identification of peaks is obtained with the users matrix.

12. Calculation

12.1. Prepare separate calibration curves for each anion of interest by plotting peak size in area, or peak height units of standards against concentration values. The system will then compute sample concentration by comparing sample peak response with the standard curve.

12.2. Report results in mg/L.

12.3. Report:
NO_2- as N
NO_3- as N or as NO_3 if desired by the client
$H(PO_4)_2$- as P

13. Precision and Accuracy - Method Detection Limit

13.1. The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations listed in Table 1A and 1B were obtained using reagent waters.

14. Calculations associated with this method:

14.1. Total Anions (TA)
mequiv. of OH + CO3 + HCO3 + SO4 + Cl + NO3 = TA

14.2. Electrochemical Balance (ECB)
Total Cations (TC) – Total Anions (TA)

14.3. Total Dissolved Solids by Summation (TDSSUM)
mg/L of 0.6(Total Alkalinity) + Na + K + Ca + Mg + SO4 + Cl + NO3 + F + SiO3 = TDSSUM

Table 1A. Chromatographic Conditions and Detection Limits in Reagent Water (Method A)

Analyte	Peak #	MDL (mg/L)
Fluoride	1	0.01
Chloride	2	0.792
Bromide	4	0.0015
Nitrate-N	5	0.0115
o-Phosphate-P	6	0.003
Sulfate	7	0.028
PCBSA	8	<1 (EDL)

Standard Conditions:

Unit:	DX 120
Columns:	as specified in 6.2.2.1
Detector:	as specified in 6.2.4
Pump Rate:	2.0 mL/min.
Eluent:	as specified in 7.3.1
Sample Loop:	25 uL

Table 1B. Chromatographic Conditions and Detection Limits In Reagent Water (Method C)

Analyte	MDL (mg/L)
Perchlorate	0.0018

Standard Conditions:

Unit:	DX500
Column:	as specified in 6.2.2.3
Detector:	as specified in 6.2.4
Pump Rate:	1.0 mL/min.
Eluent:	as specified in 7.3
Sample Loop:	740 uL

15.0 Corrective Action For Out of Control Or Unacceptable Data:

See SOP Q06 – Corrective Action

16.0 Pollution Prevention and Waste Management:

See SOP S05 – Neutralization Procedure for Acid and Alkaline Wastes
SOP S07 – Pollution Prevention

Method Variations
(a). Low Level Check Frequency EPA Method 300.0 section 10.8.
(b). Low Level Check Duplicates EPA Method 300.0 section 10.9.
(c). Stability of Standards - EPA Method 300.0 section 7.5.

(d). Blank in Calibration – EPA Method 300.0 section 9.2. and DHS-IC-Rev 0 section 10.2.
(e) LCS Acceptance Limit - California Department of Health Services IC Rev 0, section 9.3.2, 9.3.3.
(f). Laboratory Fortified Sample Matrix required for each method – EPA Method 300.0 revision 2.1, section 9.4.1.
(g) .Demonstration of Capability EPA Method 300.0 section 10.2.
(h). Reagent Water Monitoring EPA Method 300.0 section 10.5.
(i). Proof of Linear Calibration Range required for each method - EPA Method 300.0 revision 2.1, section 9.2.2.
(j). Retention Time Window – EPA Method 300.0 section 11.4.

References:

EPA SW846 method 9056

EPA Methods for the Determination of Inorganic Substances in Environmental Samples, Method 300.

California Department of Health Services: IC Rev 0

Approved by Susan K Thomas 8/25/00

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METHOD #: EPA 350.1, SM 4500-NH_3 H

TITLE: 153 Nitrogen, Ammonia (Colorimetric, Automated Phenate)

ANALYTE: Ammonia Nitrogen

1.0 Scope and Application

1.1 This method covers the determination of ammonia in drinking, surface, and saline waters, domestic and industrial wastes in the range of 0.1 to 5.0 mg/L NH3 as N. This range is for photometric measurements made at 630nm in a 10 mm tubular flow cell. Higher concentrations can be determined by sample dilution.

2.0 Summary of Method

2.1 Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside.

3.0 Sample Handling and Preservation

3.1 Preservation by addition of conc. H_2SO_4 to a pH < 2 and refrigeration at 4-C.

3.2 Safety: Safety glasses and gloves should be worn when dealing with acids and bases.

4.0 Interferences

4.1 Calcium and magnesium ions may be present in concentrations sufficient to cause precipitation problems during analysis. A 5% EDTA solution is used to prevent the precipitation of calcium and magnesium ions from river water and industrial waste. For sea water a sodium potassium tartrate solution may be used.

4.2 Sample turbidity and color may interfere with this method. Turbidity must be removed by filtration prior to analysis. Sample color that absorbs in the photometric range used will also interfere. Sample is diluted if necessary

4.3 Marked variation in acidity and alkalinity are eliminated by sample preservation with H_2SO_4. The pH is then checked to ensure that it is <2. (a) Due to the reducing nature of this environment, residual chlorine is not expected to be a problem. (b) The sample is neutralized prior to analysis by the addition of the first reagent which is a NaOH buffer. (c)

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5.0 Apparatus

5.1 Test tube rack from Lachat.

5.2 13 x 100 mm disposable culture tubes.

5.3 Lachat Quikchem Analyzer

5.4 Whatman 2 and Whatman 4 (11.0 cm) filter paper or Gelman 0.45 micron disk filters.

5.5 100 ml beakers.

5.6 1 ml, 2 ml, 5 ml, and 10 ml pipets.

5.7 25 ml, 50 ml, and 100 ml graduated cylinders.

5.8 Helium Gas (technical grade).

5.9 Digestion hot plates

6.0 Reagents (d)

6.1 Nanopure water

6.2 Carrier or preserved water: 2ml of Sulfuric acid dilute to 1 gallon with Nanopure. Degas with Helium just prior to analysis.

6.3 Sodium phenolate: Using a 1 liter Erlenmeyer flask, 68ml of 88% phenol in 500 ml. of Nanopure water. In small increments, cautiously add with agitation, 32 g of NaOH. Periodically cool flask under water faucet. When cool, dilute to 1 liter with Nanopure water.

6.4 Sodium hypochlorite solution: Dilute 250 ml. of a bleach solution containing 5.25% NaOCl (such as "Clorox") to 500 ml with Nanopure water. Available chlorine level should approximate 2 to 3%. Since "Clorox" is a proprietary product, its formulation is subject to change. The analyst must remain alert to detecting any variation in this product significant to its use in this procedure. Due to the instability of this product, storage over an extended period should be avoided.

6.5 Buffer: Disodium ethylenediamine-tetraacetate (EDTA) (5%): Dissolve 50 g of EDTA (disodium salt) and 9g of NaOH in 1 liter of Nanopure water. Degas with Helium just prior to analysis.

6.6 Sodium nitroprusside (0.05%): Dissolve 3.5 g of sodium nitroprusside in 1 liter of Nanopure water.

7.0 Standards:

7.1 Lab Control Sample (LCS) and Matrix Spikes (MS/MSD):

7.1.1 Stock Solution: EM 1000 mg/L NH_3 Standard.

7.1.2 LCS: Dilute 1 ml of stock to 1000 ml in a volumetric flask with preserved water (7.3). The concentration is 1 mg/L NH_3 or 0.78 mg/L NH_3-N

7.1.3 Acceptability: The result of the LCS analysis is compared to statistically generated acceptance ranges. If the analysis does not fall within the acceptance range,(85%-115%) the analysis is stopped until the cause is determined and the LCS is within the acceptance range.

7.2 Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

7.2.1 Spike solution: Use a 1:1 dilution of a sample and LCS. Mix well.
7.2.2 Acceptability: 70%-130%, RPD maximum 20

7.3 Method Blank

7.3.2 Use carrier from section 6.2
7.3.3 Acceptability: MB must read below RL of 0.1mg/L

Note: Since the intensity of the color used to quantify the concentration is pH dependent, the acid concentration of the carrier and the standard ammonia solutions should approximate that of the samples.

7.4 Calibration Standard:
7.4.1 Stock Ammonia Standard:
7.4.1.1 Dehydrate Ammonium Chloride (NH_4Cl) in a 105°C oven.
7.4.1.2 Allow to cool in a dessicator. Weigh out 3.819 g NH_4Cl.
7.4.1.3 Dilute to 1 liter with nanopure water in a volumetric flask.
7.4.1.4 Pour the solution into a 1 liter amber bottle. Keep out of sunlight.
7.4.2 Use the stock NH_3-N standard for the calibration standards (1000ppm).

7.4.3 Dilute 5 ml of stock standard to 1000 ml with preserved water. This will be the 5.0 mg/L working standard and the intermediate standard.

7.4.4 Dilute from the intermediate standard for the other working standards as follows:

7.4.4.1 2.5 mg/L standard: 50 ml of 5.0 mg/L diluted to 100 mL with preserved water.

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7.4.4.2 1.0 mg/L standard: 20 mL of 5.0 mg/L diluted to 100 mL with preserved water.
7.4.4.3 0.2 mg/L standard: 4 mL of 5.0 mg/L diluted to 100 mL with preserved water.
7.4.4.4 0.05 mg/L standard: 1 mL of 5.0 mg/L diluted to 100 mL with preserved water

8.0 Procedure:

8.1 Preserve samples with H_2SO_4 to a pH of <2.

8.2 Rinse all glassware with 1:1 HCl.

8.3 Use the following volumes based on sample matrix:

8.3.1 Industrial or influent Wastewater – 2-5mL.

8.3.2 Effluent Wastewater – 25-50 mL.

8.3.3 Well water - 50 mL

8.3.4 Solid - Make a 1:10 water extract, extract and swirl periodically for one hour.

8.4 Dilute all samples to a final volume of 50 mL. If less than 5 ml of sample is used, dilute with carrier otherwise Nanopure water may be used.

Note: Filter all samples. Distillation is not required since compatability data on representative samples is being generated to show that this step is not necessary however manual distillation will be required to resolve any controversies (e).

8.5 Pour samples into test tubes in the test tube rack. Analyze or the leachat.

8.6 If diluted samples read below 0.1 mg/L, re-analyze using more sample and diluting to a final volume of 50 ml.

8.7 If any sample reads above 5.0 mg/L, re-analyze using less sample.

8.8 Allows both colorimeter and recorder to warm up for 30 minutes. Obtain a stable baseline with all reagents, feeding Nanopure water through sample line.

8.9 Arrange ammonia standards in sampler. Complete loading of sampler tray with unknown samples.

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8.10 See Lachat SOP I41 for general operating instructions.

8.11 Choose method: NH4PHE

8.12 After system has stabilized with water only running through the lines and the heater temperature has reached at least 58° C, put the reagent tube into the carrier bottle.

8.13 Wait until carrier has reached the end of the board before putting the buffer tube into the reagent bottle.

8.14 Continue on in this manner, adding all of the reagents in the order in which they are numbered.

8.15 Once the baseline is stable and the temperature of the heater has returned to 58-62°C, calibration may begin.

8.16 When an acceptable calibration has been performed, submit the tray of samples.

9.0 Calculations

9.1 Prepare appropriate standard curve derived from processing ammonia standards through manifold. Compute concentration of samples by comparing sample peak areas (f) with standard curve.

9.2 Apply dilution factors to samples where less than 50ml was anlalyzed.

9.3 The reporting limit is 0.1mg/L.

9.4 Report 2 significant figures.

9.5 Inorganic Nitrogen = $NH_3N + NO_3N + NO_2N$

10.0 Definitions: See SOP Q15 – SOP Definitions

11.0 Safety: The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. A reference file of material data handling sheets is made available to all personnel involved in the chemical analysis.

See SOP S01 – Concentrated Acids and Bases
SOP S02 – Compressed Gas Cylinder Handling
SOP S03 – Spill Control Policy

12.0 Corrective Action For Out of Control Or Unacceptable Data:
See SOP Q06 - Corrective Action

13.0 Pollution Prevention and Waste Management:
See SOP S05 – Neutralization Procedure for Acid and Alkaline Wastes
SOP S06 – Disposal of Chlorinated Solvents
SOP S07 – Pollution Prevention

Method Variations

(a) Elimination of Marked Acidity or Alkalinity – Standard Methods 18th Edition 4500-NH_3 H 1b.
(b) Chlorine Pretreatment – Standard Methods 18th Edition 4500-NH_3 A 2
(c) Sample Neutralization - Standard Methods 18th Edition 4500-NH_3 A 3
(d) Reagent Recipes –Lachat Quikchem Methods NH3 Phenolate Method 10-107-06-1-B @ 3/13/99 and Standard Methods 20th Edition 4500-NH_3
(e) Distillation – 40 Code of Federal Regulations part 136.
(f) Quantification Using Peak Area– Standard Methods 18th Edition 4500-NH_3 H 5

References:

Standard Methods for the Examination of Water and Wastewater APHA, AWWA, WPCF 18th Edition.

Lachat Quikchem Methods 10-107-06-3-F @ 3/31/91

EPA Method 350.1 Methods for the Chemical Analysis of Waters and Wastes.

Approved by Jessum K. Thomas 7/10/00

8 ATTACHMENTS

8.1 ATTACHMENT A: CLEANING OF EQUIPMENT FOR WATER SAMPLING

8.2 ATTACHMENT B: STANDARD OPERATION PROCEDURES

QUALITY ASSURANCE PROJECT PLAN

FOR SAMPLING
SUSPENDED SEDIMENT CONCENTRATIONS
IN THE 31 IMPERIAL VALLEY DRAINS
FLOWING INTO THE SALTON SEA

March 2002

Prepared by and for
California Regional Water Quality Control Board Staff
Colorado River Basin Region

Quality Assurance Project Plan

for Sampling Suspended Sediment Concentrations
in the 31 Imperial Valley Drains Flowing into the Salton Sea

APPROVALS:

California Regional Water Quality Control Board

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1. PROJECT MANAGEMENT

1.1 INTRODUCTION

This Quality Assurance Project Plan (QAPP) describes the monitoring activities to characterize the suspended sediment concentrations in the 31 Imperial Valley Drains flowing directly into the Salton Sea, and the quality assurance (QA) and quality control (QC) procedures associated with these monitoring activities. This QAPP follows the format that the United States Environmental Protection Agency (USEPA) has established in its *Requirements for Quality Assurance Project Plans, EPA QA/R-5, 2001.* Further, it also complies with the QA/QC requirements specified in the *State Water Resources Control Board Quality Assurance Program Plan, 1994.*

The Quality Assurance Officer is responsible for ensuring that the QAPP commitments are implemented and followed to meet the objectives of this project. The Quality Assurance Officer will be independent from the monitoring crew generating the data for this project and has authority to change and modify this QAPP to achieve the objectives of the project.

1.2 DISTRIBUTION LIST

The following individuals will receive copies of the approved QAPP and subsequent revisions:

- Jose Angel, P.E., Division Chief*
- Doug Wylie, P.E., Project Manager*
- Joan Stormo, Senior Engineering Geologist*
- Maria de la Paz Carpio-Obeso, Ph.D., Quality Assurance Officer*
- Jeff Allred, WRCE, Field Lead Person
- Phan Le, WRCE, Field Sampler
- Jose Cortez, WRCE, Field Sampler

Also, copies of the approved QAPP and subsequent revisions will be placed in the following RWQCB files:

- TMDL QAQC (TMDL Section Quality Assurance File)
- TMDL SILT IVD (TMDL Silt Imperial Valley Drains)

* indicates approving authority

1.3 PROJECT/TASK ORGANIZATION

Specific Project responsibilities of the Regional Board staff are outlined below. A project organization chart is provided as Attachment 1.

Jose Angel, Project Supervisor, Supervising WRC Engineer, 760-776-8932

- Review and approve the QAPP and subsequent revisions.
- The primary decision maker, responsible for oversight of the project at Regional Board level.

Doug Wylie, Project Manager, Senior WRC Engineer, 760-346-6585

- Review and approve the QAPP and subsequent revisions.
- Ensure that the QAPP is implemented and followed to meet the project objectives.
- Review reports and ensure plans are implemented according to schedule.

- Conduct Health and Safety briefing for sampling team prior to each sampling event.
- Coordinate field and laboratory activities.
- Conduct project activities in accordance with the QAPP.
- Report to the Quality Assurance Officer and management regarding the project status. Prepare interim and final reports for the Quality Assurance Officer and management.

Joan Stormo, Senior Engineering Geologist, Basin Planning Unit Chief, 760-776-8982
- Review and approve the QAPP and subsequent revisions.
- Ensure that the QAPP is implemented and followed to meet the project objectives.

Maria de la Paz Carpio-Obeso, Quality Assurance Officer, Environmental Scientist, 760-674-0803
- Review and approve the QAPP and subsequent revisions.
- Ensure that the QAPP is implemented and followed to meet the project objectives.
- Review reports and ensure plans are implemented according to schedule.
- Responsible for operation of the Regional Board Laboratory.
- Responsible for coordinating lab quality assurance activities.

Jeff Allred, Field Lead Person, WRC Engineer, 760-776-8946
- Responsible for maintaining and calibrating instruments in the field.
- Responsible for coordinating filed activities and ensuring they are consistent with QAPP.
- Assist with monitoring activities as required.
- Prepare a narrative report on sampling event.
- Responsible for delivery of samples to the laboratory.
- Responsible for decontamination of sampling equipment used in field.

Nadim Zeywar, Field Sampler, Environmental Scientist, 760-776-8971
- Assist with monitoring activities as required.

Jason Voskanian, Field Sampler, SETT, 760-776-8930
- Assist with sampling activities as required.
- Responsible for delivery of samples to the laboratory.

Phan Le, WRC Engineer, 760-346-7491
- Assist with sampling activities as required.
- Responsible for calibration of metering equipment prior to sampling event.
- Responsible for assisting Lab Director with water quality analysis.

Jon Rokke, Field Sampler, WRCE, 760-776-8959.
- Assist with sampling activities as required.
- Responsible for delivery of samples to the laboratory.

Kola Olatunbosun, Field Sampler, WRCE, 760-776-8986
- Assist with sampling activities as required.
- Responsible for delivery of samples to the laboratory.

Theresa Illare, Field Sampler, Environmental Scientist, 760-776-8971
- Assist with sampling activities as required.
- Responsible for delivery of samples to the laboratory.

Maribel Rodriguez, Field Sampler, SETT, 760-776-8971
- Assist with sampling activities as required.

Jose Cortez, Field Sampler, WRC Engineer, 760-674-8142

- Prepare the Quality Assurance Project Plan (QAPP) and revisions.
- Responsible for processing data, maintaining the project database, and validating the field data.
- Assist with sampling activities as required.

1.4 PROBLEM DEFINITION/BACKGROUND

Pursuant to Section 303(d) of the Clean Water Act (CWA), the Colorado River Basin Regional Water Quality Control Board (Regional Board) is developing Siltation/Sedimentation Total Maximum Daily Loads (TMDLs) for the Imperial Valley Drains that discharge directly into the Salton Sea. The TMDLs are being developed because the list of impaired waterbodies for the Region (also known as the Region's 303(d) list) identifies the Drains as water quality limited, in part, because sediment concentrations violate the water quality standards (WQS) established by the Regional Board to protect the beneficial uses of the Drains. TMDL development requires a Source Analysis that identifies the sources of the pollutant of concern and quantifies their relative contributions. The data collection activities outlined in this QAPP are being undertaken to better characterize the sources of sediment to the Drains, and the existing sediment concentrations within them.

The Imperial Valley Drains (Drains) discharging directly into the Salton Sea are generally located around the southern perimeter of the Salton Sea The Drains are dominated by agricultural return flows from Imperial Valley. These agricultural return flows consist of surface run-off (tailwater) and subsurface drainage (tilewater), which mix with groundwater seepage. The Drains are operated by the Imperial Irrigation District (IID). Tailwater is believed to be the main source of sediment, and sediment is present in the Drains at concentrations that violate the WQS the Regional Board has established for surface waters within the watershed. Other sources and activities that contribute to the current sediment load of the Drains include dredging of the Drains, channel scouring in areas of high velocity flow, and, to a lesser degree, stormwater runoff and wind deposition. The Drains discharge a combined average flow of about 100 cfs (75,000 AFY) into the Salton Sea.

1.5 PROJECT/TASK DESCRIPTION

The overall objective of this project is to obtain valid data of known and documented quality, which can be utilized in the Source Analysis of the Imperial Valley Drains Sediment TMDLs, and in determining "baseline" sediment concentrations, from which future changes in sediment concentrations can be evaluated. Specific objectives targeted towards meeting this overall objective are to:

1. Collect representative water samples for total suspended solids (TSS) and turbidity analyses from the Drains at the sampling locations identified in Table No. 2, below;

2. Conduct field measurements of dissolved oxygen (DO), pH, temperature, turbidity, and electrical conductivity (EC) conditions in the Drains;

3. Evaluate the water quality data acquired through this Project and compare them with existing Regional Board data and data collected by IID through its Drain Quality Improvement Program;

4. Collect representative TSS and turbidity data for use, to the degree feasible, in developing an empirical relationship between TSS and turbidity for water in the IV Drains;

The initial phase of this Project consists of twelve monthly sampling events. The first event is scheduled for March, 2002. During the events, water samples will be collected and field measurements taken at the thirty-one (31) drains, which have been identified as discharging directly

into the Salton Sea. Future phases of this project may be undertaken, depending on data needs, as well as staff and funding availability, based on future revisions of this QAPP. The specific sampling locations are described in detail in Section 2.1.

1.6 DATA QUALITY OBJECTIVES AND CRITERIA

Valid data of known and documented quality is needed to meet the objectives of this project. Therefore, for the critical measurements of this project (TSS and turbidity), only data for which the data quality indicators show that the data quality objectives are being met will be considered valid. The specific data quality objectives of this project are:

- The analyses for TSS and turbidity must yield results that are of sufficient quality to be used in the development of the source analysis for the sediment TMDLs. Therefore, data obtained should be of sufficient quality to be utilized to determine the relative contributions of sediment from the Drains at the time of sampling.

- The analyses for TSS and turbidity must yield results that are of sufficient quality to be utilized, along with data from future sampling projects, in the determination of representative "baseline" suspended sediment concentrations in the Drains. Therefore, data collected must be of sufficient quality to determine the relative changes in suspended sediment concentrations in Drains over time, as land use practices and/or hydrological and/or climate conditions change.

- The analyses for TSS and turbidity will be compared to historic TSS and turbidity data for the Drains to assess the overall representativeness of the historic data for the present situation. Therefore, the detection limits that are proposed herein are, at a minimum, equal to the detection limits for TSS and turbidity used in the historic data.

- The data collected in this project should be of sufficient quality to be utilized to the extent technically feasible, along with data from future sampling projects, in the determination of a TSS/Turbidity relationship for the Drains emptying into the Salton Sea.

1.6.1 DATA QUALITY INDICATORS (ACCEPTANCE CRITERIA)

The following data quality indicators will be utilized to assess whether data generated is useable and meets the data quality objectives stated above:

1.6.1.1 Precision

Precision of the data generated will be assessed as the relative percent difference (RPD) for field duplicates and laboratory dilutions for the samples. The frequency for the field duplicates is discussed in section 2.5, below. All duplicates and dilutions should fall within a 25% RPD for TSS and a 35% RPD for turbidity, as described in Table 1, below in order for the data quality objectives to be met.

1.6.1.2 Accuracy

Accuracy will be determined using double blind spike samples for turbidity and TSS, field blanks, and equipment blanks. The frequency for the submittal of double blind spikes, field blanks, and equipment blanks is discussed in Section 2.5, below. For double blind spikes, the laboratory results

for both TSS and turbidity should be between 80 and 120 percent recovery of the true concentration of the spike, as described in Table 1, below, in order for data quality objectives to be met.

For field blanks and trip blanks, the average of all TSS measurement must be 15 mg/l or less, and the average of all turbidity measurements must be 15 NTU or less, in order to for this data quality objective to be met.

1.6.1.3 Completeness

To ensure completeness, 80 percent of the samples proposed in the design must be collected and analyzed. If less than this amount of samples is collected and analyzed, another sampling event will be required.

1.6.1.4 Comparability

Comparability will be addressed by using commonly accepted sampling and analytical techniques and by reporting data in commonly accepted units.

1.6.1.5 Representativeness

Representativeness will be assured by using a statistically significant number of samples with only one event and sampling at specific locations where a representative sample can be obtained, i.e. where flows are relatively well mixed and at least 100 feet downstream from the influence of potential sources of bias, such as direct tailwater discharges to the Drain being sampled.

Table No. 1, below, summarizes the precision, accuracy and completeness criteria.

Table 1: QA Objectives for Laboratory Data

Parameter	Matrix	Units	Precision (RPD)	Accuracy (% Recovery)	Completeness[1] (% Cmp)
TSS	Water	mg/L	25	80-120	80
Turbidity	Water	NTU	35	65-135	80

[1] Completeness criteria will not be applied to results from QC samples.

Where:

RPD = Relative Percent Difference = $\{ABS (D_1 - D_2)/[(D_1 + D_2)/2]\} \times 100$

D_1 = Results for sample 1

D_2 = Results for sample 2

ABS = Absolute value

% Recovery = Recovery of spike samples = S_s/S_e

S_s = Result of spiked sample analysis

S_e = Expected result of the spike sample analysis

$\%C_{mp} = 100x(V/n)$

V = Number of valid samples

n = Number of samples collected

1.7 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

The Project Manager will ensure that all of the field samplers have valid and current training for their field activities, as required by OSHA regulations. Currently, all sampling personnel identified in the Project/Task Organization section of this QAPP have completed the required OSHA training for the sampling activities described herein. There are no other specialized training/certification requirements needed to perform the Project's objectives.

1.8 DOCUMENTATION AND RECORDS

The Project Manager will establish and maintain a Project file for the purpose of filing and safeguarding sampling event data/records in accordance with the QAPP. The Field Lead Person, Quality Assurance Officer, and Unit Chiefs will ensure that all project data they receive/generate about the sampling events (e.g., field notes, chain of custody forms, lab analyses) is delivered to the Project Manager. The Project file shall be available for the review and inspection of the Quality Assurance Officer and accessible to the TMDL Development Unit Chief for TMDL development. The file will contain, but needs not be limited to, the following records:

- field logs/notes, quality control logs and calibration logs
- laboratory analytical reports
- preliminary data reports summarizing field activity and quality control for each sampling event
- data spreadsheets and databases
- miscellaneous correspondence related to the sampling events
- audit reports
- copies of the historic data (e.g., IID data) to be used for comparison purposes
- final report.

Field notes will be entered into bound field log notebooks with pre-numbered pages. Each page of the field logs and field data worksheets will be dated and signed by a member of the sampling team at each sampling station. At the time of sampling, the following information will be entered into the field log book:

- Observations about the weather and the sampling station.
- The latitude and longitude of the sampling station, as determined using a global positioning system (GPS) receiver.
- Identification codes, specific sampling point locations, and sampling methods for all samples taken. The instream YSI readings for temperature, DO, pH and EC.
- Sample codes and time and location of preparation for all quality control samples prepared in the field.
- Any deviations from the procedures of this QAPP.
- Any other noteworthy observations.

Quality control (QC) samples will be documented in a bound Quality Control Log with pre-numbered pages. The Quality Control Log will document the QC samples submitted to the laboratory and the results of the analysis of these QC samples. For each QC sample, the log will contain:

- The sample identification code.
- The supplier of the QC sample.
- The value reported by the supplier.
- The date of preparation and submission.
- The name and signature of the person submitting the QC sample.

- The laboratory performing the analysis.
- The analysis method.
- The reported value from the laboratory.

A YSI 6600 multiprobe water quality sonde will be used for field measurements of DO, pH, temperature, turbidity, and EC. Calibration of the YSI 6600 sonde will be documented in a bound calibration log with pre-numbered pages. The calibration log will contain:

- The date and time of calibration.
- The persons performing the calibration.
- The signature of one of the persons performing the calibration.
- All standard solutions used in calibration, including the source and date of preparation of the standard solution.
- The initial reading of the YSI when tested against each standard solution, and the temperature of each standard solution at the time of calibration.
- Any deviations from the QAPP
- Any difficulties or other relevant notes about the calibration.

Upon completion of the laboratory analysis of the samples from each sampling event, the laboratory will prepare and submit to the Project Manager a Laboratory Analytical Summary. The summary shall consist of analytical results and chain of custody forms.

A Preliminary Monthly Data Report will be produced by the Project Manager and filed with the Quality Assurance Officer within 7 to 10 days from the date the Project Manager receives all lab results for the monthly sampling event. This report will summarize the field activities and observations for the month; it will also include field measurements and the results of the laboratory analysis. This report will also include a quantitative analysis and discussion of the results of quality control activities, and what these results indicate about the quality of data generated in each sampling event. It may include recommendations for modification of this QAPP as appropriate.

The field logs, quality control log, and calibration log along with all additional documentation consisting of any laboratory records, and chain of custody forms will be stored in an organized manner by the Project Manager, and will be available upon request.

Once all of the sampling is completed for this project, a narrative report will be prepared by Project Manager for the Quality Assurance Officer and management. At a minimum, this report will discuss all the field activities, provide a qualitative and quantitative analysis of the data generated by the sampling activities, and any problems encountered and their solutions. Additionally, it will discuss any deviations from this QAPP, if any, as well as a discussion of the data quality.

2. DATA GENERATION AND ACQUISITON

2.1 SAMPLING PROCESS DESIGN

In order to meet the overall objectives stated in section 1.5 of this QAPP, this project was designed to estimate the suspended sediment concentration, as represented by total suspended solids (TSS) and turbidity, at the sampling stations in the IV Drains, and the contributions of suspended sediment to the Drains. Because accurate suspended sediment data is necessary for TMDL development, TSS and turbidity are considered critical measurements for this project, while the other baseline parameters to be measured, temperature, EC, pH and DO, are considered non-critical measurements. The sampling stations were selected to characterize the contribution of suspended sediments from Drains discharging driectly into the the Salton Sea. One sampling station has been establsihed for each Drain.

At each of the locations listed in Table 2, below, between one and four water samples will be taken (i.e. grab, grab duplicate, field spike, or field blank) and a YSI 6600 multi-parameter sonde will be used to take in-stream measurements of temperature, DO, EC and pH.

Table 2: Monitoring Stations

Sampling Location	Description[1]
ND1	Monitoring station for Niland Drain 1.
ND2	Monitoring station for Niland Drain 2.
ND3	Monitoring station for Niland Drain 3.
ND4	Monitoring station for Niland Drain 4.
ND5	Monitoring station for Niland Drain 5.
ZD	Monitoring station for Z Drain.
WD	Monitoring station for W Drain.
UD	Monitoring station for U Drain.
TD	Monitoring station T Drain.
SD	Monitoring station for S Drain.

[1] Located approximately 100 feet upstream of the outlet to the Salton Sea, unless the prescribed distance is inaccessible, as documented by field observations.

RD	Monitoring station for R Drain.
QD	Monitoring station for Q Drain.
PD	Monitoring station for P Drain.
OD	Monitoring station O Drain.
VD3	Monitoring station for Vail 3 Drain.
PUMD	Monitoring station for Pumice Drain.
VD5	Monitoring station for Vail 5 Drain.
VD5A	Monitoring station Vail 5A Drain.
VD6	Monitoring station for Vail 6 Drain.
VCD	Monitoring station for Vail Cut-Off Drain.
TRD12	Monitoring station for Trifolium 12 Drain.
TRD13	Monitoring station for Trifolium 13 Drain.
TRD14A	Monitoring station for Trifolium 14A Drain.
TRD1	Monitoring station for Trifolium 1 Drain.
TRSD	Monitoring station Trifolium Storm Drain.
TRD18	Monitoring station for Trifolium 18 Drain.
POED	Monitoring station for Poe Drain.
TRD19	Monitoring station for Trifolium 19 Drain.
TRD20	Monitoring station for Trifolium 20 Drain.
TRD22	Monitoring station for Trifolium 22 Drain.
TRD23	Monitoring station for Trifolium 23 Drain.

For all the sampling stations, there are three (3) sampling points (S1, S2, S3) distributed along the cross-sectional area of the Drain. The sampling points are to be spaced at approximately equal intervals from each other and from the edge of the drain (i.e., at a distance equal to w/4, where "w" is the top width of the cross-sectional area). Figure No. 1, below, illustrates this.

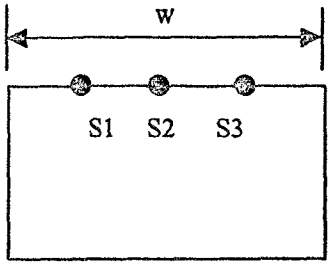

Figure 1: Sampling Points at Monitoring Stations

For the locations listed in Table 2, where the width of the drain is less than four feet and composite duplicates are required, the samples will be taken at the center point (S2) and composited into a single sample. A churn splitter will be used to make the duplicates. Where the width of the drain is greater than four feet, the samples will be taken at the three sampling points (S1, S2, S3) and composited into a single sample. A churn splitter will be used to make the duplicates.

For all sampling stations, readings of EC, DO, pH and temperature will be recorded at the center sampling point (S2) using the YSI 6600 sonde.

2.2 SAMPLING METHODS REQUIREMENTS

Sampling methods include the collection of grab samples, as well as the acquisition of readings for water quality parameters from the YSI water quality sonde.

Wherever possible, samples will be collected at the drop structure closest to the outlet of the drain where the water is thoroughly mixed. Otherwise the grab samples will be collected at approximately ½ foot below the water surface at the center sampling point (S2). Where hazardous conditions prevent midstream sampling, the grab sample will be collected at sampling location S1 or S3 and will be recorded in the field notebook. Grab samples will be collected using a swing sampler. For each sample collected, the sample bottle will be rinsed three times with native water before collection of the sample. The sample will then be placed into an ice chest packed with ice.

The YSI 6600 multi-parameter water quality sonde will be used to collect field measurements for the following parameters: DO, pH, temperature, and specific conductance (electrical conductivity) at the center point at each sampling location from about 1 foot below the water surface. When the readings have come to equilibrium, the values for these parameters will be manually recorded in the field notebook.

2.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

In general, sample-holding times will be adhered to, as prescribed by USEPA and 40 CFR 136. Specifically, the required preservation techniques and holding times for all of the constituents which the laboratory will be analyzing are listed in Table 3, below.

Table 3 : Required Containers, Preservatives, Techniques, and Holding Times

Constituent	Container	Preservation Technique	Holding Time
Turbidity	1-quart low density polyethylene bottle	Cool 4 °C	48 hours
Total Suspended Solids			7 days

Each sample container will be labeled with a unique sample identification code. All samples (including QC samples) for laboratory analyses will immediately be stored in an ice chest, and will remain in the custody of field samplers until the samples are delivered to the laboratory. The ice chest will contain sufficient ice to maintain the samples at a temperature below 4°C at all times until they are relinquished to the lab staff. All samples will be delivered with chain of custody forms. A sample chain of custody form to be used for this project is included in Attachment 2. Any violation of holding times or other sample handling and custody requirements will be documented in the quality control records and reported to the Project Manager and the Quality Assurance Officer. Any violations thereof will be taken into account when evaluating the data.

2.4 ANALYTICAL METHODS REQUIREMENTS

As prescribed by the State Water Resources Control Board's *"Quality Assurance Program Plan"*, each analytical laboratory used for sample analysis must have a written Quality Assurance Laboratory Manual describing the analytical method requirements. Water samples will be analyzed at the lab for TSS and turbidity, using USEPA approved methods as outlined in Table No. 4.

Table 4: Sampling Constituents and Methods

Constituent	USEPA Method	Reporting Limit	Units	Type of Sample
Turbidity	180.1	0.05	NTU	Depth-integrated, grab
Total Suspended Solids	160.2	4	mg/L	Depth-integrated, grab

2.5 QUALITY CONTROL REQUIREMENTS

In order to assess whether the data quality requirements of this project are being met, a number of quality control checks will be implemented. It is proposed that approximately 10 percent of all the samples analyzed be quality control (QC) samples. The calibration and maintenance of laboratory instruments and the general operation of the laboratories are subject to the requirements of the State Board Quality Assurance Program Plan and the Regional Board Quality Assurance Program for its Laboratory. All QC samples will be placed in an ice chest, and kept at 4 °C, for transport to the lab. Specifically:

- The Field Lead Person will prepare field duplicate samples during the sampling event. The field duplicate samples will be prepared from a grab sample of the water being sampled. A grab

sample will be collected as described in the section above and placed in a churn splitter where the duplicates will be made by keeping the sample water constantly mixed.

- One pair of field duplicate samples will be prepared for each day of sampling.
- Two double blind spike samples for turbidity and TSS will be prepared by an independent lab and submitted to the laboratory for analyses.

QC samples will be submitted to the lab along with the "real" surface water samples being submitted as blind spike samples. (i.e., the laboratory will not be informed in any way as to which samples are control samples and which samples are from the Drains).

Table 5: Quality Control Sample Requirements

Quality Control Samples	Number Of Samples/Event
Duplicate Samples (10%/Event, 2 for each parameter)	2
Field Blanks (1/day/event)	1
Spike Samples (10%/Event)	2

2.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, & MAINTENANCE REQUIREMENTS

All staff participating in the Project will be trained in the operation, calibration, and maintenance of the field instruments. The manufacturer's instruction manuals will be readily available for field personnel. The instruments will be maintained and calibrated in accordance with the manufacturer's instructions and recommendations. Prior to the collection of each sample, all equipment that comes into contact with the sample will be rinsed with distilled water.

2.7 INSTRUMENT CALIBRATION AND FREQUENCY

The YSI 6600 sonde will be calibrated in the laboratory prior to its initial deployment. It will then be tested in the field with known concentrations of pH, turbidity, and specific conductance. If necessary, the sonde will then be re-calibrated. The DO probe will be tested using tap water. Results of calibration measurements will be documented in the field log notebook and submitted to the Quality Assurance Officer. Table 6, below illustrates the YSI 6600 sonde specifications:

Table 6: Parameter Specifications for the YSI 6600 Multiprobe Sonde.

Parameter	Operating Range	Accuracy	Resolution	Calibration Standard
pH	0 to 14 units	± 0.2 units	0.01 units	3-pt, with pH buffered solutions
Temperature	– 5 to 50 °C	± 0.15 °C	0.01 °C	not required
DO	0 to 20 mg/L	± 0.2 mg/L	0.01 mg/L	saturated air
EC	0 to 100 mS/cm	± 1% of range	4 digits	KCl

2.8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES/CONSUMABLES

The Field Lead Person will ensure that sample bottles have no defects and have been prepared properly.

2.9 DATA ACQUISITION REQUIREMENTS (NON-DIRECT METHODS)

Only data collected from this project, historic data from the Board's Trend Monitoring Program, USGS water quality data, US Bureau of Reclamation data, and data from IID's DWQIP, which have already been approved, will be used.

2.10 DATA MANAGEMENT

Documentation and records will be kept as described in section 1.8 of this QAPP.

2.10.1 DATA STATISTICAL ANALYSIS

The Project Manager will prepare a Preliminary Monthly Data Report and submit a copy of the report to the Project Quality Assurance Officer within 7 to 10 days that the Project Manager has received all lab results for a sampling event. This report will summarize the field activities and observations; it will also include field measurements and the results of the laboratory analysis. This report will also include a quantitative analysis and discussion of the results of quality control activities, and what these results indicate about the quality of data generated in each sampling event.

The Project Manager will manage and analyze TSS and turbidity data using the Spreadsheet Excel software. Data will be entered into Excel in columns by drain and in rows by month. Descriptive Statistics (e.g. means, standard deviations, and coefficient of variation) will be computed for each column in part to ascertain the TSS loading of the drains and overall data trends. QC data will also be reviewed and entered into the spreadsheet by the Project Manager. As requested by the TMDL Development Unit Chief, the Project Manager will share data management records (e.g., spreadsheets) for TMDL development. Field data collected by sonde will also be analyzed using the Excel software. Data will be entered into Excel in columns by drain and in rows by month for each parameter collected. Descriptive Statistics (e.g. means, standard deviations, and coefficient of variation) will be computed for each column in part to ascertain the TSS loading of the drains. The results of the analysis of this data will be used qualitatively to address any discrepancies found in the lab data results (i.e. low D.O. reading may be an indicator of dredging not observed resulting in extreme TSS value). Before conducting any further statistical analyses, field and lab analyses data will be checked for potential outliers because outliers can greatly influence the statistical analyses of the data. As a means of identifying potential outliers a tolerance limit of three standard deviations (99 percent confidence interval) will be used. The following steps will be followed for thoroughly examining and dealing with outliers:

1. Conduct Outlier Statistical Test for the suspected outliers in each column as follows:

    a. Calculate the mean, the standard deviation and the coefficient of variance of the data.

    b. Calculate the statistical mean $\pm$ 2.58σ to determine any suspected values.

2. Check the field and laboratory records or daily logbook for any recording errors and to see if the samplers or the lab technicians noted any special observations or remarks regarding sample collection, handling, and lab analyses to explain the outlier.

3. For outliers whose causes of extreme values can be determined, the suspected data will be either excluded or corrected. Otherwise, the suspected data will be retained and included in the statistical analyses as a true but extreme value.

4. All the facts regarding deleting or retaining outliers should be documented in the statistical section of the final Project Report.

Statistical analyses methods such as the Dixon Type test or Chauvenet's Criterion will be conducted. The results of the analysis will be used to determine if extreme deviates are can be treated as outliers. In the case that the analysis shows that the data are not normally distributed other statistical methods of analysis will be used to normalize.

Upon completion of the last sampling event, the spreadsheet data and all data related to the sampling events will be transferred to the TMDL Development Unit for completion of the TMDLs.

3. ASSESSMENT AND OVERSIGHT

3.1 ASSESSMENT AND RESPONSE ACTIONS

Surveillance of the records and overall status of the project will be conducted by the Quality Assurance Officer to ensure that all of the requirements of the QAPP are being met. Surveillance will be conducted after each sampling event and after all laboratory results have been received for that sampling event.

A Technical Systems Audit will also be performed by the Quality Assurance Officer. During this audit, the Quality Assurance Officer will examine field activities and record-keeping procedures to assess their conformance to the QAPP. This audit will take place during the first sampling trip and any time thereafter. Any non-conformance with the QAPP will be corrected and documented. Performance Evaluations of the laboratories will be conducted through the use of quality control samples, namely split samples and matrix spike samples. A review of the laboratory's Quality Assurance for this project will also be conducted.

Prior to the submittal of the final report, an Audit of Data Quality will be performed to assess the handling of all data and to correct any errors found in the project database. A Data Quality Assessment will also be performed in which statistical tools will be used to determine whether the data met all of the assumptions that the Data Quality Objectives and data collection design were developed under, and whether the total error in the data is tolerable.

3.2 REPORTS TO MANAGEMENT

Upon completion of the project, the Project Manager will prepare a final project report. This final report will include a summary of the activities performed, the resulting data, and the quality of the resulting data, any problems encountered and their solutions and will identify any samples that indicate violations of Water Quality Standards.

4. DATA VALIDATION AND USABILITY

4.1 DATA REVIEW, VERIFICATION AND VALIDATION

Regional Board staff will be responsible for validating the project's data to ensure that QA guidelines have been followed.

4.1.1 DATA REVIEW, VERIFICATION, AND VALIDATION

After each sampling event, the Regional Board's Quality Assurance Officer will review the field notes and field data generated to assess adherence to the project sampling design in terms of the spatial distribution the sampling locations. Departures from the sampling design will be considered in the design of each subsequent phase of sampling. Deviations from the sampling design may change the data needed to characterize the system. Departures from the sampling design may also be due to unforeseen field conditions, which may require adjustment of the sampling design. Significant departures from the project sampling design and responses to those departures will be noted in the project database, as well as the Audit of Data Quality, and in the final report. In the Data Quality Assessment, the Project Quality Assurance Manager will consider the effects of any departures from the sampling design on the overall completeness of the data generated, and thus the usability of the data set for drawing conclusions.

4.1.2 VERIFICATION AND VALIDATION METHODS

Verification of adherence to the sample collection and equipment decontamination procedures contained in Section 5.3.2 of this report will be determined through the field records, Technical Systems Audit, and project surveillance identified above. All of this information will be considered in the final Audit of Data Quality. Departures from the sample collection and equipment decontamination procedures are unacceptable, and will result in data that will not be considered valid for use in this study. Unacceptable departures from sample collection procedures include the use of contaminated sampling bottles, the lack of critical sample collection information, or any other activity which would result in the cross contamination or incorrect identification of samples.

Departures from the sample handling and custody procedures contained in Section 2.3 of this report will be determined through the review of chain of custody forms and laboratory analysis forms. In order for data to be considered valid for meeting the data quality objectives of this study, all samples' chain of custody forms must be in the possession of the project manager, and strict adherence to holding times and temperatures must be followed. Data generated from samples that do not meet these requirements will not be considered valid for use in this study.

Verification of proper calibration of the YSI sonde will be performed during the audit of data quality through a review of the quality control records. Calibration values will also be assessed to determine the potential error in the field measurements. If calibration values for a particular calibration have errors that exceed acceptable error tolerances, the measurements obtained prior to that calibration, but after the previous calibration will be labeled suspect and further investigated to determine if they are valid for use in this study.

Validation of laboratory data will be performed in the Audit of Data Quality by assessing the results of QC sample analyses. Lab data will be validated for precision, accuracy, and completeness according to the criteria specified in Section 1.6

The data then will be entered into database by staff. It is conceivable, however, that errors could occur in entering the data (e.g., transposing the decimal point for a particular result or keying in the wrong Sample ID). Therefore, once a data set has been entered into the database, all records will be checked to ensure accuracy.

In case of missing data, the staff will discuss it with the laboratories submitting the data. In some cases, missing data will be denoted as missing in reports. For all missing data, and any other data requiring special explanation, qualifiers will be included in the database and in data reports. Missing data will be designated as "NR", meaning *Not Reported*.

4.1.3 RECONCILIATION WITH USER REQUIREMENTS

The Quality Assurance Manager will be responsible for validation and final approval of all data for use in this study. The final project report will contain a discussion of relevant information obtained through the Audit of Data Quality about the quality, validity, completeness and limitations of the data obtained in this study. The final project report will also contain a discussion of the results of statistical analyses performed on the data set in the Data Quality Assessment, and a final conclusion as to the adequacy of the data set for making a final determination of the impacts of TSS in the study area.

Data objectives for this project do not require a full, formal, and independent data validation. Although the data is considered legally defensible as presented herein, all records will be available for independent evaluation should the need arise at a later date.

5. HEALTH AND SAFETY PLAN

5.1 CONTAMINATION CONTAINMENT ZONES

The contaminated areas for this Project consist of and cover the entire waterways for the aforementioned waters, their banks, and the area within 2 feet of the banks. Decontamination zones will be set at least 10 feet away from the banks of the surface waters. The decontamination zone will be used for personnel decontamination and will include wash water, soap, paper towels, and trash bags. All contaminated solid waste material will be placed in trash bags for proper disposal. Only biodegradable antibacterial soap will be used at the site. Wash water runoff will be contained and disposed of in the surface waters. The Clean area will be set at least 20 feet away from the banks of the surface waters.

5.2 PERSONAL PROTECTIVE EQUIPMENT

The general concerns at the sampling sites are the potential exposure to pathogens and toxicants present in the waters being sampled, the risk of being struck by an automobile when taking samples near the roadside or off of bridges, and the risks of sunburn, excessive heat exposure, insect and possibly snake bites. In addition, the sampling crew should be aware of the risk of falling into a drain. No less than three experienced samplers will be out in the field at one time. (The sampling crew will also have a functional cellular phone in one of the vehicles).

- Any member of the sampling team has the authority to stop the sampling event when he/she determines that conditions at the site (e.g., rain, dust, local emergency, etc.) preclude safe sampling. A Hazard Evaluation Plan (HEP) will be done for each day of sampling.

- To reduce the risk of exposure to pathogens and toxicants, all samplers will wear a Face Shield, Latex Examination Gloves (inner gloves), Nitrile Gloves (outer gloves), Tyvek Suit or isolation gown, and boot covers (required for collection of all samples). The Contaminated Zone must not be entered without the aforementioned PPE.

- The following precautions will be taken to reduce the risk of being around automobile traffic. At roads, bridge crossing, and wherever traffic is reasonably expected to be present, Traffic Cones will be set at approximately 30-foot intervals as to form at least a 5-foot wide "safety corridor" between the traffic and the sampling crew. At the beginning and end of the corridor, one State vehicle must be parked as part of the "safety corridor". The parked vehicle and safety cones must be clearly visible to on-coming traffic from a distance of at least 120 feet. Samplers will also be required to wear orange vests when sampling near roads.

- To reduce the risk of heat exposure and sunburn, samplers will wear sunscreen and the vehicle will always have plenty of cold drinking water. If any of the samplers begin to experience symptoms of heat exhaustion, such as cramps or dizziness, he or she will immediately be removed from the sun and given plenty of cool liquids. If these symptoms persist, he or she will be taken to the nearest hospital.

- Extra caution should be used when working near or around the drains to reduce the risk of potentially falling in.

- To reduce the risk of insect bites, samplers will use insect repellent.

- To reduce the possibility of snakebites, samplers will check areas for snakes prior to entering the area. If a snakebite occurs, ice will be placed on the bite. The sampler will be immediately transported to the nearest medical facility.

5.3 PERSONNEL DECONTAMINATION PROCEDURES

The Clean Zone must not be entered with contaminated PPE. All team members coming out of the Contaminated Zones must immediately proceed to the Decontamination Zones and use the following decontamination procedures before proceeding to Clean Zone:

1. Remove boot covers and place them in a plastic bag;

2. Wash outer rubber gloves with antibacterial soap prior to removal of any other PPE. Place outer gloves in the storage bin labeled "Decontamination PPE No. 1";

3. Carefully remove Tyvek suit and place it in a plastic bag for contaminated articles to be discarded (making sure not to let skin contact the outside of the suit);

4. Remove face shield and place it in a plastic bag;

5. Remove latex gloves carefully to avoid contact with bare skin and dispose of them in the trash bag. Thoroughly wash hands with antibacterial soap; and

6. Dispose of wash water into surface water just sampled.

Note: everything that is touched (pens, pencils, rinse water bottles, probes, etc.) with dirty gloves could be contaminated. Avoid touching these items with bare skin.

5.3.1 EMERGENCY NUMBERS AND FACILITIES

All sampling personnel will have access to a cellular phone to call 911 in case of an emergency. The hospital nearest all sampling locations is Pioneers Memorial Hospital located at 207 West Legion, Brawley, telephone, 760- 351-3333.

In case of an emergency, sampling personnel should also contact the Regional Board Health & Safety Officer, as soon as practical at 760-346-6585 or 760-341-7491.

5.3.2 AFTER SAMPLING

Place samples into lab refrigerator or keep in an ice chest filled with wet ice; keep water drained from ice chests to avoid soaking container labels. Make copies of field notes and put original in the project binder. Contaminated equipment should be packed in designated containers for transport to the Regional Board office. Decontaminate and properly clean all items, which were exposed in the field in accordance with USGS National Field Manual for the Collection of Water-Quality Data, Chapter A3. Cleaning of Equipment for Water Sampling (See Attachment IV).

6. REFERENCES

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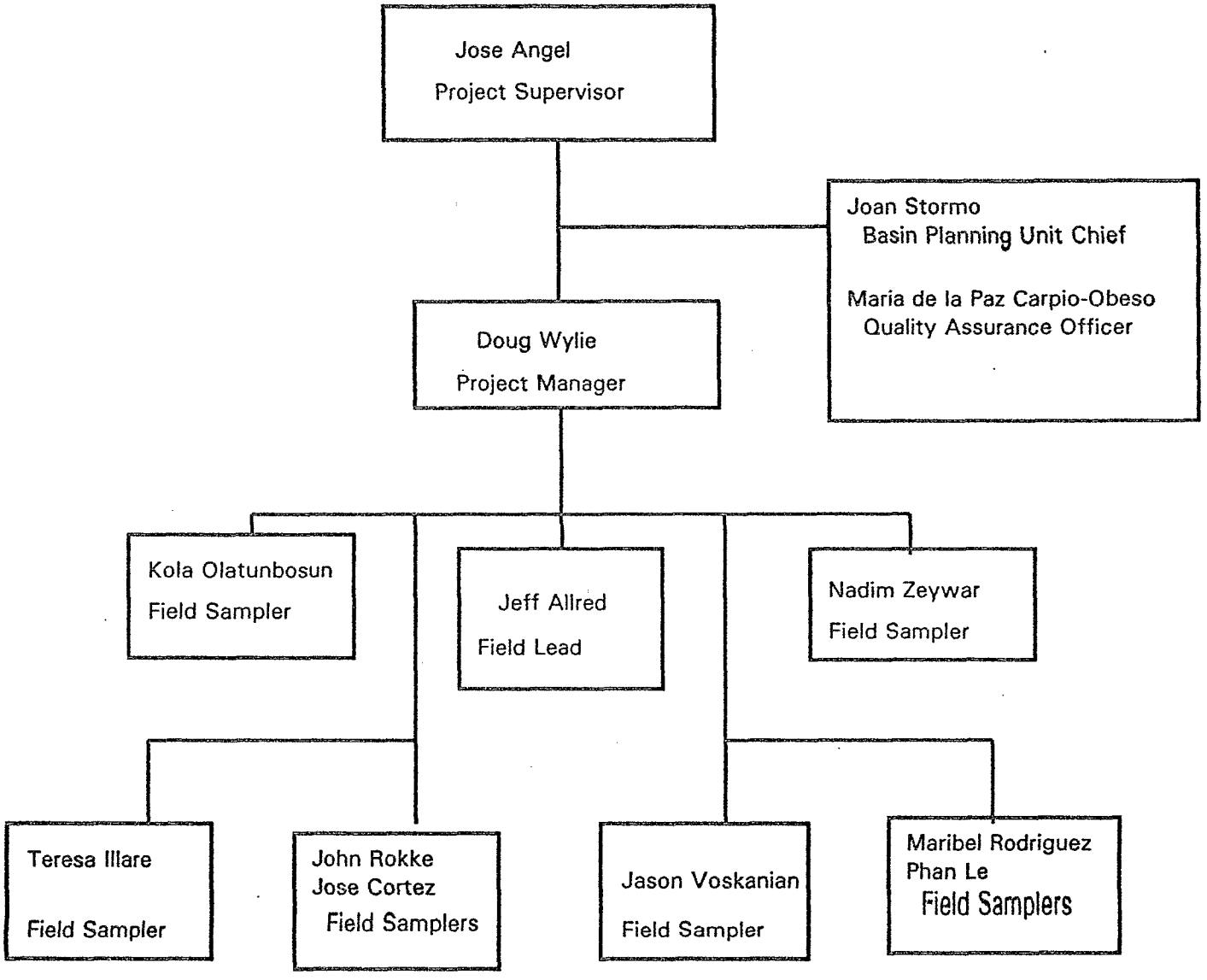
U.S. Environmental Protection Agency. Handling and Disposition of Project Records and Documents. SOP #EPA-90251.3b. U.S. Environmental Protection Agency, Washington, D.C.

U.S. Geological Survey, 1997. Field Guide For Collecting Samples For Analysis In Stream Water For The National Water-Quality Assessment Program, Open-File Report 97-401.

State Water Resources Control Board (State of California), 1994. *Quality Assurance Program Plan.*

Wilde, F.D., D.B. Radtke, Jacob Gibs, and R.T. Iwatsubo. 1998. National field Manual for the Collection of Water-Quality Data. Chapter A3. Cleaning of Equipment for Water Sampling. USGS, Reston, VA.

ATTACHMENT I, PROJECT ORGANIZATION CHART



ATTACHMENT II, SAMPLE CHAIN OF CUSTODY FORM

E.S. BABCOCK & SONS, INC.

6100 Quail Valley Court
Riverside, CA 92507

(909) 653-3351
FAX (909) 653-1662

CHAIN OF CUSTODY RECORD

Lab #s: _____ Invoice No. _____

oject No.	Project Name / Location

mplers:
gnature)

Description	Sampled		Determination Requested												Number of Containers	Condition of Sample			Remarks
	Date	Time														Sealed	Chilled	Preserved	

nquished By:	Date/Time	Received By:	Relinquished By:	Date/Time	Received By:
nquished By:	Date/Time	Received By:	Received For Lab By:	Date / Time	

Attachment III, US Fish and Wildlife Incidental Take Permit

United States Department of the Interior

FISH AND WILDLIFE SERVICE
Ecological Services
Carlsbad Fish and Wildlife Office
2730 Loker Avenue West
Carlsbad, California 92008

In Reply Refer To: FWS-IMP-TA-2404.1

NOV 0 7 2001

Ms. Teresa Newkirk
California Regional Water Quality Control Board
Colorado River Basin Region
73-720 Fred Waring Drive, Suite 100
Palm Desert, California 92260

Subject: Water Quality Monitoring Activities

Dear Ms. Newkirk:

The Fish and Wildlife Service has reviewed your proposed water quality monitoring activities in the Imperial Irrigation District drains. Some of these drains are occupied by the endangered desert pupfish (*Cyprinodon macularius*). Given the nature of the sampling activities, we concur with your conclusion that the potential for impact is very low. Based on the description and schedule of the monitoring activities provided in your letter, it is our determination that no permit under the Endangered Species Act of 1973 (as amended) will be required for this activity. Should the nature of the activity change such that the likelihood of impacts increase, we request that you contact our office so that we may work with you in determining the permitting requirements and how to minimize the potential for impacts. If you have any questions, please contact Carol Roberts of my staff at (760) 431-9440.

Sincerely,

for Nancy Gilbert
Assistant Field Supervisor

NOV 1 5 2001

ATTACHMENT IV, DECONTAMINATION PROCEDURES

Techniques of Water-Resources Investigations

Book 9
Handbooks for Water-Resources Investigations

National Field Manual for the Collection of Water-Quality Data

Chapter A3. CLEANING OF EQUIPMENT FOR WATER SAMPLING

Edited by
F.D. Wilde, D.B. Radtke, Jacob Gibs,
and R.T. Iwatsubo

USGS
science for a changing world

U.S. DEPARTMENT OF THE INTERIOR
BRUCE BABBITT, *Secretary*

U.S. GEOLOGICAL SURVEY
Thomas J. Casadevall, *Acting Director*

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Mail Stop 412
Reston, VA 20192
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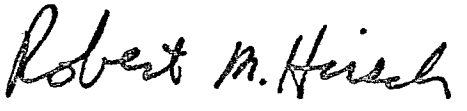
purchased from:
U.S. Geological Survey
Information Services
Box 25286, Federal Center
Denver, CO 80225

Foreword

The mission of the Water Resources Division of the U.S. Geological Survey (USGS) is to provide the information and understanding needed for wise management of the Nation's water resources. Inherent in this mission is the responsibility to collect data that accurately describe the physical, chemical, and biological attributes of water systems. These data are used for environmental and resource assessments by the USGS, other government and scientific agencies, and the general public. Reliable and objective data are essential to the credibility and impartiality of the water-resources appraisals carried out by the USGS.

The development and use of a *National Field Manual* is necessary to achieve consistency in the scientific methods and procedures used, to document those methods and procedures, and to maintain technical expertise. USGS field personnel use this manual to ensure that data collected are of the quality required to fulfill our mission.



Robert M. Hirsch
Chief Hydrologist

Techniques of Water-Resources Investigations

Book 9
Handbooks for Water-Resources Investigations

Chapters of Section A: National Field Manual for the Collection of Water-Quality Data

A1. Preparations for Water Sampling

A2. Selection of Equipment for Water Sampling

A3. Cleaning of Equipment for Water Sampling

A4. Collection of Water Samples

A5. Processing of Water Samples

A6. Field Measurements
- 6.0 General Information and Guidelines
- 6.1 Temperature
- 6.2 Dissolved Oxygen
- 6.3 Specific Electrical Conductance
- 6.4 pH
- 6.5 Reduction-Oxidation Potential (Electrode Method)
- 6.6 Alkalinity and Acid Neutralizing Capacity
- 6.7 Turbidity

A7. Biological Indicators
- 7.1 Fecal Indicator Bacteria
- 7.2 Five-Day Biochemical Oxygen Demand

A8. Bottom-Material Samples

A9. Safety in Field Activities

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[1]Bold type indicates published chapters and chapter sections, and shaded type indicates chapters and chapter sections that are in preparation.

CLEANING OF EQUIPMENT FOR WATER SAMPLING A3.

National Field Manual for the Collection of Water-Quality Data

Chapter A3.

Illustrations

Table

Chapter A3. CLEANING OF EQUIPMENT FOR WATER SAMPLING

Edited by Franceska D. Wilde, Dean B. Radtke, Jacob Gibs, and Rick T. Iwatsubo

ABSTRACT

The *National Field Manual for the Collection of Water-Quality Data (National Field Manual)* provides protocols and guidelines for U.S. Geological Survey (USGS) personnel who collect data used to assess the quality of the Nation's surface-water and ground-water resources. Chapter A3 describes procedures for cleaning the equipment used to collect and process water samples and for assessing the efficacy of the equipment-cleaning process. This chapter is designed for use with the other chapters of this field manual.

Each chapter of the *National Field Manual* is published separately and revised periodically. Newly published and revised chapters will be announced on the USGS Home Page on the World Wide Web under "New Publications of the U.S. Geological Survey." The URL for this page is <http://water.usgs.gov/lookup/get?newpubs>.

INTRODUCTION

As part of its mission, the U.S. Geological Survey (USGS) collects data needed to assess the quality of our Nation's water resources. The *National Field Manual for the Collection of Water-Quality Data (National Field Manual)* describes protocols (requirements and recommendations) and provides guidelines for USGS personnel who collect data on the Nation's surface-water and ground-water resources. Chapter A3 describes procedures for cleaning the

equipment used to collect and process samples of surface water and ground water and procedures for assessing the efficacy of the equipment-cleaning process.

The *National Field Manual* is Section A of Book 9 of the USGS publication series Techniques of Water-Resources Investigations (TWRI). Each chapter of this manual is published as a separate report. Chapter numbers are preceded by an "A" to indicate that the report is part of the *National Field Manual*. Other chapters and sections of other chapters of the *National Field Manual* are referred to in this report by the abbreviation "NFM" and the specific chapter and (or) section number. For example, general information on field measurements of ground water is covered in section 6.0.2 of Chapter A6, "Field Measurements," and would be cited as NFM 6.0.2.

PURPOSE AND SCOPE

The *National Field Manual* is targeted specifically toward field personnel in order to *(1)* establish and communicate scientifically sound methods and procedures, *(2)* provide methods that minimize data bias and, when properly applied, result in data that are reproducible within acceptable limits of variability, *(3)* encourage consistent use of field methods for the purpose of producing nationally comparable data, and (4) provide citable documentation for USGS water-quality data-collection protocols.

The equipment-cleaning procedures presented in this chapter are adequate for routine environmental conditions. A modification of the cleaning procedures might be required, for example, in order to decontaminate equipment adequately after sampling at sites where analyte concentrations are large. Modifications to the standard procedures described in this chapter must be documented and quality controlled.

REQUIREMENTS AND RECOMMENDATIONS

As used in the *National Field Manual*, the terms **required** and **recommended** have USGS-specific meanings.

Required (require, required, or requirements) pertains to USGS protocols and indicates that a specific USGS Office of Water Quality (OWQ) policy has been established on the basis of research and (or) consensus of the technical staff and has been reviewed by water-quality specialists and District[1] or other professional personnel, as appropriate. Technical memorandums or other unpublished documents that define the policy pertinent to such requirements are cited in this chapter. Personnel are instructed to use required equipment or procedures as described in this chapter. Departure from or modifications to the stipulated requirements that might be necessary to accomplish specific data-quality requirements or study objectives must be based on referenced research and good field judgment and must be quality assured and documented.

Recommended (recommend, recommended, or recommendation) pertains to USGS protocols and indicates that USGS Office of Water Quality policy recognizes that one or several alternatives to a given procedure or equipment selection are acceptable on the basis of research and (or) consensus. Specific data-quality requirements, study objectives, or other constraints affect the choice of recommended equipment or procedures. Selection from among the recommended alternatives should be based on referenced research and good field judgment, and reasons for the selection should be documented. Departure from or modifications to recommended procedures must be quality assured and documented.

[1]**District** refers to a water-data collecting organizational unit of the USGS located in any of the States or Territories of the United States.

FIELD MANUAL REVIEW AND REVISION

Chapters of the *National Field Manual* will be reviewed, revised, and reissued periodically to correct any errors, incorporate technical advances, and address additional topics. Please send comments or corrections to NFM-QW, USGS, 412 National Center, Reston, VA 20192 (or send electronic mail to nfm-owq@usgs.gov). Information regarding the status and any errata of this and other chapters can be found at the beginning of the electronic version of each chapter, located in the Publications Section of the following website: http://water.usgs.gov/lookup/get?owq.

Newly published and revised chapters will be announced on the USGS Home Page on the World Wide Web under "New Publications of the U.S. Geological Survey." The URL for this page is <http://water.usgs.gov/lookup/get?newpubs>.

ACKNOWLEDGMENTS

The information in this chapter of the *National Field Manual* is based principally on the work of Sandstrom (1990), Horowitz and others (1994), Shelton (1994), and Koterba and others (1995).

The editors wish to thank and pay tribute to R.W. Lee and S.W. McKenzie, who were responsible for final technical review and who contributed to the accuracy, quality, and usability of this report. We would like to express appreciation to the following colleague reviewers for helping to improve this report: H.D. Ardourel, B.A. Bernard, K.K. Fitzgerald, D.S. Francy, S.R. Glodt, V.J. Kelly, S.L. Lane, S.K. Sando, C.A. Silcox, and W.R. White. The editors are indebted to I.M. Collies, C.M. Eberle, B.B. Palcsak, and Chester Zenone for their valuable editorial contributions, and to C.T. Mendelsohn, L.E. Menoyo, and A.M. Weaver, whose production assistance was instrumental in maintaining the quality of the report.

Special thanks go to T.L. Miller, whose encouragement and faith in this project has been instrumental to its achievement; and to D.A. Rickert and J.R. Ward, who provided the support needed to produce a national field manual for water-quality studies.

A3. CLEANING OF EQUIPMENT FOR WATER SAMPLING

Equipment cleaning (decontamination): Applying cleaning solutions to the surfaces of equipment or using other nondestructive procedures (such as steam cleaning) to remove foreign substances that could affect the concentrations of analytes in samples.

USGS policy requires that equipment for water samples be properly cleaned before contacting the sample and that the effectiveness of cleaning procedures be quality controlled (Sandstrom, 1990; Horowitz and others, 1994; Koterba and others, 1995). The goal of equipment cleaning is to help ensure that the equipment is not a source of foreign substances that could affect the ambient concentrations or chemistry of target analytes in samples. Standard procedures are described in this chapter for when, where, and how to clean equipment constructed of various materials and to collect equipment blanks and field blanks for quality control. Space is commonly dedicated in an office laboratory for equipment cleaning and for storage of cleaning supplies. In this report this work space can include the Field Service Unit or other dedicated office space.

- Clean all sample-collection and sample-processing equipment before use.
  - Manufacturing residues must be removed from new equipment.
  - Dust and any other foreign substances must be removed from equipment that has been in storage.
  - Substances adhering to equipment from previous sampling must be removed.
- Prevent cross contamination between sampling sites by rinsing equipment with deionized water (DIW) while equipment is still wet, and then clean equipment as prescribed in this chapter before transporting it to the next site.
- Do not substitute field rinsing with sample water for the equipment-cleaning procedures described in this chapter.
- Collect equipment blanks and field blanks for quality control. A minimum of one equipment blank per year is required for each piece of equipment. The frequency of collecting blanks normally is based on study objectives and site conditions.

To help prevent sample and site contamination, be sure to use properly cleaned equipment.

SUPPLIES FOR EQUIPMENT CLEANING 3.1

By D.B. Radtke, A.J. Horowitz, and M.W. Sandstrom

The supplies commonly used to clean sample-collection and sample-processing equipment are listed in table 3-1. Cleaning supplies are to be stored in a contaminant-free cabinet. Follow safety instructions regarding the storage of chemical reagents (NFM 9).

Before gathering the cleaning supplies, check the construction materials (for example, metal, glass, or plastic) of washbasins and other cleaning items relative to the samples to be collected.

- **For analysis of inorganic constituents**—Basins, brushes, and other items used for cleaning should be constructed of a suitable nonmetallic material such as uncolored or white polypropylene, polyethylene, or other plastic. **Do not use cleaning agents or items that might leach or sorb metals if the equipment to be cleaned will be used for samples to be analyzed for trace elements.**
- **For analysis of organic compounds**—Basins and other cleaning items can be constructed of metal, glass, or plastic materials. Stainless steel is recommended if methanol will be used. **Do not use cleaning agents or items that might leach, sorb, or leave residues of organic substances that could bias or interfere with the analysis.**

CAUTION: Refer to Material Safety Data Sheets (MSDS) before handling any chemicals. Wear appropriate safety gloves, glasses, and apron when working with corrosive and oxidizing solutions. When using chemicals, work in a well-ventilated area.

Table 3-1. Supplies for cleaning equipment used for water-sampling activities

[ACS, American Chemical Society; DIW, distilled/deionized water; µS/cm, microsiemens per centimeter at 25 degrees Celsius; PBW, pesticide-grade blank water; VBW, volatiles and pesticide-grade blank water; IBW, inorganic-grade blank water; L, liter; cm, centimeter; TOC, total organic carbon; DOC, dissolved organic carbon; SOC, suspended organic carbon; NFM, *National Field Manual*; PVC, polyvinyl chloride; IBW, inorganic-grade blank water]

Item	Description and Comments
Acid solution[1]	Hydrochloric: ACS trace-element grade (5 percent by volume in DIW). Nitric: ACS trace-element grade (10 percent by volume in DIW).
Aluminum foil	Organics only. Heavy duty, for work surfaces and equipment.
Bags, plastic or fluorocarbon polymer	Sealable bags with uncolored closure strips, various sizes. Recyclable trash bags are recommended for large equipment storage.
Noncolored plastic sheeting	Clean sheeting used to provide a clean work surface.
Brushes and sponges	Uncolored; plastic components needed for inorganic work.
Distilled/deionized water (DIW)	Maximum specific electrical conductance, 1 µS/cm (usually District produced; Office of Water Quality Memorandum 92.01).
Office-produced organic-grade deionized water	Usable only as a cleaning solution and only as specified in the text. Must not be used to substitute for PBW or VBW.[2]
Detergent	Nonphosphate laboratory soap (for example, Liquinox™).
Gloves, disposable	Powderless, noncolored vinyl, latex, or nitrile (latex or nitrile for use with methanol), assorted sizes.
Inorganic-grade blank water (IBW)[2]	Blank water with certificate of analysis prepared and (or) quality assured by the analyzing laboratory. IBW is required for blank samples.
Jerricans or carboys	For waste solutions and as neutralization container. Neutralization container: 25- to 30-L, polyethylene, wide-mouth, with layer of marble chips. Methanol waste container: Appropriate for flammable liquid.
Methanol	ACS pesticide grade. Methanol is the organic solvent in common use for equipment cleaning, but study requirements might dictate use of a different ACS pesticide-grade solvent.
Neutralization materials	Marble landscape chips (1- to 2-cm chips recommended).[3]
Pesticide-grade blank water (PBW)[2]; volatile-grade blank water (VBW)[2]	Blank water prepared and (or) quality assured by the analyzing laboratory; required for collecting blank samples as follows: PBW for pesticide analysis; VBW for volatile compounds analysis and pesticide analysis; and either PBW or VBW for TOC, DOC, and SOC analyses.
Safety equipment and guidelines (NFM 9)	For example, Material Safety Data Sheets (MSDS), safety glasses, chemical spill kit, apron, emergency phone numbers.

Table 3-1. Supplies for cleaning equipment used for water-sampling activities—*Continued*

Item	Description and Comments
Standpipes for submersible pump	Plastic, glass, or other suitable material; for example, pipette jars or capped PVC casing; one standpipe labeled for blank water and one each for each cleaning solution. (Do not use PVC for methanol.)
Tapwater	If quality is questionable, substitute DIW. Tapwater is more effective for initial and rapid removal of detergent residue.
Tissues	Laboratory grade, lint free, various sizes (for example, Kimwipes™).
Washbasins	One washbasin for each cleaning solution; white or uncolored. Plastic, nonleaching. (Stainless steel is required for methanol.)
Wash bottles (dispenser or squeeze)	Labeled to indicate contents (for example, ACID, DIW, TAP). Fluorocarbon polymer needed for methanol, PBW, VBW, and IBW.

[1]Hydrochloric acid is required if analyzing for nitrogen species; otherwise, nitric acid is acceptable.
[2]PBW and VBW can be obtained from the USGS National Water Quality Laboratory (NWQL). IBW can be obtained from the USGS Quality of Water Service Unit.
[3]Agricultural limestone, soda ash, baking soda, and crushed shells are not recommended (Horowitz and others, 1994).

CAUTION: Methanol is extremely flammable and potentially explosive, emits noxious fumes, and is absorbed through the skin. Observe safety practices when handling methanol or other organic solvents.

- **Wear safety gloves, glasses, and apron.**
- **Work in a well-ventilated area and away from an open flame or sparks.**
- **Make sure that all electrically powered equipment is grounded; alternating current equipment must have a ground-fault interrupter.**
- **Inspect electrical wiring for cuts, breaks, or abrasions where the metal wire is exposed.**
  - **Exposed wires can cause sparks if a short to ground occurs.**
  - **Replace faulty wires—do not rely on fixing with electrical tape.**

CLEANING PROCEDURES 3.2

By A.J. Horowitz and M.W. Sandstrom

Equipment should be cleaned in an area protected from airborne or other sources of contamination. Procedures to remove contaminants to concentrations below the targeted method-detection levels can vary, depending on the cleaning supplies used, the type of equipment being cleaned, the solubility and concentration of contaminant(s), and the length of time equipment is exposed to contaminant(s). **Examine equipment-blank and field-blank data to determine whether adjustments to the cleaning protocol are needed (section 3.4).**

The cleaning procedure to be used depends on the type(s) of water samples that will be collected and processed. Figure 3-1 summarizes the sequence of cleaning procedures for equipment used to collect samples for inorganic and (or) organic analytes (Sandstrom, 1990; Horowitz and others, 1994; and Koterba and others, 1995).

- **Inspect equipment for stains, cuts, or abrasions. Replace parts as needed.**
  - Replace chipped or cracked glassware.
  - Replace bent sampler nozzles or samplers with bent fins (surface-water samplers).
  - Replace tubing if mold, mildew, or imbedded sediment cannot be removed.
  - Replace cracked or severely crimped O-rings.
  - Repair pump intakes and antibacksiphons that have loose or missing screws.
  - Check the flow manifold and sample tubing to ensure that valves and quick-connect fittings are in good working order; repair or replace as necessary to eliminate any problems.
  - Recoat chipped surface-water samplers with epoxy paint or "plasti-coat." Such samplers must be recoated before use.

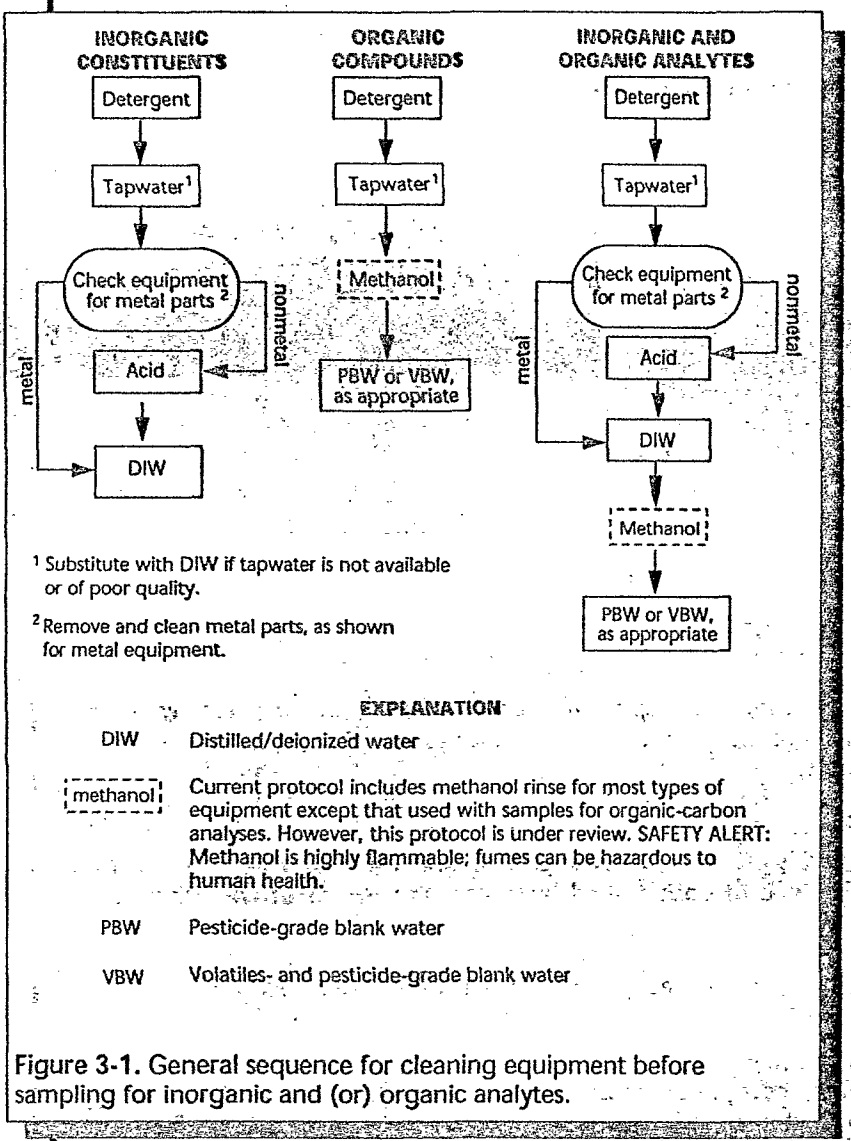

Figure 3-1. General sequence for cleaning equipment before sampling for inorganic and (or) organic analytes.

- **Rinse equipment with DIW directly after use while equipment is still wet and before cleaning procedures are implemented.**
- **Place cleaned equipment in doubled storage bags.**

Do not allow collection and processing equipment to sit uncleaned in a field vehicle or elsewhere between field trips

CLEANING OF EQUIPMENT USED TO SAMPLE FOR INORGANIC CONSTITUENTS 3.2.1

Cleaning of equipment used to collect and process water for analysis of inorganic constituents involves a five-step office-laboratory procedure or a five-step field-site procedure. These procedures are effective for cleaning equipment exposed to water containing concentrations of as much as 50,000 µg/L of iron, 5,000 µg/L each of manganese and zinc, 400 µg/L of copper, 125 µg/L of cobalt, and large concentrations of the other trace elements (Horowitz and others, 1994). The cleaning procedures are summarized in figures 3-2 and 3-3. (These procedures do not apply to field-measurement instruments—see NFM 6.)

Equipment should be cleaned periodically in the office laboratory, where complete disassembly is more practical and more thorough procedures are possible. Compared to cleaning at the field site, cleaning procedures carried out in the office laboratory involve longer exposure of equipment to cleaning solutions, more frequent change of cleaning solution, and greater volumes of rinse water.

- To minimize field cleaning of equipment between sampling sites, preclean a separate set of equipment for each site.

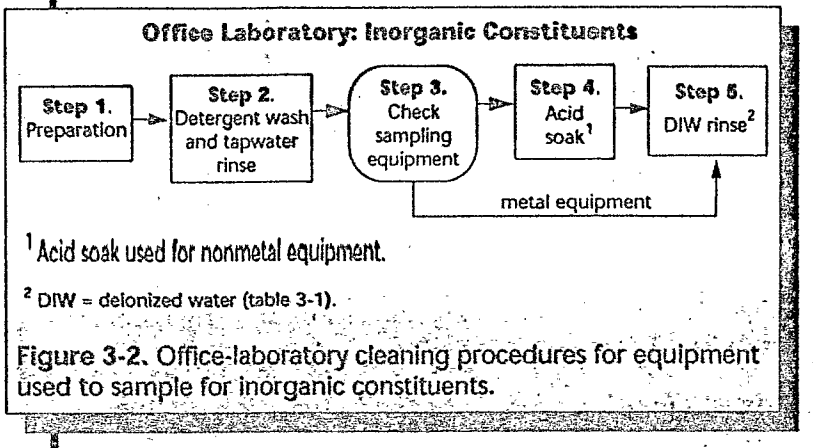

Figure 3-2. Office-laboratory cleaning procedures for equipment used to sample for inorganic constituents.

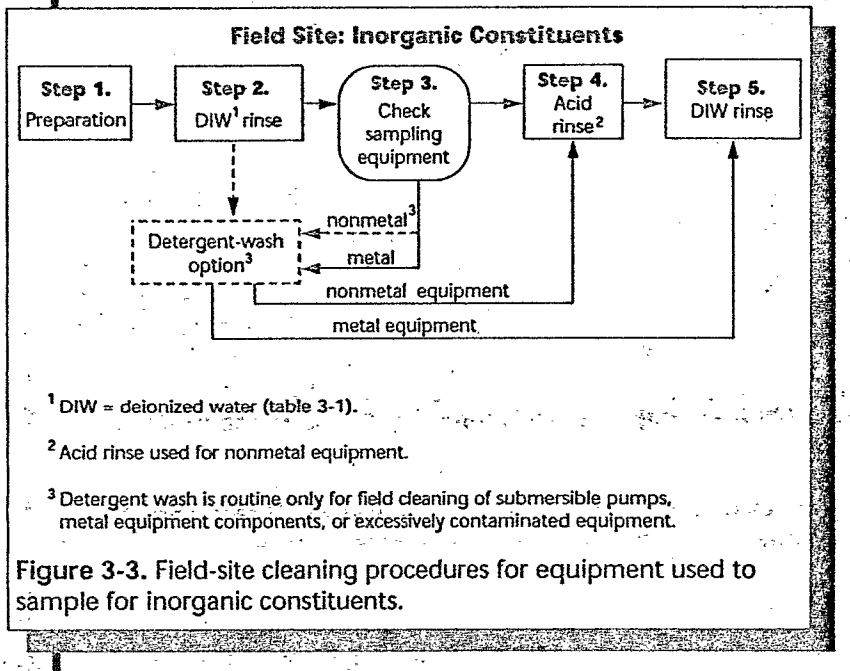

Figure 3-3. Field-site cleaning procedures for equipment used to sample for inorganic constituents.

- If individual or dedicated sets of equipment for each field site are not available or cannot be precleaned, clean the equipment onsite and process additional field blanks during each field trip (Horowitz and others, 1994; Koterba and others, 1995).

- Return excessively contaminated equipment to the office laboratory for rigorous cleaning before reuse.

- After cleaning, document completion of and any modifications to the cleaning procedures.

Equipment-cleaning procedures for inorganic constituents

Standard procedures for office-laboratory and field-site cleaning of equipment used to collect and process samples for analysis of inorganic constituent are described below and summarized in figures 3-2 and 3-3. Not all the steps listed apply to all equipment, however. For example,

- Omit detergent step when cleaning plastic bags for surface-water samplers.

- Omit acid step when cleaning submersible pumps, the churn-splitter spigot, or other equipment constructed of stainless steel or other metallic material.

- Omit detergent and acid steps when cleaning sample bottles.

Be sure to check the specific procedures for sample bottles and other selected equipment listed in section 3.3 before proceeding with the office-laboratory and field-site procedures.

Step 1. Preparation at the office laboratory or field site (figs. 3-2 and 3-3).

a. Prepare a contaminant-free space for cleaning and drying the cleaning supplies and sample-collection and sample-processing equipment.

i. Gather the cleaning supplies, the equipment to be cleaned, and the plastic bags or other material with which to wrap the cleaned equipment. Check table 3-1 for the cleaning supplies needed.

ii. Place clean plastic sheeting over the work surface.

iii. Put on disposable, powderless gloves[2], a laboratory coat or apron, and safety glasses.

iv. Prepare the detergent solution, using a nonphosphate, laboratory-grade detergent.

- **Office laboratory** (fig. 3-2). Use 0.1- to 2-percent solution, volume-to-volume (v/v), using a higher concentration for dirtier equipment.
- **Field site** (fig. 3-3). Use 0.1- to 0.2-percent solution, v/v.

v. Prepare the acid solution, using a 5-percent v/v dilution of ACS trace-element-grade hydrochloric acid (HCl) in DIW.

- **Add the acid to the water**, not water to acid (NFM 9).
- If nitric acid (HNO_3) will be used, prepare a 10-percent solution (v/v) of ACS trace-element-grade acid in DIW.

vi. Label each washbasin, standpipe, and wash bottle to indicate the solution it will contain. Use a black waterproof marker.

vii. Unwrap the equipment to be cleaned and discard the storage bags. Change gloves.

b. Clean the items used to clean the equipment.

i. Fill washbasins and (or) standpipes with the nonphosphate detergent solution. Put wash bottles, scrub brushes, and other small items used for cleaning into a washbasin. **Soak for 30 minutes.**

ii. Scrub interior and exterior sides of basins and standpipes with soft scrub brushes. Fill wash bottles with a soapy solution and shake vigorously.

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[2]Refers to laboratory gloves that are nonpowdered on the inside and intended for disposal after one use. Glove materials must be appropriate for the work to be carried out and the solutions and equipment to be contacted. For example, vinyl gloves are appropriate for most sampling activities but not when working with methanol or other organic solvents.

iii. Rinse all items thoroughly with tapwater to remove detergent residue. No detergent bubbles should appear when fresh tapwater is agitated in the basin, standpipe, or wash bottle.

iv. Rinse washbasins with DIW.

v. Pour 5-percent HCl (or 10 percent HNO_3) solution into washbasins, standpipes, and wash bottles. Soak for 30 minutes. **Do not soak items with metal parts (exposed or hidden) in an acid solution.**

vi. Discard used acid solution into a neutralization container containing a bottom layer of marble chips (Step 4d).

vii. Rinse washbasins, standpipes, and wash bottles with DIW. Dispose of DIW using directions in Step 4d.

c. Disassemble sample-collection and sample-processing equipment. Change gloves.

- Submersible pumps should be disassembled periodically for office cleaning, but they are not usually disassembled for field cleaning.
- Processing and preservation chamber frames should be cleaned periodically using office-laboratory cleaning procedures. Field cleaning is needed only if the cover is slipped over the frame instead of being clipped to the inside of the frame.

Step 2. Detergent wash and tapwater rinse—Office laboratory (fig. 3-2).

a. Place small equipment parts into washbasin labeled for detergent and fill with a 0.1- to 2-percent solution of nonphosphate laboratory detergent. The amount of detergent depends on the hardness of the tapwater and the degree to which the equipment is dirty or contaminated.

b. Soak equipment and tubing for 30 minutes: fill tubing with solution and keep submerged.

c. Scrub exterior and interior of equipment surfaces to the extent possible, using a firm sponge or soft brush to remove any adhering material such as oil and grease, sediment, algae, and chemical deposits. Pay particular attention to grooves and crevices, O-rings, nozzles, and other spaces where inorganic or organic materials might be trapped. Change gloves.

d. Rinse equipment thoroughly with warm tapwater to remove detergent residue. Equipment rinsing is completed when no soap bubbles appear after the rinse water is agitated. Change gloves.

Step 2. DIW rinse and detergent-wash option—Field site (fig. 3-3).

For the DIW rinse:

a. Rinse equipment and tubing with DIW. Pay particular attention to removing material from grooves and crevices, O-rings, nozzles, and places where materials might be trapped. Note that equipment should already have had one DIW rinse directly after contact with sample water and before the equipment had a chance to dry.

b. Change gloves. Proceed to field detergent-wash option only for metal equipment components or for equipment that has become excessively contaminated.

For the detergent-wash option:

A field detergent wash is used for between-site cleaning of submersible pumps, metal components of equipment, or for equipment that has become greasy or otherwise coated and requires detergent to remove foreign materials; specific instructions for submersible pumps are given in section 3.3.9.

a. Place small equipment, tubing, and parts into basin labeled "detergent" and fill with a 0.1- to 0.2-percent detergent solution. Soak for about 10 minutes, or keep equipment assembled and circulate the solution through pump tubing for 5 to 10 cycles.

b. Scrub equipment surfaces with a firm sponge or soft brush to remove any adhering material such as oil and grease, sediment, algae, or chemical deposits. Pay particular attention to grooves and crevices, O-rings, nozzles, and other places where materials might be trapped. Change gloves.

c. Rinse equipment thoroughly with tapwater to remove detergent residue. Use DIW if tapwater is unavailable or is suspected of having a quality so poor as to contaminate the equipment. If necessary, use a wash bottle filled with DIW or tapwater to rinse hard-to-reach places; pump tapwater through assembled equipment for five or more tubing volumes. Equipment rinsing is complete when no soap bubbles appear after agitating the rinse water. If nonmetal equipment has been detergent-washed, go to Step 4.

d. Place equipment into acid-solution washbasin. Change gloves.

Step 3. Check equipment—Office laboratory and field site (figs. 3-2 and 3-3).

- Nonmetal equipment or equipment with removable metal parts: remove any metal parts and go to Step 4.
- Metal equipment components or excessively contaminated equipment: go to Step 2, detergent-wash option at the field site and then to Step 5, DIW rinse.

Step 4. Acid soak/rinse—Office laboratory and field site (figs. 3-2 and 3-3).

For equipment constructed primarily of glass or fluorocarbon polymer or some other plastic, soak (office laboratory) or rinse (field site) in a 5-percent (v/v) HCl solution to remove any remaining organic films and inorganic deposits.

> TECHNICAL NOTE: A 10-percent (v/v) HNO_3 solution can be used instead of HCl if samples to be collected with the equipment will not be analyzed for nitrogen species.

CAUTION: Wear safety glasses and other protective apparel when working with acids.

a. Place nonmetal equipment and tubing into the washbasin labeled "acid solution."

b. **Office laboratory.** Fill basin with dilute HCl solution (see TECHNICAL NOTE above). Soak equipment and tubing for 30 minutes. Carefully swirl the acid solution several times during the 30-minute soak to enhance removal of mineral encrustations.

c. **Field site.** Using a wash bottle filled with 5-percent HCl solution (see TECHNICAL NOTE above), rinse exterior of equipment and tubing. Pump acid solution through the equipment and tubing, using a peristaltic pump.

d. Carefully pour or pump the used acid solution into a neutralization container with marble chips covering the bottom (table 3-1). Do not reuse the acid solution.

   - Do not fill the neutralization container more than three-fourths full of acid solution.
   - Ventilate container and workspace to allow for safe escape of carbon dioxide gas during dissolution of marble chips.

- Check the solution pH periodically using narrow range pH indicator strips. Neutralization is complete when the solution pH is greater than 6.0 or the original DIW pH.
- Discard the neutral solution, as appropriate.
- Rinse the container with tapwater but retain any undissolved marble chips. Replenish chips to form a layer on the bottom of the neutralization container.

Step 5. DIW rinse—Office laboratory or field site (figs. 3-2 and 3-3).

a. Place equipment into the cleaned washbasin labeled DIW. Change gloves.

b. **Office laboratory.** Rinse exterior and interior of each piece of equipment and tubing thoroughly with DIW and place on a clean surface to dry or into a clean IBW washbasin if blank samples will be collected to quality control the cleaning procedures.

c. **Field site.** Pump DIW through equipment.

d. Pour or discharge DIW rinse water into neutralization container. Change gloves.

e. Continue DIW rinsing until rinse-water pH is greater than 6.0 or the original DIW pH.

f. Allow equipment to air dry in an area free from potential airborne contaminants.

Storage of clean equipment

- Place dry, clean equipment inside doubled plastic bags. For small equipment, parts, and tubing, use sealable plastic bags.
- Place the churn splitter and funnel into doubled plastic bags and then place churn splitter inside of the churn carrier.

Clean equipment at the sampling site while equipment is still wet and before leaving for the next site.

CLEANING OF EQUIPMENT USED TO SAMPLE FOR ORGANIC COMPOUNDS 3.2.2

Nearly identical procedures are used in the office laboratory and at the field site to clean equipment used to sample for organic compounds. The office laboratory provides an environment in which equipment can be cleaned over an extended time using greater volumes of cleaning and rinsing solutions than in the field. The five-step cleaning procedure summarized in figure 3-4 is described in this section. If inorganic constituents also will be sampled for, check the sequence of cleaning solution to be used as shown in figure 3-1 before proceeding.

- Preclean a separate set of equipment for each site in order to avoid field cleaning of equipment between sampling sites. Always rinse equipment with DIW directly after use, however.
- If individual or dedicated sets of equipment for each field site are not available or cannot be precleaned, field clean equipment before moving to the next sampling site and process additional field blanks for each field trip (Koterba and others, 1995).
- Collect additional field blanks after cleaning equipment that was exposed to high levels of contamination (NFM 4) and before the equipment is reused for environmental sampling.

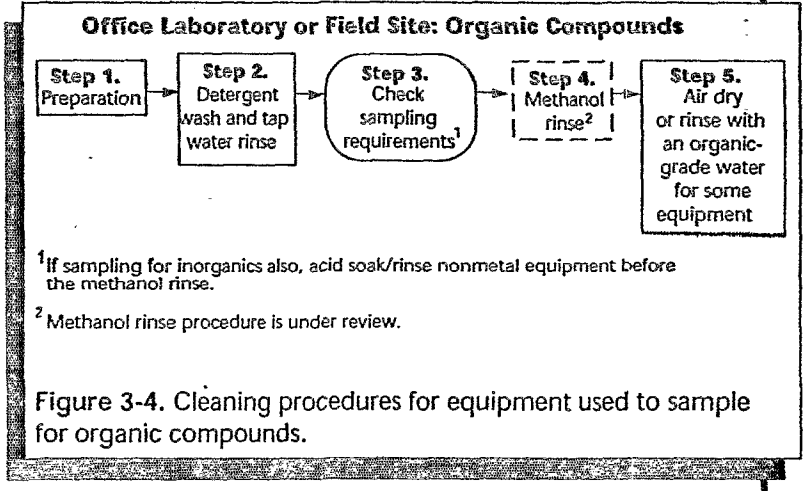


Figure 3-4. Cleaning procedures for equipment used to sample for organic compounds.

Equipment-cleaning procedure for organic compounds

Standard procedures for office-laboratory and field-site cleaning of equipment used to collect and process samples for organic-compound analysis are described below and summarized in figure 3-4. Not all the steps listed apply to all equipment, however. For example,

- **Omit any cleaning procedure for sample bottles for organic compounds.** Bottles for organic analyses arrive from the laboratory capped and ready for use and should not be rinsed by field personnel. Discard bottles if received uncapped.
- **Omit the methanol rinse when cleaning the equipment used to collect and process samples for total, dissolved, and suspended organic carbon (TOC, DOC, SOC).** If equipment (such as a submersible pump) that has been in contact with methanol or other organic solvent must be used for TOC, DOC, or SOC sampling, flush the equipment with copious quantities of sample water before collecting the sample; collection of a blank sample for DOC quality control is recommended.

Be sure to check the specific procedures for selected equipment listed in section 3.3 before proceeding with the office-laboratory and field-site procedures.

Step 1. Preparation (fig. 3-4).

a. Prepare a contaminant-free space for cleaning and drying the cleaning supplies and sample-collection and sample-processing equipment.

   i. Gather the cleaning supplies, the equipment to be cleaned, and clean storage bags and aluminum foil with which to wrap the cleaned equipment. (Check table 3-1 for the cleaning supplies needed.)

   ii. Cover the cleaning area with aluminum foil or fluorocarbon polymer sheeting.

iii. Put on disposable, powderless gloves,[3] a laboratory coat or apron, and safety glasses. **Gloves provide protection from direct contact with solvents only for a limited period of time.**

iv. Prepare the detergent solution, using nonphosphate laboratory-grade detergent. A 0.1- to 0.2-percent (v/v) solution is normally of sufficient strength, unless equipment is very oily or greasy. **Do not use greater than a 0.2-percent solution for field cleaning.**

b. Clean the items used to clean the equipment.

i. Label each washbasin, standpipe, and wash bottle with a black waterproof marker to indicate the solution it will contain.

ii. Follow Steps 2–5, listed below, to clean the washbasins, standpipes, wash bottles, and other items to be used for equipment cleaning.

c. Disassemble sample-collection and sample-processing equipment. Submersible pumps should be disassembled periodically for office cleaning but usually are not disassembled for field cleaning.

[3]Refers to laboratory gloves that are nonpowdered on the inside and intended for disposal after one use. Glove materials must be appropriate for the work to be carried out and the solutions and equipment to be contacted. For example, vinyl gloves are appropriate for most sampling activities but not when working with methanol or other organic solvents. Use solvent-resistant gloves when cleaning with organic solvents. Latex or nitrile disposable, powderless gloves are appropriate when using methanol.

Step 2. Detergent wash and tapwater rinse (fig. 3-4).

a. Place small equipment parts into washbasin labeled for detergent. Fill washbasin with a 0.2-percent solution of nonphosphate, laboratory-grade detergent. (The specific concentration of detergent solution depends on how contaminated the equipment might be and on the hardness of the tapwater.) Change gloves.

   - **Office laboratory.** Soak equipment in detergent solution for 10 to 30 minutes.
   - **Field site.** Rinse equipment exterior and interior with detergent solution.

b. Scrub the exterior and interior of equipment surfaces to the extent possible, using a firm sponge or soft brush to remove any adhering material such as oil and grease, sediment, algae, or chemical deposits. Pay particular attention to removing material from areas where inorganic or organic materials might be trapped, such as grooves and crevices, O-rings, and nozzles.

c. Place equipment into tapwater washbasin.

d. Rinse equipment thoroughly with tapwater to remove detergent residue. Use an organic-grade water (PBW, VBW, or office-produced) if tapwater is unavailable or is of a quality so poor as to contaminate the equipment. If necessary, use a wash bottle filled with organic-grade water or tapwater to rinse hard-to-reach places. Equipment rinsing is complete if no detergent bubbles appear when rinse water is agitated. Change gloves.

Step 3. Check sampling requirements (fig. 3-4).

a. If samples will be collected for organic analysis only, go to Step 4.

b. If samples will be collected for inorganic analysis in addition to organic analysis, follow the procedure for the acid wash and DIW rinse before proceeding with the methanol rinse (see figs. 3-1 and 3-4).

Step 4. Methanol rinse[4] (fig. 3-4).

a. Change to gloves that are chemically resistant to any solvent being used. Place cleaned equipment into a clean stainless steel or organic-solvent-resistant washbasin. Methanol-rinse area must be outside of the field vehicle and away from the sample-processing site. **Sample-collection, -processing, and -preservation areas must remain free of solvent vapors.**

CAUTION: Use methanol or other organic solvents sparingly and work under a fume hood or in a well-ventilated area away from where an open flame or sparks can occur. Wear safety gloves, glasses, and apron.

b. Use pesticide-grade methanol (or appropriate organic solvent) dispensed from a methanol fluorocarbon-polymer wash bottle (office laboratory) or pumped through tubing (field site) (see TECHNICAL NOTE below).

i. Rinse equipment exterior and interior with a minimum amount of methanol.

ii. Rinse interior of pump tubing with methanol.

- Do not rinse exterior of pump tubing with methanol.
- **Do not rinse pump tubing with methanol or any organic solvent if TOC, DOC, or SOC samples will be withdrawn through that tubing.**

[4]Current (1998) cleaning protocol dictates the use of methanol to remove contaminants from equipment to be used to collect samples for analysis of organic compounds. This protocol is under review.

iii. Place equipment components and tubing on a clean aluminum foil surface.

iv. Pour or discharge used methanol (or other organic solvent) into an appropriate waste container for flammable liquids (Water Resources Division Memorandum 94.007). Change gloves. Dispose of gloves used for methanol rinse appropriately.

TECHNICAL NOTE: Rinse with dichloromethane or hexane if the methanol rinse is not sufficient to clean equipment contaminated with excessive concentrations of hydrophobic organic compounds. If rinsing with dichloromethane or hexane, use pesticide-grade solutions, wear nitrile gloves, and use only on dry equipment (dichloromethane and hexane are not soluble in water). Do not rinse equipment with any organic solvent if equipment will be used for TOC, DOC, or SOC samples.

Step 5. Air dry equipment or rinse with organic-grade water (fig. 3-4).

a. Allow methanol-rinsed equipment to air dry in an area free from dust and potential airborne contaminants (place an aluminum foil tent loosely over the drying equipment).

b. If it is not practical for the methanol to evaporate from the interior of equipment components or sample tubing, either

- dry by blowing clean, filtered, inert gas through equipment; or
- rinse methanol from equipment with pesticide-grade or volatile-grade blank water, dispensed from a wash bottle or pumped with a valveless fluid metering pump.

Storage of clean equipment

Cover all equipment orifices with aluminum foil or fluorocarbon polymer bags, then place equipment into sealable storage bags. Isolate equipment used to collect trace-element samples from aluminum foil.

SPECIFIC PROCEDURES FOR CLEANING SELECTED TYPES OF EQUIPMENT 3.3

By A.J. Horowitz, M.W. Sandstrom, and F.D. Wilde

The equipment-cleaning steps described in sections 3.2.1 and 3.2.2 apply to most, but not all, equipment. This section describes the cleaning procedures needed for specific equipment for which the general protocols are modified or do not apply, or for which more detailed instructions might be useful. Wear appropriate disposable, powderless gloves throughout each cleaning procedure, changing gloves with each change in cleaning solution and as described in section 3.2.

INORGANIC-SAMPLE BOTTLE CLEANING PROCEDURES 3.3.1

Bottles for samples to be analyzed for inorganic constituents include translucent colorless polyethylene, opaque brown polyethylene, and transparent glass bottles.Translucent polyethylene bottles that were acid rinsed at the laboratory should arrive capped with colorless, translucent plastic caps. Glass bottles for samples for mercury analysis also are acid rinsed and should arrive capped.

- **Discard acid-rinsed bottles that are received uncapped.**
- A cleaning procedure is required for bottles that will contain samples to be analyzed for trace elements and is recommended for bottles that will contain samples to be analyzed for major ions and nutrients.

Before leaving for the field, clean polyethylene and glass sample bottles, including acid-rinsed bottles, as described in the steps that follow:

1. Put on powderless, vinyl gloves.
2. Fill each bottle about one-quarter full of DIW and cap.
3. Shake vigorously and decant DIW.

4. Repeat the DIW rinse (Steps 2 and 3 above) two more times.
5. Following the last rinse, fill each bottle half full with DIW and cap the bottle.
6. Rinse exterior of bottle with DIW and dry with lint-free laboratory tissue.
7. Store bottles in doubled plastic bags.

3.3.2 CHURN SPLITTER CLEANING PROCEDURES

Plastic churn splitters are used primarily for samples to be analyzed for inorganic constituents (NFM 2). Avoid the need to field-clean the churn splitter by using a separate, precleaned churn splitter at each field site to be sampled, if possible.

When using the detergent wash/tapwater rinse for the churn splitter—Office-laboratory procedure (fig. 3-2, Step 2):

1. Fill churn splitter through the funnel with detergent solution.
2. Soak for 30 minutes.
3. Scrub interior and exterior surfaces with a soft brush, taking care not to abrade the surface.
4. Pay particular attention to cleaning the paddle and the area around the spigot.
5. Make sure spigot and funnel are free of sediment, including fine particulates (clay), organic matter, and stains.
6. Drain some of the cleaning solution through the spigot before discarding the remaining solution.
7. Fill churn through the funnel splitter about one-third full with tapwater; swirl and shake churn vigorously to remove detergent residues. Allow tapwater to pass through the spigot.
8. Repeat rinse procedure until no bubbles remain in rinse water after the water is agitated.

When using the acid rinse for the churn splitter—Office-laboratory or field-site procedures (figs. 3-2 and 3-3, Step 4):

1. Do not allow acid solution to contact the outside of churn splitter, or the churn spigot.

2. Do not pass acid solution through the spigot.

3. Decant acid solution by pouring out of the top of the churn into the neutralization container.

When using the DIW rinse for the churn splitter—Office-laboratory or field-site procedures (figs. 3-2 and 3-3, Step 5):

1. Fill the churn splitter through the funnel with DIW to about one-third full.

2. Swirl the DIW vigorously and pour it out of the top of the churn into the neutralization container.

3. Repeat the fill-and-swirl procedures of 1 and 2 above at least twice, checking the pH of the DIW after each swirl with narrow-range pH indicator strips.

4. **Pass a portion of the DIW through the spigot only after the DIW pH equals or is greater than either 6.0 or the pH of the DIW before acidification. Pour the rest of the DIW into the neutralization container.**

For storage of a cleaned churn splitter—Office-laboratory or field-site procedures:

1. Package a clean, dry churn splitter in two new plastic bags and loosely tie or secure with a nonmetal clip. If a churn splitter must be packaged while wet, use within 1 to 3 days and (or) keep chilled to prevent bacterial growth.

2. Place entire package into the churn carrier.

3.3.3 CONE SPLITTER CLEANING PROCEDURES

The fluorocarbon-polymer cone splitter (NFM 2) is appropriate for splitting samples for inorganic or organic analyses. When cleaning the cone splitter (Office of Water Quality Technical Memorandum 97.03), pay particular attention to removing foreign material from threaded and hard-to-access parts. Field cleaning can be minimized by having separate, precleaned cone splitters available for each site and by keeping a supply of clean tubes to replace the used tubes for each site to be sampled.

When inorganic constituents will be analyzed in samples processed through the cone splitter:

Office laboratory. Follow the steps as described for figure 3-2.

Field site. Referring to figure 3-3:

1. Prepare the field site as described in section 3.2.1. Put on disposable, powderless gloves.
2. Rinse the splitter thoroughly with deionized water.
3. Inspect the cone splitter. If it looks dirty, is suspected of being contaminated, or was allowed to dry between field sites without a thorough DIW rinse, or if the splitter will be used for sampling both inorganic and organic analytes, use the detergent-wash option. Change gloves.
4. Acid rinse by passing 1 L of 5-percent HCl solution through the cone splitter. Collect used acid solution into a neutralization container. Change gloves.
5. Rinse the cone splitter with at least 3 L of deionized water. Collect the rinse solution into a neutralization container. Change gloves.
6. Allow the cone splitter to dry and then store in a clean plastic bag. Seal the bag and store in a second plastic bag or plastic storage container for transport to the next site. A cone splitter that is packaged into bags while wet should be used within 1 to 3 days and (or) kept chilled to prevent bacterial growth.

When organic compounds will be analyzed in samples processed through the cone splitter (fig. 3-4):

Office Laboratory. Follow the steps described for figure 3-4.

Field Site.

1. Prepare site as described in section 3.2.2. Put on appropriate disposable, powderless gloves; if a solvent will be used, select gloves that will withstand contact with the solvent.
2. Detergent wash and rinse equipment as described for figure 3-4.
3. Check equipment and sampling requirements. If splitter will also be used for inorganics sampling, follow acid-rinse directions before rinsing with methanol or other organic solvent.
4. Proceed with the methanol (or other organic solvent) rinse, if required (section 3.2.2).
   - **Do not use any organic solvent if the cone splitter will contact samples for analysis of TOC, DOC, or SOC.**
   - If samples processed through a splitter will be analyzed for TOC, DOC, or SOC, rerinse the splitter thoroughly to completely remove residues from the detergent wash. Use PBW, VBW, or other organic-grade water for the final rinse if complete methanol evaporation is impractical. If the cone splitter will not be used to process samples for inorganic constituents at the next site, wrap nozzle and other orifices in aluminum foil.

For storage of a cleaned cone splitter:

1. Allow the cone splitter to air dry.
2. Place the cone splitter into a clean plastic bag and seal.
3. Store in a second plastic bag or plastic storage container for transport to the next site.

If a cone splitter must be packaged while wet, use within 1 to 3 days and (or) keep chilled to prevent bacterial growth.

3.3.4 FILTRATION EQUIPMENT CLEANING PROCEDURES

Filtration equipment includes disposable capsule filters and various plate-filter and pressure-filter assemblies. Cleaning procedures for these types of equipment are described below.

3.3.4.A Disposable Capsule Filter Cleaning Procedure

The disposable capsule filter has a one-time use for processing samples to be analyzed for inorganic constituents but must be cleaned before use. The filter can be prerinsed in the office laboratory instead of at the field site as long as it is kept chilled and used in less than 1 day. After filtering the sample, clean or replace the sample-delivery tubing and discard the capsule filter. The cleaning procedure described below comprises sufficient cleaning of the filter for analysis of inorganic constituents at the parts-per-billion (ppb) concentration level (Horowitz and others, 1994).

To clean the disposable capsule filter, pump 1 L of DIW to the filter through precleaned tubing (section 3.3.5) as follows (refer to NFM 5.2.1.A for additional instructions):

1. Use Clean Hands/Dirty Hands techniques described in NFM 4. Remember: the Dirty Hands team member performs operations that are outside of the processing chamber and the Clean Hands team member performs operations that are inside the chamber. Put on disposable, powderless gloves.

2. In a processing chamber, remove the capsule filter from the protective bags. Attach pump tubing to the inlet connector of the capsule filter, keeping the tubing as short as possible. **Make sure the direction of flow through the capsule filter matches the direction-of-flow arrow on the side of the filter.**

3. Pump 1 L of DIW through the capsule filter; discharge waste rinse water through a sink funnel or to a toss bottle.
   - Operate the pump at a low speed.
   - Hold the capsule filter so the arrow is pointing up at an acute angle from the horizontal plane. (This expels trapped air from the capsule; do not allow water to spray onto chamber walls.)
4. Remove tubing from the DIW reservoir and continue to operate the pump in the forward, mid-range speed position to drain as much of the DIW that remains in the capsule filter as possible. While the pump is operating, shake the capsule filter to help remove any entrained DIW.
5. Detach the capsule filter from the peristaltic pump tubing, put into a clean, sealable plastic bag, and store chilled until ready for use at the next site.

Plate-Filter Assembly Cleaning Procedure 3.3.4.B

To clean filtration equipment used for samples to be analyzed for inorganic or organic analytes, consult sections 3.2.1 and 3.2.2, respectively. Use Clean Hands/Dirty Hands techniques, as appropriate (NFM 4).

- **Preclean in the office laboratory** one plate-filter assembly per site to be sampled, if possible, in order to save the time that would be needed to clean the plate-filter assembly during the field effort.
- **During the detergent wash and (or) DIW rinse,** pay particular attention to grooves and crevices, O-rings, and support structures for the filter, where sediment or organic matter might be lodged. Detergent wash and DIW rinse the pressure valve.
- **Remove and discard the used filter at the field site;** rinse the filter assembly immediately with DIW while still wet from filtering the sample, even if a clean filter assembly is available for the next site.

When field cleaning the plastic plate-filter assembly:

1. Disassemble the plate-filter assembly inside the processing chamber while it is still wet from the sample water and while wearing disposable, powderless gloves.

   a. Remove the used filter media carefully to avoid spilling any of the filter cake.

   b. Place the filter media into a sealable plastic bag. Seal and pass the bag out of the chamber. Change gloves.

2. DIW rinse all components of the plate-filter assembly, including the exterior and interior of the tubing and the pressure valve, dispensing the DIW from a wash bottle. Pay particular attention to grooves and crevices, O-rings, and support structures for the membrane filter, where inorganic or organic materials might be lodged. Change gloves.

3. Inspect the plastic plate-filter assembly. Use the detergent-wash option described in figure 3-3 (Step 2) if the filter assembly looks dirty, is suspected of being contaminated, or was allowed to dry after use without first rinsing thoroughly with deionized water.

4. Reassemble the plate-filter assembly, reattaching the piece of tubing to the outlet of the filter assembly and placing the discharge end of the tube through the drain or disposal funnel in the bottom of the processing chamber to the acid-neutralization container. Reconnect the filter assembly to the peristaltic pump with the sample tubing. Change gloves.

5. To acid rinse the plate-filter assembly, pump 1 L of 5-percent HCl solution (or 10-percent HNO_3) solution through the plate-filter assembly. Check that the acid solution is being discharged into the acid-neutralization container. Alternately squeeze and release the tubing at the outlet to force the acid solution to cover and rinse all interior surfaces of the filtration assembly. (Be careful not to force tubing from the outlet by squeezing tubing for too long.)

6. To DIW rinse the plate-filter assembly, pump 2 L of deionized water through the assembly, using the same squeeze-and-release method described above in 5 for the acid rinse. Ensure that all the rinse water is being discharged to the acid-neutralization container. After confirming that the pH of the acid rinse solution is greater than 6.0 or the original pH of the DIW, appropriately discard solutions from the neutralization container.

7. For storage, place the cleaned plate-filter assembly and tubing into clean double bags for temporary storage until use at the next site. If wet when bagged, store for no longer than 24 hours and (or) chill to prevent bacterial growth. The filter assembly must be dry if stored for more than 24 hours.

Always remove the used filter media from the plate-filter assembly before cleaning and storage.

When field cleaning the aluminum plate-filter assembly, use the general cleaning instructions in section 3.2.2 for figure 3-4, as follows:

1. Inspect the aluminum (or stainless steel) plate-filter assembly for damage or excessive contamination and replace if necessary.

2. Wearing disposable, powderless gloves, prepare the area to be used for cleaning the plate-filter assembly by lining the table or counter surface with aluminum foil.

3. Disassemble the filter assembly and remove the used glass-fiber filter media carefully to avoid spilling any of the filter cake. Place used filter media into a sealable plastic bag, seal the bag, and put aside for disposal. Place components of the plate-filter assembly and tubing into a washbasin for detergent. Change gloves.

4. Detergent wash by using a 0.1- to 0.2-percent nonphosphate-detergent solution. Scrub each component of the filter assembly with a soft brush to remove any adhering material such as oil and grease, sediment, algae, or chemical deposits. Pay particular attention to grooves and crevices, O-rings, and support structures for the glass-fiber filter, where inorganic or organic materials might be lodged. Pump detergent solution through tubing. Place components of the plate-filter assembly onto a clean, aluminum-foil-covered surface.

5. Discard detergent solution from basin, rinse basin with tapwater, and place components of the plate-filter assembly into the basin. Change gloves.

6. Rinse each component thoroughly to remove detergent residue, paying particular attention to grooves and crevices. Use a wash bottle filled with DIW or tapwater to rinse hard-to-reach places. Place rinsed components onto a dry section of clean aluminum foil or basin. Change gloves. If the assembly will be rinsed with

methanol or other organic solvent, change to disposable, solvent-resistant gloves, and place components of the filter assembly into a clean, solvent-resistant washbasin.

7. Rinse plate-filter assembly components with pesticide-grade methanol or an equivalent grade for other organic solvents. Do not methanol rinse any tubing or filtration assembly to be used for collecting or processing samples for TOC, DOC, or SOC analysis. The instructions for the methanol rinse apply also for use of any other organic solvent. **Rinse the equipment with methanol while outside of the field vehicle and downwind of sampling activity.**
   a. Dispense methanol from a fluorocarbon-polymer wash bottle. Rinse all sample-contacting surfaces of filter-assembly components and tubing over a solvent-resistant basin or waste container. **Methanol-laced rinse water must be collected into an appropriate waste container designed for flammable liquids.**
   b. Place methanol-rinsed equipment components onto a clean aluminum foil surface to air dry. (Cover equipment components loosely with an aluminum foil tent, if concerned about airborne contaminants.)
8. Reassemble the plate-filter assembly. Wrap nozzles with aluminum foil and seal filter assembly in plastic bags. Double bag for transport or for long-term storage.

3.3.4.C Pressure-Filter Assembly Cleaning Procedure

The cleaning procedures described in section 3.2.2 for figure 3-4 do not apply to the filtration assembly used for samples to be analyzed for DOC and SOC. The filtration assembly for processing organic-carbon samples is a gas-pressurized apparatus constructed of either stainless steel or fluorocarbon-polymer material.

- **Do not bring the pressure-filter assembly in contact with methanol or other organic solvent or organic-solvent vapors.**
- In general, office-produced organic-grade water that is prepared by being passed through appropriate columns to remove organic compounds is of adequate purity for cleaning this equipment. PBW or VBW also can be used. Office-produced organic-grade water, however, must not be substituted for blank samples.

▶ **Do not clean the pressure-filter assembly with detergent.** Exception: see Step 3 below.

When using office-laboratory or field-site cleaning procedures for cleaning the pressure-filter assembly:

1. Wearing disposable, powderless gloves, disassemble the pressure-filter assembly before it dries and place components into a clean washbasin. Change gloves.

2. Using office-produced organic-grade water, thoroughly rinse the pressure-filter assembly and place it into a washbasin or onto a clean surface. Generally, these steps are sufficient to field clean the pressure-filter assembly.

   - If necessary, use a soft-bristled toothbrush to remove sediment, chemical deposits, and other foreign material from threaded components, gaskets, O-rings, support screens, grooves, and nozzles. Take care not to scratch or mar inner surfaces when scrubbing.

   - Rinse the pressure-filter assembly thoroughly with office-produced organic-grade water or PBW or VBW.

3. If the pressure-filter assembly is very dirty or contaminated, clean as follows:

   a. Disassemble and soak assembly for at least 1 hour in a 0.1-percent solution of nonphosphate laboratory-grade detergent.

   b. Scrub with a soft-bristled toothbrush, as described above in 2.

   c. Rinse repeatedly with office-produced organic-grade water, being sure to remove all traces of detergent.

4. Place all components of the pressure-filter assembly onto aluminum foil and allow to air dry thoroughly under a protective aluminum foil tent.

5. Reassemble the pressure-filter assembly, wrap nozzles in aluminum foil, and seal in a storage bag.

Do not use methanol or other organic solvents on the equipment used to filter samples for organic-carbon analyses.

3.3.5 SAMPLE TUBING CLEANING PROCEDURES

Cleaning procedures are described below for the tubing and nozzles used with peristaltic and valveless metering pumps. Cleaning procedures for submersible pump tubing are described in section 3.3.9.B. Wear appropriate, disposable, powderless gloves throughout the cleaning process, changing gloves with each change in cleaning solution as indicated throughout section 3.2.

- Preclean the number of tubing sections needed at each site in the office laboratory rather than recleaning tubing in the field, in order to save time during field work. Place into doubled plastic bags and store tubing dry or store wet tubing chilled to prevent bacterial growth. If bacterial growth is present, reclean tubing before use.
- Use disposable tubing if possible, especially at contaminated sites, to avoid the cleaning process and prevent the possibility of cross contamination.

When using office-laboratory or field-site procedures for cleaning plastic (including fluorocarbon-polymer) sample tubing used for samples to be analyzed for inorganic constituents, follow the general sequence of procedures described for figures 3-2 or 3-3, and those described for filtration assemblies (section 3.3.4).

To summarize the key steps for figures 3-2 or 3-3:

1. Pump 1 L of 5-percent HCl solution through the tubing, discharging the used acid solution into a neutralization container. Pinch and release tubing near tubing outlet while pumping the acid through to ensure that all interior surfaces are acid rinsed.
2. Pump 2 L of DIW through tubing, using the same pinch-and-release method. Discharge used DIW to an acid-neutralization container, and check that the rinse-water pH is greater than 6.0 or the original DIW pH.
3. Discard neutralized solutions appropriately.
4. Clean stainless steel connections or metal tubing using detergent-wash and tapwater/DIW rinse procedures.

When using office-laboratory or field-site procedures for cleaning tubing for organic-compound samples:

Follow the general sequence of procedures described for figures 3-1 and 3-4. Proceed with the methanol rinse after the detergent wash and tapwater rinse. If samples also will be collected for inorganic-constituent analysis, however, acid rinse nonmetallic tubing and components after the detergent wash/tapwater rinse and before continuing to the methanol rinse. When cleaning sample tubing:

1. Pump 1 L of nonphosphate, laboratory-grade detergent solution through tubing, followed by sufficient tapwater or DIW to remove detergent residue. Pinch and release tubing near tubing outlet while pumping the solution to ensure that all interior surfaces are cleaned.
2. Place discharge end of tubing from peristaltic or valveless metering pump over methanol waste container.
   - Pass one tubing volume of methanol through the same pump system used for filtration, using the same pinch-and-release method.
   - Short sections of tubing can be held over the waste container while dispensing the methanol from a fluorocarbon-polymer wash bottle instead of pumping the methanol through the tubing.
   - **Do not methanol rinse tubing to be used for samples for TOC, DOC, or SOC analysis.**
3. Store tubing in doubled plastic bags.

CAUTION: Do not use methanol around equipment that can create electrical sparks (see section 3.3.9.B).

3.3.6 PROCESSING AND PRESERVATION CHAMBERS AND FLOWTHROUGH CHAMBER CLEANING PROCEDURES

Processing and preservation chambers used to protect samples from atmospheric contamination generally are portable and are assembled at the field site. Large, clear plastic bags usually are clipped to the inside of the frame rather than stretched over the frame. Plastic clips are used to hold the cover tightly in place. When the bag is clipped to the inside, it is not necessary to field clean the chamber frame.

The flowthrough chamber, used when monitoring ground-water field measurements, is connected inline to the pump sampler. The flowthrough chamber should be kept free of sediment and dirt or deposits on the chamber walls. Air dry and store the chambers in sealable plastic bags.

When cleaning the processing and preservation chambers:

Office laboratory. Clean the frame of portable chambers in the office with detergent solution, then rinse thoroughly with tapwater and dry and store in plastic bags.

Field site. Frames require regular cleaning after each use at a site if chamber covers are stretched over the outside of the frame rather than clipped to the frame.

1. Discard the used bag.
2. Wipe the chamber frame with DIW.
3. Replace chamber cover only when the next samples are ready to be processed.
4. If the processing chamber is a fixed installation, clean out any spilled sample water, solid materials, or wash solutions, and swab down the inside using deionized water and lint-free laboratory tissue.
5. Use detergent solution followed by a thorough tapwater or DIW rinse if a spill has contaminated the chamber.
6. Store chamber frames in plastic bags.

When cleaning the flowthrough chamber:

1. Clean the flowthrough chamber in the office laboratory with detergent solution and rinse thoroughly with tapwater, followed by DIW. **Do not use acid solution or methanol.**
2. If the flowthrough chamber needs to be field cleaned, remove measurement sensors and clean with a dilute detergent solution; rinse thoroughly with tapwater followed by DIW.

RADON SAMPLER CLEANING PROCEDURE 3.3.7

Soak radon samplers in a detergent solution for 10 minutes and rinse thoroughly with tapwater to remove detergent residue; follow with three to five rinses with DIW. **Do not use methanol.** Air dry the radon sampler and store in doubled plastic bags.

3.3.8 SURFACE-WATER SAMPLER CLEANING PROCEDURES

Disassemble surface-water samplers for cleaning and follow the sequence of procedures described in section 3.2 and figures 3-2, 3-3, or 3-4, as appropriate.

When using office-laboratory procedures for cleaning surface-water samplers:

1. Periodically disassemble samplers for office-laboratory cleaning. **Discard the bag sampler bag after one use**—do not attempt to scrub or detergent wash the used bag. Prepare cleaning solutions, cleaning equipment, and cleaning area as described in section 3.2.

2. Soak components in detergent solution for 30 minutes. Put on appropriate disposable, powderless gloves. Scrub components with a soft brush or sponge and rinse thoroughly (section 3.2.1 or 3.2.2). Change gloves.

3. Check the sequence of cleaning procedures shown in figure 3-1.

   a. If the sampler is used for sampling inorganic constituents, soak each nonmetallic component in a 5-percent trace-metal-grade HCl solution for 30 minutes, followed by copious rinsing with DIW (section 3.2.1). **Acid rinse only nonmetal parts.** Change gloves.

      - Acid must not contact the metal collar on the DH-81 sampler.
      - Make sure that the nozzle is unscrewed from the cap.

   b. If the sampler is used for collecting organic-compound samples, rinse each component with pesticide-grade methanol dispensed from a fluorocarbon-polymer wash bottle and allow to air dry (section 3.2.2). **Do not methanol rinse tubing or components that will contact TOC, DOC, or SOC samples.** Change gloves.

4. If collecting an equipment blank (section 3.4), change gloves and rinse each component with the appropriate blank water before collecting the blank sample.

5. Reassemble the sampler. If the sampler is dedicated to sampling for organic compounds, double wrap the sampler nozzle in aluminum foil. Place the sampler into double plastic bags and seal for storage and transport.

When using field-site procedures for cleaning surface-water samplers:

1. Unwrap precleaned washbasins (one for each cleaning solution to be used).

2. Disassemble the used sampler into its component parts (bottle, cap, nozzle) so that all of the pieces can be thoroughly wetted with the various rinses. **Discard the previously used bag-sampler bag** (do not attempt to clean it for reuse).

3. Wearing appropriate disposable gloves, thoroughly rinse the sampler components with DIW. Use a stream of DIW from the wash bottle, if required.

4. Check whether target analytes are inorganic constituents, organic compounds, or both. Review figure 3-1 for the appropriate cleaning sequence.

   a. If a sampler will be used for collecting samples for analysis of inorganic constituents only, change gloves and

      i. Thoroughly rinse the sampler components with tapwater or DIW.

      ii. Acid rinse nonmetallic components over a container using a stream of dilute acid solution from the appropriate wash bottle, if required.

      iii. Thoroughly rerinse the sampler components with DIW over the same washbasin, if possible (see section 3.2.1). Change gloves.

      iv. Place each component on a clean, plastic surface. Pour used acid solution and DIW rinse water into neutralization container.

      v. Check the pH of the solution in the neutralization container. Discard when solution pH is greater than 6.0 or the original DIW pH. Change gloves.

   b. If a sampler will be used for collecting samples for analysis of organic compounds only, change gloves and

      i. Detergent wash, then rinse sampler components thoroughly with tapwater or DIW until agitated rinse water produces no more suds. Change to solvent-resistant gloves.

      ii. Rinse sampler components with pesticide-grade methanol (section 3.2.2), collecting the used methanol into an appropriate container for safe storage until appropriate disposal is arranged.

iii. Place each component on a clean, aluminum-foil-covered surface to air dry and cover loosely with an aluminum foil tent, if airborne contaminants are a concern. Change gloves.

c. If sampler will be used for collecting samples for both organic and inorganic analyses, change gloves and

i. Proceed with a detergent wash and thorough tapwater and (or) DIW rinse.

ii. Acid rinse and DIW rinse nonmetallic components, as described above, discarding used solutions appropriately. Change to solvent-resistant gloves.

iii. Rinse with methanol, if needed, as described above.

iv. Place cleaned items on a clean plastic surface to air dry.

5. **Reassemble sampler. If the sampler is dedicated to sampling for** organic compounds, double-wrap sampler nozzle in aluminum foil. Place sampler into doubled plastic bags for storage and transport.

Do not use methanol or other organic solvents on equipment used to collect organic-carbon samples.

GROUND-WATER SAMPLER CLEANING PROCEDURES 3.3.9

Ground water is sampled with nonpumping samplers (such as bailers, syringe samplers, and the Kemmerer sampler) and with pumping samplers (such as peristaltic and valveless metering pumps and submersible pumps). Office-laboratory cleaning procedures are used before a sampler is used for the first time, after the sampler has been in long-term storage, and whenever the sampler has become excessively contaminated. Field-site cleaning procedures are used after sampling at a field site and before proceeding to the next sampling site. Caveats and modifications that apply to the general office-laboratory and field-site cleaning procedures (section 3.2) are described in this section. The cleaning procedures used should be documented on field forms.

The rinse with methanol, or other organic solvent, is under review and appropriate only for samplers being used to collect samples for organic-compound analysis. **Solvents are never used to clean equipment when sampling for TOC, DOC, or SOC.** Dispose of used methanol and all other cleaning solutions appropriately.

> TECHNICAL NOTE: Sampler components made of fluorocarbon-polymer plastic generally can withstand a solvent rinse with methanol. Check with the manufacturer before using an organic solvent on pump components constructed of any other plastic material.

3.3.9.A Cleaning of Bailers and Other Nonpumping Samplers

Office-laboratory procedure. Clean nonpumping samplers in a designated area of the office laboratory. Follow the procedures described for figures 3-2 and 3-4, as appropriate for equipment used to sample for inorganic constituents or organic compounds, respectively.

Field-site procedure. Follow the field-site cleaning procedures described for figures 3-3 and 3-4, as appropriate for equipment used to sample for inorganic constituents or organic compounds, respectively.

- Rinse the outside of the sampler with DIW directly after use.
- After filling the sampler with each cleaning solution, shake the sampler vigorously and drain solution through the bottom-emptying device, spigot, or nozzle of the sampler.
- If the sampler looks very dirty or is contaminated, disassemble and clean sampler components using the office-laboratory procedure.

3.3.9.B Cleaning of Submersible Pumps and Submersible-Pump Tubing

The general sequence shown in figure 3-5 is appropriate for cleaning most submersible pumps. The field-site cleaning procedure (described below after the office-laboratory procedure) is sufficient for routine cleaning of the pump in most cases. Collection of blank samples for quality control must be included as a standard protocol for every study in order to document and ensure the efficacy of the cleaning procedure for the field conditions encountered.

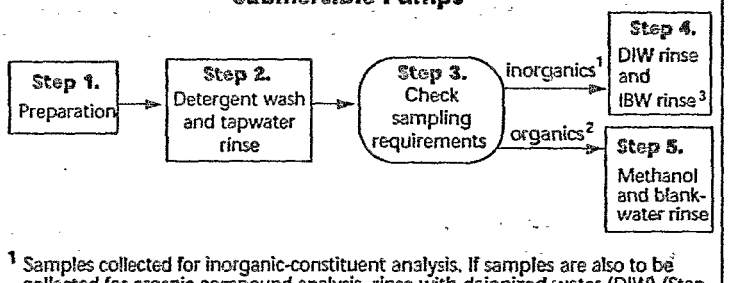
- Fluorocarbon-polymer tubing used to collect water containing large concentrations of volatile organic compounds (VOCs) can be difficult to clean adequately.

- Collect additional blanks if VOC concentrations in last sample collected through the tubing were greater than 500 µg/L.
- Pump tubing should be replaced rather than cleaned if VOC concentrations in last sample exceeded about 700 µg/L.

▶ Most submersible pumps have a stainless steel casing and other metal parts and should not be acid rinsed.

- To clean pumps that are excessively contaminated, a dilute acid rinse followed by copious water rinsing can be used occasionally without damaging the pump.
- Repeated rinsing with dilute acid solution can pit or corrode the pump's stainless steel surface. If the surface appears dulled, the pump must not be used for collecting trace-metal samples.

▶ Lubrication water inside water-lubricated pumps (for example, the Grundfos RediFlo2™) can become contaminated and cause contamination of subsequent samples. Replace the lubrication water with VBW each time after sampling and when cleaning the pump. Follow manufacturer's instructions.



[1] Samples collected for inorganic-constituent analysis. If samples are also to be collected for organic-compound analysis, rinse with deionized water (DIW) (Step 4), then methanol (Step 5).

[2] Samples collected for organic-compound analysis. Methanol rinse procedure is under review. When sampling for total, dissolved, or suspended organic carbon, do not methanol rinse; complete cleaning by rinsing with organic-grade water.

[3] A rinse with inorganic blank water (IBW) is needed only if collecting a blank sample for inorganics analysis directly after cleaning the pump.

Figure 3-5. Cleaning procedures for submersible pumps.

Office-laboratory pump-cleaning procedure:

Use office-laboratory procedures about once a year and more frequently if results of the pump blank or other information indicate that the pump is contaminated.

Step 1. Preparation.

a. Wearing appropriate gloves, prepare several gallons of a laboratory-grade nonphosphate detergent solution (about 0.1 or 0.2 percent, v/v; use up to 2-percent solution for excessively contaminated pump systems).

b. Preclean washbasins and standpipes (section 3.2).

c. Place pump into sink or waste basin and scrub exterior surfaces with soft brush and detergent solution; rinse thoroughly with tapwater.

d. Disassemble the pump and place components into a detergent-solution washbasin.

Step 2. Detergent wash and tapwater rinse pump components and tubing.

a. Soak pump components in the detergent solution for 30 minutes.

b. Scrub pump components with soft sponge or brush.

c. Rinse thoroughly with tapwater.

d. Raise discharge end of tubing above the rest of the tubing. Using a peristaltic or valveless fluid metering pump, fill the pump tubing with fresh detergent solution until solution rises to the end of the tubing. Plug the tubing end(s).

e. After 30 minutes remove plug from discharge end of tubing and flush detergent solution from tubing by pumping copious amounts of tapwater through the tubing. Change gloves.

Step 3. Check sampling requirements.

- If pump will be used for collecting samples for inorganic-constituent analysis, reassemble the pump and go to Step 4.
- Complete Step 4 if pump will be used for collecting samples for analysis of both inorganic and organic analytes before proceeding to Step 5.
- If the pump will be used for collecting samples for organic-compound analyses only, go to Step 5.

Step 4. DIW rinse.

a. Place pump components into DIW washbasin and dispense DIW from a wash bottle to thoroughly rinse all pump components.

b. Using a peristaltic pump and appropriate clean tubing, pump DIW through the sample tubing to rinse.

c. Reassemble pump and connect pump tubing. Change gloves.

d. If collecting equipment blanks to verify that the pump has been adequately cleaned (section 3.4):

   i. Rinse a clean standpipe dedicated to blank water with blank water.

   ii. Insert pump into blank-water standpipe only after pump exterior has been rinsed with blank water or air dried after the methanol rinse.

   iii. Pour IBW into the standpipe and pump at least one tubing volume to waste before collecting the blank sample.

Step 5. Rinse with blank water followed by a methanol rinse.

a **Change to latex or nitrile gloves.** Put pump components into solvent-resistant washbasin.

b. Working under a fume hood, dispense methanol (or appropriate solvent) from a fluorocarbon-polymer wash bottle to rinse each pump component and the exterior pump casing. Collect the used solvent into a nonflammable container for storage until disposal.

- **Do not reuse methanol or other solvents.**
- **Work under a fume hood, if possible, or in a well-ventilated area outside of the office laboratory, as methanol fumes can contaminate other equipment.**

c. Place methanol-rinsed components on a clean, aluminum foil surface and allow the pump components and casing to completely air dry before reassembling the pump (see section 3.2.2).

d. Using a valveless fluid metering pump and fluorocarbon-polymer tubing, pump about 2 L of methanol through sample tubing and to the methanol waste container.

e. Reassemble the pump and connect the pump tubing. Change gloves and dispose of the methanol-contaminated gloves appropriately.

f. Pour an organic-grade water (PBW or VBW) into a clean PBW/VBW standpipe. Insert pump and pass about two tubing volumes of organic-grade blank water (PBW or VBW) through the pump and tubing to waste.

CAUTION: Pumping methanol or other flammable solvents through an electrical pump system could be dangerous in the event of sparks. Methanol emits noxious fumes and is absorbed through the skin. Wear a mask, safety glasses, and other protective apparel to protect yourself when working with organic solvents.

Field-site cleaning procedure for submersible pumps and pump tubing:

Step 1. Preparation.

a. Preclean the standpipes (one standpipe for each cleaning solution to be used, as described in 3.2.1). The standpipes need to be of sufficient height to supply necessary head for proper pump operation. Separate standpipes are designated for detergent solution and tapwater rinse, DIW rinse, methanol rinse, and blank water (IBW/PBW/VBW). Double-bag each cleaned standpipe for transport to the field site.

b. Estimate the volumes of cleaning solutions and blank water that will be needed for the field effort (refer to fig. 3-6).

c. Prepare the volumes of cleaning solutions needed for the field effort, using appropriate bottles for short-term storage and transport.

The volume of storage in tubing, V_s, of a set of pump-reel and extension tubing can be estimated[1,2] as follows:

$$V_s = [(L_p \times C_p) + (L_e \times C_e) + V_{sp}] \times C_{sp}$$

where,

V_s is volume of storage in tubing, in gallons
L_p is length of pump-tubing segment being cleaned, in feet
L_e is length of extension tubing, in feet
C_p (or C_e) = 0.023 liter per foot for a 3/8-inch inside-diameter (ID) tubing
or = 0.041 liter per foot for a 1/2-inch ID tubing
V_{sp} is volume of solution needed to fill standpipe to minimum level required to operate pump, in liters[1]
C_{sp} = 0.264 gallon per liter.

Examples

Given:

1. L_p - sample-wetted tubing segment is 100 feet for a pump-reel system that has a 1/2-inch ID tubing;
2. L_e - two, 10-foot, 3/8-inch-ID pieces of extension tubing, one running from pump-reel outlet to sample collection chamber, and another running from chamber back to pump-reel (return-flow tubing to standpipe); and
3. V_{sp} - minimum volume[1] of solution required in standpipe to operate pump is 0.8 liter.

To estimate the volume of detergent solution needed for the detergent wash cycle:

$$V_s = [(100 \times 0.041) + (20 \times 0.023) + 0.8] \times 0.264 = 1.4 \text{ gallons}$$

The volume of office-produced deionized water needed to displace detergent solution and the volume of laboratory-produced organic-grade blank water needed to displace 2 liters of methanol just pumped into a system, ideally, would each be estimated to equal V_s[1,2].

[1]Estimate assumes no mixing of two solutions and ignores potential for detergent to adhere to tubing walls. Outflow from the discharge end of tubing should be checked for sudsing to determine that detergent has been removed.

[2]Estimate assumes no mixing at interface of two solutions and ignores potential for methanol to adhere to tubing walls. It is recommended that an additional 0.1 gallon (~ 0.4 liter) of blank water (pesticide-grade blank water or volatile-grade blank water) be used for each 10 feet of tubing to remove methanol residues from sample-wetted sections of tubing. Thus in the example above, another 1.1 (= (100 + 10) x (0.1/10)) gallons (4.2 liters) of blank water would be pumped from the system. This implies a total of about 2.5 (= 1.4 + 1.1) gallons (9.6 liters) of blank water would be used to remove methanol from the equipment setup.

[3]The minimum volume corresponds to the level of solution in the standpipe, which, if maintained, allows pump to operate without introducing air through the pump intake. Once this level is reached, remove pump, and measure this volume.

Figure 3-6. Estimation of cleaning-solution volumes for standpipe, pump, and pump tubing. [From Koterba and others, 1995, table 24.]

Step 2. Detergent wash and tapwater rinse.

a. Put on disposable, powderless gloves (usually vinyl). Rest pump in a washbasin or pail partially filled with detergent solution and clean exterior of pump and tubing with a soft brush. Rinse thoroughly with tapwater. (DIW can be substituted for tapwater, but is less efficient in detergent removal and requires a greater volume of water than tapwater.)

b. Place pump into standpipe, add detergent solution to level above pump intake, and route intake and discharge end of pump tubing to the standpipe.

c. Begin pumping:

   i. Record the pumping rate.

   ii. Record the time it takes to fill the sample tubing.

   iii. Calculate the time it takes for a segment of solution to complete one cycle (fig. 3-6).

d. Circulate detergent solution for about three cycles through the tubing and back to the standpipe. If possible, pump detergent solution through tubing at alternating high and low speeds, and (or) introduce air segments between aliquots of the detergent solution to increase cleaning efficiency.

e. Remove the discharge end of tubing from the standpipe and pump about two tubing volumes of detergent solution to waste, adding fresh solution to the standpipe as needed. Remove pump from standpipe.

f. Rinse detergent from standpipe with tapwater until sudsing stops.

g. Rinse pump exterior with tapwater. Place rinsed pump into standpipe; add tapwater/DIW to level above pump intake. Begin pumping through sample tubing. Do not recirculate rinse water, but add water as needed to maintain water level above pump intake. Continue for five or more tubing volumes. Direct rinse water to waste, away from the vicinity of the wellhead and sampling area and (or) contain as required for disposal.

h. Collect rinse water into a small bottle and stop the pump. Shake the bottle—if sudsing is observed in the rinse water, continue the rinse procedure until no suds appear in the rinse water. Change gloves.

Step 3. Check sampling requirements.

— If a pump will be used to collect samples for inorganic-constituent analysis, go to Step 4.

— Complete Step 4 if a pump will be used to collect samples for analysis of both inorganic and organic analytes and go to Step 5.

— If a pump will be used to collect samples for organic-compound analysis only, go to Step 5.

Step 4. DIW rinse.

A separate DIW rinse is not required if DIW was substituted for tapwater.

a. Use a clean DIW-dedicated standpipe, not the tapwater standpipe, and rinse with DIW. Rinse pump exterior with DIW to remove any detergent residue. Place pump into the DIW standpipe and add DIW to level above pump intake. Change gloves.

b. Start pumping DIW. Rinse DIW through sample tubing without recirculating, using about 3 tubing volumes of DIW. Keep the DIW level above pump intake.

c. Collect DIW rinse water in a clean bottle, shake, and check for suds. Continue to DIW rinse until rinse water is free of suds.

d. If collecting field blanks to verify that the pump has been adequately cleaned (section 3.4):

   i. Change gloves. Rinse clean blank-water standpipe with IBW. Rinse pump exterior with blank water.

   ii. Place pump into the standpipe and add IBW to cover the pump intake.

   iii. Turn on pump and displace any water residing in the pump and tubing. Continue pumping IBW for one tubing volume before collecting the blank sample.

Step 5. Methanol rinse.

Make certain that the pump or other nearby electrically powered equipment is grounded, the power cord is intact, and potential sources of sparks do not exist before rinsing pump with methanol.

TECHNICAL NOTES:

- Inspect the integrity of the seals and O-rings on the pump-motor/pump-body housing. Water inside the motor housing may indicate that methanol vapors could enter the motor. Direct-current motors inherently spark because of the commutator ring. AC motors might spark if the insulation is frayed or burnt on the motor windings or any associated wiring.

- If flammable liquids are required for cleaning electrical pump systems, use extreme caution. Vapors from solvents such as methanol can ignite if a disruption in the motor lead-insulation system occurs in the vapor-enriched zone. (Ignition from a spark from an AC induction-type motor in good operating condition is not a concern if rated as using the National Electrical Code (NEC) at Class 1, Group D.[5])

a. Change to latex or nitrile gloves. Wear safety glasses and apron. Work in a well-ventilated area outside of the field van and downwind of the sampling area.

b. Place pump into a clean, dedicated, solvent-resistant standpipe and route discharge end of sample tubing to a methanol waste container. Add methanol solution to level above pump intake.

c. Pump about 2 L of methanol through sample tubing into methanol waste container, keeping the level of solution above pump intake. The operator should stand back from the pump as a safety precaution in the event that an electrical spark ignites the methanol. Carefully put any unused methanol from bottom of standpipe into methanol waste container. Let methanol in the standpipe evaporate to dryness. Change gloves.

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[5]NEC Class 1; Group D: Areas in which flammable gases or vapors may be present in the air in sufficient quantities to be explosive; atmospheres such as acetone, alcohol, ammonia, benzene, benzol, butane, gasoline, hexane, lacquer solvent vapors, naphtha, natural gas, propane, or gas or vapors of equivalent hazard (Cole-Parmer Instrument Company, 1997).

d. Rinse pump exterior with organic-grade water and place pump into standpipe. Add organic-grade water to the standpipe to push the methanol out of the tubing and into the methanol waste container. Pump at least an additional 0.1 gallon (about 0.38 L) of organic-grade water through the system for every 10 ft (about 3.05 m) of methanol-wetted tubing to the methanol waste container after used methanol is collected.

TECHNICAL NOTE: The recommended organic-grade water is PBW or VBW (supplied by NWQL for blank samples). Office-produced organic-grade water might not be of adequate purity, especially after being stored, and its use requires collection of additional blank samples for quality control (see section 3.4).

e. Repeat d above with blank water (PBW or VBW) pumped from a blank-water standpipe if blank samples will be collected for analysis of organic compounds.

Use of methanol is not recommended as a routine procedure for field cleaning of the pump. A methanol rinse is most safely accomplished as an office laboratory procedure.

Storage of the cleaned submersible pump and tubing:

1. Place pump into two clean, noncontaminating storage bags and close bags.
2. Cover the pump reel and tubing with doubled plastic bags or sheeting for transport to the next site.

For long-term storage (longer than 3 days), the pump and exterior and interior of the tubing must be dry before being placed into plastic bags. Tubing can be dried by blowing filtered air or filtered (inert) gas through the tubing. If tubing cannot be dried, store chilled to prevent bacterial growth. If bacterial growth has occurred, reclean before use.

3.4 QUALITY CONTROL FOR EQUIPMENT-CLEANING PROCEDURES

By A.J. Horowitz, M.W. Sandstrom, and F.D. Wilde

Quality-control samples are required for any sampling and analysis program. Without quality-control information, the quality of the environmental data collected can be neither evaluated nor qualified. If the user has no means of knowing the associated errors, the data cannot be interpreted properly.

The purpose for obtaining quality-control (QC) samples following equipment cleaning is to ensure that the equipment and the procedures used for cleaning the equipment do not contaminate or otherwise affect the environmental samples that were or will be collected. The QC sample used to assess the adequacy of cleaning procedures before field work commences is called the equipment blank.

- **Blank water.** Blank water is used to develop specific types of QC samples (National Water Quality Laboratory Memorandum 92.01). The water is a solution that is free of analyte(s) of interest at a specified detection level. USGS personnel are required to use blank water that has been analyzed and certified to be of a specific grade and composition.
  - Use IBW to collect blank samples for analysis of inorganic constituents.
  - Use PBW to collect blank samples for analysis of pesticides. (Do not use PBW when collecting samples for VOC analysis.)
  - Use VBW to process blank samples for analysis of VOCs. VBW is also suitable as a blank sample for pesticide analysis.
  - Use PBW or VBW as the quality-control sample for total and dissolved organic-carbon analysis (TOC and DOC). This cannot be documented as a blank sample because neither PBW nor VBW is certified to be free of organic carbon.

- **Equipment blank.** An equipment blank is blank water that is processed under controlled conditions in the office laboratory by being passed sequentially through each component of the sample processing and collection equipment. **An equipment blank represents an entire sampling system (fig.3-7) and is required:**
  - Annually.
  - When a cleaning procedure is followed for the first time.
  - When new equipment will be used for the first time.

To fulfill equipment-blank requirements:

1. Allow enough time in the study workplan to collect the annual equipment blank, complete laboratory analyses, and review analytical results before field work for the study commences.
2. Process the annual equipment blank in a clean, controlled environment in the office laboratory, after the equipment has been cleaned using office-laboratory procedures.
3. Analyze the annual equipment-blank data before collecting and processing the first water-quality sample of either the fiscal year or the study.
   - If the equipment-blank data indicate that the equipment does not introduce contaminants that will bias study results, sampling can proceed.
   - If the equipment-blank data indicate unacceptable concentrations of analytes of interest, the cause must be identified and the equipment or cleaning procedures must be changed or modified before sampling can proceed.

Plan ahead. Assess equipment-blank data before environmental samples are collected.

- **Field blank.** The field blank is blank water that is processed at the field site by being passed sequentially through each component of the equipment being used to collect environmental samples. The procedure for processing the field blank, like the equipment blank, can also result in a set of sequentially collected blank samples (fig. 3-7) (Horowitz and others, 1994). Other types of blank samples also are collected at the field site (NFM 4). **At least one field blank per sampling run is recommended; the numbers and distribution of QC samples depend on study objectives, the target analytes, and site conditions.**

  - Process field blanks through clean equipment.
  - If equipment is used at several sites during a field trip, process a field-equipment blank after the last sample has been collected and again after the equipment has undergone the prescribed field-cleaning procedures.
  - If multiple sets of office-cleaned equipment are used during a field trip, process a field blank at any site during the course of the trip. In this case, the blank must be processed before sampling to avoid contaminating the blank with residues from an environmental sample.
  - Process field blanks onsite and under the same conditions as the environmental sample.

Before filling the QC sample bottle with the appropriate blank water:

1. Check that sample bottles are clean, are the correct type, and are labeled correctly.
2. Check the certificate of analysis for the lot of blank water to be sure that it is appropriate for quality control of target analytes.
3. Record the date and lot number of the IBW, PBW, and (or) VBW used and of the preservative used. To the extent possible, use preservative from the same lot number for an entire sampling trip for both the environmental and quality-control samples.
4. Rinse sample bottles for inorganic constituents three times with a small quantity of the blank water.

Use the following strategy for QC data collection and analysis:

1. For inorganic-constituent samples, initially send only the final equipment-blank sample for the routine inorganic blank-sample analysis or for inorganic analytes targeted by the study.

   - Archive the remaining sequentially processed blank samples (fig.3-7) until the inorganic-constituent analysis of the equipment-blank sample has been received.

   - Do not archive blank samples for organic-compound analysis.

2. Check the analytical results for the equipment blank and field blanks as soon as possible and before the next field trip.

   - If analytical results indicate that the equipment is clean within acceptable limits, the equipment may be used for field work without additional testing or analysis.

   - **Use of equipment is not recommended if analysis of the equipment blank sample indicates greater than acceptable analyte concentrations.**

3. Additional QC data collection and (or) analysis is required if the equipment blank has greater than acceptable analyte concentrations.

   - **For inorganic-sample analysis.** Submit the rest of the sequential blank samples for laboratory analysis and use the analytical results from the sequential blank samples to identify potential source(s) of contamination. Modify equipment-cleaning procedures if contamination can be remedied by a change in cleaning procedure. Repeat collection of equipment blanks until the blank data verify that the equipment is suitable for use.

   - **For organic-sample analysis.** Modify the equipment cleaning procedure if the source of contamination is known or suspected and contamination can be remedied by a change in cleaning procedure. If the source of contamination is not known, reclean equipment using office-laboratory procedures and collect and analyze blanks for each part of the sampling system that could be a source of contamination. Repeat collection of equipment blanks until the blank data verify that the equipment is suitable for use.

The equipment blank is the last sample of a set of sequentially processed blanks collected in the office laboratory and documents the suitability of the equipment for the samples that are to be collected and analyzed. **Field blanks** are collected in the field in the same manner as the equipment blank but document the effectiveness of the field-cleaning procedures plus any ambient contamination.

- Surface water: collect the series of five sequential blank samples listed below for routine surface-water sampling.
- Ground water: collect the source-solution blank (Sample 1) and either a sampler blank (Sample 2) or pump blank (Sample 4) (depending on the type of sampling device being used) along with the filter blank (Sample 5) .

Sample 1. SS blank	**Source solution (SS)** Put on disposable gloves. Pour the IBW, PBW, or VBW directly into appropriate SS blank-sample bottle.[1] Add chemical treatment and (or) chill, as required for the analytes of interest.
Sample 2. Sampler blank	**SS + Sampler** Bottle or bag sampler: Fill sampler container with SS; attach sampler cap and nozzle; decant sample into blank-sample bottle through the nozzle. Preserve sample (add chemical treatment and (or) chill) as required (NFM 5). Bailer or thief sampler: Fill sampler with SS; install bottom-emptying device; empty sample into blank-sample bottle through the bottom-emptying device. Preserve sample, as required. Submersible or nonsubmersible pumps: Go to Sample 4 (Pump blank).
Sample 3. Splitter blank	**SS + Sampler + Splitter[2]** If a cone or churn splitter is used, decant remainder of the SS into sampler container, and then through splitter (through nozzle or bottom-emptying device). Refill sampler container with SS to fill churn with 3 to 5 liters of water. Alternatively, pour enough SS from samplers through cone splitter to fill splitter-blank bottle. Collect SS into blank-sample bottle through churn spigot or cone-splitter exit port(s). Preserve sample, as required.
Sample 4. Pump blank	**SS + Sampler + Splitter + Pump** Nonsubmersible pump (peristaltic, vacuum, or valveless metering pump): Secure intake end of clean pump tubing into churn splitter or into a subsample split with the cone splitter. Pump some sample to waste to rinse tubing, and fill pump-blank bottle directly from the discharge end. Preserve sample, as required. Submersible pump: Place pump in blank-water standpipe and fill standpipe with enough SS to cover pump intake and allow for drawdown. Start pump at low pumping rate, discharge 0.5 liter of SS to waste, then fill blank-sample bottle with SS. Preserve sample, as required.
Sample 5. Filter or equipment blank	**SS + Sampler + Splitter + Pump + Filter** Pump SS through a prerinsed filtration assembly (plate filter or capsule filter); pump the first aliquot to waste and then pump SS directly into the blank-sample bottle. Preserve sample, as required.

[1]Process the source-solution blank in the protected environment of the office laboratory only, not in the field (NFM 4).

[2]For ground-water quality control: A splitter blank is included if a cone splitter is used; a standpipe blank often is collected if a submersible pump is used.

Figure 3-7. Sequence of sample collection to obtain the equipment blank

CONVERSION FACTORS AND ABBREVIATIONS

CONVERSION FACTORS

Multiply	By	To obtain
centimeter (cm)	0.3937	inch
meter	3.281	foot
milliliter (mL)	0.06102	$inch^3$ or cubic inch
liter (L)	0.2642	gallon
microgram (µg)	3.53×10^{-8}	ounce

Temperature: Water and air temperature are given in degrees Celsius (°C), which can be converted to degrees Fahrenheit (°F) by use of the following equation:

$$°F = 1.8(°C) + 32$$

ABBREVIATIONS

DIW	deionized water
DOC	dissolved organic carbon
HCl	hydrochloric acid
HNO_3^-	nitric acid
IBW	inorganic-grade blank water, laboratory-certified free of trace elements and other inorganic constituents
µg/L	micrograms per liter
µS/cm	microsiemens per centimeter at 25°C
MSDS	Material Safety Data Sheet
NFM	*National Field Manual for the Collection of Water-Quality Data*
NWQL	National Water Quality Laboratory of the U.S. Geological Survey
OWQ	Office of Water Quality of the U.S. Geological Survey
PBW	pesticide-grade blank water, certified free of pesticide organic compounds by the NWQL
PVC	polyvinyl chloride
QC	quality control
QWSU	Quality of Water Service Unit
SOC	suspended organic carbon
SS	source solution
TOC	total organic carbon
TWRI	Techniques of Water-Resources Investigations
URL	Uniform Resource Locator
USGS	U.S. Geological Survey
VBW	volatiles-grade blank water, certified free of volatile compounds by the NWQL
v/v	volume to volume

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Water Quality

Memo No.	Title	Date
qw 92.01	Distilled/Deionized Water for District Operations	Dec. 20, 1991
qw 97.03	Protocols for Cleaning a Teflon Cone Splitter to Produce Contaminant-Free Subsamples for Subsequent Determinations of Trace Elements	Feb. 7, 1997

National Water Quality Laboratory (NWQL)

Memo No.	Title	Date
92.01	Technology Transfer—Availability of Equipment Blank Water for Inorganic and Organic Analysis	Mar. 25; 1992

Water Resources Division

Memo No.	Title	Date
wrd 94.007	Safety--Storage, Transportation, Handling and Disposal of Methyl Alcohol	Dec. 3, 1993

PUBLICATIONS ON TECHNIQUES OF WATER-RESOURCES INVESTIGATIONS

The U.S. Geological Survey publishes a series of manuals describing procedures for planning and conducting specialized work in water-resources investigations. The material is grouped under major subject headings called books and is further divided into sections and chapters. For example, Section A of Book 9 (Handbooks for Water-Resources Investigations) pertains to collection of water-quality data. The chapter, which is the unit of publication, is limited to a narrow field of subject matter. This format permits flexibility in revision and publication as the need arises.

The Techniques of Water-Resources Investigations (TWRI) reports listed below are for sale by the U.S. Geological Survey, Branch of Information Services, Box 25286, Federal Center, Denver, CO 80225 (authorized agent of the Superintendent of Documents, Government Printing Office). Prepayment is required. Remittance should be sent by check or money order payable to the U.S. Geological Survey. Prices are not included because they are subject to change. Current prices can be obtained by writing to the above address. When ordering or inquiring about prices for any of these publications, please give the title, book number, chapter number, and "U.S. Geological Survey Techniques of Water-Resources Investigations." An updated list of TWRI reports can be found by accessing the World Wide Web url: http://water.usgs.gov/lookup/get?TWRI.

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5-A1.Methods for determination of inorganic substances in water and fluvial sediments, by M.J. Fishman and L.C. Friedman, editors: USGS—TWRI Book 5, Chapter A1. 1989. 545 pages.

5-A2.Determination of minor elements in water by emission spectroscopy, by P.R. Barnett and E.C. Mallory, Jr.: USGS—TWRI Book 5, Chapter A2. 1971. 31 pages.

5-A3.Methods for the determination of organic substances in water and fluvial sediments, edited by R.L. Wershaw, M.J. Fishman, R.R. Grabbe, and L.E. Lowe: USGS—TWRI Book 5, Chapter A3. 1987. 80 pages.

5-A4.Methods for collection and analysis of aquatic biological and microbiological samples, by L.J. Britton and P.E. Greeson, editors: USGS—TWRI Book 5, Chapter A4. 1989. 363 pages.

5-A5.Methods for determination of radioactive substances in water and fluvial sediments, by L.L. Thatcher, V.J. Janzer, and K.W. Edwards: USGS—TWRI Book 5, Chapter A5. 1977. 95 pages.

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Book 6. Modeling Techniques

Section A. Ground Water

6-A1.A modular three-dimensional finite-difference ground-water flow model, by M. G. McDonald and A. W. Harbaugh: USGS—TWRI Book 6, Chapter A1. 1988. 586 pages.

6-A2.Documentation of a computer program to simulate aquifer-system compaction using the modular finite-difference ground-water flow model, by S.A. Leake and D.E. Prudic: USGS—TWRI Book 6, Chapter A2. 1991. 68 pages.

6-A3.A modular finite-element model (MODFE) for areal and axisymmetric ground-water-flow problems, Part 1: Model Description and User's Manual, by L. J. Torak: USGS—TWRI Book 6, Chapter A3. 1993. 136 pages.

6-A4.A modular finite-element model (MODFE) for areal and axisymmetric ground-water-flow problems, Part 2: Derivation of finite-element equations and comparisons with analytical solutions, by R.L. Cooley: USGS—TWRI Book 6, Chapter A4. 1992. 108 pages.

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6-A6.A coupled surface-water and ground-water flow model (MODBRANCH) for simulation of stream-aquifer interaction by E.D. Swain and Eliezer J. Wexler: USGS—TWRI Book 6, Chapter A6, 1996. 125 pages.

Book 7. Automated Data Processing and Computations

Section C. Computer Programs

7-C1.Finite difference model for aquifer simulation in two dimensions with results of numerical experiments, by P.C. Trescott, G.F. Pinder, and S.P. Larson: USGS—TWRI Book 7, Chapter C1. 1976. 116 pages.

7-C2.Computer model of two-dimensional solute transport and dispersion in ground water, by L.F. Konikow and J.D. Bredehoeft: USGS—TWRI Book 7, Chapter C2. 1978. 90 pages.

7-C3.A model for simulation of flow in singular and interconnected channels, by R.W. Schaffranek, R.A. Baltzer, and D.E. Goldberg: USGS—TWRI Book 7, Chapter C3. 1981. 110 pages.

Book 8. Instrumentation

Section A. Instruments for Measurement of Water Level

8-A1.Methods of measuring water levels in deep wells, by M.S. Garber and F.C. Koopman: USGS—TWRI Book 8, Chapter A1. 1968. 23 pages.

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Section B. Instruments for Measurement of Discharge

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Book 9. Handbooks for Water-Resources Investigations

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9-A2.Selection of equipment for water sampling, by F.D. Wilde, D.B. Radtke, Jacob Gibs, and R.T. Iwatsubo, editors: USGS—TWRI Book 9, Chapter A2. 1998. Variously paged.

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9-A6.Field measurements, by F.D. Wilde and D.B. Radtke, editors: USGS—TWRI Book 9, Chapter A6. 1998. Variously paged.

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ATTACHMENT V, LABORATORY STANDARD OPERATING PROCEDURES

Date Effective: 07/01/01 I36
Revision #2.3
Page: 1

Standard Operating Procedure: Edward S. Babcock & Sons

METHOD #: EPA 180.1
Standard Methods 2130 B

TITLE: Turbidity (Nephelometric)

1.0 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters in the range of turbidity from 0.05 to 4000 nephelometric turbidity units (NTU).

NOTE 1: NTU's are considered comparable to the previously reported Formazin Turbidity Units (FTU) and Jackson Turbidity Units (JTU).

2.0 Summary of Method

2.1 The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity. Readings, in NTU's, are made in a nephelometer designed according to specifications outlined in Apparatus. A standard suspension of Formazin, prepared under closely defined conditions, is used to calibrate the instrument.

2.1.1 Formazin polymer is used as the turbidity reference suspension for water because it is more reproducible than other types of standards previously used for turbidity standards.

2.1.2 A commercially available polymer standard is also approved for use for the National Interim Primary Drinking Water Regulations. This standard is identified as AMCO-AEPA-1 available from Amco Standard International, Inc.

3.0 Sample Handling and Preservation

3.1 Samples may be stored in either plastic or glass.

3.2 Preservation consists of refrigeration or icing to 4-C, to minimize microbiological decomposition of solids.

3.3 Analysis must be performed within *24 hours* per 40CFR section 136, Table II. Samples must be stored at 4°C.

4.0 Interferences

4.1 The presence of floating debris and coarse sediments which settle out rapidly will give low readings. Finely divided air bubbles will affect the results in a positive manner.

4.2 The presence of true color, that is the color of water which is due to dissolved substances which absorb light, will cause turbidities to be low, although this effect is generally not significant with finished waters.

5.0 Apparatus

5.1 The 2100N Hach turbidimeter consists of a nephelometer with light source for illuminating the sample a photo-electric detector with a readout device to indicate the intensity of light scattered at right angles to the path of the incident light. The turbidimeter is designed so that little stray light reaches the detector in the absence of turbidity should be free from significant drift after a short warm-up period.

5.2 The sensitivity of the instrument permits detection of a turbidity difference of 0.05 unit or less in waters having turbidities less than 1 unit. (The minimum detection level reported is 0.05 NTU.) The instrument is able to measure from 0.05 to 4000 units turbidity. Several ranges are available to obtain both adequate coverage and sufficient sensitivity for low turbidities.

5.3 The sample tubes are made of clear, colorless glass. They must be kept scrupulously clean, both inside and out, and discarded when they become scratched or etched. They must not be handled at all where the light beam from the instrument strikes them, so they are provided with sufficient extra length so that they may be handled at the top. Check all tubes before analysis by reading a D.I. blank.

5.4 Differences in physical design of turbidimeters will cause differences in measured values for turbidity even though the same suspension is used for calibration. To minimize such differences, the following design criteria are observed:

5.4.1 Light source: Tungsten lamp operated at a color temperature between 2200-3000-K.

5.4.2 Distance traversed by incident light and scattered light within the sample tube: Total not to exceed 10 cm.

5.4.3 Detector: Centered at 90- to the incident light path and not to exceed +/- 30 C- from 90 C. The Detector, and filter system if used, shall have a spectral peak response between 400 and 600 nm.

5.5 Standard laboratory glassware: volumetric flasks, beakers, graduated cylinders, pipets.

Note: All glassware is cleaned immediately prior to and after use by thorough rinsing with three portions of D.I. water. If glassware still appears dirty, further steps are taken, by use of one of the following: Alconox and hot water, 1:1 acid rinse, acetone or appropriate solvent rinse. Glassware is always finished with a final D.I. rinse.

6.0 Reagents and Standards

6.1 Turbidity-free water: D.I. water is used as long as it does not read above 0.1 NTU. If it does read high, Nanopure water is used.

6.2 Stock formazin turbidity suspension: 4000 NTU Formazine Solution purchased for supplier, stored at room temperature until manufacturer specified expiration date. Two sources are purchased, one for calibration standard preparation and the second source for calibration verification (LCS) preparation.

6.3 Standard formazin turbidity suspension prepared:

6.3.1 LCS : Prepare daily as specified below for from noncalibration stock source

6.3.2 Calibration Standards: Prepare each time a calibration is necessary as specified below for instrument calibration.

For: Standard Concentration, NTU	Pipette mls From Stock	*Dilute To: (Volume in mls) with D.I.
4000	fill cell	0
1000	25	100
800	20	100
400	10	100
200	5	100
80	2	100
40	1	100
20	1	200
8	2	1000
4	1	1000
0.9	0.2	1000

7.0 Procedure

7.1 Turbidimeter calibration: The manufacturer's operating instructions are followed which specify calibration every 90 days for USEPA reporting. However, should the Electronic P.C. Board, the Photo Detectors, or the Light Source be replaced or if very carefully prepared Formazine Suspensions indicate a need for recalibration, this will be done more often.

7.2 Calibration

7.2.1 Always mix the contents of each cell by inverting several times before placing in the Optical Well for reading.

7.2.2 Keep the outside surface of the cell clean and dry. Apply a drop of silicone oil to the outside and wipe with a cloth or tissue. Finish with a chem wipe.

7.2.3 When placing any standards in the well, always use the light shield to cover the well in order to keep out ambient light.

7.3 Carry out the following steps:
7.3.1 First place a cell of D.I. water in the cell holder.
7.3.2 Press the CAL key.
7.3.3 Press the ENTER key.
7.3.4 The instrument will advance to the next standard, display the expected value, and the S1 light.
7.3.5 Place the 20 NTU standard in the cell holder.
7.3.6 Press ENTER.
7.3.7 Once the instrument displays the next standard value and S2 light, place that standard in the cell holder, press ENTER and so on until all the standards have been read.
7.3.8 The standard values are: 20, 200, 1000, and 4000.
7.3.9 After the last standard is processed, press the CAL key.

7.4 Analysis:
7.4.1 Clean and rinse the cell with D.I., wiping all excess water from the sides with tissue. Apply a drop of silicone oil to the outside and wipe with a cloth or tissue. Finish with a chem wipe.
7.4.2 Check the calibration by reading the 0.9 Lab Control. If it is not within range repour or remake it. If it is still out, examine the instrument for problems. The calibration may have to be repeated. Sample results may not be taken until a Lab Control falls within the acceptance ranges.
7.4.3 Shake the sample and pour it into the cell. Wipe the cell with lens paper to make sure there are not smudges, then put the cell into the turbidity meter with the line on the cell pointing foreword. If the sample reading is higher than the 0.90 range, the calibration must be checked using a lab control (from the previous table) that encompasses the sample's range. Record the lowest stabilized reading before the sample particles have settled.
7.4.4 *Do not dilute the sample!* (Standard Methods, 20th Edition, Method 2130B 4a and Hach Model 2100N Turbidity Meter Manual 2.3.8.) Readings above 4000 are reported as >4000 NTU.

8.0 Calculation and Reporting Requirements:
8.1 Results are reported in NTU's.

8.2 Report results to 2 significant figures.

8.3 Detection Limit for Reporting Purposes (RL) = 0.05 NTU

9.0 QA/QC Requirements:
9.1 The 0.9 NTU LCS serves as an Initial Calibration check analyzed at the beginning of the analysis. Another LCS, made at a different concentration is analyzed at the end of the run to verify calibration.

9.2 If the lab control does not read within the limits of 85-115%, re-make and read again. If the lab control still does not read correctly, re-calibrate the instrument.

9.3 Duplicates are analyzed with each batch of 20 samples. Results must have a RPD ± 20%.

9.4 An MDL study is completed at a minimum of once per year, or whenever major equipment or procedural changes are made. Standards are spiked at the reporting limit and a minimum of seven replicates is analyzed. See QA Manual section 23 for calculation. Results must be below the reporting limit.

9.5 Initial Demonstration of Capability: Prior to analysis of samples or when a significant change is made to the method, an Initial Demonstration of Capability Study is performed. This is accomplished by analysis of four replicates of a QC sample made at a concentration 10 times the MDL. Acceptance ranges are 80-120% with a maximum %RSD of 20.

9.6 Performance Evaluation Studies performed twice a year serve as documentation of continuing proficiency.

10.0 Definitions: See SOP Q15 - SOP Definitions

11.0 Safety: General laboratory safety procedures are adequate for this analysis.

12.0 Corrective Action for Out of Control Or Unacceptable Data:
See SOP Q06 - Corrective Action

13.0 Pollution Prevention and Waste Management:
See SOP S07 - Pollution Prevention

14.0 Method Performance
Refer to MDL studies, Initial Demonstration of Capability Studies, and laboratory control charts maintained in the QC Office.

Revision Log

Rev.2.3 - 07/01/01: added Revision Log, and sections: 5.5, 9.1, 9.4, 9.5, 9.6, 14.0, edited sections 6.2, 9.2.

Bibliography

1. EPA Method 180.1 (1993) Methods for the Chemical Analysis of Waters and Wastes.

2. Standard Methods for the Examination of Water and Wastewater, APHA/AWWA/WEF, 18th Edition, Method 2130B.

Approved by Susan K Thomas 07/03/01

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RESIDUE, TOTAL SUSPENDED
Edward S. Babcock & Sons
STANDARD OPERATING PROCEDURE
(EPA Method 160.2)
(SM 2540 D)

1.0 Scope and Application:

1.1 This method is applicable to all aqueous samples.

2.0 Working Range:

2.1 The working range is 5mg/L to 2000mg/L.

3.0 Method Summary:

3.1 100mls of sample is filtered through a pretared filter. The residue that remains on this filter after drying in a 105 degree Celsius oven is considered the suspended solid portion of the sample.

4.0 Sample Collection, Preservation and Holding Time:

4.1 The sample must be unpreserved. It must be stored at 4 degrees Celsius until analysis. Analysis must take place within 7 days of sampling per CFR part 136. Table II.

5.0 Interferences:

5.1 Exclude large floating particles or submerged agglomerates of nonhomogeneous materials from the sample if it is determined that their inclusion is not desired. Nonrepresentative particulates such as leaves and sticks may also be excluded.

5.2 To avoid water entrapment, limit the sample size to that yielding no more than 200mg residue on the filter. (This would be a final result of 2000mg/L since we are analyzing 100mls of sample.)

5.3 For samples high in dissolved solids thoroughly wash the filter with D.I. water after the sample has passed through the filter.

5.4 Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter. If more than 10

minutes are required to complete filtration. Increase filter size or decrease sample size.

6.0 Apparatus and Standards

6.1 Side-arm flask of sufficient capacity for sample size selected.

6.2 Filtration apparatus: Membrane filter funnel with a Gelman type A/E glass fiber filter disk with a suitable diameter for the funnel.

6.3 Drying oven, for operation at 105°C ± 2°C.

6.4 Vacuum aspirator.

6.5 Desiccator.

6.6 Balance with a sensitivity of 0.1 mg. Calibrated on a daily basis with 0.1 g and 100 g class "S" weights on a daily basis. Calibration must be within ± 0.5%. If values are not within these limits, recalibrate the balance.

6.7 *Filter garages* to hold glass fiber filters.

6.8 Standard laboratory glassware: volumetric flasks, beakers, graduated cylinders, pipets.

Note: All glassware is cleaned immediately prior to and after use by thorough rinsing with three portions of D.I. water. If glassware still appears dirty, further steps are taken, by use of one of the following: Alconox and hot water, 1:1 acid rinse, acetone or appropriate solvent rinse. Glassware is always finished with a final D.I. rinse.

6.9 Stock : Cellulose

6.10 Lab Control: 500mg of cellulose is weighed into a liter of D.I. water. This solution is kept at room temperature for up to a year.

7.0 Procedure

7.1 Prepare glass fiber filters by rinsing three times with D.I. water and heating at 105°C for a minimum of 1 hour. *A constant weight study is performed yearly to establish the minimum time required to bring the filter to a constant weight.*

7.2 Take hot filters out of 105°C oven.

7.3 Cool filters to room temperature in a desiccator. Use forceps when handling filters. Weigh on balance for tare weight and record. Place the filter onto the filtering apparatus. Wet filter with a small amount of D.I. to seat it.

7.4 Mix sample well by *shaking* sample bottle. Measure an appropriate volume of sample in a graduated cylinder. For normal samples use 100 ml. For samples with a lot of suspended matter, a smaller volume of sample may be used 50 ml to 10 ml. A larger filtering apparatus may be necessary as well. Use a 200 ml sample volume for samples expected to contain very minute amounts of suspended material Filter through apparatus collecting suspended residue on filter. Rinse cylinder and filter 2 to 3 times with a small amount of D.I. water. Suction three minutes after filtration or *until no visible free liquid is present.*

7.5 Place samples in 105°C oven for 1.5 hours which is longer than the *time proven to be sufficient to bring the sample to a constant weight.*

7.6 Cool filters in a desiccator and weigh filters for final weight. Record weight.

8.0 Calculation:

$$\frac{(A-B) \times 1,000,000}{\text{\# ml of sample used}}$$

Where A = Weight in grams of filter with residue, and

B = Tare weight in grams of filter.

8.1 Alternatively, you may subtract the actual numbers in the weight readings (without any decimal points) and multiply the difference by the factor of 100/(ml of sample used).

8.2 The detection limit for this procedure is 5 mg/L.

8.3 Report all results to two significant figures.

9.0 Quality Control:

9.1 Duplicates are analyzed at least once in every analytical batch and at a minimum of once for every 20 samples. The Relative Percent Difference is calculated and compared to the acceptance limit for the analysis. If the RPD does not fall within the acceptance limit which is a maximum of 40, the analysis is re-run. If the RPD still does not fall within the acceptance limit, the supervisor is informed. If it is determined that the matrix of the sample interfered with the analysis, this is noted on the worksheet.

9.2 A method blank is analyzed with every batch of samples. It must read ± 5mg/L.

9.3 A lab control is analyzed with every batch of samples. 500mg of cellulose is weighed into a liter of D.I. water. 100 ml of this solution is analyzed. Results must be ± 20%.

9.4 An MDL study is completed at a minimum of once per year, or whenever major equipment or procedural changes are made. Standards are spiked at the reporting limit and a minimum of seven replicates is analyzed. See QA Manual section 23 for calculation. Results must be below the reporting limit.

9.5 Initial Demonstration of Capability: Prior to analysis of samples or when a significant change is made to the method, an Initial Demonstration of Capability Study is performed. This is accomplished by analysis of four replicates of a QC sample made at a concentration 10 times the MDL. Acceptance ranges are 80-120% with a maximum %RSD of 20.

9.6 Performance Evaluation Studies performed twice a year serve as documentation of continuing proficiency.

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10.0 Corrective Action For Out of Control Or Unacceptable Data:
See SOP Q06 – Corrective Action

11.0 Pollution Prevention and Waste Management:
SOP S07 - Pollution Prevention

12.0 Definitions: See SOP Q15 - SOP Definitions

13.0 Safety

13.1 General laboratory safety procedures are sufficient for this analysis. Recommended safety equipment includes gloves and safety glasses.

14.0 Method Performance:
Refer to MDL studies, Initial Demonstration of Capability Studies, and laboratory control charts maintained in the QC Office.

Revision Log

Rev.2.2 - 07/01/01: added Revision Log, and sections:6.8, 6.9, 6.10, 9.4, 9.5, 9.6 , edited sections:1.6, 14.0

10.0 References

Standard Methods For the Examination of Water and Wastewater 18th Edition APHA/AWWA/WEF 2540D.

Methods for the Chemical Analysis of Waters and Wastes EPA 160.2.

Approved by Susan K. Thomas 07/03/01

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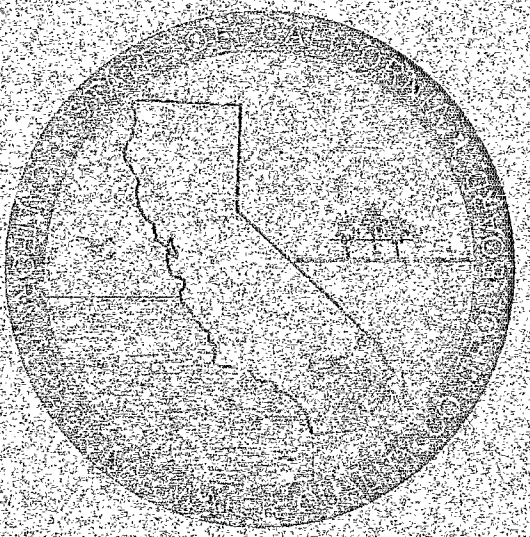

QUALITY ASSURANCE PROJECT PLAN FOR NEW RIVER SILTATION/SEDIMENTATION TMDL IMPLEMENTATION

March 2003
Revision 0 3/4/03

Prepared by and for
State of California Regional Water Quality Control Board Staff
Colorado River Basin

QUALITY ASSURANCE PROJECT PLAN FOR NEW RIVER SILTATION/SEDIMENTATION TMDL IMPLEMENTATION

March 2003
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Prepared by and for
State of California Regional Water Quality Control Board Staff
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Quality Assurance Project Plan
For New River Siltation/Sedimentation TMDL Implementation

APPROVALS:

California Regional Water Quality Control Board

Jose Angel, PE, Supervising WRC Engineer
Watershed Protection Division Chief
Quality Assurance Officer

3/7/03
Date

Doug Wylie

Doug Wylie, PE, Senior WRC Engineer
NPS/TMDL Implementation Unit Chief
Project Manager

3-7-03
Date

Teresa Gonzales, Senior Environmental Scientist
TMDL Development Unit Chief

3-7-03
Date

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1. PROJECT MANAGEMENT

1.1 INTRODUCTION

This Quality Assurance Project Plan (QAPP) describes the quality assurance (QA) and quality control (QC) procedures associated with the monitoring activities to characterize the suspended sediment in the New River. The activities are scheduled in response to certain requirements to be specified by the California Regional Water Quality Control Board, Colorado River Basin (Regional Board) Basin Plan (Basin Plan), which will address the implementation section of the New River Sedimentation/Siltation Total Maximum Daily Load (TMDL). The amendment (Appendix IV) specifies monitoring requirements for the New River identical to monitoring requirements for the Alamo River. Currently, the Basin Plan amendment states that the "Regional Board will conduct monitoring activities for the New River Sedimentation/Siltation TMDL pursuant to a Regional Board Quality Assurance Project Plan for the New River (QAPP-NR)." "The objectives of the monitoring program shall include collection of water quality data for assessment of water quality standards attainment, verification of pollution source allocations, calibration or modification of selected models (if any), evaluation of point and nonpoint source control implementation and effectiveness, evaluation of in-stream water quality, evaluation of temporal and spatial trends in water quality, and modification of the TMDL as necessary."

This QAPP follows the format that the United States Environmental Protection Agency (USEPA) established in its *Requirements for Quality Assurance Project Plans, EPA QA/R-5, 2001.* Further, it also complies with the QA/QC requirements specified in the *State Water Resources Control Board Quality Assurance Program Plan, June 2001.*

The Project Manager and the QA Officer/Division Chief may, upon mutual concurrence, request modification of this QAPP to achieve the objectives of the project. The process for QAPP modification consists of incorporating the necessary changes into the QAPP document, obtaining approval signatures, and distributing the revised document to project personnel.

1.2 DISTRIBUTION LIST

The following individuals will receive copies of the approved QAPP and subsequent revisions:

- Jose Angel, PE, Supervising WRCE, Watershed Protection Division Chief/ QA Officer*
- Doug Wylie, PE, Sr. WRCE, NPS/TMDL Implementation Unit Chief, Project Manager *
- Teresa Gonzales, Sr. ES, TMDL Development Unit Chief*

* Indicates approving authority

1.3 PROJECT/TASK ORGANIZATION

Specific responsibilities of the Regional Board staff are outlined below. A project organization chart is provided as Appendix 1.

Jose L. Angel, P.E., Supervising WRC Engineer, Watershed Protection Division Chief/QA Officer, (760) 776-8932

- Reviews and approves the QAPP and subsequent revisions.
- Ensures that the QAPP is implemented and followed to meet the project objectives.
- Oversees validation activities for field and lab data.

Doug Wylie, PE, Senior WRC Engineer, Project Manager, (760) 346-6585

- Reviews and approves the QAPP and subsequent revisions.
- Ensures that the QAPP is implemented and followed to meet the project objectives.
- Ensures that field personnel have appropriate training and certification for field activities.
- Reviews field reports.
- Ensures plans are implemented according to schedule.
- Prepares annual reports.
- Manages sampling data.
- Performs field sampling as necessary.
- Conducts Health and Safety briefing for field samplers prior to each sampling event.
- Coordinates field and laboratory activities.
- Reports project status to the QA Officer and Division Chief.
- Responsible for evaluating data to ensure TMDL compliance.

Teresa Gonzales, Senior Environmental Scientist, TMDL Development Unit Chief, (760) 776-8931

- Reviews and approves QAPP and subsequent revisions.
- Responsible for TMDL development.

Maria de la Paz Carpio-Obeso, PhD, Environmental Scientist, Regional Board Lab Director, (760) 674-0803

- Responsible for Regional Board Laboratory.

Phan Le, WRC Engineer, Deputy Lab Director, (760) 346-7491

- Responsible for calibration of equipment prior to sampling event.
- Responsible for performing water quality analysis as required.
- Assists with sampling activities as required.

Jeff Allred, WRC Engineer, Lead Field Sampler, (760) 776-8946

- Writes and Revises the Quality Assurance Project Plan (QAPP)
- Coordinates field activities and ensures they are consistent with the QAPP
- Conducts assignment briefing for field samplers prior to each sampling event.
- Ensures that sample containers have no defects and have been prepared properly.
- Conducts sampling activities as required.
- Prepares a summary report for each sampling event.
- Coordinates delivery of samples to the laboratory.
- Coordinates decontamination of equipment.

Field Samplers

- Assists with sampling activities as required.

1.4 PROBLEM DEFINITION/BACKGROUND

Pursuant to Section 303(d) of the Clean Water Act (CWA), the Regional Board is scheduled to approve a Siltation/Sedimentation TMDL for the New River in 2003. The TMDL was developed because sediment concentrations in the river violate the water quality standards (WQS) established by the Regional Board to protect the beneficial uses of the river. The Implementation Plan of the TMDL identifies the monitoring and tracking of the pollutant of concern to determine compliance with the TMDL.

The implementation section of the TMDL divides the New River into three "drainshed" areas. The monitoring and tracking program associated with the TMDL requires water quality monitoring of TSS and turbidity metabolites at the boundaries of these three regions in order to track compliance of the dischargers within each drainshed. Data collection activities outlined in this QAPP are being undertaken to execute the implementation section of the TMDL.

1.5 PROJECT/TASK DESCRIPTION

The overall objective of this project is to obtain valid data of known and documented quality, which can be utilized in determining the compliance with the water quality objectives as set forth in the New River Sediment TMDL. Specifically, the purpose of this project is to:

1. Collect representative water samples for TSS, turbidity, DDT, DDE, and DDD from the New River at the sampling locations identified in Table No. 2, below; and
2. Record field measurements (physical parameters) including turbidity, pH, temperature, dissolved oxygen (DO), and electrical conductivity (EC).

This project consists of monthly sampling events, in which water samples will be collected and field measurements taken at four sampling stations. Sampling events may include sampling at locations on the New River or within drains as necessary to pinpoint sources of contamination (i.e. drainsheds, drains, or individual fields). Predetermined sampling locations are described in detail in Section 2.1, Sampling Process Design.

1.6 DATA QUALITY OBJECTIVES

Valid data of known and documented quality are needed to meet the objectives of this project. Therefore, for the critical measurements of this project (TSS and turbidity), only data which meet QA criteria will be considered valid. The specific data quality objectives of this project are:

- Analyses for TSS and turbidity must yield results that are of sufficient quality to be used in the execution of the implementation for the New River Sediment TMDL. Therefore, data obtained should be of sufficient quality to be utilized to determine the contributions of sediment from each drainshed of the New River, and the resulting sediment concentrations

within the New River, at the time of sampling.

- The data generated in this project should be of sufficient quality to be utilized, along with data from future sampling projects, in the determination of a TSS/Turbidity relationship for the New River and the Agricultural Drains emptying into the New River.

Table No. 1, below, summarizes the precision, accuracy, and completeness criteria.

Table 1: QA Objectives for Laboratory Data

Analyte	Matrix	Units	Precision (RPD)	Accuracy (% Recovery)	Completeness (% Comp)
TSS	Water	mg/L	25	80-120[2,3]	95
Turbidity	Water	NTU	35	65-135[2,3]	95
DDT	Water	µg/L	25	N/A	95
DDE	Water	µg/L	25	N/A	95
DDD	Water	µg/L	25	N/A	95
Field Measurement					
Turbidity	Water	NTU	35	65-135[2,3]	95
Temperature	Water	^{0}C	0.01	+/- 0.15	N/A
pH	Water	pH units	0.01	+/-0.2	N/A
DO	Water	mg/L	N/A	+/- 2% of reading or 0.2 mg/L[4]	N/A
EC	Water	µmhos/cm	N/A	+/- 0.5% of reading + 0.001 mS/cm	N/A

[1] Completeness criteria will not be applied to results from QC samples.
[2] For blanks, the average of all TSS and turbidity measurements must be 5mg/L or 5 NTU or less, respectively.
[3] All projects requiring submittal of spike samples will be coordinated in a manner such that one set of spikes will be submitted to the laboratory for all projects.
[4] Whichever is greater.

1.6.1 Data Quality Indicators (Acceptance Criteria)

The following data quality indicators will be utilized to assess whether data generated are useable and meet the data quality objectives stated above:

1.6.1.1 Precision

Precision is defined as the degree of refinement of a measurement. Precision of the data generated will be assessed as the relative percent difference (RPD) for field and laboratory duplicates.

$$RPD = \frac{ABS(C_1 - C_2)*100}{\left(\frac{C_1 + C_2}{2}\right)}$$

RPD = Relative Percent Difference
D_1 = Results for sample 1
D_2 = Results for sample 2
ABS = Absolute value

1.6.1.2 Accuracy

Accuracy is defined as the degree of refinement of a measurement to the actual value or standard. Accuracy for turbidity and TSS will be determined by calculating percent recovery for double blind spike samples and field blanks. For field blanks, the average of all TSS measurements must be 5 mg/l or less, and the average of all turbidity measurements must be 5 NTU or less, in order for this data quality objective to be met. All projects requiring submittal of spike samples will be coordinated in a manner such that one set of spikes will submitted to the laboratory for all projects.

$$\%R = 100 * \frac{C_M}{C_R}$$

%R = Percent recovery
C_M = Measured concentration of reference material (RM).
C_R = Actual concentration of reference material (RM).

1.6.1.3 Completeness

Completeness is defined as a measure of how many collected samples actually yield valid and useable data. A minimum of 95% completeness is expected for this project. This will result in a sufficient amount of data to meet the previously stated requirements.

$$\%C = 100 * \frac{V}{T}$$

%C = Percent complete
V = Total number of measurements or laboratory results judged valid
T = Total number of measurements or laboratory results

1.7 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

The Project Manager will ensure that all of the field samplers have valid and current training for their field activities, as required by OSHA regulations. Currently, all sampling personnel identified in the Project/Task Organization section of this QAPP have completed the required OSHA training for the sampling activities described herein. There are no other specialized training/certification requirements needed to perform the Project's objectives.

1.8 DOCUMENTATION AND RECORDS

1.8.1 Project Working File

The Project Manager will establish and maintain a Project Working File for maintaining sampling records. The Lead Field Sampler and QA Officer will ensure that all received/generated data (e.g., field notes, chain-of-custody forms, lab analyses) are delivered to the Project Manager. The file will contain, but need not be limited to:

- Field data sheets
- Calibration logs
- Laboratory reports
- Data reports summarizing field activity and quality control for each sampling event
- Data spreadsheets

- Correspondence
- Quality control reports
- Validation reports
- Sampling Event Summary Reports
- Annual Reports

1.8.2 Field Datasheets

The Lead Field Sampler will use field datasheets to document field activities and data for each sampling event. Each field datasheet will be dated and signed by a sampling team member at each sampling station. At the time of sampling, the following information will be recorded in the field datasheet:

- Weather observations
- Sampling station latitude and longitude, using a global positioning system (GPS) unit, if not previously recorded
- Sample identification code and sampling method for all samples taken
- In-situ measurements for temperature, pH, DO, and EC
- Field turbidity measurement
- Sample identification code, and time and location of preparation, for all quality control samples prepared in the field
- Any deviations from the QAPP
- Any noteworthy observations

1.8.3 Sampling Event Summary Report

The Lead Field Sampler will prepare a sampling event summary report for each sampling event, which will be submitted to the Project Manager. The reports will be due within 7 days following each sampling event. The reports will summarize:

- Any deviations from the QAPP
- Any problems encountered and how the problems were addressed
- Recommendations as appropriate

1.8.4 Quality Control Log Notebook

The QA Officer will use a bound quality control log notebook with pre-numbered pages to document the quality control (QC) samples submitted to the laboratory and the analysis results. For each QC sample, the quality control log notebook will contain the:

- Sample identification code
- Supplier of the QC sample
- Value reported by the supplier

- Date of preparation and submission
- Name and signature of the person submitting the QC sample
- Laboratory performing the analysis
- Analysis method
- Reported value from the laboratory

1.8.5 Calibration Log Notebook

The Deputy Lab Director will use a calibration log notebook to document calibration activities performed on sampling equipment prior to each sampling event. The calibration log notebook will be bound and will have pre-numbered pages. For each calibration event, the calibration log notebook will contain:

- Date and time of calibration
- Person(s) performing the calibration
- Signature of one of the persons performing the calibration
- All standard solutions used in calibration, including the source and date of preparation of the standard solution
- The initial reading of the YSI 6600 multiprobe sonde when tested against each standard solution, and the temperature of each standard solution at the time of calibration
- Any problems encountered and how the problems were addressed

1.8.6 Laboratory Analytical Summaries

The Division Chief will request that the laboratory prepare and submit to the Project Manager a laboratory analytical summary for each sampling event upon completion of the laboratory's analysis of samples. This summary will include analytical results, analytical methods, problems encountered, QC results, and chain of custody forms.

1.8.7 Quality Assurance Reports

The QA Officer will prepare quality assurance reports, including a technical systems audit for each sampling event, performance evaluations of laboratories, and an annual data quality assessment. These reports are described in more detail in Section 4.1.

2. MEASUREMENT/DATA ACQUISITON

2.1 SAMPLING PROCESS DESIGN

In order to meet the overall objectives stated in Section 1.5 of this QAPP, this project was designed to estimate the suspended sediment concentration, as represented by total suspended solids (TSS) and turbidity, at several sampling stations along the New River. Also, to pinpoint sources, sampling locations may be located as necessary within the New River watershed. Because accurate suspended sediment data are necessary for TMDL implementation, TSS and turbidity are considered critical measurements for this project, while the other baseline parameters, temperature, EC, pH and DO, are considered non-critical measurements.

The New River sampling stations were selected to characterize the changes in water quality in each of the three drainsheds. Sampling stations are located at the upstream and downstream ends of each drainshed. As previously stated, when it is necessary to locate a source, additional sampling stations may be designated and established on an as-needed basis within the New River watershed.

At each of the locations listed in Table 2 below, water samples will be taken as described, and a YSI 6600 multi-parameter sonde will be used to take in-stream measurements of temperature, DO, EC and pH. Field turbidity will be measured using a Hach 2100P portable turbidimeter.

Table 2: Monitoring Stations

Sampling Location[1]	No.[2]	Description
NR-B	NR-0	Monitoring station for Mexico's sediment contribution to the New River at the Boundary, to be located at the intersection of the New River and the International Boundary.
NR-EH	NR-1	Monitoring station for the Lower New River drainshed, to be located at the Evan Hewes Road Bridge and the New River.
NR-2	NR-2	Monitoring station for the Middle New River drainshed, to be located at Drop Structure #2 of the New River.
NR-O	NR-Outlet	Monitoring station for the Upper New River drainshed, to be located at the USGS sampling station downstream of Lack Road Bridge.

[1]A location may be changed provided the prescribed location is inaccessible or haphazard, as documented by field observations.
[2]Denotes sampling location as stated in the Basin Plan.

Sampling station NR-2 listed in Table 2 is a drop structure. Because the water is well mixed as it cascades over this structure, a single grab sample is typically representative of the sediment concentration throughout the river at the given cross-section. The mixing effect of the structures also introduces oxygen into the water. Therefore, YSI readings should be taken on the upstream side of the structure. When samples are taken at alternative locations, which are not located at a drop structure and are greater than six (6) feet wide, three (3) sampling points (S1, S2, and S3) will be distributed along the cross-sectional area of the river/drain. The sampling points are to be

spaced at approximately equal intervals from each other and from the edge of the river/drain (i.e., at a distance equal to w/4, where "w" is the top width of the cross-sectional area). Figure No. 1, below, illustrates this. A sample composed of a grab sample from each cross-section will be obtained and homogenized using a churn splitter. The composite sample will be used for lab analyses.

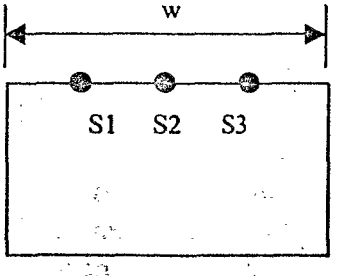


Figure 1: Sampling Points at Non-Well-Mixed Sampling Locations

For the drop structure location listed in Table 2 and for small drain sampling stations where there are small cross sections and well-mixed flow, only one grab sample at the center point (S2), if possible, will be taken to characterize TSS and turbidity. Also, at sampling points along the river and the main drains where the depth is less than 2 feet, well mixed flow is assumed, and only grab samples at the center point (S2) will be taken.

For all sampling stations, YSI readings of EC, DO, pH and temperature will be recorded at the center sampling point (S2) if possible. Also, a Hach 2100P field turbidimeter will be used to obtain turbidity measurements at the same sampling point. Minimum sample collection frequency is noted in Table 3 below.

Table 3: Minimum Sample Frequency

Constituent	Sample Frequency
Turbidity	Monthly
Total Suspended Solids	Monthly
DDT	Quarterly
DDE	Quarterly
DDD	Quarterly

2.2 SAMPLING METHODS REQUIREMENTS

Sampling methods include the collection of grab samples, as well as the acquisition of readings for water quality parameters using the YSI water quality sonde. A clean sample collection bottle will be used at each sampling station. Inaccuracies in the lab analyses due to sorption of analytes to the swing sample collection bottle, and churn splitter if appropriate, will be negated by rinsing each three times with native water prior to collection of the sample. The grab samples will be

collected at approximately midstream (sampling point S2). Where hazardous conditions prevent midstream sampling, the grab sample will be collected at sampling location S1 or S3. Grab samples will be collected at approximately ½-foot below the water surface using a swing sampler, and immediately transferred to a churn splitter. While churning the sample in the churn splitter, the sample will be distributed into the appropriately labeled sample bottle and placed into an ice chest with a sufficient quantity wet ice.

The YSI 6600 multi-parameter water quality sonde will be used to collect field measurements for the following parameters: DO, pH, temperature, and EC at the center point at each sampling location from about 1-foot below the water surface. Where hazardous conditions prevent midstream sampling, the YSI measurements will be taken at sampling location S1 or S3. After at least two minutes or when the readings have reached equilibrium, the values for these parameters will be manually recorded in the field data sheet. Turbidity measurements using the Hach 2100P turbidimeter will be performed on the collected grab samples. In the event that a field turbidity measurement falls outside of the range of the equipment (1000 NTU), the turbidity values will be reported as >1,000 NTU. This out-of-range data may be used in assessing compliance, but will not be used in calculating the relationship between TSS and turbidity.

2.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Sample-holding times will be adhered to, as prescribed by USEPA and 40 CFR 136. Specifically, the required preservation techniques and holding times for all of the constituents which the laboratory will be analizing are listed in Table 4, below.

Table 4: Required Containers, Preservatives, Techniques, and Holding Times

Constituent	Container	Preservation Technique	Holding Time
Turbidity	1-L glass or low density polyethylene bottle	Cool 4 °C	48 hours
Total Suspended Solids			7 days
DDT, DDE, DDD	2-L Amber Glass bottle	Cool 4 °C	7 days

Each sample container will be labeled with a unique sample identification code. All samples (including QC samples) for laboratory analyses will immediately be stored in an ice chest, and will remain in the custody of Regional Board staff until the samples are delivered to the laboratory. The ice chest will contain sufficient ice to maintain the samples at a temperature below 4°C at all times until they are relinquished to the lab staff. All samples will be delivered with chain of custody forms. A sample chain of custody form to be used for this project is included in Appendix II. Any violation of holding times or other sample handling and custody requirements will be documented in the quality control records and reported to the Project Manager and the QA Officer. Any violations thereof will be taken into account when evaluating the data.

2.4 ANALYTICAL METHODS REQUIREMENTS

As prescribed by the State Water Resources Control Board's "*Quality Assurance Program Plan*", each analytical laboratory used for sample analysis must have a written QA Laboratory Manual describing the analytical method requirements. The lab will use USEPA approved methods as outlined in Table No. 5.

Table 5: Sampling Constituents and Methods

Constituent	USEPA Method	Reporting Limit	Units
Turbidity	180.1	0.10	NTU
Total Suspended Solids	160.2	1.0	mg/L
DDT, DDE, DDD	8081	0.025-2.0	µg/L

2.5 QUALITY CONTROL REQUIREMENTS

In order to assess whether the data quality requirements of this project are being met, a number of quality control checks will be implemented. The calibration and maintenance of Regional Board Laboratory instruments and the general operation of the Regional Board laboratory are subject to the requirements of the State Board Quality Assurance Program Plan and the Regional Board Quality Assurance Program for the Laboratory. All QC samples will be placed in an ice chest, and kept at 4 °C, for transport to the lab. Specifically:

- Field blank samples will be collected in the field by dispensing deionized water into the appropriately labeled sample container. The reported value is used to check for laboratory accuracy. One field blank will be collected and submitted for analysis for each constituent, for each day of sampling.

- Field duplicate samples will be prepared from a grab sample of the water being sampled. A grab sample will be collected as described above and split using a churn splitter, into the appropriately labeled containers. Ten percent of the samples collected will be field duplicates, with a minimum of one set of field duplicate samples for each analyte collected per day of sampling.

- Double blind spike samples for turbidity and TSS (one each) will be prepared by an independent lab and submitted to the laboratory for analyses once per year. All projects requiring submittal of spike samples will be coordinated in a manner such that, when possible, one set of spikes will be submitted to the laboratory for all projects.

- To ensure preservation requirements are met, a random sample will be chosen by the laboratory from each ice chest for temperature measurement.

QC samples will be submitted to the lab along with the "real" surface water samples being submitted (i.e., the laboratory will not be informed in any way as to which samples are control

samples and which samples are from the aforementioned surface waters). A summary of Quality Control Sample Requirements is located in Table 6.

Table 6: Quality Control Sample Requirements

Quality Control Samples	Number of Samples	Frequency
*Duplicate Samples (All parameters)	10%	Per event
Field Blanks (All parameters)	1	Per day/event
Spike Samples (TSS, Turbidity only)	1	Annually
Temperature blank[1]	1	Per ice chest

[1]Temperature blank may be omitted if temperature is read from a random sample bottle using an infrared thermometer.

2.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, & MAINTENANCE REQUIREMENTS

All Regional Board staff participating in the project will be trained in the operation, calibration, and maintenance of the field instruments. The manufacturer's instruction manuals will be readily available for field personnel. The instruments will be maintained and calibrated in accordance with the manufacturer's instructions and recommendations. Calibration, inspection, and maintenance of field instruments are performed by laboratory personnel prior to all sampling events.

2.7 INSTRUMENT CALIBRATION AND FREQUENCY

The YSI 6600 sonde and the Hach 2100P Turbidimeter will be calibrated in the Regional Board laboratory prior to each sampling event. Dissolved oxygen will be calibrated prior to each sample collection, using ambient air. Pre and post sampling calibration check will be performed at each sampling site by field personnel. This process involves checking and recording the DO output. If it is within 2% of saturation, recalibration of DO is required. The pre and post calibration check data (DO output) will be recorded on the field datasheet. If recalibration is required, calibration data will be recorded on a calibration log sheet. A post-calibration for all parameters will be performed when the sonde is returned to the office, or as soon as practical. Results of calibration measurements will be documented in the field log notebook and submitted to the QA Officer. Table 7, below illustrates the YSI 6600 sonde and the Hach 2100P specifications.

Table 7: Specifications for the YSI 6600 Sonde & Hach 2100P Turbidimeter.

Parameter	Operating Range	Accuracy	Resolution	Calibration Standard
pH	0 to 14 units	± 0.2 units	0.01 units	2-pt, with pH buffered solutions
Temperature	– 5 to 45 °C	± 0.15 °C	0.01 °C	not required
DO	0 to 20 mg/L	± 0.2 mg/L	0.01 mg/L	saturated air
EC	0 to 100 mS/cm	± 1% of range	4 digits	KCl
Turbidity	0-1000 NTU	+/- 1% or 0.01 NTU[1]	0.01 NTU	Gelex Standards

[1]Whichever is greater (with Gelex standards).

2.8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES/CONSUMABLES

The Lead Field Sampler will ensure that the sample bottles have no defects, and that all sample bottles have been prepared properly.

No other special requirements are needed.

2.9 DATA ACQUISITION REQUIREMENTS (NON-DIRECT METHODS)

Only data collected from this Project and historic data from the TMDL development program, which have already passed QC criteria, will be used. No data will be used from other sources unless the data also meet the QA requirements set herein.

2.10 DATA MANAGEMENT

The Project Manager will maintain field datasheets and chain of custody forms in the project file. Field measurement data will be uploaded from the YSI using ECOLAB software, and analyses results will be obtained in electronic form from the lab. The Project Manager will submit all data in MS Excel format to the QA Officer. After verification and approval by the QA Officer, the Project Manager will download the data into the project database (MS Excel format) and store it on the Regional Board's local area network (LAN). After a period of twelve months, statistical analyses of the data from each sampling point will be employed to calculate an annual average to be used in determining compliance of each drainshed as per the Implementation Section of the TMDL.

3. ASSESSMENT AND OVERSIGHT

3.1 ASSESSMENT AND RESPONSE ACTIONS

Surveillance of the records and overall status of the project will be conducted by the QA Officer to ensure that all of the requirements of the QAPP are being met. Surveillance will be conducted after each sampling event, after all laboratory results have been received for that sampling event. A technical systems audit will also be performed by the QA Officer, as discussed in Section 4.1.1. Also, an annual data quality assessment of the applicability of the data will be performed to assess the handling of all data and to correct any errors found in the project database (see Section 4.1.3).

3.2 REPORTS TO MANAGEMENT

The Project Manager will prepare quarterly and annual project reports. The quarterly project reports will include a summary of the activities performed, the resulting data, and the quality of the resulting data, any problems encountered and their solutions and will identify any samples that indicate violations of Water Quality Standards. The annual project reports will include a statistical analysis of the results indicating drainshed loading, any decrease or increase in loading at the drainshed boundaries, drainsheds which are out of compliance, recommendations for TMDL modification, and the relationship of TSS to turbidity.

4. DATA VALIDATION AND USABILITY

4.1 DATA REVIEW, VERIFICATION AND VALIDATION

Data objectives for this project do not require a full, formal, and independent data validation. The data has no legal requirement for independent validation. Although the data are considered legally defensible as presented herein, all records will be available for independent evaluation should the need arise at a later date.

The QA Officer will be responsible for validating the project's data to ensure that QA guidelines have been followed by performing:

4.1.1 Technical Systems Audit

The QA Officer will prepare or oversee a technical systems audit for each sampling event, after all laboratory results are received. Data will be validated to determine if the collection and analyses procedure conformed to the QAPP. The review will consider field notes, field datasheets, chain of custody forms, laboratory analysis forms, and calibration assessment (determines potential error in field measurements). Documentation of results will occur within 15 days, and will describe data reviewed, review criteria, and data usability.

Unacceptable departures from sample collection procedures include the use of contaminated sampling bottles, the lack of critical sample collection information, or any other activity which would result in the cross contamination or incorrect identification of samples.

Departures from the sample handling and custody procedures contained in Section 2.3 of this report will be determined through the review of chain of custody forms and laboratory analysis forms. In order for data to be considered valid for meeting the data quality objectives of this study, all chain of custody forms must be in the possession of the Project Manager, and strict adherence to holding times and temperatures must be followed. Data generated from samples that do not meet these requirements will not be considered valid for use in this study.

Verification of proper calibration of the YSI sonde and the Hach 2100P turbidimeter will be performed during the audit of data quality through a review of the quality control records. Calibration values will also be assessed to determine the potential error in the field measurements. If calibration values have errors that exceed acceptable error tolerances, the measurements obtained prior to that calibration, but after the previous calibration, will be labeled suspect and further investigated to determine if they are valid for use in this study.

The QA Officer will then ensure that data are entered into the database. It is conceivable, however, that errors could occur in entering the data (e.g., transposing the decimal point for a particular result or keying in the wrong Sample ID). Therefore, once a data set has been entered into the database, all records will be checked to ensure accuracy.

4.1.2 Performance Evaluation of Laboratories

Validation of laboratory data will be performed in the Audit of Data Quality by assessing the results of QC sample analyses. Lab data will be validated for precision, accuracy, and completeness according to the criteria specified in Section 1.6.

In the event that QC analyses do not meet the specified criteria, the data will be labeled as "suspect", the lab will be notified, and the field notes will be re-evaluated. Data sets corresponding to any value that cannot be confirmed, based on the acceptable criteria in Table 1, will be rejected.

In case of missing data, the QA Officer will discuss it with the laboratories submitting the data. In some cases, missing data will be denoted as missing in reports. For all missing data, and any other data requiring special explanation, qualifiers will be included in the database and in data reports. Missing data will be designated as "NR", meaning *Not Reported.*

4.1.3 Data Quality Assessment

The QA Officer will prepare annually an audit of data quality reports, which takes into account all Technical Systems Audits and includes verification of calibration and instrument drift results for the YSI 6600 multi-probe sonde and Hach 2100P turbidimeter, and the results of the laboratory QC samples. Precision, accuracy, and completeness results for laboratory data will also be included in the reports. Also, the reports will assess whether the total error in the data is tolerable, and whether significant departures from the QAPP reduce data set completeness (and thus reduce data set usability for drawing conclusions).

Significant departures from the QAPP will be noted in these reports, and the resulting data will not be validated. Unacceptable departures include, but are not limited to:

- Cross-contamination
- Lack of critical sample collection information
- Violation of sample holding times and temperatures

5. HEALTH AND SAFETY PLAN

5.1 CONTAMINATION CONTAINMENT ZONES

The contaminated areas for this Project consist of and cover the entire waterways for the aforementioned waters, their banks, the area within 2 feet of the banks, and the bridge at the Outlet. Decontamination zones will be set at least 10 feet away from the banks of the surface waters. The decontamination zone will be used for personnel decontamination and will include wash water, soap, paper towels, and trash bags. All contaminated solid waste material will be placed in trash bags for proper disposal. Only biodegradable antibacterial soap will be used at the site. Wash water runoff will be contained and disposed of in the surface waters. The Clean area will be set at least 20 feet away from the banks of the surface waters.

5.2 PERSONAL PROTECTIVE EQUIPMENT

The general concerns at the sampling sites are the potential exposure to toxicants present in the waters being sampled, risk of sunburn, excessive heat exposure, insect bite, and possibly snakebites. In addition, the sampling crew should be aware of the risk of falling into the waterways. No less than two experienced samplers will be out in the field at one time. (The sampling crew will also have a functional cellular phone in one of the vehicles).

- Any member of the sampling team has the authority to stop the sampling event when he/she determines that conditions at the site (e.g., rain, dust, local emergency, etc.) preclude safe sampling. A Hazard Evaluation Plan (HEP) will be done for each day of sampling. The lead field sampler will be responsible for preparing the HEP.

- To reduce the risk of exposure while collecting/transporting samples, Latex Examination Gloves must be worn. The Contaminated Zone must not be entered without the aforementioned Personal Protective Equipment (PPE).

- To reduce the risk of heat exposure and sunburn, samplers should wear sunscreen and carry in their vehicle cold drinking water. If any of the samplers begin to experience symptoms of heat exhaustion, such as cramps or dizziness, he or she will immediately be removed from the sun and given plenty of cool liquids. If these symptoms persist, he or she will be taken to the nearest hospital.

- Extra caution should be used when working near or around the drains to reduce the risk of potentially falling in.

- To reduce the risk of insect bites, samplers should use insect repellent.

- To reduce the possibility of snakebite, samplers will check areas for snakes prior to entering the area. If snakebite occurs, ice will be placed on the bite. The sampler will be immediately transported to the nearest medical facility.

5.3 PERSONNEL DECONTAMINATION PROCEDURES

The Clean Zone must not be entered with contaminated PPE. All team members coming out of the Contaminated Zones must immediately proceed to the Decontamination Zones and use the following decontamination procedures before proceeding to Clean Zone:

1. Remove latex gloves carefully to avoid contact with bare skin and dispose of them in the trash bag. Thoroughly wash hands with antibacterial soap; and

2. Dispose of wash water into surface water just sampled.

Note: everything that is touched (pens, pencils, rinse water bottles, probes, etc.) with dirty gloves could be contaminated. Avoid touching these items with bare skin.

5.3.1 Emergency Numbers and Facilities

All sampling personnel will have access to a cellular phone to call 911 in case of an emergency. The hospital nearest the sampling locations are listed in Table 8 below:

Table 8: Nearest Hospitals to Sampling Locations

Sampling Location	Medical Facility	Address	Phone #
NR-B	EL Centro Regional Medical Center	Imperial and Ross Ave., El Centro	(760) 339-7100
NR-EH	EL Centro Regional Medical Center	Imperial and Ross Ave., El Centro	(760) 339-7100
NR-D2	Pioneers Memorial Hospital	207 West Legion, Brawley	(760) 351-3333
AR-O	Pioneers Memorial Hospital	207 West Legion, Brawley	(760) 351-3333

In case of an emergency, sampling personnel should also contact the Regional Board Safety Officer, Doug Wylie, as soon as practical at 760-346-6585 or 760-341-7491.

5.3.2 After Sampling

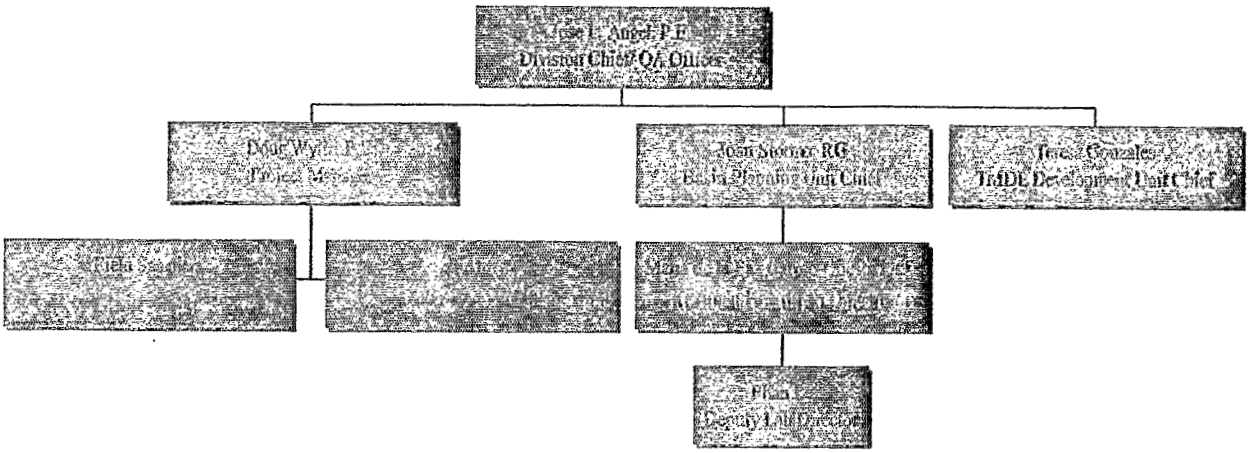
Place samples into Regional Board lab refrigerator or keep in an ice chest filled with wet ice; keep water drained from ice chests to avoid soaking container labels. Make copies of field notes and put original in the project working file. Contaminated equipment should be packed in designated containers for transport to the Regional Board office. Decontaminate and properly clean ALL items, which were exposed in the field in accordance with USGS National Field Manual for the Collection of Water-Quality Data, Chapter A3. Cleaning of Equipment for Water Sampling (See Attachment IV).

6. REFERENCES

U.S. Environmental Protection Agency. 2001. EPA Requirements for Quality Assurance Project Plans, EPA QA/R5. EPA Publication number 240/B-01/003. U.S. Environmental Protection Agency, Washington, D.C.

State Water Resources Control Board (State of California), 2001. Quality Assurance Program Plan.

APPENDIX I, PROJECT ORGANIZATION CHART



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Appendix II, Sample Chain of Custody Form

E.S. Babcock & Sons, Inc.
6100 Quail Valley Court Riverside, CA 92507
(909) 653-3351 • FAX (909) 653-1662

Chain of Custody & Sample Information Record

Client: Contact: Phone No.

Project Name: _____

Project Location:

Turn Around Time: Routine 3-5 Days 48 Hours 24 Hours *(Rushes Require Approval, Additional Charges May Apply)*

Sampler Information

Name: _____

Employer: _____

Signature: _____

ESB #	Sample ID	Date	Time	# of Containers & Preservatives: Unpreserved	H_2SO_4	HCl	HNO_3	$Na_2S_2O_3$	NaOH			Total # of Containers	Analysis Requested	Matrix	Notes
														DW = Drinking Water WW = Wastewater GW = Groundwater S = Soil SG = Sludge L = Liquid M = Miscellaneous	

Relinquished By (sign)	Print Name / Company	Date / Time	Received By (Sign)	Print Name / Company

(For Lab Use Only) *Sample Integrity Upon Receipt*

Sample(s) Submitted on Ice? Yes No Temperature °C

Custody Seal(s) Intact? Yes No N/A

Sample(s) Intact? Yes No

Lab No. _____

Page _____ of _____

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APPENDIX III, BASIN PLAN AMENDMENT

An Amendment to the Water Quality Control Plan for the Colorado River Basin Region to Establish the New River Sedimentation/Siltation Total Maximum Daily Load

AMENDMENT

(Proposed changes are in reference to the May 23, 2002 version of the Basin Plan. Proposed additions are denoted by underlined text, proposed deletions are denoted by ~~strikethrough~~ text)

Page 4-19, change "~~VI. TOTAL MAXIMUM DAILY LOADS~~" TO "V. TOTAL MAXIMUM DAILY LOADS" and add the following new subsequent Section and renumber accordingly:

C. New River Sedimentation/Siltation TMDL

1. TMDL ELEMENTS

Table 4-3: New River Sedimentation/Siltation TMDL Elements

ELEMENT	
Problem Statement (impaired water quality standard)	Excess delivery of sediment to the New River has resulted in degraded conditions that impairs designated beneficial uses: warm freshwater habitat; wildlife habitat; preservation of threatened, rare, and endangered species habitat; contact- and non-contact recreation; freshwater replenishment. As the New River discharges into the Salton Sea, sediment also threatens the same beneficial uses of the Salton Sea. Sediment serves as a carrier for DDT, DDT metabolites, and other insoluble pesticides including toxaphene, which pose a threat to aquatic and avian communities and people feeding on fish from the New River; and suspended solids concentrations, sediment loads, and turbidity levels are in violation of water quality objectives. These current concentrations, loads, and levels are also forming objectionable bottom deposits, which are also adversely affecting the beneficial uses of New River.

(This table is continued on the following page.)

Table C-1: New River Sedimentation/Siltation TMDL Elements (continued)

ELEMENT	CURRENT CONDITIONS
Numeric Target	200 mg/L Total Suspended Solids (annual average)[1]
Source Analysis	**Source** — **tons/year** Agricultural Drain Discharges: 137,715 In-Stream Erosion & Wind Deposition: 6,409 NPDES Permitted Facilities: 356 International Boundary 11,265 Total: 155,745

ELEMENT	LOAD ALLOCATIONS
Margin of Safety	6,409 tons/year (corresponds to 10 mg/L)
Seasonal Variations and Critical Conditions	Both the flow and sedimentation regimes within the New River watershed are relatively stable, and the sediment and water sources within the watershed are relatively uniform and widespread; therefore, this TMDL does not include provisions other than the established load allocations and implementation plan for seasonal variations or critical conditions. Staff's analysis of potential water transfers out of the watershed indicate that the transfers are not likely to affect compliance with this TMDL, but could cause other water quality problems that will need to be addressed by the parties responsible for the transfers.
Loading Capacity	127,881 tons/year

(This table is continued on the following page.)

[1] The numeric target is a goal that translates current silt/sediment-related Basin Plan narrative objectives and shall not be used for enforcement purposes.

Table C-1: New River Sedimentation/Siltation TMDL Elements (continued)

<table>
<tr><td>ELEMENT</td><td colspan="3"></td></tr>
<tr><td rowspan="4">Load Allocations and Wasteload Allocations</td><td colspan="3">Load Allocations:
• Natural sources of sediment to the New River, including erosion and wind deposition, are allocated 6,409 tons/year.
• Waste discharges from nonpoint sources into the New River shall not exceed the load allocations specified below:</td></tr>
<tr><td>River Reach</td><td># of IID Drains Identified within Reach</td><td>Sediment Load Allocation (tons/year)[1,2]</td></tr>
<tr><td>New River immediately downstream of the International Boundary, at the USGS gauging station, a point identified hereafter at "NR-0"</td><td>None</td><td>11,265</td></tr>
<tr><td>Reach 1: Downstream from the International Boundary to the intersection of the Evan Hewes Road Bridge and the New River Channel, a point identified hereafter as "NR-1"</td><td>14</td><td>20,730</td></tr>
<tr><td></td><td>Reach 2: This reach encompasses the river from NR-1 to Drop Structure 2, a point upstream of the Rutheford Road Bridge hereafter referred to as "NR-2".</td><td>17</td><td>32,350</td></tr>
</table>

(This table is continued on the following page.)

Table C-1: New River Sedimentation/Siltation TMDL Elements (continued)

ELEMENT			
Load Allocations and Wasteload Allocations	Reach 3: This reach covers the river from NR-2 to the point where it intersects the Lack Road Bridge, a point hereafter referred to as "NR-Outlet."	23	35,835
	Direct Outfalls to River	# of IID Drains Identified	Sediment Load Allocation (tons/year)[1,2]
	Tailwater outfalls discharging directly to the New River.	[a]	14,884
	Natural Sources		
	Natural Sources		6,409
	Waste Load Allocations: • The discharge from point sources (NPDES permits) shall not exceed the total suspended solids limits specified under 40 CFR 122 et seq., and the corresponding mass loading rates.		

Footnotes for Table No. C-1:

1. The sediment load allocation for any particular applicable reach shall be distributed proportionately amongst the agricultural drains within that particular reach based on the relative flow contribution of each drain to the total flow contribution to the reach from the drains within the reach. The Regional Board's Executive Officer shall determine the proportional load amongst the agricultural drains within that particular reach. The sediment load allocation will be reviewed by the Regional Board's Executive Officer every three years following TMDL implementation.
2. The sediment load allocations have been calculated based on the estimated individual average drain flows within the reach for the 1995-2000 period. At lower or higher drain flows, the average annual load allocation for a particular reach shall not exceed the load given by:
   $LA_R = (180)*(Q_R)*(0.0013597)$, where:
   LA_R = Load Allocation for any of the New River reaches identified above (tons/yr).
   Q_R = Reach Flow (ac-ft) = Total flow contribution to the reach from the drains within the reach (ac-ft).
   The sediment load allocation will be reviewed by the Executive Officer every three years following TMDL implementation.

[a]. The number of outfalls has not been determined.

TMDL attainment shall be in accordance with the schedule contained in Table C-2, below:

Table C-2: Interim Numeric Targets for Attainment of the TMDL

Phase	Time Period[1]	Estimated Percent Load Reduction[2]	Interim Target (mg/L)[3]
Phase 1	Years 1 – 3	5%	229
Phase 2	Years 4 – 6	7%	213
Phase 3	Years 7 – 9	4%	204
Phase 4		2%	200

	Years 10 – 12		

Footnotes for Table No. C-2:

1. Year 1 refers to the effective date to start TMDL implementation, which shall be one year after USEPA approves the TMDL. For example, if USEPA approves the TMDL on November 15, 2002, Year 1 is November 15, 2003, which makes Year 3 November 15, 2005, which makes Year 4 November 15, 2006, and so on.
2. Percent reductions indicate the reduction required in total suspended sediment load from the average concentration of the New River at the beginning of each phase, beginning with the 1980-2001 average concentration of 306 mg/L.
3. These interim targets are goals which translate current silt/sediment related Basin Plan narrative objectives and are not intended to specifically be used for enforcement purposes.

Page 4-25, Edit subsequent Section ~~1. IMPLEMENTATION ACTIONS AND REGULATIONS FOR ATTAINMENT OF ALAMO RIVER SEDIMENTATION/SILTATION TMDL"~~ change to "1. IMPLEMENTATION ACTIONS AND REGULATIONS FOR ATTAINMENT OF SEDIMENTATION/SILTATION TMDLs"

Page 4-25, Edit Subsequent Section "1.1 DESIGNATED MANAGEMENT ACTIONS" and change to:

- Farmers/growers discharging waste into the New River and Alamo River in a manner that causes or could cause violation of load allocations and/or exceedance of the Sediment/Silt numeric target;

Page 4-25, Edit Subsequent Section "1.1.1 Farmers/growers Water Quality Management Plans" and change to:

The farmers/growers shall submit self-determined sediment control programs to the Regional Board **by: ~~{insert the date that corresponds 15 months following the date of USEPA TMDL approval}~~*.**

Table 4-4 Date that Corresponds to 15 months following the date of USEPA TMDL Approval *

TMDL	Date (15 months after USEPA Approval
Alamo River	
New River	

Edit Subsequent Section "1.1.2 The Imperial Irrigation District" and change to:
By: ~~{insert the date that corresponds to 15 months following the date of USEPA TMDL approval}~~*

Table 4-5 Date that Corresponds to 15 months following the date of USEPA TMDL Approval *

TMDL	Date (15 months after USEPA Approval
Alamo River	
New River	

the Imperial Irrigation District shall submit to the Regional Board a revised Drain Water Quality Improvement Plan (DWQIP) with a proposed program to control and monitor water quality impacts caused by drain maintenance operations within the Alamo and New River Watershed and dredging operations in the Alamo and New Rivers.

---

* Note: Upon USEPA TMDL approval, this parenthetical "formula" will be replaced by the date certain, based on the date of approval.

* Note: Upon USEPA TMDL approval, this parenthetical "formula" will be replaced by the date certain, based on the date of approval. The Executive Officer shall be responsible for determining proportional sediment load allocations amongst the agricultural drains.

a. Drain and ~~New~~ River Deltas Maintenance

- Reduction in drain cleaning and dredging activities to the practical extent allowed by the implementation of on- and off-field sediment control BMPs by the farmers/growers and the BMP effectiveness in reducing silt built up in the drains and the New and Alamo River Deltas to avoid impacts on sensitive resources.

b. Drain Water Quality Monitoring Plan
The revised DWQIP shall consist of a proposed program to monitor:

- Water quality impacts caused by dredging operations in the drains and to monitor the effects that dredging operations in the New and Alamo River Deltas have on the ~~river's~~ rivers' water quality standards;
- Representative samples from the water column of all major drains and a representative number of the small drains tributary to the New and Alamo Rivers for analyses of flow, TSS, Turbidity, and nutrients.

c. Information on Agricultural Dischargers

No later than ~~{insert date that corresponds to 16 months following the date of USEPA TMDL approval}~~*;

Table 4-6 Date that Corresponds to 16 months following the date of USEPA TMDL Approval

TMDL	Date (16 months after USEPA Approval
Alamo River	
New River	

Page 4-27, Edit Subsequent Section "1.1.3. United States Environmental Protection Agency (USEPA) and U.S. Section of the International Boundary and Water Commission (IBWC)" and change to:

By: ~~{insert the date that corresponds to 15 months following the date of USEPA TMDL approval}~~*

Table 4-7 Date that Corresponds to 15 months following the date of USEPA TMDL Approval *

TMDL	Date (15 months after USEPA Approval
Alamo River	
New River	

the USEPA and/or the U.S. Section of the IBWC shall submit to the Regional Board a technical report pursuant to Section 13225 of the California Water Code describing the proposed control

* Note: Upon USEPA approval, this parenthetical "formula" will be replaced by the date certain, based on the date of approval.

measures, monitoring plan and reporting procedures, and quality assurance procedures the U.S. Government proposes to take to ensure that discharges of wastes from Mexico do not violate or contribute to a violation of ~~this~~ these ~~TMDL~~ TMDLs, particularly a violation of the Load Allocation immediately downstream of the International Boundary, at the ~~point~~ points identified as "AR-0~~.~~" and "NR-0."

Edit Subsequent Section "1.2 RECOMMENDED MANAGEMENT ACTIONS FOR FARMERS/GROWERS AND DRAINAGE MANAGEMENT" and change to:

Implementation of BMPs should normally include: (1) consideration of specific site conditions; (2) monitoring to assure that practices are properly applied and are effective; (3) improvement of a BMP or implementation of additional BMPs or other management practices when needed to resolve a deficiency and; (4) mitigation of a problem where the practices are not effective. The practices listed herein are a compilation of BMPs recommended by the Technical Advisory Committee for the Silt TMDL for the Alamo and New Rivers (Silt TAC), the Natural Resources Conservation Services Field Office Technical Guide (NRCS FOTG), the IID, and the University of California Cooperative Extension (Holtville Field Station). Inclusion of practices herein is not meant to imply or establish a prescriptive list of 'one size fits all' preferred practices for the drainage basins tributary to the Alamo and New ~~River~~ Rivers.

Edit Subsequent Section Title "1.2.3 ESTIMATED COST OF IMPLEMENTATION AND SOURCES OF FINANCING" and change to "1.2.3 ESTIMATED COST OF IMPLEMENTATION AND SOURCES OF FINANCING FOR THE NEW AND ALAMO RIVERS"

Edit Subsequent Section 1.3.1 IMPERIAL COUNTY FARM BUREAU VOLUNTARY WATERSHED PROGRAM and change to:

a. ICFB WATERSHED PROGRAM PLAN
The Imperial County Farm Bureau should:

- **By: ~~{insert the date that corresponds to 13 months following the date of USEPA TMDL approval}~~*;**

Table 4-8 Date that Corresponds to 13 months following the date of USEPA TMDL Approval *

TMDL	Date (13 months after USEPA Approval
Alamo River	
New River	

~~Issue~~ issue letters to all potential program participants within the Alamo and New Rivers watersheds that describes the ICFB Voluntary Watershed Program.

- **By: ~~By {insert the date that corresponds to 15 months following the date of USEPA TMDL approval}~~*;**

Table 4-9 Date that Corresponds to 15 months following the date of USEPA TMDL Approval *

* Note: Upon USEPA TMDL approval, this parenthetical "formula" will be replaced by the date certain, based on the date of approval.

TMDL	Date (15 months after USEPA Approval
Alamo River	
New River	

provide the Regional Board with a list of program participants, organized by subwatershed ("drainshed").

- By: ~~{insert the date that corresponds to 15 months following the date of USEPA TMDL approval}*;~~

Table 4-10 Date that Corresponds to 15 months following the date of USEPA TMDL Approval *

TMDL	Date (15 months after USEPA Approval
Alamo River	
New River	

submit the ICFB Watershed Program Plan to the Regional Board. The Plan should (1) identify measurable environmental and programmatic goals; (2) describe aggressive, reasonable milestones and timelines for the development and implementation of TMDL outreach plans; (3) describe aggressive, reasonable milestones and timelines for the development of sub-watershed ("drainshed") plans; (4) describe a commitment to develop and implement a tracking and reporting program.

b. ICFB TRACKING AND REPORTING PROCEDURES
The Imperial County Farm Bureau should also:

- By: ~~{insert the date that corresponds to 16 months following the date of USEPA TMDL approval}*;~~

Table 4-11 Date that Corresponds to 16 months following the date of USEPA TMDL Approval *

TMDL	Date (16 months after USEPA Approval
Alamo River	
New River	

submit a plan describing the process and procedures for tracking and reporting implementation of BMPs (and other proven management practices) and BMP performance to the Regional Board's Executive Officer.

- Implement the tracking and reporting procedures.
- Submit semi-monthly written reports assessing trends in the data and level of adoption of the process and procedures throughout each of the sub-watersheds ("drainsheds") to the Executive Officer.

Submit a yearly summary report to the Executive Officer by 15th of February of each year.

Page 4-32, Edit " VI. ACTIONS OF OTHER AUTHORITIES" change to "VII. ACTIONS OF OTHER AUTHORITIES"

Page 6-3, Edit "II. REGIONAL BOARD MONITORING", SUBSECTION "B. COMPLIANCE MONITORING", SUBSEQUENT SECTION "~~1. Recommended Biomonitoring (Toxicity Monitoring) Programs~~" change to "2. Recommended Biomonitoring (Toxicity Monitoring) Programs"

Page 6-4, Edit under subsequent Sections the following:

~~2.~~ 3. New River Pathogen TMDL

~~3.~~ 4. Alamo River Sedimentation/Siltation TMDL

5. New River Sedimentation/Siltation TMDL

~~3.1.~~ 5.1 Compliance Assurance and Enforcement

~~3.2~~ 5.2 Monitoring and Tracking

Page 6-5, Edit Section

- **Water Quality Monitoring and Assessment and add the Subsection "Alamo River" directly beneath the Section title. Add the subsequent Subsection "New River" with the following text:**

Monitoring activities are contingent upon adequate programmatic funding. The Regional Board will conduct monitoring activities for the New River Sedimentation/Siltation TMDL pursuant to a Regional Board Quality Assurance Project Plan for the New River *(QAPP-NR)*. The QAPP-NR shall be developed by Regional Board staff and be ready for implementation within 180 days following USEPA approval of this TMDL. The Regional Board's Executive Officer shall approve the QAPP-NR and monitoring plan after determining that the QAPP-NR and monitoring plan satisfy the objectives and requirements of this Section 5.2. The objectives of the monitoring program shall include collection of water quality data for:

- Assessment of water quality standards attainment,
- Verification of pollution source allocations,
- Calibration or modification of selected models (if any),
- Evaluation of point and nonpoint source control implementation and effectiveness,
- Evaluation of in-stream water quality,
- Evaluation of temporal and spatial trends in water quality, and
- Modification of the TMDL as necessary.

The monitoring program shall include a sufficient number of sampling locations and sampling points per location along the New River and major drain tributaries to the river. Monthly grab samples from the above-mentioned surface waters shall be collected and analyzed for the following parameters:

- Flow (to be obtained from IID or USGS)
- Dissolved Oxygen
- pH

- Temperature
- Field turbidity
- Laboratory turbidity
- Total suspended solids
- Quarterly monitoring of DDT and DDT metabolites
- Fecal coliform organisms
- E. Coli
- Fecal streptococci
- Enterococci

The Regional Board will track activities implemented by dischargers and responsible parties and surveillance conducted for the New River Sedimentation/Siltation TMDL pursuant to an implementation tracking plan (ITP). Regional Board staff will develop the ITP within 180 days following USEPA approval of this TMDL. The Regional Board's Executive Officer shall approve the ITP after determining that the ITP satisfies the objectives and requirements of this Section 5.2. The objectives of Regional Board Surveillance and implementation tracking are:

- Assess/track/account for practices already in place;
- Measure the attainment of Milestones;
- Determine compliance with NPDES permits, WLAs, and LAs; and
- Report progress toward implementation of NPS water quality control, in accordance with the SWRCB NPS Program Plan (PROSIP).

QUALITY ASSURANCE PROJECT PLAN FOR ALAMO RIVER SILTATION/SEDIMENTATION TMDL IMPLEMENTATION

February 2003
Revision 0 2/10/03

Prepared by and for
State of California Regional Water Quality Control Board Staff
Colorado River Basin

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Quality Assurance Project Plan
For Alamo River Siltation/Sedimentation TMDL Implementation

APPROVALS:

California Regional Water Quality Control Board

Doug Wylie — 2-11-03

for Jose Angel, PE, Supervising WRC Engineer — Date
Watershed Protection Division Chief
Quality Assurance Officer

Doug Wylie — 2-11-03

Doug Wylie, PE, Senior WRC Engineer — Date
NPS/TMDL Implementation Unit Chief
Project Manager

Teresa Gonzales — 2/10/03

Teresa Gonzales, Senior Environmental Scientist — Date
TMDL Development Unit Chief

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1. PROJECT MANAGEMENT

1.1 INTRODUCTION

This Quality Assurance Project Plan (QAPP) describes the quality assurance (QA) and quality control (QC) procedures associated with the monitoring activities to characterize the suspended sediment in the Alamo River. The activities are scheduled in response to certain requirements specified by the California Regional Water Quality Control Board, Colorado River Basin (Regional Board) Basin Plan (Basin Plan), which addresses the implementation section of the Alamo River Sedimentation/Siltation Total Maximum Daily Load (TMDL). Specifically, Chapter 6, Section II.B.3.1 (Revised May 2002) states that "Regional Board water quality monitoring activities for the Alamo River Sedimentation/Siltation TMDL Monitoring and Tracking Program shall be conducted pursuant to a Quality Assurance Project Plan for the Alamo River (QAPP-AR). The QAPP-AR shall: (1) include a sufficient number of sampling stations along the Alamo River to determine progress towards compliance with the TMDL and overall water quality improvement; (2) provide for monthly monitoring of flow, field turbidity, laboratory turbidity, total suspended solids in the river; and (3) provide for quarterly monitoring of DDT and DDT metabolites in the river's water column."

This QAPP follows the format that the United States Environmental Protection Agency (USEPA) established in its *Requirements for Quality Assurance Project Plans, EPA QA/R-5, 2001.* Further, it also complies with the QA/QC requirements specified in the *State Water Resources Control Board Quality Assurance Program Plan, June 2001.*

The Project Manager and the QA Officer/Division Chief may, upon mutual concurrence, request modification of this QAPP to achieve the objectives of the project. The process for QAPP modification consists of incorporating the necessary changes into the QAPP document, obtaining approval signatures, and distributing the revised document to project personnel.

1.2 DISTRIBUTION LIST

The following individuals will receive copies of the approved QAPP and subsequent revisions:

- Jose Angel, PE, Supervising WRCE, Watershed Protection Division Chief/ QA Officer*
- Doug Wylie, PE, Sr. WRCE, NPS/TMDL Implementation Unit Chief, Project Manager *
- Teresa Gonzales, Sr. ES, TMDL Development Unit Chief*

* Indicates approving authority

1.3 PROJECT/TASK ORGANIZATION

Specific responsibilities of the Regional Board staff are outlined below. A project organization chart is provided as Appendix 1.

Jose L. Angel, P.E., Supervising WRC Engineer, Watershed Protection Division Chief/QA Officer, (760) 776-8932

- Reviews and approves the QAPP and subsequent revisions.
- Ensures that the QAPP is implemented and followed to meet the project objectives.
- Oversees validation activities for field and lab data.

Doug Wylie, PE, Senior WRC Engineer, Project Manager, (760) 346-6585

- Reviews and approves the QAPP and subsequent revisions.
- Ensures that the QAPP is implemented and followed to meet the project objectives.
- Ensures that field personnel have appropriate training and certification for field activities.
- Reviews field reports.
- Ensures plans are implemented according to schedule.
- Prepares annual reports.
- Manages sampling data.
- Performs field sampling as necessary.
- Conducts Health and Safety briefing for field samplers prior to each sampling event.
- Coordinates field and laboratory activities.
- Reports project status to the QA Officer and Division Chief.
- Responsible for evaluating data to ensure TMDL compliance.

Teresa Gonzales, Senior Environmental Scientist, TMDL Development Unit Chief, (760) 776-8931

- Reviews and approves QAPP and subsequent revisions.
- Responsible for TMDL development.

Maria de la Paz Carpio-Obeso, PhD, Environmental Scientist, Regional Board Lab Director, (760) 674-0803

- Responsible for Regional Board Laboratory.

Phan Le, WRC Engineer, Deputy Lab Director, (760) 346-7491

- Responsible for calibration of equipment prior to sampling event.
- Responsible for performing water quality analysis as required.
- Assists with sampling activities as required.

Jeff Allred, WRC Engineer, Lead Field Sampler, (760) 776-8946

- Writes and Revises the Quality Assurance Project Plan (QAPP)
- Coordinates field activities and ensures they are consistent with the QAPP
- Conducts assignment briefing for field samplers prior to each sampling event.
- Ensures that sample containers have no defects and have been prepared properly.
- Conducts sampling activities as required.
- Prepares a summary report for each sampling event.
- Coordinates delivery of samples to the laboratory.
- Coordinates decontamination of equipment.

Nadim Zeywar, PhD, Environmental Scientist, Field Sampler (760) 776-8942.

- Assists with sampling activities as required.

Theresa Illare, Environmental Scientist, Field Sampler (760) 776-8971.

- Assists with sampling activities as required.

Sheila Ault, Environmental Scientist, Field Sampler, (760) 776-8960.

- Assists with sampling activities as required.

Ivory Reyburn, Environmental Scientist, Field Sampler, (760) 776-8933.

- Assists with sampling activities as required.

Logan Raub, Environmental Scientist, Field Sampler, (760) 776-8966.

- Assists with sampling activities as required.

1.4 PROBLEM DEFINITION/BACKGROUND

Pursuant to Section 303(d) of the Clean Water Act (CWA), the Regional Board approved a Siltation/Sedimentation TMDL for the Alamo River in 2001. The TMDL was developed because sediment concentrations in the river violate the water quality standards (WQS) established by the Regional Board to protect the beneficial uses of the river. The Implementation Plan of the TMDL identifies the monitoring and tracking of the pollutant of concern to determine compliance with the TMDL.

The implementation section of the TMDL divides the Alamo River into six "drainshed" areas. The monitoring and tracking program associated with the TMDL requires water quality monitoring of TSS and turbidity metabolites at the boundaries of these six regions in order to track compliance of the dischargers within each drainshed. Data collection activities outlined in this QAPP are being undertaken to execute the implementation section of the QAPP.

1.5 PROJECT/TASK DESCRIPTION

The overall objective of this project is to obtain valid data of known and documented quality, which can be utilized in determining the compliance with the water quality objectives as set forth in the Alamo River Sediment TMDL. Specifically, the purpose of this project is to:

1. Collect representative water samples for TSS, turbidity, DDT, DDE, and DDD from the Alamo River at the sampling locations identified in Table No. 2, below; and
2. Record field measurements (physical parameters) including turbidity, pH, temperature, dissolved oxygen (DO), and electrical conductivity (EC).

This project consists of monthly sampling events, in which water samples will be collected and field measurements taken at seven sampling stations. Sampling events may include sampling at locations on the Alamo River or within drains as necessary to pinpoint sources of contamination (i.e. drainsheds, drains, or individual fields). Predetermined sampling locations are described in detail in Section 2.1, Sampling Process Design.

1.6 DATA QUALITY OBJECTIVES

Valid data of known and documented quality are needed to meet the objectives of this project. Therefore, for the critical measurements of this project (TSS and turbidity), only data which meet QA criteria will be considered valid. The specific data quality objectives of this project are:

- Analyses for TSS and turbidity must yield results that are of sufficient quality to be used in the execution of the implementation for the Alamo River Sediment TMDL. Therefore, data obtained should be of sufficient quality to be utilized to determine the contributions of sediment from each drainshed of the Alamo River, and the resulting sediment concentrations within the Alamo River, at the time of sampling.

- The data generated in this project should be of sufficient quality to be utilized, along with data from future sampling projects, in the determination of a TSS/Turbidity relationship for the Alamo River and the Agricultural Drains emptying into the Alamo River.

Table No. 1, below, summarizes the precision, accuracy, and completeness criteria.

Table 1: QA Objectives for Laboratory Data

Analyte	Matrix	Units	Precision (RPD)	Accuracy (% Recovery)	Completeness (% Cmp)
TSS	Water	mg/L	25	80-120[2,3]	95
Turbidity	Water	NTU	35	65-135[2,3]	95
DDT	Water	µg/L	25	N/A	95
DDE	Water	µg/L	25	N/A	95
DDD	Water	µg/L	25	N/A	95
Field Measurement					
Turbidity	Water	NTU	35	65-135[2,3]	95
Temperature	Water	^{0}C	0.01	+/- 0.15	N/A
pH	Water	pH units	0.01	+/-0.2	N/A
DO	Water	mg/L	N/A	+/- 2% of reading or 0.2 mg/L[4]	N/A
EC	Water	µmhos/cm	N/A	+/- 0.5% of reading + 0.001 mS/cm	N/A

[1] Completeness criteria will not be applied to results from QC samples.
[2] For blanks, the average of all TSS and turbidity measurements must be 5mg/L or 5 NTU or less, respectively.
[3] All projects requiring submittal of spike samples will be coordinated in a manner such that one set of spikes will be submitted to the laboratory for all projects.
[4] Whichever is greater.

1.6.1 Data Quality Indicators (Acceptance Criteria)

The following data quality indicators will be utilized to assess whether data generated are useable and meet the data quality objectives stated above:

1.6.1.1 Precision

Precision is defined as the degree of refinement of a measurement. Precision of the data generated will be assessed as the relative percent difference (RPD) for field and laboratory duplicates.

$$RPD = \frac{ABS(C_1 - C_2)*100}{\left(\frac{C_1 + C_2}{2}\right)}$$

RPD = Relative Percent Difference
D_1 = Results for sample 1
D_2 = Results for sample 2
ABS = Absolute value

1.6.1.2 Accuracy

Accuracy is defined as the degree of refinement of a measurement to the actual value or standard. Accuracy for turbidity and TSS will be determined by calculating percent recovery for double blind spike samples and field blanks. For field blanks, the average of all TSS measurements must be 5 mg/l or less, and the average of all turbidity measurements must be 5 NTU or less, in order for this data quality objective to be met. All projects requiring submittal of spike samples will be coordinated in a manner such that one set of spikes will submitted to the laboratory for all projects.

$$\%R = 100*\frac{C_M}{C_R}$$

%R = Percent recovery
C_M = Measured concentration of reference material (RM).
C_R = Actual concentration of reference material (RM).

1.6.1.3 Completeness

Completeness is defined as a measure of how many collected samples actually yield valid and useable data. A minimum of 95% completeness is expected for this project. This will result in a sufficient amount of data to meet the previously stated requirements.

$$\%C = 100*\frac{V}{T}$$

%C = Percent complete
V = Total number of measurements or laboratory results judged valid
T = Total number of measurements or laboratory results

1.7 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

The Project Manager will ensure that all of the field samplers have valid and current training for their field activities, as required by OSHA regulations. Currently, all sampling personnel identified in the Project/Task Organization section of this QAPP have completed the required OSHA training for the sampling activities described herein. There are no other specialized training/certification requirements needed to perform the Project's objectives.

1.8 DOCUMENTATION AND RECORDS

1.8.1 Project Working File

The Project Manager will establish and maintain a Project Working File for maintaining sampling records. The Lead Field Sampler and QA Officer will ensure that all received/generated data (e.g., field notes, chain-of-custody forms, lab analyses) are delivered to the Project Manager. The file will contain, but need not be limited to:

- Field data sheets
- Calibration logs
- Laboratory reports
- Data reports summarizing field activity and quality control for each sampling event
- Data spreadsheets
- Correspondence
- Quality control reports
- Validation reports
- Sampling Event Summary Reports
- Annual Reports

1.8.2 Field Datasheets

The Lead Field Sampler will use field datasheets to document field activities and data for each sampling event. Each field datasheet will be dated and signed by a sampling team member at each sampling station. At the time of sampling, the following information will be recorded in the field log notebook:

- Weather observations
- Sampling station latitude and longitude, using a global positioning system (GPS) unit, if not previously recorded
- Sample identification code and sampling method for all samples taken
- In-situ measurements for temperature, pH, DO, and EC
- Field turbidity measurement
- Sample identification code, and time and location of preparation, for all quality control samples prepared in the field
- Any deviations from the QAPP
- Any noteworthy observations

1.8.3 Sampling Event Summary Report

The Lead Field Sampler will prepare a sampling event summary report for each sampling event, which will be submitted to the Project Manager. The reports will be due within 7 days following each sampling event. The reports will summarize:

- Any deviations from the QAPP
- Any problems encountered and how the problems were addressed
- Recommendations as appropriate

1.8.4 Quality Control Log Notebook

The QA Officer will use a bound quality control log notebook with pre-numbered pages to document the quality control (QC) samples submitted to the laboratory and the analysis results. For each QC sample, the quality control log notebook will contain the:

- Sample identification code
- Supplier of the QC sample
- Value reported by the supplier
- Date of preparation and submission
- Name and signature of the person submitting the QC sample
- Laboratory performing the analysis
- Analysis method
- Reported value from the laboratory

1.8.5 Calibration Log Notebook

The Deputy Lab Director will use a calibration log notebook to document calibration activities performed on sampling equipment prior to each sampling event. The calibration log notebook will be bound and will have pre-numbered pages. For each calibration event, the calibration log notebook will contain:

- Date and time of calibration
- Person(s) performing the calibration
- Signature of one of the persons performing the calibration
- All standard solutions used in calibration, including the source and date of preparation of the standard solution
- The initial reading of the YSI 6600 multiprobe sonde when tested against each standard solution, and the temperature of each standard solution at the time of calibration
- Any problems encountered and how the problems were addressed

1.8.6 Laboratory Analytical Summaries

The Division Chief will request that the laboratory prepare and submit to the Project Manager a laboratory analytical summary for each sampling event upon completion of the laboratory's analysis of samples. This summary will include analytical results, analytical methods, problems encountered, QC results, and chain of custody forms.

1.8.7 Quality Assurance Reports

The QA Officer will prepare quality assurance reports, including a technical systems audit for each sampling event, performance evaluations of laboratories, and an annual data quality assessment. These reports are described in more detail in Section 4.1.

2. MEASUREMENT/DATA ACQUISITON

2.1 SAMPLING PROCESS DESIGN

In order to meet the overall objectives stated in Section 1.5 of this QAPP, this project was designed to estimate the suspended sediment concentration, as represented by total suspended solids (TSS) and turbidity, at several sampling stations along the Alamo River. Also, to pinpoint sources, sampling locations may be located as necessary within the Alamo River watershed. Because accurate suspended sediment data are necessary for TMDL implementation, TSS and turbidity are considered critical measurements for this project, while the other baseline parameters, temperature, EC, pH and DO, are considered non-critical measurements.

The Alamo River sampling stations were selected to characterize the changes in water quality in each of the six drainsheds. Sampling stations are located at the upstream and downstream ends of each drainshed. As previously stated, when it is necessary to locate a source, additional sampling stations may be designated and established on an as-needed basis within the Alamo River watershed.

At each of the locations listed in Table 2 below, water samples will be taken as described, and a YSI 6600 multi-parameter sonde will be used to take in-stream measurements of temperature, DO, EC and pH. Field turbidity will be measured using a Hach 2100P portable turbidimeter.

Table 2: Monitoring Stations

Sampling Location[1]	AR-Y[2]	Description[1]
AR-B	AR-0	Monitoring station for Mexico's sediment contribution to the Alamo River at the Boundary, to be located at the intersection of the Alamo River and the International Boundary.
ARD10	AR-1	Monitoring station for the Lower Alamo River drainshed, to be located at Drop Structure #10 of the Alamo River.
ARD8	AR-2	Monitoring station for the Central Drain drainshed, to be located at Drop Structure #8 of the Alamo River.
ARD6A	AR-3	Monitoring station for the Holtville Main Drain drainshed, to be located at Drop Structure #6a of the Alamo River.
ARD6	AR-4	Monitoring station for the Rose Drain drainshed, to be located at Drop Structure #6 of the Alamo River.
ARD3	AR-5	Monitoring station for the Central Alamo River drainshed, to be located at Drop Structure #3 of the Alamo River.
AR-O	AR-Outlet	Monitoring station for the Upper Alamo River drainshed, to be located at the intersection of Garst Road Bridge and the Alamo River.

[1]A location may be changed provided the prescribed location is inaccessible or a haphazard, as documented by field observations.
[2]Denotes sampling location as stated in Table 4.1 of the Basin Plan (Revised May 2002).

All sampling stations (except for AR-O) listed in Table 2 are drop structures. Because the water is well mixed as it cascades over these structures, a single grab sample is typically representative

of the sediment concentration throughout the river at the given cross-section. The mixing effect of the structures also introduces oxygen into the water. Therefore, YSI readings should be taken on the upstream side of the structures. When samples are taken at alternative locations, which are not located at a drop structure and are greater than six (6) feet wide, three (3) sampling points (S1, S2, and S3) will be distributed along the cross-sectional area of the river/drain. The sampling points are to be spaced at approximately equal intervals from each other and from the edge of the river/drain (i.e., at a distance equal to w/4, where "w" is the top width of the cross-sectional area). Figure No. 1, below, illustrates this. A sample composed of a grab sample from each cross-section will be obtained and homogenized using a churn splitter. The composite sample will be used for lab analyses.

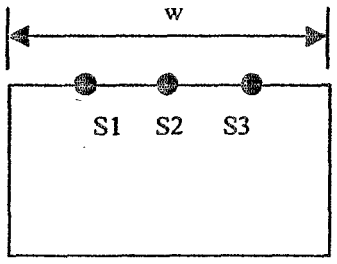


Figure 1: Sampling Points at Non-Well-Mixed Sampling Locations

For the drop structure locations listed in Table 2, and for small drain sampling stations where there are small cross sections and well-mixed flow, only one grab sample at the center point (S2) will be taken to characterize TSS and turbidity. Also, at sampling points along the river and the main drains where the depth is less than 2 feet, well mixed flow is assumed, and only grab samples at the center point (S2) will be taken.

For all sampling stations, YSI readings of EC, DO, pH and temperature will be recorded at the center sampling point (S2) if possible. Also, a Hach 2100P field turbidimeter will be used to obtain turbidity measurements at the same sampling point. Minimum sample collection frequency is noted in Table 3 below.

Table 3: Minimum Sample Frequency

Constituent	Sample Frequency
Turbidity	Monthly
Total Suspended Solids	Monthly
DDT	Quarterly
DDE	Quarterly
DDD	Quarterly

2.2 SAMPLING METHODS REQUIREMENTS

Sampling methods include the collection of grab samples, as well as the acquisition of readings for water quality parameters using the YSI water quality sonde. A clean sample collection bottle will be used at each sampling station. Inaccuracies in the lab analyses due to sorption of analytes to the swing sample collection bottle, and churn splitter if appropriate, will be negated by rinsing each three times with native water prior to collection of the sample. The grab samples will be collected at approximately midstream (sampling point S2). Where hazardous conditions prevent midstream sampling, the grab sample will be collected at sampling location S1 or S3. Grab samples will be collected at approximately ½-foot below the water surface using a swing sampler, and immediately transferred to a churn splitter. While churning the sample in the churn splitter, the sample will be distributed into the appropriately labeled sample bottle and placed into an ice chest with a sufficient quantity wet ice.

The YSI 6600 multi-parameter water quality sonde will be used to collect field measurements for the following parameters: DO, pH, temperature, and EC at the center point at each sampling location from about 1-foot below the water surface. Where hazardous conditions prevent midstream sampling, the YSI measurements will be taken at sampling location S1 or S3. After at least two minutes or when the readings have reached equilibrium, the values for these parameters will be manually recorded in the field data sheet. Turbidity measurements using the Hach 2100P turbidimeter will be performed on the collected grab samples. In the event that a field turbidity measurement falls outside of the range of the equipment (1000 NTU), the turbidity values will be reported as >1,000 NTU. This out-of-range data may be used in assessing compliance, but will not be used in calculating the relationship between TSS and turbidity.

2.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Sample-holding times will be adhered to, as prescribed by USEPA and 40 CFR 136. Specifically, the required preservation techniques and holding times for all of the constituents which the laboratory will be analizing are listed in Table 4, below.

Table 4: Required Containers, Preservatives, Techniques, and Holding Times

Constituent	Container	Preservation Technique	Holding Time
Turbidity	1-L low density polyethylene bottle	Cool 4 °C	48 hours
Total Suspended Solids			7 days
DDT, DDE, DDD	2-L Amber Glass bottle	Cool 4 °C	7 days

Each sample container will be labeled with a unique sample identification code. All samples (including QC samples) for laboratory analyses will immediately be stored in an ice chest, and will remain in the custody of Regional Board staff until the samples are delivered to the laboratory. The ice chest will contain sufficient ice to maintain the samples at a temperature below 4°C at all times until they are relinquished to the lab staff. All samples will be delivered with chain of custody forms. A sample chain of custody form to be used for this project is

included in Appendix II. Any violation of holding times or other sample handling and custody requirements will be documented in the quality control records and reported to the Project Manager and the QA Officer. Any violations thereof will be taken into account when evaluating the data.

2.4 ANALYTICAL METHODS REQUIREMENTS

As prescribed by the State Water Resources Control Board's "*Quality Assurance Program Plan*", each analytical laboratory used for sample analysis must have a written QA Laboratory Manual describing the analytical method requirements. The lab will use USEPA approved methods as outlined in Table No. 5.

Table 5: Sampling Constituents and Methods

Constituent	USEPA Method	Reporting Limit	Units
Turbidity	180.1	0.10	NTU
Total Suspended Solids	160.2	1.0	mg/L
DDT, DDE, DDD	8081	0.025-2.0	µg/L

2.5 QUALITY CONTROL REQUIREMENTS

In order to assess whether the data quality requirements of this project are being met, a number of quality control checks will be implemented. The calibration and maintenance of Regional Board Laboratory instruments and the general operation of the Regional Board laboratory are subject to the requirements of the State Board Quality Assurance Program Plan and the Regional Board Quality Assurance Program for the Laboratory. All QC samples will be placed in an ice chest, and kept at 4 °C, for transport to the lab. Specifically:

- Field blank samples will be collected in the field by dispensing deionized water into the appropriately labeled sample container. The reported value is used to check for laboratory accuracy. One field blank will be collected and submitted for analysis for each constituent, for each day of sampling.

- Field duplicate samples will be prepared from a grab sample of the water being sampled. A grab sample will be collected as described above and split using a churn splitter, into the appropriately labeled containers. Ten percent of the samples collected will be field duplicates, with a minimum of one set of field duplicate samples for each analyte collected per day of sampling.

- Double blind spike samples for turbidity and TSS (one each) will be prepared by an independent lab and submitted to the laboratory for analyses once per year. All projects requiring submittal of spike samples will be coordinated in a manner such that, when possible, one set of spikes will be submitted to the laboratory for all projects.

- To ensure preservation requirements are met, a random sample will be chosen by the laboratory from each ice chest for temperature measurement.

QC samples will be submitted to the lab along with the "real" surface water samples being submitted (i.e., the laboratory will not be informed in any way as to which samples are control samples and which samples are from the aforementioned surface waters). A summary of Quality Control Sample Requirements is located in Table 6.

Table 6: Quality Control Sample Requirements

Quality Control Samples	Number of Samples	Frequency
*Duplicate Samples (All parameters)	10%	Per event
Field Blanks (All parameters)	1	Per day/event
Spike Samples (TSS, Turbidity only)	1	Annually
Temperature blank[1]	1	Per ice chest

[1]Temperature blank may be omitted if temperature is read from a random sample bottle using an infrared thermometer.

2.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, & MAINTENANCE REQUIREMENTS

All Regional Board staff participating in the project will be trained in the operation, calibration, and maintenance of the field instruments. The manufacturer's instruction manuals will be readily available for field personnel. The instruments will be maintained and calibrated in accordance with the manufacturer's instructions and recommendations. Calibration, inspection, and maintenance of field instruments are performed by laboratory personnel prior to all sampling events.

2.7 INSTRUMENT CALIBRATION AND FREQUENCY

The YSI 6600 sonde and the Hach 2100P Turbidimeter will be calibrated in the Regional Board laboratory prior to each sampling event. Dissolved oxygen will be calibrated prior to each sample collection, using ambient air. Pre and post sampling calibration check will be performed at each sampling site by field personnel. This process involves checking and recording the DO output. If it is within 2% of saturation, recalibration of DO is required. The pre and post calibration check data (DO output) will be recorded on the field datasheet. If recalibration is required, calibration data will be recorded on a calibration log sheet. A post-calibration for all parameters will be performed when the sonde is returned to the office, or as soon as practical. Results of calibration measurements will be documented in the field log notebook and submitted to the QA Officer. Table 7, below illustrates the YSI 6600 sonde and the Hach 2100P specifications.

Table 7: Specifications for the YSI 6600 Sonde & Hach 2100P Turbidimeter.

Parameter	Operating Range	Accuracy	Resolution	Calibration Standard
pH	0 to 14 units	± 0.2 units	0.01 units	2-pt, with pH buffered solutions
Temperature	– 5 to 45 °C	± 0.15 °C	0.01 °C	not required
DO	0 to 20 mg/L	± 0.2 mg/L	0.01 mg/L	saturated air
EC	0 to 100 mS/cm	± 1% of range	4 digits	KCl
Turbidity	0-1000 NTU	+/- 1% or 0.01 NTU[1]	0.01 NTU	Gelex Standards

[1]Whichever is greater (with Gelex standards).

2.8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES/CONSUMABLES

The Lead Field Sampler will ensure that the sample bottles have no defects, and that all sample bottles have been prepared properly.

No other special requirements are needed.

2.9 DATA ACQUISITION REQUIREMENTS (NON-DIRECT METHODS)

Only data collected from this Project and historic data from the TMDL development program, which have already passed QC criteria, will be used. No data will be used from other sources unless the data also meet the QA requirements set herein.

2.10 DATA MANAGEMENT

The Project Manager will maintain field datasheets and chain of custody forms in the project file. Field measurement data will be uploaded from the YSI using ECOLAB software, and analyses results will be obtained in electronic form from the lab. The Project Manager will submit all data in MS Excel format to the QA Officer. After verification and approval by the QA Officer, the Project Manager will download the data into the project database (MS Excel format) and store it on the Regional Board's local area network (LAN). After a period of twelve months, statistical analyses of the data from each sampling point will be employed to calculate an annual average to be used in determining compliance of each drainshed as per the Implementation Section of the TMDL.

3. ASSESSMENT AND OVERSIGHT

3.1 ASSESSMENT AND RESPONSE ACTIONS

Surveillance of the records and overall status of the project will be conducted by the QA Officer to ensure that all of the requirements of the QAPP are being met. Surveillance will be conducted after each sampling event, after all laboratory results have been received for that sampling event. A technical systems audit will also be performed by the QA Officer, as discussed in Section 4.1.1. Also, an annual data quality assessment of the applicability of the data will be performed to assess the handling of all data and to correct any errors found in the project database (see Section 4.1.3).

3.2 REPORTS TO MANAGEMENT

The Project Manager will prepare quarterly and annual project reports. The quarterly project reports will include a summary of the activities performed, the resulting data, and the quality of the resulting data, any problems encountered and their solutions and will identify any samples that indicate violations of Water Quality Standards. The annual project reports will include a statistical analysis of the results indicating drainshed loading, any decrease or increase in loading at the drainshed boundaries, drainsheds which are out of compliance, recommendations for TMDL modification, and the relationship of TSS to turbidity.

4. DATA VALIDATION AND USABILITY

4.1 DATA REVIEW, VERIFICATION AND VALIDATION

Data objectives for this project do not require a full, formal, and independent data validation. The data has no legal requirement for independent validation. Although the data are considered legally defensible as presented herein, all records will be available for independent evaluation should the need arise at a later date.

The QA Officer will be responsible for validating the project's data to ensure that QA guidelines have been followed by performing:

4.1.1 Technical Systems Audit

The QA Officer will prepare or oversee a technical systems audit for each sampling event, after all laboratory results are received. Data will be validated to determine if the collection and analyses procedure conformed to the QAPP. The review will consider field notes, field datasheets, chain of custody forms, laboratory analysis forms, and calibration assessment (determines potential error in field measurements). Documentation of results will occur within 15 days, and will describe data reviewed, review criteria, and data usability.

Unacceptable departures from sample collection procedures include the use of contaminated sampling bottles, the lack of critical sample collection information, or any other activity which would result in the cross contamination or incorrect identification of samples.

Departures from the sample handling and custody procedures contained in Section 2.3 of this report will be determined through the review of chain of custody forms and laboratory analysis forms. In order for data to be considered valid for meeting the data quality objectives of this study, all chain of custody forms must be in the possession of the Project Manager, and strict adherence to holding times and temperatures must be followed. Data generated from samples that do not meet these requirements will not be considered valid for use in this study.

Verification of proper calibration of the YSI sonde and the Hach 2100P turbidimeter will be performed during the audit of data quality through a review of the quality control records. Calibration values will also be assessed to determine the potential error in the field measurements. If calibration values have errors that exceed acceptable error tolerances, the measurements obtained prior to that calibration, but after the previous calibration, will be labeled suspect and further investigated to determine if they are valid for use in this study.

The QA Officer will then ensure that data are entered into the database. It is conceivable, however, that errors could occur in entering the data (e.g., transposing the decimal point for a particular result or keying in the wrong Sample ID). Therefore, once a data set has been entered into the database, all records will be checked to ensure accuracy.

4.1.2 Performance Evaluation of Laboratories

Validation of laboratory data will be performed in the Audit of Data Quality by assessing the results of QC sample analyses. Lab data will be validated for precision, accuracy, and completeness according to the criteria specified in Section 1.6.

In the event that QC analyses do not meet the specified criteria, the data will be labeled as "suspect", the lab will be notified, and the field notes will be re-evaluated. Data sets corresponding to any value that cannot be confirmed, based on the acceptable criteria in Table 1, will be rejected.

In case of missing data, the QA Officer will discuss it with the laboratories submitting the data. In some cases, missing data will be denoted as missing in reports. For all missing data, and any other data requiring special explanation, qualifiers will be included in the database and in data reports. Missing data will be designated as "NR", meaning *Not Reported.*

4.1.3 Data Quality Assessment

The QA Officer will prepare annually an audit of data quality reports, which takes into account all Technical Systems Audits and includes verification of calibration and instrument drift results for the YSI 6600 multi-probe sonde and Hach 2100P turbidimeter, and the results of the laboratory QC samples. Precision, accuracy, and completeness results for laboratory data will also be included in the reports. Also, the reports will assess whether the total error in the data is tolerable, and whether significant departures from the QAPP reduce data set completeness (and thus reduce data set usability for drawing conclusions).

Significant departures from the QAPP will be noted in these reports, and the resulting data will not be validated. Unacceptable departures include, but are not limited to:

- Cross-contamination
- Lack of critical sample collection information
- Violation of sample holding times and temperatures

5. HEALTH AND SAFETY PLAN

5.1 CONTAMINATION CONTAINMENT ZONES

The contaminated areas for this Project consist of and cover the entire waterways for the aforementioned waters, their banks, the area within 2 feet of the banks, and the bridge at the Outlet. Decontamination zones will be set at least 10 feet away from the banks of the surface waters. The decontamination zone will be used for personnel decontamination and will include wash water, soap, paper towels, and trash bags. All contaminated solid waste material will be placed in trash bags for proper disposal. Only biodegradable antibacterial soap will be used at the site. Wash water runoff will be contained and disposed of in the surface waters. The Clean area will be set at least 20 feet away from the banks of the surface waters.

5.2 PERSONAL PROTECTIVE EQUIPMENT

The general concerns at the sampling sites are the potential exposure to toxicants present in the waters being sampled, risk of sunburn, excessive heat exposure, insect bite, and possibly snakebites. In addition, the sampling crew should be aware of the risk of falling into the waterways. No less than two experienced samplers will be out in the field at one time. (The sampling crew will also have a functional cellular phone in one of the vehicles).

- Any member of the sampling team has the authority to stop the sampling event when he/she determines that conditions at the site (e.g., rain, dust, local emergency, etc.) preclude safe sampling. A Hazard Evaluation Plan (HEP) will be done for each day of sampling. The lead field sampler will be responsible for preparing the HEP.

- To reduce the risk of exposure while collecting/transporting samples, Latex Examination Gloves must be worn. The Contaminated Zone must not be entered without the aforementioned Personal Protective Equipment (PPE).

- To reduce the risk of heat exposure and sunburn, samplers should wear sunscreen and carry in their vehicle cold drinking water. If any of the samplers begin to experience symptoms of heat exhaustion, such as cramps or dizziness, he or she will immediately be removed from the sun and given plenty of cool liquids. If these symptoms persist, he or she will be taken to the nearest hospital.

- Extra caution should be used when working near or around the drains to reduce the risk of potentially falling in.

- To reduce the risk of insect bites, samplers should use insect repellent.

- To reduce the possibility of snakebite, samplers will check areas for snakes prior to entering the area. If snakebite occurs, ice will be placed on the bite. The sampler will be immediately transported to the nearest medical facility.

5.3 PERSONNEL DECONTAMINATION PROCEDURES

The Clean Zone must not be entered with contaminated PPE. All team members coming out of the Contaminated Zones must immediately proceed to the Decontamination Zones and use the following decontamination procedures before proceeding to Clean Zone:

1. Remove latex gloves carefully to avoid contact with bare skin and dispose of them in the trash bag. Thoroughly wash hands with antibacterial soap; and

2. Dispose of wash water into surface water just sampled.

Note: everything that is touched (pens, pencils, rinse water bottles, probes, etc.) with dirty gloves could be contaminated. Avoid touching these items with bare skin.

5.3.1 Emergency Numbers and Facilities

All sampling personnel will have access to a cellular phone to call 911 in case of an emergency. The hospital nearest the sampling locations are listed in Table 8 below:

Table 8: Nearest Hospitals to Sampling Locations

Sampling Location	Medical Facility	Address	Phone
AR-B	EL Centro Regional Medical Center	Imperial and Ross Ave., El Centro	(760) 339-7100
ARD10	EL Centro Regional Medical Center	Imperial and Ross Ave., El Centro	(760) 339-7100
ARD8	EL Centro Regional Medical Center	Imperial and Ross Ave., El Centro	(760) 339-7100
ARD6a	Pioneers Memorial Hospital	207 West Legion, Brawley	(760) 351-3333
ARD6	Pioneers Memorial Hospital	207 West Legion, Brawley	(760) 351-3333
ARD3	Pioneers Memorial Hospital	207 West Legion, Brawley	(760) 351-3333
AR-O	Pioneers Memorial Hospital	207 West Legion, Brawley	(760) 351-3333

In case of an emergency, sampling personnel should also contact the Regional Board Safety Officer, Doug Wylie, as soon as practical at 760-346-6585 or 760-341-7491.

5.3.2 After Sampling

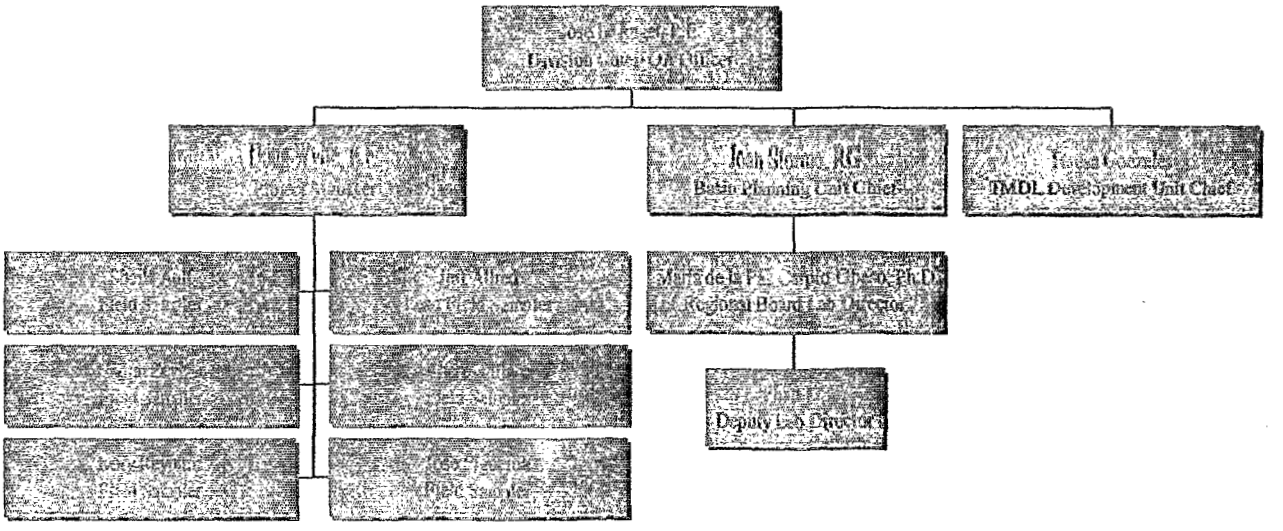
Place samples into Regional Board lab refrigerator or keep in an ice chest filled with wet ice; keep water drained from ice chests to avoid soaking container labels. Make copies of field notes and put original in the project working file. Contaminated equipment should be packed in designated containers for transport to the Regional Board office. Decontaminate and properly clean ALL items, which were exposed in the field in accordance with USGS National Field Manual for the Collection of Water-Quality Data, Chapter A3. Cleaning of Equipment for Water Sampling (See Attachment IV).

6. REFERENCES

U.S. Environmental Protection Agency. 2001. EPA Requirements for Quality Assurance Project Plans, EPA QA/R5. EPA Publication number 240/B-01/003. U.S. Environmental Protection Agency, Washington, D.C.

State Water Resources Control Board (State of California), 2001. Quality Assurance Program Plan.

APPENDIX I, PROJECT ORGANIZATION CHART



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APPENDIX II, SAMPLE CHAIN OF CUSTODY FORM

Techniques of Water-Resources Investigations

Book 9
Handbooks for Water-Resources Investigations

National Field Manual for the Collection of Water-Quality Data

Chapter A3. CLEANING OF EQUIPMENT FOR WATER SAMPLING

Edited by
F.D. Wilde, D.B. Radtke, Jacob Gibs,
and R.T. Iwatsubo

USGS
science for a changing world

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Foreword

The mission of the Water Resources Division of the U.S. Geological Survey (USGS) is to provide the information and understanding needed for wise management of the Nation's water resources. Inherent in this mission is the responsibility to collect data that accurately describe the physical, chemical, and biological attributes of water systems. These data are used for environmental and resource assessments by the USGS, other government and scientific agencies, and the general public. Reliable and objective data are essential to the credibility and impartiality of the water-resources appraisals carried out by the USGS.

The development and use of a *National Field Manual* is necessary to achieve consistency in the scientific methods and procedures used, to document those methods and procedures, and to maintain technical expertise. USGS field personnel use this manual to ensure that data collected are of the quality required to fulfill our mission.

Robert M. Hirsch

Robert M. Hirsch
Chief Hydrologist

Techniques of Water-Resources Investigations

Book 9
Handbooks for Water-Resources Investigations

Chapters of Section A: National Field Manual for the Collection of Water-Quality Data

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[1]Bold type indicates published chapters and chapter sections, and shaded type indicates chapters and chapter sections that are in preparation.

CLEANING OF EQUIPMENT FOR WATER SAMPLING A3.

National Field Manual for the Collection of Water-Quality Data

Chapter A3.

E.S. Babcock & Sons, Inc.
6100 Quail Valley Court Riverside, CA 92507
(909) 653-3351 • FAX (909) 653-1662

Chain of Custody & Sample Information Record

Client: Contact: Phone No.

Project Name: _____
Project Location:

Turn Around Time: Routine 3-5 Days 48 Hours 24 Hours *(Rushes Require Approval, Additional Charges May Apply)*

Sampler Information

Name: _____
Employer: _____
Signature: _____

ESB #	Sample ID	Date	Time	# of Containers & Preservatives: Unpreserved	H_2SO_4	HCl	HNO_3	$Na_2S_2O_3$	NaOH			Total # of Containers	Analysis Requested	Matrix	Notes
														DW = Drinking Water WW = Wastewater GW = Groundwater S = Soil SG = Sludge L = Liquid M = Miscellaneous	

Relinquished By (sign)	Print Name / Company	Date / Time	Received By (Sign)	Print Name / Company

(For Lab Use Only) *Sample Integrity Upon Receipt*

Sample(s) Submitted on Ice? Yes No Temperature
Custody Seal(s) Intact? Yes No N/A °C
Sample(s) Intact? Yes No

Lab No. _____

Page ___ of ___

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APPENDIX III, DECONTAMINATION PROCEDURES

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Illustrations

Table

Chapter A3. CLEANING OF EQUIPMENT FOR WATER SAMPLING

Edited by Franceska D. Wilde, Dean B. Radtke, Jacob Gibs, and Rick T. Iwatsubo

ABSTRACT

The *National Field Manual for the Collection of Water-Quality Data (National Field Manual)* provides protocols and guidelines for U.S. Geological Survey (USGS) personnel who collect data used to assess the quality of the Nation's surface-water and ground-water resources. Chapter A3 describes procedures for cleaning the equipment used to collect and process water samples and for assessing the efficacy of the equipment-cleaning process. This chapter is designed for use with the other chapters of this field manual.

Each chapter of the *National Field Manual* is published separately and revised periodically. Newly published and revised chapters will be announced on the USGS Home Page on the World Wide Web under "New Publications of the U.S. Geological Survey." The URL for this page is <http://water.usgs.gov/lookup/get?newpubs>.

INTRODUCTION

As part of its mission, the U.S. Geological Survey (USGS) collects data needed to assess the quality of our Nation's water resources. The *National Field Manual for the Collection of Water-Quality Data (National Field Manual)* describes protocols (requirements and recommendations) and provides guidelines for USGS personnel who collect data on the Nation's surface-water and ground-water resources. Chapter A3 describes procedures for cleaning the

equipment used to collect and process samples of surface water and ground water and procedures for assessing the efficacy of the equipment-cleaning process.

The *National Field Manual* is Section A of Book 9 of the USGS publication series Techniques of Water-Resources Investigations (TWRI). Each chapter of this manual is published as a separate report. Chapter numbers are preceded by an "A" to indicate that the report is part of the *National Field Manual*. Other chapters and sections of other chapters of the *National Field Manual* are referred to in this report by the abbreviation "NFM" and the specific chapter and (or) section number. For example, general information on field measurements of ground water is covered in section 6.0.2 of Chapter A6, "Field Measurements," and would be cited as NFM 6.0.2.

PURPOSE AND SCOPE

The *National Field Manual* is targeted specifically toward field personnel in order to *(1)* establish and communicate scientifically sound methods and procedures, *(2)* provide methods that minimize data bias and, when properly applied, result in data that are reproducible within acceptable limits of variability, *(3)* encourage consistent use of field methods for the purpose of producing nationally comparable data, and (4) provide citable documentation for USGS water-quality data-collection protocols.

The equipment-cleaning procedures presented in this chapter are adequate for routine environmental conditions. A modification of the cleaning procedures might be required, for example, in order to decontaminate equipment adequately after sampling at sites where analyte concentrations are large. Modifications to the standard procedures described in this chapter must be documented and quality controlled.

REQUIREMENTS AND RECOMMENDATIONS

As used in the *National Field Manual*, the terms **required** and **recommended** have USGS-specific meanings.

Required (require, required, or requirements) pertains to USGS protocols and indicates that a specific USGS Office of Water Quality (OWQ) policy has been established on the basis of research and (or) consensus of the technical staff and has been reviewed by water-quality specialists and District[1] or other professional personnel, as appropriate. Technical memorandums or other unpublished documents that define the policy pertinent to such requirements are cited in this chapter. Personnel are instructed to use required equipment or procedures as described in this chapter. Departure from or modifications to the stipulated requirements that might be necessary to accomplish specific data-quality requirements or study objectives must be based on referenced research and good field judgment and must be quality assured and documented.

Recommended (recommend, recommended, or recommendation) pertains to USGS protocols and indicates that USGS Office of Water Quality policy recognizes that one or several alternatives to a given procedure or equipment selection are acceptable on the basis of research and (or) consensus. Specific data-quality requirements, study objectives, or other constraints affect the choice of recommended equipment or procedures. Selection from among the recommended alternatives should be based on referenced research and good field judgment, and reasons for the selection should be documented. Departure from or modifications to recommended procedures must be quality assured and documented.

[1]**District** refers to a water-data collecting organizational unit of the USGS located in any of the States or Territories of the United States.

FIELD MANUAL REVIEW AND REVISION

Chapters of the *National Field Manual* will be reviewed, revised, and reissued periodically to correct any errors, incorporate technical advances, and address additional topics. Please send comments or corrections to NFM-QW, USGS, 412 National Center, Reston, VA 20192 (or send electronic mail to nfm-owq@usgs.gov). Information regarding the status and any errata of this and other chapters can be found at the beginning of the electronic version of each chapter, located in the Publications Section of the following website: http://water.usgs.gov/lookup/get?owq.

Newly published and revised chapters will be announced on the USGS Home Page on the World Wide Web under "New Publications of the U.S. Geological Survey." The URL for this page is <http://water.usgs.gov/lookup/get?newpubs>.

ACKNOWLEDGMENTS

The information in this chapter of the *National Field Manual* is based principally on the work of Sandstrom (1990), Horowitz and others (1994), Shelton (1994), and Koterba and others (1995).

The editors wish to thank and pay tribute to R.W. Lee and S.W. McKenzie, who were responsible for final technical review and who contributed to the accuracy, quality, and usability of this report. We would like to express appreciation to the following colleague reviewers for helping to improve this report: H.D. Ardourel, B.A. Bernard, K.K. Fitzgerald, D.S. Francy, S.R. Glodt, V.J. Kelly, S.L. Lane, S.K. Sando, C.A. Silcox, and W.R. White. The editors are indebted to I.M. Collies, C.M. Eberle, B.B. Palcsak, and Chester Zenone for their valuable editorial contributions, and to C.T. Mendelsohn, L.E. Menoyo, and A.M. Weaver, whose production assistance was instrumental in maintaining the quality of the report.

Special thanks go to T.L. Miller, whose encouragement and faith in this project has been instrumental to its achievement; and to D.A. Rickert and J.R. Ward, who provided the support needed to produce a national field manual for water-quality studies.

A3. CLEANING OF EQUIPMENT FOR WATER SAMPLING

Equipment cleaning (decontamination): Applying cleaning solutions to the surfaces of equipment or using other nondestructive procedures (such as steam cleaning) to remove foreign substances that could affect the concentrations of analytes in samples.

USGS policy requires that equipment for water samples be properly cleaned before contacting the sample and that the effectiveness of cleaning procedures be quality controlled (Sandstrom, 1990; Horowitz and others, 1994; Koterba and others, 1995). The goal of equipment cleaning is to help ensure that the equipment is not a source of foreign substances that could affect the ambient concentrations or chemistry of target analytes in samples. Standard procedures are described in this chapter for when, where, and how to clean equipment constructed of various materials and to collect equipment blanks and field blanks for quality control. Space is commonly dedicated in an office laboratory for equipment cleaning and for storage of cleaning supplies. In this report this work space can include the Field Service Unit or other dedicated office space.

- Clean all sample-collection and sample-processing equipment before use.
  - Manufacturing residues must be removed from new equipment.
  - Dust and any other foreign substances must be removed from equipment that has been in storage.
  - Substances adhering to equipment from previous sampling must be removed.
- Prevent cross contamination between sampling sites by rinsing equipment with deionized water (DIW) while equipment is still wet, and then clean equipment as prescribed in this chapter before transporting it to the next site.
- Do not substitute field rinsing with sample water for the equipment-cleaning procedures described in this chapter.
- Collect equipment blanks and field blanks for quality control. A minimum of one equipment blank per year is required for each piece of equipment. The frequency of collecting blanks normally is based on study objectives and site conditions.

To help prevent sample and site contamination, be sure to use properly cleaned equipment.

SUPPLIES FOR EQUIPMENT CLEANING 3.1

By D.B. Radtke, A.J. Horowitz, and M.W. Sandstrom

The supplies commonly used to clean sample-collection and sample-processing equipment are listed in table 3-1. Cleaning supplies are to be stored in a contaminant-free cabinet. Follow safety instructions regarding the storage of chemical reagents (NFM 9).

Before gathering the cleaning supplies, check the construction materials (for example, metal, glass, or plastic) of washbasins and other cleaning items relative to the samples to be collected.

- **For analysis of inorganic constituents**—Basins, brushes, and other items used for cleaning should be constructed of a suitable nonmetallic material such as uncolored or white polypropylene, polyethylene, or other plastic. **Do not use cleaning agents or items that might leach or sorb metals if the equipment to be cleaned will be used for samples to be analyzed for trace elements.**
- **For analysis of organic compounds**—Basins and other cleaning items can be constructed of metal, glass, or plastic materials. Stainless steel is recommended if methanol will be used. **Do not use cleaning agents or items that might leach, sorb, or leave residues of organic substances that could bias or interfere with the analysis.**

CAUTION: Refer to Material Safety Data Sheets (MSDS) before handling any chemicals.

- **Wear appropriate safety gloves, glasses, and apron when working with corrosive and oxidizing solutions.**
- **When using chemicals, work in a well-ventilated area.**

Table 3-1. Supplies for cleaning equipment used for water-sampling activities

[ACS, American Chemical Society; DIW, distilled/deionized water; µS/cm, microsiemens per centimeter at 25 degrees Celsius; PBW, pesticide-grade blank water; VBW, volatiles and pesticide-grade blank water; IBW, inorganic-grade blank water; L, liter; cm, centimeter; TOC, total organic carbon; DOC, dissolved organic carbon; SOC, suspended organic carbon; NFM, *National Field Manual*; PVC, polyvinyl chloride; IBW, inorganic-grade blank water]

Item	Description and Comments
Acid solution[1]	Hydrochloric: ACS trace-element grade (5 percent by volume in DIW). Nitric: ACS trace-element grade (10 percent by volume in DIW).
Aluminum foil	Organics only: Heavy duty, for work surfaces and equipment.
Bags, plastic or fluorocarbon polymer	Sealable bags with uncolored closure strips, various sizes. Recyclable trash bags are recommended for large equipment storage.
Noncolored plastic sheeting	Clean sheeting used to provide a clean work surface.
Brushes and sponges	Uncolored; plastic components needed for inorganic work.
Distilled/deionized water (DIW)	Maximum specific electrical conductance, 1 µS/cm (usually District produced; Office of Water Quality Memorandum 92.01).
Office-produced organic-grade deionized water	Usable only as a cleaning solution and only as specified in the text. Must not be used to substitute for PBW or VBW.[2]
Detergent	Nonphosphate laboratory soap (for example, Liquinox™).
Gloves, disposable	Powderless, noncolored vinyl, latex, or nitrile (latex or nitrile for use with methanol), assorted sizes.
Inorganic-grade blank water (IBW)[2]	Blank water with certificate of analysis prepared and (or) quality assured by the analyzing laboratory. IBW is required for blank samples.
Jerricans or carboys	For waste solutions and as neutralization container. Neutralization container: 25- to 30-L, polyethylene, wide-mouth, with layer of marble chips. Methanol waste container: Appropriate for flammable liquid.
Methanol	ACS pesticide grade. Methanol is the organic solvent in common use for equipment cleaning, but study requirements might dictate use of a different ACS pesticide-grade solvent.
Neutralization materials	Marble landscape chips (1- to 2-cm chips recommended).[3]
Pesticide-grade blank water (PBW)[2]; volatile-grade blank water (VBW)[2]	Blank water prepared and (or) quality assured by the analyzing laboratory; required for collecting blank samples as follows: PBW for pesticide analysis; VBW for volatile compounds analysis and pesticide analysis; and either PBW or VBW for TOC, DOC, and SOC analyses.
Safety equipment and guidelines (NFM 9)	For example, Material Safety Data Sheets (MSDS), safety glasses, chemical spill kit, apron, emergency phone numbers.

Table 3-1. Supplies for cleaning equipment used for water-sampling activities—*Continued*

Item	Description and Comments
Standpipes for submersible pump	Plastic, glass, or other suitable material; for example, pipette jars or capped PVC casing; one standpipe labeled for blank water and one each for each cleaning solution. (Do not use PVC for methanol.)
Tapwater	If quality is questionable, substitute DIW. Tapwater is more effective for initial and rapid removal of detergent residue.
Tissues	Laboratory grade, lint free, various sizes (for example, Kimwipes™).
Washbasins	One washbasin for each cleaning solution; white or uncolored. Plastic, nonleaching. (Stainless steel is required for methanol.)
Wash bottles (dispenser or squeeze)	Labeled to indicate contents (for example, ACID, DIW, TAP). Fluorocarbon polymer needed for methanol, PBW, VBW, and IBW.

[1]Hydrochloric acid is required if analyzing for nitrogen species; otherwise, nitric acid is acceptable.
[2]PBW and VBW can be obtained from the USGS National Water Quality Laboratory (NWQL). IBW can be obtained from the USGS Quality of Water Service Unit.
[3]Agricultural limestone, soda ash, baking soda, and crushed shells are not recommended (Horowitz and others, 1994).

CAUTION: Methanol is extremely flammable and potentially explosive, emits noxious fumes, and is absorbed through the skin. Observe safety practices when handling methanol or other organic solvents.

- **Wear safety gloves, glasses, and apron.**
- **Work in a well-ventilated area and away from an open flame or sparks.**
- **Make sure that all electrically powered equipment is grounded; alternating current equipment must have a ground-fault interrupter.**
- **Inspect electrical wiring for cuts, breaks, or abrasions where the metal wire is exposed.**
  - **Exposed wires can cause sparks if a short to ground occurs.**
  - **Replace faulty wires—do not rely on fixing with electrical tape.**

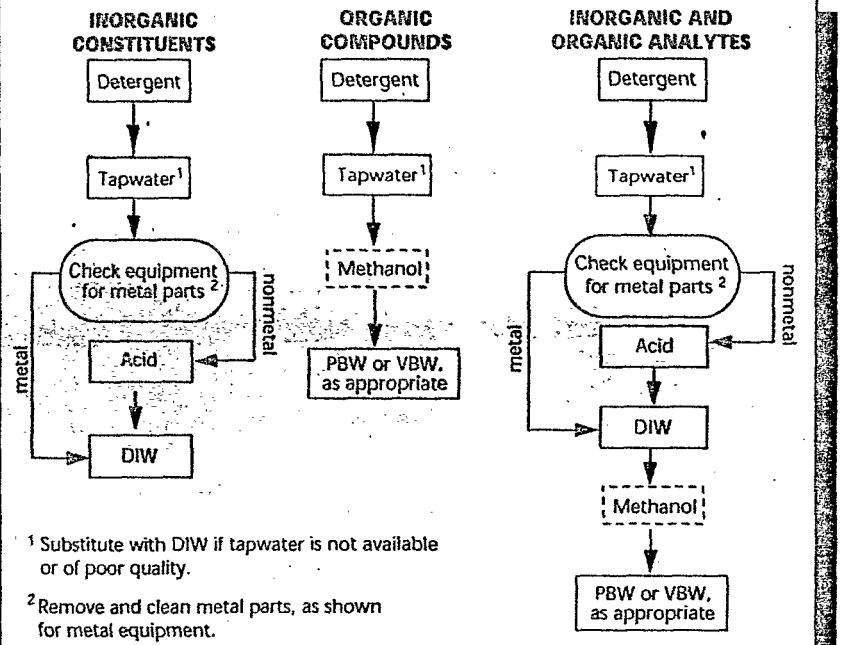
CLEANING PROCEDURES 3.2

By A.J. Horowitz and M.W. Sandstrom

Equipment should be cleaned in an area protected from airborne or other sources of contamination. Procedures to remove contaminants to concentrations below the targeted method-detection levels can vary, depending on the cleaning supplies used, the type of equipment being cleaned, the solubility and concentration of contaminant(s), and the length of time equipment is exposed to contaminant(s). **Examine equipment-blank and field-blank data to determine whether adjustments to the cleaning protocol are needed (section 3.4).**

The cleaning procedure to be used depends on the type(s) of water samples that will be collected and processed. Figure 3-1 summarizes the sequence of cleaning procedures for equipment used to collect samples for inorganic and (or) organic analytes (Sandstrom, 1990; Horowitz and others, 1994; and Koterba and others, 1995).

- **Inspect equipment for stains, cuts, or abrasions. Replace parts as needed.**
  - Replace chipped or cracked glassware.
  - Replace bent sampler nozzles or samplers with bent fins (surface-water samplers).
  - Replace tubing if mold, mildew, or imbedded sediment cannot be removed.
  - Replace cracked or severely crimped O-rings.
  - Repair pump intakes and antibacksiphons that have loose or missing screws.
  - Check the flow manifold and sample tubing to ensure that valves and quick-connect fittings are in good working order; repair or replace as necessary to eliminate any problems.
  - Recoat chipped surface-water samplers with epoxy paint or "plasti-coat." Such samplers must be recoated before use.



[1] Substitute with DIW if tapwater is not available or of poor quality.

[2] Remove and clean metal parts, as shown for metal equipment.

EXPLANATION

DIW	Distilled/deionized water
methanol	Current protocol includes methanol rinse for most types of equipment except that used with samples for organic-carbon analyses. However, this protocol is under review. SAFETY ALERT: Methanol is highly flammable; fumes can be hazardous to human health.
PBW	Pesticide-grade blank water
VBW	Volatiles- and pesticide-grade blank water

Figure 3-1. General sequence for cleaning equipment before sampling for inorganic and (or) organic analytes.

- **Rinse equipment with DIW directly after use while equipment is still wet and before cleaning procedures are implemented.**
- **Place cleaned equipment in doubled storage bags.**

Do not allow collection and processing equipment to sit uncleaned in a field vehicle or elsewhere between field trips.

CLEANING OF EQUIPMENT USED TO SAMPLE FOR INORGANIC CONSTITUENTS 3.2.1

Cleaning of equipment used to collect and process water for analysis of inorganic constituents involves a five-step office-laboratory procedure or a five-step field-site procedure. These procedures are effective for cleaning equipment exposed to water containing concentrations of as much as 50,000 µg/L of iron, 5,000 µg/L each of manganese and zinc, 400 µg/L of copper, 125 µg/L of cobalt, and large concentrations of the other trace elements (Horowitz and others, 1994). The cleaning procedures are summarized in figures 3-2 and 3-3. (These procedures do not apply to field-measurement instruments—see NFM 6.)

Equipment should be cleaned periodically in the office laboratory, where complete disassembly is more practical and more thorough procedures are possible. Compared to cleaning at the field site, cleaning procedures carried out in the office laboratory involve longer exposure of equipment to cleaning solutions, more frequent change of cleaning solution, and greater volumes of rinse water.

- To minimize field cleaning of equipment between sampling sites, preclean a separate set of equipment for each site.

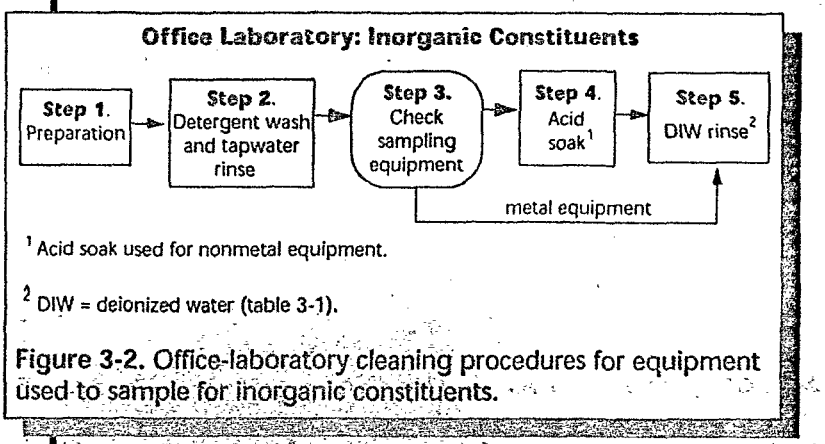


Figure 3-2. Office-laboratory cleaning procedures for equipment used to sample for inorganic constituents.

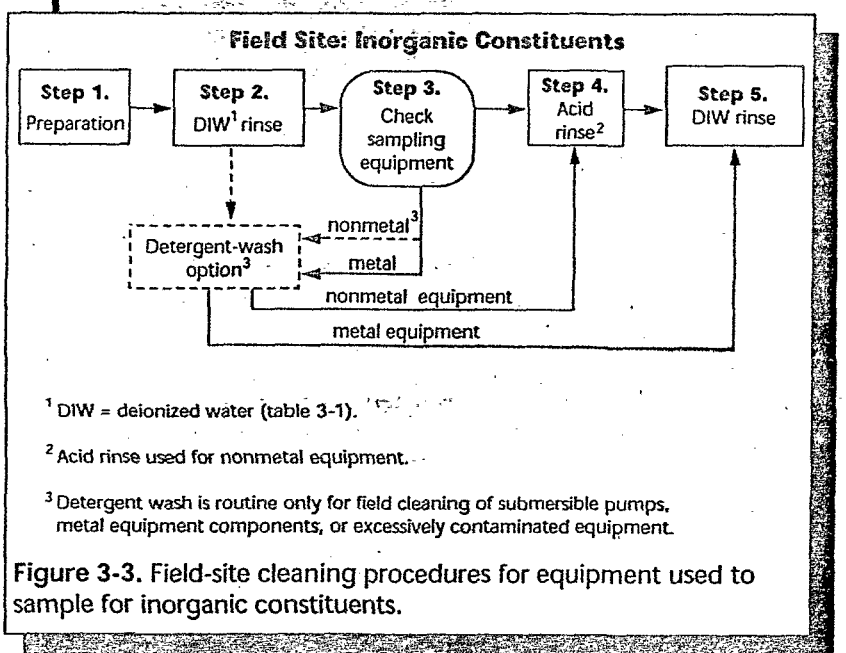


Figure 3-3. Field-site cleaning procedures for equipment used to sample for inorganic constituents.

- If individual or dedicated sets of equipment for each field site are not available or cannot be precleaned, clean the equipment onsite and process additional field blanks during each field trip (Horowitz and others, 1994; Koterba and others, 1995).

- Return excessively contaminated equipment to the office laboratory for rigorous cleaning before reuse.

- After cleaning, document completion of and any modifications to the cleaning procedures.

Equipment-cleaning procedures for inorganic constituents

Standard procedures for office-laboratory and field-site cleaning of equipment used to collect and process samples for analysis of inorganic constituent are described below and summarized in figures 3-2 and 3-3. Not all the steps listed apply to all equipment, however. For example,

- Omit detergent step when cleaning plastic bags for surface-water samplers.

- Omit acid step when cleaning submersible pumps, the churn-splitter spigot, or other equipment constructed of stainless steel or other metallic material.

- Omit detergent and acid steps when cleaning sample bottles.

Be sure to check the specific procedures for sample bottles and other selected equipment listed in section 3.3 before proceeding with the office-laboratory and field-site procedures.

Step 1. Preparation at the office laboratory or field site (figs. 3-2 and 3-3).

a. Prepare a contaminant-free space for cleaning and drying the cleaning supplies and sample-collection and sample-processing equipment.

i. Gather the cleaning supplies, the equipment to be cleaned, and the plastic bags or other material with which to wrap the cleaned equipment. Check table 3-1 for the cleaning supplies needed.

ii. Place clean plastic sheeting over the work surface.

iii. Put on disposable, powderless gloves[2], a laboratory coat or apron, and safety glasses.

iv. Prepare the detergent solution, using a nonphosphate, laboratory-grade detergent.

- **Office laboratory** (fig. 3-2). Use 0.1- to 2-percent solution, volume-to-volume (v/v), using a higher concentration for dirtier equipment.
- **Field site** (fig. 3-3). Use 0.1- to 0.2-percent solution, v/v.

v. Prepare the acid solution, using a 5-percent v/v dilution of ACS trace-element-grade hydrochloric acid (HCl) in DIW.

- **Add the acid to the water**, not water to acid (NFM 9).
- If nitric acid (HNO_3^-) will be used, prepare a 10-percent solution (v/v) of ACS trace-element-grade acid in DIW.

vi. Label each washbasin, standpipe, and wash bottle to indicate the solution it will contain. Use a black waterproof marker.

vii. Unwrap the equipment to be cleaned and discard the storage bags. Change gloves.

b. Clean the items used to clean the equipment.

i. Fill washbasins and (or) standpipes with the nonphosphate detergent solution. Put wash bottles, scrub brushes, and other small items used for cleaning into a washbasin. **Soak for 30 minutes.**

ii. Scrub interior and exterior sides of basins and standpipes with soft scrub brushes. Fill wash bottles with a soapy solution and shake vigorously.

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[2]Refers to laboratory gloves that are nonpowdered on the inside and intended for disposal after one use. Glove materials must be appropriate for the work to be carried out and the solutions and equipment to be contacted. For example, vinyl gloves are appropriate for most sampling activities but not when working with methanol or other organic solvents.

iii. Rinse all items thoroughly with tapwater to remove detergent residue. No detergent bubbles should appear when fresh tapwater is agitated in the basin, standpipe, or wash bottle.

iv. Rinse washbasins with DIW.

v. Pour 5-percent HCl (or 10 percent HNO_3^-) solution into washbasins, standpipes, and wash bottles. Soak for 30 minutes. **Do not soak items with metal parts (exposed or hidden) in an acid solution.**

vi. Discard used acid solution into a neutralization container containing a bottom layer of marble chips (Step 4d).

vii. Rinse washbasins, standpipes, and wash bottles with DIW. Dispose of DIW using directions in Step 4d.

c. Disassemble sample-collection and sample-processing equipment. Change gloves.

- Submersible pumps should be disassembled periodically for office cleaning, but they are not usually disassembled for field cleaning.
- Processing and preservation chamber frames should be cleaned periodically using office-laboratory cleaning procedures. Field cleaning is needed only if the cover is slipped over the frame instead of being clipped to the inside of the frame.

Step 2. Detergent wash and tapwater rinse—Office laboratory (fig. 3-2).

a. Place small equipment parts into washbasin labeled for detergent and fill with a 0.1- to 2-percent solution of nonphosphate laboratory detergent. The amount of detergent depends on the hardness of the tapwater and the degree to which the equipment is dirty or contaminated.

b. Soak equipment and tubing for 30 minutes: fill tubing with solution and keep submerged.

c. Scrub exterior and interior of equipment surfaces to the extent possible, using a firm sponge or soft brush to remove any adhering material such as oil and grease, sediment, algae, and chemical deposits. Pay particular attention to grooves and crevices, O-rings, nozzles, and other spaces where inorganic or organic materials might be trapped. Change gloves.

d. Rinse equipment thoroughly with warm tapwater to remove detergent residue. Equipment rinsing is completed when no soap bubbles appear after the rinse water is agitated. Change gloves.

Step 2. DIW rinse and detergent-wash option—Field site (fig. 3-3).

For the DIW rinse:

a. Rinse equipment and tubing with DIW. Pay particular attention to removing material from grooves and crevices, O-rings, nozzles, and places where materials might be trapped. Note that equipment should already have had one DIW rinse directly after contact with sample water and before the equipment had a chance to dry.

b. Change gloves. Proceed to field detergent-wash option only for metal equipment components or for equipment that has become excessively contaminated.

For the detergent-wash option:

A field detergent wash is used for between-site cleaning of submersible pumps, metal components of equipment, or for equipment that has become greasy or otherwise coated and requires detergent to remove foreign materials; specific instructions for submersible pumps are given in section 3.3.9.

a. Place small equipment, tubing, and parts into basin labeled "detergent" and fill with a 0.1- to 0.2-percent detergent solution. Soak for about 10 minutes, or keep equipment assembled and circulate the solution through pump tubing for 5 to 10 cycles.

b. Scrub equipment surfaces with a firm sponge or soft brush to remove any adhering material such as oil and grease, sediment, algae, or chemical deposits. Pay particular attention to grooves and crevices, O-rings, nozzles, and other places where materials might be trapped. Change gloves.

c. Rinse equipment thoroughly with tapwater to remove detergent residue. Use DIW if tapwater is unavailable or is suspected of having a quality so poor as to contaminate the equipment. If necessary, use a wash bottle filled with DIW or tapwater to rinse hard-to-reach places; pump tapwater through assembled equipment for five or more tubing volumes. Equipment rinsing is complete when no soap bubbles appear after agitating the rinse water. If nonmetal equipment has been detergent-washed, go to Step 4.

d. Place equipment into acid-solution washbasin. Change gloves.

Step 3. Check equipment—Office laboratory and field site (figs. 3-2 and 3-3).

— Nonmetal equipment or equipment with removable metal parts: remove any metal parts and go to Step 4.

— Metal equipment components or excessively contaminated equipment: go to Step 2, detergent-wash option at the field site and then to Step 5, DIW rinse.

Step 4. Acid soak/rinse—Office laboratory and field site (figs. 3-2 and 3-3).

For equipment constructed primarily of glass or fluorocarbon polymer or some other plastic, soak (office laboratory) or rinse (field site) in a 5-percent (v/v) HCl solution to remove any remaining organic films and inorganic deposits.

TECHNICAL NOTE: A 10-percent (v/v) HNO_3^- solution can be used instead of HCl if samples to be collected with the equipment will not be analyzed for nitrogen species.

CAUTION: Wear safety glasses and other protective apparel when working with acids.

a. Place nonmetal equipment and tubing into the washbasin labeled "acid solution."

b. **Office laboratory.** Fill basin with dilute HCl solution (see TECHNICAL NOTE above). Soak equipment and tubing for 30 minutes. Carefully swirl the acid solution several times during the 30-minute soak to enhance removal of mineral encrustations.

c. **Field site.** Using a wash bottle filled with 5-percent HCl solution (see TECHNICAL NOTE above), rinse exterior of equipment and tubing. Pump acid solution through the equipment and tubing, using a peristaltic pump.

d. Carefully pour or pump the used acid solution into a neutralization container with marble chips covering the bottom (table 3-1). Do not reuse the acid solution.

- Do not fill the neutralization container more than three-fourths full of acid solution.
- Ventilate container and workspace to allow for safe escape of carbon dioxide gas during dissolution of marble chips.

- Check the solution pH periodically using narrow range pH indicator strips. Neutralization is complete when the solution pH is greater than 6.0 or the original DIW pH.
- Discard the neutral solution, as appropriate.
- Rinse the container with tapwater but retain any undissolved marble chips. Replenish chips to form a layer on the bottom of the neutralization container.

Step 5. DIW rinse—Office laboratory or field site (figs. 3-2 and 3-3).

a. Place equipment into the cleaned washbasin labeled DIW. Change gloves.

b. **Office laboratory.** Rinse exterior and interior of each piece of equipment and tubing thoroughly with DIW and place on a clean surface to dry or into a clean IBW washbasin if blank samples will be collected to quality control the cleaning procedures.

c. **Field site.** Pump DIW through equipment.

d. Pour or discharge DIW rinse water into neutralization container. Change gloves.

e. Continue DIW rinsing until rinse-water pH is greater than 6.0 or the original DIW pH.

f. Allow equipment to air dry in an area free from potential airborne contaminants.

Storage of clean equipment

- Place dry, clean equipment inside doubled plastic bags. For small equipment, parts, and tubing, use sealable plastic bags.
- Place the churn splitter and funnel into doubled plastic bags and then place churn splitter inside of the churn carrier.

Clean equipment at the sampling site while equipment is still wet and before leaving for the next site.

CLEANING OF EQUIPMENT USED TO SAMPLE FOR ORGANIC COMPOUNDS 3.2.2

Nearly identical procedures are used in the office laboratory and at the field site to clean equipment used to sample for organic compounds. The office laboratory provides an environment in which equipment can be cleaned over an extended time using greater volumes of cleaning and rinsing solutions than in the field. The five-step cleaning procedure summarized in figure 3-4 is described in this section. If inorganic constituents also will be sampled for, check the sequence of cleaning solution to be used as shown in figure 3-1 before proceeding.

- Preclean a separate set of equipment for each site in order to avoid field cleaning of equipment between sampling sites. Always rinse equipment with DIW directly after use, however.
- If individual or dedicated sets of equipment for each field site are not available or cannot be precleaned, field clean equipment before moving to the next sampling site and process additional field blanks for each field trip (Koterba and others, 1995).
- Collect additional field blanks after cleaning equipment that was exposed to high levels of contamination (NFM 4) and before the equipment is reused for environmental sampling.

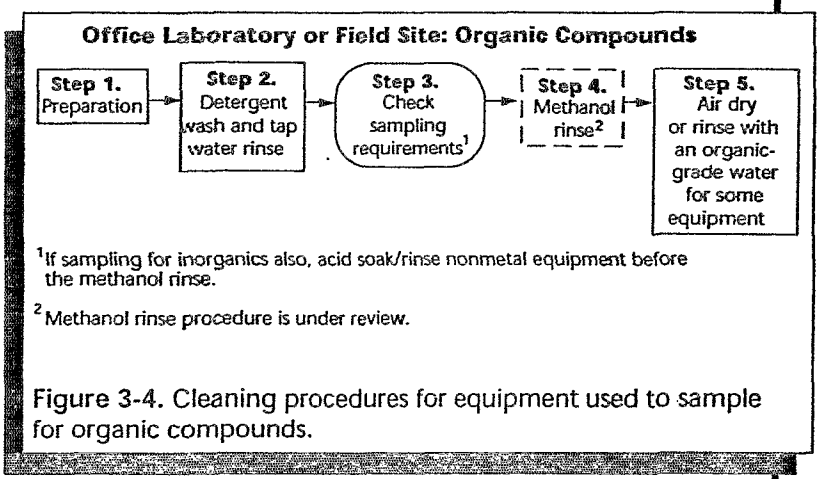


Figure 3-4. Cleaning procedures for equipment used to sample for organic compounds.

Equipment-cleaning procedure for organic compounds

Standard procedures for office-laboratory and field-site cleaning of equipment used to collect and process samples for organic-compound analysis are described below and summarized in figure 3-4. Not all the steps listed apply to all equipment, however. For example,

- **Omit any cleaning procedure for sample bottles for organic compounds.** Bottles for organic analyses arrive from the laboratory capped and ready for use and should not be rinsed by field personnel. Discard bottles if received uncapped.
- **Omit the methanol rinse when cleaning the equipment used to collect and process samples for total, dissolved, and suspended organic carbon (TOC, DOC, SOC).** If equipment (such as a submersible pump) that has been in contact with methanol or other organic solvent must be used for TOC, DOC, or SOC sampling, flush the equipment with copious quantities of sample water before collecting the sample; collection of a blank sample for DOC quality control is recommended.

Be sure to check the specific procedures for selected equipment listed in section 3.3 before proceeding with the office-laboratory and field-site procedures.

Step 1. Preparation (fig. 3-4).

a. Prepare a contaminant-free space for cleaning and drying the cleaning supplies and sample-collection and sample-processing equipment.

   i. Gather the cleaning supplies, the equipment to be cleaned, and clean storage bags and aluminum foil with which to wrap the cleaned equipment. (Check table 3-1 for the cleaning supplies needed.)

   ii. Cover the cleaning area with aluminum foil or fluorocarbon polymer sheeting.

iii. Put on disposable, powderless gloves,[3] a laboratory coat or apron, and safety glasses. **Gloves provide protection from direct contact with solvents only for a limited period of time.**

iv. Prepare the detergent solution, using nonphosphate laboratory-grade detergent. A 0.1- to 0.2-percent (v/v) solution is normally of sufficient strength, unless equipment is very oily or greasy. **Do not use greater than a 0.2-percent solution for field cleaning.**

b. Clean the items used to clean the equipment.

i. Label each washbasin, standpipe, and wash bottle with a black waterproof marker to indicate the solution it will contain.

ii. Follow Steps 2–5, listed below, to clean the washbasins, standpipes, wash bottles, and other items to be used for equipment cleaning.

c. Disassemble sample-collection and sample-processing equipment. Submersible pumps should be disassembled periodically for office cleaning but usually are not disassembled for field cleaning.

[3]Refers to laboratory gloves that are nonpowdered on the inside and intended for disposal after one use. Glove materials must be appropriate for the work to be carried out and the solutions and equipment to be contacted. For example, vinyl gloves are appropriate for most sampling activities but not when working with methanol or other organic solvents. Use solvent-resistant gloves when cleaning with organic solvents. Latex or nitrile disposable, powderless gloves are appropriate when using methanol.

Step 2. Detergent wash and tapwater rinse (fig. 3-4).

a. Place small equipment parts into washbasin labeled for detergent. Fill washbasin with a 0.2-percent solution of nonphosphate, laboratory-grade detergent. (The specific concentration of detergent solution depends on how contaminated the equipment might be and on the hardness of the tapwater.) Change gloves.

- **Office laboratory.** Soak equipment in detergent solution for 10 to 30 minutes.
- **Field site.** Rinse equipment exterior and interior with detergent solution.

b. Scrub the exterior and interior of equipment surfaces to the extent possible, using a firm sponge or soft brush to remove any adhering material such as oil and grease, sediment, algae, or chemical deposits. Pay particular attention to removing material from areas where inorganic or organic materials might be trapped, such as grooves and crevices, O-rings, and nozzles.

c. Place equipment into tapwater washbasin.

d. Rinse equipment thoroughly with tapwater to remove detergent residue. Use an organic-grade water (PBW, VBW, or office-produced) if tapwater is unavailable or is of a quality so poor as to contaminate the equipment. If necessary, use a wash bottle filled with organic-grade water or tapwater to rinse hard-to-reach places. Equipment rinsing is complete if no detergent bubbles appear when rinse water is agitated. Change gloves.

Step 3. Check sampling requirements (fig. 3-4).

a. If samples will be collected for organic analysis only, go to Step 4.

b. If samples will be collected for inorganic analysis in addition to organic analysis, follow the procedure for the acid wash and DIW rinse before proceeding with the methanol rinse (see figs. 3-1 and 3-4).

Step 4. Methanol rinse[4] (fig. 3-4).

a. Change to gloves that are chemically resistant to any solvent being used. Place cleaned equipment into a clean stainless steel or organic-solvent-resistant washbasin. Methanol-rinse area must be outside of the field vehicle and away from the sample-processing site. **Sample-collection, -processing, and -preservation areas must remain free of solvent vapors.**

CAUTION: Use methanol or other organic solvents sparingly and work under a fume hood or in a well-ventilated area, away from where an open flame or sparks can occur. Wear safety gloves, glasses, and apron.

b. Use pesticide-grade methanol (or appropriate organic solvent) dispensed from a methanol fluorocarbon-polymer wash bottle (office laboratory) or pumped through tubing (field site) (see TECHNICAL NOTE below).

i. Rinse equipment exterior and interior with a minimum amount of methanol.

ii. Rinse interior of pump tubing with methanol.

- Do not rinse exterior of pump tubing with methanol.
- **Do not rinse pump tubing with methanol or any organic solvent if TOC, DOC, or SOC samples will be withdrawn through that tubing.**

[4]Current (1998) cleaning protocol dictates the use of methanol to remove contaminants from equipment to be used to collect samples for analysis of organic compounds. This protocol is under review.

iii. Place equipment components and tubing on a clean aluminum foil surface.

iv. Pour or discharge used methanol (or other organic solvent) into an appropriate waste container for flammable liquids (Water Resources Division Memorandum 94.007). Change gloves. Dispose of gloves used for methanol rinse appropriately.

TECHNICAL NOTE: Rinse with dichloromethane or hexane if the methanol rinse is not sufficient to clean equipment contaminated with excessive concentrations of hydrophobic organic compounds. If rinsing with dichloromethane or hexane, use pesticide-grade solutions, wear nitrile gloves, and use only on dry equipment (dichloromethane and hexane are not soluble in water). Do not rinse equipment with any organic solvent if equipment will be used for TOC, DOC, or SOC samples.

Step 5. Air dry equipment or rinse with organic-grade water (fig. 3-4).

a. Allow methanol-rinsed equipment to air dry in an area free from dust and potential airborne contaminants (place an aluminum foil tent loosely over the drying equipment).

b. If it is not practical for the methanol to evaporate from the interior of equipment components or sample tubing, either

- dry by blowing clean, filtered, inert gas through equipment; or
- rinse methanol from equipment with pesticide-grade or volatile-grade blank water, dispensed from a wash bottle or pumped with a valveless fluid metering pump.

Storage of clean equipment

Cover all equipment orifices with aluminum foil or fluorocarbon polymer bags, then place equipment into sealable storage bags. Isolate equipment used to collect trace-element samples from aluminum foil.

SPECIFIC PROCEDURES FOR CLEANING SELECTED TYPES OF EQUIPMENT 3.3

By A.J. Horowitz, M.W. Sandstrom, and F.D. Wilde

The equipment-cleaning steps described in sections 3.2.1 and 3.2.2 apply to most, but not all, equipment. This section describes the cleaning procedures needed for specific equipment for which the general protocols are modified or do not apply, or for which more detailed instructions might be useful. Wear appropriate disposable, powderless gloves throughout each cleaning procedure, changing gloves with each change in cleaning solution and as described in section 3.2.

INORGANIC-SAMPLE BOTTLE CLEANING PROCEDURES 3.3.1

Bottles for samples to be analyzed for inorganic constituents include translucent colorless polyethylene, opaque brown polyethylene, and transparent glass bottles.Translucent polyethylene bottles that were acid rinsed at the laboratory should arrive capped with colorless, translucent plastic caps. Glass bottles for samples for mercury analysis also are acid rinsed and should arrive capped.

- **Discard acid-rinsed bottles that are received uncapped.**
- A cleaning procedure is required for bottles that will contain samples to be analyzed for trace elements and is recommended for bottles that will contain samples to be analyzed for major ions and nutrients.

Before leaving for the field, clean polyethylene and glass sample bottles, including acid-rinsed bottles, as described in the steps that follow:

1. Put on powderless, vinyl gloves.
2. Fill each bottle about one-quarter full of DIW and cap.
3. Shake vigorously and decant DIW.

4. Repeat the DIW rinse (Steps 2 and 3 above) two more times.
5. Following the last rinse, fill each bottle half full with DIW and cap the bottle.
6. Rinse exterior of bottle with DIW and dry with lint-free laboratory tissue.
7. Store bottles in doubled plastic bags.

3.3.2 CHURN SPLITTER CLEANING PROCEDURES

Plastic churn splitters are used primarily for samples to be analyzed for inorganic constituents (NFM 2). Avoid the need to field-clean the churn splitter by using a separate, precleaned churn splitter at each field site to be sampled, if possible.

When using the detergent wash/tapwater rinse for the churn splitter—Office-laboratory procedure (fig. 3-2, Step 2):

1. Fill churn splitter through the funnel with detergent solution.
2. Soak for 30 minutes.
3. Scrub interior and exterior surfaces with a soft brush, taking care not to abrade the surface.
4. Pay particular attention to cleaning the paddle and the area around the spigot.
5. Make sure spigot and funnel are free of sediment, including fine particulates (clay), organic matter, and stains.
6. Drain some of the cleaning solution through the spigot before discarding the remaining solution.
7. Fill churn through the funnel splitter about one-third full with tapwater; swirl and shake churn vigorously to remove detergent residues. Allow tapwater to pass through the spigot.
8. Repeat rinse procedure until no bubbles remain in rinse water after the water is agitated.

When using the acid rinse for the churn splitter—Office-laboratory or field-site procedures (figs. 3-2 and 3-3, Step 4):

1. Do not allow acid solution to contact the outside of churn splitter, or the churn spigot.

2. Do not pass acid solution through the spigot.

3. Decant acid solution by pouring out of the top of the churn into the neutralization container.

When using the DIW rinse for the churn splitter—Office-laboratory or field-site procedures (figs. 3-2 and 3-3, Step 5):

1. Fill the churn splitter through the funnel with DIW to about one-third full.

2. Swirl the DIW vigorously and pour it out of the top of the churn into the neutralization container.

3. Repeat the fill-and-swirl procedures of 1 and 2 above at least twice, checking the pH of the DIW after each swirl with narrow-range pH indicator strips.

4. **Pass a portion of the DIW through the spigot only after the DIW pH equals or is greater than either 6.0 or the pH of the DIW before acidification. Pour the rest of the DIW into the neutralization container.**

For storage of a cleaned churn splitter—Office-laboratory or field-site procedures:

1. Package a clean, dry churn splitter in two new plastic bags and loosely tie or secure with a nonmetal clip. If a churn splitter must be packaged while wet, use within 1 to 3 days and (or) keep chilled to prevent bacterial growth.

2. Place entire package into the churn carrier.

3.3.3 CONE SPLITTER CLEANING PROCEDURES

The fluorocarbon-polymer cone splitter (NFM 2) is appropriate for splitting samples for inorganic or organic analyses. When cleaning the cone splitter (Office of Water Quality Technical Memorandum 97.03), pay particular attention to removing foreign material from threaded and hard-to-access parts. Field cleaning can be minimized by having separate, precleaned cone splitters available for each site and by keeping a supply of clean tubes to replace the used tubes for each site to be sampled.

When inorganic constituents will be analyzed in samples processed through the cone splitter:

Office laboratory. Follow the steps as described for figure 3-2.

Field site. Referring to figure 3-3:

1. Prepare the field site as described in section 3.2.1. Put on disposable, powderless gloves.
2. Rinse the splitter thoroughly with deionized water.
3. Inspect the cone splitter. If it looks dirty, is suspected of being contaminated, or was allowed to dry between field sites without a thorough DIW rinse, or if the splitter will be used for sampling both inorganic and organic analytes, use the detergent-wash option. Change gloves.
4. Acid rinse by passing 1 L of 5-percent HCl solution through the cone splitter. Collect used acid solution into a neutralization container. Change gloves.
5. Rinse the cone splitter with at least 3 L of deionized water. Collect the rinse solution into a neutralization container. Change gloves.
6. Allow the cone splitter to dry and then store in a clean plastic bag. Seal the bag and store in a second plastic bag or plastic storage container for transport to the next site. A cone splitter that is packaged into bags while wet should be used within 1 to 3 days and (or) kept chilled to prevent bacterial growth.

When organic compounds will be analyzed in samples processed through the cone splitter (fig. 3-4):

Office Laboratory. Follow the steps described for figure 3-4.

Field Site.

1. Prepare site as described in section 3.2.2. Put on appropriate disposable, powderless gloves; if a solvent will be used, select gloves that will withstand contact with the solvent.
2. Detergent wash and rinse equipment as described for figure 3-4.
3. Check equipment and sampling requirements. If splitter will also be used for inorganics sampling, follow acid-rinse directions before rinsing with methanol or other organic solvent.
4. Proceed with the methanol (or other organic solvent) rinse, if required (section 3.2.2).

   - **Do not use any organic solvent if the cone splitter will contact samples for analysis of TOC, DOC, or SOC.**
   - If samples processed through a splitter will be analyzed for TOC, DOC, or SOC, rerinse the splitter thoroughly to completely remove residues from the detergent wash. Use PBW, VBW, or other organic-grade water for the final rinse if complete methanol evaporation is impractical. If the cone splitter will not be used to process samples for inorganic constituents at the next site, wrap nozzle and other orifices in aluminum foil.

For storage of a cleaned cone splitter:

1. Allow the cone splitter to air dry.
2. Place the cone splitter into a clean plastic bag and seal.
3. Store in a second plastic bag or plastic storage container for transport to the next site.

If a cone splitter must be packaged while wet, use within 1 to 3 days and (or) keep chilled to prevent bacterial growth.

3.3.4 FILTRATION EQUIPMENT CLEANING PROCEDURES

Filtration equipment includes disposable capsule filters and various plate-filter and pressure-filter assemblies. Cleaning procedures for these types of equipment are described below.

3.3.4.A Disposable Capsule Filter Cleaning Procedure

The disposable capsule filter has a one-time use for processing samples to be analyzed for inorganic constituents but must be cleaned before use. The filter can be prerinsed in the office laboratory instead of at the field site as long as it is kept chilled and used in less than 1 day. After filtering the sample, clean or replace the sample-delivery tubing and discard the capsule filter. The cleaning procedure described below comprises sufficient cleaning of the filter for analysis of inorganic constituents at the parts-per-billion (ppb) concentration level (Horowitz and others, 1994).

To clean the disposable capsule filter, pump 1 L of DIW to the filter through precleaned tubing (section 3.3.5) as follows (refer to NFM 5.2.1.A for additional instructions):

1. Use Clean Hands/Dirty Hands techniques described in NFM 4. Remember: the Dirty Hands team member performs operations that are outside of the processing chamber and the Clean Hands team member performs operations that are inside the chamber. Put on disposable, powderless gloves.

2. In a processing chamber, remove the capsule filter from the protective bags. Attach pump tubing to the inlet connector of the capsule filter, keeping the tubing as short as possible. **Make sure the direction of flow through the capsule filter matches the direction-of-flow arrow on the side of the filter.**

3. Pump 1 L of DIW through the capsule filter; discharge waste rinse water through a sink funnel or to a toss bottle.
   - Operate the pump at a low speed.
   - Hold the capsule filter so the arrow is pointing up at an acute angle from the horizontal plane. (This expels trapped air from the capsule; do not allow water to spray onto chamber walls.)
4. Remove tubing from the DIW reservoir and continue to operate the pump in the forward, mid-range speed position to drain as much of the DIW that remains in the capsule filter as possible. While the pump is operating, shake the capsule filter to help remove any entrained DIW.
5. Detach the capsule filter from the peristaltic pump tubing, put into a clean, sealable plastic bag, and store chilled until ready for use at the next site.

Plate-Filter Assembly Cleaning Procedure 3.3.4.B

To clean filtration equipment used for samples to be analyzed for inorganic or organic analytes, consult sections 3.2.1 and 3.2.2, respectively. Use Clean Hands/Dirty Hands techniques, as appropriate (NFM 4).

- **Preclean in the office laboratory** one plate-filter assembly per site to be sampled, if possible, in order to save the time that would be needed to clean the plate-filter assembly during the field effort.
- **During the detergent wash and (or) DIW rinse,** pay particular attention to grooves and crevices, O-rings, and support structures for the filter, where sediment or organic matter might be lodged. Detergent wash and DIW rinse the pressure valve.
- **Remove and discard the used filter at the field site;** rinse the filter assembly immediately with DIW while still wet from filtering the sample, even if a clean filter assembly is available for the next site.

When field cleaning the plastic plate-filter assembly:

1. Disassemble the plate-filter assembly inside the processing chamber while it is still wet from the sample water and while wearing disposable, powderless gloves.

   a. Remove the used filter media carefully to avoid spilling any of the filter cake.

   b. Place the filter media into a sealable plastic bag. Seal and pass the bag out of the chamber. Change gloves.

2. DIW rinse all components of the plate-filter assembly, including the exterior and interior of the tubing and the pressure valve, dispensing the DIW from a wash bottle. Pay particular attention to grooves and crevices, O-rings, and support structures for the membrane filter, where inorganic or organic materials might be lodged. Change gloves.

3. Inspect the plastic plate-filter assembly. Use the detergent-wash option described in figure 3-3 (Step 2) if the filter assembly looks dirty, is suspected of being contaminated, or was allowed to dry after use without first rinsing thoroughly with deionized water.

4. Reassemble the plate-filter assembly, reattaching the piece of tubing to the outlet of the filter assembly and placing the discharge end of the tube through the drain or disposal funnel in the bottom of the processing chamber to the acid-neutralization container. Reconnect the filter assembly to the peristaltic pump with the sample tubing. Change gloves.

5. To acid rinse the plate-filter assembly, pump 1 L of 5-percent HCl solution (or 10-percent HNO_3 solution) through the plate-filter assembly. Check that the acid solution is being discharged into the acid-neutralization container. Alternately squeeze and release the tubing at the outlet to force the acid solution to cover and rinse all interior surfaces of the filtration assembly. (Be careful not to force tubing from the outlet by squeezing tubing for too long.)

6. To DIW rinse the plate-filter assembly, pump 2 L of deionized water through the assembly, using the same squeeze-and-release method described above in 5 for the acid rinse. Ensure that all the rinse water is being discharged to the acid-neutralization container. After confirming that the pH of the acid rinse solution is greater than 6.0 or the original pH of the DIW, appropriately discard solutions from the neutralization container.

7. For storage, place the cleaned plate-filter assembly and tubing into clean double bags for temporary storage until use at the next site. If wet when bagged, store for no longer than 24 hours and (or) chill to prevent bacterial growth. The filter assembly must be dry if stored for more than 24 hours.

Always remove the used filter media from the plate-filter assembly before cleaning and storage.

When field cleaning the aluminum plate-filter assembly, use the general cleaning instructions in section 3.2.2 for figure 3-4, as follows:

1. Inspect the aluminum (or stainless steel) plate-filter assembly for damage or excessive contamination and replace if necessary.

2. Wearing disposable, powderless gloves, prepare the area to be used for cleaning the plate-filter assembly by lining the table or counter surface with aluminum foil.

3. Disassemble the filter assembly and remove the used glass-fiber filter media carefully to avoid spilling any of the filter cake. Place used filter media into a sealable plastic bag, seal the bag, and put aside for disposal. Place components of the plate-filter assembly and tubing into a washbasin for detergent. Change gloves.

4. Detergent wash by using a 0.1- to 0.2-percent nonphosphate-detergent solution. Scrub each component of the filter assembly with a soft brush to remove any adhering material such as oil and grease, sediment, algae, or chemical deposits. Pay particular attention to grooves and crevices, O-rings, and support structures for the glass-fiber filter, where inorganic or organic materials might be lodged. Pump detergent solution through tubing. Place components of the plate-filter assembly onto a clean, aluminum-foil-covered surface.

5. Discard detergent solution from basin, rinse basin with tapwater, and place components of the plate-filter assembly into the basin. Change gloves.

6. Rinse each component thoroughly to remove detergent residue, paying particular attention to grooves and crevices. Use a wash bottle filled with DIW or tapwater to rinse hard-to-reach places. Place rinsed components onto a dry section of clean aluminum foil or basin. Change gloves. If the assembly will be rinsed with

methanol or other organic solvent, change to disposable, solvent-resistant gloves, and place components of the filter assembly into a clean, solvent-resistant washbasin.

7. Rinse plate-filter assembly components with pesticide-grade methanol or an equivalent grade for other organic solvents. Do not methanol rinse any tubing or filtration assembly to be used for collecting or processing samples for TOC, DOC, or SOC analysis. The instructions for the methanol rinse apply also for use of any other organic solvent. **Rinse the equipment with methanol while outside of the field vehicle and downwind of sampling activity.**

   a. Dispense methanol from a fluorocarbon-polymer wash bottle. Rinse all sample-contacting surfaces of filter-assembly components and tubing over a solvent-resistant basin or waste container. **Methanol-laced rinse water must be collected into an appropriate waste container designed for flammable liquids.**

   b. Place methanol-rinsed equipment components onto a clean aluminum foil surface to air dry. (Cover equipment components loosely with an aluminum foil tent, if concerned about airborne contaminants.)

8. Reassemble the plate-filter assembly. Wrap nozzles with aluminum foil and seal filter assembly in plastic bags. Double bag for transport or for long-term storage.

3.3.4.C Pressure-Filter Assembly Cleaning Procedure

The cleaning procedures described in section 3.2.2 for figure 3-4 do not apply to the filtration assembly used for samples to be analyzed for DOC and SOC. The filtration assembly for processing organic-carbon samples is a gas-pressurized apparatus constructed of either stainless steel or fluorocarbon-polymer material.

- **Do not bring the pressure-filter assembly in contact with methanol or other organic solvent or organic-solvent vapors.**
- In general, office-produced organic-grade water that is prepared by being passed through appropriate columns to remove organic compounds is of adequate purity for cleaning this equipment. PBW or VBW also can be used. Office-produced organic-grade water, however, must not be substituted for blank samples.

▶ **Do not clean the pressure-filter assembly with detergent.** Exception: see Step 3 below.

When using office-laboratory or field-site cleaning procedures for cleaning the pressure-filter assembly:

1. Wearing disposable, powderless gloves, disassemble the pressure-filter assembly before it dries and place components into a clean washbasin. Change gloves.

2. Using office-produced organic-grade water, thoroughly rinse the pressure-filter assembly and place it into a washbasin or onto a clean surface. Generally, these steps are sufficient to field clean the pressure-filter assembly.

   - If necessary, use a soft-bristled toothbrush to remove sediment, chemical deposits, and other foreign material from threaded components, gaskets, O-rings, support screens, grooves, and nozzles. Take care not to scratch or mar inner surfaces when scrubbing.

   - Rinse the pressure-filter assembly thoroughly with office-produced organic-grade water or PBW or VBW.

3. If the pressure-filter assembly is very dirty or contaminated, clean as follows:

   a. Disassemble and soak assembly for at least 1 hour in a 0.1-percent solution of nonphosphate laboratory-grade detergent.

   b. Scrub with a soft-bristled toothbrush, as described above in 2.

   c. Rinse repeatedly with office-produced organic-grade water, being sure to remove all traces of detergent.

4. Place all components of the pressure-filter assembly onto aluminum foil and allow to air dry thoroughly under a protective aluminum foil tent.

5. Reassemble the pressure-filter assembly, wrap nozzles in aluminum foil, and seal in a storage bag.

Do not use methanol or other organic solvents on the equipment used to filter samples for organic-carbon analyses.

3.3.5 SAMPLE TUBING CLEANING PROCEDURES

Cleaning procedures are described below for the tubing and nozzles used with peristaltic and valveless metering pumps. Cleaning procedures for submersible pump tubing are described in section 3.3.9.B. Wear appropriate, disposable, powderless gloves throughout the cleaning process, changing gloves with each change in cleaning solution as indicated throughout section 3.2.

- Preclean the number of tubing sections needed at each site in the office laboratory rather than recleaning tubing in the field, in order to save time during field work. Place into doubled plastic bags and store tubing dry or store wet tubing chilled to prevent bacterial growth. If bacterial growth is present, reclean tubing before use.
- Use disposable tubing if possible, especially at contaminated sites, to avoid the cleaning process and prevent the possibility of cross contamination.

When using office-laboratory or field-site procedures for cleaning plastic (including fluorocarbon-polymer) sample tubing used for samples to be analyzed for inorganic constituents, follow the general sequence of procedures described for figures 3-2 or 3-3, and those described for filtration assemblies (section 3.3.4).

To summarize the key steps for figures 3-2 or 3-3:

1. Pump 1 L of 5-percent HCl solution through the tubing, discharging the used acid solution into a neutralization container. Pinch and release tubing near tubing outlet while pumping the acid through to ensure that all interior surfaces are acid rinsed.
2. Pump 2 L of DIW through tubing, using the same pinch-and-release method. Discharge used DIW to an acid-neutralization container, and check that the rinse-water pH is greater than 6.0 or the original DIW pH.
3. Discard neutralized solutions appropriately.
4. Clean stainless steel connections or metal tubing using detergent-wash and tapwater/DIW rinse procedures.

When using office-laboratory or field-site procedures for cleaning tubing for organic-compound samples:

Follow the general sequence of procedures described for figures 3-1 and 3-4. Proceed with the methanol rinse after the detergent wash and tapwater rinse. If samples also will be collected for inorganic-constituent analysis, however, acid rinse nonmetallic tubing and components after the detergent wash/tapwater rinse and before continuing to the methanol rinse. When cleaning sample tubing:

1. Pump 1 L of nonphosphate, laboratory-grade detergent solution through tubing, followed by sufficient tapwater or DIW to remove detergent residue. Pinch and release tubing near tubing outlet while pumping the solution to ensure that all interior surfaces are cleaned.
2. Place discharge end of tubing from peristaltic or valveless metering pump over methanol waste container.
   - Pass one tubing volume of methanol through the same pump system used for filtration, using the same pinch-and-release method.
   - Short sections of tubing can be held over the waste container while dispensing the methanol from a fluorocarbon-polymer wash bottle instead of pumping the methanol through the tubing.
   - **Do not methanol rinse tubing to be used for samples for TOC, DOC, or SOC analysis.**
3. Store tubing in doubled plastic bags.

CAUTION: Do not use methanol around equipment that can create electrical sparks (see section 3.3.9.B).

3.3.6 PROCESSING AND PRESERVATION CHAMBERS AND FLOWTHROUGH CHAMBER CLEANING PROCEDURES

Processing and preservation chambers used to protect samples from atmospheric contamination generally are portable and are assembled at the field site. Large, clear plastic bags usually are clipped to the inside of the frame rather than stretched over the frame. Plastic clips are used to hold the cover tightly in place. When the bag is clipped to the inside, it is not necessary to field clean the chamber frame.

The flowthrough chamber, used when monitoring ground-water field measurements, is connected inline to the pump sampler. The flowthrough chamber should be kept free of sediment and dirt or deposits on the chamber walls. Air dry and store the chambers in sealable plastic bags.

When cleaning the processing and preservation chambers:

Office laboratory. Clean the frame of portable chambers in the office with detergent solution, then rinse thoroughly with tapwater and dry and store in plastic bags.

Field site. Frames require regular cleaning after each use at a site if chamber covers are stretched over the outside of the frame rather than clipped to the frame.

1. Discard the used bag.
2. Wipe the chamber frame with DIW.
3. Replace chamber cover only when the next samples are ready to be processed.
4. If the processing chamber is a fixed installation, clean out any spilled sample water, solid materials, or wash solutions, and swab down the inside using deionized water and lint-free laboratory tissue.
5. Use detergent solution followed by a thorough tapwater or DIW rinse if a spill has contaminated the chamber.
6. Store chamber frames in plastic bags.

When cleaning the flowthrough chamber:

1. Clean the flowthrough chamber in the office laboratory with detergent solution and rinse thoroughly with tapwater, followed by DIW. **Do not use acid solution or methanol.**
2. If the flowthrough chamber needs to be field cleaned, remove measurement sensors and clean with a dilute detergent solution; rinse thoroughly with tapwater followed by DIW.

RADON SAMPLER CLEANING PROCEDURE 3.3.7

Soak radon samplers in a detergent solution for 10 minutes and rinse thoroughly with tapwater to remove detergent residue; follow with three to five rinses with DIW. **Do not use methanol.** Air dry the radon sampler and store in doubled plastic bags.

3.3.8 SURFACE-WATER SAMPLER CLEANING PROCEDURES

Disassemble surface-water samplers for cleaning and follow the sequence of procedures described in section 3.2 and figures 3-2, 3-3, or 3-4, as appropriate.

When using office-laboratory procedures for cleaning surface-water samplers:

1. Periodically disassemble samplers for office-laboratory cleaning. **Discard the bag sampler bag after one use**—do not attempt to scrub or detergent wash the used bag. Prepare cleaning solutions, cleaning equipment, and cleaning area as described in section 3.2.

2. Soak components in detergent solution for 30 minutes. Put on appropriate disposable, powderless gloves. Scrub components with a soft brush or sponge and rinse thoroughly (section 3.2.1 or 3.2.2). Change gloves.

3. Check the sequence of cleaning procedures shown in figure 3-1.

   a. If the sampler is used for sampling inorganic constituents, soak each nonmetallic component in a 5-percent trace-metal-grade HCl solution for 30 minutes, followed by copious rinsing with DIW (section 3.2.1). **Acid rinse only nonmetal parts.** Change gloves.

      - Acid must not contact the metal collar on the DH-81 sampler.
      - Make sure that the nozzle is unscrewed from the cap.

   b. If the sampler is used for collecting organic-compound samples, rinse each component with pesticide-grade methanol dispensed from a fluorocarbon-polymer wash bottle and allow to air dry (section 3.2.2). **Do not methanol rinse tubing or components that will contact TOC, DOC, or SOC samples.** Change gloves.

4. If collecting an equipment blank (section 3.4), change gloves and rinse each component with the appropriate blank water before collecting the blank sample.

5. Reassemble the sampler. If the sampler is dedicated to sampling for organic compounds, double wrap the sampler nozzle in aluminum foil. Place the sampler into double plastic bags and seal for storage and transport.

When using field-site procedures for cleaning surface-water samplers:

1. Unwrap precleaned washbasins (one for each cleaning solution to be used).

2. Disassemble the used sampler into its component parts (bottle, cap, nozzle) so that all of the pieces can be thoroughly wetted with the various rinses. **Discard the previously used bag-sampler bag** (do not attempt to clean it for reuse).

3. Wearing appropriate disposable gloves, thoroughly rinse the sampler components with DIW. Use a stream of DIW from the wash bottle, if required.

4. Check whether target analytes are inorganic constituents, organic compounds, or both. Review figure 3-1 for the appropriate cleaning sequence.

   a. If a sampler will be used for collecting samples for analysis of inorganic constituents only, change gloves and

      i. Thoroughly rinse the sampler components with tapwater or DIW.

      ii. Acid rinse nonmetallic components over a container using a stream of dilute acid solution from the appropriate wash bottle, if required.

      iii. Thoroughly rerinse the sampler components with DIW over the same washbasin, if possible (see section 3.2.1). Change gloves.

      iv. Place each component on a clean, plastic surface. Pour used acid solution and DIW rinse water into neutralization container.

      v. Check the pH of the solution in the neutralization container. Discard when solution pH is greater than 6.0 or the original DIW pH. Change gloves.

   b. If a sampler will be used for collecting samples for analysis of organic compounds only, change gloves and

      i. Detergent wash, then rinse sampler components thoroughly with tapwater or DIW until agitated rinse water produces no more suds. Change to solvent-resistant gloves.

      ii. Rinse sampler components with pesticide-grade methanol (section 3.2.2), collecting the used methanol into an appropriate container for safe storage until appropriate disposal is arranged.

iii. Place each component on a clean, aluminum-foil-covered surface to air dry and cover loosely with an aluminum foil tent, if airborne contaminants are a concern. Change gloves.

c. If sampler will be used for collecting samples for both organic and inorganic analyses, change gloves and

i. Proceed with a detergent wash and thorough tapwater and (or) DIW rinse.

ii. Acid rinse and DIW rinse nonmetallic components, as described above, discarding used solutions appropriately. Change to solvent-resistant gloves.

iii. Rinse with methanol, if needed, as described above.

iv. Place cleaned items on a clean plastic surface to air dry.

5. Reassemble sampler. If the sampler is dedicated to sampling for organic compounds, double-wrap sampler nozzle in aluminum foil. Place sampler into doubled plastic bags for storage and transport.

Do not use methanol or other organic solvents on equipment used to collect organic carbon samples

GROUND-WATER SAMPLER CLEANING PROCEDURES 3.3.9

Ground water is sampled with nonpumping samplers (such as bailers, syringe samplers, and the Kemmerer sampler) and with pumping samplers (such as peristaltic and valveless metering pumps and submersible pumps). Office-laboratory cleaning procedures are used before a sampler is used for the first time, after the sampler has been in long-term storage, and whenever the sampler has become excessively contaminated. Field-site cleaning procedures are used after sampling at a field site and before proceeding to the next sampling site. Caveats and modifications that apply to the general office-laboratory and field-site cleaning procedures (section 3.2) are described in this section. The cleaning procedures used should be documented on field forms.

The rinse with methanol, or other organic solvent, is under review and appropriate only for samplers being used to collect samples for organic-compound analysis. **Solvents are never used to clean equipment when sampling for TOC, DOC, or SOC.** Dispose of used methanol and all other cleaning solutions appropriately.

TECHNICAL NOTE: Sampler components made of fluorocarbon-polymer plastic generally can withstand a solvent rinse with methanol. Check with the manufacturer before using an organic solvent on pump components constructed of any other plastic material.

3.3.9.A Cleaning of Bailers and Other Nonpumping Samplers

Office-laboratory procedure. Clean nonpumping samplers in a designated area of the office laboratory. Follow the procedures described for figures 3-2 and 3-4, as appropriate for equipment used to sample for inorganic constituents or organic compounds, respectively.

Field-site procedure. Follow the field-site cleaning procedures described for figures 3-3 and 3-4, as appropriate for equipment used to sample for inorganic constituents or organic compounds, respectively.

- Rinse the outside of the sampler with DIW directly after use.
- After filling the sampler with each cleaning solution, shake the sampler vigorously and drain solution through the bottom-emptying device, spigot, or nozzle of the sampler.
- If the sampler looks very dirty or is contaminated, disassemble and clean sampler components using the office-laboratory procedure.

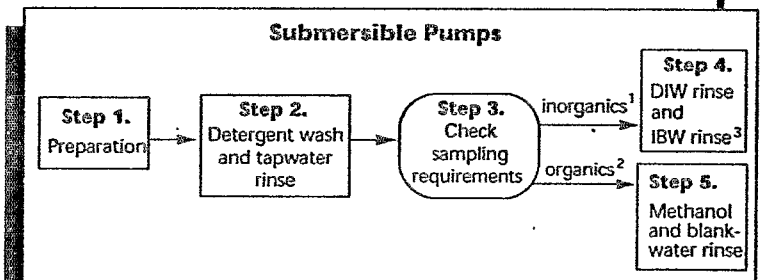
3.3.9.B Cleaning of Submersible Pumps and Submersible-Pump Tubing

The general sequence shown in figure 3-5 is appropriate for cleaning most submersible pumps. The field-site cleaning procedure (described below after the office-laboratory procedure) is sufficient for routine cleaning of the pump in most cases. Collection of blank samples for quality control must be included as a standard protocol for every study in order to document and ensure the efficacy of the cleaning procedure for the field conditions encountered.

- Fluorocarbon-polymer tubing used to collect water containing large concentrations of volatile organic compounds (VOCs) can be difficult to clean adequately.

- Collect additional blanks if VOC concentrations in last sample collected through the tubing were greater than 500 µg/L.
- Pump tubing should be replaced rather than cleaned if VOC concentrations in last sample exceeded about 700 µg/L.

- Most submersible pumps have a stainless steel casing and other metal parts and should not be acid rinsed.
  - To clean pumps that are excessively contaminated, a dilute acid rinse followed by copious water rinsing can be used occasionally without damaging the pump.
  - Repeated rinsing with dilute acid solution can pit or corrode the pump's stainless steel surface. If the surface appears dulled, the pump must not be used for collecting trace-metal samples.
- Lubrication water inside water-lubricated pumps (for example, the Grundfos RediFlo2™) can become contaminated and cause contamination of subsequent samples. Replace the lubrication water with VBW each time after sampling and when cleaning the pump. Follow manufacturer's instructions.



[1] Samples collected for inorganic-constituent analysis. If samples are also to be collected for organic-compound analysis, rinse with deionized water (DIW) (Step 4), then methanol (Step 5).

[2] Samples collected for organic-compound analysis. Methanol rinse procedure is under review. When sampling for total, dissolved, or suspended organic carbon, do not methanol rinse; complete cleaning by rinsing with organic-grade water.

[3] A rinse with inorganic blank water (IBW) is needed only if collecting a blank sample for inorganics analysis directly after cleaning the pump.

Figure 3-5. Cleaning procedures for submersible pumps.

Office-laboratory pump-cleaning procedure:

Use office-laboratory procedures about once a year and more frequently if results of the pump blank or other information indicate that the pump is contaminated.

Step 1. Preparation.

a. Wearing appropriate gloves, prepare several gallons of a laboratory-grade nonphosphate detergent solution (about 0.1 or 0.2 percent, v/v; use up to 2-percent solution for excessively contaminated pump systems).

b. Preclean washbasins and standpipes (section 3.2).

c. Place pump into sink or waste basin and scrub exterior surfaces with soft brush and detergent solution; rinse thoroughly with tapwater.

d. Disassemble the pump and place components into a detergent-solution washbasin.

Step 2. Detergent wash and tapwater rinse pump components and tubing.

a. Soak pump components in the detergent solution for 30 minutes.

b. Scrub pump components with soft sponge or brush.

c. Rinse thoroughly with tapwater.

d. Raise discharge end of tubing above the rest of the tubing. Using a peristaltic or valveless fluid metering pump, fill the pump tubing with fresh detergent solution until solution rises to the end of the tubing. Plug the tubing end(s).

e. After 30 minutes remove plug from discharge end of tubing and flush detergent solution from tubing by pumping copious amounts of tapwater through the tubing. Change gloves.

Step 3. Check sampling requirements.

- If pump will be used for collecting samples for inorganic-constituent analysis, reassemble the pump and go to Step 4.
- Complete Step 4 if pump will be used for collecting samples for analysis of both inorganic and organic analytes before proceeding to Step 5.
- If the pump will be used for collecting samples for organic-compound analyses only, go to Step 5.

Step 4. DIW rinse.

a. Place pump components into DIW washbasin and dispense DIW from a wash bottle to thoroughly rinse all pump components.

b. Using a peristaltic pump and appropriate clean tubing, pump DIW through the sample tubing to rinse.

c. Reassemble pump and connect pump tubing. Change gloves.

d. If collecting equipment blanks to verify that the pump has been adequately cleaned (section 3.4):

   i. Rinse a clean standpipe dedicated to blank water with blank water.

   ii. Insert pump into blank-water standpipe only after pump exterior has been rinsed with blank water or air dried after the methanol rinse.

   iii. Pour IBW into the standpipe and pump at least one tubing volume to waste before collecting the blank sample.

Step 5. Rinse with blank water followed by a methanol rinse.

a **Change to latex or nitrile gloves.** Put pump components into solvent-resistant washbasin.

b. Working under a fume hood, dispense methanol (or appropriate solvent) from a fluorocarbon-polymer wash bottle to rinse each pump component and the exterior pump casing. Collect the used solvent into a nonflammable container for storage until disposal.

- **Do not reuse methanol or other solvents.**
- **Work under a fume hood, if possible, or in a well-ventilated area outside of the office laboratory, as methanol fumes can contaminate other equipment.**

c. Place methanol-rinsed components on a clean, aluminum foil surface and allow the pump components and casing to completely air dry before reassembling the pump (see section 3.2.2).

d. Using a valveless fluid metering pump and fluorocarbon-polymer tubing, pump about 2 L of methanol through sample tubing and to the methanol waste container.

e. Reassemble the pump and connect the pump tubing. Change gloves and dispose of the methanol-contaminated gloves appropriately.

f. Pour an organic-grade water (PBW or VBW) into a clean PBW/VBW standpipe. Insert pump and pass about two tubing volumes of organic-grade blank water (PBW or VBW) through the pump and tubing to waste.

CAUTION: Pumping methanol or other flammable solvents through an electrical pump system could be dangerous in the event of sparks. Methanol emits noxious fumes and is absorbed through the skin. Wear a mask, safety glasses and other protective apparel to protect yourself when working with organic solvents.

Field-site cleaning procedure for submersible pumps and pump tubing:

Step 1. Preparation.

a. Preclean the standpipes (one standpipe for each cleaning solution to be used, as described in 3.2.1). The standpipes need to be of sufficient height to supply necessary head for proper pump operation. Separate standpipes are designated for detergent solution and tapwater rinse, DIW rinse, methanol rinse, and blank water (IBW/PBW/VBW). Double-bag each cleaned standpipe for transport to the field site.

b. Estimate the volumes of cleaning solutions and blank water that will be needed for the field effort (refer to fig. 3-6).

c. Prepare the volumes of cleaning solutions needed for the field effort, using appropriate bottles for short-term storage and transport.

The volume of storage in tubing, V_s, of a set of pump-reel and extension tubing can be estimated[1,2] as follows:

$$V_s = [(L_p \times C_p) + (L_e \times C_e) + V_{sp}] \times C_{sp}$$

where,

V_s is volume of storage in tubing, in gallons
L_p is length of pump-tubing segment being cleaned, in feet
L_e is length of extension tubing, in feet
C_p (or C_e) = 0.023 liter per foot for a 3/8-inch inside-diameter (ID) tubing
or = 0.041 liter per foot for a 1/2-inch ID tubing
V_{sp} is volume of solution needed to fill standpipe to minimum level required to operate pump, in liters[1]
C_{sp} = 0.264 gallon per liter.

Examples

Given:

1. L_p - sample-wetted tubing segment is 100 feet for a pump-reel system that has a 1/2-inch ID tubing;
2. L_e - two, 10-foot, 3/8-inch-ID pieces of extension tubing, one running from pump-reel outlet to sample collection chamber, and another running from chamber back to pump-reel (return-flow tubing to standpipe); and
3. V_{sp} - minimum volume[1] of solution required in standpipe to operate pump is 0.8 liter.

To estimate the volume of detergent solution needed for the detergent wash cycle:

$$V_s = [(100 \times 0.041) + (20 \times 0.023) + 0.8] \times 0.264 = 1.4 \text{ gallons}$$

The volume of office-produced deionized water needed to displace detergent solution and the volume of laboratory-produced organic-grade blank water needed to displace 2 liters of methanol just pumped into a system, ideally, would each be estimated to equal V_s[1,2].

[1]Estimate assumes no mixing of two solutions and ignores potential for detergent to adhere to tubing walls. Outflow from the discharge end of tubing should be checked for sudsing to determine that detergent has been removed.

[2]Estimate assumes no mixing at interface of two solutions and ignores potential for methanol to adhere to tubing walls. It is recommended that an additional 0.1 gallon (~ 0.4 liter) of blank water (pesticide-grade blank water or volatile-grade blank water) be used for each 10 feet of tubing to remove methanol residues from sample-wetted sections of tubing. Thus in the example above, another 1.1 (= (100 + 10) x (0.1/10)) gallons (4.2 liters) of blank water would be pumped from the system. This implies a total of about 2.5 (= 1.4 + 1.1) gallons (9.6 liters) of blank water would be used to remove methanol from the equipment setup.

[3]The minimum volume corresponds to the level of solution in the standpipe, which, if maintained, allows pump to operate without introducing air through the pump intake. Once this level is reached, remove pump, and measure this volume.

Figure 3-6. Estimation of cleaning-solution volumes for standpipe, pump, and pump tubing. [From Koterba and others, 1995, table 24.]

Step 2. Detergent wash and tapwater rinse.

a. Put on disposable, powderless gloves (usually vinyl). Rest pump in a washbasin or pail partially filled with detergent solution and clean exterior of pump and tubing with a soft brush. Rinse thoroughly with tapwater. (DIW can be substituted for tapwater, but is less efficient in detergent removal and requires a greater volume of water than tapwater.)

b. Place pump into standpipe, add detergent solution to level above pump intake, and route intake and discharge end of pump tubing to the standpipe.

c. Begin pumping:

   i. Record the pumping rate.

   ii. Record the time it takes to fill the sample tubing.

   iii. Calculate the time it takes for a segment of solution to complete one cycle (fig. 3-6).

d. Circulate detergent solution for about three cycles through the tubing and back to the standpipe. If possible, pump detergent solution through tubing at alternating high and low speeds, and (or) introduce air segments between aliquots of the detergent solution to increase cleaning efficiency.

e. Remove the discharge end of tubing from the standpipe and pump about two tubing volumes of detergent solution to waste, adding fresh solution to the standpipe as needed. Remove pump from standpipe.

f. Rinse detergent from standpipe with tapwater until sudsing stops.

g. Rinse pump exterior with tapwater. Place rinsed pump into standpipe; add tapwater/DIW to level above pump intake. Begin pumping through sample tubing. Do not recirculate rinse water, but add water as needed to maintain water level above pump intake. Continue for five or more tubing volumes. Direct rinse water to waste, away from the vicinity of the wellhead and sampling area and (or) contain as required for disposal.

h. Collect rinse water into a small bottle and stop the pump. Shake the bottle—if sudsing is observed in the rinse water, continue the rinse procedure until no suds appear in the rinse water. Change gloves.

Step 3. Check sampling requirements.

— If a pump will be used to collect samples for inorganic-constituent analysis, go to Step 4.

— Complete Step 4 if a pump will be used to collect samples for analysis of both inorganic and organic analytes and go to Step 5.

— If a pump will be used to collect samples for organic-compound analysis only, go to Step 5.

Step 4. DIW rinse.

A separate DIW rinse is not required if DIW was substituted for tapwater.

a. Use a clean DIW-dedicated standpipe, not the tapwater standpipe, and rinse with DIW. Rinse pump exterior with DIW to remove any detergent residue. Place pump into the DIW standpipe and add DIW to level above pump intake. Change gloves.

b. Start pumping DIW. Rinse DIW through sample tubing without recirculating, using about 3 tubing volumes of DIW. Keep the DIW level above pump intake.

c. Collect DIW rinse water in a clean bottle, shake, and check for suds. Continue to DIW rinse until rinse water is free of suds.

d. If collecting field blanks to verify that the pump has been adequately cleaned (section 3.4):

   i. Change gloves. Rinse clean blank-water standpipe with IBW. Rinse pump exterior with blank water.

   ii. Place pump into the standpipe and add IBW to cover the pump intake.

   iii. Turn on pump and displace any water residing in the pump and tubing. Continue pumping IBW for one tubing volume before collecting the blank sample.

Step 5. Methanol rinse.

Make certain that the pump or other nearby electrically powered equipment is grounded, the power cord is intact, and potential sources of sparks do not exist before rinsing pump with methanol.

TECHNICAL NOTES:

- Inspect the integrity of the seals and O-rings on the pump-motor/pump-body housing. Water inside the motor housing may indicate that methanol vapors could enter the motor. Direct-current motors inherently spark because of the commutator ring. AC motors might spark if the insulation is frayed or burnt on the motor windings or any associated wiring.

- If flammable liquids are required for cleaning electrical pump systems, use extreme caution. Vapors from solvents such as methanol can ignite if a disruption in the motor lead-insulation system occurs in the vapor-enriched zone. (Ignition from a spark from an AC induction-type motor in good operating condition is not a concern if rated as using the National Electrical Code (NEC) at Class 1, Group D.[5])

a. Change to latex or nitrile gloves. Wear safety glasses and apron. Work in a well-ventilated area outside of the field van and downwind of the sampling area.

b. Place pump into a clean, dedicated, solvent-resistant standpipe and route discharge end of sample tubing to a methanol waste container. Add methanol solution to level above pump intake.

c. Pump about 2 L of methanol through sample tubing into methanol waste container, keeping the level of solution above pump intake. The operator should stand back from the pump as a safety precaution in the event that an electrical spark ignites the methanol. Carefully put any unused methanol from bottom of standpipe into methanol waste container. Let methanol in the standpipe evaporate to dryness. Change gloves.

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[5]NEC Class 1; Group D: Areas in which flammable gases or vapors may be present in the air in sufficient quantities to be explosive; atmospheres such as acetone, alcohol, ammonia, benzene, benzol, butane, gasoline, hexane, lacquer solvent vapors, naphtha, natural gas, propane, or gas or vapors of equivalent hazard (Cole-Parmer Instrument Company, 1997).

d. Rinse pump exterior with organic-grade water and place pump into standpipe. Add organic-grade water to the standpipe to push the methanol out of the tubing and into the methanol waste container. Pump at least an additional 0.1 gallon (about 0.38 L) of organic-grade water through the system for every 10 ft (about 3.05 m) of methanol-wetted tubing to the methanol waste container after used methanol is collected.

TECHNICAL NOTE: The recommended organic-grade water is PBW or VBW (supplied by NWQL for blank samples). Office-produced organic-grade water might not be of adequate purity, especially after being stored, and its use requires collection of additional blank samples for quality control (see section 3.4).

e. Repeat d above with blank water (PBW or VBW) pumped from a blank-water standpipe if blank samples will be collected for analysis of organic compounds.

Use of methanol is not recommended as a routine procedure for field cleaning of the pump. A methanol rinse is most safely accomplished as an office laboratory procedure.

Storage of the cleaned submersible pump and tubing:

1. Place pump into two clean, noncontaminating storage bags and close bags.
2. Cover the pump reel and tubing with doubled plastic bags or sheeting for transport to the next site.

For long-term storage (longer than 3 days), the pump and exterior and interior of the tubing must be dry before being placed into plastic bags. Tubing can be dried by blowing filtered air or filtered (inert) gas through the tubing. If tubing cannot be dried, store chilled to prevent bacterial growth. If bacterial growth has occurred, reclean before use.

QUALITY CONTROL FOR EQUIPMENT-CLEANING PROCEDURES 3.4

By A.J. Horowitz, M.W. Sandstrom, and F.D. Wilde

Quality-control samples are required for any sampling and analysis program. Without quality-control information, the quality of the environmental data collected can be neither evaluated nor qualified. If the user has no means of knowing the associated errors, the data cannot be interpreted properly.

The purpose for obtaining quality-control (QC) samples following equipment cleaning is to ensure that the equipment and the procedures used for cleaning the equipment do not contaminate or otherwise affect the environmental samples that were or will be collected. The QC sample used to assess the adequacy of cleaning procedures before field work commences is called the equipment blank.

- **Blank water.** Blank water is used to develop specific types of QC samples (National Water Quality Laboratory Memorandum 92.01). The water is a solution that is free of analyte(s) of interest at a specified detection level. USGS personnel are required to use blank water that has been analyzed and certified to be of a specific grade and composition.

  - Use IBW to collect blank samples for analysis of inorganic constituents.
  - Use PBW to collect blank samples for analysis of pesticides. (Do not use PBW when collecting samples for VOC analysis.)
  - Use VBW to process blank samples for analysis of VOCs. VBW is also suitable as a blank sample for pesticide analysis.
  - Use PBW or VBW as the quality-control sample for total and dissolved organic-carbon analysis (TOC and DOC). This cannot be documented as a blank sample because neither PBW nor VBW is certified to be free of organic carbon.

- **Equipment blank.** An equipment blank is blank water that is processed under controlled conditions in the office laboratory by being passed sequentially through each component of the sample processing and collection equipment. **An equipment blank represents an entire sampling system (fig.3-7) and is required:**
  - Annually.
  - When a cleaning procedure is followed for the first time.
  - When new equipment will be used for the first time.

To fulfill equipment-blank requirements:

1. Allow enough time in the study workplan to collect the annual equipment blank, complete laboratory analyses, and review analytical results before field work for the study commences.
2. Process the annual equipment blank in a clean, controlled environment in the office laboratory, after the equipment has been cleaned using office-laboratory procedures.
3. Analyze the annual equipment-blank data before collecting and processing the first water-quality sample of either the fiscal year or the study.
   - If the equipment-blank data indicate that the equipment does not introduce contaminants that will bias study results, sampling can proceed.
   - If the equipment-blank data indicate unacceptable concentrations of analytes of interest, the cause must be identified and the equipment or cleaning procedures must be changed or modified before sampling can proceed.

Plan ahead! Assess equipment-blank data before environmental samples are collected.

- **Field blank.** The field blank is blank water that is processed at the field site by being passed sequentially through each component of the equipment being used to collect environmental samples. The procedure for processing the field blank, like the equipment blank, can also result in a set of sequentially collected blank samples (fig. 3-7) (Horowitz and others, 1994). Other types of blank samples also are collected at the field site (NFM 4). **At least one field blank per sampling run is recommended; the numbers and distribution of QC samples depend on study objectives, the target analytes, and site conditions.**

  - Process field blanks through clean equipment.
  - If equipment is used at several sites during a field trip, process a field-equipment blank after the last sample has been collected and again after the equipment has undergone the prescribed field-cleaning procedures.
  - If multiple sets of office-cleaned equipment are used during a field trip, process a field blank at any site during the course of the trip. In this case, the blank must be processed before sampling to avoid contaminating the blank with residues from an environmental sample.
  - Process field blanks onsite and under the same conditions as the environmental sample.

Before filling the QC sample bottle with the appropriate blank water:

1. Check that sample bottles are clean, are the correct type, and are labeled correctly.
2. Check the certificate of analysis for the lot of blank water to be sure that it is appropriate for quality control of target analytes.
3. Record the date and lot number of the IBW, PBW, and (or) VBW used and of the preservative used. To the extent possible, use preservative from the same lot number for an entire sampling trip for both the environmental and quality-control samples.
4. Rinse sample bottles for inorganic constituents three times with a small quantity of the blank water.

Use the following strategy for QC data collection and analysis:

1. For inorganic-constituent samples, initially send only the final equipment-blank sample for the routine inorganic blank-sample analysis or for inorganic analytes targeted by the study.

   - Archive the remaining sequentially processed blank samples (fig.3-7) until the inorganic-constituent analysis of the equipment-blank sample has been received.
   - Do not archive blank samples for organic-compound analysis.

2. Check the analytical results for the equipment blank and field blanks as soon as possible and before the next field trip.

   - If analytical results indicate that the equipment is clean within acceptable limits, the equipment may be used for field work without additional testing or analysis.
   - **Use of equipment is not recommended if analysis of the equipment blank sample indicates greater than acceptable analyte concentrations.**

3. Additional QC data collection and (or) analysis is required if the equipment blank has greater than acceptable analyte concentrations.

   - **For inorganic-sample analysis.** Submit the rest of the sequential blank samples for laboratory analysis and use the analytical results from the sequential blank samples to identify potential source(s) of contamination. Modify equipment-cleaning procedures if contamination can be remedied by a change in cleaning procedure. Repeat collection of equipment blanks until the blank data verify that the equipment is suitable for use.
   - **For organic-sample analysis.** Modify the equipment cleaning procedure if the source of contamination is known or suspected and contamination can be remedied by a change in cleaning procedure. If the source of contamination is not known, reclean equipment using office-laboratory procedures and collect and analyze blanks for each part of the sampling system that could be a source of contamination. Repeat collection of equipment blanks until the blank data verify that the equipment is suitable for use.

The equipment blank is the last sample of a set of sequentially processed blanks collected in the office laboratory and documents the suitability of the equipment for the samples that are to be collected and analyzed. **Field blanks** are collected in the field in the same manner as the equipment blank but document the effectiveness of the field-cleaning procedures plus any ambient contamination.

- Surface water: collect the series of five sequential blank samples listed below for routine surface-water sampling.
- Ground water: collect the source-solution blank (Sample 1) and either a sampler blank (Sample 2) or pump blank (Sample 4) (depending on the type of sampling device being used) along with the filter blank (Sample 5).

Sample 1. SS blank	**Source solution (SS)** Put on disposable gloves. Pour the IBW, PBW, or VBW directly into appropriate SS blank-sample bottle.[1] Add chemical treatment and (or) chill, as required for the analytes of interest.
Sample 2. Sampler blank	**SS + Sampler** Bottle or bag sampler: Fill sampler container with SS; attach sampler cap and nozzle; decant sample into blank-sample bottle through the nozzle. Preserve sample (add chemical treatment and (or) chill) as required (NFM 5). Bailer or thief sampler: Fill sampler with SS; install bottom-emptying device; empty sample into blank-sample bottle through the bottom-emptying device. Preserve sample, as required. Submersible or nonsubmersible pumps: Go to Sample 4 (Pump blank).
Sample 3. Splitter blank	**SS + Sampler + Splitter[2]** If a cone or churn splitter is used, decant remainder of the SS into sampler container, and then through splitter (through nozzle or bottom-emptying device). Refill sampler container with SS to fill churn with 3 to 5 liters of water. Alternatively, pour enough SS from samplers through cone splitter to fill splitter-blank bottle. Collect SS into blank-sample bottle through churn spigot or cone-splitter exit port(s). Preserve sample, as required.
Sample 4. Pump blank	**SS + Sampler + Splitter + Pump** Nonsubmersible pump (peristaltic, vacuum, or valveless metering pump): Secure intake end of clean pump tubing into churn splitter or into a subsample split with the cone splitter. Pump some sample to waste to rinse tubing, and fill pump-blank bottle directly from the discharge end. Preserve sample, as required. Submersible pump: Place pump in blank-water standpipe and fill standpipe with enough SS to cover pump intake and allow for drawdown. Start pump at low pumping rate, discharge 0.5 liter of SS to waste, then fill blank-sample bottle with SS. Preserve sample, as required.
Sample 5. Filter or equipment blank	**SS + Sampler + Splitter + Pump + Filter** Pump SS through a prerinsed filtration assembly (plate filter or capsule filter); pump the first aliquot to waste and then pump SS directly into the blank-sample bottle. Preserve sample, as required.

[1]Process the source-solution blank in the protected environment of the office laboratory only, not in the field (NFM 4).

[2]For ground-water quality control: A splitter blank is included if a cone splitter is used; a standpipe blank often is collected if a submersible pump is used.

Figure 3-7. Sequence of sample collection to obtain the equipment blank

PUBLICATIONS ON TECHNIQUES OF WATER-RESOURCES INVESTIGATIONS

The U.S. Geological Survey publishes a series of manuals describing procedures for planning and conducting specialized work in water-resources investigations. The material is grouped under major subject headings called books and is further divided into sections and chapters. For example, Section A of Book 9 (Handbooks for Water-Resources Investigations) pertains to collection of water-quality data. The chapter, which is the unit of publication, is limited to a narrow field of subject matter. This format permits flexibility in revision and publication as the need arises.

The Techniques of Water-Resources Investigations (TWRI) reports listed below are for sale by the U.S. Geological Survey, Branch of Information Services, Box 25286, Federal Center, Denver, CO 80225 (authorized agent of the Superintendent of Documents, Government Printing Office). Prepayment is required. Remittance should be sent by check or money order payable to the U.S. Geological Survey. Prices are not included because they are subject to change. Current prices can be obtained by writing to the above address. When ordering or inquiring about prices for any of these publications, please give the title, book number, chapter number, and "U.S. Geological Survey Techniques of Water-Resources Investigations." An updated list of TWRI reports can be found by accessing the World Wide Web url: http://water.usgs.gov/lookup/get?TWRI.

Book 1. Collection of Water Data by Direct Measurement

Section D. Water Quality

1-D1. Water temperature—influential factors, field measurement, and data presentation, by H.H. Stevens, Jr., J.F. Ficke, and G.F. Smoot: USGS—TWRI Book 1, Chapter D1. 1975. 65 pages.

1-D2. Guidelines for collection and field analysis of ground-water samples for selected unstable constituents, by W.W. Wood: USGS—TWRI Book 1, Chapter D2. 1976. 24 pages.

Book 2. Collection of Environmental Data

Section D. Surface Geophysical Methods

2-D1. Application of surface geophysics to ground-water investigations, by A.A.R. Zohdy, G.P. Eaton, and D.R. Mabey: USGS—TWRI Book 2, Chapter D1. 1974. 116 pages.

2-D2. Application of seismic-refraction techniques to hydrologic studies, by F.P. Haeni: USGS—TWRI Book 2, Chapter D2. 1988. 86 pages.

Section E. Subsurface Geophysical Methods

2-E1.Application of borehole geophysics to water-resources investigations, by W.S. Keys and L.M. MacCary: USGS—TWRI Book 2, Chapter E1. 1971. 126 pages.

2-E2.Borehole geophysics applied to ground-water investigations, by W.S. Keys: USGS—TWRI Book 2, Chapter E2. 1990. 150 pages.

Section F. Drilling and Sampling Methods

2-F1.Application of drilling, coring, and sampling techniques to test holes and wells, by Eugene Shuter and W.E. Teasdale: USGS—TWRI Book 2, Chapter F1. 1989. 97 pages.

Book 3. Applications of Hydraulics

Section A. Surface-Water Techniques

3-A1.General field and office procedures for indirect discharge measurements, by M.A. Benson and Tate Dalrymple: USGS—TWRI Book 3, Chapter A1. 1967. 30 pages.

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http://water.usgs.gov/lookup/get?techmemo.

Water Quality

Memo No.	Title	Date
qw 92.01	Distilled/Deionized Water for District Operations	Dec. 20, 1991
qw 97.03	Protocols for Cleaning a Teflon Cone Splitter to Produce Contaminant-Free Subsamples for Subsequent Determinations of Trace Elements	Feb. 7, 1997

National Water Quality Laboratory (NWQL)

Memo No.	Title	Date
92.01	Technology Transfer—Availability of Equipment Blank Water for Inorganic and Organic Analysis	Mar. 25, 1992

Water Resources Division

Memo No.	Title	Date
wrd 94.007	Safety--Storage, Transportation, Handling and Disposal of Methyl Alcohol	Dec. 3, 1993

CONVERSION FACTORS AND ABBREVIATIONS

CONVERSION FACTORS

Multiply	By	To obtain
centimeter (cm)	0.3937	inch
meter	3.281	foot
milliliter (mL)	0.06102	$inch^3$ or cubic inch
liter (L)	0.2642	gallon
microgram (µg)	3.53×10^{-8}	ounce

Temperature: Water and air temperature are given in degrees Celsius (°C), which can be converted to degrees Fahrenheit (°F) by use of the following equation:

$$°F = 1.8(°C) + 32$$

ABBREVIATIONS

DIW	deionized water
DOC	dissolved organic carbon
HCl	hydrochloric acid
HNO_3	nitric acid
IBW	inorganic-grade blank water, laboratory-certified free of trace elements and other inorganic constituents
µg/L	micrograms per liter
µS/cm	microsiemens per centimeter at 25°C
MSDS	Material Safety Data Sheet
NFM	*National Field Manual for the Collection of Water-Quality Data*
NWQL	National Water Quality Laboratory of the U.S. Geological Survey
OWQ	Office of Water Quality of the U.S. Geological Survey
PBW	pesticide-grade blank water, certified free of pesticide organic compounds by the NWQL
PVC	polyvinyl chloride
QC	quality control
QWSU	Quality of Water Service Unit
SOC	suspended organic carbon
SS	source solution
TOC	total organic carbon
TWRI	Techniques of Water-Resources Investigations
URL	Uniform Resource Locator
USGS	U.S. Geological Survey
VBW	volatiles-grade blank water, certified free of volatile compounds by the NWQL
v/v	volume to volume

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APPENDIX IV, LABORATORY STANDARD OPERATING PROCEDURES

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Standard Operating Procedure: Edward S. Babcock & Sons

METHOD #: EPA 180.1
Standard Methods 2130 B

TITLE: Turbidity (Nephelometric)

1.0 Scope and Application
1.1 This method is applicable to drinking, surface, and saline waters in the range of turbidity from 0.05 to 4000 nephelometric turbidity units (NTU).

NOTE 1: NTU's are considered comparable to the previously reported Formazin Turbidity Units (FTU) and Jackson Turbidity Units (JTU).

2.0 Summary of Method
2.1 The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity. Readings, in NTU's, are made in a nephelometer designed according to specifications outlined in Apparatus. A standard suspension of Formazin, prepared under closely defined conditions, is used to calibrate the instrument.

2.1.1 Formazin polymer is used as the turbidity reference suspension for water because it is more reproducible than other types of standards previously used for turbidity standards.

2.1.2 A commercially available polymer standard is also approved for use for the National Interim Primary Drinking Water Regulations. This standard is identified as AMCO-AEPA-1 available from Amco Standard International, Inc.

3.0 Sample Handling and Preservation
3.1 Samples may be stored in either plastic or glass.

3.2 Preservation consists of refrigeration or icing to 4-C. to minimize microbiological decomposition of solids.

3.3 Analysis must be performed within *24 hours* per 40CFR section 136, Table II. Samples must be stored at 4°C.

4.0 Interferences
4.1 The presence of floating debris and coarse sediments which settle out rapidly will give low readings. Finely divided air bubbles will affect the results in a positive manner.

4.2 The presence of true color, that is the color of water which is due to dissolved substances which absorb light, will cause turbidities to be low, although this effect is generally not significant with finished waters.

5.0 Apparatus

5.1 The 2100N Hach turbidimeter consists of a nephelometer with light source for illuminating the sample a photo-electric detector with a readout device to indicate the intensity of light scattered at right angles to the path of the incident light. The turbidimeter is designed so that little stray light reaches the detector in the absence of turbidity should be free from significant drift after a short warm-up period.

5.2 The sensitivity of the instrument permits detection of a turbidity difference of 0.05 unit or less in waters having turbidities less than 1 unit. (The minimum detection level reported is 0.05 NTU.) The instrument is able to measure from 0.05 to 4000 units turbidity. Several ranges are available to obtain both adequate coverage and sufficient sensitivity for low turbidities.

5.3 The sample tubes are made of clear, colorless glass. They must be kept scrupulously clean, both inside and out, and discarded when they become scratched or etched. They must not be handled at all where the light beam from the instrument strikes them, so they are provided with sufficient extra length so that they may be handled at the top. Check all tubes before analysis by reading a D.I. blank.

5.4 Differences in physical design of turbidimeters will cause differences in measured values for turbidity even though the same suspension is used for calibration. To minimize such differences, the following design criteria are observed:

5.4.1 Light source: Tungsten lamp operated at a color temperature between 2200-3000-K.

5.4.2 Distance traversed by incident light and scattered light within the sample tube: Total not to exceed 10 cm.

5.4.3 Detector: Centered at 90- to the incident light path and not to exceed +/- 30 C- from 90 C. The Detector, and filter system if used, shall have a spectral peak response between 400 and 600 nm.

5.5 Standard laboratory glassware: volumetric flasks, beakers, graduated cylinders, pipets.

Note: All glassware is cleaned immediately prior to and after use by thorough rinsing with three portions of D.I. water. If glassware still appears dirty, further steps are taken, by use of one of the following: Alconox and hot water, 1:1 acid rinse, acetone or appropriate solvent rinse. Glassware is always finished with a final D.I. rinse.

6.0 Reagents and Standards
6.1 Turbidity-free water: D.I. water is used as long as it does not read above 0.1 NTU. If it does read high, Nanopure water is used.

6.2 Stock formazin turbidity suspension: 4000 NTU Formazine Solution purchased for supplier, stored at room temperature until Manufacturer specified expiration date. Two sources are purchased, one for calibration standard preparation and the second source for calibration verification (LCS) preparation.

6.3 Standard formazin turbidity suspension prepared:
6.3.1 LCS : Prepare daily as specified below for from noncalibration stock source
6.3.2 Calibration Standards: Prepare each time a calibration is necessary as specified below for instrument calibration.

For: Standard Concentration NTU	Pipette mls From Stock	*Dilute To: (Volume in mls) with D.I.
4000	fill cell	0
1000	25	100
800	20	100
400	10	100
200	5	100
80	2	100
40	1	100
20	1	200
8	2	1000
4	1	1000
0.9	0.2	1000

7.0 Procedure
7.1 Turbidimeter calibration: The manufacturer's operating instructions are followed which specify calibration every 90 days for USEPA reporting. However, should the Electronic P.C. Board, the Photo Detectors, or the Light Source be replaced or if very carefully prepared Formazine Suspensions indicate a need for recalibration, this will be done more often.

7.2 Calibration
7.2.1 Always mix the contents of each cell by inverting several times before placing in the Optical Well for reading.

7.2.2 Keep the outside surface of the cell clean and dry. Apply a drop of silicone oil to the outside and wipe with a cloth or tissue. Finish with a chem wipe.

7.2.3 When placing any standards in the well, always use the light shield to cover the well in order to keep out ambient light.

7.3 Carry out the following steps:
7.3.1 First place a cell of D.I. water in the cell holder.
7.3.2 Press the CAL key.
7.3.3 Press the ENTER key.
7.3.4 The instrument will advance to the next standard, display the expected value, and the S1 light.
7.3.5 Place the 20 NTU standard in the cell holder.
7.3.6 Press ENTER.
7.3.7 Once the instrument displays the next standard value and S# light, place that standard in the cell holder, press ENTER and so on until all the standards have been read.
7.3.8 The standard values are: 20, 200, 1000, and 4000.
7.3.9 After the last standard is processed, press the CAL key.

7.4 Analysis:
7.4.1 Clean and rinse the cell with D.I., wiping all excess water from the sides with tissue. Apply a drop of silicone oil to the outside and wipe with a cloth or tissue. Finish with a chem wipe.
7.4.2 Check the calibration by reading the 0.9 Lab Control. If it is not within range repour or remake it. If it is still out, examine the instrument for problems. The calibration may have to be repeated. Sample results may not be taken until a Lab Control falls within the acceptance ranges.
7.4.3 Shake the sample and pour it into the cell. Wipe the cell with lens paper to make sure there are not smudges, then put the cell into the turbidity meter with the line on the cell pointing foreword. If the sample reading is higher than the 0.90 range, the calibration must be checked using a lab control (from the previous table) that encompasses the sample's range. Record the lowest stabilized reading before the sample particles have settled.
7.4.4 *Do not dilute the sample!* (Standard Methods, 20th Edition, Method 2130B 4a and Hach Model 2100N Turbidity Meter Manual 2.3.8.) Readings above 4000 are reported as >4000 NTU.

8.0 Calculation and Reporting Requirements:
8.1 Results are reported in NTU's.

8.2 Report results to 2 significant figures.

8.3 Detection Limit for Reporting Purposes (RL) = 0.05 NTU

9.0 QA/QC Requirements:
9.1 The 0.9 NTU LCS serves as an Initial Calibration check analyzed at the beginning of the analysis. Another LCS, made at a different concentration is analyzed at the end of the run to verify calibration.

9.2 If the lab control does not read within the limits of 85-115%, re-make and read again. If the lab control still does not read correctly, re-calibrate the instrument.

9.3 Duplicates are analyzed with each batch of 20 samples. Results must have a RPD ± 20%.

9.4 An MDL study is completed at a minimum of once per year, or whenever major equipment or procedural changes are made. Standards are spiked at the reporting limit and a minimum of seven replicates is analyzed. See QA Manual section 23 for calculation. Results must be below the reporting limit.

9.5 Initial Demonstration of Capability: Prior to analysis of samples or when a significant change is made to the method, an Initial Demonstration of Capability Study is performed. This is accomplished by analysis of four replicates of a QC sample made at a concentration 10 times the MDL. Acceptance ranges are 80-120% with a maximum %RSD of 20.

9.6 Performance Evaluation Studies performed twice a year serve as documentation of continuing proficiency.

10.0 Definitions: See SOP Q15 - SOP Definitions

11.0 Safety: General laboratory safety procedures are adequate for this analysis.

12.0 Corrective Action for Out of Control Or Unacceptable Data:
See SOP Q06 - Corrective Action

13.0 Pollution Prevention and Waste Management:
See SOP S07 - Pollution Prevention

14.0 Method Performance
Refer to MDL studies, Initial Demonstration of Capability Studies, and laboratory control charts maintained in the QC Office.

Revision Log

Rev.2.3 - 07/01/01: added Revision Log, and sections: 5.5, 9.1, 9.4, 9.5, 9.6, 14.0, edited sections 6.2, 9.2.

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Approved by Susan K Thomas 07/03/01

RESIDUE, TOTAL SUSPENDED
Edward S. Babcock & Sons
STANDARD OPERATING PROCEDURE
(EPA Method 160.2)
(SM 2540 D)

1.0 Scope and Application:

1.1 This method is applicable to all aqueous samples.

2.0 Working Range:

2.1 The working range is 5mg/L to 2000mg/L.

3.0 Method Summary:

3.1 100mls of sample is filtered through a pretared filter. The residue that remains on this filter after drying in a 105 degree Celsius oven is considered the suspended solid portion of the sample.

4.0 Sample Collection, Preservation and Holding Time:

4.1 The sample must be unpreserved. It must be stored at 4 degrees Celsius until analysis. Analysis must take place within 7 days of sampling per CFR part 136, Table II.

5.0 Interferences:

5.1 Exclude large floating particles or submerged agglomerates of nonhomogeneous materials from the sample if it is determined that their inclusion is not desired. Nonrepresentative particulates such as leaves and sticks may also be excluded.

5.2 To avoid water entrapment, limit the sample size to that yielding no more than 200mg residue on the filter. (This would be a final result of 2000mg/L since we are analyzing 100mls of sample.)

5.3 For samples high in dissolved solids thoroughly wash the filter with D.I. water after the sample has passed through the filter.

5.4 Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter. If more than 10

minutes are required to complete filtration. Increase filter size or decrease sample size.

6.0 Apparatus and Standards

6.1 Side-arm flask of sufficient capacity for sample size selected.

6.2 Filtration apparatus: Membrane filter funnel with a Gelman type A/E glass fiber filter disk with a suitable diameter for the funnel.

6.3 Drying oven, for operation at 105°C ± 2°C.

6.4 Vacuum aspirator.

6.5 Desiccator.

6.6 Balance with a sensitivity of 0.1 mg. Calibrated on a daily basis with 0.1 g and 100 g class "S" weights on a daily basis. Calibration must be within ± 0.5%. If values are not within these limits, recalibrate the balance.

6.7 Filter garages to hold glass fiber filters.

6.8 Standard laboratory glassware: volumetric flasks, beakers, graduated cylinders, pipets.

Note: All glassware is cleaned immediately prior to and after use by thorough rinsing with three portions of D.I. water. If glassware still appears dirty, further steps are taken, by use of one of the following: Alconox and hot water, 1:1 acid rinse, acetone or appropriate solvent rinse. Glassware is always finished with a final D.I. rinse.

6.9 Stock : Cellulose

6.10 Lab Control: 500mg of cellulose is weighed into a liter of D.I. water. This solution is kept at room temperature for up to a year.

7.0 Procedure

7.1 Prepare glass fiber filters by rinsing three times with D.I. water and heating at 105°C for a minimum of 1 hour. A constant weight study is performed yearly to establish the minimum time required to bring the filter to a constant weight.

7.2 Take hot filters out of 105°C oven.

7.3 Cool filters to room temperature in a desiccator. Use forceps when handling filters. Weigh on balance for tare weight and record. Place the filter onto the filtering apparatus. Wet filter with a small amount of D.I. to seat it.

7.4 Mix sample well by shaking sample bottle. Measure an appropriate volume of sample in a graduated cylinder. For normal samples use 100 ml. For samples with a lot of suspended matter, a smaller volume of sample may be used 50 ml to 10 ml. A larger filtering apparatus may be necessary as well. Use a 200 ml sample volume for samples expected to contain very minute amounts of suspended material Filter through apparatus collecting suspended residue on filter. Rinse cylinder and filter 2 to 3 times with a small amount of D.I. water. Suction three minutes after filtration or until no visible free liquid is present.

7.5 Place samples in 105°C oven for 1.5 hours which is longer than the time proven to be sufficient to bring the sample to a constant weight.

7.6 Cool filters in a desiccator and weigh filters for final weight. Record weight.

8.0 Calculation:

$$\frac{(A-B) \times 1,000,000}{\text{\# ml of sample used}}$$

Where A = Weight in grams of filter with residue, and

B = Tare weight in grams of filter.

8.1 Alternatively, you may subtract the actual numbers in the weight readings (without any decimal points) and multiply the difference by the factor of 100/(ml of sample used).

8.2 The detection limit for this procedure is 5 mg/L.

8.3 Report all results to two significant figures.

9.0 Quality Control:

9.1 Duplicates are analyzed at least once in every analytical batch and at a minimum of once for every 20 samples. The Relative Percent Difference is calculated and compared to the acceptance limit for the analysis. If the RPD does not fall within the acceptance limit which is a maximum of 40, the analysis is re-run. If the RPD still does not fall within the acceptance limit, the supervisor is informed. If it is determined that the matrix of the sample interfered with the analysis, this is noted on the worksheet.

9.2 A method blank is analyzed with every batch of samples. It must read ± 5mg/L.

9.3 A lab control is analyzed with every batch of samples. 500mg of cellulose is weighed into a liter of D.I. water. 100 ml of this solution is analyzed. Results must be ± 20%.

9.4 An MDL study is completed at a minimum of once per year, or whenever major equipment or procedural changes are made. Standards are spiked at the reporting limit and a minimum of seven replicates is analyzed. See QA Manual section 23 for calculation. Results must be below the reporting limit.

9.5 Initial Demonstration of Capability: Prior to analysis of samples or when a significant change is made to the method, an Initial Demonstration of Capability Study is performed. This is accomplished by analysis of four replicates of a QC sample made at a concentration 10 times the MDL. Acceptance ranges are 80-120% with a maximum %RSD of 20.

9.6 Performance Evaluation Studies performed twice a year serve as documentation of continuing proficiency.

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10.0 Corrective Action For Out of Control Or Unacceptable Data:
See SOP Q06 – Corrective Action

11.0 Pollution Prevention and Waste Management:
SOP S07 • Pollution Prevention

12.0 Definitions: See SOP Q15 - SOP Definitions

13.0 Safety

13.1 General laboratory safety procedures are sufficient for this analysis. Recommended safety equipment includes gloves and safety glasses.

14.0 Method Performance:
Refer to MDL studies, Initial Demonstration of Capability Studies, and laboratory control charts maintained in the QC Office.

Revision Log

Rev.2.2 - 07/01/01: added Revision Log, and sections:6.8, 6.9, 6.10, 9.4, 9.5, 9.6 , edited sections:1.6, 14.0

10.0 References

Standard Methods For the Examination of Water and Wastewater 18th Edition APHA/AWWA/WEF 2540D.

Methods for the Chemical Analysis of Waters and Wastes EPA 160.2.

Approved by Susan K Thomas 07/03/01

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Standard Operating Procedure
Edward S. Babcock & Sons

METHOD #: 8081B
TITLE: ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY
INSTRUMENTATION: Gas Chromatography

1.0 SCOPE AND APPLICATION

1.1 Method 8081 is used to determine the concentrations of various organochlorine pesticides in xtracts from solid and liquid matrices, using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).. The compounds listed below are determined by a dual-column analysis system. Analytes in parenthesis are not certified by NELAP.

Compound	CAS Registry No.
Aldrin	309-00-2
alpha-BHC	319-84-6
beta-BHC	319-85-7
gamma-BHC (Lindane)	58-89-9
delta-BHC	319-86-8
(Chlorobenzilate)	510-15-6
(alpha-Chlordane)	5103-71-9
(gamma-Chlordane)	5103-74-2
Chlordane - not otherwise specified	57-74-9
(DBCP)	96-12-8
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
(Diallate)	2303-16-4
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
(Endrin ketone)	53494-70-5
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Hexachlorobenzene	118-74-1
Hexachlorocyclopentadiene	77-47-4
(Isodrin)	465-73-6
Kepone	143-50-0
Methoxychlor	72-43-5
Toxaphene	8001-35-2

1.2 Several multi-component mixtures (i.e., Chlordane and Toxaphene) are listed as target analytes. When samples contain more than one multi-component analyte, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of multi-component analytes that have been subjected to environmental degradation or degradation by treatment technologies. These result in "weathered" multi-component mixtures that may have significant differences in peak patterns than those of standards.

1.3 The dual-column option is used. The option allows a hardware configuration of two analytical columns joined to a single injection port. The option allows one injection to be used for dual-column analysis. Analysts are cautioned that the dual-column option may not be appropriate when the instrument is subject to mechanical stress, many samples are to be run in a short period, or when contaminated samples are analyzed.

1.4 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs (GC) and skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.5 Extracts suitable for analysis by this method may also be analyzed for organophosphorus pesticides (Method 8141). Some extracts may also be suitable for triazine herbicide analysis, if low recoveries (normally samples taken for triazine analysis must be preserved) are not a problem.

1.6 The following compounds may also be determined using this method. Analytes in parenthesis are not certified by NELAP

Compound	CAS Registry No.
(Alachlor)	15972-60-8
(Captafol)	2425-06-1
(Chloroneb)	2675-77-6
(Chloropropylate)	5836-10-2
Chlorothalonil	1897-45-6
(DCPA)	1861-32-1
(Dichlone)	117-80-6
(Dicofol)	115-32-2
(Etridiazole)	2593-15-9
(Halowax-1000)	58718-66-4
(Halowax-1001)	58718-67-5
(Halowax-1013)	12616-35-2
(Halowax-1014)	12616-36-3
(Halowax-1051)	2234-13-1
(Halowax-1099)	39450-05-0
(Mirex)	2385-85-5
(Nitrofen)	1836-75-5
(PCNB)	82-68-8
(Permethrin) (cis + trans)	52645-53-1
(Perthane)	72-56-0
Propachlor	1918-16-7
(Strobane)	8001-50-1
(trans-Nonachlor)	39765-80-5
(Trifluralin)	1582-09-8

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (approximately 1 L for liquids, 2 g to 10 g for solids) is extracted using the appropriate matrix-specific sample extraction technique.

2.2 Liquid samples are extracted at neutral pH with methylene chloride using Method 3510 (separatory funnel).

2.3 Solid samples are extracted with hexane-acetone (1 – 1) using Method 3550 (ultrasonic extraction).

2.4 A variety of cleanup steps may be applied to the extract, depending on the nature of the matrix interferences and the target analytes. Florisil (Method 3620), clean up is most commonly used.

2.5 After cleanup, the extract is analyzed by injecting a 2 uL sample into a gas chromatograph with a narrow- or wide-bore fused-silica capillary column and electron capture detector (GC/ECD).

3.0 INTERFERENCES

3.1 Sources of interference in this method can be grouped into three broad categories.

3.1.1 Contaminated solvents, reagents, or sample processing hardware.

3.1.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

3.1.3 Compounds extracted from the sample matrix to which the detector will respond.

3.1.4 Interferences co-extracted from the samples will vary considerably from waste to waste. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

3.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

3.2.1 These materials may be removed prior to analysis using Method 3640 (Gel Permeation Cleanup) or Method 3630 (Silica Gel Cleanup).

3.2.2 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations.

3.2.3 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled.

3.2.4 Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

3.3 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot

water, and rinses with tap water and organic-free reagent water. Drain the glassware and dry it, or rinse with methanol and drain. Store dry glassware in a clean environment.

3.4 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. Method 3660 is suggested for removal of sulfur. Since the recovery of Endrin aldehyde (using the TBA procedure) is drastically reduced, this compound must be determined prior to sulfur cleanup.

3.5 Waxes, lipids, and other high molecular weight materials can be removed by Method 3640 (gel-permeation cleanup).

3.6 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Certain co-eluting organophosphorus pesticides are eliminated by Method 3640 (gel-permeation cleanup - pesticide option). Co-eluting chlorophenols may be eliminated by using Method 3630 (silica gel), Method 3620 (Florisil), or Method 3610 (alumina). Polychlorinated biphenyls (PCBs) also may interfere with the analysis of the organochlorine pesticides. The problem may be most severe for the analysis of multicomponent analytes such as Chlordane, Toxaphene, and Strobane. If PCBs are known or expected to occur in samples, the analyst should consult Methods 3620 and 3630 for techniques that may be used to separate the pesticides from the PCBs.

3.7 The following compounds may coelute using the dual-column analysis scheme. In general, the HP-5 column resolves fewer compounds that the HP-1701.

HP-5 Permethrin/Heptachlor epoxide
Endosulfan I/alpha-Chlordane
Perthane/Endrin
Endosulfan II/Chloropropylate/Chlorobenzilate
4,4'-DDT/Endosulfan sulfate
Methoxychlor/Dicofol

HP-1701 Chlorothalonil/beta-BHC
delta-BHC/DCPA/Permethrin
alpha-Chlordane/trans-Nonachlor

Nitrofen, Dichlone, Carbophenothion, Dichloran exhibit extensive peak tailing on both columns. Simazine and Atrazine give poor responses on the ECD detector. Triazine compounds should be analyzed using Method 8141 (NPD option).

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph - An analytical system complete with gas chromatograph suitable for on-column and split-splitless injection and all required accessories including autosampler, analytical columns, gases, electron capture detectors (ECD), and data system.

4.1.1 Primary Gas Chromatograph: HP 5890.
4.1.2 Detector: Electron Capture Detector.
4.1.3 Turbochrome/ Data Capture
4.1.4 Primary column: HP5MS 30m x 0.25mm ID, 0.25 µm thickness.
4.1.5 Confirmatory Gas Chromatograph: Hewlett-Packard 5890.

water, and rinses with tap water and organic-free reagent water. Drain the glassware and dry it, or rinse with methanol and drain. Store dry glassware in a clean environment.

3.4 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. Method 3660 is suggested for removal of sulfur. Since the recovery of Endrin aldehyde (using the TBA procedure) is drastically reduced, this compound must be determined prior to sulfur cleanup.

3.5 Waxes, lipids, and other high molecular weight materials can be removed by Method 3640 (gel-permeation cleanup).

3.6 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Certain co-eluting organophosphorus pesticides are eliminated by Method 3640 (gel-permeation cleanup - pesticide option). Co-eluting chlorophenols may be eliminated by using Method 3630 (silica gel), Method 3620 (Florisil), or Method 3610 (alumina). Polychlorinated biphenyls (PCBs) also may interfere with the analysis of the organochlorine pesticides. The problem may be most severe for the analysis of multicomponent analytes such as Chlordane, Toxaphene, and Strobane. If PCBs are known or expected to occur in samples, the analyst should consult Methods 3620 and 3630 for techniques that may be used to separate the pesticides from the PCBs.

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Endosulfan I/alpha-Chlordane
Perthane/Endrin
Endosulfan II/Chloropropylate/Chlorobenzilate
4,4'-DDT/Endosulfan sulfate
Methoxychlor/Dicofol

HP-1701 Chlorothalonil/beta-BHC
delta-BHC/DCPA/Permethrin
alpha-Chlordane/trans-Nonachlor

Nitrofen, Dichlone, Carbophenothion, Dichloran exhibit extensive peak tailing on both columns. Simazine and Atrazine give poor responses on the ECD detector. Triazine compounds should be analyzed using Method 8141 (NPD option).

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph - An analytical system complete with gas chromatograph suitable for on-column and split-splitless injection and all required accessories including autosampler, analytical columns, gases, electron capture detectors (ECD), and data system.

4.1.1 Primary Gas Chromatograph: HP 5890.
4.1.2 Detector: Electron Capture Detector.
4.1.3 Turbochrome/ Data Capture
4.1.4 Primary column: HPSMS 30m x 0.25mm ID, 0.25 µm thickness.
4.1.5 Confirmatory Gas Chromatograph: Hewlett-Packard 5890.

4.1.6 Confirmatory Detector: Electron Capture Detector.
4.1.7 Confirmatory Column: HP-1701 30 m X 0.25 mm ID, 0.25 µm phase thickness.
4.1.8 Column Conditions:
4.1.8.1 Injector Temp: 220°C
4.1.8.2 Detector Temp: 300°C
4.1.8.3 Initial Temp: 80°C
4.1.8.4 Initial Hold: 0.25 min.
4.1.8.5 Rate: 20°C/min.
4.1.8.6 Temp: 150°C
4.1.8.7 Hold Time: 0 min
4.1.8.8 Rate: 4°C/min
4.1.8.9 Temp: 235°C
4.1.8.10 Hold Time: 0 min
4.1.8.11 Rate: 20°C/min
4.1.8.12 Final Temp: 270°C
4.1.8.13 Final Hold: 3.25 min
4.1.8.14 He Carrier head pressure set to 160kPa.
4.1.8.15 N_2 Make-up set to 60 mL/min.

5.0 REAGENTS

5.1 Reagent grade or pesticide grade chemicals are used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
5.2 Solvents used in the extraction and cleanup procedures and for standards include n-hexane, methylene chloride, MtBE, and acetone.
5.3 Organic-free reagent water
5.4 Stock standard solutions – Stock standards are purchased from a certified manufacturer. Solutions are stored in sealed vials, protected from light, at 4°C. *Manufacturer expiration dates are observed.* Stock standards can be replaced sooner if comparison with laboratory fortified blanks, or QC samples indicate a problem. See standard log for recipes.

NOTE: All other standard solutions are replaced after six months or sooner if routine QC indicates a problem.

5.5 Calibration standards are prepared at five different concentrations for single component analytes through dilution of the stock standards with hexane containing 10% acetone. The concentrations correspond to the expected range of concentrations found in real samples and bracket the linear range of the detector. Solutions are stored in sealed vials, protected from light, at 4°C. Solutions are replaced after 6 months or sooner if QC samples indicate a problem. See standard log for recipes. Calibration standards are made from a source separate from the LFB and LFSM. The lowest calibration standard is below the reporting limit but above the method detection limit. The rest of the calibrators bracket the expected working range of the samples. Calibration concentrations are 5ppb, 10ppb, 25ppb, 50ppb, 75ppb, 100ppb for single peak compounds. For multipeak compounds a single point

preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements

7.2.1 If a sample is of biological origin, or contains high molecular weight materials, the use of Method 3640 (GPC cleanup - pesticide option) is recommended. Frequently, one of the adsorption chromatographic cleanups (alumina, silica gel, or Florisil) may also be required following the GPC cleanup.

7.2.2 Method 3610 (alumina) may be used to remove phthalate esters.

7.2.3 Method 3620 (Florisil) may be used to separate organochlorine pesticides from aliphatic compounds, aromatics, and nitrogen-containing compounds.

7.2.4 Method 3630 (silica gel) may be used to separate single component organochlorine pesticides from some interferants.

7.2.5 Elemental sulfur, which may be present in certain sediments and industrial wastes, interferes with the electron capture gas chromatography of certain pesticides. Sulfur should be removed by the technique described in Method 3660.

7.3 Calibration

7.3.1 For single component analytes, a five point calibration is performed for a linear (first order) model, six point calibration for a quadratic (second order) model, and seven point for a polynomial (third order) model. A single point calibration near the mid-point of the expected calibration range is performed for each multi-component analyte *that is detected in the sample*. Each run begins with a low level calibration check for each multi-component analyte. This is intended to demonstrate that the pattern is recognizable, the analyst is familiar with the retention times on each column and that if any multi-component were present, it would be detected.

7.3.2 For calibration verification see section 7.4.2.

7.3.3 A 2 uL injection volume of each calibration standard is used.

7.3.4 Calibration factors - The calibration factor for each analyte at each concentration, the mean calibration factor, and the relative standard deviation (RSD) of the calibration factors, are calculated using the formula below for the calculation of response factors.

7.3.4.1 Calculate the calibration factor for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

7.3.4.2 Calculate the mean calibration factor for each analyte as:

$$\text{mean CF} = \overline{CF} = \frac{\Sigma^{n}_{i=1} CFi}{n}$$

where n is the number of standards analyzed.

7.3.4.3 Calculate the standard deviation (SD) and the RSD of the calibration factors for each analyte as:

$$SD = \left[\frac{\overline{n}_{i=1} (CFi - CF)^2}{n-1}\right]^{0.5}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

7.3.4.4 A linear curve must have a correlation coefficient, $r \geq 0.99$. A nonlinear curve must have a coefficient of determination, $r^2 > 0.99$. The analyst is responsible for determining the best curve fit (eg. 2nd or 3rd order polynomials).

7.3.5 Retention time windows - Absolute retention times are used for compound identification. Retention time windows are crucial to the identification of target compounds.

7.3.5.1 Before establishing the retention time windows, make sure the gas chromatographic system is operating within optimum conditions.

7.3.5.2 Retention time window studies are performed as stated in Method 8000 section 7.6. The experience of the analyst weighs heavily in the interpretation of chromatograms.

7.4 Gas chromatographic analysis of sample extracts

7.4.1 The same GC operating conditions used for the initial calibration is employed for samples analyses.

7.4.2 The calibration is verified at the beginning of each run by the analysis of a midpoint calibration check from a second source for all single-component analytes. A low level calibration check from a second source for each multi-component analyte is also injected, see section 7.3.1. Every 10 samples, and at the end of the run, analysts alternate the use of high and low concentration mixtures of single-component analytes for calibration verification. A *low level* calibration check for one multi-component analyte is injected at the end of the run. The

analyst alternates each day between PCB 1016/1260 mix, Toxaphene, and Chlordane.

7.4.2.1 The calibration factor for each single component analyte should not exceed a +/- 15 percent difference from the mean calibration factor calculated for the initial calibration. If a non-linear calibration model or a linear model not through the origin has been employed for the initial calibration, consult Sec. 7 of Method 8000 for the specifics of calibration verification.

$$\% \text{ Difference} = \frac{\overline{CF} - CF(V)}{\overline{CF}} \times 100$$

7.4.2.2 If this criterion is exceeded for any analyte, calculate the average percent difference across all analytes If the average of the responses is within the +/- 15% limit then the calibration has been verified. *We do not provide the data user with a list of those analytes exceeding the limit.* If the average of the responses is not within the +/- 15%, check the instrument operating conditions, if necessary, restore them to the original settings, and inject another aliquot of the calibration verification standard. If the average response still exceeds 15%, a new calibration is performed.

7.4.3 Each analyte in each standard must fall within its respective retention time window. If not, the gas chromatographic system must either be adjusted so that a second analysis of the standard does result in all analytes falling within their retention time windows, or a new initial calibration is performed and new retention time windows established.

7.4.4 Inject a 3 uL aliquot of the concentrated sample extract. Record the volume injected to the nearest 0.05 uL and the resulting peak size in area units.

7.4.5 Tentative identification of an analyte occurs when a peak from a sample extract falls within the absolute retention time window. All results are reported from the primary column and confirmed using the secondary column, unless analytical conditions and quality control samples indicate that the secondary column results are more accurate. See ESB SOP Q20 for details. The primary column for all of the analytes specified in section 1.1 and 1.6 is column A except for 4,4'-DDE and Dieldrin which coelude on column A. Column B is the primary column for 4,4'-DDE and Dieldrin. If in any case the secondary column is used for quantification, the analyst will document her reasoning for doing so.

7.4.6 When using the external calibration procedure, determine the quantity of each component peak in the sample chromatogram that corresponds to the compounds used for calibration purposes, as follows. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined.

7.4.6.1 For aqueous samples

$$\text{Concentration (ug/L)} = \frac{(Ax)(Vt)(D)}{(\overline{CF})(Vi)(Vs)}$$

where:

Ax = Area (or height) of the peak for the analyte in the sample.
Vt = Total volume of the concentrated extract (uL).
D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

$\overline{CF}$ = Mean calibration factor from the initial calibration (area/ng).
Vi = Volume of the extract injected (uL). The injection volume for samples and calibration standards is the same. For purge-and-trap analysis, Vi is not applicable and therefore is set at 1.
Vs = Volume of the aqueous sample extracted in mL. If units of liters are used for this term, multiply the results by 1000.

Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to ug/L.

7.4.6.2 For non-aqueous samples

$$\text{Concentration (ug/kg)} = \frac{(Ax)(Vt)(D)}{(\overline{CF})(Vi)(Ws)}$$

where Ax, Vt, D, CF, and Vi are the same as for aqueous samples, and

Ws = Weight of sample extracted (g). The wet weight or dry weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to ug/kg.

7.4.6.3 If the responses exceed the calibration range of the system, dilute the extract and reanalyze. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

7.4.7 Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard(s), or calibration standards interspersed within the samples. The results from these bracketing standards must meet the calibration

verification criteria. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent misquantitations and possible false negative results, and re-injection of the sample extracts may be required. However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e., > 15%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the verification standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits was detected in a sample extract, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 15% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present (i.e., a false negative result).

7.4.8 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements.

7.4.9 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

7.4.10 Identification of mixtures (i.e. Chlordane and Toxaphene) is based on the characteristic "fingerprint" retention time and shape of the indicator peak(s); and quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peak(s) of the same retention time and shape generated using either internal or external calibration procedures.

7.4.11 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. Rerun the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix..

7.4.11.1 DDT and endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated with high boiling residue from sample injection or when the injector contains metal fittings. Check for degradation problems by injecting a standard containing only 4,4'-DDT and endrin. Presence of 4,4'-DDE, 4,4'-DDD, endrin ketone or endrin indicates breakdown. If degradation of either DDT or endrin exceeds 15%, take corrective action before proceeding with calibration.

Calculate percent breakdown as follows:

$$\%\ \text{breakdown of} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \times 100$$

$$\%\ \text{breakdown of endrin} = \frac{\text{sum of degradation peak areas(aldehyde+ketone)}}{\text{sum of all peak areas(endrin+aldehyde+ketone)}} \times 100$$

7.4.11.2 The breakdown of DDT and endrin should be measured before samples are analyzed and at the beginning of each 12 hour shift. Injector maintenance and recalibration should be completed if the breakdown is greater than 15% for either compound.

7.5 Quantitation of multi-component analytes - Multi-component analytes present problems in measurement. See EPA Method 8081B 7.6 for suggestions for handling Toxaphene, Strobane, Chlordane, BHC, and DDT.

7.6 Suggested chromatographic system maintenance - When system performance does not meet the established QC requirements, corrective action is required, and may include one or more of the following.

7.6.1 Splitter connections - For dual-columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter. Break off the first few centimeters (up to 30 cm) of the injection port side of the column. Remove the columns and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

7.6.2 GC injector ports can be of critical concern, especially in the analysis of DDT and Endrin. Injectors that are contaminated, chemically active, or too hot can cause the degradation ("breakdown") of the analytes. Endrin and DDT breakdown to endrin aldehyde, endrin ketone, DDD, or DDE. When such breakdown is observed, clean and deactivate the injector port, break off at least 30 cm of the column and remount it. Check the injector temperature and lower it to 205-C, if required. Endrin and DDT breakdown are less of a problem when ambient on-column injectors are used.

7.6.3 Metal injector body - Turn off the oven and remove the analytical columns when the oven has cooled. Remove the glass injection port insert (instruments with on-column injection). Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

7.6.3.1 Place a beaker beneath the injector port inside the oven. Using a wash bottle, serially rinse the entire inside of the injector port with acetone and then toluene, catching the rinsate in the beaker

7.6.3.2 Prepare a solution of a deactivating agent (Sylon-CT or equivalent) following manufacturer's directions. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, rinse the injector body with toluene, methanol, acetone, then hexane. Reassemble the injector and replace the columns.

7.6.4 Column rinsing - The column should be rinsed with several column volumes of an appropriate solvent. Both polar and nonpolar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone. Methylene chloride is a good final rinse and in some cases may be the only solvent required. The column should then be filled with methylene chloride and allowed to stand flooded overnight to allow materials within the stationary phase to migrate into the solvent. The column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen.

8.0 QUALITY CONTROL
Note: See also ESB SOP Q01 for general QC requirements

8.1 Minimum quality control (QC) requirements are initial demonstration of laboratory capability, determination of surrogate compound recoveries in each sample and blank, analysis of laboratory reagent blanks,laboratory fortified samples, laboratory fortified blanks, and QC samples.

8.2 Laboratory Reagent Blanks -- Before processing any samples, the analyst must demonstrate that all glassware and reagent interferences are under control. Each time a set of samples is extracted or reagents are changed, a laboratory reagent blank (LRB) is analyzed. This blank is analyzed every 20 samples per matrix type. If within the retention time window of any analyte of interest the LRB produces a peak above the reporting limit, that would prevent the determination of that analyte determine the source of contamination and eliminate the interference before processing samples. Samples results reported must be accompanied with a note if the method blank exceeds a concentration greater than 1/10 of the measured concentration of the sample or is greater than 1/10 of the specified regulatory limit if known, however blank results below the MDL are considered to be ND and will not require a note.

8.3 INITIAL DEMONSTRATION OF CAPABILITY

8.3.1 Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made.

8.3.2 The quality control (QC) reference sample concentrate selected contained each analyte of interest at *20 µg/kg, except methoxychlor which was at 80µg/kg*. For analysis of Chlordane or Toxaphene, the QC reference sample concentrate was selected at the *reporting limit*.

8.3.3 Calculate the average recovery and the standard deviation of the recoveries of the analytes in each of the four QC reference samples. Refer to Sec. 8.0 of Method 8000 for procedures for evaluating method performance.

8.4 ASSESSING SURROGATE RECOVERY

8.4.1 When surrogate recovery from a sample or method blank is not within historically generated acceptance limits, check calculations to locate possible errors, fortifying solutions for degradation, contamination or other obvious abnormalities, and instrument performance. If those steps do not reveal the cause of the problem, reanalyze the extract.

8.4.2 If a blank extract reanalysis fails the recovery criterion, the problem must be identified and corrected before continuing.

8.4.3 If sample extract reanalysis meets the surrogate recovery criterion, report only data for the reanalyzed extract. If sample extract reanalysis continues to fail the surrogate recovery criterion, report all data for that sample as suspect.

8.5 ASSESSING LABORATORY PERFORMANCE - LABORATORY FORTIFIED BLANK

8.5.1 A laboratory fortified blank (LFB) and duplicate are analyzed with every twenty samples or one per sample set (all samples extracted within a 24-h period) whichever is greater. Acceptance ranges are generated from historical data and updated on a monthly basis. See QC Limit Summary for most current limits. If the recovery of any analyte falls outside the control limits that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

8.6 MDL studies are performed whenever there is a change in instrumentation, or a major modification to the analysis. Standards are spiked at the reporting limit and a minimum of seven replicates are analyzed. See QA Manual section 23 for calculation. Results must be below the reporting limit.

8.7 Twice a year, we participate in an external performance evaluation study. A QC sample from an outside source (ERA) is analyzed. This study serves as documentation of continuing proficiency.

8.8 ASSESSING METHOD PERFORMANCE - LABORATORY FORTIFIED SAMPLE MATRIX

8.8.1 The laboratory adds a known concentration to a minimum of 5% of the routine samples per matrix type or one sample concentration per set, whichever is greater.

8.8.2 Acceptance ranges are generated from historical data and updated on a monthly basis. See QC Limit Summary for most current limits.

8.8.3 If the recovery of any such analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control, the recovery problem encountered with the dosed sample is judged to be matrix related, not system related. The result for that analyte in the unfortified sample is labeled

suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.0 SAFETY

9.1 See SOP S02 – Compressed Gas Cylinder Handling
SOP S03 – Spill Control Policy

10.0 DEFINITIONS

10.1 See SOP Q15 – SOP Definitions

11.0 CORRECTIVE ACTION FOR OUT OF CONTROL / UNACCEPTABLE DATA

11.1 See SOP Q06 – Corrective Action

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 See SOP S05 – Neutralization Procedure for Acid and Alkaline Wastes
SOP S06 – Disposal of Chlorinated Solvents
SOP S07 – Pollution Prevention

13.0 METHOD PERFORMANCE

13.1 See Method 8081B Tables 9 – 16.

13.1.1 Our intralaboratory generated data is expected to achieve similar results. Refer to MDL studies and laboratory control charts maintained in the QC Office.

14.0 REVISION LOG

Rev.2.0 – 05/19/01: added Revision Log, reporting limits, eliminated sections: 1.7, 8.9, added sections 5.6.3, 5.7, 8.3.4, 14, edited sections 1.1, 3.7, 4.1.8, 5.4, 5.5, 6.2, 7.3.1, 7.3.4, 7.4.2, 7.4.5, 7.4.12, 8.3.2, 8.4.1, 8.5.1

Rev 2.1 – 09/20/01; added 8.0 note, edited sections: 1.1, 1.6, 5.5, 7.3.1, 7.3.4.3, 7.3.4.4, 7.3.5.2, 7.4.2, 7.4.5, 7.4.11, 7.4.12, 7.5, 8.2, 8.4, 8.5.1, 8.6, 8.8.1, 8.8.2.

15.0 REFERENCES

15.1 EPA 8081B Methods for the Chemical Analysis of Waters and Wastes.
15.2 (SW-846 Prop. Update IV, January 1998)

Note: All *italicized items* are an indication of a variation from the method.

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Reporting Limits	Liquids		Solids	
4,4'-DDD	1.1	ug/L	1.1	ug/kg
4,4'-DDE	0.4	ug/L	0.4	ug/kg
4,4'-DDT	1.2	ug/L	1.2	ug/kg
a-BHC	0.3	ug/L	0.3	ug/kg
Aldrin	0.4	ug/L	0.4	ug/kg
b-BHC	0.6	ug/L	0.6	ug/kg
BZ-198	1	ug/L	1	ug/kg
Chlordane	1.4	ug/L	1.4	ug/kg
Chloroneb		ug/L	1000	ug/kg
d-BHC	0.9	ug/L	0.9	ug/kg
Dibromochloropropane		ug/L	20	ug/kg
Dieldrin	0.2	ug/L	0.2	ug/kg
Endosulfan I	1.4	ug/L	1.4	ug/kg
Endosulfan II	0.4	ug/L	0.4	ug/kg
Endosulfan Sulfate	6.6	ug/L	6.6	ug/kg
Endrin	0.6	ug/L	0.6	ug/kg
Endrin Aldehyde	2.3	ug/L	2.3	ug/kg
Heptachlor	0.3	ug/L	0.3	ug/kg
Heptachlor Epoxide	8.3	ug/L	8.3	ug/kg
Hexachlorobenzene		ug/L	100	ug/kg
Lindane	0.4	ug/L	0.4	ug/kg
Methoxychlor	18	ug/L	18	ug/kg
Mirex		ug/L	33	ug/kg
Toxaphene	1	ug/L	1	ug/kg
Trifluralin		ug/L	1000	ug/kg

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QUALITY ASSURANCE PROJECT PLAN FOR NEW RIVER PATHOGENS TMDL IMPLEMENTATION

March 2003
First Draft 3/17/03

Prepared by and for
State of California Regional Water Quality Control Board Staff
Colorado River Basin

Quality Assurance Project Plan
For New River Pathogens TMDL Implementation

APPROVALS:

California Regional Water Quality Control Board

Jose Angel, PE, Supervising WRC Engineer — Date 4/24/03
Watershed Protection Division Chief
Quality Assurance Officer

Doug Wylie, PE, Senior WRC Engineer — Date 3/26/03
NPS/TMDL Implementation Unit Chief
Project Manager

Teresa Gonzales, Senior Environmental Scientist — Date 3/26/03
TMDL Development Unit Chief

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1. PROJECT MANAGEMENT

1.1 INTRODUCTION

This Quality Assurance Project Plan (QAPP) describes the quality assurance (QA) and quality control (QC) procedures associated with the monitoring of pathogen indicators in the New River. The activities are scheduled in response to certain requirements specified by the California Regional Water Quality Control Board, Colorado River Basin (Regional Board) Basin Plan (Basin Plan), which addresses the implementation section of the New River Pathogens Total Maximum Daily Load (TMDL). Regional Board staff are required to develop a New River Pathogens TMDL Implementation QAPP and conduct monitoring activities in accordance with the QAPP's requirements. "The objectives of the monitoring program shall include collection of water quality data for: assessment of water quality standards attainment, verification of pollution source allocations, calibration or modification of selected models (if any), evaluation of point and nonpoint source control implementation and effectiveness, evaluation of in-stream water quality, evaluation of temporal and spatial trends in water quality, and modification of the TMDL as necessary."

This QAPP follows the format that the United States Environmental Protection Agency (USEPA) established in its *Requirements for Quality Assurance Project Plans, EPA QA/R-5, 2001.* Further, it also complies with the QA/QC requirements specified in the *State Water Resources Control Board Quality Assurance Program Plan, June 2001.*

The Project Manager and the QA Officer/Division Chief may, upon mutual concurrence, request modification of this QAPP to achieve the objectives of the project. The process for QAPP modification consists of incorporating the necessary changes into the QAPP document, obtaining approval signatures, and distributing the revised document to project personnel.

1.2 DISTRIBUTION LIST

The following individuals have approving authority of this QAPP and subsequent revisions:

- Jose Angel, PE, Supervising WRCE, Watershed Protection Division Chief/ QA Officer
- Doug Wylie, PE, Sr. WRCE, NPS/TMDL Implementation Unit Chief, Project Manager
- Teresa Gonzales, Sr. ES, TMDL Development Unit Chief

1.3 PROJECT/TASK ORGANIZATION

Specific responsibilities of the Regional Board staff are outlined below. A project organization chart is provided as Appendix I.

Jose L. Angel, P.E., Supervising WRC Engineer, Watershed Protection Division Chief/QA Officer, (760) 776-8932

- Reviews and approves the QAPP and subsequent revisions.

- Ensures that the QAPP is implemented and followed to meet the project objectives.
- Oversees validation activities for field and lab data.

Doug Wylie, PE, Senior WRC Engineer, Project Manager, (760) 346-6585

- Reviews and approves the QAPP and subsequent revisions.
- Ensures that the QAPP is implemented and followed to meet the project objectives.
- Ensures that field personnel have appropriate training and certification for field activities.
- Reviews field reports.
- Ensures plans are implemented according to schedule.
- Prepares annual reports.
- Manages sampling data.
- Performs field sampling as necessary.
- Conducts Health and Safety briefing for field samplers prior to each sampling event.
- Coordinates field and laboratory activities.
- Reports project status to the QA Officer and Division Chief.
- Responsible for evaluating data to ensure TMDL compliance.

Teresa Gonzales, Senior Environmental Scientist, TMDL Development Unit Chief, (760) 776-8931

- Reviews and approves QAPP and subsequent revisions.

Maria de la Paz Carpio-Obeso, Ph.D., Environmental Scientist, Regional Board Lab Director, (760) 674-0803

- Responsible for Regional Board Laboratory.

Phan Le, WRC Engineer, Deputy Lab Director, (760) 346-7491

- Responsible for calibration of equipment prior to sampling event.
- Responsible for performing water quality analysis as required.
- Assists with sampling activities as required.

Jeff Allred, WRC Engineer, Lead Field Sampler, (760) 776-8946

- Coordinates field activities and ensures they are consistent with the QAPP
- Conducts assignment briefing for field samplers prior to each sampling event.
- Ensures that sample containers have no defects and have been prepared properly.
- Conducts sampling activities as required.
- Prepares a summary report for each sampling event.
- Coordinates delivery of samples to the laboratory.
- Coordinates decontamination of equipment.

Field Samplers

- Assist with sampling activities as required.

1.4 PROBLEM DEFINITION/BACKGROUND

Pursuant to Section 303(d) of the Clean Water Act (CWA), the Regional Board has developed a Pathogens TMDL for the New River. The TMDL was developed because pathogens concentrations in the river violate the water quality standards (WQSs) established by the Regional Board to protect the beneficial uses of the river. The Implementation Plan of the TMDL identifies the monitoring and tracking of the pollutant of concern to determine compliance with the TMDL.

The monitoring and tracking program associated with the New River Pathogens TMDL requires water quality monitoring of pathogen-indicator organisms (i.e. bacteria) at "a sufficient number of sampling locations and sampling points per location along the New River and major drain tributaries to the river." Data collection activities outlined in this QAPP are being undertaken to execute the implementation section of the TMDL.

1.5 PROJECT/TASK DESCRIPTION

The overall objective of this project is to obtain valid data of known and documented quality, which can be utilized in determining the compliance with the water quality objectives as set forth in the New River Pathogens TMDL. Specifically, the purpose of this project is to:

1. Collect representative water samples for the pathogen-indicator organisms fecal coliform, E. coli, fecal streptococci, and enterococci from the New River at the sampling locations identified in Table No. 2, below; and
2. Record field measurements (physical parameters) including pH, temperature, dissolved oxygen (DO), and electrical conductivity (EC).

This project consists of monthly sampling events, in which water samples will be collected and field measurements taken at four sampling stations. Sampling events may include sampling at locations other than the four sampling stationed predetermined on the New River or within drains as necessary to pinpoint sources of contamination (i.e. drainsheds, drains, or individual fields). Predetermined sampling locations are described in detail in Section 2.1, Sampling Process Design.

1.6 DATA QUALITY OBJECTIVES

Valid data of known and documented quality are needed to meet the objectives of this project. Therefore, for the critical measurements of this project (fecal coliform, E. coli, fecal streptococci, and enterococci), only data that meet QA objectives will be considered valid. The specific data quality objectives of this project are:

- Samples collected should be of sufficient quality to be used for laboratory analysis.
- Laboratory analyses for the pathogen indicators fecal coliform, E. coli, fecal streptococci, and enterococci must yield results that are of sufficient quality to be used in the implementation of the New River Pathogens TMDL.
- The data generated in this project should be of sufficient quality to be utilized, along with

data from future sampling projects, in the determination of the contributions of pathogen indicators from each source of the New River, and the concentrations within the New River, at the time of sampling.

Table No. 1, below, summarizes the precision, accuracy, and completeness criteria.

Table 1: QA Objectives for Laboratory Data

Analyte	Matrix	Units	Precision (RPD)	Accuracy (% Recovery)	Completeness (% Comp)
Fecal Coliform	Water	MPN/100mL	Must fall within 95% confidence interval[1]	Not Applicable	95
E. Coli	Water	MPN/100mL	Must fall within 95% confidence interval[1]	Not Applicable	95
Fecal Streptococci	Water	MPN/100mL	Must fall within 95% confidence interval[1]	Not Applicable	95
Fecal Enterococci	Water	MPN/100mL	Must fall within 95% confidence interval[1]	Not Applicable	95
Field Measurement					
Temperature	Water	^{0}C	0.01	+/- 0.15	N/A
pH	Water	pH units	0.01	+/-0.2	N/A
DO	Water	Mg/L	N/A	+/- 2% of reading or 0.2 mg/L[(4)]	N/A
EC	Water	µmhos/cm	N/A	+/- 0.5% of reading + 0.001 mS/cm	N/A

1 Listed in Table 9221.IV of Clesceri et. Al 1995

1.6.1 Data Quality Indicators (Acceptance Criteria)

The following data quality indicators will be utilized to assess whether data generated are useable and meet the data quality objectives stated above:

1.6.1.1 Precision

Precision is defined as the degree of refinement of a measurement. Precision of the data generated will be assessed as the relative percent difference (RPD) for field and laboratory duplicates.

$$RPD = \frac{ABS(C_1 - C_2)*100}{\left(\frac{C_1 + C_2}{2}\right)}$$

RPD = Relative Percent Difference
D_1 = Results for sample 1
D_2 = Results for sample 2
ABS = Absolute value

1.6.1.2 Accuracy

Accuracy is not applicable for this monitoring program.

1.6.1.3 Completeness

Completeness is defined as a measure of how many collected samples actually yield valid and useable data. A minimum of 95% completeness is expected for this project. This will result in a sufficient amount of data to meet the previously stated requirements.

$$\%C = 100 * \frac{V}{T}$$

%C = Percent complete
V = Total number of measurements or laboratory results judged valid
T = Total number of measurements or laboratory results

1.7 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

The Project Manager will ensure that all of the field samplers have valid and current training for their field activities, as required by OSHA regulations. Currently, all sampling personnel identified in the Project/Task Organization section of this QAPP have completed the required OSHA training for the sampling activities described herein. There are no other specialized training/certification requirements needed to perform the Project's objectives.

1.8 DOCUMENTATION AND RECORDS

1.8.1 Project Working File

The Project Manager will establish and maintain a Project Working File for maintaining sampling records. The Lead Field Sampler and QA Officer will ensure that all received/generated data (e.g., field notes, chain-of-custody forms, lab analyses) are delivered to the Project Manager. The file will contain, but need not be limited to:

- Field data sheets
- Calibration logs
- Laboratory reports
- Data reports summarizing field activity and quality control for each sampling event
- Data spreadsheets
- Correspondence
- Quality control reports
- Validation reports
- Sampling Event Summary Reports
- Annual Reports

1.8.2 Field Datasheets

The Lead Field Sampler will use field datasheets to document field activities and data for each sampling event. Each field datasheet will be dated and signed by a sampling team member at each sampling station. At the time of sampling, the following information will be recorded in the field datasheet:

- Weather observations
- Sampling station latitude and longitude, using a global positioning system (GPS) unit, if not previously recorded
- Sample identification code and sampling method for all samples taken
- In-situ measurements for temperature, pH, DO, and EC
- Field turbidity measurement
- Sample identification code, and time and location of preparation, for all quality control samples prepared in the field
- Any deviations from the QAPP
- Any noteworthy observations

1.8.3 Sampling Event Summary Report

The Lead Field Sampler will prepare a sampling event summary report for each sampling event, which will be submitted to the Project Manager. The reports will be due within 7 days following each sampling event. The reports will summarize:

- Any deviations from the QAPP
- Any problems encountered and how the problems were addressed
- Recommendations as appropriate

1.8.4 Quality Control Log Notebook

The QA Officer will use a bound quality control log notebook with pre-numbered pages to document the quality control (QC) samples submitted to the laboratory and the analysis results. For each QC sample, the quality control log notebook will contain the:

- Sample identification code
- Supplier of the QC sample
- Value reported by the supplier
- Date of preparation and submission
- Name and signature of the person submitting the QC sample
- Laboratory performing the analysis
- Analysis method
- Reported value from the laboratory

1.8.5 Calibration Log Notebook

The Deputy Lab Director will use a calibration log notebook to document calibration activities performed on sampling equipment prior to each sampling event. The calibration log notebook will be bound and will have pre-numbered pages. For each calibration event, the calibration log notebook will contain:

- Date and time of calibration
- Person(s) performing the calibration
- Signature of one of the persons performing the calibration
- All standard solutions used in calibration, including the source and date of preparation of the standard solution
- The initial reading of the YSI 6600 multiprobe sonde when tested against each standard solution, and the temperature of each standard solution at the time of calibration
- Any problems encountered and how the problems were addressed

1.8.6 Laboratory Analytical Summaries

The Division Chief will request that the laboratory prepare and submit to the Project Manager a laboratory analytical summary for each sampling event upon completion of the laboratory's analysis of samples. This summary will include analytical results, analytical methods, problems encountered, QC results, and chain of custody forms.

1.8.7 Quality Assurance Reports

The QA Officer will prepare quality assurance reports, including a technical systems audit for each sampling event, performance evaluations of laboratories, and an annual data quality assessment. These reports are described in more detail in Section 4.1.

2. MEASUREMENT/DATA ACQUISITON

2.1 SAMPLING PROCESS DESIGN

In order to meet the overall objectives stated in Section 1.5 of this QAPP, this project was designed to estimate the pathogen concentration, as represented by the pathogen-indicator organisms fecal coliform, E. coli, fecal streptococci, and enterococci at several sampling stations in the New River. Additionally, to pinpoint specific sources, sampling locations may be added as necessary within the New River watershed. Because accurate patogens data are necessary for TMDL implementation, fecal coliform, E. coli, fecal streptococci, and enterococci are considered critical measurements for this project, while the other baseline parameters, temperature, EC, pH and DO, are considered non-critical measurements.

The New River sampling stations were selected to characterize the changes in water quality in each of three drainsheds in the U.S. and the contribution from Mexico. Sampling stations are located at the upstream and downstream ends of each drainshed.

At each of the locations listed in Table 2 below, water samples will be taken as described, and a YSI 6600 multi-parameter sonde will be used to collect in-stream measurements of temperature, DO, EC and pH. Field turbidity will be measured using a Hach 2100P portable turbidimeter.

Table 2: Monitoring Stations

Sampling Location	Alias	Description
NR-B	NR-0	Monitoring station for Mexico's pathogens contribution to the New River at the Boundary, to be located at the intersection of the New River and the International Boundary.
NR-EH	NR-1	Monitoring station for the Lower New River drainshed, to be located at the Evan Hewes Road Bridge and the New River.
NR-2	NR-2	Monitoring station for the Middle New River drainshed, to be located at Drop Structure #2 of the New River.
NR-O	NR-Outlet	Monitoring station for the Upper New River drainshed, to be located at the USGS sampling station downstream of Lack Road Bridge.

[1]A location may be changed provided the prescribed location is inaccessible or a hazardous, as documented by field observations.

Sampling station NR-2 listed in Table 2 is a drop structure. Because the water is well mixed as it cascades over this structure, a single grab sample is typically representative of the pathogen indicators concentration throughout the river at the given cross-section. The mixing effect of the structure also introduces oxygen into the water. Therefore, YSI readings should be taken on the upstream side of the structures. When samples are taken at alternative locations, which are not located at a drop structure and are greater than six (6) feet wide, three (3) sampling points (S1, S2, and S3) will be distributed along the cross-sectional area of the river/drain. The sampling points are to be spaced at approximately equal intervals from each other and from the edge of

the river/drain (i.e., at a distance equal to w/4, where "w" is the top width of the cross-sectional area). Figure No. 1, below, illustrates this. A sample composed of a grab sample from each cross-section will be obtained and homogenized using a churn splitter. The composite sample will be used for lab analyses.

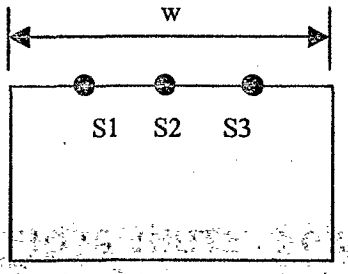


Figure 1: Sampling Points at Non-Well-Mixed Sampling Locations

For the drop structure location listed in Table 2 and for small drain sampling stations where there are small cross sections and well-mixed flow, only one grab sample at the center point (S2), if possible, will be taken to characterize fecal coliform, E. coli, fecal streptococci, and enterococci. Also, at sampling points along the river and the main drains where the depth is less than 2 feet, well mixed flow is assumed, and only grab samples at the center point (S2) will be taken.

For all sampling stations, YSI readings of EC, DO, pH and temperature will be recorded at the center sampling point (S2) if possible. Minimum sample collection frequency is noted in Table 3 below.

Table 3: Minimum Sample Frequency

Constituent	Sample Frequency
Fecal Coliform	Monthly
E. Coli	
Fecal Streptococci	
Fecal Enterococci	

2.2 SAMPLING METHODS REQUIREMENTS

Sampling methods include the collection of grab samples, as well as the acquisition of readings for water quality parameters using the YSI water quality sonde. A clean sample collection bottle will be used at each sampling station. Inaccuracies in the lab analyses due to sorption of analytes to the swing sample collection bottle, and churn splitter if appropriate, will be negated by rinsing each three times with native water prior to collection of the sample. The grab samples will be collected at approximately midstream (sampling point S2). Where hazardous conditions prevent midstream sampling, the grab sample will be collected at sampling location S1 or S3. Grab samples will be collected at approximately ½-foot below the water surface using a swing

sampler, and immediately transferred to a churn splitter. While churning the sample in the churn splitter, the sample will be distributed into the appropriately labeled sample bottle and placed into an ice chest with a sufficient quantity of wet ice.

The YSI 6600 multi-parameter water quality sonde will be used to collect field measurements for the following parameters: DO, pH, temperature, and EC at the center point at each sampling location from about 1-foot below the water surface. Where hazardous conditions prevent midstream sampling, the YSI measurements will be taken at sampling location S1 or S3. After at least two minutes or when the readings have reached equilibrium, the values for these parameters will be manually recorded in the field data sheet.

2.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Sample-holding times will be adhered to, as prescribed by USEPA and 40 CFR 136. Specifically, the required preservation techniques and holding times for all of the constituents which the laboratory will be analizing are listed in Table 4, below.

Table 4: Required Containers, Preservatives, Techniques, and Holding Times

Constituent	Container	Preservation Technique	Holding Time
Fecal Coliform	100-mL plastic	Cool below 4 °C; Sodium Thiosulfate Preservative (to neutralize any chlorine present)	6 hours
E. Coli			
Fecal Streptococci			
Fecal Enterococci			

Each sample container will be labeled with a unique sample identification code. All samples (including QC samples) for laboratory analyses will immediately be stored in an ice chest, and will remain in the custody of Regional Board staff until the samples are delivered to the laboratory. The ice chest will contain sufficient ice to maintain the samples at a temperature below 4°C at all times until they are relinquished to the lab staff. All samples will be delivered with chain of custody forms. A sample chain of custody form to be used for this project is included in Appendix II. Any violation of holding times or other sample handling and custody requirements will be documented in the quality control records and reported to the Project Manager and the QA Officer. Any violations thereof will be taken into account when evaluating the data.

2.4 ANALYTICAL METHODS REQUIREMENTS

As prescribed by the State Water Resources Control Board's "*Quality Assurance Program Plan*", each analytical laboratory used for sample analysis must have a written QA Laboratory Manual describing the analytical method requirements. The lab will use USEPA approved methods as outlined in Table No. 5.

Table 5: Sampling Constituents and Methods

Constituent	USEPA Method	Reporting Limit	Units
Fecal Coliform	Standard Method 9221E1	2MPN/100 mL	MPN/100 mL
E. Coli	Standard Method 9221F		
Fecal Streptococci	Standard Method 9230B		
Fecal Enterococci			

2.5 QUALITY CONTROL REQUIREMENTS

In order to assess whether the data quality requirements of this project are being met, a number of quality control checks will be implemented. All QC samples will be placed in an ice chest, and kept at 4 °C, for transport to the lab. Specifically:

- Field blank samples will be collected in the field by dispensing deionized water into the appropriately labeled sample container. The reported value is used to check for laboratory accuracy. One field blank will be collected and submitted for analysis for each constituent, for each day of sampling.

- Field duplicate samples will be prepared from a grab sample of the water being sampled. A grab sample will be collected as described above and split using a churn splitter, into the appropriately labeled containers. Ten percent of the samples collected will be field duplicates, with a minimum of one set of field duplicate samples for each analyte collected per day of sampling.

- To ensure preservation requirements are met, a random sample will be chosen by the laboratory from each ice chest for temperature measurement.

QC samples will be submitted to the lab along with the "real" surface water samples being submitted (i.e., the laboratory will not be informed in any way as to which samples are control samples and which samples are from the aforementioned surface waters). A summary of Quality Control Sample Requirements is located in Table 6.

Table 6: Quality Control Sample Requirements

Quality Control Samples	Number of Samples	Frequency

Duplicate Samples (All parameters)	10% (1 minimum)	Per day/event
Field Blanks (All parameters)	1	Per day/event
Temperature blank[1]	1	Per ice chest

[1]Temperature blank may be omitted if temperature is read from a random sample bottle using an infrared thermometer.

2.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, & MAINTENANCE REQUIREMENTS

All Regional Board staff participating in the project will be trained in the operation, calibration, and maintenance of the field instruments. The manufacturer's instruction manuals will be readily available for field personnel. The instruments will be maintained and calibrated in accordance with the manufacturer's instructions and recommendations. Calibration, inspection, and maintenance of field instruments are performed by laboratory personnel prior to all sampling events.

2.7 INSTRUMENT CALIBRATION AND FREQUENCY

The YSI 6600 sonde will be calibrated in the Regional Board laboratory prior to each sampling event. Dissolved oxygen will be calibrated prior to each sample collection, using ambient air. Field personnel will perform pre- and post- sampling calibration check for dissolved oxygen at each sampling site. This process involves checking and recording the DO output. If it is within 2% of saturation, recalibration of DO is required. The pre and post calibration check data (DO output) will be recorded on the field datasheet. If recalibration is required, calibration data will be recorded on a calibration log sheet. A post-calibration for all parameters will be performed when the sonde is returned to the office, or as soon as practical. Results of calibration measurements will be documented in the field log notebook and submitted to the QA Officer. Table 7, below illustrates the YSI 6600 sonde specifications.

Table 7: Specifications for the YSI 6600 Sonde & Hach 2100P Turbidimeter.

Parameter	Operating Range	Accuracy	Resolution	Calibration Standard
pH	0 to 14 units	± 0.2 units	0.01 units	2-pt, with pH buffered solutions
Temperature	– 5 to 45 °C	± 0.15 °C	0.01 °C	not required
DO	0 to 20 mg/L	± 0.2 mg/L	0.01 mg/L	saturated air
EC	0 to 100 mS/cm	± 1% of range	4 digits	KCl

[1]Whichever is greater (with Gelex standards).

2.8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES/CONSUMABLES

The Lead Field Sampler will ensure that the sample bottles have no defects, and that all sample bottles have been prepared properly.

No other special requirements are needed.

2.9 DATA ACQUISITION REQUIREMENTS (NON-DIRECT METHODS)

Only data collected from this Project and historic data from the TMDL development program, which have already passed QC criteria, will be used. No data will be used from other sources unless the data also meet the QA requirements set herein.

2.10 DATA MANAGEMENT

The Project Manager will maintain field datasheets and chain of custody forms in the project file. Field measurement data will be uploaded from the YSI using ECOLAB software, and analyses results will be obtained in electronic form from the lab. The Project Manager will submit all data in MS Excel format to the QA Officer. After verification and approval by the QA Officer, the Project Manager will download the data into the project database (MS Excel format) and store it on the Regional Board's local area network (LAN). Statistical analyses of the data from each sampling point will be employed and used in determining compliance per the Implementation Section of the TMDL.

2.11 ASSESSMENT AND RESPONSE ACTIONS

Surveillance of the records and overall status of the project will be conducted by the QA Officer to ensure that all of the requirements of the QAPP are being met. Surveillance will be conducted after each sampling event, after all laboratory results have been received for that sampling event. A technical systems audit will also be performed by the QA Officer, as discussed in Section 4.1.1. Also, an annual data quality assessment of the applicability of the data will be performed to assess the handling of all data and to correct any errors found in the project database (see Section 4.1.3).

2.12 REPORTS TO MANAGEMENT

The Project Manager will prepare quarterly and annual project reports. The quarterly project reports will include a summary of the activities performed, the resulting data, and the quality of the resulting data, any problems encountered and their solutions and will identify any samples that indicate violations of Water Quality Standards. The annual project reports will include a statistical analysis of the results indicating drainshed loading, any decrease or increase in loading at the drainshed boundaries, drainsheds which are out of compliance, recommendations for TMDL modification, and the relationship of TSS to turbidity.

3. DATA VALIDATION AND USABILITY

Data objectives for this project do not require a full, formal, and independent data validation. The data has no legal requirement for independent validation. Although the data are considered legally defensible as presented herein, all records will be available for independent evaluation should the need arise at a later date.

The QA Officer will be responsible for validating the project's data to ensure that QA guidelines have been followed, by performing:

The QA Officer will ensure that QA guidelines were followed, by performing:

(a) a Technical Systems Audit for each sampling event, after all laboratory results are received. Data will be validated if collected and analyzed in conformance to the QAPP. The review will take into account field notes, field datasheets, chain-of-custody forms, laboratory analysis forms, and calibration assessment (determines potential error in field measurements). Documentation of results will occur within 15 days, and will describe data reviewed, review criteria, and data usability.

(b) Performance Evaluations of laboratories, through the use of quality control samples, namely field blank and field duplicate samples.

(c) a final Audit of Data Quality, which takes into account all Technical Systems Audits, and includes verification of proper calibration of the YSI 6600 multiprobe sonde and the results of laboratory QC samples. Laboratory results will be validated for precision, accuracy, and completeness.

(d) a Data Quality Assessment, in which statistical tools determine whether data met all Data Quality Objectives, whether the total error in the data is tolerable, and whether significant departures from the QAPP reduce data set completeness (and thus reduce data set usability for drawing conclusions).

Significant departures from the QAPP will be noted in these reports, and resulting data will not be validated (and thus will be excluded from the data set). Unacceptable departures include, but are not limited to:

- cross-contamination
- lack of critical sample collection information
- violation of sample holding times and temperatures

Regional Board staff will discuss missing analysis data with the laboratory that submitted the data. All missing data will be designated with "NR" (meaning "Not Reported").

4.1 CONTAMINATION CONTAINMENT ZONES

The contaminated areas for this Project consist of and cover the entire waterways for the aforementioned waters, their banks, and the area within 2 feet of the banks. Decontamination zones will be set at least 10 feet away from the banks of the surface waters. The decontamination zone will be used for personnel decontamination and will include wash water, soap, paper towels, and trash bags. All contaminated solid waste material will be placed in trash bags for proper disposal. Only biodegradable antibacterial soap will be used at the site. Wash water runoff will be contained and disposed of in the surface waters. The Clean area will be set at least 20 feet away from the banks of the surface waters.

4.2 PERSONAL PROTECTIVE EQUIPMENT

The general concerns at the sampling sites are the potential exposure to toxicants and pathogens present in the waters being sampled, risk of sunburn, excessive heat exposure, insect bite, and possibly snakebites. In addition, the sampling crew should be aware of the risk of falling into the waterways. No less than two experienced samplers will be out in the field at one time. (The sampling crew will also have a functional cellular phone in one of the vehicles).

- Any member of the sampling team has the authority to stop the sampling event when he/she determines that conditions at the site (e.g., rain, dust, local emergency, etc.) preclude safe sampling. A Hazard Evaluation Plan (HEP) will be done for each day of sampling. The lead field sampler will be responsible for preparing the HEP.

- To reduce the risk of exposure while collecting/transporting samples, Latex Examination Gloves must be worn. The Contaminated Zone must not be entered without the aforementioned Personal Protective Equipment (PPE).

- To reduce the risk of heat exposure and sunburn, samplers should wear sunscreen and carry in their vehicle cold drinking water. If any of the samplers begin to experience symptoms of heat exhaustion, such as cramps or dizziness, he or she will immediately be removed from the sun and given plenty of cool liquids. If these symptoms persist, he or she will be taken to the nearest hospital.

- Extra caution should be used when working near or around the drains to reduce the risk of potentially falling in.

- To reduce the risk of insect bites, samplers should use insect repellent.

- To reduce the possibility of snakebite, samplers will check areas for snakes prior to entering the area. If snakebite occurs, ice will be placed on the bite. The sampler will be immediately transported to the nearest medical facility.

4.3 PERSONNEL DECONTAMINATION PROCEDURES

The Clean Zone must not be entered with contaminated PPE. All team members coming out of the Contaminated Zones must immediately proceed to the Decontamination Zones and use the following decontamination procedures before proceeding to Clean Zone:

1. Remove latex gloves carefully to avoid contact with bare skin and dispose of them in the trash bag. Thoroughly wash hands with antibacterial soap; and

2. Dispose of wash water into surface water just sampled.

Note: everything that is touched (pens, pencils, rinse water bottles, probes, etc.) with dirty gloves could be contaminated. Avoid touching these items with bare skin.

4.3.1 Emergency Numbers and Facilities

All sampling personnel will have access to a cellular phone to call 911 in case of an emergency. The hospital nearest the sampling locations are listed in Table 8 below:

Table 8: Nearest Hospitals to Sampling Locations

Sampling Location	Medical Facility	Address	Phone
NR-B	EL Centro Regional Medical Center	Imperial and Ross Ave., El Centro	(760) 339-7100
NR-EH	EL Centro Regional Medical Center	Imperial and Ross Ave., El Centro	(760) 339-7100
NR-D2	Pioneers Memorial Hospital	207 West Legion, Brawley	(760) 351-3333
AR-0	Pioneers Memorial Hospital	207 West Legion, Brawley	(760) 351-3333

In case of an emergency, sampling personnel should also contact the Regional Board Safety Officer, Doug Wylie, as soon as practical at 760-346-6585 or 760-341-7491.

4.3.2 After Sampling

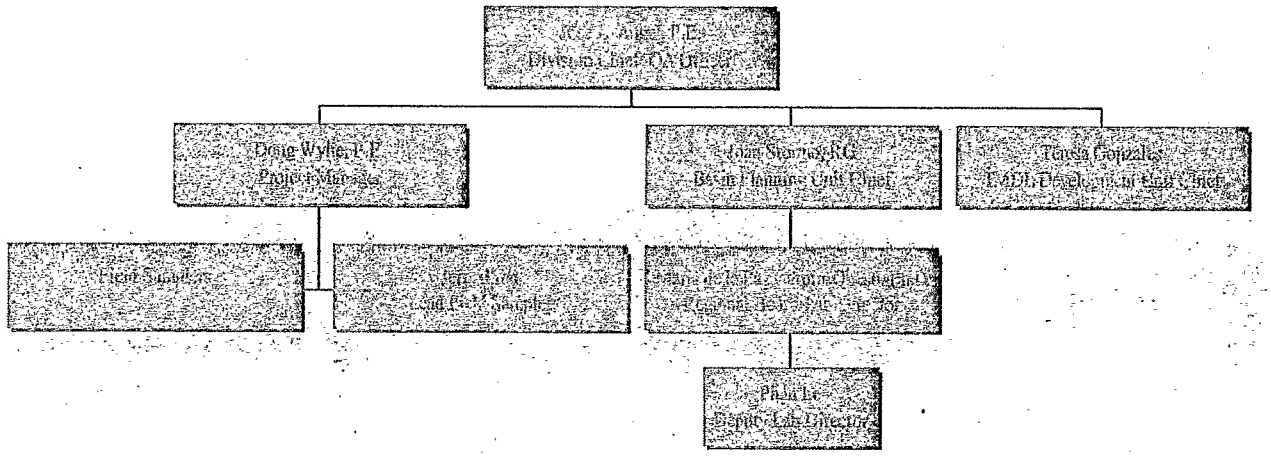
Place samples into Regional Board lab refrigerator or keep in an ice chest filled with wet ice; keep water drained from ice chests to avoid soaking container labels. Make copies of field notes and put original in the project working file. Contaminated equipment should be packed in designated containers for transport to the Regional Board office. Decontaminate and properly clean ALL items, which were exposed in the field in accordance with USGS National Field Manual for the Collection of Water-Quality Data, Chapter A3. Cleaning of Equipment for Water Sampling (See Attachment IV).

5. REFERENCES

U.S. Environmental Protection Agency. 2001. EPA Requirements for Quality Assurance Project Plans, EPA QA/R5. EPA Publication number 240/B-01/003. U.S. Environmental Protection Agency, Washington, D.C.

State Water Resources Control Board (State of California), 2001. Quality Assurance Program Plan.

APPENDIX I, PROJECT ORGANIZATION CHART



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Appendix II, Sample Chain of Custody Form

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E.S. Babcock & Sons, Inc.
6100 Quail Valley Court Riverside, CA 92507
(909) 653-3351 • FAX (909) 653-1662

Chain of Custody & Sample Information Record

Client: Contact: Phone No.

Project Name: _____

Project Location:

Turn Around Time: Routine 3-5 Days 48 Hours 24 Hours *(Rushes Require Approval, Additional Charges May Apply)*

Sampler Information

Name: _____

Employer: _____

Signature: _____

ESB #	Sample ID	Date	Time	# of Containers & Preservatives: Unpreserved	H_2SO_4	HCl	HNO_3	$Na_2S_2O_3$	NaOH			Total # of Containers	Analysis Requested	Matrix	Notes
														DW = Drinking Water WW = Wastewater GW = Groundwater S = Soil SG = Sludge L = Liquid M = Miscellaneous	

Relinquished By (sign)	Print Name / Company	Date / Time	Received By (Sign)	Print Name / Company

(For Lab Use Only) *Sample Integrity Upon Receipt*

Sample(s) Submitted on Ice? Yes No Temperature

Custody Seal(s) Intact? Yes No N/A ºC

Sample(s) Intact? Yes No

Lab No. _____

Page _____ of _____

Appendix II, Sample Chain of Custody Form

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APPENDIX IV, BASIN PLAN AMENDMENT

Terry Tamminen
Secretary for Environmental Protection

State Water Resources Control Board

Division of Water Quality

Arthur G. Baggett Jr., Chair
1001 I Street • Sacramento, California 95814 • (916) 341-5455
Mailing Address: P.O. Box 100 • Sacramento, California • 95812-0100
Fax (916) 341-5584 • http://www.swrcb.ca.gov

Arnold Schwarzenegger
Governor

TELECOPY TRANSMITTAL SHEET

Date: 7/16/04

To: Amy King Fax: 619 232 2392

Phone: 619 525 7017

From: Melissa Phone: 916 341-527

Number of pages being transmitted (including this transmittal sheet): 16

If you have difficulties in receiving all pages, please telephone transmitter: _____

Message: Here are the SOA's from Star Charter (LTOO).

IID-421 (R16-87)

IMPERIAL IRRIGATION DISTRICT
P.O. Box 937 - 333 East Barioni Boulevard
Imperial, California 92251
(760)339-9220 General Information

LETTER OF TRANSMITTAL

Date: July 7, 2004	Job No.
Attn:	
RE: SOPs for IID water monitoring data	

TO **Melenee Emanuel**

State Water Resources Control Board
Division of Water Quality, Monitoring
1001 I Street
P.O. Box 944213
Sacramento, CA 95812

WE ARE SENDING YOU ☒ Attached ☐ Under separate cover via _____ the following items:

☐ Book ☐ CD Disk ☐ 3.5" Floppy Disk ☐ Samples ☐ Specifications
☐ Copy of letter ☐ Zip Disk ☐ Data Pages ☒ Reports ☐ Drawings

			DESCRIPTION
1	copy		Monthly anion/cation water monitoring Standard Operating Procedure (New River, Alamo River, All American Canal)
1	copy		Monthly anion/cation water monitoring Standard Operating Procedure (Salton Sea)

THESE ARE TRANSMITTED as checked below:

☐ For Approval ☐ Approved as Submitted ☐ Resubmit _____ Copies for approval
☐ For your use ☐ Approved as Noted ☐ Submit _____ Copies for distribution
☒ As requested ☐ Returned for corrections ☐ Return _____ Corrected prints
☐ For review and comment ☐ _____
☐ FOR BIDS DUE _____ 19 ____ ☐ PRINTS RETURNED AFTER LOAN TO US

REMARKS The attached SOPs are being submitted as supplementary information for water monitoring data submitted on 6/10/04. An SOP for IID's monthly coliform samples is not available.

COPY TO _____

SIGNED: Steve Charlton
Steve Charlton
Resources Planning & Management

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

Procedure: DCO-SILTTDS-01

Date Created/ Revision: 7/26/00, 3/6/01

Printed: 3/6/01 - 12:57

Associated Procedures: LAB-TST-01

Object: To collect water samples along the All American Canal using the DH-59 Sampler and the Grab method.

Schedule: Monthly

Personnel: Two (2) Data Technicians

Equipment:
DH-59 water sampler
Cable and reel
Life jackets
Work gloves
Sample containers (18 one-pint bottles; three one-quart and four-½ gallon jars)
Basket for Grab samples
Paper caps for pint bottles
Thermometer with 0°F to 120°F range
Pencil for labeling and noting observations
Heavy duty string or cord, 20-foot minimum length
Timepiece
IID keys
IID-430A (R3 12-70) Water and silt samples form (7 minimum)
IID Log book

Sites:
All American Canal (Coor. N32.70599 W114.96191) – approximately 6500 ft. downstream of Drop 1. Take Gordon's Well turn-off of I-8, 500' west along AAC bank, see *Fig.2.*
East Highline Canal (Coor. N32.70390 W115.28436) – approximately 2000 ft. downstream of Heading on west bank, see *Fig.3.*
Alamo River Inlet (Coor. N32.67455 W115.36996) – outlet headwall of river crossing at All American Canal, Grab Sample only, see *Fig.4.*
Central Main Canal (Coor. N32.69530 W115.46595) – approximately 4500 ft. downstream of Heading, near Acacia Heading off Bowker Road, see *Fig.5.*
Westside Main Canal (Coor. N32.67917 W115.67744) – approximately 500 ft. downstream of Hwy 98 crossing, see *Fig.6.*
Alamo River Outlet (Coor. N33.19865 W115.59621) – approximately 400 ft. upstream of intersection at Garst Rd. and Alamo River, Grab Sample only, see *Fig.7.*
New River Outlet (Coor. N33.10471 W115.66434) – approximately 4500 ft. west of the intersection at Lack Rd. and Vail Canal, Grab Sample only, see *Fig.8.*

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

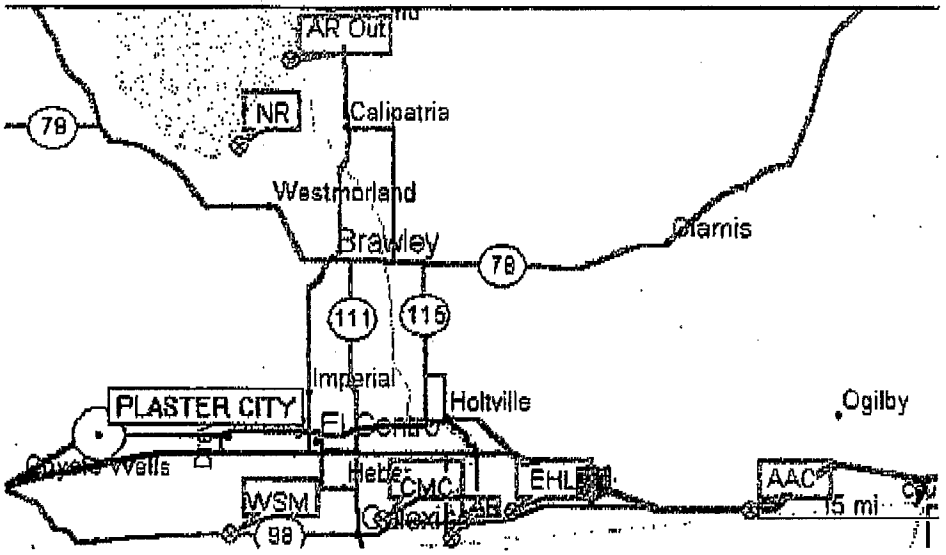


Fig. 1 – General location of water sampling sites: Alamo River Outlet (AR Out), New River Outlet (NR), Westside Main (WSM), Central Main (CMC), Alamo River Inlet (AR), East Highline (EHL), and All American Canal @ Drop 1 (AAC).

Following is a pictorial of all sites, refer to *Sites* on page 1.

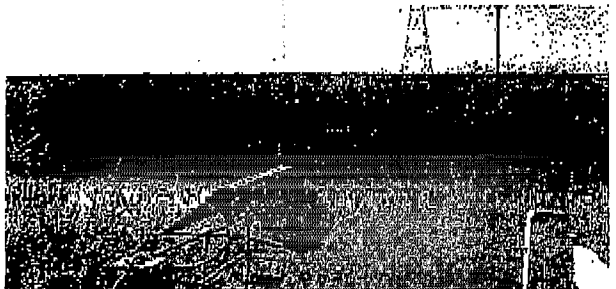


Fig. 2 – All American Canal at Drop 1, meter-cart in foreground

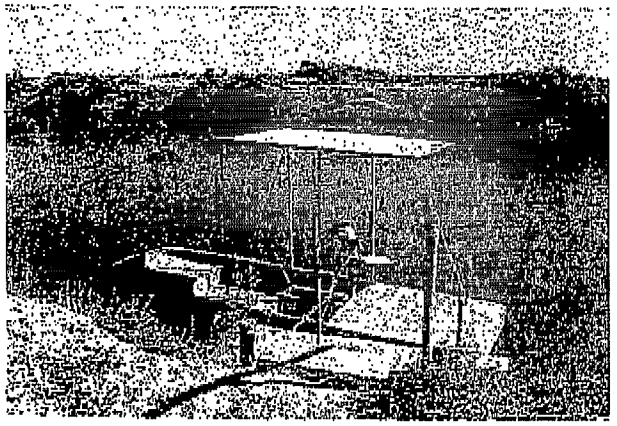


Fig. 3 – East Highline, meter-boat in foreground.

Fig. 4 – Alamo River @ Inlet, temperature and Grab Sample taken only.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

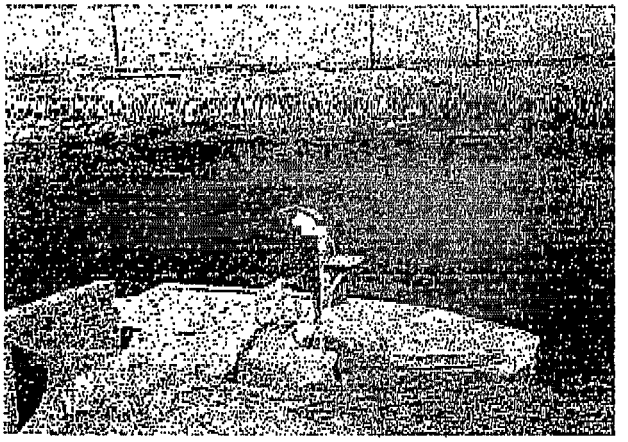


Fig. 5 – Central Main Canal. Meter-boat in foreground.

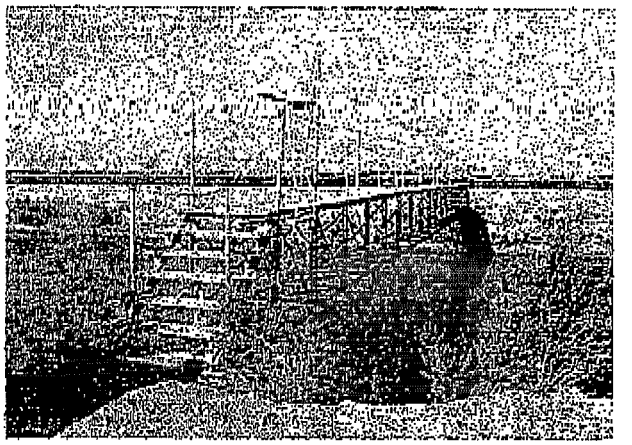


Fig. 6 – Westside Main Canal. Pictured is the meter-bridge that spans the channel.

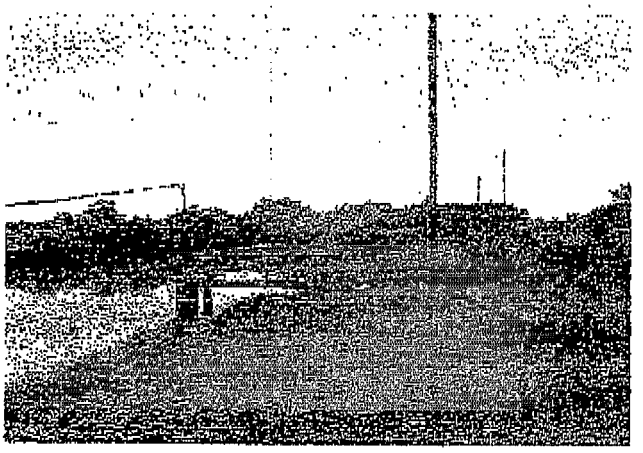


Fig. 7 – Alamo River Outlet. Meter-cart in background. Temperature and Grab Sample taken only.

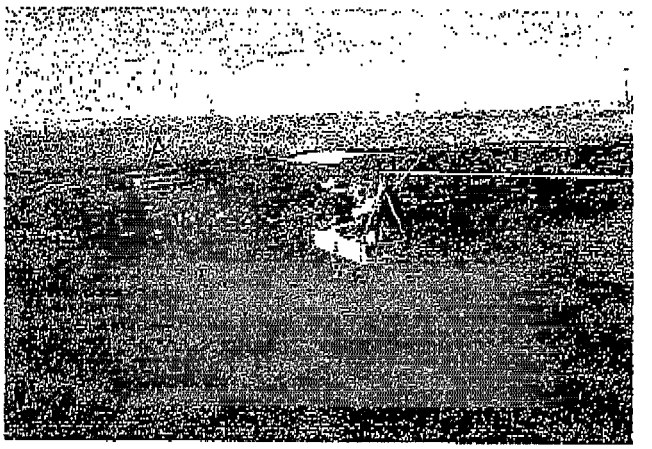


Fig. 8 – *New* River Outlet. Meter-cart centered in picture. Temperature and Grab Sample taken only.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

Step 1 *Collect equipment*

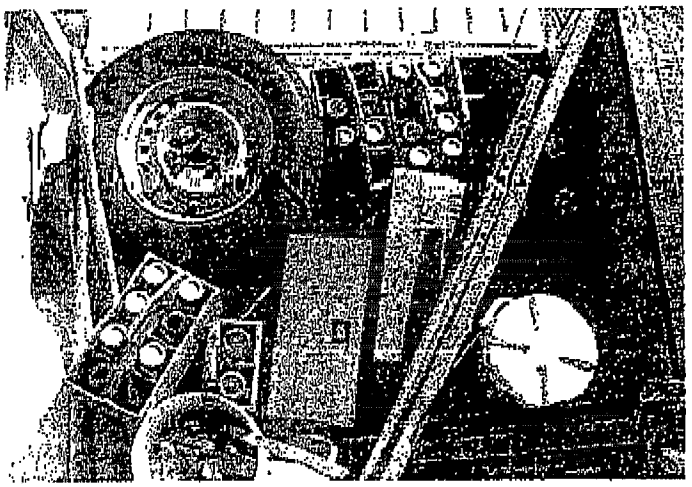


Fig. 9 – Pictured are several pieces of equipment: (A) one-pint bottles, (B) ½ gallon jars, (C) DH59 Sampler, (D) reel and cable assembly, (E) one-quart jars

Fig.10 – Data Technician outfitted with proper gear; life jacket, rubber gloves, and work boots with non-slip soles.

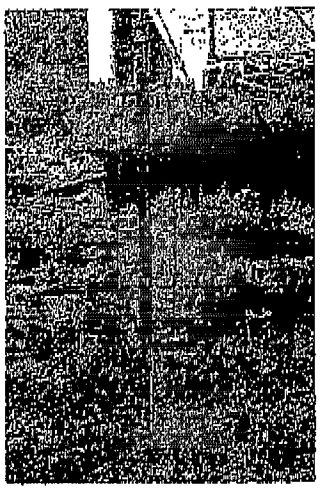


Fig.11 – Circled is a 0°F to 120°F thermometer enclosed in protective metal case.

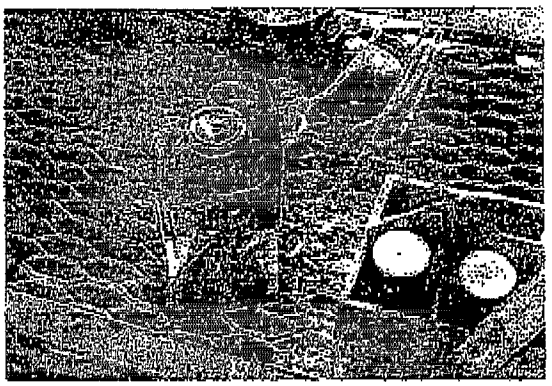


Fig.12 – Close-up view of Grab Sample ½ gallon jar in metal basket.

DATE SAMPLED _____ TIME _____ DATE TO LAB. _____

BOTTLE NO. _____ LAB NO. _____

LOCATION OF SAMPLE _____

DISCHARGE _____ WATER TEMP. _____

METHOD OF SAMPLING _____

REMARKS _____

SAMPLED BY _____

DATA TESTED _____ TESTED BY _____

IID-430A (R3 12-70) - WATER AND SILT SAMPLES

Fig.13 – An example of IID-430A (R3 12-70) form.

Fig.14 – Close-up view of reel/cable assembly. Data Technician labeling paper caps for one-pint specimens.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

Step 2 *Proper use of Safety Harness*

The Alamo River crossing at Boundary requires the use of a Safety Harness. The following is a breakdown of the proper use of safety harness. A safety harness should be used whenever there is a possibility of falling during sample retrieval.

Figure 15 – Drape harness over shoulders. Make sure straps are not twisted or frayed. Buckle top front belt.

Figure 16 – Pull straps from underneath and buckle to bottom set of belts.

Figure 17 – Exposed is hoop for attaching lifeline.

Figure 18 – Attach lifeline to a solid anchor point. In this case, the base of a railing is sufficient. See *Fig. 4*, page 2 for example of harness in action.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

Sub-procedure on the use of the DH-59 Sampler and Temperature.

Note: This aspect applies to AAC Drop 1, EHL, CMC, and WSM sites

Step 1 *Assemble Sampler*

Insert one-pint bottle into cavity of DH-59 Sampler. Be sure that a gasket rests between bottle and backside of nozzle.

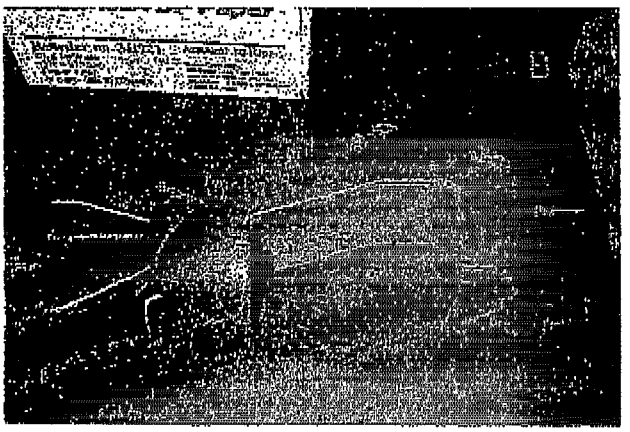


Figure 15 – Shown is the DH-59 Sampler. Arrow (A) points to spring-loaded pull, (B) points to nozzle tip.

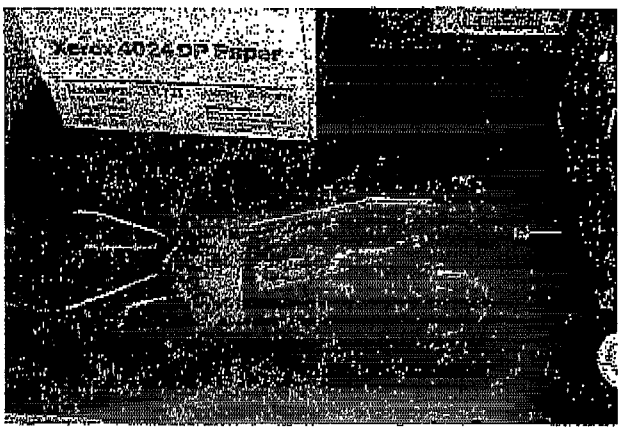


Figure 16 – DH-59 with bottle in place. Arrow (C) points to location of gasket.

Step 2 *Take temperature*

Place thermometer somewhere nearby the sampling in channel. Allow at least ten (10) minutes for temperature to stabilize. Once temperature has stabilized, log result on IID-430A (R3 12-70) Water and silt samples form and log book.

Fig.17 – Drape thermometer's cord over convenient platform and allow completely submerged for approximately ten (10) minutes to stabilize.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

Step 3 *Lower DH-59 onto water surface*

After securing cable/reel assembly to observation platform, attach DH-59 to cable/reel assembly. Lower DH-59 to water surface. Set timepiece to zero; begin lowering DH-59 through stream flow. Once DH-59 touches bottom, retract at the same rate. It should take approximately 15 seconds to complete a sampling. An indication of a proper sample is a not quite full bottle. If bottle is completely full, sample is bad. Discard and redo. If acceptable, place a paper cap with site name to seal sample.
NOTE: Samples are to be retrieved in evenly spaced intervals; six (6) samples from AAC Drop 1, four (4) from remaining sites. Refer to Page 1 "Sites" for listing.

Figure 18 – Attach cable from reel to DH-59

Figure 19 – Close up view of reel assembly.

Figure 20 – Technician with all needed equipment in meter-cart@ AAC Drop 1.

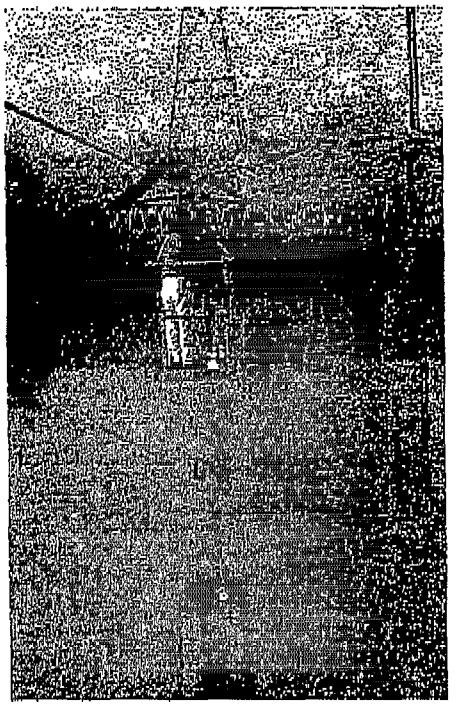


Figure 21 – DH-59 at water surface. Set timepiece to zero, time begins when nozzle (arrow) is submerged.

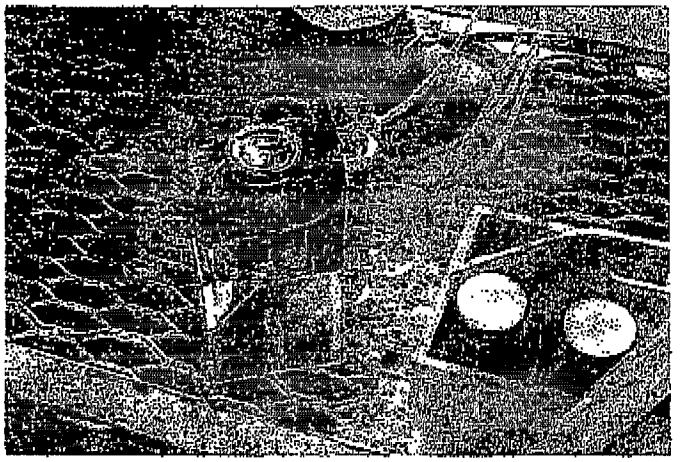
IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

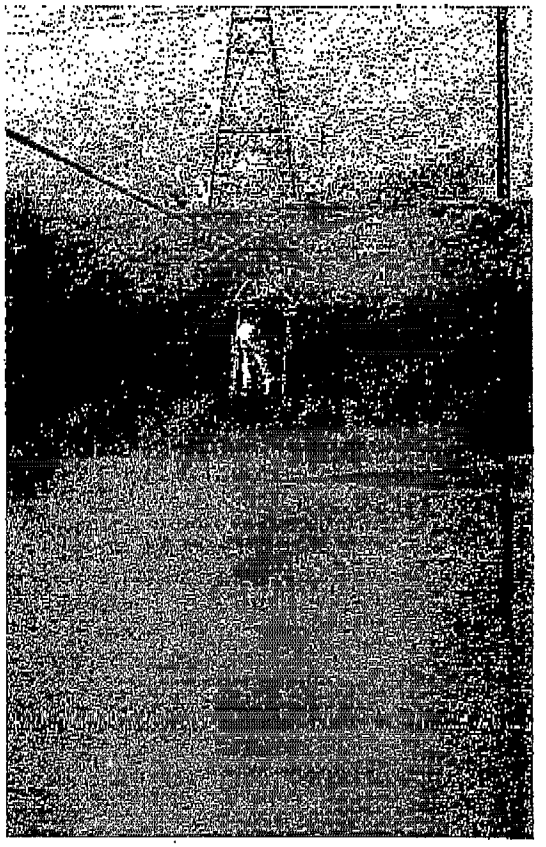
Sub-procedure on the use of the Grab Sampler and Temperature*

Note: This aspect applies to AAC Drop 1, EHL, Alamo River Inlet, CMC, WSM, New River Outlet, and Alamo River Outlet sites.
*Refer to **Sub-procedure on the use of the DH-59 Sampler and Temperature**, Step 2, page 5, for detail on temperature readings.

Step 1 *Collect Grab Sample*

When near center of stream, collect a sample of water in a glass jar. The technique used is akin to dunking. ½-gallon jar samples are taken at AAC Drop 1, New River, Alamo River Inlet and Outlet. These sites require the use of a basket and rope.
One-quart jar samples are taken at EHL, CMC, and WSM, refer to *Fig.24* for example.
Write resulting sample collection into logbook





Figures 22 & 23 – Above, close-up view of basket/rope device for retrieving Grab samples. To the right, dunking of ½-gallon jar with basket/rope device at AAC Drop 1 site.

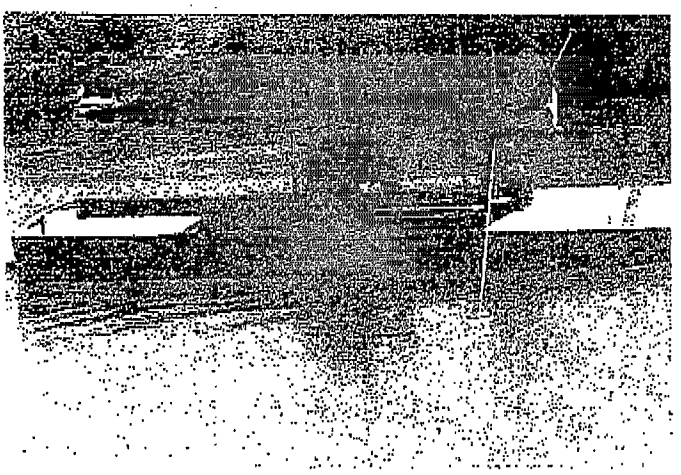


Fig.24 – East Highline site. An alternative method to retrieve a Grab Sample is by dunking manually several times in order to get a relative composite.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

These final steps apply to both sub-procedures

Step 2 *Terminate Session*

Gather all equipment used for collecting samples. Place DH-59 in case, jars in box, one-pint bottles in carrying case. Make sure all samples are sealed and protected from spilling during transport.

Step 3 *Place samples in Laboratory*

Place all collected samples in laboratory, located on southeast corner of El Centro Steam Plant (Biological Control building). Include all completed IID 430A slips.

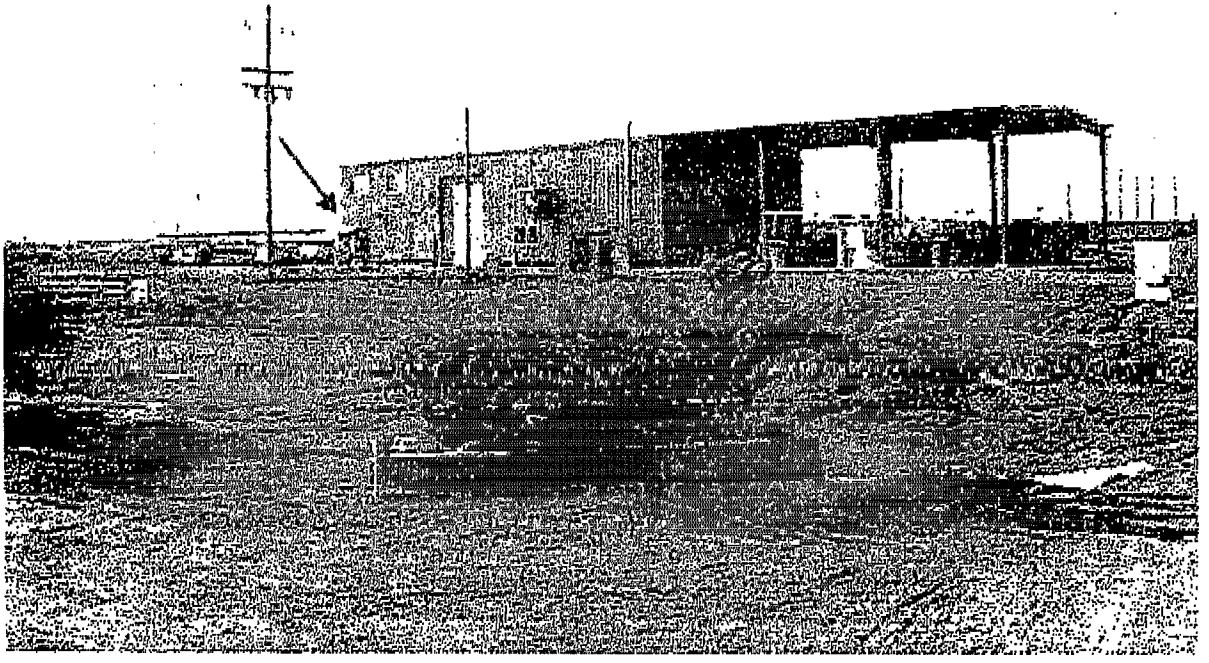


Fig.25 – Picture of Biological Center building, located on southeast corner of El Centro Steam Plant's yard. Red arrow points to entryway for laboratory.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

Procedure:	DCO-SS-01
Date created/ Revision:	12/19/00, 3/26/01
Printed:	3/26/01 07:44
Associated Procedures:	DCO-SILTTDS-01
Object:	To collect water samples around the Salton Sea using the Grab method.
Schedule:	Bi-annually. Samples collected in the months of July and November.
Personnel:	Two Data Technicians
Equipment:	Labeled Sample containers (five-½ gallon jars) with lids Basket with handle for Grab samples Thermometer with 0°F to 120°F range Pencil for noting observations Timepiece IID-430A (R3 12-70) Water and silt samples form (5 minimum) Rubber waders Rubber gloves
Sites:	Between Rivers (Coor. N33.13988° W115.66645°) – near sump pump S-307. Approximately 3000 ft. north along dike from the western end of Young Rd., near the center of Section 7, T.12 S., R.13 E. Bertram Station (Coor. N33.35805° W115.76081°) – Walk approximately 1100 ft. southeasterly from a point on Hwy 111 which is 1.4 miles northwesterly of Bombay Beach and near MCI marker labeled *64840*, near the SW corner of the NW ¼ of Section 24, T.9 S., R.11 E. Desert Beach (Coor. N33.51551° W115.93545°) – Enter the townsite of North Shore on Desert Beach Drive. Site is approximately 200 ft. southwesterly of southwest corner of North Shore Marina RV Park, near the center of east line of the NE ¼ Section 9, T.9 S., R.9 E. Salton Sea Beach (Coor. N33.375090° W116.00645°) – approximately 1.1 miles east of Hwy 86 along Brawley Ave in the townsite of Salton Sea Beach, near the center of the SW ¼ of Section 23, T.9 S., R.9 E. Sandy Beach (Coor. N33.17685° W115.83465°) – Turn-off 3.3 miles north of Three Flags Ranch turn-off. Proceed approximately 3.1 miles east, near the SE corner of the SE ¼ of Section 20, T.11 S., R.11 E.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

Overall site view

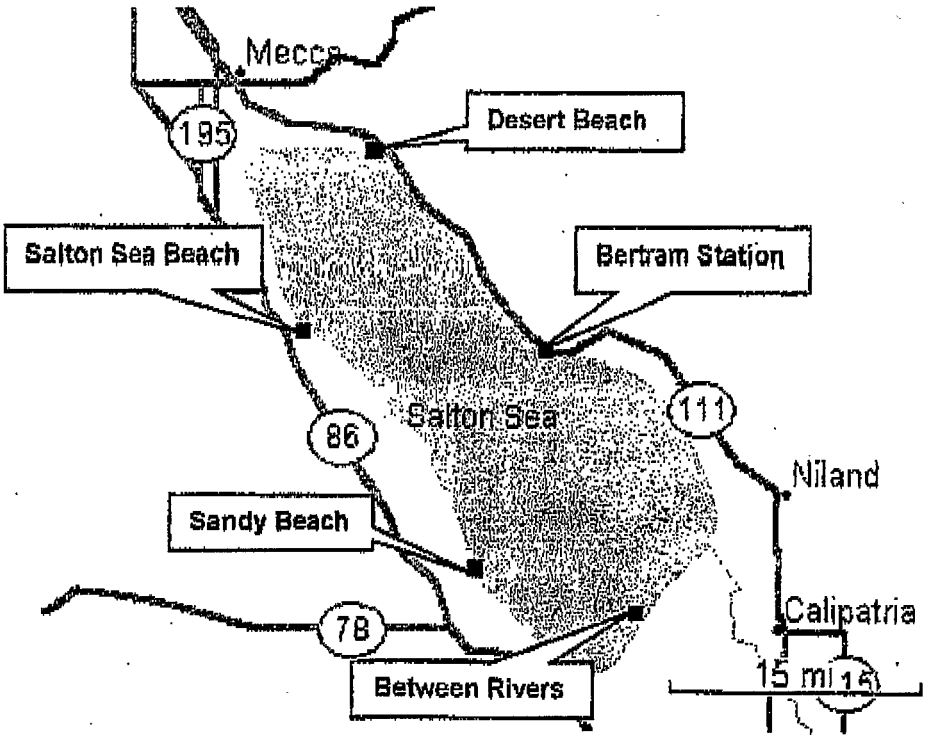


Figure 1 – Illustration of all sites around the Salton Sea.

Figure 2 – Between Rivers site. Sample taken near discharge point of pumps S-307 and WP-9.

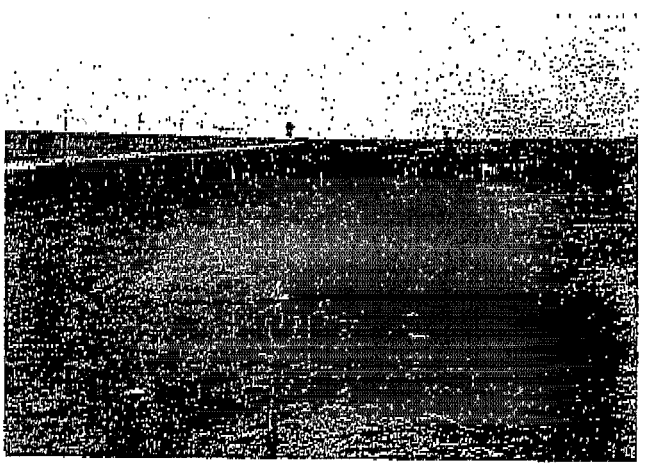


Figure 3 – Bertram Station entry site. Point is marked solely with a wooden lathe and a rubber glove. The adjacent area is protected by the State. Vehicular traffic is prohibited.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

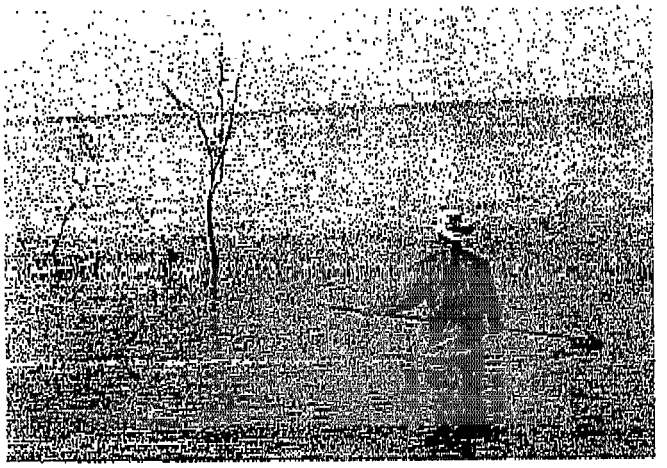


Figure 4 – Data Technician collecting sample using pole and basket at Desert Beach. Use rubber waders to access site.

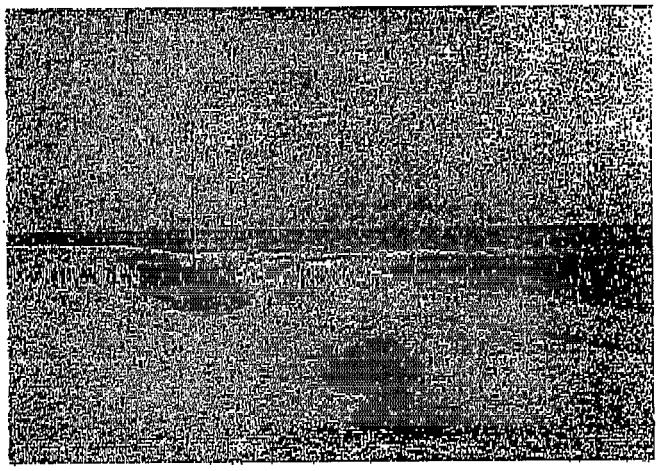


Figure 5 – Salton Sea Beach shoreline. Use rubber waders at this site also.

Figure 6 – Turn-off entrance from Highway 86 for Sandy Beach. Located approximately 3.3 miles northwest of Three Flags Ranch.

Figure 7 – Assembled sampling device.

FIELD PROCEDURE

Step 1 *Assemble Sampling device*

After putting on rubber gloves and waders, place sample jar into basket/pole device. Secure with spring-loaded outer ring.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

Step 2 *Technique for collecting sample*

Carefully walk out into Sea. Make sure footing is secure with each step. Go out to a depth of approximately 3 feet. With a sweeping motion, dunk sample jar repeatedly until full. Typically 3 to 4 passes, refer to *Figure 4* for example.

Step 3 *Take temperature*

Immerse thermometer nearby on the shore and let stabilize for approximately 10 minutes.

Step 4 *Log results*

Fill out form IID-430A (R3 12-70) Water and Silt Samples, for each site. Fields of importance are the (1) DATE SAMPLED, (2) TIME, (3) LOCATION OF SAMPLE, (4) TEMPERATURE, (5) METHOD OF SAMPLING, & (6) SAMPLED BY. Refer to *Figure 8* for example of form.

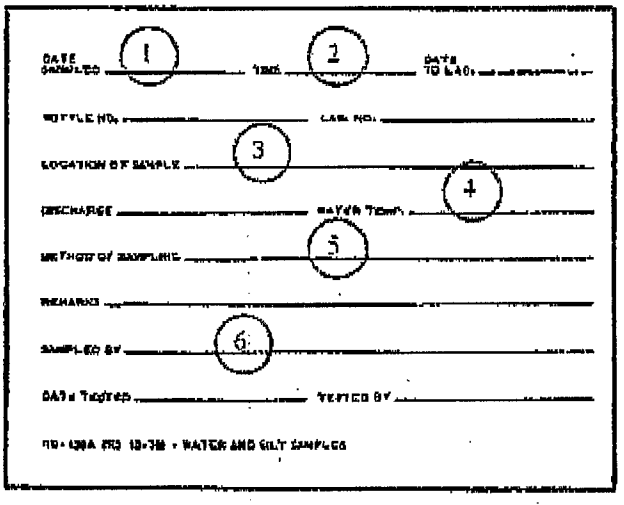


Figure 8 – An example of IID 430A form.

Step 5 *Deliver Samples*

After collecting samples from all sites, secure them so as not to spill during transport. Deliver samples directly to ATS Laboratory in Brawley, refer to *Fig. 9.*

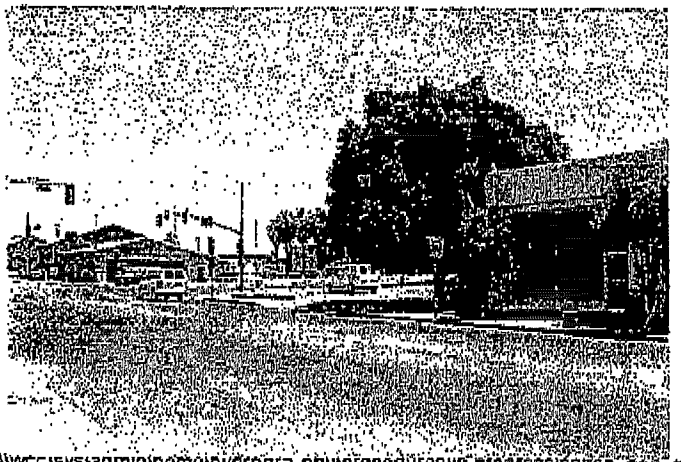


Figure 9 – ATS Laboratory in Brawley. Located near the southeast corner of 8th and Main Street.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

OFFICE PROCEDURE

Step 6 *Assign Laboratory Numbers to Samples*

A three-ring binder, labeled **Silt/TDS Log Sheets**, with *Laboratory Numbers Index* (form IID-442C R2 8-65), located at WCC's Hydrography Unit, contains a sequential list of Lab numbers. Follow format of previous entries. Write corresponding numbers onto slips. Make copies of slips. Send originals to Water Department, Engineering Services, Technical Resources and Planning Unit.

Step 7 *File copies*

File copies of slips in file cabinet, located in Hydrography Unit's Data Technician office area. Place in folder labeled with corresponding numerical sequence. Once ATS Laboratory submits a report of the chemical breakdown, attach copy of relative slips to this report and file. Duty completed.

CLINICAL LABORATORY of SAN BERNARDINO

QUALITY ASSURANCE MANUAL

Revision 3.0 – June 24,2004

_X_ Controlled ___ Uncontrolled

Issued to: MELANIE EMANUAL

Copy No.: QA 62204A Date: 6/22/04

Approved Signatures:

Richard Kelso (Laboratory Director) Date

Joseph LaVoie (Quality Manager) 6/22/04 Date

The information in this CLSB QA Manual is intended for the addressee as issued and noted above. To remain compliant with quality control standards this manual is not to be duplicated.

CLINICAL LABORATORY of SAN BERNARDINO
21881 Barton Road, Grand Terrace, CA 92313
Phone: (909) 825 - 7693 Fax: (909) 825 - 7696

FOREWARD

The following document was prepared in accordance with the USEPA guidelines specified in "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" (QAMS-005/80). It is the intent of CLSB to meet or exceed the QA/QC requirements set by USEPA or other appropriate governmental or private entities and to assure that all analytical data generated are scientifically valid, defensible, comparable and of known acceptable precision and accuracy.

Richard Kelso
Laboratory Director

W. Joseph LaVoie
QA Manager

6/24/04

Clinical Laboratory of San Bernardino

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Table of Contents

1.0 INTRODUCTION

The purpose of the Clinical Laboratory of San Bernardino (CLSB) Quality Assurance Manual is to document the minimum quality assurance requirements for the laboratory. This Quality Assurance Manual provides ready reference for analysts and clients on CLSB's policy pertaining to the accuracy and reliability of analytical tests performed in the laboratory.

The policies contained within this CLSB Quality Assurance Manual are to be applied to all laboratory operations. The manual is updated as required to provide for the addition of new methods and procedures as they are developed.

1.1 CLSB ANALYTICAL SERVICES

Clinical Laboratory of San Bernardino (CLSB) is an environmental testing laboratory providing a wide range of analytical services to both the public and private sectors. CLSB laboratories are located in Grand Terrace, California and features modern facilities and equipment. The staff is comprised of chemists, scientists, and technicians from a broad range of academic and environmental disciplines. The staff recognizes the need for high quality and legally defensive data, and the impact that this data has on the decisions of our clientele. It is our company mission to provide our customers with high quality, and cost effective laboratory services that will meet and/or exceed our customers' expectations.

1.2 LABORATORY ORGANIZATION AND RESPONSIBILITY

Since the demands on an environmental testing laboratory can be great and diverse in nature, the CLSB laboratories are structured into distinct and effective departments. These departments have clearly defined objectives and responsibilities that are directly involved in the analytical testing process. The structure of CLSB provides a framework for high quality analytical operations for which the Quality Assurance manual is the blueprint. The minimum responsibilities of laboratory personnel are defined as follows with the laboratory organization outlined in Figure 1-1.

- **President**

  The President is responsible for the management of the entire laboratory both financial and technical. It is the President's job to implement corporate goals, objectives and policies. The President is in direct communication with the Laboratory Director and Quality Assurance Manager.

- **Laboratory Director**
  Ultimate responsibility for laboratory operations and Quality Assurance is that of the Laboratory Director. The Laboratory Director communicates with the Quality Assurance Manager and Laboratory Manager to ensure that the CLSB Quality Assurance Manual

and SOPs are followed as written. The Laboratory Director works with each department supervisor to implement the QA/QC procedures of this manual. It is the Laboratory

Director's job to see that the non-laboratory departments (administration, data processing, etc.) of CLSB work with their laboratory counterparts to achieve high quality results.

- **Laboratory Manager**

The Laboratory Manager supports the Laboratory Director with the daily operation of the laboratory, working closely with Project Managers, Department Supervisors and clients.

- **Department Supervisors**

CLSB is divided into four analytical departments: Inorganics, Organics, Radiochemical and Microbiology; each department having its own supervisor. The department supervisors provide supervision of group operations, implement the laboratory quality assurance plan, ensure proper scheduling and execution of analyses, assure that proper analysis techniques are being used (use of approved SOPs), review all data before it is released to Quality Control, and report all discrepancies to the QA department.

- **Client Services/Project Management**

Client Services/Project Management serves as the primary laboratory contact for CLSB clientele. Any changes in the scope of work will be processed through this department. The department monitors the progress and timeliness of analytical work; reviews ongoing work orders and all subsequent final laboratory reports for accuracy and adherence to the QA Plan.

- **Field Services**

Field Services has the responsibility of proper sampling and transportation of samples to and from CLSB. Field service personnel are required to know CLSB's QA policies and report to the Field Services Manager.

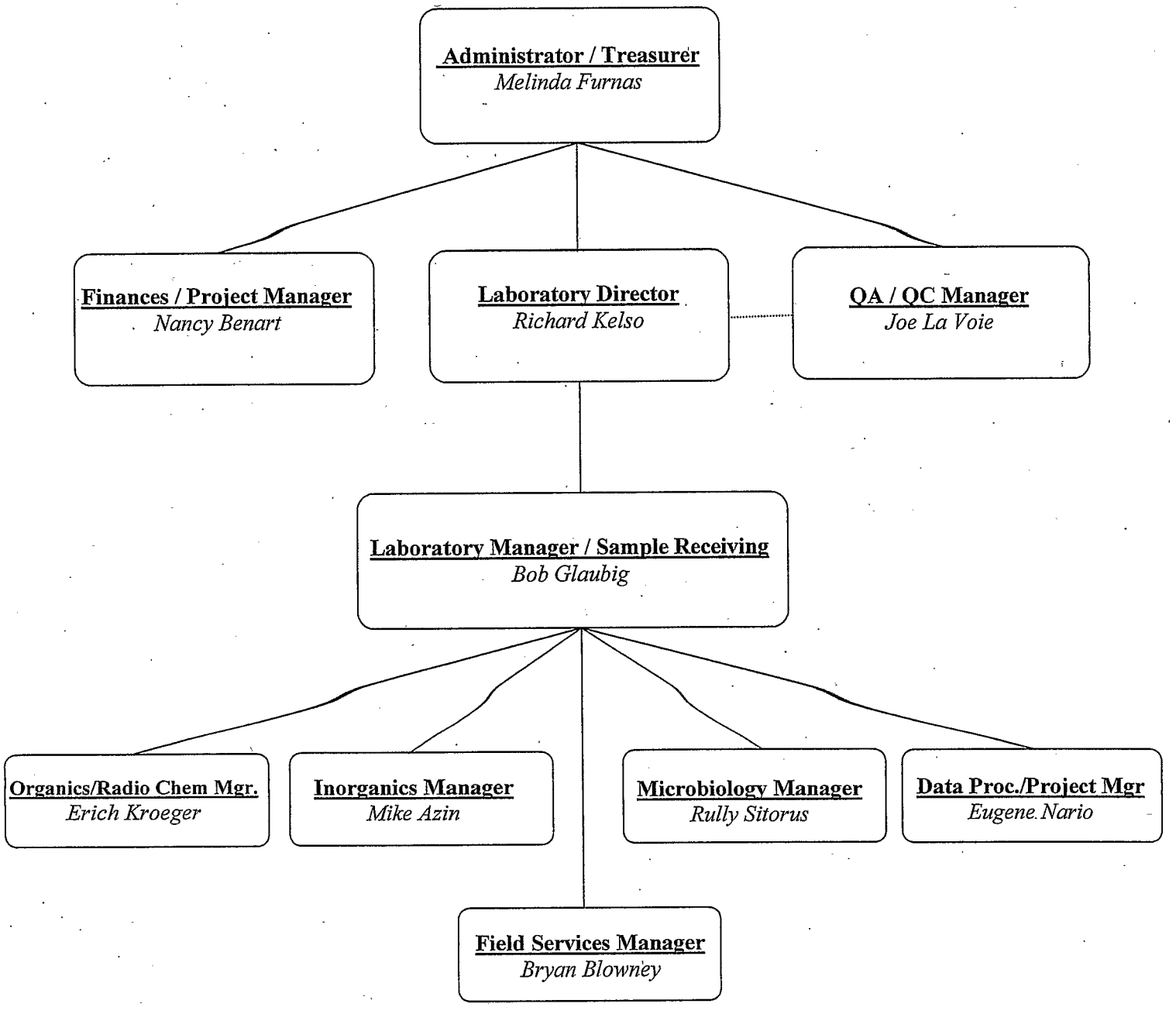
- **Administration**

Administration is responsible for the management of financial operations, including accounting and procurement of all laboratory items. It is the Administration's job to ensure that purchased items and services meet the QA Plan requirements and perform as outlined in this document.

- **Data Control**

Data Control (Data Management) is responsible for sorting, reviewing, distributing, filing, and archiving all data generated by CLSB. This department works with the QC department to help implement QC standards through the use of electronic programming.

Clinical Laboratory of San Bernardino, Inc.
6/22/04



1.3 OBJECTIVES OF THE QA PROGRAM

The primary objective of the Quality Assurance program is to produce quality data which is of known precision and accuracy and legally defensible. This will ensure that the data can be relied on to represent the true value for a given sample.

1.4 CLSB QUALITY ASSURANCE PROGRAM

The CLSB Quality Assurance/Quality Control Program is an essential part of any analytical procedure. The program has been integrated into every phase of laboratory operations. The program detects and corrects problems in the measurement process to ensure that all data is valid, of known precision and accuracy, and is legally defensible. Secondly, it is designed to monitor and control the quality of the data generated by the laboratory, thus ensuring that errors are kept to an acceptable level and corrective action is taken when necessary.

1.5 QA MANUAL SUMMARY

The organization of this manual is presented in the table of contents. Essentially the manual follows the logical progression of analytical work and the application of the Quality Assurance Program in the laboratory. The program can be divided into four major areas.

1.5.1 Pre-Analytical Procedures

The pre-analytical work includes the various aspects of sampling, preservation and storage, documentation, materials and standards, and calibration.

1.5.2 Procedures Concurrent with Analysis

This group of procedures includes Quality Control steps such as blanks, spikes, replicates, etc., as well as analytical methodology.

1.5.3 Data Reduction and Evaluation

Both QC and sample data must be evaluated and a QC check performed to ensure the data obtained is valid and falls within acceptable precision and accuracy limits.

1.5.4 Data Reporting and Record Maintenance

Specific reporting formats may be required for different projects, but all data must be reviewed before being released. Additionally, records are maintained to allow access for future inquiries concerning the results.

1.6 ANALYTES AND ANALYTICAL METHODS

The principle methods used for the analysis of drinking water and wastewater come from USEPA procedures and APHA's "Standard Methods" 18th Ed. All analysis performed at CLSB comply with these methods and are listed in Table 1-2.

CLSB has written SOPs for the bench level analyst using these methods that comply with all federal and state regulations.

ANALYTICAL METHODS

A. ORGANIC CHEMICAL TESTING

	Water
1. EDB and DBCP	EPA 504.1
2. Glyphosate	EPA 547
3.. Purgeable Organic Compounds by GC/MS	EPA 524.2
4.. Benzo-(A)-pyrene	EPA 550.1
5. Haloacetic Acids	EPA 552.2
6.. Oil & Grease	EPA 1664

Table 1-2 Analytical Methods

ANALYTICAL METHODS (cont.)

B. INORGANIC CHEMICAL TESTING

	Water
1. Aluminum	EPA 200.7
2. Antimony	SM3113B
3. Arsenic	SM3113B
4. Barium	200.7
5. Beryllium	SM3113B
6. Boron	EPA 200.7
7. Cadmium	SM3113B
8. Chloride	EPA 300.0
9. Calcium	200.7
10. Chromium, hex	EPA 218.6
11. Chromium, total	SM3113B
12. Copper	EPA 200.7
13. Cobalt	EPA 200.7
14. Fluoride	EPA 300.0/340.2
15. Iron	EPA 200.7
16. Lead	SM3113B
17 Magnesium	EPA 200.7
18. Manganese	EPA 200.7
19. Mercury	EPA 245.2
20. Molybdenum	EPA 200.7
21. Nickel	SM3113B
22. Nitrate /Nitrite	EPA 300.0 /353.2
23. Perchlorate	EPA 314.0
24. Phosphorus	EPA 365.2
25. Potassium	EPA 200.7
26. Selenium	EPA 200.7
27. Silicon	EPA 200.7
28.. Silver	SM3113B
29.. Sodium	EPA 200.7
30. Sulfate	EPA 300.0
31. Sulfide	EPA 376.2
32. Thallium	EPA 200.7/200.9
33. Vanadium	EPA 200.9
34. COD	Hach 8000
35. Cyanide	SM4500 CN –F

Table 1-2 Analytical Methods (cont.)

ANALYTICAL METHODS (cont.)

C. DRINKING WATER AND WASTEWATER TESTING

		Method
1.	General Mineral	
a.	Calcium, Magnesium, Sodium, Potassium ,Silica	EPA 200.7
b.	Alkalinity	SM2320B
c.	Anions	EPA300.0-EPA 300.1
d.	Fluoride	EPA 340.2
e.	Acidity	EPA 305.1
f.	Calcium	SM 4500 –Ca D.
2.	General Physical	
a.	pH	EPA 150.1
b.	Specific Conductance	EPA 120.1
c.	Total Dissolved Solids	EPA160.2,SM2540C
d.	Turbidity	EPA 180.1
e.	Methylene Blue Active Substances (MBAS)	EPA425.1/SM 5540C
f.	Volatile Residue	EPA 160.4
g.	Settleable Residue	EPA 160.5
h.	Total Residue	SM2540B
i	Filterable Residue	SM2540C
j	Odor	SM2150A
k.	Color	SM2120B
l.	Conductivity	SM2510B
3.	Primary/Secondary Inorganic	
a.	Aluminum, Barium, Copper, Iron, Manganese, Silver, and Zinc	EPA200.7
b.	Arsenic, Antimony .	SM3113B
c.	Cadmium	SM3113B
d.	Chromium	SM3113B
e.	Lead	SM3113B
f.	Mercury	EPA 245.2
g.	Selenium	SM3113B
h.	Chlorine	SM 4500 Cl G
i	COD	Hach 8000 Method
j	Ammonia	EPA 350.1
k.	Kjehldahl Nitrogen	EPA 351.2
l.	Dissolved Oxygen	EPA 360.1
m.	Total Phosphorous	EPA 365.2
n .	Orthophosphate	EPA 365.2

4.	Regulated Organic	
a.	Volatile Organic Compounds	EPA 524.1
b.	DBCP and EDB	EPA 504.1
c.	Pesticides	EPA 531.1
d.	Glyphosate	EPA 547
e.	Benzo -(A) –Pyrene	EPA 550.1
f.	Halocetic Acid	EPA 552.2

Table 1-2 Analytical Methods (cont.)

ANALYTICAL METHODS (cont.)

D.. Radiochemistry of drinking water and wastewater .	Method
1. Gross Alpha	EPA 900.0
2. Gross Beta	EPA 900.0
3. Uranium	EPA 908.0
4. Radon –222	SM 7500 – Rn

E. MICROBIOLOGICAL TESTING

		Method
1.	Total, Fecal, E. Coli Coliforms by Multiple Tube Fermentation	9221A*
2.	Total & E. Coli Coliforms by MMO-MUG	9223B *
3.	Heterotrophic Plate Count	9215B *
4.	Total Coliform by Multiple Tubes Fermentation	9221B *
5.	Fecal/E. Coli by Multiple Tubes Fermentation	9221E *
6.	Fecal Strep Bacteria	9230B *
7.	BOD /CBOD	5210B *

* Standard Method - 19th Ed.

Table 1-2 Analytical Methods (cont.)

2.0 SAMPLE RECEIPT, PRESERVATION AND STORAGE

To provide representative samples for analysis, both field and laboratory personnel must satisfactorily perform their duties. Field sampling is a critical part of the analytical process and can have a direct effect on data quality. All samples must be properly collected, preserved, and transported to the laboratory before analysis.

2.1 SAMPLING PROCEDURES AND DOCUMENTATION

Proper sampling in the field requires consideration of many aspects including:

- Sampling technique
- Containers used
- Labeling the containers
- Preservation and Storage
- Transportation
- Documentation
- Identification of analysis required to give useful results for the intended purpose

The items discussed in this section touch on several of these key elements in environmental sampling and analysis.

2.1.1 Chain of Custody

An overriding consideration for accurate analytical results is the ability to demonstrate that the samples have been obtained from the locations stated and that they have reached the laboratory without alteration. To accomplish this, evidence of collection, shipment, laboratory receipt, and laboratory custody must be documented.

Documentation is accomplished through a "chain of custody" (COC) form that records each sample and the individuals responsible for sample collection, shipment and receipt. A sample is considered in custody if it is:

- In a person's actual possession
- In view after being in physical possession
- Locked up so that no one can tamper with it after having been in physical custody
- In a secured area, restricted to authorized personnel

Figure 2-1 represents a chain of custody form (COC) that is used by CLSB personnel in collecting and shipping samples.

Each individual who has the samples in their possession signs the COC form. Preparation of the COC shall be as follows:

- The person collecting the samples shall initiate the chain of custody record, in the field. Samples can be grouped for shipment and can use a common COC form.
- The record shall be completed in the field to indicate project, sampling team, and other necessary information.
- If the person collecting the sample does not transport the samples to the laboratory, or deliver the sample containers for shipment, the first block for: Relinquished By_____, Received By _____ shall be completed in the field.
- The person transporting the samples to the laboratory or delivering them for shipment shall sign the record form as: Relinquished By _____.
- If the samples are shipped to the laboratory by commercial carrier, the COC form shall be sealed in a watertight container, placed in the shipping container, and the container sealed prior to giving it to the carrier.
- If the samples are directly transported to the laboratory, the COC form shall be kept in possession of the person delivering the samples.
- For samples shipped by commercial carrier, the weigh bill shall serve as an extension of the chain of custody record between the final field custodian and receipt in the laboratory.
- Upon receipt in the laboratory, the Sample Control Manager, or representative, shall open the shipping containers, compare the contents with the COC record and sign and date the record.
- If discrepancies occur, the sample in question shall be segregated from normal sample storage and the field personnel immediately notified.
- COC forms shall be maintained with the records for a specific project, becoming a part of the data package.

Multipart COC forms may be used so that a copy can be returned to the person shipping the samples after they are received in the laboratory and after the laboratory disposes of the samples. Otherwise, photocopies will be made and distributed.

2.1.1.1 Forms for Microbiology

Three forms are for the exclusive use of the Microbiology Department. The receipt of sample, data and results are not entered into the LIMS system.

2.1.1.1.1 Coliform Bacteria, 15 Tube Dilution, Water/Wastewater Form, Figure 2.2

This form is for the collection of water/wastewater samples for the above described analysis. The form has all sample collection information and all data reporting sections included. The form is completed when analysis of samples is finished. After the form is reviewed by the laboratory director, a copy is kept in the internal filing system and a copy is sent to the client.

2.1.1.1.2 Coliform Bacteria Report Form, Figure 2.3

This form is for the collection of drinking water for the analysis for Coliform Bacteria. As above, the form has all collection information and all data reporting sections included. The form is complete when the analysis data is entered. After the form is reviewed by the laboratory director, a copy is kept in the internal filing system and a copy is sent to the client.

2.1.1.1.3 General Physical Analysis Form, Figure 2.4

This form is for the collection of drinking water for general physical analysis. As above, the form has all collection information and all data reporting sections included. The form is complete when the analysis data is entered. After the form is reviewed by the laboratory director, a copy is kept in the internal filing system and a copy is sent to the client.

Clinical Laboratory of San Bernardino, Inc. CHAIN OF CUSTODY PAGE ___ OF ___

CLIENT:

Address:

System Number:

Phone No.: Fax No.:

Project Name:

Sampled By:

Comments:

CLIENT JOB NUMBER

(Company Use Only)

DESTINATION LABORATORY

☐ CLINICAL LABORATORY OF SAN BERNARDINO, INC. (909) 825-7693

☐ OTHER _____

PRESERVATIVES

ANALYSIS REQUESTED

TURN AROUND TIME

DATE	TIME	SAMPLE IDENTIFICATION	MATRIX	NUMBER OF CONTAINERS

PRESERVATIVES: (1) $Na_2S_2O_3$ (2) HNO_3/HCl (3) COLD (4)

TURN AROUND TIME: (10) 10 DAY (5) 5 DAY RUSH (2) 2 DAY RUSH ALL TURN AROUND TIMES ARE EXPRESSED AS WORKING DAYS. SOME ANALYSES MAY TAKE LONGER THAN CHOSEN TIME DUE TO METHOD LIMITATIONS.

RELINQUISHED BY (SIGN)	PRINT NAME / COMPANY	DATE / TIME	RECEIVED BY (SIGN)	PRINT NAME / COMPANY

REC'D AT LAB BY: DATE / TIME: CONDITIONS / COMMENTS:

SHIPPED VIA ☐ FED X ☐ GOLDEN STATE ☐ GREYHOUND ☐ CLIENT ☐ OTHER _____

2.1.2 FIELD COLLECTION AND SHIPMENT

Prior to collecting samples, the collection team must consider the analyses to be performed so that proper sample containers and shipping containers can be assembled and proper preservatives added to containers. In addition, field logs, record sheets, chain of custody forms (COC) and analysis records must be assembled.

All records required for proper documentation must be completed by the field team. The primary documenting record is the COC form, the Sample Receiving Log-in Sheet and the records in the LIMS system as discussed in Section 2.2.

In addition to initiating the COC form, field personnel are responsible for uniquely identifying and labeling samples, providing proper preservation, and packaging samples to preclude breakage during shipment.

2.1.2.1 Labeling

Every sample shall be labeled to identify:

- Project or job number
- Sample location (such as well number)
- Sampling date and time
- Person obtaining the sample
- Sample preservation/conditioning method, if applicable
- Analysis requested

2.1.2.2 Sample Containers and Preservation

Containers provided by CLSB have been purchased from commercial suppliers and are certified as to being cleaned per USEPA procedures for low level chemical analysis. Samples must be placed in containers compatible with the intended analysis and properly preserved. Also, collectors of samples must consider the time interval between acquiring the sample and analysis (holding time) so that the sample is representative. Table 2-1 provides requirements for various analytical parameters with respect to preservation, method and maximum holding time between collection and analysis.

2.1.2.3 Sample Transportation

Shipping containers are to be sealed prior to shipment, whether shipped by direct transport by field personnel or commercial carrier. The only exception is if sufficient holding time exists so that the samples can be held in the field, but it will be necessary to re-ice the containers prior to or during transport.

2.1.2.4 Request for Analysis

The final step in providing information to the laboratory is the "Analysis Requested" portion of the COC form. The Analysis Requested, included on the CLSB COC form (Figure 2-1), shall be completed by the field personnel and included with the COC record. Any other form, provided by the client, that details the requested analysis may be substituted for the COC form provided sufficient information is included. It is imperative that the "Analysis Requested" information be provided to enable the lab to comply with maximum allowable sample holding times.

2.2 RECEIPT OF SAMPLES AND CHAIN OF CUSTODY

Samples are stored either in a cold room at 4° C or in a refrigerator or freezer depending on the type of samples and analysis. Samples for volatile analyses, such as EPA 601/602, are stored in separate refrigerators.

The Sample Receiving staff will receive the samples and:

- Examine all samples and determine if proper temperature and preservation have been maintained during shipment. If samples have been damaged during shipment, the remaining samples shall be carefully examined to determine whether they were affected. Any samples affected shall also be considered damaged. It will be noted on the COC record that specific samples were damaged. The Sample Receiving supervisor is notified and the client is contacted as to the damage.
- Compare samples received against those listed on the Chain of Custody.
- Verify that sample holding times have not been exceeded.
- Sign and date any Chain of Custody form and attach any waybill to the Chain of Custody.
- Place the samples in proper laboratory storage.
- Enter the client name in the laboratory Sample Log-in Sheet.
- Enter all login information into the computer information system.
- Issue and distribute a work order to the appropriate analytical department.
- Place the completed chain of custody records in the project file.

After sample receipt and inspection, the log-in personnel will sign the COC form. For samples delivered by mail or by a third party, the client should include a signed COC form with all the required information. A signed copy of the COC form will be included in the final report and in a QC package report to be kept in our archive room.

2.3 PRESERVATION, STORAGE AND DISPOSAL

2.3.1 Sample Preservation

Preservation of samples is addressed in several of the references in Section 2.1. Additionally, Table 2-1 summarizes preservation methods.

2.3.2 Laboratory Storage of Samples

The primary consideration for sample storage is:

- The extraction and analysis of samples within the prescribed holding times for the parameters of interest.

The requirements of Table 2-1 for holding times shall be used. Placing of samples in the proper storage environment is the responsibility of the Sample Control Manager, who should notify the Laboratory Director, or department supervisor, if there are any samples that must be analyzed immediately because of holding-time requirements.

2.3.3 Sample Disposal

Ultimate disposition of the samples is addressed in CLSB's Haz-Waste Disposal Plan. There are several possibilities for sample disposition:

- The sample may be completely consumed during analysis.
- Samples may be returned to the client or location of sampling for disposal.
- The samples may be stored after analysis. Proper environmental control and holding time must be observed if reanalysis is anticipated. Otherwise, environmental conditions for storage will not be observed.

The Sample Control Manager shall determine disposition of samples if not specified on the COC (Figure 2-1). In general, CLSB does not maintain samples and extracts longer than 30 days beyond receipt of analytical data by the customer, unless otherwise specified.

2.4 Initiation of Testing Program

As stated in Section 2.1.1, the COC form (including the Analysis Request) shall be submitted with the samples to the laboratory.

If the analytical program is not defined with the sample shipment, the Sample Control Manager shall immediately notify the responsible field personnel for definition of the analysis program. If the samples are external to CLSB, the client shall be contacted to determine the testing program. The Sample Control Manager will store the samples as appropriate. The COC form, Sample Log-in Sheet, and the LIMS record remain the primary sample documents.

The Laboratory Director or Laboratory Manager is responsible for prioritizing samples on the basis of holding time and required turn around time.

ANALYSIS	METHOD	PRESERVATIVE	HOLD TIME
GC VOLATILES:			
EDB / DBCP	EPA 504/8011	$Na_2S_2O_3$	14 days
Purgeable Aromatics or BTEX	EPA 602/8020	$Na_2S_2O_3$	14 days
Purgeable Aromatics & Halocarbons	EPA 502.2/8021	$Na_2S_2O_3$, HCl	14 days
Purgeable Halocarbons	EPA 601/8010	$Na_2S_2O_3$	14 days
Purgeable Non-Halogenated Hydrocarbons	EPA 8015	None	14 days
PETROLEUM FUELS:			
TPH Gasoline, BTEX, MTBE	EPA 8015m/602/8020	HCl	14 days
Oxygenates	EPA 8260m	HCl	14 days
TPH Extractables (Diesel/Oil)	EPA 8015m	None	7 days
Oil & Grease	EPA 1664	HCl	28 days
TRPH	EPA 1664 SGT	HCl	28 days
GC/HPLC SEMI-VOLATILES:			
Phthalate Esters	EPA 506	None	14 days
Benzo(a)pyrene	EPA 550	None	7 days
Formaldehyde	EPA 8315	None	3 days
Nitroaromatics/Nitroamines (Explosives)	EPA 8330	None	7 days
Polynuclear Aromatic Hydrocarbons	EPA 610/8310	None	7 days
Acrolein & Acrylonitrile	EPA 8316	None	14 days
Phenols	EPA 420.1	H_2SO_4	28 days
PCBs:			
Polychlorinated Biphenyls (PCBs)-water	EPA 508/608/8082	None	7 days
Polychlorinated Biphenyls (PCBs)-oil	EPA 8082	None	7 days
Pesticides and PCBs	EPA 8080/508/608	None	7 days
PESTICIDES/HERBICIDES:			
Carbamates	EPA 531/632	None	7 days
Chlorophenoxyacid Herbicides	EPA 8151A/515	None	7 days
Organochlorine Pesticides	EPA 8081A/508	None	7 days
Organophosphorus Pesticides	EPA 8141A	None	7 days
Triazine Pesticides	EPA 507	None	7 days
GC/MS:			
Volatile Organic Compounds	EPA 624/8240/8260	HCl	14 days
Volatile Organic Compounds-drinking water	EPA 524.2	HCl	14 days, 24 hrs w/o HCl
Semi-volatile Organic Compounds	EPA 625/8270	None	7 days
Phenols	EPA 625/8270	None	7 days
Phthalate Esters	EPA 625/8270	None	7 days
METALS ANALYSIS:			
Metals	EPA 6010/7000/200	HNO_3	6 months
Mercury	EPA 245.1/7040	HNO_3	28 days
Hexavalent Chromium	EPA 7196/7199/218.6	None	24 hours

TABLE 2-1

ANALYSIS	METHOD	PRESERVATIVE	HOLD TIME
INORGANIC ANALYSIS:			
Alkalinity	EPA 310.1	None	14 days
Ammonia	EPA 350.2	H_2SO_4	28 days
BOD	EPA 405.1	None	48 hours
Bromide	EPA 300.0	None	28 days
Chlorate	EPA 300.0	None	28 days
Chloride	EPA 300.0	None	28 days
COD	EPA 410.4	H_2SO_4	28 days
Color	EPA 140.1	None	48 hours
Conductivity	EPA 120.1	None	28 days
Corrosivity-Langlier Index	SM 2330B	None	14 days
Cyanide	EPA 335.2/9010	NaOH	14 days
Dissolved Oxygen	EPA 360.1	None	Immediate
Flash Point	EPA 1010	None	N/A
Fluoride	EPA 340.2	None	28 days
Hardness	EPA 200.7	HNO_3	6 months
Iodide	EPA 300.0	None	28 days
MBAS Surfactant	EPA 425.1	None	48 hours
Nitrate	EPA 300.0/353.2	None	48 hours
Nitrite	EPA 300.0/353.2	None	48 hours
Odor	EPA 110.2	None	24 hours
Ortho-phosphate	SM 4500-P	None	48 hours
Paint Filter Liquids Test	EPA 9095	None	N/A
Perchlorate	EPA 300.0/314.0	None	28 days
Percent Moisture	SM 2540B		
pH	EPA 150.1/9040	None	Immediate
Total Phosphorus	SM 4500-P	H_2SO_4	28 days
Reactivity	SW 846	None	N/A
Silica	EPA 200.7/6010	None	28 days
Specific Gravity	SM 2710F	None	N/A
Sulfate	EPA 300.0	None	28 days
Sulfide	EPA 376.2/9030	Zn-Ac	7 days
Total Dissolved Solids	EPA 160.1	None	7 days
Total Kjeldahl Nitrogen	EPA 351.3	H_2SO_4	28 days
Total Organic Carbon	EPA 415.1	H_2SO_4	28 days
Total Settleable Solids	EPA 160.5	None	48 hours
Total Solids	EPA 160.3	None	7 days
Total Suspended Solids	EPA 160.2	None	7 days
Turbidity	EPA 180.1	None	48 hours
MICROBIOLOGY:			
Coliform bacteria-drinking water	MMO-MUG/SM 9221	$Na_2S_2O_3$	24 hours
Coliform bacteria-waste water	MMO-MUG/SM 9221		8 hours
Heterotrophic Plate Count	SM 9215	$Na_2S_2O_3$	24 hours
Fecal Streptococci	SM 9230	$Na_2S_2O_3$	8 hours (24hrs D.W.)

TABLE 2-1

SAMPLE LOG-IN FORM

CLS# **S**_____ COC# _____Date received _____

Received by: _____

Delivered by: _____

Packaging: cooler _____ DOT packing _____ other (specify) _____

Did package come with shipping slip? . . . . . yes no

If yes, tracking number : _____

Were custody seals present? . . . . . yes no

If yes, how many?_____ Date _____ Signed?. . . . yes no Intact? . . . . yes no

Custody papers sealed? . . . . . yes no Inside packaging? . . . . . yes no

Did sample containers arrive intact?. . . . . yes no

If no, list any samples broken _____

Shipping preservation: blue ice_____ ice_____ none_____ other_____

Temperature upon receipt: _____ °C IR/THERM Blank . . . yes no

Geiger counter test . . . . . Negative Positive

Chain of custody filled out properly? . . . . . not present yes no

Did receiver sign chain of custody in correct place? . . . . . yes no

Were sample labels complete?

(ID, date, time, signature, preservative) . . . . . yes no

Did sample labels agree with chain of custody? . . . . . yes no

Were correct containers used for tests indicated? . . . . . yes no

If no, was pink slip filled out noting discrepancy? . . . . . yes no

Also, if no, was customer called and notified? . . . . . yes no

Were correct preservatives verified?. . . . . N/A yes no

If yes, pH of non-volatile samples _____

Was sufficient amount of sample delivered? . . . . . yes no

Were bubbles absent in VOA samples? . . . . . N/A yes no

Date samples were logged in _____ By whom:_____

Additional comments: _____

Figure 2-2

3.0 MATERIALS AND STANDARDS / SOURCES AND PREPARATION

The quality of reagents, solvents, gases, water and laboratory vessels used in analyses must be known so that their effect upon analytical results can be defined. Materials purchased by CLSB meet the requirements stated below or as denoted in specific analytical procedures and are controlled as stated. Requirements must also be met for internally prepared materials such as water.

The Quality Control Manager or other person as assigned by the Laboratory Director will retain responsibility for purchasing materials and controlling them in the laboratory. The duties of the materials coordinator include:

- Specifying in purchase orders suitable grades of materials (grade should be defined by the QC Manager or Laboratory Director).
- Verifying upon receipt that materials meet requirements and that, as applicable, material certificates are provided and maintained in the laboratory record system.
- The identification and proper storage of materials.
- Verifying that material storage is properly maintained and material is removed from use when expired.

3.1 REQUIREMENTS FOR REAGENTS, SOLVENTS, AND GAS

Chemical reagents, solvents and gas are available in a variety of grades of purity, ranging from technical grade to ultra-pure grades. The purity required varies with the analytical method. The parameter measured, and the sensitivity / specificity of the detection system are important factors in determining the purity of the reagents required.

Standards are obtained from NIST and commercial sources and are traceable to EPA, NIST CRADA (Cooperative Research And Development Agreement) or A2LA (American Association for Laboratory Accreditation). Our commercial suppliers include credible companies such as Ultra-Scientific, Chem Service, VWR, Aldrich,...etc. Certificates for all standards are obtained and kept in a department log book/file that is available for review and inspection. If two sources of a standard are used, at least one standard shall have a certificate and the other shall be traceable to the certified standard through comparative study. All standards, stock or working, are labeled by their name and traceable to the standard logbooks. Expiration dates are also found on the labels and in the logbooks. No expired standards shall be used. All logbooks pertaining to standards and standard traceability are audited by the QA/QC department.

Carrier gas, solvents, acids and deionized water are checked on a batch wise basis. In this way it is possible to avoid systematic contamination of samples without repeating a set of samples, as would be the case if we relied only on method blanks to detect such contamination.

3.1.1 General Inorganic Analyses

In general, Analytical Reagent Grade (AR) reagents and solvents are adequate for inorganic analyses. Primary standard reagents shall be used for standardizing all volumetric solutions. All prepared reagents shall be checked for accuracy.

Individual analytical methods specify the reagents that require frequent standardization or special treatment. To minimize potential deterioration, the analyst should prepare a limited volume of such reagents, depending on the quantity required over a given period of time.

3.1.2 Trace Metals Analyses

All standards used for atomic absorption and emission spectroscopy shall be spectro-quality. It is recommended that other reagents and solvents also be spectro-quality, although, in some cases, AR grade may be satisfactory. Standards are prepared by the analyst, or purchased directly from suppliers provided the materials meet the requirements of the analytical method.

In general, fuel and oxidant gas used for atomic absorption can be commercial grade.

Compressed air can be commercially supplied, zero grade or supplied by laboratory air compressors if adequate pressure is maintained and the air is filtered to remove oil, water and possible trace metals.

3.1.3 Organic Chemical Analyses

AR is the minimum acceptable grade for materials used for organic analyses; use reference grade standards only as necessary. Special note should be made of the assay of standard materials.

Some GC detectors require that solvents, standards and samples be free of certain classes of compounds. For example, use of the flame photometric detector requires that reagents and solvents be free of sulfur and phosphorus interference.

Pesticide-quality solvents are required for low-concentration work. AR grade solvents are adequate for analyzing industrial waste samples. However, the contents of each solvent lot must be checked to determine suitability for the analyses. Similarly, all analytical reagents and other chemicals must also be routinely checked.

3.1.4 Water

Deionized water is used for dilution, preparation of reagent solutions and final rinsing of glassware. Water quality shall be determined daily by measuring specific conductance and shall be recorded in a logbook. A resistance equal to or greater than 18.3 megohms/cm at 25° C is required. This is equivalent to less than 0.1 mg/L of ionized material.

Organic-free water is required for microbiological and volatile organic analyses. Organic-free water may be verified by the purge-and-trap technique on the GC.

When determining trace organics by solvent extraction and gas chromatography, specialty water such as HPLC grade water with sufficiently low background must be used. Pre-extraction of the water with the solvent used in the analysis may be helpful in eliminating organic compounds in the water.

3.1.5 Compressed Air

Compressed air must be free of oil, water and dirt and of high quality, dry grade. The usual quality of compressed air for laboratory use is Ultra Zero.

3.2 CONTAINERS

Containers used in the laboratories can affect the quality of results. Material composition, volumetric tolerances and cleaning are important considerations in laboratory containers. Sample containers are discussed in Section 2.1.

3.2.1 Composition of Laboratory Containers

Soft glass containers are not recommended for general use, especially for the storage of reagents. The glass recommended for general use is chemically resistant borosilicate glass, such as is manufactured under the trade names of Pyrex or Kimax. This glassware is satisfactory for analyses performed by CLSB unless otherwise noted in the sampling or testing procedure. The use of plastic vessels, containers and other apparatus made of Teflon, polyethylene, polystyrene and polypropylene is desirable for certain specified applications.

The following guidelines should be considered when selecting the material composition of laboratory vessels:

- Borosilicate or polyethylene bottles are to be used for the storage of reagents and standard solutions, unless otherwise specified.

- Plastic containers should not be used for reagents and solvents in organic analyses.

- Dilute metal solutions have a tendency to plate out on container walls over long periods of time; therefore, standard solutions should be prepared at the time of analysis.

- The use of disposable glassware is satisfactory for some analyses, such as the use of disposable test tubes for use with some automatic samplers.

- Plastic bottles of polyethylene and Teflon are satisfactory, in general, for the shipment of water samples. However, strong mineral acids such as sulfuric acid and organic solvents readily attack polyethylene.

- Borosilicate glassware is not completely inert, particularly to alkalis. Standard solutions of silica, boron and the alkali metals should be stored in polyethylene bottles.

3.2.2 Volumetric Container Specifications

CLSB shall use glassware of sufficient accuracy as required for each analytical procedure. This includes volumetric flasks, volumetric pipettes and accurately calibrated burets. Less accurate types of glassware, including graduated cylinders and serological and measuring pipettes have specific uses when less exact volumes are permitted by the analytical procedure.

In general, volumetric containers will not be calibrated by CLSB unless required by a specific analytical method. However, volumetric glassware shall be purchased with the objective of meeting the correct end use of the container in an analytical procedure.

3.2.3 Glassware Cleaning Requirements

Methods of cleaning glassware are selected according to the substances that are to be removed and the analytical analysis required.

For inorganic analytical uses, all glassware will be placed into detergent water immediately after use and must not be allowed to dry. After a thorough soaking, glassware will be scrubbed and rinsed at least 3 times with DI water. Glassware will also be rinsed twice with 5% nitric acid solution and then rinsed with D.I. water, air dried and stored in an upright position.

Glassware used for phosphate determinations will not be washed with detergents containing phosphates. This glassware must be thoroughly rinsed with tap water and deionized water. For ammonia and Kjeldahl nitrogen determination, the glassware must be rinsed with ammonia-free water.

Glassware used in the determination of trace organic constituents in water should be as free as possible of organic contaminants. Glassware used for organic analysis should be soaked in hot water containing detergent for two hours, then scrubbed, re-soaked in chem-solve for two hours, rinsed with D.I. water and allowed to dry. Once the glassware has dried it will be rinsed with methanol, air-dried and stored with open end sealed with aluminum foil.

Sampling bottles are all purchased certified clean, but if not they will follow the above procedure for cleaning, depending on the analysis requested. Bottles used for the collection of samples for organic analyses are rinsed successively with acid cleaning solution, tap water, deionized water, and, finally, several times with a redistilled solvent such as acetone, hexane, petroleum ether or chloroform. Caps should be washed with detergent, rinsed with tap water, deionized water and solvent. Liners are treated in the same way as bottles and are stored in a sealed container.

Alternate methods for cleaning may be used if it is demonstrated (such as by blank analysis) that the result is satisfactory. Also, disposable glassware may be used if applicable to the analytical procedure.

3.3 STORING AND MAINTAINING REAGENTS AND SOLVENTS

The following shall apply for storing and maintaining reagents and solvents:

- All standards and reagents will be logged into the standard logbook, and the work standard logbook, upon receipt or formulation of.
- Standard reagents and solvents are stored in accordance with the manufacturer's recommendations.
- Light-sensitive standard reagents or solvents are stored in a cool, dark place.
- Organic reagent standards are stored at 4° C ± 2°.
- Organic reference materials are stored at 4° C ± 2°.
- Standards are not maintained longer than recommended by the manufacturer or as specified in the analytical method.

4.0 INSTRUMENT CALIBRATION, MAINTENANCE AND REPAIR

Modern environmental chemical analysis is heavily dependent on properly maintained and calibrated instruments. The sensitivity and reliability of these high precision instruments require periodic maintenance and calibration to assure precise and accurate measurements. Therefore, CLSB standard procedures include routine instrument calibration and maintenance.

4.1 CALIBRATION

The calibration program verifies that equipment is of the proper type, range, accuracy and precision to provide data compatible with specified requirements. All instruments and equipment that measure a quantity, or whose performance is expected at a stated level, are subject to calibration.

This section of the QA Manual prescribes the practices used by the laboratory to implement a calibration program. Implementation is the responsibility of the laboratory management and analysts. The Quality Assurance Manager shall review the implementation of the program.

Two types of calibration are discussed in this section:

- Operational calibration is routinely performed as part of instrument use, such as the development of a standard curve for use with an atomic absorption spectrophotometer. Operational calibration is generally performed for instrument systems.

- Periodic calibration that is performed at prescribed intervals for equipment, such as balances and ovens. In general, equipment that can be calibrated periodically is a distinct, single purpose unit and is relatively stable in performance.

4.1.1 Calibration Program

The program of calibration for laboratory instruments contains the following elements:

4.1.1.1 Calibration Procedures

Whenever possible, recognized procedures, such as those published by ASTM or the USEPA, or procedures provided by manufacturers, shall be used by CLSB. If established procedures are not available, a procedure shall be developed considering the type of equipment, stability characteristics of the equipment, required accuracy and the effect of operational error on the quantities measured. As a minimum, the procedures shall include:

- Equipment to be calibrated
- Reference standards used for calibration

- Calibration technique and sequential actions
- Acceptable performance tolerances
- Frequency of calibration
- Calibration documentation format

4.1.1.2 Calibration Frequency

Instruments and equipment shall be calibrated at prescribed intervals and/or as part of the operational use of the equipment.

Frequency shall be based on the type of equipment, inherent stability, manufacturer's recommendations, values provided in recognized standards, intended use, effect of error upon the measurement process, and prior experience. Calibration frequency is given in the method working SOP's that are in every CLSB laboratory.

4.1.1.3 Calibration Reference Standards

Two types of reference standards are used within every CLSB laboratory for calibration:

- PHYSICAL STANDARDS such as weights for calibrating balances and certified thermometers for calibrating working thermometers and ovens. These are generally used for periodic calibration.

- CHEMICAL STANDARDS such as Standard Reference Materials (SRMs) provided by the National Institute of Standards and Technology (NIST), EPA check standards, laboratory control standards or working (calibration) standards.

Whenever possible, physical reference standards shall have known relationships to nationally recognized standards (e.g., NIST) or accepted values of natural physical constants. If national standards do not exist, the basis for the reference standard shall be documented.

Physical reference standards shall be used only for calibration and shall be stored separately from equipment used in analyses.

In general, physical reference standards shall be at least four to ten times as accurate as the requirements for the equipment that they are used to calibrate; physical standards should be recalibrated every three years by a certified external agency.

Whenever possible, chemical reference standards shall be directly traceable to NIST SRMs. If SRMs are not available, compounds of certified high purity will be used to prepare calibration standards.

4.1.1.4 Calibration Records

Records shall be maintained for each piece of equipment subject to calibration. Records demonstrating accuracy of reference standards shall also be maintained.

Records for periodically calibrated equipment shall include, as appropriate:

- Identification number of equipment and type of equipment
- Calibration frequency and acceptable tolerances
- Identification of calibration procedure used
- Date calibration was performed
- Identity of CLSB personnel and/or external agencies performing calibration
- Reference standards used for calibration
- Calibration data
- Certificates or statements of calibration provided by manufacturers and external agencies, and traceability to national standards
- Information regarding calibration acceptance or failure and any repair of failed equipment

Records for periodically calibrated equipment shall be maintained by the Instrument or in a secure location that is accessible.

For instruments and equipment that are calibrated on an operational basis, calibration generally consists of determining instrumental response against compounds of known composition and concentration or the preparation of a standard response curve of the same compound at different concentrations. Records of these calibrations are maintained in several ways:

- The calibration data are kept with analytical sample data, and/or
- A logbook is prepared for each instrument that contains all calibration data.

The former method provides response factor information directly with the analytical raw data so that the data can be readily processed and verified. The latter method provides an ongoing record of the calibration undertaken for a specific instrument.

CLSB LABS

ION CHROMATOGRAPHY RUN LOG
EPA M300.0, IC 101

Date: _____ Analyst: _____ Anion: _____

Check Stnd ID: _____ Spike Stnd ID: _____ Calibration Date: _____

	SAMPLE ID	DF		SAMPLE ID	DF
1			25		
2			26		
3			27		
4			28		
5			29		
6			30		
7			31		
8			32		
9			33		
10			34		
11			35		
12			36		
13			37		
14			38		
15			39		
16			40		
17			41		
18			42		
19			43		
20			44		
21			45		
22			46		
23			47		
24			48		

Batch #:_____

Figure 4-1 Instrument Use Log

4.1.2 Operational Calibration

Operational calibration is performed as part of the analytical procedure. Included are the analysis of a method blank and the preparation of a standard response (standard calibration) curve.

A brief discussion of the analysis of method blanks and preparation of standard curves and guidelines for the major instrument systems within the laboratory follows.

4.1.2.1 General Calibration Procedures

The initial phase of a laboratory-testing program requires the selection and certification of the method best suited for an individual parameter. Certification, or verification, is the elimination or minimization of determinate errors that may be due to analyst error, the use of less-than-optimum equipment, reagents, solvents or gases. The quality of materials, even though they are AR grade or better, may vary from one source to another. The analyst must determine, through the use of reagent and/or solvent blanks, if materials are free from interfering substances that could affect the analysis. Other steps in certifying the method include the determination of a method blank and the preparation of a standard calibration curve.

4.1.2.1.1 Method Blank

After determining the individual reagent or solvent blanks, the analyst defines the method blank to determine if the cumulative blank interferes with the analysis. The method blank is defined by following the procedure step by step, including the addition of all of the reagents and solvents, in the quantity required by the method. If the cumulative blank interferes with the determination, steps must be taken to eliminate or reduce the interference to a level that will permit the combination of solvents and reagents to be used.

A method blank should be determined whenever an analysis is made. The number of blanks is determined by the method of analysis and the number of samples analyzed at a given time.

4.1.2.1.2 Standard Calibration Curve

Concurrent with the preparation of reagent and method blanks, a standard calibration curve is prepared for the instrumentation. Preparation of a standard calibration curve is accomplished by using calibration standards.

Calibration standards are also referred to as "working standards". They are prepared by mixing the species to be analyzed into the solvent that is to be introduced into the instrument.

The concentrations of the calibration standards are chosen to cover the working range of the instrument. All sample measurements are made within this working range. The calibration curve is prepared by plotting instrument response versus concentration of the species analyzed. Actual sample concentrations are then read directly from the calibration curve or determined by interpolation. Data reduction is done manually and/or by electronic data systems.

4.1.2.2 Calibration of the Gas Chromatograph and Gas Chromatograph/Mass Spectrometers

Calibration of the gas chromatographs or gas chromatograph/mass spectrometers for organic compound analyses is performed simultaneously with the standardization of the instrument. A five-point standard curve is initially analyzed to calibrate instrument response and to define the working range of the instrument for the compounds of interest.

After initial calibration is established, mid-point calibration standards are run to confirm continuing instrument calibration. The acceptance criteria are method specific and are strictly adhered to.

Response Factors (RF) are to be calculated for each compound at each concentration level (acceptable response factors are given in the individual method SOP's). These RF will be averaged to generate the mean RF for each compound over the range of the standard curve. The mean response factor will be used to calculate the sample concentration of the compound of interest. When sample responses exceed the range of the standard curve, the sample will be diluted to fall within the range of the standard curve and be reanalyzed. The results of the daily GC standardization will be tabulated and filed with the corresponding sample analyses.

4.1.2.3 Calibration of Inductively Coupled Plasma Spectrometer (ICP) and Atomic Absorption Spectrophotometer (AA)

The ICP and AA are standardized for the metal of interest by the analysis of a set of calibration standards prepared by diluting a stock solution of known concentration. Working standards are prepared by dilution of the stock standard. The concentration of the calibration standards is chosen so as to cover the working range of the instrument. Subsequently, all sample measurements are made within this working range. Once the working standards are prepared, they are analyzed on the ICP or AA, and the instrument response is calibrated to provide a direct readout in milligrams of metal per liter of water.

The calibration is accomplished by inputting the metal concentration equivalent to the readout in absorbance units during analysis of the working standards.

Once the instrument has been initially calibrated, the analysis of the working standards is repeated during sample analysis to standardize instrument response during analysis and confirm the calibration settings. A typical analysis sequence is as follows:

- Working standards are prepared by dilution of a stock standard solution for the metal of interest.
- A calibration curve within the working range of the instrument is established by analysis of five working standards.
- The working standards are reanalyzed to confirm the calibration settings. If the calibration settings are not confirmed, the instrument is recalibrated.
- The samples are analyzed for the metal of interest.
- During sample analysis, a midpoint standard is analyzed to monitor instrument stability. If the analysis indicates that instrument calibration has changed, the instrument is recalibrated and the analysis is repeated.
- Following completion of the sample analyses, the working standards are reanalyzed to confirm calibration settings. If calibration settings are confirmed, the analysis is completed. However, if the calibration settings are not confirmed, the problem is corrected and the analyses are repeated.
- Analysis data may be input (if available) into a computer data file for later calculation and normalization for matrix effects.

4.1.3 Periodic Calibration

Periodic calibration shall be performed for equipment such as balances, thermometers, ovens and furnaces that are required in analytical methods, but that are not routinely calibrated as part of the analytical procedure. Documentation of calibration shall be kept for each equipment item.

Calibration requirements are determined within the laboratory depending upon the equipment used and its operating function. Following is an example for the calibration of balances with examples of a calibration data sheet to serve as a guideline for the preparation of laboratory-specific procedures.

4.1.3.1 Balances

All balances shall be calibrated weekly using weights traceable to the National Institute of Standards and Technology (NIST). Calibration weights shall be Class S or better. Balances are calibrated by an external agency three times per year.

Calibration of balances shall be to approximately 95 percent of balance capacity. Acceptance for balances that are direct reading to 0.01 gram shall be ± 0.01 g for 0 to 100 g and ± 0.1 percent of the applied weight for more than 100 g. Figure 4-2 provides an example data sheet that can be used for balance calibration.

Balance #: _____ Balance Name:_____ Balance S/N:_____

Date	Check Weight	Balance Reading	In Range? *	Comment	Initial

* Tolerance ranges are in the front of this logbook

Figure 4-2 Balance Log

4.2 INSTRUMENT MAINTENANCE AND REPAIR

The purpose of instrument maintenance is to maintain proper equipment performance and to prevent instruments and equipment from failing during use. An adequate maintenance program increases reliability of a measurement system and will include equipment cleaning, lubricating, reconditioning, adjustment and/or testing.

Within the laboratory, the Laboratory Director is responsible for preparation and documentation of the program. Department Supervisors shall implement the program, and the QC Manager shall review implementation to verify compliance.

CLSB's maintenance program considers several factors:

4.2.1 Instruments, equipment and parts that are subject to wear, deterioration or other change in operational characteristics without periodic maintenance.

4.2.2 The availability of spare parts within the laboratory to minimize downtime.

4.2.3 Frequency that preventive maintenance is required.

Preventive maintenance is performed on a routine basis and documented by department supervisors in a maintenance logbook assigned to each instrument. It should be noted if parts are replaced or if the instrument has deteriorated from use, etc. Figure 4-3 illustrates one type of maintenance log currently in use.

Instrument ID: _____

Date	Time	Name	Maintenance/Service/Parts/Remarks

Figure 4-3 Instrument Maintenance Log

5.0 QUALITY CONTROL SAMPLE ANALYSIS

This section discusses samples that are routinely added to the normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. Quality control samples are of known content and concentration (with the exception of field blanks) to ensure that accuracy and precision can be determined and control charts can be prepared. Evaluation of this data is discussed in Section 8.1.

The following is a discussion of the major types of quality control samples. QC samples will be analyzed as recommended herein, unless analytical procedures prescribe other specific QC sample analysis. If the procedure is specific, the procedural requirements will be met.

As stated, Section 8.1 presents the statistical analyses of these samples.

5.1 ANALYSES AND FREQUENCY OF BLANKS

5.1.1 Trip Blank Analyses

Volatile organics samples are susceptible to contamination by diffusion of organic contaminants through the Teflon-faced silicone rubber septum of the sample vial; therefore, trip blanks shall be analyzed to monitor for possible sample contamination during shipment. Trip blanks will be prepared by filling two VOA vials with organic-free water and shipping the blanks with the field kit. Trip blanks accompany the sample bottles through collection and shipment to the laboratory and are stored with the samples. Following the analyses, if the trip blanks indicate possible contamination of the samples, depending upon the nature and extent of the contamination, the samples may be corrected for the trip blank concentration or the sources re-sampled.

Results of trip blank analyses should be maintained with the corresponding sample analytical data in the project file.

5.1.2 Method Blank Analyses

A method blank is a volume of deionized laboratory water for water samples, or a purified solid matrix for soil/sediment samples, carried through the entire analytical procedure. The volume or weight of the blank must be approximately equal to the sample volume or sample weight processed. A method blank should be performed with each analytical batch of samples. Analysis of the blank verifies that method interferences caused by contaminants in solvents, reagents, glassware and other sample processing are known and minimized. Results of method blank analyses will be maintained with the corresponding analytical data in the project file. For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank of any analyte of concern must be no higher than the detection limit.

5.1.3 Holding Blank Analyses

On a regular basis, a set of VOA vials containing organic-free water are prepared and stored along with client samples in the VOA-storage refrigerator. After one week, the holding blanks are analyzed by GC for possible organic contamination. If compounds are detected, the QC Manager will initiate a corrective action.

5.2 ANALYSES AND FREQUENCY OF REPLICATES

5.2.1 Replicate Sample Analyses

Replicate analyses are performed to evaluate the precision of an analysis. Results of the replicate analyses are used to determine the relative difference between replicate samples. Criteria for evaluating replicate sample results are provided in Section 8.1. A replicate analysis should be performed on every group of twenty samples analyzed. Replicate analysis results should be summarized on the quality control data summary form.

The frequency of replicates is specified in many analyses, and CLSB analyzes, as a minimum, the percentage of replicate specified. The replicate aliquots are carried through the entire workup and analytical process.

Care is taken to assure that soils and hazardous wastes are replicated at least as frequently as waters and wastewaters.

5.2.2 Blind Replicate Analysis

A blind replicate sample is a replicate sample that has been introduced as a separate sample by the Quality Control Manager during the log-in process or prior to analysis. Evaluation of the replicate is discussed in Section 8.1. This data is reported to and summarized by the Quality Control Manager.

5.3 ANALYSES AND FREQUENCY OF SPIKED SAMPLES

Samples are spiked with known amounts of chemical entities being measured in order to determine the percent recovery.

5.3.1 Matrix Spikes

At least one matrix spike (MS) and one matrix spike duplicate (MSD) will be analyzed per analytical batch (if not enough sample is available for a matrix spike, then a Laboratory Control Sample and a Laboratory Control Sample Duplicate will be used as QC samples). A matrix spike is defined as a sample matrix that has predetermined quantities of stock solutions of certain analytes added prior to sample extraction/digestion and analysis.

To evaluate the effect of the sample matrix upon analytical methodology, a separate aliquot sample should be spiked with the analyte of interest and analyzed with the sample. The percent recovery for the respective analyte will then be calculated.

If the percent recovery falls outside quality control limits, the data should be evaluated and the sample reanalyzed if criteria are not met. Matrix spike results should be summarized on the quality control data summary sheets.

5.3.2 Regulatory Spikes

When sample analysis requires values within a specified percent recovery of a regulatory limit, the sample will be spiked with a standard of concentration (suggested in the method) and the spiked sample analyzed. Recoveries are calculated and reported on a percent basis. In this manner, the spike serves to provide information on accuracy of the procedure.

5.3.3 Replicate Spikes

Certain methods specify running replicate spikes. A regulatory spike is a subpart of replicate spike. Frequently, the replicate spike is run at one to five times the concentration of the observed sample value or at one to five times the background level, depending on method requirements.

5.4 STANDARDS AND REFERENCE MATERIALS

Standards and reference materials will be obtained per procedures specified in Section 3. Proper laboratory procedure requires the use of the following types of standards and reference materials.

5.4.1 Quality Control Samples

A Liquid Control Sample (LCS) will be processed with each analytical batch. A LCS is defined as a known matrix spiked with compound(s) representative of the target compounds, which is run through the entire analytical procedure. Only the LCS needs to be reported, if the MS or MSD fail their parameters. The results of the LCS are compared to control limits established for both precision and bias to help determine the usability of the data.

5.4.2 Working or Calibration Standards

Calibration of instruments such as GC, ICP, and AA requires use of standard solutions. These calibration standards are carefully prepared by volumetric or gravimetric methods and standardized against the laboratory control standards before use in the laboratory.

Because instrument response and calibration curves are subject to change and can vary from day to day, a midpoint standard or check standard will be analyzed at the beginning of analysis, every 10 samples thereafter and at the end of sample analysis.

Analysis of this standard is necessary to verify the standard curve and may serve in some cases to be sufficient for calibration. This value should be entered in the instrument calibration log whenever performed.

5.4.3 Internal Standards

An internal standard is a known amount of a compound not normally found in environmental samples added to each sample before analysis. The area of internal standard peak in the calibration standard is used as a reference to monitor the area of the internal standard peak in the sample. The internal standard peak in the sample is the reference peak used for the calculation of the concentrations of the compounds present in the sample. The retention time of the internal standard is monitored by the analyst to ensure there is minimal change.

5.4.4 Surrogates

A surrogate is an organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. Surrogates are typically spiked into the samples prior to extraction and thereby provide recovery data for sample workup. Although such data is typically derived from compounds closely related to the compounds under investigation, it is not compound-specific and in the strict sense should not be used for making corrections for recovery. Since the information is provided with every sample, it is nevertheless very useful in detecting both sample-specific and systematic recovery problems. Surrogates will be run on all organic analysis including spikes and blanks. If surrogate recoveries exceed their specified control limits corrective action will be implemented as specified in the individual method SOPs.

5.4.5 Certified Reference Materials

On a regular basis, the Quality Control Manager should introduce a group of prepared verification samples (Certified Reference Materials) into the analytical testing regime. Results of these data will be summarized, evaluated and presented to laboratory management for review and corrective actions, if appropriate. The data are reported to and summarized by the Quality Control Manager. Certified Reference Materials are acquired from sources which are accredited by the State of California to provide such samples.

5.4.6 QC Batches

A number of samples of similar matrix, origin and composition which are analyzed together with the same method sequence and the same lots of reagents with manipulations common to each sample within the same time period or in continuous sequential time periods shall be known as a QC batch.

The number of total samples in a QC batch should not exceed twenty samples plus the number of samples required to perform QC evaluation of the twenty initial samples.

A sequential number will be assigned to each QC batch prior to batching a number of samples. This number will be obtained by using the next sequential number available as recorded in the QC Batch Log Book.

The QC Batch Log Book will contain information pertaining to each QC batch and will include the sequential batch number, date, analysis to be performed, analyst who assigned the batch, the sample numbers to be analyzed, and additional notes as required.

5.5 INTER/INTRA-LABORATORY PERFORMANCE EVALUATIONS

The performance of CLSB's laboratories is monitored by the participation in the Environmental Laboratory Accreditation Program of the State of California. This program includes the Water Pollution (WP) and Water Supply (WS) programs. Under these programs, blind samples with known concentrations are analyzed annually. CLSB obtains intra-laboratory PE samples from commercial companies such as Environmental Resource Associates.

The chemists, the Laboratory Manager and the Laboratory Director are kept informed of all inter/intra-laboratory performance evaluations. If a method fails or is found to be suspect, appropriate corrective actions are taken immediately. If any results are found to be outside the established control limits, the method will be evaluated and the problem resolved prior to performing any additional tests.

6.0 PERFORMANCE AND SYSTEMS AUDIT

A QA audit is an independent assessment of the measurement system. The purpose of the performance audit is to qualitatively and quantitatively assess the data output generated at any level within the laboratory during the data collection. The results of the audit are formulated into a report detailing the overall system performance and deficiencies, plus any recommendations.

6.1 QUALITY ASSURANCE AUDITS

The QA Manager will perform performance audits. Audits are considered an essential part of the CLSB Quality Assurance Program. CLSB conducts two types of audits; a system audit to qualitatively evaluate the operational details of the QA program, and a performance audit to evaluate the quantitative outputs of all measurement systems.

These audits are combined into one summary audit. The audit includes: (1) laboratory inspection to ensure the laboratory, instruments and equipment, etc., are kept in good condition, and all records of standard preparation, calibration, sample preparations, etc., are documented; (2) data validation: selected tests/reports will be audited, and the complete QC package from log-in to report generation will be checked; (3) Assessment of QC sample analysis; (4) Record filing and retreivability. A checklist will be used by CLSB QA personnel when performing audits to assure that nothing is overlooked. Major elements of the audits are listed below:

- o SOPs are available and updated
- o Standards are not expired
- o Lab notebooks have been signed and reviewed
- 0 Instrument performance and logs are updated
- o Properly trained chemist(s) are performing analysis
- o Traceability of all analysis
- o Safety practices of laboratory personnel

The audit results will be documented and given to the laboratory director and all managers, as well as being available for review by the company President.

6.2 SUBCONTRACT LABORATORIES

CLSB periodically sends samples to other laboratories for analysis which are not performed by CLSB. Before CLSB sends samples to a contract laboratory, CLSB requires a current ELAP certificate, QA Manual, and WS/WP results. In addition, CLSB may submit QA samples to assure sample integrity.

7.0 ANALYTICAL PROCEDURES

CLSB utilizes USEPA prescribed methods whenever applicable. Other sources of analytical methods may be used for other analyses if widely recognized by industrial and government laboratories. Industry standard methods are published by USEPA, American Public Health Association (APHA), American Society for Testing and Materials (ASTM), National Institute for Occupational Safety and Health (NIOSH), and American Industrial Hygiene Association (AIHA).

A brief summary of the method sources for performing "certified" analyses, as well as other commonly used references, is located in Table 7-1.

Analysis will be performed in accordance with the methods cited herein unless specific project requirements or needs dictate modification of the cited methods or adoption of an alternate method. If analysis is performed in an alternate manner, the method shall be documented in the project records.

Accurate environmental analysis involves the need for several activities to be performed in coordination with or coincidental to actual analysis; e.g. 1.) sample procurement and storage (Section 2) to preserve sample integrity, 2.) Instrument calibration, 3.) Analysis of QC samples and standards to assess recovery, matrix affects, range within linearity, 4.) Extraction of the analytes from the matrix. Each of these aspects is discussed elsewhere in this manual.

7.1 SOPs

CLSB relies heavily on the use of Standard Operating Procedures (SOPs). CLSB's SOPs not only include the instrumentation and method procedures but also include all aspects of the complete analytical process, from sample receipt to waste disposal.

No procedure or task is accepted for use until an appropriate SOP has been written and approved by both the QA/QC Manager and Laboratory Director. The QA/QC Manager reviews all SOPs annually. SOPs are kept in the appropriate lab areas, readily available to each analyst.

All laboratory method SOPs should include the following elements if applicable:

1) Title of the method.
2) Effective date of the method.
3) Scope and application including a list of analytes, matrices and detection limits.
4) Summary of the method.
5) Definition of terms.
6) Health and safety considerations.
7) Sample handling and preservation considerations.
8) Effect of potential interferences.
9) Apparatus and materials including reagents, equipment and instruments.
10) Quality control criteria defined in detail .
11) Procedures for the analysis of samples.
12) Documentation: a list of items to be included in the project folder.
13) References for Method .
14) A sample run log is attached to SOP.
15) Standards Preparation is in SOP in detail for CAL ,CCV,LCS , MS &MSD.

SOPs are written in a numbered outline format with the following major headings:

1.0 Purpose
2.0 Scope and Application
3.0 Method Detection Limits
4.0 Applicable Matrx or Matrices
5.0 Method Summary
6.0 Definitions
7.0 Contamination and Interferences
8.0 Apparatus and Materials
9.0 Reagents and Standards
10.0 Sample Collection , preservation , shipment and storage .
11.0 Quality Control
12.0 Calibration and Standarization
13.0 Procedure
14.0 Calculations
15.0 Method Performance

16.0 Pollution Prevention.
17.0 Data Assessment and Acceptance Criteria
18.0 Corrective Actions for Out of Control Data
19.0 Contingencies for Out of Control Data.
20.0 Waste Management .
21.0 References
22.0 Tables , Diagrams , Flowcharts ,etc.
23.0 Training and Qualification Validation
24 0 Health and Safety

Definition and Procedure for the Determination of the Method Detection Limit,□ Code of Federal Regulations (CFR) 40, Part136, Appendix B, Revised July 1995.

Drinking Water Methods from Methods for the Determination of Organic Compounds in Drinking Water,□ EPA 600/4- 88/039, December 1988, Revised July 1991.

Drinking Water Methods from Methods for the Determination of Organic Compounds in Drinking Water-Supplement I,□ EPA 600/4-90/020, July 1990.

Drinking Water Methods from Methods for the Determination of Organic Compounds in Drinking Water-Supplement II,□ EPA 600/R-92/129, August 1992.

Inductively Coupled Plasma-Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes,□ CFR 40, Part 136, Appendix C, Revised July 1995.

Methods for Chemical Analysis of Water and Wastes (MCAWW),□ EPA 600/4-79-020, Revised, March 1983.

Methods for the Determination of Metals in Environmental Samples-Supplement I,□ EPA 600/R-94-111, May 1994.

Methods for the Determination of Inorganic Substances in Environmental Samples,□ EPA 600/R-93-100, August 1993.

"Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater," EPA 600/4-82-057, July 1982.

Methods for Organic Chemical analysis of Municipal and Industrial Wastewater,□ CFR 40, Part 136, Appendix A, July 1995.

"Methods for the Determination of Organic Compounds in Drinking Water", EPA 600/4-88/039, December 1988.

Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater - Volume I,□ EPA 821/R-93-010-A, August 1993, Revision 1.

Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater - Volume II,□ EPA 821/R-93-010B, August 1993.

Method 1664: N-Hexane Extractable Material (HEM) and Silical Gel Treated N-Hexane Extractable Material (SGT- HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons), EPA 821/B-94-0046, April 1995.

Precision and Recovery Statements for Methods for Measuring Metals,□ CFR 40, Part 136, Appendix D, Revised July 1995

"Standard Methods for the Examination of Waste and Wastewater," 18th Ed. APHA-AWWA-WPCF, 1992.

Technical Notes on Drinking Water Methods,□ EPA 600/R-94-173, October 1994

A number of additional methods are summarized in the "AB 1803 Methods Manual" issued by the California Department of Health Services, 1984.1994.

Test Methods for Evaluating Solid Waste,□ USEPA SW-846, December 1996, Third Edition Update III.

Federal Register, 40 CFR Part 136, Oct. 26, 1984.

Table 7-1 Basic References for Analytical Methods

8.0 QUALITY CONTROL DATA PROCESSING AND VALIDATION

Data processing and validation within the analytical laboratory ensure that the reported results will correctly represent the analyses performed. This function has two primary activities:

- The processing of quality control sample results to demonstrate that analyses are within laboratory prescribed bounds for accuracy, precision and completeness.
- Sample reduction and validation to demonstrate that numerical computation of data is correct and that it is correctly reported.

This section discusses the computation process and Section 9.0 discusses maintenance of resulting records.

8.1 PROCESSING OF QUALITY CONTROL DATA

This section discusses the analytical treatment of the data resulting from the quality control samples discussed in Section 5.

8.1.1 Assessment of Data Precision and Accuracy

All data generated must be evaluated for precision and accuracy by the following procedure. Quality control sample analyses are performed as appropriate for organic or inorganic samples as discussed in Section 5. The protocol used will be in accordance with specific method analytical procedures if QC requirements are stated in the procedure.

8.1.1.1 Frequency and Types of QC Samples :

Reagent or Method Blank - A reagent and/or method blank is prepared and analyzed with each batch of samples.

Trip Blank - Trip blanks are analyzed to determine possible sample contamination during collection and shipment to the laboratory. Trip blanks are applicable to volatile organics analysis (VOA) where volatile contaminants can be introduced from ambient air on site, during shipment, and in the laboratory.

Calibration Curve - A calibration curve consisting of standards and a reagent blank are prepared for each parameter. If the standard curve is within acceptance criteria for the method in use, the standard curve will be verified by the analysis of a midpoint standard.

Initial and Continuing Calibration Verification (ICV & CCV) - A Standard of reagent water or solvent that is spiked with a standard of the analytes from a second source standard . It is used to verify the calibration curve initially and continuously . These samples are run every ten samples with CCB. Recovery of the analyte is recorded .

Initial Calibration and Continuing Calibration Blank (ICB and CCB) – A Reagent blank that is run after CCV to check system cleanliness .

Liquid Control Samples(LCS) – A reagent water or solvent that is spiked with analytes from a second source standard . This sample is run through the entire analytical procedure as a sample and recovery of analyte is recorded .

Matrix Spike & Duplicate - As a minimum, one sample in every sample set of twenty samples is spiked twice at a mid-concentration level to provide a final concentration within the expected range of the samples.

Blind Replicate - A blind replicate, unknown to the analyst, is introduced by the Quality Control Department quarterly. Blind replicates are routinely used for the analysis of metals, water quality parameters and organics analyses that do not require separate extraction.

Certified Reference Materials - Certified Reference Materials are introduced at least annually into the testing scheme by the Quality Control Manager to evaluate the testing procedure and the analyst's performance.

Check Standards - A check standard consisting of deionized water spiked with the parameter of interest is analyzed. Check standards are routinely used for the analysis of metals, water quality parameters and some organics parameters.

Surrogate Standard Spike - Every sample is spiked with the required and appropriate surrogate standards prior to extraction and analysis for volatile and semi-volatile organic compounds.

Internal standards - Internal standards are added to samples as prescribed in each specific method.

Quality Control Samples - Quality Control Samples are required twice per analytical batch.

8.1.1.2 Acceptable Limits of QC Samples

When the analyses of a sample set are completed, the results will be reviewed and evaluated to assess the validity of the data set. Review is based on the following criteria:

Method Blank Evaluation - The method blank results are evaluated for high readings characteristic of background contamination. If high blank values are observed, laboratory glassware and reagents should be checked for contamination and the analysis halted until the system can be brought under control. For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank of any analyte of concern must be no higher than the reporting limit.

Trip Blank Evaluation - Trip blank results are evaluated for high readings similar to the reagent and/or method blanks described above. If high trip blank readings are encountered, the procedure for sample collection, shipment and laboratory analysis should be reviewed. If both the reagent and/or method blanks and the trip blanks exhibit significant background contamination, the source of contamination is probably within the laboratory. In the case of VOA, ambient air in the laboratory and reagents should be checked as possible sources of contamination. High trip blank readings for other parameters may be due to contaminated sample bottles or cross-contamination due to sample leakage and poorly sealed sample containers.

Calibration Standard Evaluation - The calibration curve is evaluated to determine linearity through its full range, and that sample values are within the range defined by the low and high standards. If the curve is not linear, sample values must be corrected for nonlinearity by deriving sample concentrations from a graph or by using an appropriate algorithm to fit a nonlinear curve to the standards.

Replicate Sample Evaluation - Replicate sample analysis for the sample set is used to determine the precision of the analytical method for the sample matrix. The replicate results are used to calculate the precision as defined by the relative percent difference (RPD). The precision value, RPD, should be plotted on control charts for the parameter determined. If the precision value exceeds the warning limit for the given parameter, the appropriate Department Supervisor, Laboratory Director or the Quality Control Manager is notified. If the precision value exceeds the control limit, the sample set must be reanalyzed for the parameter in question.

Matrix Spike Evaluation - The observed recovery of the spike versus the theoretical spike recovery is used to calculate accuracy as defined by the percent recovery. The accuracy value, (percent recovery) may be plotted on a control chart for the parameter determined. If the accuracy value exceeds the warning limit for the given parameter, the appropriate Supervisor, Manager or the Quality Control Manager is notified.

Blind Replicate Evaluation - The blind replicate analysis is evaluated in the same manner as described above for the replicate sample analysis and is treated as a replicate result for purposes of evaluating the precision of the analytical method.

Reference Standard Evaluation - Standard Reference Materials analyses are compared with true values and acceptable ranges. Values outside the acceptable ranges require corrective action to determine the source of error and provide correction action. All sample analyses should be halted pending this evaluation. Following correction of the problem, the Standard Reference Material should be reanalyzed.

Quality Control Sample Evaluation - The results of the Quality Control Sample analysis are compared with the true values, and the percent recovery of the sample is calculated. If correction is required, the control sample and the samples in its batch should be reanalyzed to demonstrate that the corrective action has been successful.

Surrogate Standard Evaluation - The results of surrogate standard determinations are compared with the true values spiked into the sample matrix prior to extraction and analysis and the percent recoveries of the surrogate standards are determined. For aqueous matrices, these percent recoveries should be compared with the laboratory generated control limits.

8.1.2 Statistical Evaluation of QC Data

As part of the analytical quality control program, CLSB determines precision and accuracy for each parameter analyzed. These values can be used as control limits for pass/fail criteria.

Initially, when these data are compiled, the evaluation is applied over a broad concentration range. As more data is accumulated, precision and accuracy determinations are updated and criteria developed to define precision and accuracy over specific concentration ranges.

8.1.2.1 Control Chart Evaluation

Precision and accuracy criteria will be applied to each parameter that is analyzed. When analysis of a sample set is completed, the quality control data may be reviewed and evaluated through the use of control charts to validate the data set. Control charts are derived from data that has been entered into the LIMS.

Control charts may be established for all major analytical parameters.

A minimum of seven measurements of precision and accuracy are required before control limits of two standard deviations shall be considered valid. Once established, control limits are updated as additional precision and accuracy data become available by the Quality Control Manager.

8.1.2.1.1 Analytical Precision

General Considerations

To determine the precision of the method and/or laboratory analyst, a routine program of replicate analyses is performed. The results of the replicate analyses are used to calculate the relative percent difference (RPD), which is the governing quality control parameter for precision.

The RPD for replicate analyses is defined as 100 times the difference (range) of each replicate set, divided by the average value (mean) of the replicate set. For replicate results D_1 and D_2, the RPD is calculated from:

$$RPD = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}}$$

When the RPD is obtained for at least seven replicate pairs, the average RPD and the standard deviation are calculated using:

$$\bar{m} = \frac{\sum_{i=1}^{n} m_i}{n}$$

and

$$S_m = \sqrt{\frac{\sum_{i-1}^{n} (m_i - \bar{m})^2}{n-1}}$$

where,

m_i = the RPD of a replicate pair,
m = the average of the Relative Percent Difference determination,
S_m = the standard deviation of the data set of RPD determinations,
n = the number of RPD determinations.

When constructing a control chart for a specific parameter, the Warning and Control Limits are then calculated from the following:

Upper Control Limit = $\bar{m} + 3S_m$
Lower Control Limit = $\bar{m} - 3S_m$
Upper Warning Limit = $\bar{m} + 2S_m$
Lower Warning Limit = $\bar{m} - 2S_m$

A control chart is established by plotting the RPD of each replicate pair on a graph generated as follows:

- The average of the RPD determinations for the original data set is established as the midpoint on the Y-axis of the graph.
- The Upper Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis above the mean of the RPD determinations.
- The calculated RPD of each replicate pair is plotted on the graph to determine whether the RPD is within the Warning and Control Limits of the Control Chart.

If the RPD plots between the Warning and Control Limits, the group leader, laboratory director or quality control manager is notified for a decision as to how to proceed.

- If the RPD plots outside the Control Limits, the data set is invalid and the analysis is stopped until the source of error has been determined and corrective action taken. Once the error source has been resolved, the data set is reanalyzed.

8.1.2.1.2 Analytical Accuracy

When a program for evaluation of analytical accuracy is established, the evaluation is applied over the entire range of spiking concentrations. As more data are accumulated, the evaluation procedure is refined to define the analytical accuracy of the method over specific concentration ranges.

To determine the accuracy of an analytical method and/or the laboratory analysis, a periodic program of sample spiking is conducted. The results of sample spiking are used to calculate the quality control parameter for accuracy evaluation, the Percent Recovery (%R).

The %R is defined as the observed concentration minus the sample concentration, divided by the true concentration of the spike, all multiplied by 100.

$$\%R = \frac{O_i - O_s}{T_1} x100$$

where

%R = The Percent Recovery,
O_i = The Observed Spiked Sample Concentration,
O_s = The Sample Concentration and
T_1 = The True Concentration of the Spike.

When the Percent Recovery is obtained for at least ten spiked samples, the mean percent recovery and the standard deviation are calculated using the formulae:

$$\%\ \overline{R} = \frac{\sum_{i=1}^{n} \%R_i}{n}$$

and

$$S_R = \sqrt{\frac{\sum_{i=1}^{n} (\%R_i - \%\overline{R})^2}{n-1}}$$

where

%R	=	the Mean Percent Recovery
$\%R_i$	=	the Percent Recovery of a Single Spiked Sample,
n	=	the number of results and
S_R	=	the Standard Deviation of the data set of Percent Recovery determinations.

The Warning and Control Limits are then calculated from the following equations:

Upper Control Limit = $\%R+3S_R$
Lower Control Limit = $\%R-3S_R$
Upper Warning Limit = $\%R+2S_R$
Lower Warning Limit = $\%R-2S_R$

A control chart (as shown in Figure 8-1) is generated by plotting the Percent Recovery data on a graph as follows:

- The average of the Percent Recovery determinations for the original data set is established as the midpoint on the Y-axis on the graph.
- The Upper Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis above the mean of the Percent Recovery determinations.

- The Lower Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis below the mean of the Percent Recovery determinations.

- The calculated Percent Recovery of each spiked sample is plotted on the graph to determine whether the Percent Recovery is within the Warning and Control Limits of the Control Chart.

- If the Percent Recovery plots between the Warning and Control Limits, the group leader, laboratory director or quality control manager is notified for a decision as to how to proceed.

- If the Percent Recovery plots outside the Control Limits, the data set is invalid and the analysis is stopped until the source of error has been determined and corrective action taken. Once the source has been corrected, the data set is reanalyzed.

- When an additional ten "Percent Recoveries" have been determined, the Warning and Control Limits are recalculated for the entire data set and the Control Chart for the corresponding parameter is updated.

The Quality Control Manager maintains all control charts.

8.1.2.2 Corrective Action/Out-of-Control Situations

In general, any result falling outside of control limits (generally set at ± 3 standard deviation units) will require initiation of corrective action. Whenever this situation occurs, it will be immediately brought to the attention of the QA Manager and the Laboratory Director.

The nature of corrective action will vary depending on interpretation of the seriousness of the situation by the QA Manager. Isolated outliers may be impossible to explain, and if warranted by previous and subsequent data, the outlier may be ignored. Consecutive recurrence of outliers will be viewed as indicative of a problem situation, and the process will be reviewed.

Most commonly, the out-of-control situation will require a series of corrective measures instituted to re-establish analytical validity. All analysis with the implicated method and instrumentation will be stopped until the problem is identified and resolved.

Review of recent historical data will be made to determine the time of the first variance from valid data, and data collected after that time discarded. Whenever possible all analyses performed after the last valid control check will be repeated.

Immediately following resolution of the out-of-control situation, an increased percentage of spikes and replicates will assure the situation is back to normal. This will continue until the QA Manager is satisfied that total resolution of the problem has occurred.

Control Chart

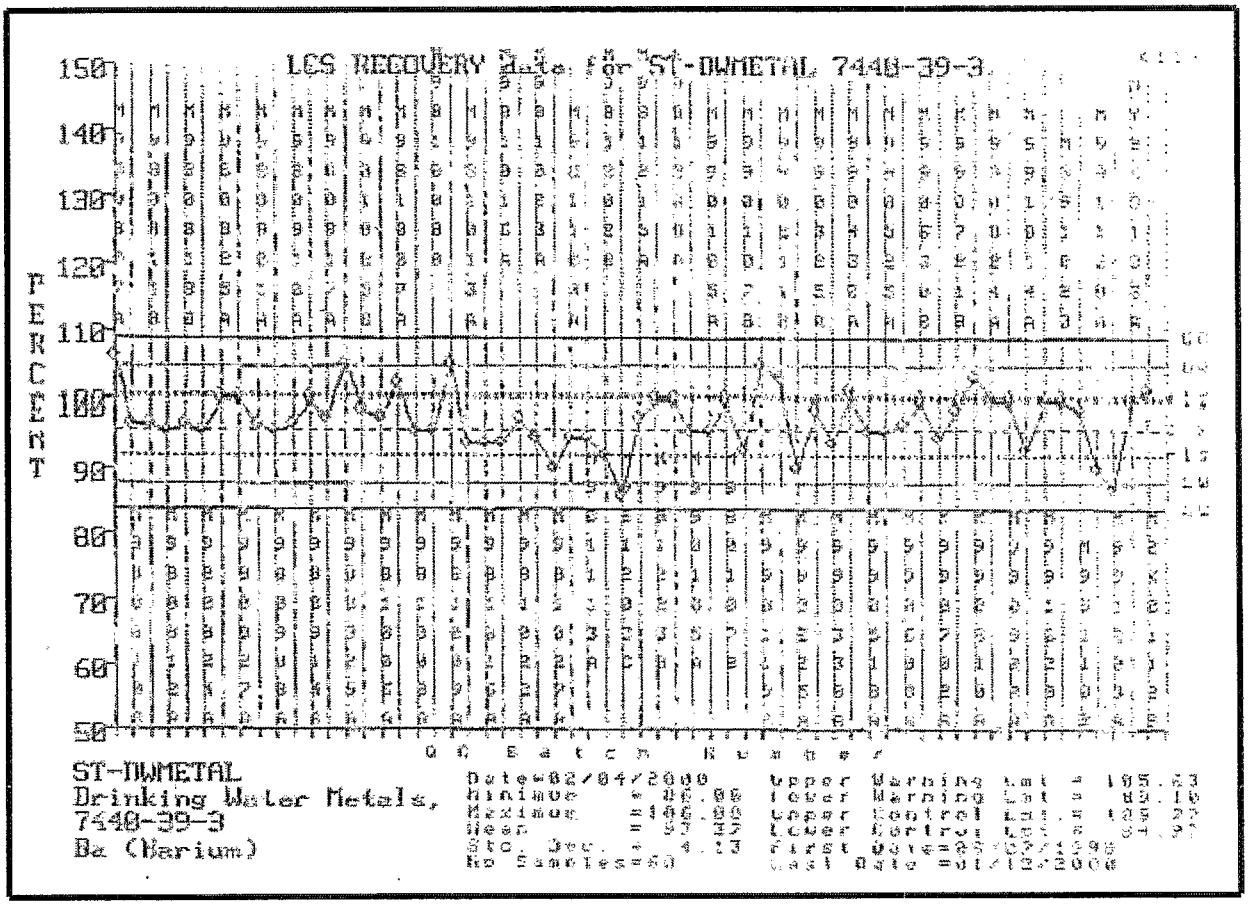


Figure 8-1 QC Control Chart

8.2 DATA VALIDATION

Data validation begins with the processing of data (including QC data) and continues through review of the final data and the reporting of analytical results. The analyst, independent of the data acquisition and processing, will perform data processing. The department supervisor reviews (validates) that the data processing has been correctly performed and continues by verifying that the reported analytical results correspond to the data acquired and processed. Final review of the data to be reported is by the Laboratory Director.

8.2.1 Data Processing

In general, an analyst will process data by:

1. Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets or
2. Input of raw data for computer processing or
3. Direct acquisition and processing of raw data by a data processing system (computer).

If the data is manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors, dilution factors and calibration constants. If calculations are not performed directly on the data sheet, calculations should be done on standard calculation paper and attached to the data sheets. The analyst shall sign and date in ink each page of calculations. Full signature and date in ink are required in all instances.

For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed shall be evident and the input signed and dated by the analyst.

If data are directly acquired from instrumentation and processed, the analyst shall verify that the following are correct: project and sample numbers, calibration constants and response factors, output parameters such as units and numerical values used for detection limits (if a value is reported as less than). The analyst shall sign and date the resulting output.

8.2.2 Review of Data Processing

Following is a discussion of the method to be used for reviewing (checking) data processing.

- The analyst performing the data processing shall give to another analyst, independent of the work, the data package. The package shall include, as appropriate, raw data, data sheets, strip charts, computer input/output, calculations, sources for input parameters such as response factors.

- The independent analyst (checker) shall review the data for:

  * Appropriateness of equations used.
  * Correctness of numerical input.
  * Numerical correctness of all calculations.
  * This should be done by performing numerical computations.
  * Correct interpretation of strip charts.

- All entries and calculations that the checker reviews shall be marked in ink with a check mark. The checking process must be thorough enough to validate that the results are correct. If the checker disagrees with any part of the computations, the checker shall mark through the number with a single line and place the revised number above it.

- Any changes made by the checker shall be re-checked by the originator. If the originator agrees with the change, no action is necessary. If the originator disagrees, the originator and checker must resolve the difference so they agree with the result presented.

- The checker shall sign originals and date in ink all pages of the data package (except for groups of printouts such as chromatograms). Signing and dating indicates that the reviewer agrees with the calculations and that the originator has agreed to any changes made.

- If the data have been processed by computer, the reviewer shall also check the input entries. If the checker disagrees with the input, the number should be marked through with a line and the corrected number indicated above it. Corrections must be re-checked by the originator as discussed above.

- If an input error is identified and the data has been processed, it will be necessary to reprocess the data. In this event, the checker shall mark the second set of input to indicate agreement with the input changes. The checker shall sign and date in ink the computer input to indicate agreement.

- Raw data that are automatically acquired and processed do not require any validation at this point beyond that previously discussed.

- The reviewed data are maintained as discussed in Section 9.

8.2.3 Review of Data Reporting

Review of data reports is required to verify that information reported by the laboratory corresponds with processed analytical results. Intermediate steps performed after the processed data are checked to prepare the data report (such as data summaries) do not require validation. Preparation of the report is the responsibility of the department supervisor or laboratory director.

After the draft data report is prepared (generally in tabular form), the reported results should be checked against the reviewed processed data so that transcription errors do not occur. The checking process follows:

- Using the draft report, all data entries are checked. The checker can be an analyst or department supervisor. The checker is not required to be independent of the work because only the transcription from the reviewed data to data report is being checked.

- The draft data report should be checked so that the items cited for data presentation in Section 9.0 are complete and correct. Corrected entries are marked through with a single line and the correct entry is provided. The reviewer will indicate that corrections have been made in the report by placing a second check mark by the correction after comparing the change with the revised copy. The checker shall sign and date every page of the data report in ink.

- Use of the draft data report results in checkprint that should be maintained as a record to demonstrate the review.

- If data printouts, such as chromatograms are included in the data report, review is not required for the data printout.

9.0 DATA REPORTS AND RECORDS MANAGEMENT

9.1 DATA REPORTS

The format and content of a data report is dependent upon project needs, such as: whether or not explanatory text is required, client or contract requirements, and government agency reporting formats. However, the final data presentation shall be checked in accordance with data verification requirements of Section 9 and approved by the Laboratory Director.

Data presentation reports also include:

- Sample identification number used by CLSB and/or the sample identification provided to the laboratory, if different than identification used in the laboratory.
- Chemical parameters analyzed, reported values and units of measurement.
- Reporting limit of the analytical procedure if the reported value is less than the reporting limit.
- Data for a chemical parameter are reported with consistent significant figures for all samples.
- Results of Quality Control sample analysis if appropriate.

9.2 RECORDS MANAGEMENT

CLSB maintains all records in two categories. Specific regulatory or contractual demands may require additional documentation and in these instances, records shall be maintained as externally required.

9.2.1 Project Specific Documents

These are records and documents pertinent to a project. Examples of individual project specific documents are correspondence, chain of custody and data reports.

9.2.2 General Laboratory Operation Documents

These documents demonstrate overall laboratory operation, such as instrument log books and control charts. These records will directly affect the data for a specific project, but in general their applicability is not limited to one project.

9.3 RETENTION OF RECORDS

Records and files with be archived chronologically by subject and retained for 5 years.

Clinical Laboratory of San Bernardino, Inc.

June 22,2004

To: Melanie Emanual
State Water Resources Board
Division of Water Quality
1001 I Steet
P.O. Box 944213
Sacramento , CA 95812

Dear Melanie ,

I,m sending you a oopy of our QA Manual for your review. We are expanding our services at present and plan to add more services soon , especially in Organics Department . If you have further questions please call me .

Yours Truly,

Joe LaVoie , QC Manager
Clinical Laboratory
21881 Barton Road
Grand Terrace , CA 92313

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- Cation & Anion Analyses
- ~~EHR~~ Title 22 Analyses

CLINICAL LABORATORY CONTROLLED DOCUMENTS

QUALITY ASSURANCE MANUAL

6/2204 **QA62204A**

CLINICAL LABORATORY of SAN BERNARDINO

QUALITY ASSURANCE MANUAL

Revision 3.0 – June 24,2004

_X_ Controlled ___ Uncontrolled

Issued to: MELANIE EMANUAL

Copy No.: QA 62204A Date: 6/22/04

Approved Signatures:

Richard Kelso (Laboratory Director) Date

Joseph LaVoie (Quality Manager) 6/22/04 Date

The information in this CLSB QA Manual is intended for the addressee as issued and noted above. To remain compliant with quality control standards this manual is not to be duplicated.

CLINICAL LABORATORY of SAN BERNARDINO
21881 Barton Road, Grand Terrace, CA 92313
Phone: (909) 825 - 7693 Fax: (909) 825 - 7696

FOREWARD

The following document was prepared in accordance with the USEPA guidelines specified in "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" (QAMS-005/80). It is the intent of CLSB to meet or exceed the QA/QC requirements set by USEPA or other appropriate governmental or private entities and to assure that all analytical data generated are scientifically valid, defensible, comparable and of known acceptable precision and accuracy.

Richard Kelso
Laboratory Director

W. Joseph LaVoie
QA Manager

6/24/04

Clinical Laboratory of San Bernardino

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1.0 INTRODUCTION

The purpose of the Clinical Laboratory of San Bernardino (CLSB) Quality Assurance Manual is to document the minimum quality assurance requirements for the laboratory. This Quality Assurance Manual provides ready reference for analysts and clients on CLSB's policy pertaining to the accuracy and reliability of analytical tests performed in the laboratory.

The policies contained within this CLSB Quality Assurance Manual are to be applied to all laboratory operations. The manual is updated as required to provide for the addition of new methods and procedures as they are developed.

1.1 CLSB ANALYTICAL SERVICES

Clinical Laboratory of San Bernardino (CLSB) is an environmental testing laboratory providing a wide range of analytical services to both the public and private sectors. CLSB laboratories are located in Grand Terrace, California and features modern facilities and equipment. The staff is comprised of chemists, scientists, and technicians from a broad range of academic and environmental disciplines. The staff recognizes the need for high quality and legally defensive data, and the impact that this data has on the decisions of our clientele. It is our company mission to provide our customers with high quality, and cost effective laboratory services that will meet and/or exceed our customers' expectations.

1.2 LABORATORY ORGANIZATION AND RESPONSIBILITY

Since the demands on an environmental testing laboratory can be great and diverse in nature, the CLSB laboratories are structured into distinct and effective departments. These departments have clearly defined objectives and responsibilities that are directly involved in the analytical testing process. The structure of CLSB provides a framework for high quality analytical operations for which the Quality Assurance manual is the blueprint. The minimum responsibilities of laboratory personnel are defined as follows with the laboratory organization outlined in Figure 1-1.

- **President**

  The President is responsible for the management of the entire laboratory both financial and technical. It is the President's job to implement corporate goals, objectives and policies. The President is in direct communication with the Laboratory Director and Quality Assurance Manager.

- **Laboratory Director**
  Ultimate responsibility for laboratory operations and Quality Assurance is that of the Laboratory Director. The Laboratory Director communicates with the Quality Assurance Manager and Laboratory Manager to ensure that the CLSB Quality Assurance Manual

and SOPs are followed as written. The Laboratory Director works with each department supervisor to implement the QA/QC procedures of this manual. It is the Laboratory

Director's job to see that the non-laboratory departments (administration, data processing, etc.) of CLSB work with their laboratory counterparts to achieve high quality results.

- **Laboratory Manager**

The Laboratory Manager supports the Laboratory Director with the daily operation of the laboratory, working closely with Project Managers, Department Supervisors and clients.

- **Department Supervisors**

CLSB is divided into four analytical departments: Inorganics, Organics, Radiochemical and Microbiology; each department having its own supervisor. The department supervisors provide supervision of group operations, implement the laboratory quality assurance plan, ensure proper scheduling and execution of analyses, assure that proper analysis techniques are being used (use of approved SOPs), review all data before it is released to Quality Control, and report all discrepancies to the QA department.

- **Client Services/Project Management**

Client Services/Project Management serves as the primary laboratory contact for CLSB clientele. Any changes in the scope of work will be processed through this department. The department monitors the progress and timeliness of analytical work; reviews ongoing work orders and all subsequent final laboratory reports for accuracy and adherence to the QA Plan.

- **Field Services**

Field Services has the responsibility of proper sampling and transportation of samples to and from CLSB. Field service personnel are required to know CLSB's QA policies and report to the Field Services Manager.

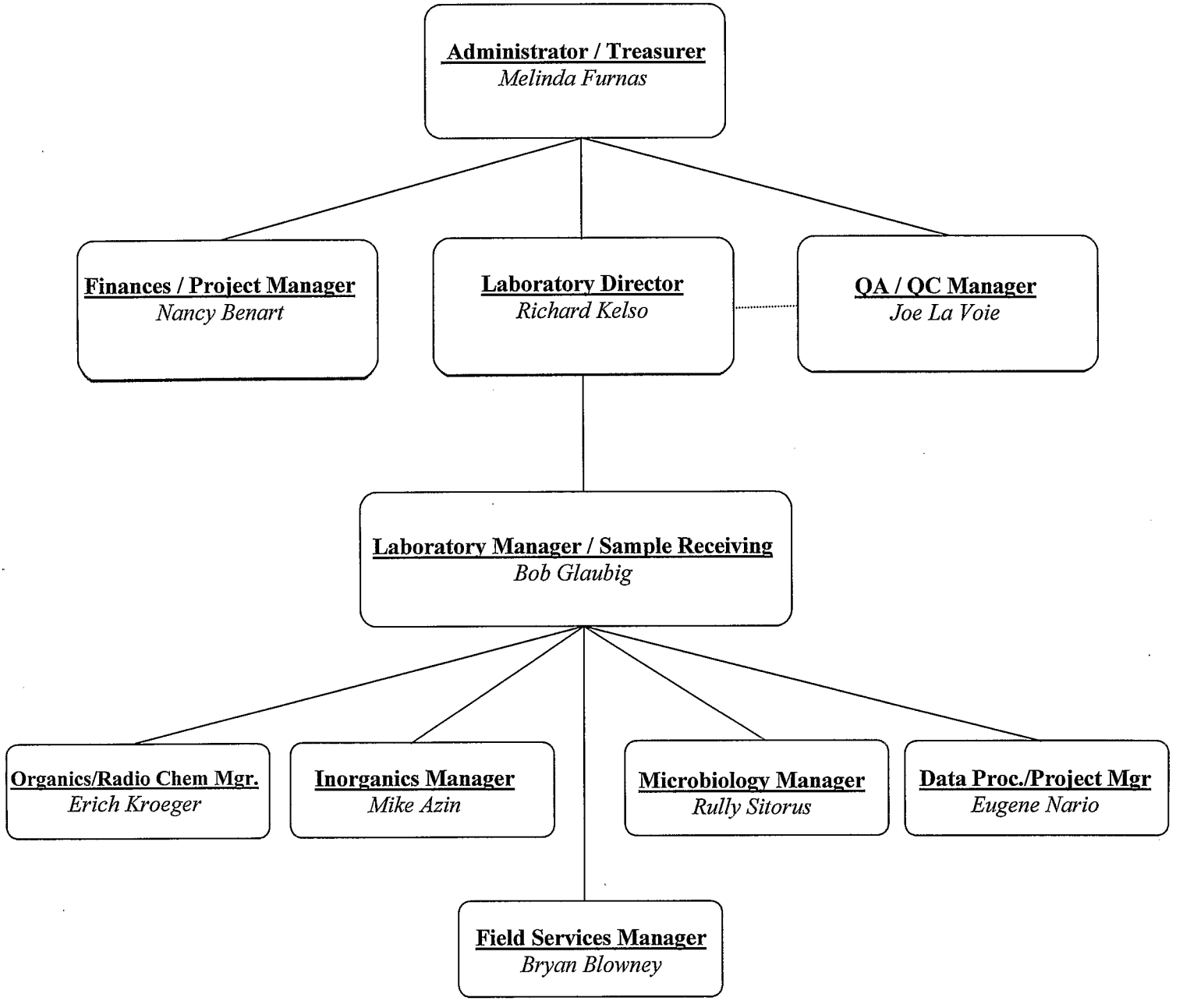
- **Administration**

Administration is responsible for the management of financial operations, including accounting and procurement of all laboratory items. It is the Administration's job to ensure that purchased items and services meet the QA Plan requirements and perform as outlined in this document.

- **Data Control**

Data Control (Data Management) is responsible for sorting, reviewing, distributing, filing, and archiving all data generated by CLSB. This department works with the QC department to help implement QC standards through the use of electronic programming.

Clinical Laboratory of San Bernardino, Inc.
6/22/04



1.3 OBJECTIVES OF THE QA PROGRAM

The primary objective of the Quality Assurance program is to produce quality data which is of known precision and accuracy and legally defensible. This will ensure that the data can be relied on to represent the true value for a given sample.

1.4 CLSB QUALITY ASSURANCE PROGRAM

The CLSB Quality Assurance/Quality Control Program is an essential part of any analytical procedure. The program has been integrated into every phase of laboratory operations. The program detects and corrects problems in the measurement process to ensure that all data is valid, of known precision and accuracy, and is legally defensible. Secondly, it is designed to monitor and control the quality of the data generated by the laboratory, thus ensuring that errors are kept to an acceptable level and corrective action is taken when necessary.

1.5 QA MANUAL SUMMARY

The organization of this manual is presented in the table of contents. Essentially the manual follows the logical progression of analytical work and the application of the Quality Assurance Program in the laboratory. The program can be divided into four major areas.

1.5.1 Pre-Analytical Procedures

The pre-analytical work includes the various aspects of sampling, preservation and storage, documentation, materials and standards, and calibration.

1.5.2 Procedures Concurrent with Analysis

This group of procedures includes Quality Control steps such as blanks, spikes, replicates, etc., as well as analytical methodology.

1.5.3 Data Reduction and Evaluation

Both QC and sample data must be evaluated and a QC check performed to ensure the data obtained is valid and falls within acceptable precision and accuracy limits.

1.5.4 Data Reporting and Record Maintenance

Specific reporting formats may be required for different projects, but all data must be reviewed before being released. Additionally, records are maintained to allow access for future inquiries concerning the results.

1.6 ANALYTES AND ANALYTICAL METHODS

The principle methods used for the analysis of drinking water and wastewater come from USEPA procedures and APHA's "Standard Methods" 18th Ed. All analysis performed at CLSB comply with these methods and are listed in Table 1-2.

CLSB has written SOPs for the bench level analyst using these methods that comply with all federal and state regulations.

ANALYTICAL METHODS

A. ORGANIC CHEMICAL TESTING

	Water
1. EDB and DBCP	EPA 504.1
2. Glyphosate	EPA 547
3.. Purgeable Organic Compounds by GC/MS	EPA 524.2
4.. Benzo-(A)-pyrene	EPA 550.1
5. Haloacetic Acids	EPA 552.2
6.. Oil & Grease	EPA 1664

Table 1-2 Analytical Methods

ANALYTICAL METHODS (cont.)

B. INORGANIC CHEMICAL TESTING

	Water
1. Aluminum	EPA 200.7
2. Antimony	SM3113B
3. Arsenic	SM3113B
4. Barium	200.7
5. Beryllium	SM3113B
6. Boron	EPA 200.7
7. Cadmium	SM3113B
8. Chloride	EPA 300.0
9. Calcium	200.7
10. Chromium, hex	EPA 218.6
11. Chromium, total	SM3113B
12. Copper	EPA 200.7
13. Cobalt	EPA 200.7
14. Fluoride	EPA 300.0/340.2
15. Iron	EPA 200.7
16. Lead	SM3113B
17 Magnesium	EPA 200.7
18. Manganese	EPA 200.7
19. Mercury	EPA 245.2
20. Molybdenum	EPA 200.7
21. Nickel	SM3113B
22. Nitrate /Nitrite	EPA 300.0 /353.2
23. Perchlorate	EPA 314.0
24. Phosphorus	EPA 365.2
25. Potassium	EPA 200.7
26. Selenium	EPA 200.7
27. Silicon	EPA 200.7
28.. Silver	SM3113B
29.. Sodium	EPA 200.7
30. Sulfate	EPA 300.0
31. Sulfide	EPA 376.2
32. Thallium	EPA 200.7/200.9
33. Vanadium	EPA 200.9
34. COD	Hach 8000
35. Cyanide	SM4500 CN –F

Table 1-2 Analytical Methods (cont.)

ANALYTICAL METHODS (cont.)

C. DRINKING WATER AND WASTEWATER TESTING

		Method
1.	General Mineral	
a.	Calcium, Magnesium, Sodium, Potassium ,Silica	EPA 200.7
b.	Alkalinity	SM2320B
c.	Anions	EPA300.0-EPA 300.1
d.	Fluoride	EPA 340.2
e.	Acidity	EPA 305.1
f.	Calcium	SM 4500 –Ca D.
2.	General Physical	
a.	pH	EPA 150.1
b.	Specific Conductance	EPA 120.1
c.	Total Dissolved Solids	EPA160.2,SM2540C
d.	Turbidity	EPA 180.1
e.	Methylene Blue Active Substances (MBAS)	EPA425.1/SM 5540C
f.	Volatile Residue	EPA 160.4
g.	Settleable Residue	EPA 160.5
h.	Total Residue	SM2540B
i	Filterable Residue	SM2540C
j	Odor	SM2150A
k.	Color	SM2120B
l.	Conductivity	SM2510B
3.	Primary/Secondary Inorganic	
a.	Aluminum, Barium, Copper, Iron, Manganese, Silver, and Zinc	EPA200.7
b.	Arsenic, Antimony .	SM3113B
c.	Cadmium	SM3113B
d.	Chromium	SM3113B
e.	Lead	SM3113B
f.	Mercury	EPA 245.2
g.	Selenium	SM3113B
h.	Chlorine	SM 4500 Cl G
i	COD	Hach 8000 Method
j	Ammonia	EPA 350.1
k.	Kjehldahl Nitrogen	EPA 351.2
l.	Dissolved Oxygen	EPA 360.1
m.	Total Phosphorous	EPA 365.2
n .	Orthophosphate	EPA 365.2

4.	Regulated Organic		
	a.	Volatile Organic Compounds	EPA 524.1
	b.	DBCP and EDB	EPA 504.1
	c.	Pesticides	EPA 531.1
	d.	Glyphosate	EPA 547
	e.	Benzo -(A) –Pyrene	EPA 550.1
	f.	Halocetic Acid	EPA 552.2

Table 1-2 Analytical Methods (cont.)

ANALYTICAL METHODS (cont.)

D.. Radiochemistry of drinking water and wastewater .	Method
1. Gross Alpha	EPA 900.0
2. Gross Beta	EPA 900.0
3. Uranium	EPA 908.0
4. Radon –222	SM 7500 – Rn

E. MICROBIOLOGICAL TESTING

		Method
1.	Total, Fecal, E. Coli Coliforms by Multiple Tube Fermentation	9221A*
2.	Total & E. Coli Coliforms by MMO-MUG	9223B *
3.	Heterotrophic Plate Count	9215B *
4.	Total Coliform by Multiple Tubes Fermentation	9221B *
5.	Fecal/E. Coli by Multiple Tubes Fermentation	9221E *
6.	Fecal Strep Bacteria	9230B *
7.	BOD /CBOD	5210B *

* Standard Method - 19th Ed.

Table 1-2 Analytical Methods (cont.)

2.0 SAMPLE RECEIPT, PRESERVATION AND STORAGE

To provide representative samples for analysis, both field and laboratory personnel must satisfactorily perform their duties. Field sampling is a critical part of the analytical process and can have a direct effect on data quality. All samples must be properly collected, preserved, and transported to the laboratory before analysis.

2.1 SAMPLING PROCEDURES AND DOCUMENTATION

Proper sampling in the field requires consideration of many aspects including:

- Sampling technique
- Containers used
- Labeling the containers
- Preservation and Storage
- Transportation
- Documentation
- Identification of analysis required to give useful results for the intended purpose

The items discussed in this section touch on several of these key elements in environmental sampling and analysis.

2.1.1 Chain of Custody

An overriding consideration for accurate analytical results is the ability to demonstrate that the samples have been obtained from the locations stated and that they have reached the laboratory without alteration. To accomplish this, evidence of collection, shipment, laboratory receipt, and laboratory custody must be documented.

Documentation is accomplished through a "chain of custody" (COC) form that records each sample and the individuals responsible for sample collection, shipment and receipt. A sample is considered in custody if it is:

- In a person's actual possession
- In view after being in physical possession
- Locked up so that no one can tamper with it after having been in physical custody
- In a secured area, restricted to authorized personnel

Figure 2-1 represents a chain of custody form (COC) that is used by CLSB personnel in collecting and shipping samples.

Each individual who has the samples in their possession signs the COC form. Preparation of the COC shall be as follows:

- The person collecting the samples shall initiate the chain of custody record, in the field. Samples can be grouped for shipment and can use a common COC form.
- The record shall be completed in the field to indicate project, sampling team, and other necessary information.
- If the person collecting the sample does not transport the samples to the laboratory, or deliver the sample containers for shipment, the first block for: Relinquished By_____, Received By _____ shall be completed in the field.
- The person transporting the samples to the laboratory or delivering them for shipment shall sign the record form as: Relinquished By _____.
- If the samples are shipped to the laboratory by commercial carrier, the COC form shall be sealed in a watertight container, placed in the shipping container, and the container sealed prior to giving it to the carrier.
- If the samples are directly transported to the laboratory, the COC form shall be kept in possession of the person delivering the samples.
- For samples shipped by commercial carrier, the weigh bill shall serve as an extension of the chain of custody record between the final field custodian and receipt in the laboratory.
- Upon receipt in the laboratory, the Sample Control Manager, or representative, shall open the shipping containers, compare the contents with the COC record and sign and date the record.
- If discrepancies occur, the sample in question shall be segregated from normal sample storage and the field personnel immediately notified.
- COC forms shall be maintained with the records for a specific project, becoming a part of the data package.

Multipart COC forms may be used so that a copy can be returned to the person shipping the samples after they are received in the laboratory and after the laboratory disposes of the samples. Otherwise, photocopies will be made and distributed.

2.1.1.1 Forms for Microbiology

Three forms are for the exclusive use of the Microbiology Department. The receipt of sample, data and results are not entered into the LIMS system.

2.1.1.1.1 Coliform Bacteria, 15 Tube Dilution, Water/Wastewater Form, Figure 2.2

This form is for the collection of water/wastewater samples for the above described analysis. The form has all sample collection information and all data reporting sections included. The form is completed when analysis of samples is finished. After the form is reviewed by the laboratory director, a copy is kept in the internal filing system and a copy is sent to the client.

2.1.1.1.2 Coliform Bacteria Report Form, Figure 2.3

This form is for the collection of drinking water for the analysis for Coliform Bacteria. As above, the form has all collection information and all data reporting sections included. The form is complete when the analysis data is entered. After the form is reviewed by the laboratory director, a copy is kept in the internal filing system and a copy is sent to the client.

2.1.1.1.3 General Physical Analysis Form, Figure 2.4

This form is for the collection of drinking water for general physical analysis. As above, the form has all collection information and all data reporting sections included. The form is complete when the analysis data is entered. After the form is reviewed by the laboratory director, a copy is kept in the internal filing system and a copy is sent to the client.

Clinical Laboratory of San Bernardino, Inc. CHAIN OF CUSTODY PAGE ___ OF ___

CLIENT:

Address:

System Number:

Phone No.: Fax No.:

Project Name:

Sampled By:

Comments:

CLIENT JOB NUMBER

(Company Use Only)

DESTINATION LABORATORY

☐ CLINICAL LABORATORY OF SAN BERNARDINO, INC. (909) 825-7693

☐ OTHER _____

PRESERVATIVES

ANALYSIS REQUESTED

TURN AROUND TIME

DATE	TIME	SAMPLE IDENTIFICATION	MATRIX	NUMBER OF CONTAINERS

PRESERVATIVES: (1) $Na_2S_2O_3$ (2) HNO_3/HCl (3) COLD (4)

TURN AROUND TIME: (10) 10 DAY (5) 5 DAY RUSH (2) 2 DAY RUSH ALL TURN AROUND TIMES ARE EXPRESSED AS WORKING DAYS. SOME ANALYSES MAY TAKE LONGER THAN CHOSEN TIME DUE TO METHOD LIMITATIONS.

RELINQUISHED BY (SIGN)	PRINT NAME / COMPANY	DATE / TIME	RECEIVED BY (SIGN)	PRINT NAME / COMPANY

REC'D AT LAB BY: DATE / TIME: CONDITIONS / COMMENTS:

SHIPPED VIA ☐ FED X ☐ GOLDEN STATE ☐ GREYHOUND ☐ CLIENT ☐ OTHER _____

2.1.2 FIELD COLLECTION AND SHIPMENT

Prior to collecting samples, the collection team must consider the analyses to be performed so that proper sample containers and shipping containers can be assembled and proper preservatives added to containers. In addition, field logs, record sheets, chain of custody forms (COC) and analysis records must be assembled.

All records required for proper documentation must be completed by the field team. The primary documenting record is the COC form, the Sample Receiving Log-in Sheet and the records in the LIMS system as discussed in Section 2.2.

In addition to initiating the COC form, field personnel are responsible for uniquely identifying and labeling samples, providing proper preservation, and packaging samples to preclude breakage during shipment.

2.1.2.1 Labeling

Every sample shall be labeled to identify:

- Project or job number
- Sample location (such as well number)
- Sampling date and time
- Person obtaining the sample
- Sample preservation/conditioning method, if applicable
- Analysis requested

2.1.2.2 Sample Containers and Preservation

Containers provided by CLSB have been purchased from commercial suppliers and are certified as to being cleaned per USEPA procedures for low level chemical analysis. Samples must be placed in containers compatible with the intended analysis and properly preserved. Also, collectors of samples must consider the time interval between acquiring the sample and analysis (holding time) so that the sample is representative. Table 2-1 provides requirements for various analytical parameters with respect to preservation, method and maximum holding time between collection and analysis.

2.1.2.3 Sample Transportation

Shipping containers are to be sealed prior to shipment, whether shipped by direct transport by field personnel or commercial carrier. The only exception is if sufficient holding time exists so that the samples can be held in the field, but it will be necessary to re-ice the containers prior to or during transport.

2.1.2.4 Request for Analysis

The final step in providing information to the laboratory is the "Analysis Requested" portion of the COC form. The Analysis Requested, included on the CLSB COC form (Figure 2-1), shall be completed by the field personnel and included with the COC record. Any other form, provided by the client, that details the requested analysis may be substituted for the COC form provided sufficient information is included. It is imperative that the "Analysis Requested" information be provided to enable the lab to comply with maximum allowable sample holding times.

2.2 RECEIPT OF SAMPLES AND CHAIN OF CUSTODY

Samples are stored either in a cold room at 4° C or in a refrigerator or freezer depending on the type of samples and analysis. Samples for volatile analyses, such as EPA 601/602, are stored in separate refrigerators.

The Sample Receiving staff will receive the samples and:

- Examine all samples and determine if proper temperature and preservation have been maintained during shipment. If samples have been damaged during shipment, the remaining samples shall be carefully examined to determine whether they were affected. Any samples affected shall also be considered damaged. It will be noted on the COC record that specific samples were damaged. The Sample Receiving supervisor is notified and the client is contacted as to the damage.
- Compare samples received against those listed on the Chain of Custody.
- Verify that sample holding times have not been exceeded.
- Sign and date any Chain of Custody form and attach any waybill to the Chain of Custody.
- Place the samples in proper laboratory storage.
- Enter the client name in the laboratory Sample Log-in Sheet.
- Enter all login information into the computer information system.
- Issue and distribute a work order to the appropriate analytical department.
- Place the completed chain of custody records in the project file.

After sample receipt and inspection, the log-in personnel will sign the COC form. For samples delivered by mail or by a third party, the client should include a signed COC form with all the required information. A signed copy of the COC form will be included in the final report and in a QC package report to be kept in our archive room.

2.3 PRESERVATION, STORAGE AND DISPOSAL

2.3.1 Sample Preservation

Preservation of samples is addressed in several of the references in Section 2.1. Additionally, Table 2-1 summarizes preservation methods.

2.3.2 Laboratory Storage of Samples

The primary consideration for sample storage is:

- The extraction and analysis of samples within the prescribed holding times for the parameters of interest.

The requirements of Table 2-1 for holding times shall be used. Placing of samples in the proper storage environment is the responsibility of the Sample Control Manager, who should notify the Laboratory Director, or department supervisor, if there are any samples that must be analyzed immediately because of holding-time requirements.

2.3.3 Sample Disposal

Ultimate disposition of the samples is addressed in CLSB's Haz-Waste Disposal Plan. There are several possibilities for sample disposition:

- The sample may be completely consumed during analysis.
- Samples may be returned to the client or location of sampling for disposal.
- The samples may be stored after analysis. Proper environmental control and holding time must be observed if reanalysis is anticipated. Otherwise, environmental conditions for storage will not be observed.

The Sample Control Manager shall determine disposition of samples if not specified on the COC (Figure 2-1). In general, CLSB does not maintain samples and extracts longer than 30 days beyond receipt of analytical data by the customer, unless otherwise specified.

2.4 Initiation of Testing Program

As stated in Section 2.1.1, the COC form (including the Analysis Request) shall be submitted with the samples to the laboratory.

If the analytical program is not defined with the sample shipment, the Sample Control Manager shall immediately notify the responsible field personnel for definition of the analysis program. If the samples are external to CLSB, the client shall be contacted to determine the testing program. The Sample Control Manager will store the samples as appropriate. The COC form, Sample Log-in Sheet, and the LIMS record remain the primary sample documents.

The Laboratory Director or Laboratory Manager is responsible for prioritizing samples on the basis of holding time and required turn around time.

ANALYSIS	METHOD	PRESERVATIVE	HOLD TIME
GC VOLATILES:			
EDB / DBCP	EPA 504/8011	$Na_2S_2O_3$	14 days
Purgeable Aromatics or BTEX	EPA 602/8020	$Na_2S_2O_3$	14 days
Purgeable Aromatics & Halocarbons	EPA 502.2/8021	$Na_2S_2O_3$, HCl	14 days
Purgeable Halocarbons	EPA 601/8010	$Na_2S_2O_3$	14 days
Purgeable Non-Halogenated Hydrocarbons	EPA 8015	None	14 days
PETROLEUM FUELS:			
TPH Gasoline, BTEX, MTBE	EPA 8015m/602/8020	HCl	14 days
Oxygenates	EPA 8260m	HCl	14 days
TPH Extractables (Diesel/Oil)	EPA 8015m	None	7 days
Oil & Grease	EPA 1664	HCl	28 days
TRPH	EPA 1664 SGT	HCl	28 days
GC/HPLC SEMI-VOLATILES:			
Phthalate Esters	EPA 506	None	14 days
Benzo(a)pyrene	EPA 550	None	7 days
Formaldehyde	EPA 8315	None	3 days
Nitroaromatics/Nitroamines (Explosives)	EPA 8330	None	7 days
Polynuclear Aromatic Hydrocarbons	EPA 610/8310	None	7 days
Acrolein & Acrylonitrile	EPA 8316	None	14 days
Phenols	EPA 420.1	H_2SO_4	28 days
PCBs:			
Polychlorinated Biphenyls (PCBs)-water	EPA 508/608/8082	None	7 days
Polychlorinated Biphenyls (PCBs)-oil	EPA 8082	None	7 days
Pesticides and PCBs	EPA 8080/508/608	None	7 days
PESTICIDES/HERBICIDES:			
Carbamates	EPA 531/632	None	7 days
Chlorophenoxyacid Herbicides	EPA 8151A/515	None	7 days
Organochlorine Pesticides	EPA 8081A/508	None	7 days
Organophosphorus Pesticides	EPA 8141A	None	7 days
Triazine Pesticides	EPA 507	None	7 days
GC/MS:			
Volatile Organic Compounds	EPA 624/8240/8260	HCl	14 days
Volatile Organic Compounds-drinking water	EPA 524.2	HCl	14 days, 24 hrs w/o HCl
Semi-volatile Organic Compounds	EPA 625/8270	None	7 days
Phenols	EPA 625/8270	None	7 days
Phthalate Esters	EPA 625/8270	None	7 days
METALS ANALYSIS:			
Metals	EPA 6010/7000/200	HNO_3	6 months
Mercury	EPA 245.1/7040	HNO_3	28 days
Hexavalent Chromium	EPA 7196/7199/218.6	None	24 hours

TABLE 2-1

ANALYSIS	METHOD	PRESERVATIVE	HOLD TIME
INORGANIC ANALYSIS:			
Alkalinity	EPA 310.1	None	14 days
Ammonia	EPA 350.2	H_2SO_4	28 days
BOD	EPA 405.1	None	48 hours
Bromide	EPA 300.0	None	28 days
Chlorate	EPA 300.0	None	28 days
Chloride	EPA 300.0	None	28 days
COD	EPA 410.4	H_2SO_4	28 days
Color	EPA 140.1	None	48 hours
Conductivity	EPA 120.1	None	28 days
Corrosivity-Langlier Index	SM 2330B	None	14 days
Cyanide	EPA 335.2/9010	NaOH	14 days
Dissolved Oxygen	EPA 360.1	None	Immediate
Flash Point	EPA 1010	None	N/A
Fluoride	EPA 340.2	None	28 days
Hardness	EPA 200.7	HNO_3	6 months
Iodide	EPA 300.0	None	28 days
MBAS Surfactant	EPA 425.1	None	48 hours
Nitrate	EPA 300.0/353.2	None	48 hours
Nitrite	EPA 300.0/353.2	None	48 hours
Odor	EPA 110.2	None	24 hours
Ortho-phosphate	SM 4500-P	None	48 hours
Paint Filter Liquids Test	EPA 9095	None	N/A
Perchlorate	EPA 300.0/314.0	None	28 days
Percent Moisture	SM 2540B		
pH	EPA 150.1/9040	None	Immediate
Total Phosphorus	SM 4500-P	H_2SO_4	28 days
Reactivity	SW 846	None	N/A
Silica	EPA 200.7/6010	None	28 days
Specific Gravity	SM 2710F	None	N/A
Sulfate	EPA 300.0	None	28 days
Sulfide	EPA 376.2/9030	Zn-Ac	7 days
Total Dissolved Solids	EPA 160.1	None	7 days
Total Kjeldahl Nitrogen	EPA 351.3	H_2SO_4	28 days
Total Organic Carbon	EPA 415.1	H_2SO_4	28 days
Total Settleable Solids	EPA 160.5	None	48 hours
Total Solids	EPA 160.3	None	7 days
Total Suspended Solids	EPA 160.2	None	7 days
Turbidity	EPA 180.1	None	48 hours
MICROBIOLOGY:			
Coliform bacteria-drinking water	MMO-MUG/SM 9221	$Na_2S_2O_3$	24 hours
Coliform bacteria-waste water	MMO-MUG/SM 9221		8 hours
Heterotrophic Plate Count	SM 9215	$Na_2S_2O_3$	24 hours
Fecal Streptococci	SM 9230	$Na_2S_2O_3$	8 hours (24hrs D.W.)

TABLE 2-1

SAMPLE LOG-IN FORM

CLS# **S**_____ COC# _____Date received _____

Received by: _____

Delivered by: _____

Packaging: cooler _____ DOT packing _____ other (specify) _____

Did package come with shipping slip? . . . . . .yes no

If yes, tracking number : _____

Were custody seals present? . . . . . yes no

If yes, how many?_____ Date _____ Signed?. . . . yes no Intact? . . . . yes no

Custody papers sealed? . . . . . yes no Inside packaging? . . . . . yes no

Did sample containers arrive intact?. . . . . yes no

If no, list any samples broken _____

Shipping preservation: blue ice_____ ice_____ none_____ other_____

Temperature upon receipt: _____ °C IR/THERM Blank . . . yes no

Geiger counter test . . . . . Negative Positive

Chain of custody filled out properly? . . . . . not present yes no

Did receiver sign chain of custody in correct place? . . . . . yes no

Were sample labels complete?

(ID, date, time, signature, preservative) . . . . . yes no

Did sample labels agree with chain of custody? . . . . . yes no

Were correct containers used for tests indicated? . . . . . yes no

If no, was pink slip filled out noting discrepancy? . . . . . yes no

Also, if no, was customer called and notified? . . . . . yes no

Were correct preservatives verified?. . . . . N/A yes no

If yes, pH of non-volatile samples _____

Was sufficient amount of sample delivered? . . . . . .yes no

Were bubbles absent in VOA samples? . . . . . N/A yes no

Date samples were logged in _____ By whom:_____

Additional comments: _____

Figure 2-2

3.0 MATERIALS AND STANDARDS / SOURCES AND PREPARATION

The quality of reagents, solvents, gases, water and laboratory vessels used in analyses must be known so that their effect upon analytical results can be defined. Materials purchased by CLSB meet the requirements stated below or as denoted in specific analytical procedures and are controlled as stated. Requirements must also be met for internally prepared materials such as water.

The Quality Control Manager or other person as assigned by the Laboratory Director will retain responsibility for purchasing materials and controlling them in the laboratory. The duties of the materials coordinator include:

- Specifying in purchase orders suitable grades of materials (grade should be defined by the QC Manager or Laboratory Director).
- Verifying upon receipt that materials meet requirements and that, as applicable, material certificates are provided and maintained in the laboratory record system.
- The identification and proper storage of materials.
- Verifying that material storage is properly maintained and material is removed from use when expired.

3.1 REQUIREMENTS FOR REAGENTS, SOLVENTS, AND GAS

Chemical reagents, solvents and gas are available in a variety of grades of purity, ranging from technical grade to ultra-pure grades. The purity required varies with the analytical method. The parameter measured, and the sensitivity / specificity of the detection system are important factors in determining the purity of the reagents required.

Standards are obtained from NIST and commercial sources and are traceable to EPA, NIST CRADA (Cooperative Research And Development Agreement) or A2LA (American Association for Laboratory Accreditation). Our commercial suppliers include credible companies such as Ultra-Scientific, Chem Service, VWR, Aldrich,...etc. Certificates for all standards are obtained and kept in a department log book/file that is available for review and inspection. If two sources of a standard are used, at least one standard shall have a certificate and the other shall be traceable to the certified standard through comparative study. All standards, stock or working, are labeled by their name and traceable to the standard logbooks. Expiration dates are also found on the labels and in the logbooks. No expired standards shall be used. All logbooks pertaining to standards and standard traceability are audited by the QA/QC department.

Carrier gas, solvents, acids and deionized water are checked on a batch wise basis. In this way it is possible to avoid systematic contamination of samples without repeating a set of samples, as would be the case if we relied only on method blanks to detect such contamination.

3.1.1 General Inorganic Analyses

In general, Analytical Reagent Grade (AR) reagents and solvents are adequate for inorganic analyses. Primary standard reagents shall be used for standardizing all volumetric solutions. All prepared reagents shall be checked for accuracy.

Individual analytical methods specify the reagents that require frequent standardization or special treatment. To minimize potential deterioration, the analyst should prepare a limited volume of such reagents, depending on the quantity required over a given period of time.

3.1.2 Trace Metals Analyses

All standards used for atomic absorption and emission spectroscopy shall be spectro-quality. It is recommended that other reagents and solvents also be spectro-quality, although, in some cases, AR grade may be satisfactory. Standards are prepared by the analyst, or purchased directly from suppliers provided the materials meet the requirements of the analytical method.

In general, fuel and oxidant gas used for atomic absorption can be commercial grade.

Compressed air can be commercially supplied, zero grade or supplied by laboratory air compressors if adequate pressure is maintained and the air is filtered to remove oil, water and possible trace metals.

3.1.3 Organic Chemical Analyses

AR is the minimum acceptable grade for materials used for organic analyses; use reference grade standards only as necessary. Special note should be made of the assay of standard materials.

Some GC detectors require that solvents, standards and samples be free of certain classes of compounds. For example, use of the flame photometric detector requires that reagents and solvents be free of sulfur and phosphorus interference.

Pesticide-quality solvents are required for low-concentration work. AR grade solvents are adequate for analyzing industrial waste samples. However, the contents of each solvent lot must be checked to determine suitability for the analyses. Similarly, all analytical reagents and other chemicals must also be routinely checked.

3.1.4 Water

Deionized water is used for dilution, preparation of reagent solutions and final rinsing of glassware. Water quality shall be determined daily by measuring specific conductance and shall be recorded in a logbook. A resistance equal to or greater than 18.3 megohms/cm at 25° C is required. This is equivalent to less than 0.1 mg/L of ionized material.

Organic-free water is required for microbiological and volatile organic analyses. Organic-free water may be verified by the purge-and-trap technique on the GC.

When determining trace organics by solvent extraction and gas chromatography, specialty water such as HPLC grade water with sufficiently low background must be used. Pre-extraction of the water with the solvent used in the analysis may be helpful in eliminating organic compounds in the water.

3.1.5 Compressed Air

Compressed air must be free of oil, water and dirt and of high quality, dry grade. The usual quality of compressed air for laboratory use is Ultra Zero.

3.2 CONTAINERS

Containers used in the laboratories can affect the quality of results. Material composition, volumetric tolerances and cleaning are important considerations in laboratory containers. Sample containers are discussed in Section 2.1.

3.2.1 Composition of Laboratory Containers

Soft glass containers are not recommended for general use, especially for the storage of reagents. The glass recommended for general use is chemically resistant borosilicate glass, such as is manufactured under the trade names of Pyrex or Kimax. This glassware is satisfactory for analyses performed by CLSB unless otherwise noted in the sampling or testing procedure. The use of plastic vessels, containers and other apparatus made of Teflon, polyethylene, polystyrene and polypropylene is desirable for certain specified applications.

The following guidelines should be considered when selecting the material composition of laboratory vessels:

- Borosilicate or polyethylene bottles are to be used for the storage of reagents and standard solutions, unless otherwise specified.
- Plastic containers should not be used for reagents and solvents in organic analyses.

- Dilute metal solutions have a tendency to plate out on container walls over long periods of time; therefore, standard solutions should be prepared at the time of analysis.

- The use of disposable glassware is satisfactory for some analyses, such as the use of disposable test tubes for use with some automatic samplers.

- Plastic bottles of polyethylene and Teflon are satisfactory, in general, for the shipment of water samples. However, strong mineral acids such as sulfuric acid and organic solvents readily attack polyethylene.

- Borosilicate glassware is not completely inert, particularly to alkalis. Standard solutions of silica, boron and the alkali metals should be stored in polyethylene bottles.

3.2.2 Volumetric Container Specifications

CLSB shall use glassware of sufficient accuracy as required for each analytical procedure. This includes volumetric flasks, volumetric pipettes and accurately calibrated burets. Less accurate types of glassware, including graduated cylinders and serological and measuring pipettes have specific uses when less exact volumes are permitted by the analytical procedure.

In general, volumetric containers will not be calibrated by CLSB unless required by a specific analytical method. However, volumetric glassware shall be purchased with the objective of meeting the correct end use of the container in an analytical procedure.

3.2.3 Glassware Cleaning Requirements

Methods of cleaning glassware are selected according to the substances that are to be removed and the analytical analysis required.

For inorganic analytical uses, all glassware will be placed into detergent water immediately after use and must not be allowed to dry. After a thorough soaking, glassware will be scrubbed and rinsed at least 3 times with DI water. Glassware will also be rinsed twice with 5% nitric acid solution and then rinsed with D.I. water, air dried and stored in an upright position.

Glassware used for phosphate determinations will not be washed with detergents containing phosphates. This glassware must be thoroughly rinsed with tap water and deionized water. For ammonia and Kjeldahl nitrogen determination, the glassware must be rinsed with ammonia-free water.

Glassware used in the determination of trace organic constituents in water should be as free as possible of organic contaminants. Glassware used for organic analysis should be soaked in hot water containing detergent for two hours, then scrubbed, re-soaked in chem-solve for two hours, rinsed with D.I. water and allowed to dry. Once the glassware has dried it will be rinsed with methanol, air-dried and stored with open end sealed with aluminum foil.

Sampling bottles are all purchased certified clean, but if not they will follow the above procedure for cleaning, depending on the analysis requested. Bottles used for the collection of samples for organic analyses are rinsed successively with acid cleaning solution, tap water, deionized water, and, finally, several times with a redistilled solvent such as acetone, hexane, petroleum ether or chloroform. Caps should be washed with detergent, rinsed with tap water, deionized water and solvent. Liners are treated in the same way as bottles and are stored in a sealed container.

Alternate methods for cleaning may be used if it is demonstrated (such as by blank analysis) that the result is satisfactory. Also, disposable glassware may be used if applicable to the analytical procedure.

3.3 STORING AND MAINTAINING REAGENTS AND SOLVENTS

The following shall apply for storing and maintaining reagents and solvents:

- All standards and reagents will be logged into the standard logbook, and the work standard logbook, upon receipt or formulation of.
- Standard reagents and solvents are stored in accordance with the manufacturer's recommendations.
- Light-sensitive standard reagents or solvents are stored in a cool, dark place.
- Organic reagent standards are stored at 4^0 C $\pm$ 2^0.
- Organic reference materials are stored at 4° C ± 2°.
- Standards are not maintained longer than recommended by the manufacturer or as specified in the analytical method.

4.0 INSTRUMENT CALIBRATION, MAINTENANCE AND REPAIR

Modern environmental chemical analysis is heavily dependent on properly maintained and calibrated instruments. The sensitivity and reliability of these high precision instruments require periodic maintenance and calibration to assure precise and accurate measurements. Therefore, CLSB standard procedures include routine instrument calibration and maintenance.

4.1 CALIBRATION

The calibration program verifies that equipment is of the proper type, range, accuracy and precision to provide data compatible with specified requirements. All instruments and equipment that measure a quantity, or whose performance is expected at a stated level, are subject to calibration.

This section of the QA Manual prescribes the practices used by the laboratory to implement a calibration program. Implementation is the responsibility of the laboratory management and analysts. The Quality Assurance Manager shall review the implementation of the program.

Two types of calibration are discussed in this section:

- Operational calibration is routinely performed as part of instrument use, such as the development of a standard curve for use with an atomic absorption spectrophotometer. Operational calibration is generally performed for instrument systems.

- Periodic calibration that is performed at prescribed intervals for equipment, such as balances and ovens. In general, equipment that can be calibrated periodically is a distinct, single purpose unit and is relatively stable in performance.

4.1.1 Calibration Program

The program of calibration for laboratory instruments contains the following elements:

4.1.1.1 Calibration Procedures

Whenever possible, recognized procedures, such as those published by ASTM or the USEPA, or procedures provided by manufacturers, shall be used by CLSB. If established procedures are not available, a procedure shall be developed considering the type of equipment, stability characteristics of the equipment, required accuracy and the effect of operational error on the quantities measured. As a minimum, the procedures shall include:

- Equipment to be calibrated
- Reference standards used for calibration

- Calibration technique and sequential actions
- Acceptable performance tolerances
- Frequency of calibration
- Calibration documentation format

4.1.1.2 Calibration Frequency

Instruments and equipment shall be calibrated at prescribed intervals and/or as part of the operational use of the equipment.

Frequency shall be based on the type of equipment, inherent stability, manufacturer's recommendations, values provided in recognized standards, intended use, effect of error upon the measurement process, and prior experience. Calibration frequency is given in the method working SOP's that are in every CLSB laboratory.

4.1.1.3 Calibration Reference Standards

Two types of reference standards are used within every CLSB laboratory for calibration:

- PHYSICAL STANDARDS such as weights for calibrating balances and certified thermometers for calibrating working thermometers and ovens. These are generally used for periodic calibration.

- CHEMICAL STANDARDS such as Standard Reference Materials (SRMs) provided by the National Institute of Standards and Technology (NIST), EPA check standards, laboratory control standards or working (calibration) standards.

Whenever possible, physical reference standards shall have known relationships to nationally recognized standards (e.g., NIST) or accepted values of natural physical constants. If national standards do not exist, the basis for the reference standard shall be documented.

Physical reference standards shall be used only for calibration and shall be stored separately from equipment used in analyses.

In general, physical reference standards shall be at least four to ten times as accurate as the requirements for the equipment that they are used to calibrate; physical standards should be recalibrated every three years by a certified external agency.

Whenever possible, chemical reference standards shall be directly traceable to NIST SRMs. If SRMs are not available, compounds of certified high purity will be used to prepare calibration standards.

4.1.1.4 Calibration Records

Records shall be maintained for each piece of equipment subject to calibration. Records demonstrating accuracy of reference standards shall also be maintained.

Records for periodically calibrated equipment shall include, as appropriate:

- Identification number of equipment and type of equipment
- Calibration frequency and acceptable tolerances
- Identification of calibration procedure used
- Date calibration was performed
- Identity of CLSB personnel and/or external agencies performing calibration
- Reference standards used for calibration
- Calibration data
- Certificates or statements of calibration provided by manufacturers and external agencies, and traceability to national standards
- Information regarding calibration acceptance or failure and any repair of failed equipment

Records for periodically calibrated equipment shall be maintained by the Instrument or in a secure location that is accessible.

For instruments and equipment that are calibrated on an operational basis, calibration generally consists of determining instrumental response against compounds of known composition and concentration or the preparation of a standard response curve of the same compound at different concentrations. Records of these calibrations are maintained in several ways:

- The calibration data are kept with analytical sample data, and/or
- A logbook is prepared for each instrument that contains all calibration data.

The former method provides response factor information directly with the analytical raw data so that the data can be readily processed and verified. The latter method provides an ongoing record of the calibration undertaken for a specific instrument.

CLSB LABS

ION CHROMATOGRAPHY RUN LOG
EPA M300.0, IC 101

Date: _____ Analyst: _____ Anion: _____

Check Stnd ID: _____ Spike Stnd ID: _____ Calibration Date: _____

	SAMPLE ID	DF		SAMPLE ID	DF
1			25		
2			26		
3			27		
4			28		
5			29		
6			30		
7			31		
8			32		
9			33		
10			34		
11			35		
12			36		
13			37		
14			38		
15			39		
16			40		
17			41		
18			42		
19			43		
20			44		
21			45		
22			46		
23			47		
24			48		

Batch #:_____

Figure 4-1 Instrument Use Log

4.1.2 Operational Calibration

Operational calibration is performed as part of the analytical procedure. Included are the analysis of a method blank and the preparation of a standard response (standard calibration) curve.

A brief discussion of the analysis of method blanks and preparation of standard curves and guidelines for the major instrument systems within the laboratory follows.

4.1.2.1 General Calibration Procedures

The initial phase of a laboratory-testing program requires the selection and certification of the method best suited for an individual parameter. Certification, or verification, is the elimination or minimization of determinate errors that may be due to analyst error, the use of less-than-optimum equipment, reagents, solvents or gases. The quality of materials, even though they are AR grade or better, may vary from one source to another. The analyst must determine, through the use of reagent and/or solvent blanks, if materials are free from interfering substances that could affect the analysis. Other steps in certifying the method include the determination of a method blank and the preparation of a standard calibration curve.

4.1.2.1.1 Method Blank

After determining the individual reagent or solvent blanks, the analyst defines the method blank to determine if the cumulative blank interferes with the analysis. The method blank is defined by following the procedure step by step, including the addition of all of the reagents and solvents, in the quantity required by the method. If the cumulative blank interferes with the determination, steps must be taken to eliminate or reduce the interference to a level that will permit the combination of solvents and reagents to be used.

A method blank should be determined whenever an analysis is made. The number of blanks is determined by the method of analysis and the number of samples analyzed at a given time.

4.1.2.1.2 Standard Calibration Curve

Concurrent with the preparation of reagent and method blanks, a standard calibration curve is prepared for the instrumentation. Preparation of a standard calibration curve is accomplished by using calibration standards.

Calibration standards are also referred to as "working standards". They are prepared by mixing the species to be analyzed into the solvent that is to be introduced into the instrument.

The concentrations of the calibration standards are chosen to cover the working range of the instrument. All sample measurements are made within this working range. The calibration curve is prepared by plotting instrument response versus concentration of the species analyzed. Actual sample concentrations are then read directly from the calibration curve or determined by interpolation. Data reduction is done manually and/or by electronic data systems.

4.1.2.2 Calibration of the Gas Chromatograph and Gas Chromatograph/Mass Spectrometers

Calibration of the gas chromatographs or gas chromatograph/mass spectrometers for organic compound analyses is performed simultaneously with the standardization of the instrument. A five-point standard curve is initially analyzed to calibrate instrument response and to define the working range of the instrument for the compounds of interest.

After initial calibration is established, mid-point calibration standards are run to confirm continuing instrument calibration. The acceptance criteria are method specific and are strictly adhered to.

Response Factors (RF) are to be calculated for each compound at each concentration level (acceptable response factors are given in the individual method SOP's). These RF will be averaged to generate the mean RF for each compound over the range of the standard curve. The mean response factor will be used to calculate the sample concentration of the compound of interest. When sample responses exceed the range of the standard curve, the sample will be diluted to fall within the range of the standard curve and be reanalyzed. The results of the daily GC standardization will be tabulated and filed with the corresponding sample analyses.

4.1.2.3 Calibration of Inductively Coupled Plasma Spectrometer (ICP) and Atomic Absorption Spectrophotometer (AA)

The ICP and AA are standardized for the metal of interest by the analysis of a set of calibration standards prepared by diluting a stock solution of known concentration. Working standards are prepared by dilution of the stock standard. The concentration of the calibration standards is chosen so as to cover the working range of the instrument. Subsequently, all sample measurements are made within this working range. Once the working standards are prepared, they are analyzed on the ICP or AA, and the instrument response is calibrated to provide a direct readout in milligrams of metal per liter of water.

The calibration is accomplished by inputting the metal concentration equivalent to the readout in absorbance units during analysis of the working standards.

Once the instrument has been initially calibrated, the analysis of the working standards is repeated during sample analysis to standardize instrument response during analysis and confirm the calibration settings. A typical analysis sequence is as follows:

- Working standards are prepared by dilution of a stock standard solution for the metal of interest.
- A calibration curve within the working range of the instrument is established by analysis of five working standards.
- The working standards are reanalyzed to confirm the calibration settings. If the calibration settings are not confirmed, the instrument is recalibrated.
- The samples are analyzed for the metal of interest.
- During sample analysis, a midpoint standard is analyzed to monitor instrument stability. If the analysis indicates that instrument calibration has changed, the instrument is recalibrated and the analysis is repeated.
- Following completion of the sample analyses, the working standards are reanalyzed to confirm calibration settings. If calibration settings are confirmed, the analysis is completed. However, if the calibration settings are not confirmed, the problem is corrected and the analyses are repeated.
- Analysis data may be input (if available) into a computer data file for later calculation and normalization for matrix effects.

4.1.3 Periodic Calibration

Periodic calibration shall be performed for equipment such as balances, thermometers, ovens and furnaces that are required in analytical methods, but that are not routinely calibrated as part of the analytical procedure. Documentation of calibration shall be kept for each equipment item.

Calibration requirements are determined within the laboratory depending upon the equipment used and its operating function. Following is an example for the calibration of balances with examples of a calibration data sheet to serve as a guideline for the preparation of laboratory-specific procedures.

4.1.3.1 Balances

All balances shall be calibrated weekly using weights traceable to the National Institute of Standards and Technology (NIST). Calibration weights shall be Class S or better. Balances are calibrated by an external agency three times per year.

Calibration of balances shall be to approximately 95 percent of balance capacity. Acceptance for balances that are direct reading to 0.01 gram shall be ± 0.01 g for 0 to 100 g and ± 0.1 percent of the applied weight for more than 100 g. Figure 4-2 provides an example data sheet that can be used for balance calibration.

Balance #: _____ Balance Name:_____ Balance S/N:_____

Date	Check Weight	Balance Reading	In Range? *	Comment	Initial

* Tolerance ranges are in the front of this logbook

Figure 4-2 Balance Log

4.2 INSTRUMENT MAINTENANCE AND REPAIR

The purpose of instrument maintenance is to maintain proper equipment performance and to prevent instruments and equipment from failing during use. An adequate maintenance program increases reliability of a measurement system and will include equipment cleaning, lubricating, reconditioning, adjustment and/or testing.

Within the laboratory, the Laboratory Director is responsible for preparation and documentation of the program. Department Supervisors shall implement the program, and the QC Manager shall review implementation to verify compliance.

CLSB's maintenance program considers several factors:

4.2.1 Instruments, equipment and parts that are subject to wear, deterioration or other change in operational characteristics without periodic maintenance.

4.2.2 The availability of spare parts within the laboratory to minimize downtime.

4.2.3 Frequency that preventive maintenance is required.

Preventive maintenance is performed on a routine basis and documented by department supervisors in a maintenance logbook assigned to each instrument. It should be noted if parts are replaced or if the instrument has deteriorated from use, etc. Figure 4-3 illustrates one type of maintenance log currently in use.

Instrument ID: _____

Date	Time	Name	Maintenance/Service/Parts/Remarks

Figure 4-3 Instrument Maintenance Log

5.0 QUALITY CONTROL SAMPLE ANALYSIS

This section discusses samples that are routinely added to the normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. Quality control samples are of known content and concentration (with the exception of field blanks) to ensure that accuracy and precision can be determined and control charts can be prepared. Evaluation of this data is discussed in Section 8.1.

The following is a discussion of the major types of quality control samples. QC samples will be analyzed as recommended herein, unless analytical procedures prescribe other specific QC sample analysis. If the procedure is specific, the procedural requirements will be met.

As stated, Section 8.1 presents the statistical analyses of these samples.

5.1 ANALYSES AND FREQUENCY OF BLANKS

5.1.1 Trip Blank Analyses

Volatile organics samples are susceptible to contamination by diffusion of organic contaminants through the Teflon-faced silicone rubber septum of the sample vial; therefore, trip blanks shall be analyzed to monitor for possible sample contamination during shipment. Trip blanks will be prepared by filling two VOA vials with organic-free water and shipping the blanks with the field kit. Trip blanks accompany the sample bottles through collection and shipment to the laboratory and are stored with the samples. Following the analyses, if the trip blanks indicate possible contamination of the samples, depending upon the nature and extent of the contamination, the samples may be corrected for the trip blank concentration or the sources re-sampled.

Results of trip blank analyses should be maintained with the corresponding sample analytical data in the project file.

5.1.2 Method Blank Analyses

A method blank is a volume of deionized laboratory water for water samples, or a purified solid matrix for soil/sediment samples, carried through the entire analytical procedure. The volume or weight of the blank must be approximately equal to the sample volume or sample weight processed. A method blank should be performed with each analytical batch of samples. Analysis of the blank verifies that method interferences caused by contaminants in solvents, reagents, glassware and other sample processing are known and minimized. Results of method blank analyses will be maintained with the corresponding analytical data in the project file. For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank of any analyte of concern must be no higher than the detection limit.

5.1.3 Holding Blank Analyses

On a regular basis, a set of VOA vials containing organic-free water are prepared and stored along with client samples in the VOA-storage refrigerator. After one week, the holding blanks are analyzed by GC for possible organic contamination. If compounds are detected, the QC Manager will initiate a corrective action.

5.2 ANALYSES AND FREQUENCY OF REPLICATES

5.2.1 Replicate Sample Analyses

Replicate analyses are performed to evaluate the precision of an analysis. Results of the replicate analyses are used to determine the relative difference between replicate samples. Criteria for evaluating replicate sample results are provided in Section 8.1. A replicate analysis should be performed on every group of twenty samples analyzed. Replicate analysis results should be summarized on the quality control data summary form.

The frequency of replicates is specified in many analyses, and CLSB analyzes, as a minimum, the percentage of replicate specified. The replicate aliquots are carried through the entire workup and analytical process.

Care is taken to assure that soils and hazardous wastes are replicated at least as frequently as waters and wastewaters.

5.2.2 Blind Replicate Analysis

A blind replicate sample is a replicate sample that has been introduced as a separate sample by the Quality Control Manager during the log-in process or prior to analysis. Evaluation of the replicate is discussed in Section 8.1. This data is reported to and summarized by the Quality Control Manager.

5.3 ANALYSES AND FREQUENCY OF SPIKED SAMPLES

Samples are spiked with known amounts of chemical entities being measured in order to determine the percent recovery.

5.3.1 Matrix Spikes

At least one matrix spike (MS) and one matrix spike duplicate (MSD) will be analyzed per analytical batch (if not enough sample is available for a matrix spike, then a Laboratory Control Sample and a Laboratory Control Sample Duplicate will be used as QC samples). A matrix spike is defined as a sample matrix that has predetermined quantities of stock solutions of certain analytes added prior to sample extraction/digestion and analysis.

To evaluate the effect of the sample matrix upon analytical methodology, a separate aliquot sample should be spiked with the analyte of interest and analyzed with the sample. The percent recovery for the respective analyte will then be calculated.

If the percent recovery falls outside quality control limits, the data should be evaluated and the sample reanalyzed if criteria are not met. Matrix spike results should be summarized on the quality control data summary sheets.

5.3.2 Regulatory Spikes

When sample analysis requires values within a specified percent recovery of a regulatory limit, the sample will be spiked with a standard of concentration (suggested in the method) and the spiked sample analyzed. Recoveries are calculated and reported on a percent basis. In this manner, the spike serves to provide information on accuracy of the procedure.

5.3.3 Replicate Spikes

Certain methods specify running replicate spikes. A regulatory spike is a subpart of replicate spike. Frequently, the replicate spike is run at one to five times the concentration of the observed sample value or at one to five times the background level, depending on method requirements.

5.4 STANDARDS AND REFERENCE MATERIALS

Standards and reference materials will be obtained per procedures specified in Section 3. Proper laboratory procedure requires the use of the following types of standards and reference materials.

5.4.1 Quality Control Samples

A Liquid Control Sample (LCS) will be processed with each analytical batch. A LCS is defined as a known matrix spiked with compound(s) representative of the target compounds, which is run through the entire analytical procedure. Only the LCS needs to be reported, if the MS or MSD fail their parameters. The results of the LCS are compared to control limits established for both precision and bias to help determine the usability of the data.

5.4.2 Working or Calibration Standards

Calibration of instruments such as GC, ICP, and AA requires use of standard solutions. These calibration standards are carefully prepared by volumetric or gravimetric methods and standardized against the laboratory control standards before use in the laboratory.

Because instrument response and calibration curves are subject to change and can vary from day to day, a midpoint standard or check standard will be analyzed at the beginning of analysis, every 10 samples thereafter and at the end of sample analysis.

Analysis of this standard is necessary to verify the standard curve and may serve in some cases to be sufficient for calibration. This value should be entered in the instrument calibration log whenever performed.

5.4.3 Internal Standards

An internal standard is a known amount of a compound not normally found in environmental samples added to each sample before analysis. The area of internal standard peak in the calibration standard is used as a reference to monitor the area of the internal standard peak in the sample. The internal standard peak in the sample is the reference peak used for the calculation of the concentrations of the compounds present in the sample. The retention time of the internal standard is monitored by the analyst to ensure there is minimal change.

5.4.4 Surrogates

A surrogate is an organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. Surrogates are typically spiked into the samples prior to extraction and thereby provide recovery data for sample workup. Although such data is typically derived from compounds closely related to the compounds under investigation, it is not compound-specific and in the strict sense should not be used for making corrections for recovery. Since the information is provided with every sample, it is nevertheless very useful in detecting both sample-specific and systematic recovery problems. Surrogates will be run on all organic analysis including spikes and blanks. If surrogate recoveries exceed their specified control limits corrective action will be implemented as specified in the individual method SOPs.

5.4.5 Certified Reference Materials

On a regular basis, the Quality Control Manager should introduce a group of prepared verification samples (Certified Reference Materials) into the analytical testing regime. Results of these data will be summarized, evaluated and presented to laboratory management for review and corrective actions, if appropriate. The data are reported to and summarized by the Quality Control Manager. Certified Reference Materials are acquired from sources which are accredited by the State of California to provide such samples.

5.4.6 QC Batches

A number of samples of similar matrix, origin and composition which are analyzed together with the same method sequence and the same lots of reagents with manipulations common to each sample within the same time period or in continuous sequential time periods shall be known as a QC batch.

The number of total samples in a QC batch should not exceed twenty samples plus the number of samples required to perform QC evaluation of the twenty initial samples.

A sequential number will be assigned to each QC batch prior to batching a number of samples. This number will be obtained by using the next sequential number available as recorded in the QC Batch Log Book.

The QC Batch Log Book will contain information pertaining to each QC batch and will include the sequential batch number, date, analysis to be performed, analyst who assigned the batch, the sample numbers to be analyzed, and additional notes as required.

5.5 INTER/INTRA-LABORATORY PERFORMANCE EVALUATIONS

The performance of CLSB's laboratories is monitored by the participation in the Environmental Laboratory Accreditation Program of the State of California. This program includes the Water Pollution (WP) and Water Supply (WS) programs. Under these programs, blind samples with known concentrations are analyzed annually. CLSB obtains intra-laboratory PE samples from commercial companies such as Environmental Resource Associates.

The chemists, the Laboratory Manager and the Laboratory Director are kept informed of all inter/intra-laboratory performance evaluations. If a method fails or is found to be suspect, appropriate corrective actions are taken immediately. If any results are found to be outside the established control limits, the method will be evaluated and the problem resolved prior to performing any additional tests.

6.0 PERFORMANCE AND SYSTEMS AUDIT

A QA audit is an independent assessment of the measurement system. The purpose of the performance audit is to qualitatively and quantitatively assess the data output generated at any level within the laboratory during the data collection. The results of the audit are formulated into a report detailing the overall system performance and deficiencies, plus any recommendations.

6.1 QUALITY ASSURANCE AUDITS

The QA Manager will perform performance audits. Audits are considered an essential part of the CLSB Quality Assurance Program. CLSB conducts two types of audits; a system audit to qualitatively evaluate the operational details of the QA program, and a performance audit to evaluate the quantitative outputs of all measurement systems.

These audits are combined into one summary audit. The audit includes: (1) laboratory inspection to ensure the laboratory, instruments and equipment, etc., are kept in good condition, and all records of standard preparation, calibration, sample preparations, etc., are documented; (2) data validation: selected tests/reports will be audited, and the complete QC package from log-in to report generation will be checked; (3) Assessment of QC sample analysis; (4) Record filing and retreivability. A checklist will be used by CLSB QA personnel when performing audits to assure that nothing is overlooked. Major elements of the audits are listed below:

- o SOPs are available and updated
- o Standards are not expired
- o Lab notebooks have been signed and reviewed
- o Instrument performance and logs are updated
- o Properly trained chemist(s) are performing analysis
- o Traceability of all analysis
- o Safety practices of laboratory personnel

The audit results will be documented and given to the laboratory director and all managers, as well as being available for review by the company President.

6.2 SUBCONTRACT LABORATORIES

CLSB periodically sends samples to other laboratories for analysis which are not performed by CLSB. Before CLSB sends samples to a contract laboratory, CLSB requires a current ELAP certificate, QA Manual, and WS/WP results. In addition, CLSB may submit QA samples to assure sample integrity.

7.0 ANALYTICAL PROCEDURES

CLSB utilizes USEPA prescribed methods whenever applicable. Other sources of analytical methods may be used for other analyses if widely recognized by industrial and government laboratories. Industry standard methods are published by USEPA, American Public Health Association (APHA), American Society for Testing and Materials (ASTM), National Institute for Occupational Safety and Health (NIOSH), and American Industrial Hygiene Association (AIHA).

A brief summary of the method sources for performing "certified" analyses, as well as other commonly used references, is located in Table 7-1.

Analysis will be performed in accordance with the methods cited herein unless specific project requirements or needs dictate modification of the cited methods or adoption of an alternate method. If analysis is performed in an alternate manner, the method shall be documented in the project records.

Accurate environmental analysis involves the need for several activities to be performed in coordination with or coincidental to actual analysis; e.g. 1.) sample procurement and storage (Section 2) to preserve sample integrity, 2.) Instrument calibration, 3.) Analysis of QC samples and standards to assess recovery, matrix affects, range within linearity, 4.) Extraction of the analytes from the matrix. Each of these aspects is discussed elsewhere in this manual.

7.1 SOPs

CLSB relies heavily on the use of Standard Operating Procedures (SOPs). CLSB's SOPs not only include the instrumentation and method procedures but also include all aspects of the complete analytical process, from sample receipt to waste disposal.

No procedure or task is accepted for use until an appropriate SOP has been written and approved by both the QA/QC Manager and Laboratory Director. The QA/QC Manager reviews all SOPs annually. SOPs are kept in the appropriate lab areas, readily available to each analyst.

All laboratory method SOPs should include the following elements if applicable:

1) Title of the method.
2) Effective date of the method.
3) Scope and application including a list of analytes, matrices and detection limits.
4) Summary of the method.
5) Definition of terms.
6) Health and safety considerations.
7) Sample handling and preservation considerations.
8) Effect of potential interferences.
9) Apparatus and materials including reagents, equipment and instruments.
10) Quality control criteria defined in detail .
11) Procedures for the analysis of samples.
12) Documentation: a list of items to be included in the project folder.
13) References for Method .
14) A sample run log is attached to SOP.
15) Standards Preparation is in SOP in detail for CAL ,CCV,LCS , MS &MSD.

SOPs are written in a numbered outline format with the following major headings:

1.0 Purpose
2.0 Scope and Application
3.0 Method Detection Limits
4.0 Applicable Matrx or Matrices
5.0 Method Summary
6.0 Definitions
7.0 Contamination and Interferences
8.0 Apparatus and Materials
9.0 Reagents and Standards
10.0 Sample Collection , preservation , shipment and storage .
11.0 Quality Control
12.0 Calibration and Standarization
13.0 Procedure
14.0 Calculations
15.0 Method Performance

16.0 Pollution Prevention.
17.0 Data Assessment and Acceptance Criteria
18.0 Corrective Actions for Out of Control Data
19.0 Contingencies for Out of Control Data.
20.0 Waste Management .
21.0 References
22.0 Tables , Diagrams , Flowcharts ,etc.
23.0 Training and Qualification Validation
24 0 Health and Safety

Definition and Procedure for the Determination of the Method Detection Limit,□ Code of Federal Regulations (CFR) 40, Part136, Appendix B, Revised July 1995.

Drinking Water Methods from Methods for the Determination of Organic Compounds in Drinking Water,□ EPA 600/4- 88/039, December 1988, Revised July 1991.

Drinking Water Methods from Methods for the Determination of Organic Compounds in Drinking Water-Supplement I,□ EPA 600/4-90/020, July 1990.

Drinking Water Methods from Methods for the Determination of Organic Compounds in Drinking Water-Supplement II,□ EPA 600/R-92/129, August 1992.

Inductively Coupled Plasma-Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes,□ CFR 40, Part 136, Appendix C, Revised July 1995.

Methods for Chemical Analysis of Water and Wastes (MCAWW),□ EPA 600/4-79-020, Revised, March 1983.

Methods for the Determination of Metals in Environmental Samples-Supplement I,□ EPA 600/R-94-111, May 1994.

Methods for the Determination of Inorganic Substances in Environmental Samples,□ EPA 600/R-93-100, August 1993.

"Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater," EPA 600/4-82-057, July 1982.

Methods for Organic Chemical analysis of Municipal and Industrial Wastewater,□ CFR 40, Part 136, Appendix A, July 1995.

"Methods for the Determination of Organic Compounds in Drinking Water", EPA 600/4-88/039, December 1988.

Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater - Volume I,□ EPA 821/R-93-010-A, August 1993, Revision 1.

Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater - Volume II,□ EPA 821/R-93-010B, August 1993.

Method 1664: N-Hexane Extractable Material (HEM) and Silical Gel Treated N-Hexane Extractable Material (SGT- HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons), EPA 821/B-94-0046, April 1995.

Precision and Recovery Statements for Methods for Measuring Metals,□ CFR 40, Part 136, Appendix D, Revised July 1995

"Standard Methods for the Examination of Waste and Wastewater," 18th Ed. APHA-AWWA-WPCF, 1992.

Technical Notes on Drinking Water Methods,□ EPA 600/R-94-173, October 1994

A number of additional methods are summarized in the "AB 1803 -Methods Manual" issued by the California Department of Health Services, 1984.1994.

Test Methods for Evaluating Solid Waste,□ USEPA SW-846, December 1996, Third Edition Update III.

Federal Register, 40 CFR Part 136, Oct. 26, 1984.

Table 7-1 Basic References for Analytical Methods

8.0 QUALITY CONTROL DATA PROCESSING AND VALIDATION

Data processing and validation within the analytical laboratory ensure that the reported results will correctly represent the analyses performed. This function has two primary activities:

- The processing of quality control sample results to demonstrate that analyses are within laboratory prescribed bounds for accuracy, precision and completeness.
- Sample reduction and validation to demonstrate that numerical computation of data is correct and that it is correctly reported.

This section discusses the computation process and Section 9.0 discusses maintenance of resulting records.

8.1 PROCESSING OF QUALITY CONTROL DATA

This section discusses the analytical treatment of the data resulting from the quality control samples discussed in Section 5.

8.1.1 Assessment of Data Precision and Accuracy

All data generated must be evaluated for precision and accuracy by the following procedure. Quality control sample analyses are performed as appropriate for organic or inorganic samples as discussed in Section 5. The protocol used will be in accordance with specific method analytical procedures if QC requirements are stated in the procedure.

8.1.1.1 Frequency and Types of QC Samples :

Reagent or Method Blank - A reagent and/or method blank is prepared and analyzed with each batch of samples.

Trip Blank - Trip blanks are analyzed to determine possible sample contamination during collection and shipment to the laboratory. Trip blanks are applicable to volatile organics analysis (VOA) where volatile contaminants can be introduced from ambient air on site, during shipment, and in the laboratory.

Calibration Curve - A calibration curve consisting of standards and a reagent blank are prepared for each parameter. If the standard curve is within acceptance criteria for the method in use, the standard curve will be verified by the analysis of a midpoint standard.

Initial and Continuing Calibration Verification (ICV & CCV) - A Standard of reagent water or solvent that is spiked with a standard of the analytes from a second source standard . It is used to verify the calibration curve initially and continuously . These samples are run every ten samples with CCB. Recovery of the analyte is recorded .

Initial Calibration and Continuing Calibration Blank (ICB and CCB) – A Reagent blank that is run after CCV to check system cleanliness .

Liquid Control Samples(LCS) – A reagent water or solvent that is spiked with analytes from a second source standard . This sample is run through the entire analytical procedure as a sample and recovery of analyte is recorded .

Matrix Spike & Duplicate - As a minimum, one sample in every sample set of twenty samples is spiked twice at a mid-concentration level to provide a final concentration within the expected range of the samples.

Blind Replicate - A blind replicate, unknown to the analyst, is introduced by the Quality Control Department quarterly. Blind replicates are routinely used for the analysis of metals, water quality parameters and organics analyses that do not require separate extraction.

Certified Reference Materials - Certified Reference Materials are introduced at least annually into the testing scheme by the Quality Control Manager to evaluate the testing procedure and the analyst's performance.

Check Standards - A check standard consisting of deionized water spiked with the parameter of interest is analyzed. Check standards are routinely used for the analysis of metals, water quality parameters and some organics parameters.

Surrogate Standard Spike - Every sample is spiked with the required and appropriate surrogate standards prior to extraction and analysis for volatile and semi-volatile organic compounds.

Internal standards - Internal standards are added to samples as prescribed in each specific method.

Quality Control Samples - Quality Control Samples are required twice per analytical batch.

8.1.1.2 Acceptable Limits of QC Samples

When the analyses of a sample set are completed, the results will be reviewed and evaluated to assess the validity of the data set. Review is based on the following criteria:

Method Blank Evaluation - The method blank results are evaluated for high readings characteristic of background contamination. If high blank values are observed, laboratory glassware and reagents should be checked for contamination and the analysis halted until the system can be brought under control. For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank of any analyte of concern must be no higher than the reporting limit.

Trip Blank Evaluation - Trip blank results are evaluated for high readings similar to the reagent and/or method blanks described above. If high trip blank readings are encountered, the procedure for sample collection, shipment and laboratory analysis should be reviewed. If both the reagent and/or method blanks and the trip blanks exhibit significant background contamination, the source of contamination is probably within the laboratory. In the case of VOA, ambient air in the laboratory and reagents should be checked as possible sources of contamination. High trip blank readings for other parameters may be due to contaminated sample bottles or cross-contamination due to sample leakage and poorly sealed sample containers.

Calibration Standard Evaluation - The calibration curve is evaluated to determine linearity through its full range, and that sample values are within the range defined by the low and high standards. If the curve is not linear, sample values must be corrected for nonlinearity by deriving sample concentrations from a graph or by using an appropriate algorithm to fit a nonlinear curve to the standards.

Replicate Sample Evaluation - Replicate sample analysis for the sample set is used to determine the precision of the analytical method for the sample matrix. The replicate results are used to calculate the precision as defined by the relative percent difference (RPD). The precision value, RPD, should be plotted on control charts for the parameter determined. If the precision value exceeds the warning limit for the given parameter, the appropriate Department Supervisor, Laboratory Director or the Quality Control Manager is notified. If the precision value exceeds the control limit, the sample set must be reanalyzed for the parameter in question.

Matrix Spike Evaluation - The observed recovery of the spike versus the theoretical spike recovery is used to calculate accuracy as defined by the percent recovery. The accuracy value, (percent recovery) may be plotted on a control chart for the parameter determined. If the accuracy value exceeds the warning limit for the given parameter, the appropriate Supervisor, Manager or the Quality Control Manager is notified.

Blind Replicate Evaluation - The blind replicate analysis is evaluated in the same manner as described above for the replicate sample analysis and is treated as a replicate result for purposes of evaluating the precision of the analytical method.

Reference Standard Evaluation - Standard Reference Materials analyses are compared with true values and acceptable ranges. Values outside the acceptable ranges require corrective action to determine the source of error and provide correction action. All sample analyses should be halted pending this evaluation. Following correction of the problem, the Standard Reference Material should be reanalyzed.

Quality Control Sample Evaluation - The results of the Quality Control Sample analysis are compared with the true values, and the percent recovery of the sample is calculated. If correction is required, the control sample and the samples in its batch should be reanalyzed to demonstrate that the corrective action has been successful.

Surrogate Standard Evaluation - The results of surrogate standard determinations are compared with the true values spiked into the sample matrix prior to extraction and analysis and the percent recoveries of the surrogate standards are determined. For aqueous matrices, these percent recoveries should be compared with the laboratory generated control limits.

8.1.2 Statistical Evaluation of QC Data

As part of the analytical quality control program, CLSB determines precision and accuracy for each parameter analyzed. These values can be used as control limits for pass/fail criteria.

Initially, when these data are compiled, the evaluation is applied over a broad concentration range. As more data is accumulated, precision and accuracy determinations are updated and criteria developed to define precision and accuracy over specific concentration ranges.

8.1.2.1 Control Chart Evaluation

Precision and accuracy criteria will be applied to each parameter that is analyzed. When analysis of a sample set is completed, the quality control data may be reviewed and evaluated through the use of control charts to validate the data set. Control charts are derived from data that has been entered into the LIMS.

Control charts may be established for all major analytical parameters.

A minimum of seven measurements of precision and accuracy are required before control limits of two standard deviations shall be considered valid. Once established, control limits are updated as additional precision and accuracy data become available by the Quality Control Manager.

8.1.2.1.1 Analytical Precision

General Considerations

To determine the precision of the method and/or laboratory analyst, a routine program of replicate analyses is performed. The results of the replicate analyses are used to calculate the relative percent difference (RPD), which is the governing quality control parameter for precision.

The RPD for replicate analyses is defined as 100 times the difference (range) of each replicate set, divided by the average value (mean) of the replicate set. For replicate results D_1 and D_2, the RPD is calculated from:

$$RPD = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}}$$

When the RPD is obtained for at least seven replicate pairs, the average RPD and the standard deviation are calculated using:

$$\overline{m} = \frac{\sum_{i=1}^{n} m_i}{n}$$

and

$$S_m = \sqrt{\frac{\sum_{i-1}^{n} (m_i - \overline{m})^2}{n-1}}$$

where,

m_i = the RPD of a replicate pair,
m = the average of the Relative Percent Difference determination,
S_m = the standard deviation of the data set of RPD determinations,
n = the number of RPD determinations.

When constructing a control chart for a specific parameter, the Warning and Control Limits are then calculated from the following:

Upper Control Limit = $\overline{m} + 3S_m$
Lower Control Limit = $\overline{m} - 3S_m$
Upper Warning Limit = $\overline{m} + 2S_m$
Lower Warning Limit = $\overline{m} - 2S_m$

A control chart is established by plotting the RPD of each replicate pair on a graph generated as follows:

- The average of the RPD determinations for the original data set is established as the midpoint on the Y-axis of the graph.
- The Upper Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis above the mean of the RPD determinations.
- The calculated RPD of each replicate pair is plotted on the graph to determine whether the RPD is within the Warning and Control Limits of the Control Chart.

If the RPD plots between the Warning and Control Limits, the group leader, laboratory director or quality control manager is notified for a decision as to how to proceed.

- If the RPD plots outside the Control Limits, the data set is invalid and the analysis is stopped until the source of error has been determined and corrective action taken. Once the error source has been resolved, the data set is reanalyzed.

8.1.2.1.2 Analytical Accuracy

When a program for evaluation of analytical accuracy is established, the evaluation is applied over the entire range of spiking concentrations. As more data are accumulated, the evaluation procedure is refined to define the analytical accuracy of the method over specific concentration ranges.

To determine the accuracy of an analytical method and/or the laboratory analysis, a periodic program of sample spiking is conducted. The results of sample spiking are used to calculate the quality control parameter for accuracy evaluation, the Percent Recovery (%R).

The %R is defined as the observed concentration minus the sample concentration, divided by the true concentration of the spike, all multiplied by 100.

$$\%R = \frac{O_i - O_s}{T_1} x100$$

where

%R = The Percent Recovery,
O_i = The Observed Spiked Sample Concentration,
O_s = The Sample Concentration and
T_1 = The True Concentration of the Spike.

When the Percent Recovery is obtained for at least ten spiked samples, the mean percent recovery and the standard deviation are calculated using the formulae:

$$\%\ \overline{R} = \frac{\sum_{i=1}^{n} \%R_i}{n}$$

and

$$S_R = \sqrt{\frac{\sum_{i=1}^{n} (\%R_i - \%\overline{R})^2}{n-1}}$$

where

%R	=	the Mean Percent Recovery
$\%R_i$	=	the Percent Recovery of a Single Spiked Sample,
n	=	the number of results and
S_R	=	the Standard Deviation of the data set of Percent Recovery determinations.

The Warning and Control Limits are then calculated from the following equations:

Upper Control Limit = $\%R+3S_R$
Lower Control Limit = $\%R-3S_R$
Upper Warning Limit = $\%R+2S_R$
Lower Warning Limit = $\%R-2S_R$

A control chart (as shown in Figure 8-1) is generated by plotting the Percent Recovery data on a graph as follows:

- The average of the Percent Recovery determinations for the original data set is established as the midpoint on the Y-axis on the graph.
- The Upper Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis above the mean of the Percent Recovery determinations.

- The Lower Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis below the mean of the Percent Recovery determinations.

- The calculated Percent Recovery of each spiked sample is plotted on the graph to determine whether the Percent Recovery is within the Warning and Control Limits of the Control Chart.

- If the Percent Recovery plots between the Warning and Control Limits, the group leader, laboratory director or quality control manager is notified for a decision as to how to proceed.

- If the Percent Recovery plots outside the Control Limits, the data set is invalid and the analysis is stopped until the source of error has been determined and corrective action taken. Once the source has been corrected, the data set is reanalyzed.

- When an additional ten "Percent Recoveries" have been determined, the Warning and Control Limits are recalculated for the entire data set and the Control Chart for the corresponding parameter is updated.

The Quality Control Manager maintains all control charts.

8.1.2.2 Corrective Action/Out-of-Control Situations

In general, any result falling outside of control limits (generally set at ± 3 standard deviation units) will require initiation of corrective action. Whenever this situation occurs, it will be immediately brought to the attention of the QA Manager and the Laboratory Director.

The nature of corrective action will vary depending on interpretation of the seriousness of the situation by the QA Manager. Isolated outliers may be impossible to explain, and if warranted by previous and subsequent data, the outlier may be ignored. Consecutive recurrence of outliers will be viewed as indicative of a problem situation, and the process will be reviewed.

Most commonly, the out-of-control situation will require a series of corrective measures instituted to re-establish analytical validity. All analysis with the implicated method and instrumentation will be stopped until the problem is identified and resolved.

Review of recent historical data will be made to determine the time of the first variance from valid data, and data collected after that time discarded. Whenever possible all analyses performed after the last valid control check will be repeated.

Immediately following resolution of the out-of-control situation, an increased percentage of spikes and replicates will assure the situation is back to normal. This will continue until the QA Manager is satisfied that total resolution of the problem has occurred.

Control Chart

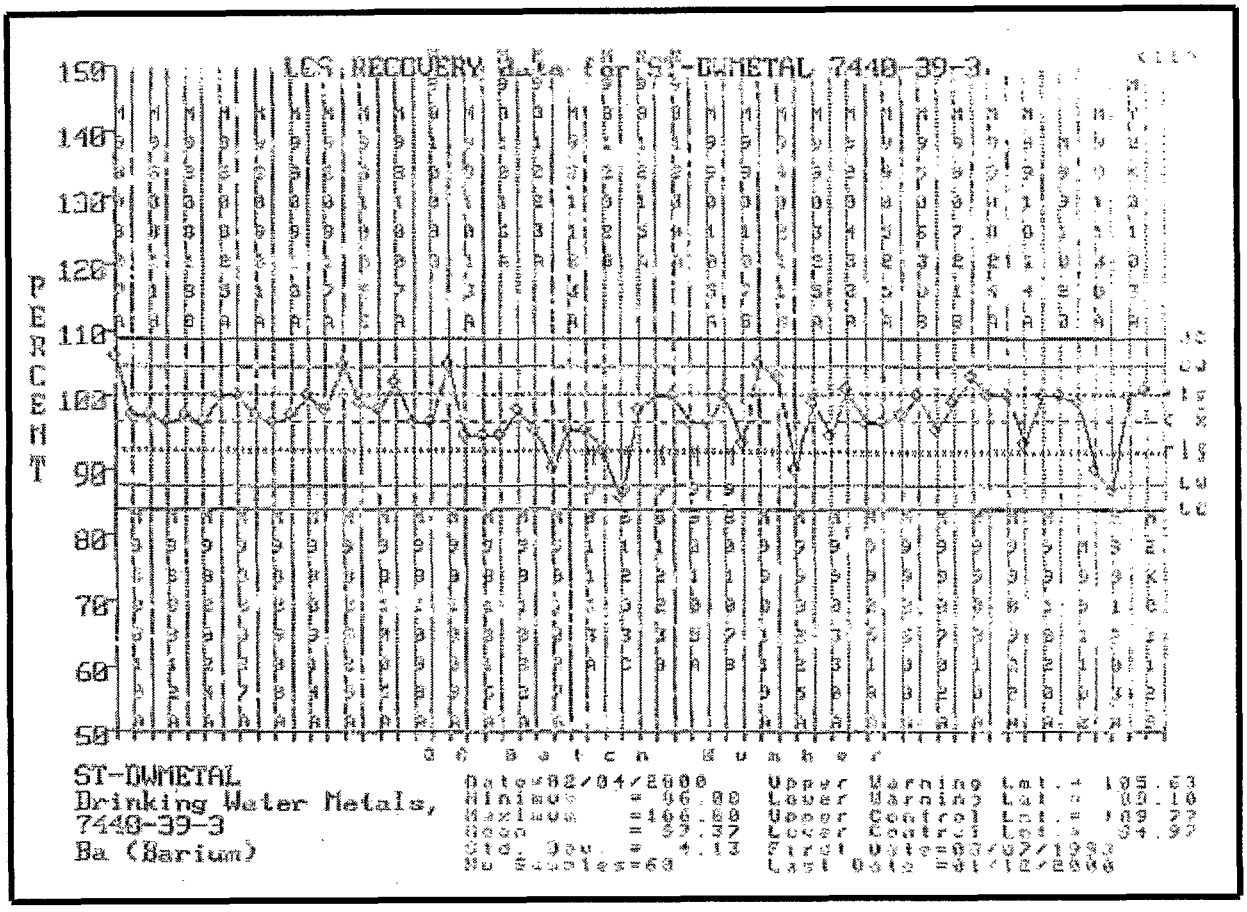


Figure 8-1 QC Control Chart

8.2 DATA VALIDATION

Data validation begins with the processing of data (including QC data) and continues through review of the final data and the reporting of analytical results. The analyst, independent of the data acquisition and processing, will perform data processing. The department supervisor reviews (validates) that the data processing has been correctly performed and continues by verifying that the reported analytical results correspond to the data acquired and processed. Final review of the data to be reported is by the Laboratory Director.

8.2.1 Data Processing

In general, an analyst will process data by:

1. Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets or
2. Input of raw data for computer processing or
3. Direct acquisition and processing of raw data by a data processing system (computer).

If the data is manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors, dilution factors and calibration constants. If calculations are not performed directly on the data sheet, calculations should be done on standard calculation paper and attached to the data sheets. The analyst shall sign and date in ink each page of calculations. Full signature and date in ink are required in all instances.

For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed shall be evident and the input signed and dated by the analyst.

If data are directly acquired from instrumentation and processed, the analyst shall verify that the following are correct: project and sample numbers, calibration constants and response factors, output parameters such as units and numerical values used for detection limits (if a value is reported as less than). The analyst shall sign and date the resulting output.

8.2.2 Review of Data Processing

Following is a discussion of the method to be used for reviewing (checking) data processing.

- The analyst performing the data processing shall give to another analyst, independent of the work, the data package. The package shall include, as appropriate, raw data, data sheets, strip charts, computer input/output, calculations, sources for input parameters such as response factors.

- The independent analyst (checker) shall review the data for:

  * Appropriateness of equations used.
  * Correctness of numerical input.
  * Numerical correctness of all calculations.
  * This should be done by performing numerical computations.
  * Correct interpretation of strip charts.

- All entries and calculations that the checker reviews shall be marked in ink with a check mark. The checking process must be thorough enough to validate that the results are correct. If the checker disagrees with any part of the computations, the checker shall mark through the number with a single line and place the revised number above it.

- Any changes made by the checker shall be re-checked by the originator. If the originator agrees with the change, no action is necessary. If the originator disagrees, the originator and checker must resolve the difference so they agree with the result presented.

- The checker shall sign originals and date in ink all pages of the data package (except for groups of printouts such as chromatograms). Signing and dating indicates that the reviewer agrees with the calculations and that the originator has agreed to any changes made.

- If the data have been processed by computer, the reviewer shall also check the input entries. If the checker disagrees with the input, the number should be marked through with a line and the corrected number indicated above it. Corrections must be re-checked by the originator as discussed above.

- If an input error is identified and the data has been processed, it will be necessary to reprocess the data. In this event, the checker shall mark the second set of input to indicate agreement with the input changes. The checker shall sign and date in ink the computer input to indicate agreement.

- Raw data that are automatically acquired and processed do not require any validation at this point beyond that previously discussed.

- The reviewed data are maintained as discussed in Section 9.

8.2.3 Review of Data Reporting

Review of data reports is required to verify that information reported by the laboratory corresponds with processed analytical results. Intermediate steps performed after the processed data are checked to prepare the data report (such as data summaries) do not require validation. Preparation of the report is the responsibility of the department supervisor or laboratory director.

After the draft data report is prepared (generally in tabular form), the reported results should be checked against the reviewed processed data so that transcription errors do not occur. The checking process follows:

o Using the draft report, all data entries are checked. The checker can be an analyst or department supervisor. The checker is not required to be independent of the work because only the transcription from the reviewed data to data report is being checked.

o The draft data report should be checked so that the items cited for data presentation in Section 9.0 are complete and correct. Corrected entries are marked through with a single line and the correct entry is provided. The reviewer will indicate that corrections have been made in the report by placing a second check mark by the correction after comparing the change with the revised copy. The checker shall sign and date every page of the data report in ink.

o Use of the draft data report results in checkprint that should be maintained as a record to demonstrate the review.

o If data printouts, such as chromatograms are included in the data report, review is not required for the data printout.

9.0 DATA REPORTS AND RECORDS MANAGEMENT

9.1 DATA REPORTS

The format and content of a data report is dependent upon project needs, such as: whether or not explanatory text is required, client or contract requirements, and government agency reporting formats. However, the final data presentation shall be checked in accordance with data verification requirements of Section 9 and approved by the Laboratory Director.

Data presentation reports also include:

- Sample identification number used by CLSB and/or the sample identification provided to the laboratory, if different than identification used in the laboratory.
- Chemical parameters analyzed, reported values and units of measurement.
- Reporting limit of the analytical procedure if the reported value is less than the reporting limit.
- Data for a chemical parameter are reported with consistent significant figures for all samples.
- Results of Quality Control sample analysis if appropriate.

9.2 RECORDS MANAGEMENT

CLSB maintains all records in two categories. Specific regulatory or contractual demands may require additional documentation and in these instances, records shall be maintained as externally required.

9.2.1 Project Specific Documents

These are records and documents pertinent to a project. Examples of individual project specific documents are correspondence, chain of custody and data reports.

9.2.2 General Laboratory Operation Documents

These documents demonstrate overall laboratory operation, such as instrument log books and control charts. These records will directly affect the data for a specific project, but in general their applicability is not limited to one project.

9.3 RETENTION OF RECORDS

Records and files with be archived chronologically by subject and retained for 5 years.

IID-421 (R16-87)

IMPERIAL IRRIGATION DISTRICT
P.O. Box 937 - 333 East Barioni Boulevard
Imperial, California 92251
(760)339-9220 General Information

LETTER OF TRANSMITTAL

Date: **July 7, 2004**	Job No.
Attn:	
RE: SOPs for IID water monitoring data	

TO **Melenee Emanuel**

State Water Resources Control Board
Division of Water Quality, Monitoring
1001 I Street
P.O. Box 944213
Sacramento, CA 95812

WE ARE SENDING YOU ☒ Attached ☐ Under separate cover via _____ the following items:

☐ Book ☐ CD Disk ☐ 3.5" Floppy Disk ☐ Samples ☐ Specifications
☐ Copy of letter ☐ Zip Disk ☐ Data Pages ☒ Reports ☐ Drawings

			DESCRIPTION
1	copy		Monthly anion/cation water monitoring Standard Operating Procedure (New River, Alamo River, All American Canal)
1	copy		Monthly anion/cation water monitoring Standard Operating Procedure (Salton Sea)

THESE ARE TRANSMITTED as checked below:

☐ For Approval ☐ Approved as Submitted ☐ Resubmit _____ Copies for approval
☐ For your use ☐ Approved as Noted ☐ Submit _____ Copies for distribution
☒ As requested ☐ Returned for corrections ☐ Return _____ Corrected prints
☐ For review and comment ☐ _____
☐ FOR BIDS DUE _____ 19 ____ ☐ PRINTS RETURNED AFTER LOAN TO US

REMARKS The attached SOPs are being submitted as supplementary information for water monitoring data submitted on 6/10/04. An SOP for IID's monthly coliform samples is not available.

COPY TO _____

SIGNED: Steve Charlton

Steve Charlton
Resources Planning & Management

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

Procedure: DCO-SILTTDS-01

Date Created/ Revision: 7/26/00, 3/6/01

Printed: 3/6/01 - 12:57

Associated Procedures: LAB-TST-01

Object: To collect water samples along the All American Canal using the DH-59 Sampler and the Grab method.

Schedule: Monthly

Personnel: Two (2) Data Technicians

Equipment:
DH-59 water sampler
Cable and reel
Life jackets
Work gloves
Sample containers (18 one-pint bottles; three one-quart and four-½ gallon jars)
Basket for Grab samples
Paper caps for pint bottles
Thermometer with 0°F to 120°F range
Pencil for labeling and noting observations
Heavy duty string or cord, 20-foot minimum length
Timepiece
IID keys
IID-430A (R3 12-70) Water and silt samples form (7 minimum)
IID Log book

Sites:
All American Canal (Coor. N32.70599 W114.96191) – approximately 6500 ft. downstream of Drop 1. Take Gordon's Well turn-off of I-8, 500' west along AAC bank, see *Fig.2.*
East Highline Canal (Coor. N32.70390 W115.28438) – approximately 2000 ft. downstream of Heading on west bank, see *Fig.3.*
Alamo River Inlet (Coor. N32.67455 W115.36996) – outlet headwall of river crossing at All American Canal, Grab Sample only, see *Fig.4.*
Central Main Canal (Coor. N32.69530 W115.46595) – approximately 4500 ft. downstream of Heading, near Acacia Heading off Bowker Road, see *Fig.5.*
Westside Main Canal (Coor. N32.67917 W115.67744) – approximately 500 ft. downstream of Hwy 98 crossing, see *Fig.6.*
Alamo River Outlet (Coor. N33.19865 W115.59621) – approximately 400 ft. upstream of intersection at Garst Rd. and Alamo River, Grab Sample only, see *Fig.7.*
New River Outlet (Coor. N33.10471 W115.66434) – approximately 4500 ft. west of the intersection at Lack Rd. and Vail Canal, Grab Sample only, see *Fig.8.*

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

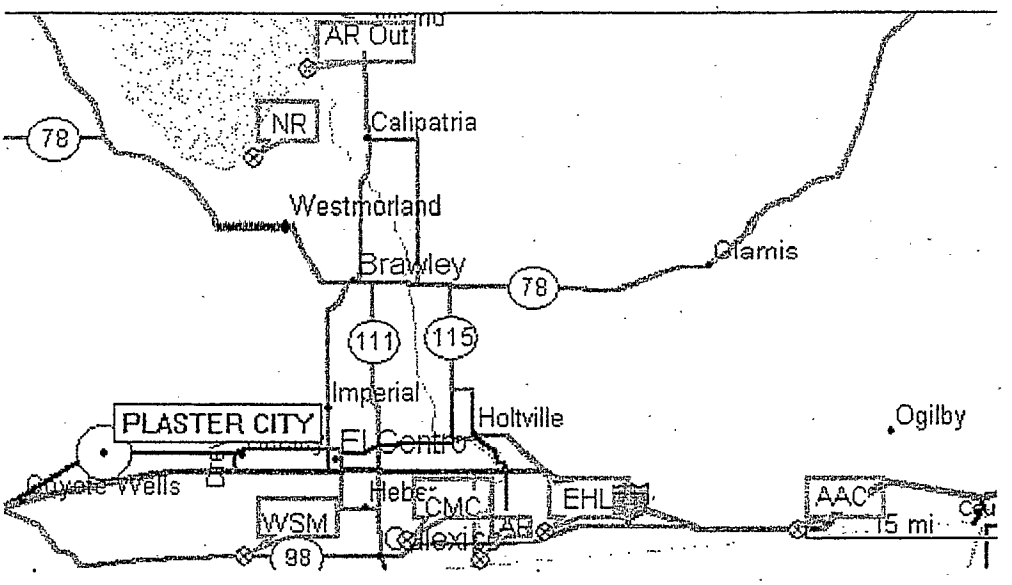


Fig. 1 – General location of water sampling sites: Alamo River Outlet (AR Out), New River Outlet (NR), Westside Main (WSM), Central Main (CMC), Alamo River Inlet (AR), East Highline (EHL), and All American Canal @ Drop 1 (AAC).

Following is a pictorial of all sites, refer to *Sites* on page 1.

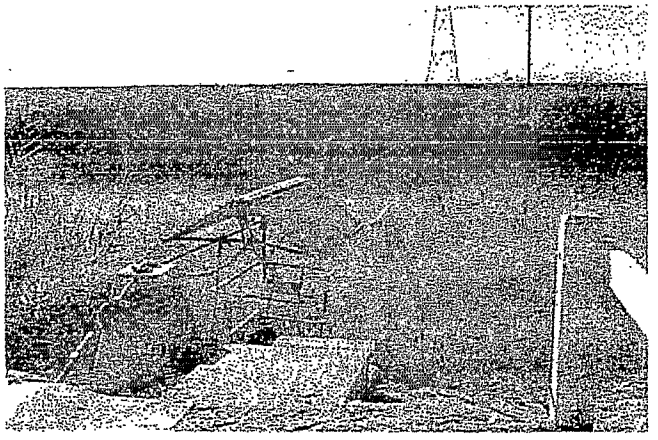


Fig. 2 – All American Canal at Drop 1, meter-cart in foreground

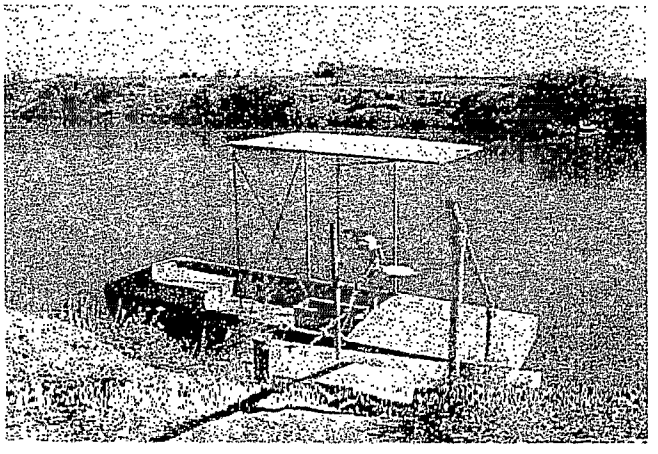


Fig. 3 – East Highline, meter-boat in foreground.

Fig. 4 – Alamo River @ Inlet, temperature and Grab Sample taken only.

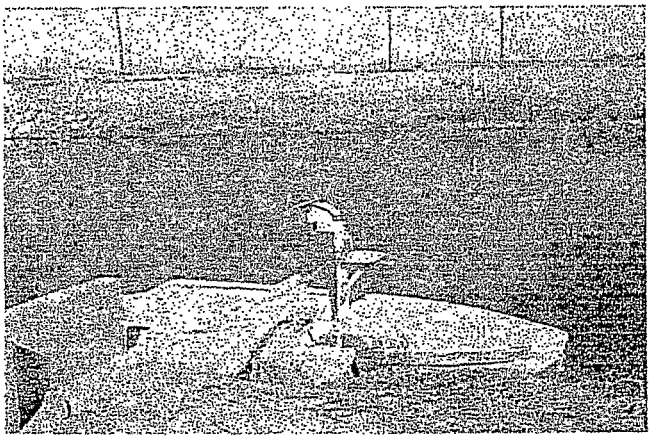


Fig. 5 – Central Main Canal. Meter-boat in foreground.

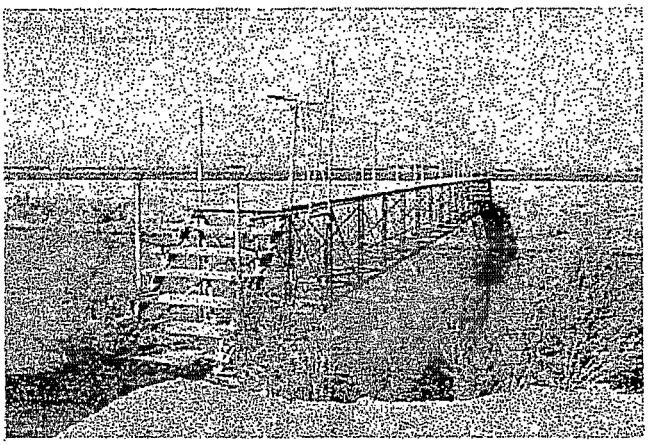


Fig. 6 – Westside Main Canal. Pictured is the meter-bridge that spans the channel.

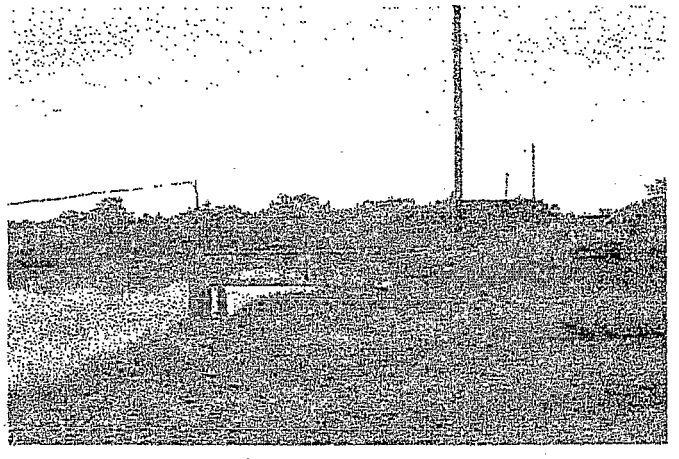


Fig. 7 – Alamo River Outlet. Meter-cart in background. Temperature and Grab Sample taken only.

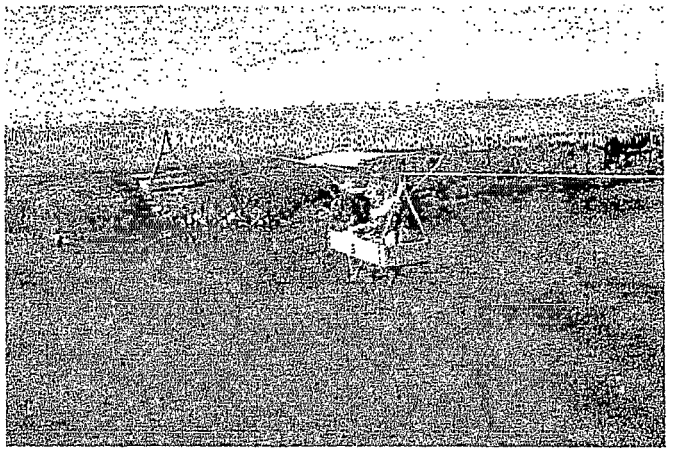
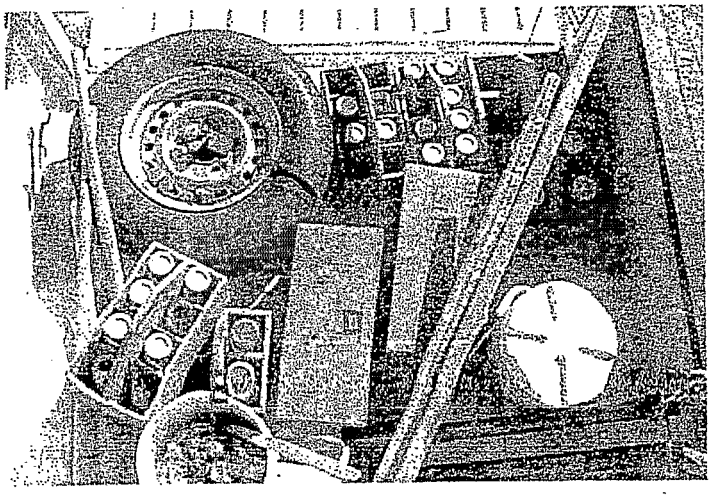


Fig. 8 – *New* River Outlet. Meter-cart centered in picture. Temperature and Grab Sample taken only.

Step 1 *Collect equipment*

Fig. 9 – Pictured are several pieces of equipment: (A) one-pint bottles, (B) ½ gallon jars, (C) DH59 Sampler, (D) reel and cable assembly, (E) one-quart jars



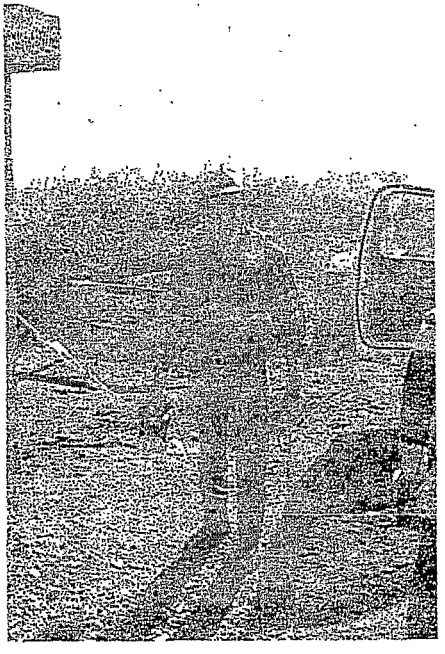


Fig.10 – Data Technician outfitted with proper gear; life jacket, rubber gloves, and work boots with non-slip soles.

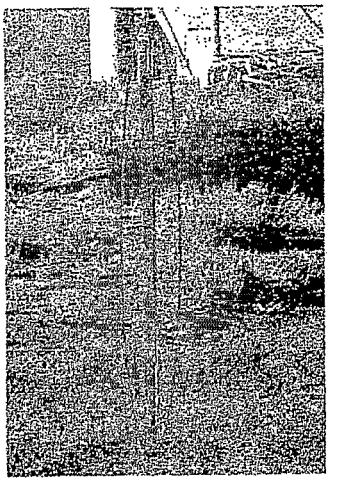


Fig.11 – Circled is a 0°F to 120°F thermometer enclosed in protective metal case.

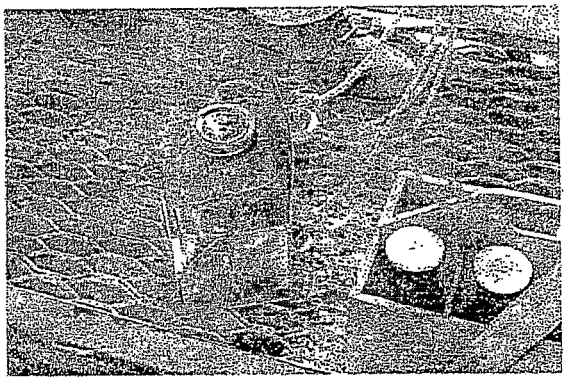


Fig.12 – Close-up view of Grab Sample ½ gallon jar in metal basket.

DATE SAMPLED _____ TIME _____ DATE TO LAB. _____

BOTTLE NO. _____ LAB. NO. _____

LOCATION OF SAMPLE _____

DISCHARGE _____ WATER TEMP. _____

METHOD OF SAMPLING _____

REMARKS _____

SAMPLED BY _____

DATE TESTED _____ TESTED BY _____

IID-430A (R3 12-70) • WATER AND SILT SAMPLES

Fig.13 – An example of IID-430A (R3 12-70) form.

Fig.14 – Close-up view of reel/cable assembly. Data Technician labeling paper caps for one-pint specimens.

Step 2 *Proper use of Safety Harness*

The Alamo River crossing at Boundary requires the use of a Safety Harness. The following is a breakdown of the proper use of safety harness. A safety harness should be used whenever there is a possibility of falling during sample retrieval.

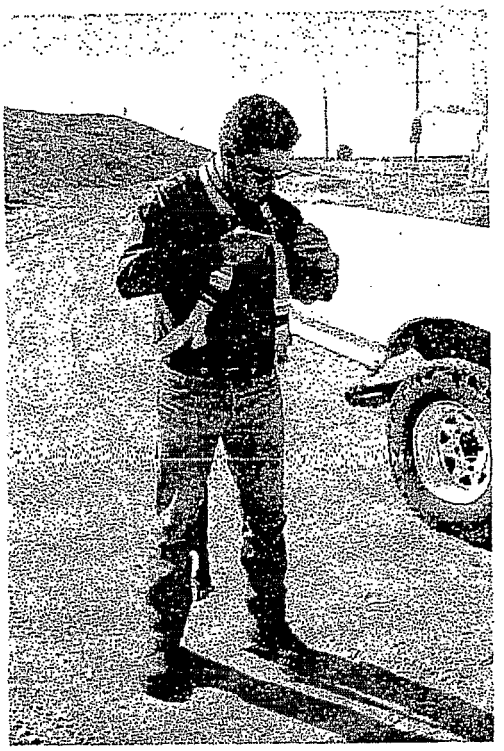


Figure 15 – Drape harness over shoulders. Make sure straps are not twisted or frayed. Buckle top front belt.

Figure 16 – Pull straps from underneath and buckle to bottom set of belts.

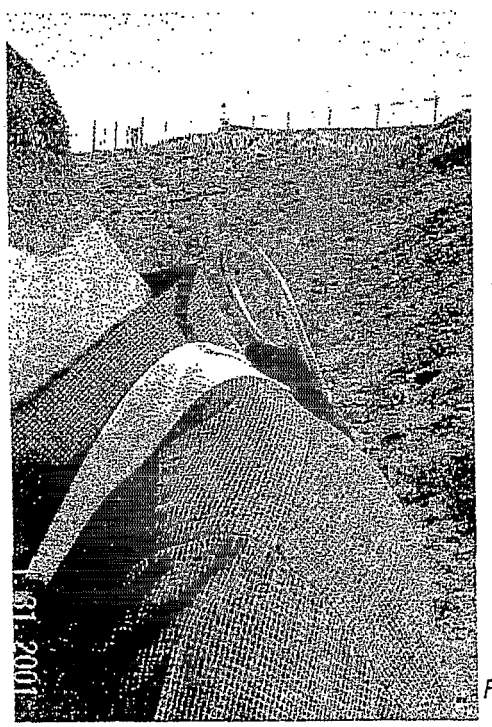


Figure 17 – Exposed is hoop for attaching lifeline.

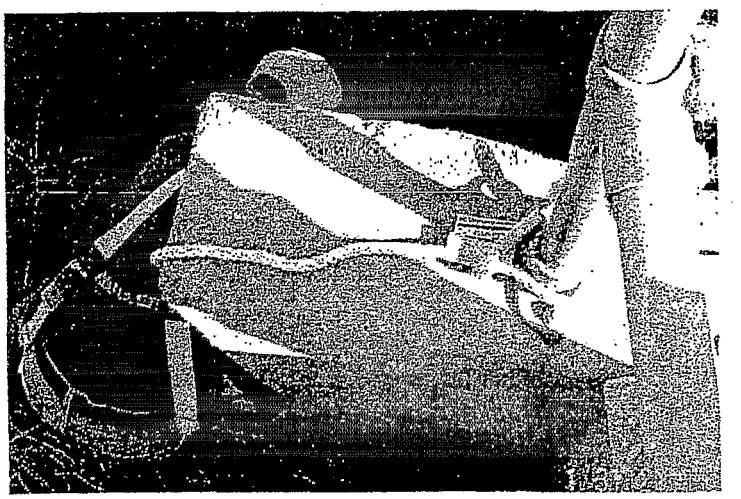


Figure 18 – Attach lifeline to a solid anchor point. In this case, the base of a railing is sufficient. See *Fig. 4*, page 2 for example of harness in action.

Sub-procedure on the use of the DH-59 Sampler and Temperature.

Note: This aspect applies to AAC Drop 1, EHL, CMC, and WSM sites

Step 1 *Assemble Sampler*

Insert one-pint bottle into cavity of DH-59 Sampler. Be sure that a gasket rests between bottle and backside of nozzle.

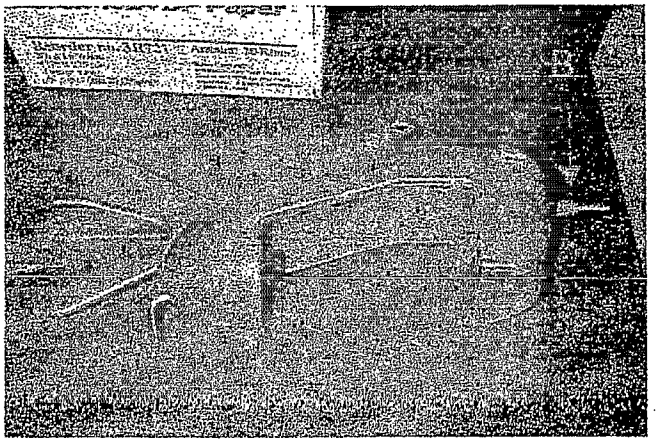


Figure 15 – Shown is the DH-59 Sampler. Arrow (A) points to spring-loaded pull, (B) points to nozzle tip.

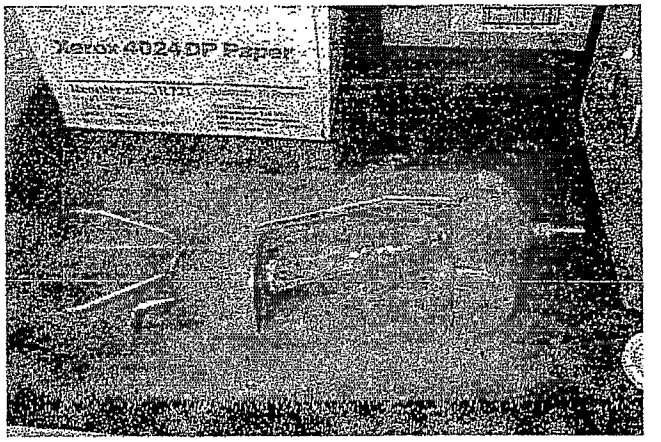


Figure 16 – DH-59 with bottle in place. Arrow (C) points to location of gasket.

Step 2 *Take temperature*

Place thermometer somewhere nearby the sampling in channel. Allow at least ten (10) minutes for temperature to stabilize. Once temperature has stabilized, log result on IID-430A (R3 12-70) Water and silt samples form and log book.

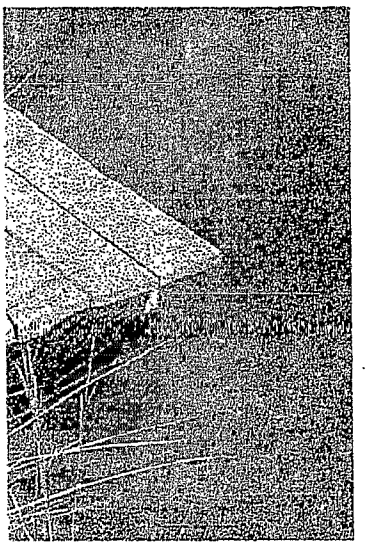


Fig.17 – Drape thermometer's cord over convenient platform and allow completely submerged for approximately ten (10) minutes to stabilize.

Step 3 *Lower DH-59 onto water surface*

After securing cable/reel assembly to observation platform, attach DH-59 to cable/reel assembly. Lower DH-59 to water surface. Set timepiece to zero; begin lowering DH-59 through stream flow. Once DH-59 touches bottom, retract at the same rate. It should take approximately 15 seconds to complete a sampling. An indication of a proper sample is a not quite full bottle. If bottle is completely full, sample is bad. Discard and redo. If acceptable, place a paper cap with site name to seal sample.
NOTE: Samples are to be retrieved in evenly spaced intervals; six (6) samples from AAC Drop 1, four (4) from remaining sites. Refer to Page 1 "Sites" for listing.

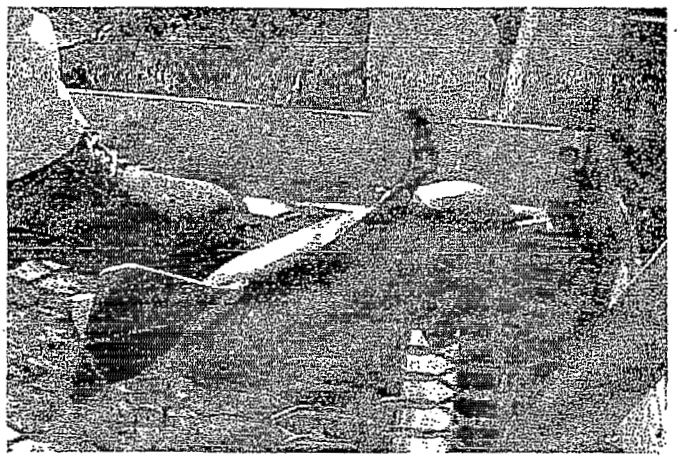


Figure 18 – Attach cable from reel to DH-59

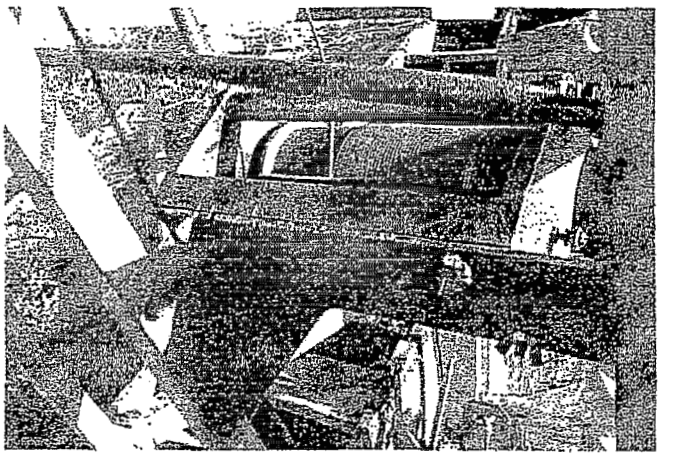


Figure 19 – Close up view of reel assembly.

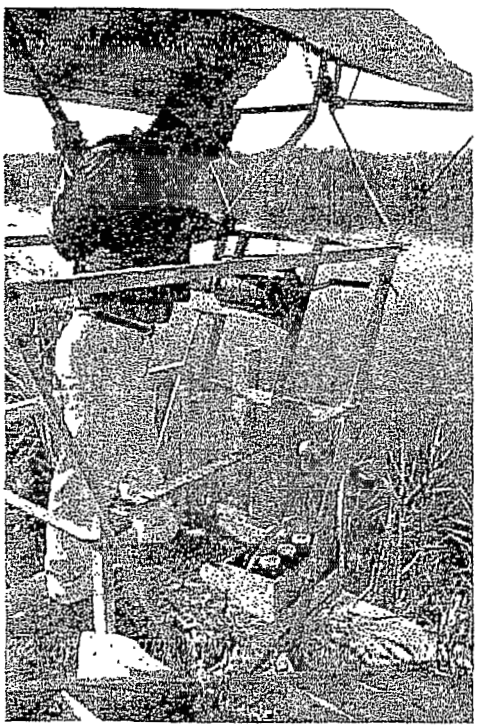


Figure 20 – Technician with all needed equipment in meter-cart@ AAC Drop 1.

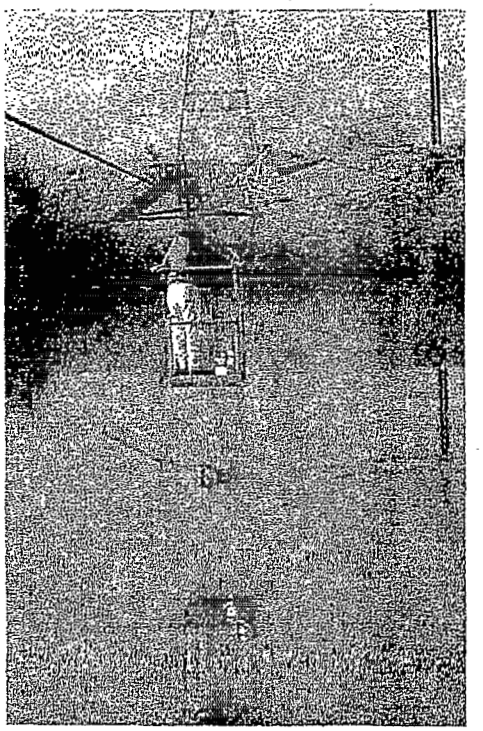


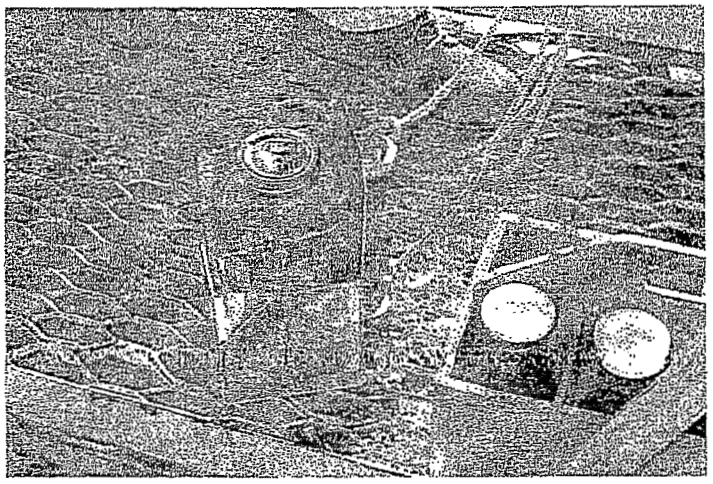
Figure 21 – DH-59 at water surface. Set timepiece to zero, time begins when nozzle (arrow) is submerged.

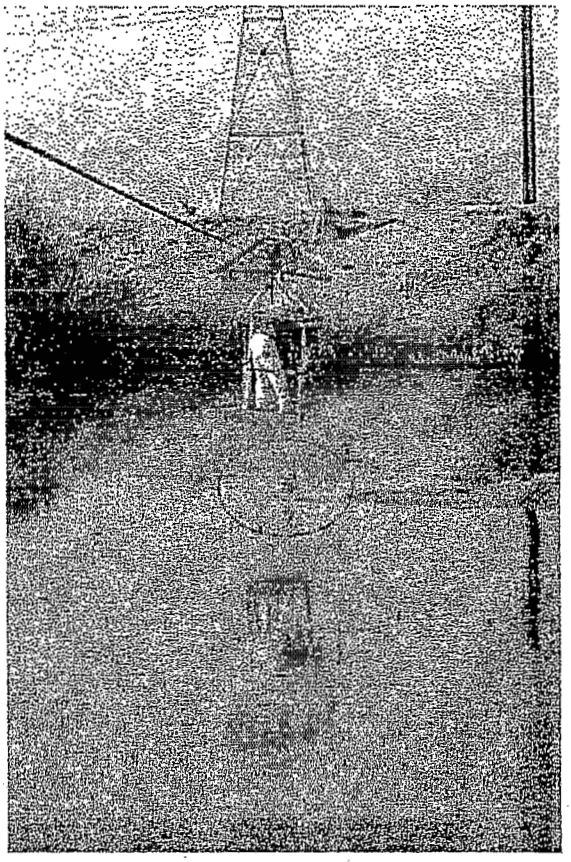
Sub-procedure on the use of the Grab Sampler and Temperature*

Note: This aspect applies to AAC Drop 1, EHL, Alamo River Inlet, CMC, WSM, New River Outlet, and Alamo River Outlet sites.
*Refer to **Sub-procedure on the use of the DH-59 Sampler and Temperature**, Step 2, page 5, for detail on temperature readings.

Step 1 *Collect Grab Sample*

When near center of stream, collect a sample of water in a glass jar. The technique used is akin to dunking. ½-gallon jar samples are taken at AAC Drop 1, New River, Alamo River Inlet and Outlet. These sites require the use of a basket and rope.
One-quart jar samples are taken at EHL, CMC, and WSM, refer to *Fig.24* for example.
Write resulting sample collection into logbook





Figures 22 & 23 – Above, close-up view of basket/rope device for retrieving Grab samples. To the right, dunking of ½-gallon jar with basket/rope device at AAC Drop 1 site.

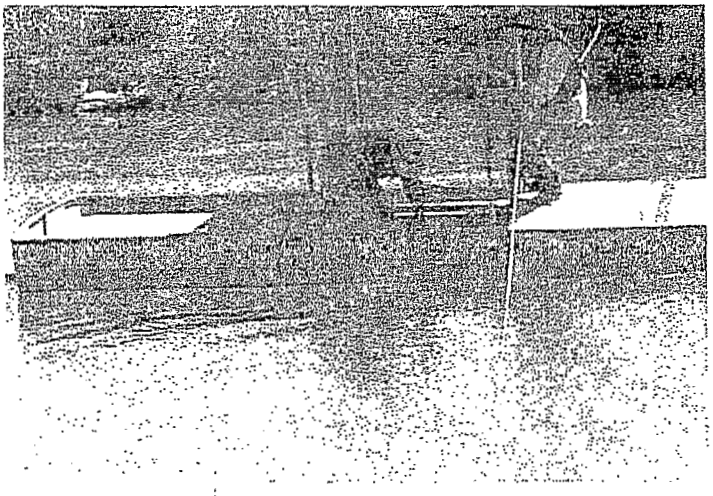


Fig.24 – East Highline site. An alternative method to retrieve a Grab Sample is by dunking manually several times in order to get a relative composite.

These final steps apply to both sub-procedures

Step 2 *Terminate Session*

Gather all equipment used for collecting samples. Place DH-59 in case, jars in box, one-pint bottles in carrying case. Make sure all samples are sealed and protected from spilling during transport.

Step 3 *Place samples in Laboratory*

Place all collected samples in laboratory, located on southeast corner of El Centro Steam Plant (Biological Control building). Include all completed IID 430A slips.

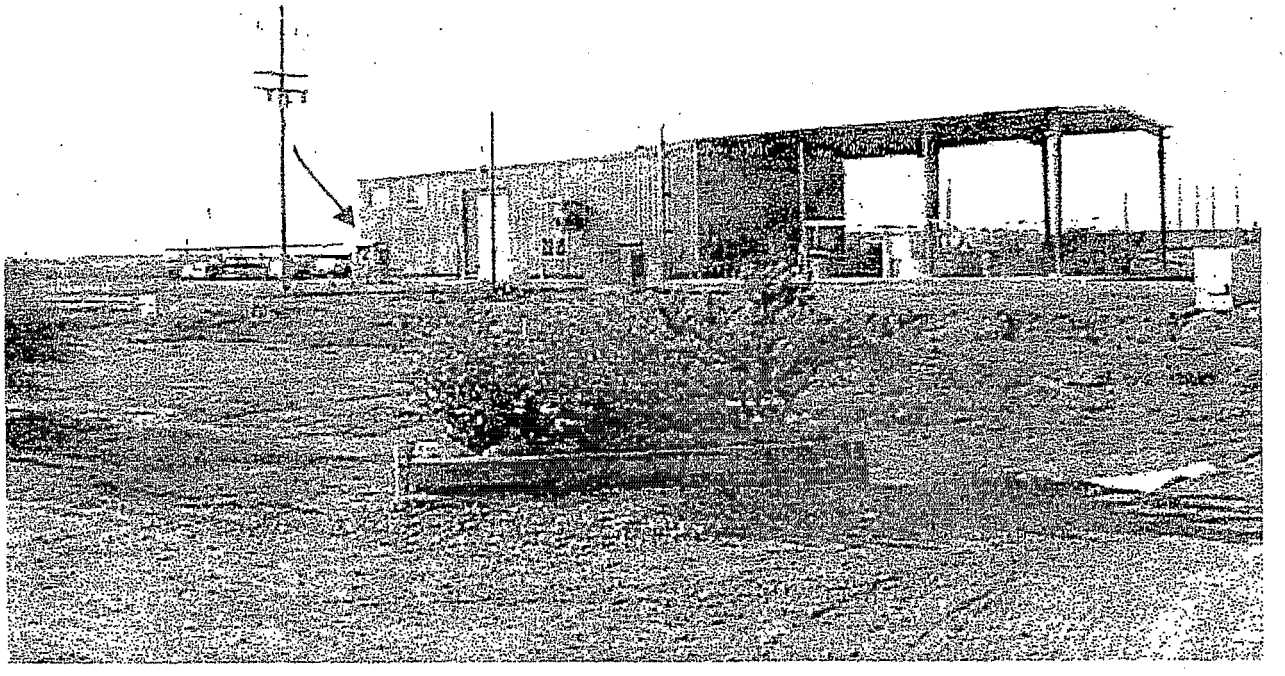


Fig.25 – Picture of Biological Center building, located on southeast corner of El Centro Steam Plant's yard. Red arrow points to entryway for laboratory.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

Procedure: DCO-SS-01

Date created/
Revision: 12/19/00, 3/26/01

Printed: 3/26/01 07:44

Associated
Procedures: DCO-SILTTDS-01

Object: To collect water samples around the Salton Sea using the Grab method.

Schedule: Bi-annually. Samples collected in the months of July and November.

Personnel: Two Data Technicians

Equipment: Labeled Sample containers (five-½ gallon jars) with lids
Basket with handle for Grab samples
Thermometer with 0°F to 120°F range
Pencil for noting observations
Timepiece
IID-430A (R3 12-70) Water and silt samples form (5 minimum)
Rubber waders
Rubber gloves

Sites:

Between Rivers (Coor. N33.13988° W115.66645°) – near sump pump S-307. Approximately 3000 ft. north along dike from the western end of Young Rd., near the center of Section 7, T.12 S., R.13 E.

Bertram Station (Coor. N33.35805° W115.76081°) – Walk approximately 1100 ft. southeasterly from a point on Hwy 111 which is 1.4 miles northwesterly of Bombay Beach and near MCI marker labeled *64840*, near the SW corner of the NW ¼ of Section 24, T.9 S., R.11 E.

Desert Beach (Coor. N33.51551° W115.93545°) – Enter the townsite of North Shore on Desert Beach Drive. Site is approximately 200 ft. southwesterly of southwest corner of North Shore Marina RV Park, near the center of east line of the NE ¼ Section 9, T.9 S., R.9 E.

Salton Sea Beach (Coor. N33.375090° W116.00645°) – approximately 1.1 miles east of Hwy 86 along Brawley Ave in the townsite of Salton Sea Beach, near the center of the SW ¼ of Section 23, T.9 S., R.9 E.

Sandy Beach (Coor. N33.17685° W115.83465°) – Turn-off 3.3 miles north of Three Flags Ranch turn-off. Proceed approximately 3.1 miles east, near the SE corner of the SE ¼ of Section 20, T.11 S., R.11 E.

Overall site view

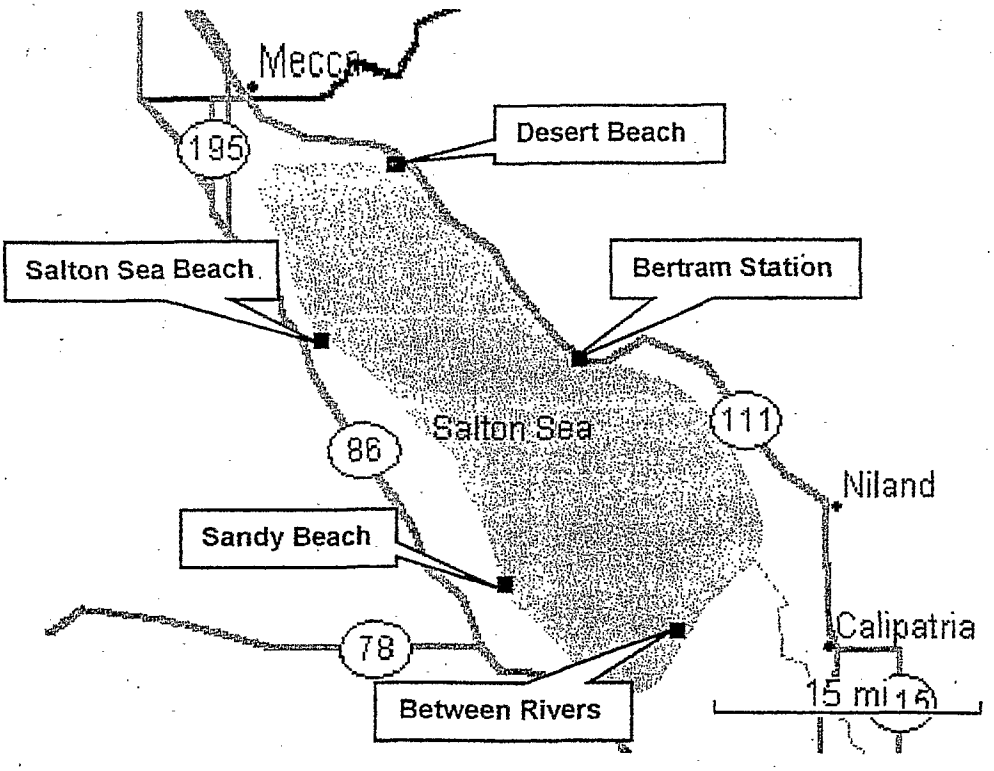


Figure 1 – Illustration of all sites around the Salton Sea.

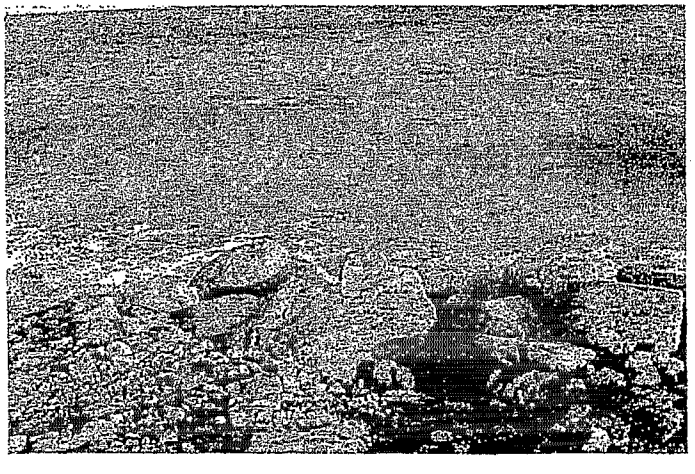


Figure 2 – Between Rivers site. Sample taken near discharge point of pumps S-307 and WP-9.

Figure 3 – Bertram Station entry site. Point is marked solely with a wooden lathe and a rubber glove. The adjacent area is protected by the State. Vehicular traffic is prohibited.

Figure 4 – Data Technician collecting sample using pole and basket at Desert Beach. Use rubber waders to access site.

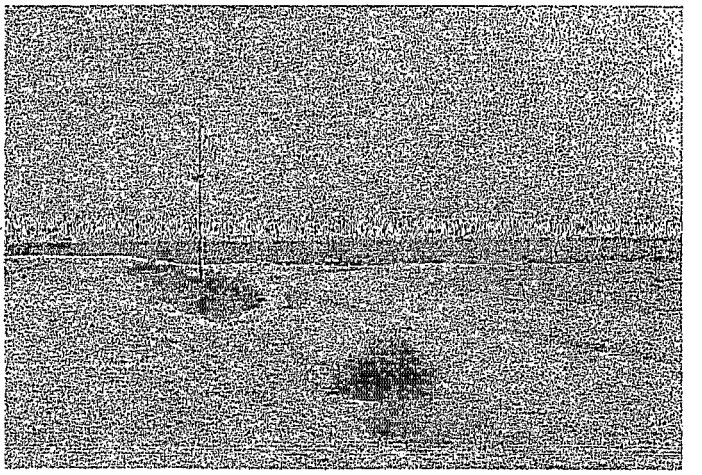


Figure 5 – Salton Sea Beach shoreline. Use rubber waders at this site also.

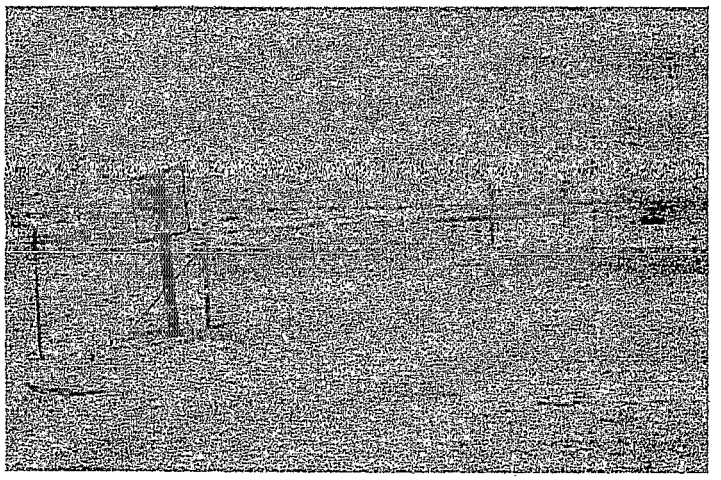


Figure 6 – Turn-off entrance from Highway 86 for Sandy Beach. Located approximately 3.3 miles northwest of Three Flags Ranch.

FIELD PROCEDURE

Step 1 *Assemble Sampling device*

After putting on rubber gloves and waders, place sample jar into basket/pole device. Secure with spring-loaded outer ring.

Figure 7 – Assembled sampling device.

Step 2 *Technique for collecting sample*

Carefully walk out into Sea. Make sure footing is secure with each step. Go out to a depth of approximately 3 feet. With a sweeping motion, dunk sample jar repeatedly until full. Typically 3 to 4 passes, refer to *Figure 4* for example.

Step 3 *Take temperature*

Immerse thermometer nearby on the shore and let stabilize for approximately 10 minutes.

Step 4 *Log results*

Fill out form IID-430A (R3 12-70) Water and Silt Samples, for each site. Fields of importance are the (1) DATE SAMPLED, (2) TIME, (3) LOCATION OF SAMPLE, (4) TEMPERATURE, (5) METHOD OF SAMPLING, & (6) SAMPLED BY. Refer to *Figure 8* for example of form.

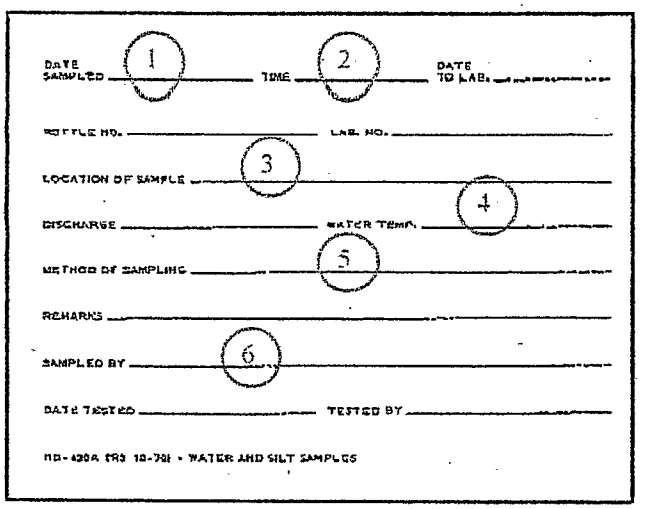


Figure 8 – An example of IID 430A form.

Step 5 *Deliver Samples*

After collecting samples from all sites, secure them so as not to spill during transport. Deliver samples directly to ATS Laboratory in Brawley, refer to *Fig. 9.*

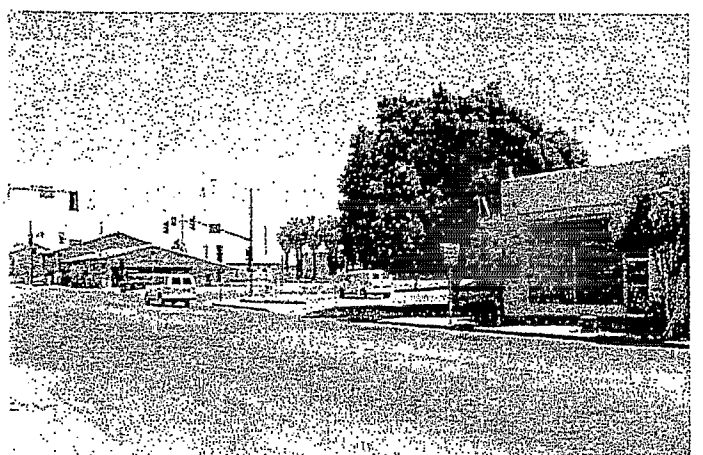


Figure 9 – ATS Laboratory in Brawley. Located near the southeast corner of 8th and Main Street.

OFFICE PROCEDURE

Step 6 *Assign Laboratory Numbers to Samples*

A three-ring binder, labeled **Silt/TDS Log Sheets,** with *Laboratory Numbers Index* (form IID-442C R2 8-65), located at WCC's Hydrography Unit, contains a sequential list of Lab numbers. Follow format of previous entries. Write corresponding numbers onto slips. Make copies of slips. Send originals to Water Department, Engineering Services, Technical Resources and Planning Unit.

Step 7 *File copies*

File copies of slips in file cabinet, located in Hydrography Unit's Data Technician office area. Place in folder labeled with corresponding numerical sequence. Once ATS Laboratory submits a report of the chemical breakdown, attach copy of relative slips to this report and file. Duty completed.

IID-421 (R16-87)

IMPERIAL IRRIGATION DISTRICT
P.O. Box 937 - 333 East Barioni Boulevard
Imperial, California 92251
(760)339-9220 General Information

LETTER OF TRANSMITTAL

Date: **July 7, 2004**	Job No.
Attn:	
RE: SOPs for IID water monitoring data	

TO **Melenee Emanuel**

State Water Resources Control Board
Division of Water Quality, Monitoring
1001 I Street
P.O. Box 944213
Sacramento, CA 95812

WE ARE SENDING YOU ☒ Attached ☐ Under separate cover via _____ the following items:

☐ Book ☐ CD Disk ☐ 3.5" Floppy Disk ☐ Samples ☐ Specifications
☐ Copy of letter ☐ Zip Disk ☐ Data Pages ☒ Reports ☐ Drawings

			DESCRIPTION
1	copy		Monthly anion/cation water monitoring Standard Operating Procedure (New River, Alamo River, All American Canal)
1	copy		Monthly anion/cation water monitoring Standard Operating Procedure (Salton Sea)

THESE ARE TRANSMITTED as checked below:

☐ For Approval ☐ Approved as Submitted ☐ Resubmit _____ Copies for approval
☐ For your use ☐ Approved as Noted ☐ Submit _____ Copies for distribution
☒ As requested ☐ Returned for corrections ☐ Return _____ Corrected prints
☐ For review and comment ☐ _____
☐ FOR BIDS DUE _____ 19 ____ ☐ PRINTS RETURNED AFTER LOAN TO US

REMARKS The attached SOPs are being submitted as supplementary information for water monitoring data submitted on 6/10/04. An SOP for IID's monthly coliform samples is not available.

COPY TO _____

SIGNED: Steve Charlton
Steve Charlton
Resources Planning & Management

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

Procedure: DCO-SILTTDS-01

Date Created/ Revision: 7/26/00, 3/6/01

Printed: 3/6/01 - 12:57

Associated Procedures: LAB-TST-01

Object: To collect water samples along the All American Canal using the DH-59 Sampler and the Grab method.

Schedule: Monthly

Personnel: Two (2) Data Technicians

Equipment:
DH-59 water sampler
Cable and reel
Life jackets
Work gloves
Sample containers (18 one-pint bottles; three one-quart and four-½ gallon jars)
Basket for Grab samples
Paper caps for pint bottles
Thermometer with 0°F to 120°F range
Pencil for labeling and noting observations
Heavy duty string or cord, 20-foot minimum length
Timepiece
IID keys
IID-430A (R3 12-70) Water and silt samples form (7 minimum)
IID Log book

Sites:
All American Canal (Coor. N32.70599 W114.96191) – approximately 6500 ft. downstream of Drop 1. Take Gordon's Well turn-off of I-8, 500' west along AAC bank, see *Fig.2.*
East Highline Canal (Coor. N32.70390 W115.28438) – approximately 2000 ft. downstream of Heading on west bank, see *Fig.3.*
Alamo River Inlet (Coor. N32.67455 W115.36996) – outlet headwall of river crossing at All American Canal, Grab Sample only, see *Fig.4.*
Central Main Canal (Coor. N32.69530 W115.46595) – approximately 4500 ft. downstream of Heading, near Acacia Heading off Bowker Road, see *Fig.5.*
Westside Main Canal (Coor. N32.67917 W115.67744) – approximately 500 ft. downstream of Hwy 98 crossing, see *Fig.6.*
Alamo River Outlet (Coor. N33.19865 W115.59621) – approximately 400 ft. upstream of intersection at Garst Rd. and Alamo River, Grab Sample only, see *Fig.7.*
New River Outlet (Coor. N33.10471 W115.66434) – approximately 4500 ft. west of the intersection at Lack Rd. and Vail Canal, Grab Sample only, see *Fig.8.*

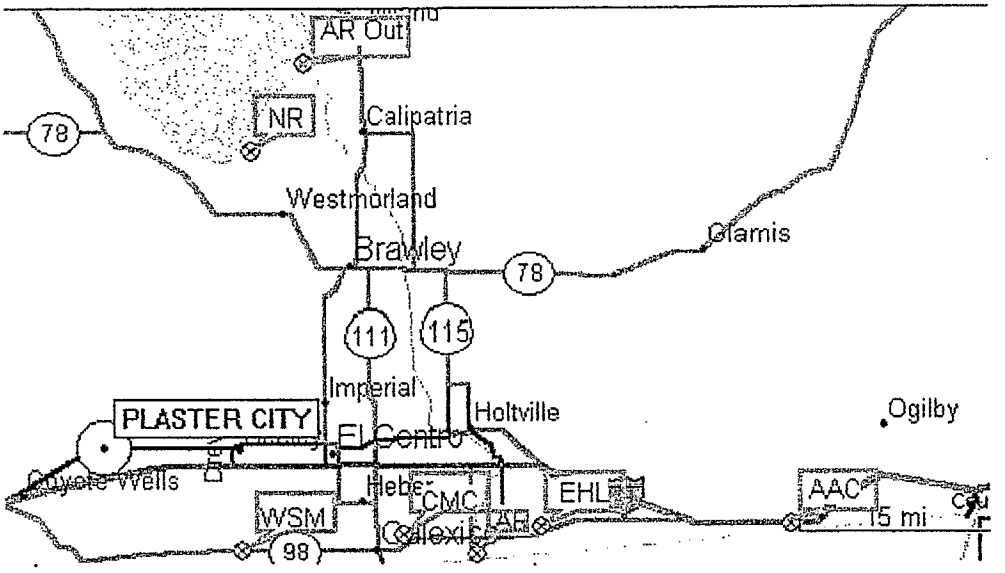


Fig. 1 – General location of water sampling sites: Alamo River Outlet (AR Out), New River Outlet (NR), Westside Main (WSM), Central Main (CMC), Alamo River Inlet (AR), East Highline (EHL), and All American Canal @ Drop 1 (AAC).

Following is a pictorial of all sites, refer to *Sites* on page 1.

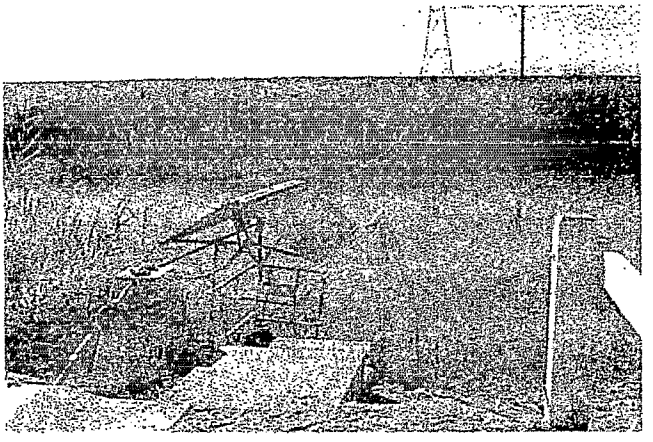


Fig. 2 – All American Canal at Drop 1, meter-cart in foreground

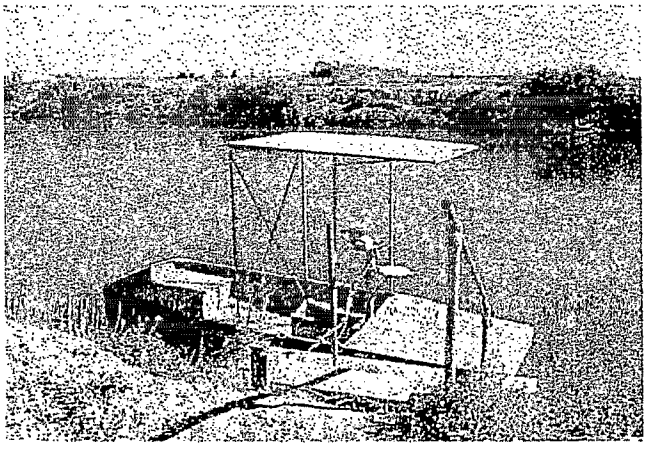


Fig. 3 – East Highline, meter-boat in foreground.

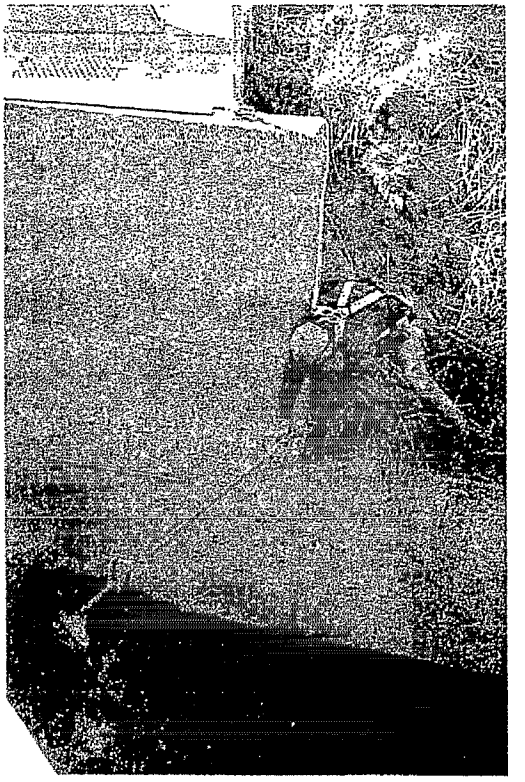


Fig. 4 – Alamo River @ Inlet, temperature and Grab Sample taken only.

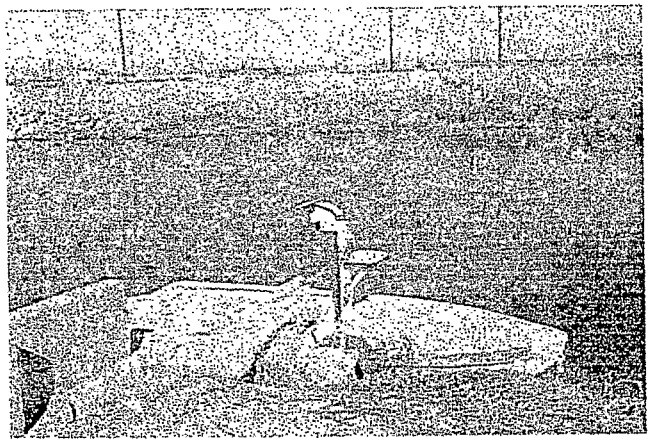


Fig. 5 – Central Main Canal. Meter-boat in foreground.

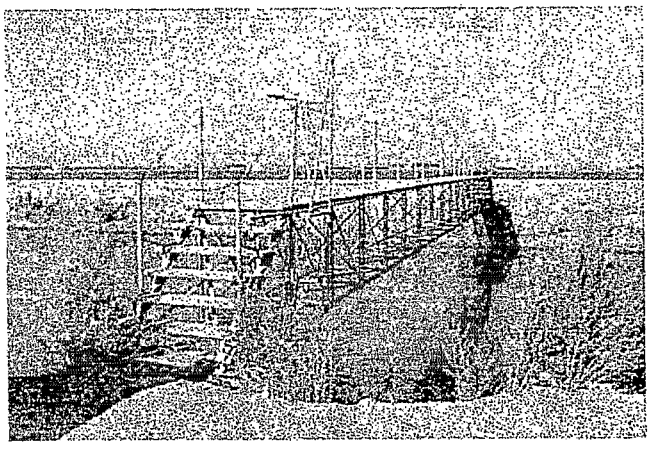


Fig. 6 – Westside Main Canal. Pictured is the meter-bridge that spans the channel.

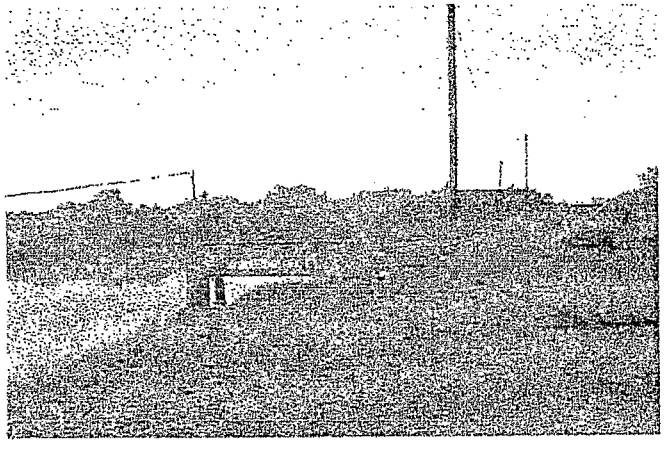


Fig. 7 – Alamo River Outlet. Meter-cart in background. Temperature and Grab Sample taken only.

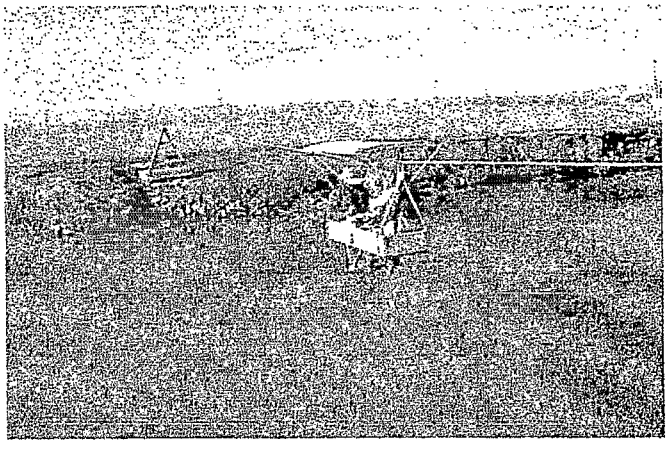


Fig. 8 – *New* River Outlet. Meter-cart centered in picture. Temperature and Grab Sample taken only.

Step 1 *Collect equipment*

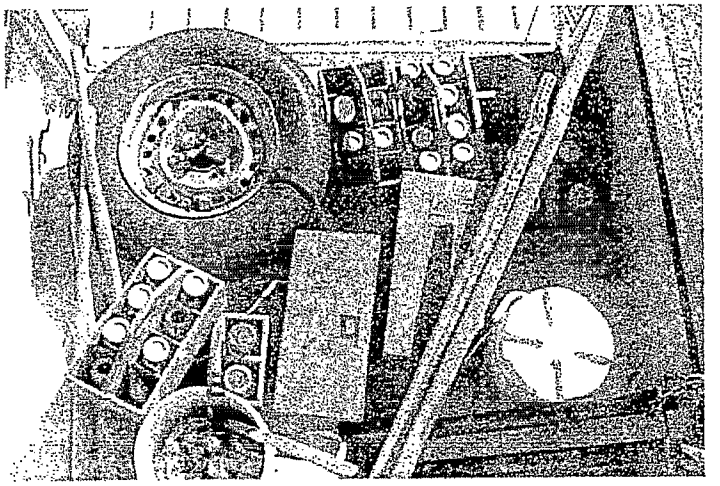


Fig. 9 – Pictured are several pieces of equipment: (A) one-pint bottles, (B) ½ gallon jars, (C) DH59 Sampler, (D) reel and cable assembly, (E) one-quart jars

Fig.10 – Data Technician outfitted with proper gear; life jacket, rubber gloves, and work boots with non-slip soles.

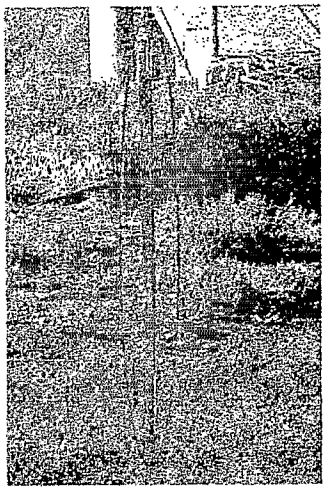


Fig.11 – Circled is a 0°F to 120°F thermometer enclosed in protective metal case.

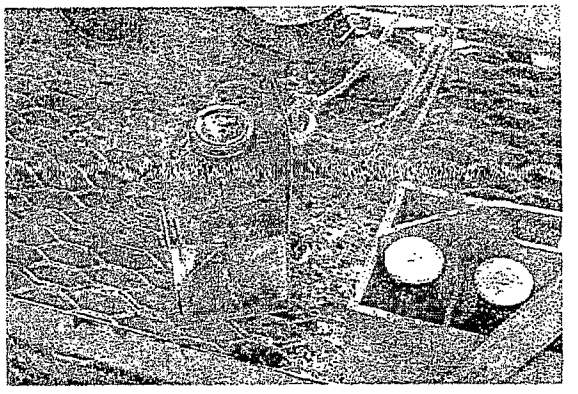


Fig.12 – Close-up view of Grab Sample ½ gallon jar in metal basket.

DATE SAMPLED _____ TIME _____ DATE TO LAB. _____

BOTTLE NO. _____ LAB. NO. _____

LOCATION OF SAMPLE _____

DISCHARGE _____ WATER TEMP. _____

METHOD OF SAMPLING _____

REMARKS _____

SAMPLED BY _____

DATE TESTED _____ TESTED BY _____

IID-430A (R3 12-70) • WATER AND SILT SAMPLES

Fig.13 – An example of IID-430A (R3 12-70) form.

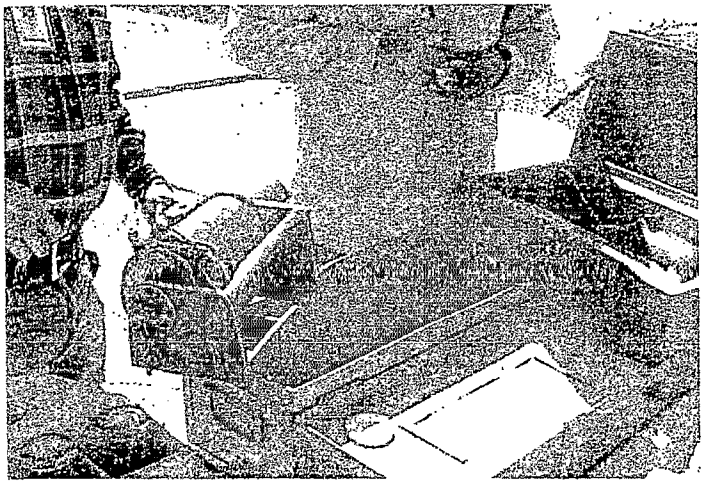


Fig.14 – Close-up view of reel/cable assembly. Data Technician labeling paper caps for one-pint specimens.

Step 2 *Proper use of Safety Harness*

The Alamo River crossing at Boundary requires the use of a Safety Harness. The following is a breakdown of the proper use of safety harness. A safety harness should be used whenever there is a possibility of falling during sample retrieval.

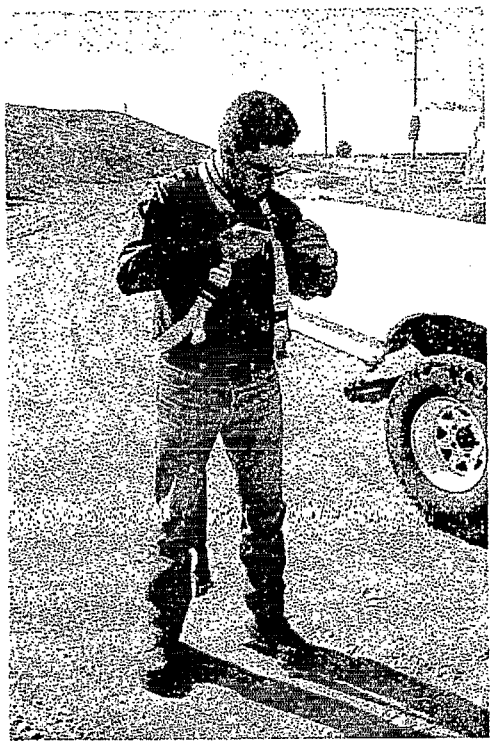


Figure 15 – Drape harness over shoulders. Make sure straps are not twisted or frayed. Buckle top front belt.

Figure 16 – Pull straps from underneath and buckle to bottom set of belts.

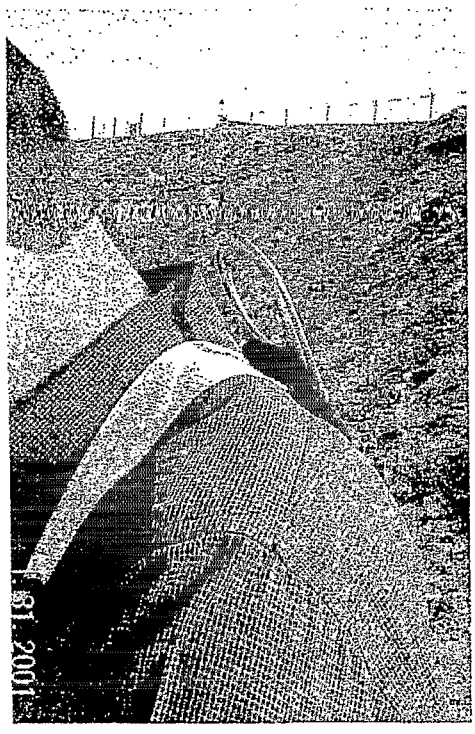


Figure 17 – Exposed is hoop for attaching lifeline.

Figure 18 – Attach lifeline to a solid anchor point. In this case, the base of a railing is sufficient. See *Fig. 4*, page 2 for example of harness in action.

Sub-procedure on the use of the DH-59 Sampler and Temperature.

Note: This aspect applies to AAC Drop 1, EHL, CMC, and WSM sites

Step 1 *Assemble Sampler*

Insert one-pint bottle into cavity of DH-59 Sampler. Be sure that a gasket rests between bottle and backside of nozzle.

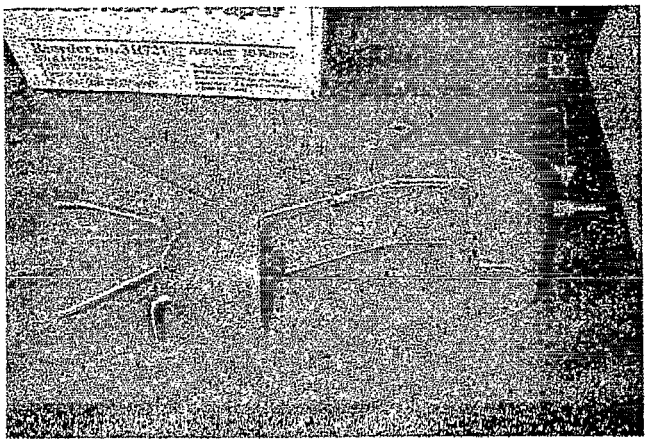


Figure 15 – Shown is the DH-59 Sampler. Arrow (A) points to spring-loaded pull, (B) points to nozzle tip.

Figure 16 – DH-59 with bottle in place. Arrow (C) points to location of gasket.

Step 2 *Take temperature*

Place thermometer somewhere nearby the sampling in channel. Allow at least ten (10) minutes for temperature to stabilize. Once temperature has stabilized, log result on IID-430A (R3 12-70) Water and silt samples form and log book.

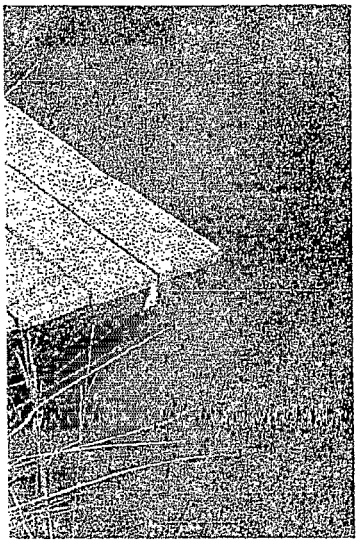


Fig.17 – Drape thermometer's cord over convenient platform and allow completely submerged for approximately ten (10) minutes to stabilize.

Step 3 *Lower DH-59 onto water surface*

After securing cable/reel assembly to observation platform, attach DH-59 to cable/reel assembly. Lower DH-59 to water surface. Set timepiece to zero; begin lowering DH-59 through stream flow. Once DH-59 touches bottom, retract at the same rate. It should take approximately 15 seconds to complete a sampling. An indication of a proper sample is a not quite full bottle. If bottle is completely full, sample is bad. Discard and redo. If acceptable, place a paper cap with site name to seal sample.
NOTE: Samples are to be retrieved in evenly spaced intervals; six (6) samples from AAC Drop 1, four (4) from remaining sites. Refer to Page 1 "Sites" for listing.

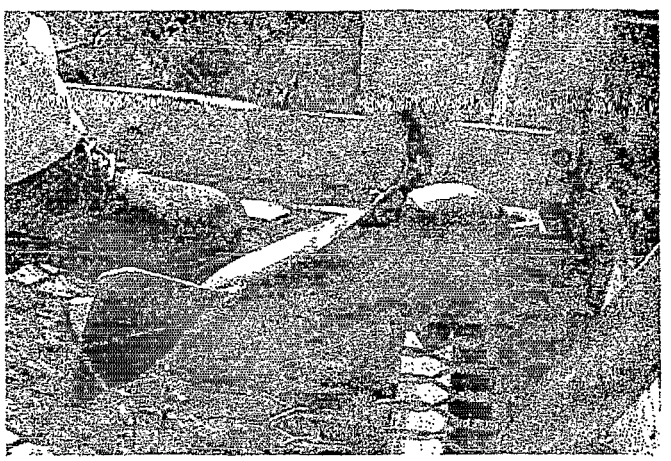


Figure 18 – Attach cable from reel to DH-59

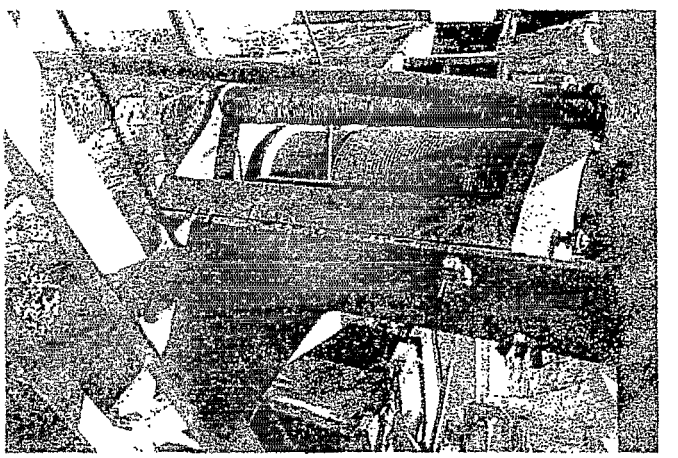


Figure 19 – Close up view of reel assembly.

Figure 20 – Technician with all needed equipment in meter-cart@ AAC Drop 1.

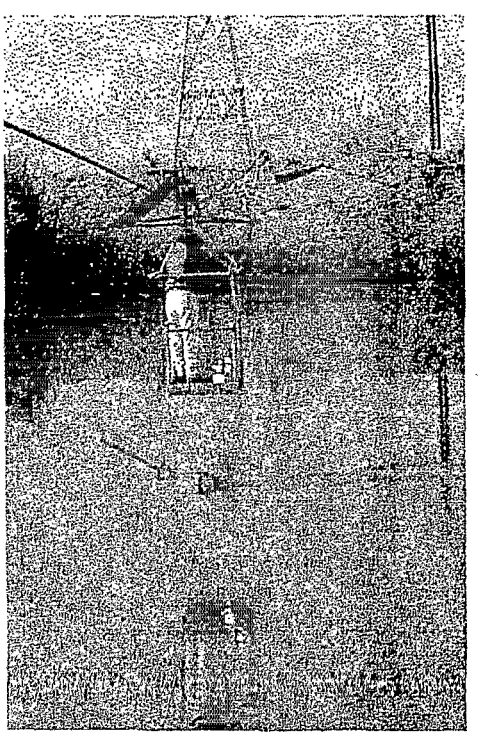


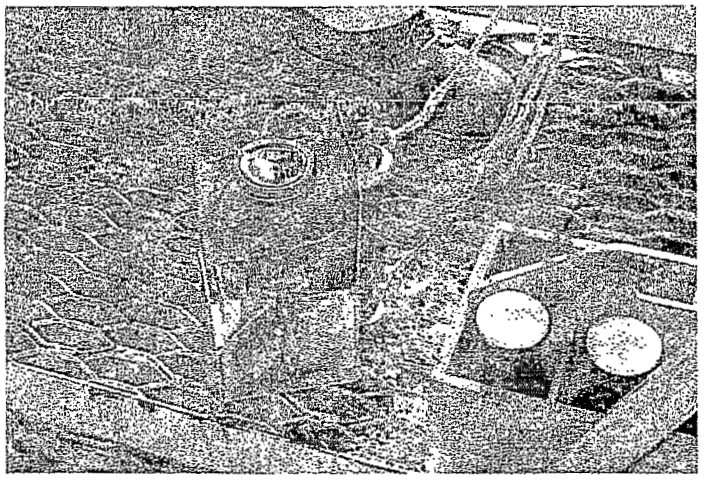
Figure 21 – DH-59 at water surface. Set timepiece to zero, time begins when nozzle (arrow) is submerged.

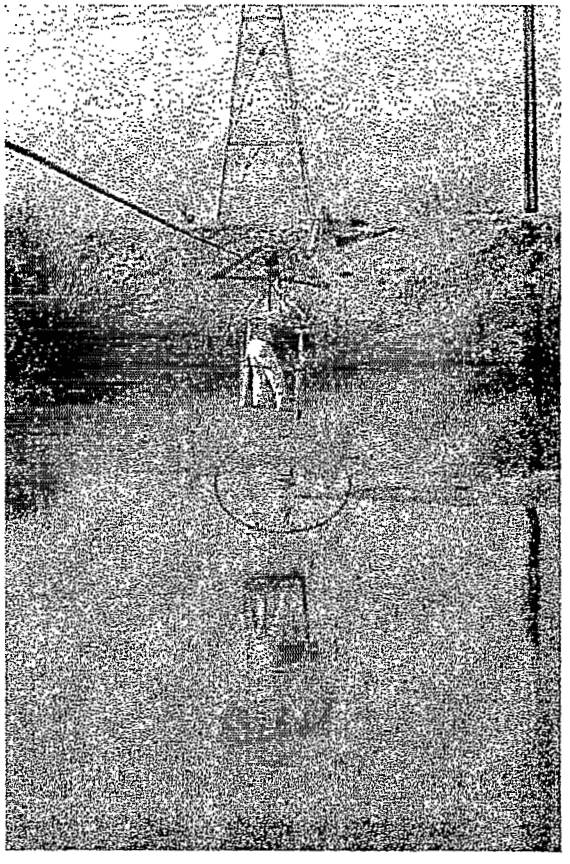
Sub-procedure on the use of the Grab Sampler and Temperature*

Note: This aspect applies to AAC Drop 1, EHL, Alamo River Inlet, CMC, WSM, New River Outlet, and Alamo River Outlet sites.
*Refer to **Sub-procedure on the use of the DH-59 Sampler and Temperature**, Step 2, page 5, for detail on temperature readings.

Step 1 *Collect Grab Sample*

When near center of stream, collect a sample of water in a glass jar. The technique used is akin to dunking. ½-gallon jar samples are taken at AAC Drop 1, New River, Alamo River Inlet and Outlet. These sites require the use of a basket and rope.
One-quart jar samples are taken at EHL, CMC, and WSM, refer to *Fig.24* for example.
Write resulting sample collection into logbook





Figures 22 & 23 – Above, close-up view of basket/rope device for retrieving Grab samples. To the right, dunking of ½-gallon jar with basket/rope device at AAC Drop 1 site.

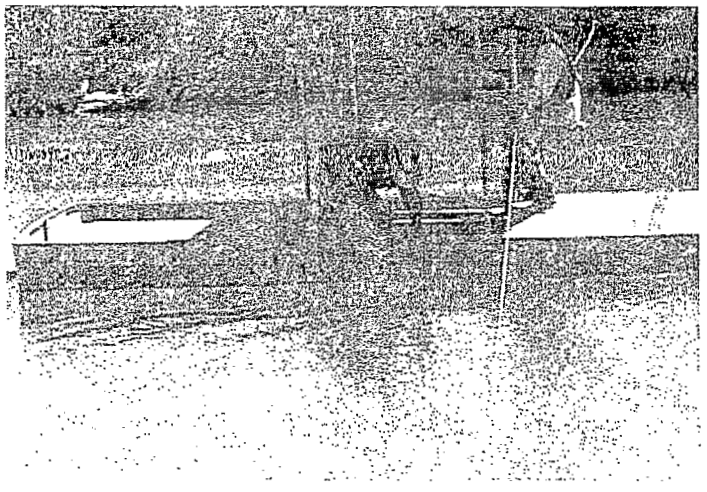


Fig.24 – East Highline site. An alternative method to retrieve a Grab Sample is by dunking manually several times in order to get a relative composite.

These final steps apply to both sub-procedures

Step 2 *Terminate Session*

Gather all equipment used for collecting samples. Place DH-59 in case, jars in box, one-pint bottles in carrying case. Make sure all samples are sealed and protected from spilling during transport.

Step 3 *Place samples in Laboratory*

Place all collected samples in laboratory, located on southeast corner of El Centro Steam Plant (Biological Control building). Include all completed IID 430A slips.

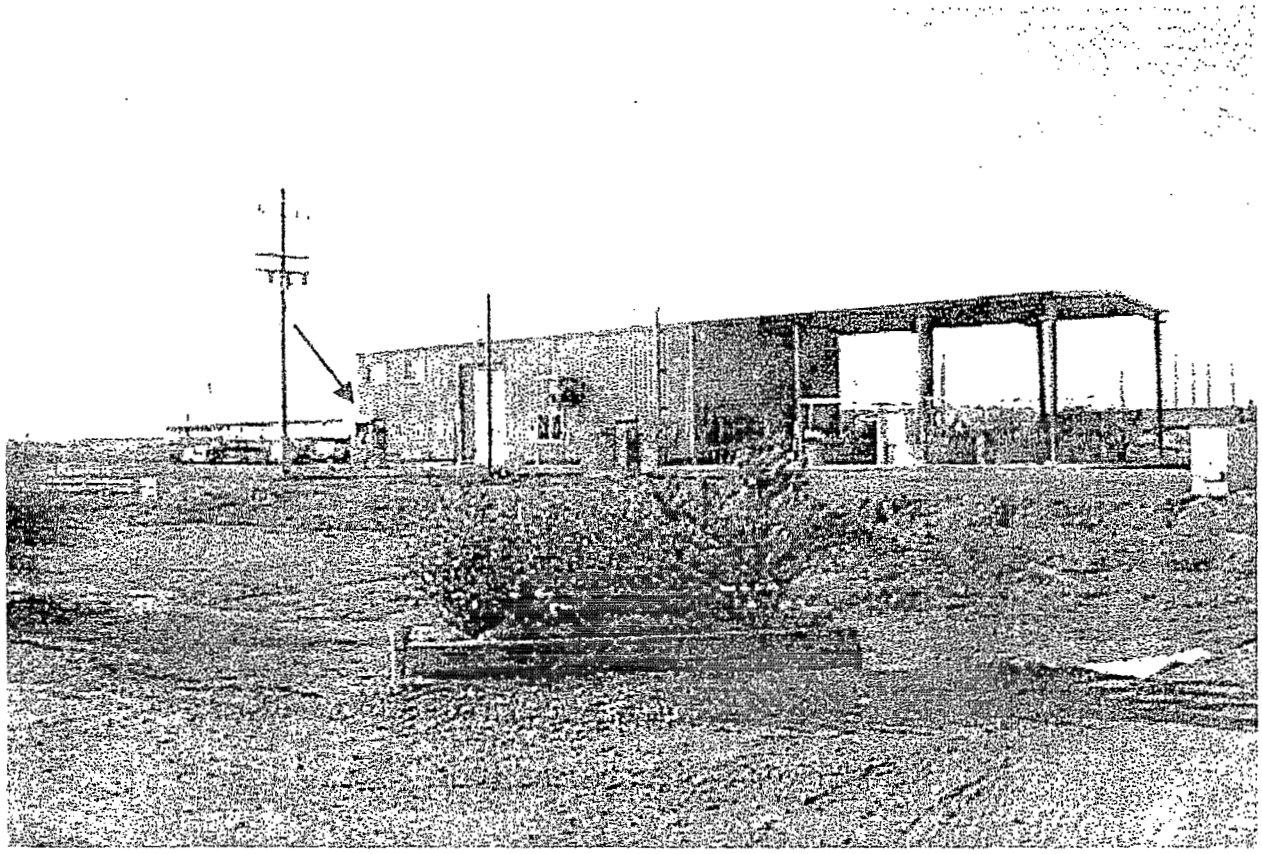


Fig.25 – Picture of Biological Center building, located on southeast corner of El Centro Steam Plant's yard. Red arrow points to entryway for laboratory.

IMPERIAL IRRIGATION DISTRICT
Hydrography Unit

Procedure: DCO-SS-01

Date created/
Revision: 12/19/00, 3/26/01

Printed: 3/26/01 07:44

Associated
Procedures: DCO-SILTTDS-01

Object: To collect water samples around the Salton Sea using the Grab method.

Schedule: Bi-annually. Samples collected in the months of July and November.

Personnel: Two Data Technicians

Equipment: Labeled Sample containers (five-½ gallon jars) with lids
Basket with handle for Grab samples
Thermometer with 0°F to 120°F range
Pencil for noting observations
Timepiece
IID-430A (R3 12-70) Water and silt samples form (5 minimum)
Rubber waders
Rubber gloves

Sites:
Between Rivers (Coor. N33.13988° W115.66645°) – near sump pump S-307. Approximately 3000 ft. north along dike from the western end of Young Rd., near the center of Section 7, T.12 S., R.13 E.

Bertram Station (Coor. N33.35805° W115.76081°) – Walk approximately 1100 ft. southeasterly from a point on Hwy 111 which is 1.4 miles northwesterly of Bombay Beach and near MCI marker labeled *64840*, near the SW corner of the NW ¼ of Section 24, T.9 S., R.11 E.

Desert Beach (Coor. N33.51551° W115.93545°) – Enter the townsite of North Shore on Desert Beach Drive. Site is approximately 200 ft. southwesterly of southwest corner of North Shore Marina RV Park, near the center of east line of the NE ¼ Section 9, T.9 S., R.9 E.

Salton Sea Beach (Coor. N33.375090° W116.00645°) – approximately 1.1 miles east of Hwy 86 along Brawley Ave in the townsite of Salton Sea Beach, near the center of the SW ¼ of Section 23, T.9 S., R.9 E.

Sandy Beach (Coor. N33.17685° W115.83465°) – Turn-off 3.3 miles north of Three Flags Ranch turn-off. Proceed approximately 3.1 miles east, near the SE corner of the SE ¼ of Section 20, T.11 S., R.11 E.

Overall site view

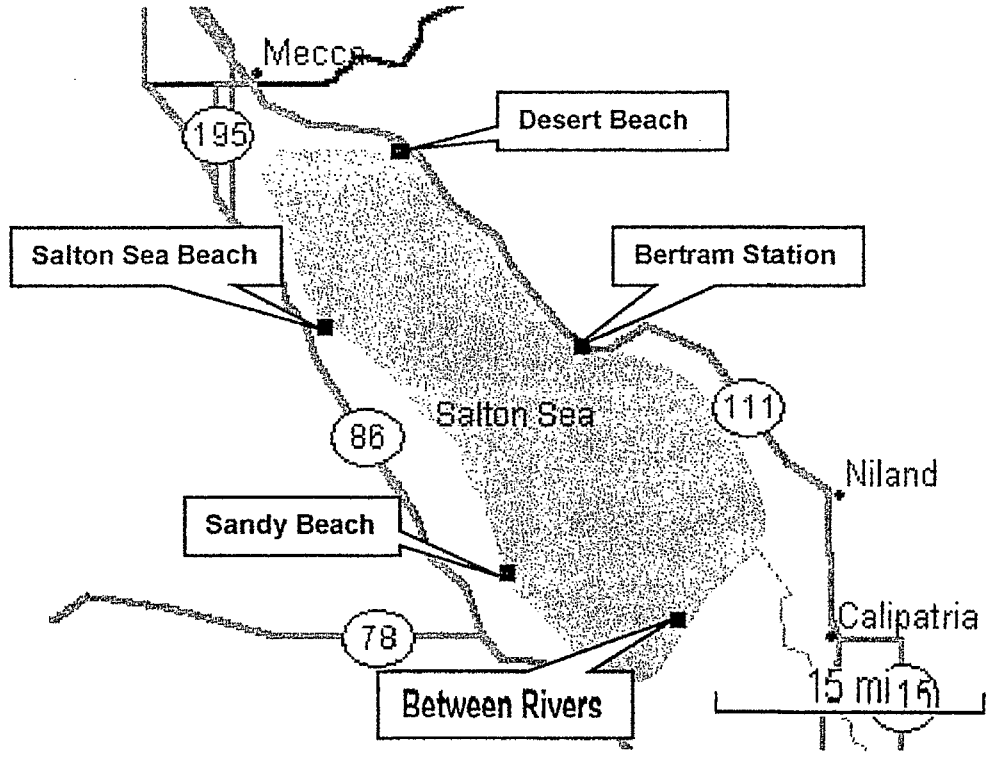


Figure 1 – Illustration of all sites around the Salton Sea.

Figure 2 – Between Rivers site. Sample taken near discharge point of pumps S-307 and WP-9.

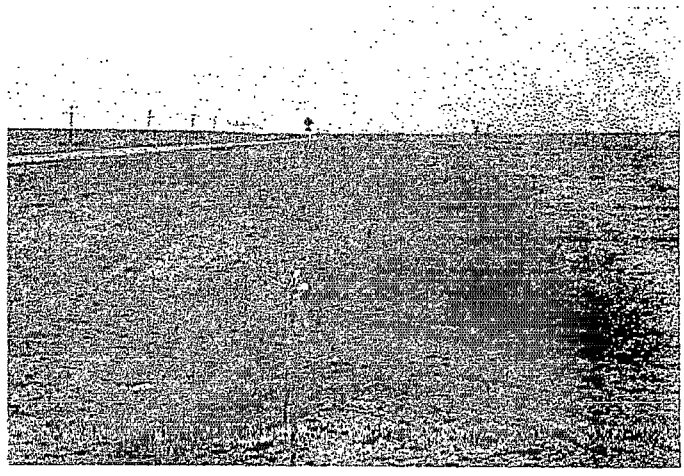


Figure 3 – Bertram Station entry site. Point is marked solely with a wooden lathe and a rubber glove. The adjacent area is protected by the State. Vehicular traffic is prohibited.

Figure 4 – Data Technician collecting sample using pole and basket at Desert Beach. Use rubber waders to access site.

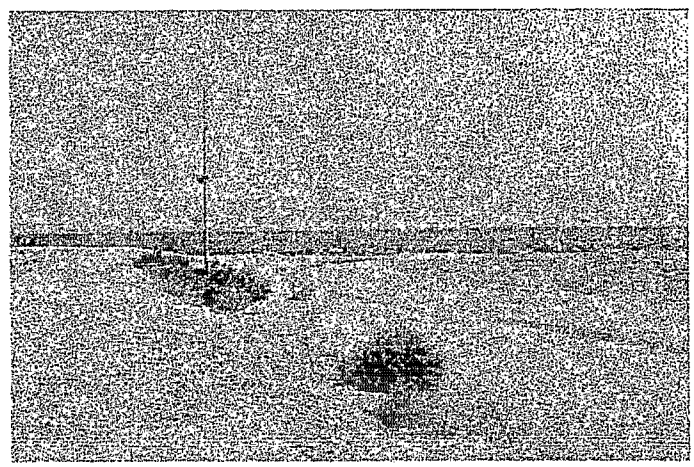


Figure 5 – Salton Sea Beach shoreline. Use rubber waders at this site also.

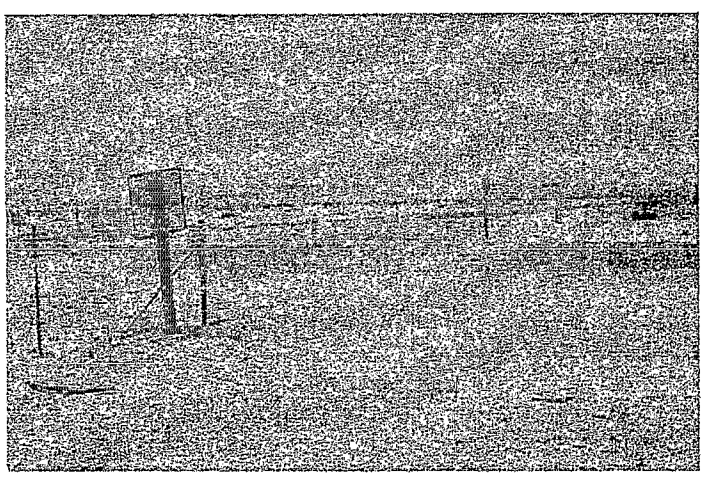


Figure 6 – Turn-off entrance from Highway 86 for Sandy Beach. Located approximately 3.3 miles northwest of Three Flags Ranch.

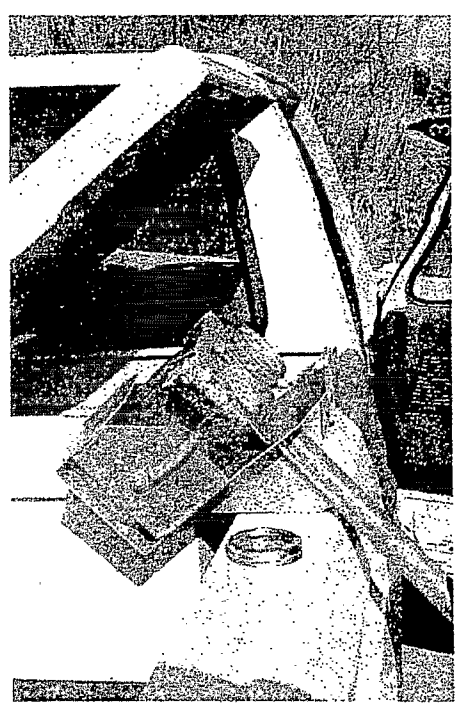


Figure 7 – Assembled sampling device.

FIELD PROCEDURE

Step 1 *Assemble Sampling device*

After putting on rubber gloves and waders, place sample jar into basket/pole device. Secure with spring-loaded outer ring.

Step 2 *Technique for collecting sample*

Carefully walk out into Sea. Make sure footing is secure with each step. Go out to a depth of approximately 3 feet. With a sweeping motion, dunk sample jar repeatedly until full. Typically 3 to 4 passes, refer to *Figure 4* for example.

Step 3 *Take temperature*

Immerse thermometer nearby on the shore and let stabilize for approximately 10 minutes.

Step 4 *Log results*

Fill out form IID-430A (R3 12-70) Water and Silt Samples, for each site. Fields of importance are the (1) DATE SAMPLED, (2) TIME, (3) LOCATION OF SAMPLE, (4) TEMPERATURE, (5) METHOD OF SAMPLING, & (6) SAMPLED BY. Refer to *Figure 8* for example of form.

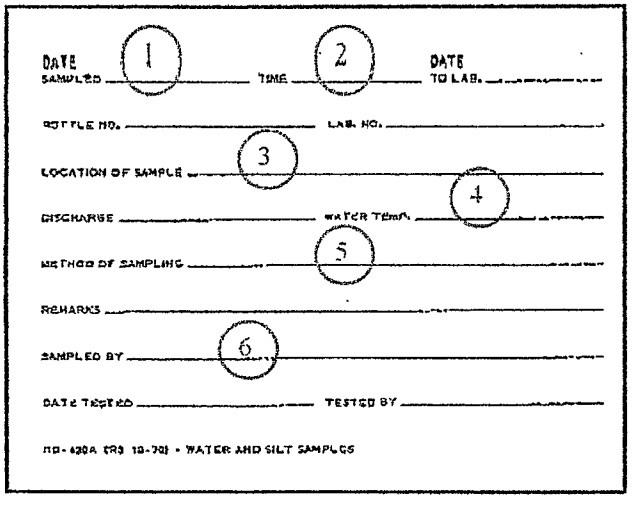


Figure 8 – An example of IID 430A form.

Step 5 *Deliver Samples*

After collecting samples from all sites, secure them so as not to spill during transport. Deliver samples directly to ATS Laboratory in Brawley, refer to *Fig. 9.*

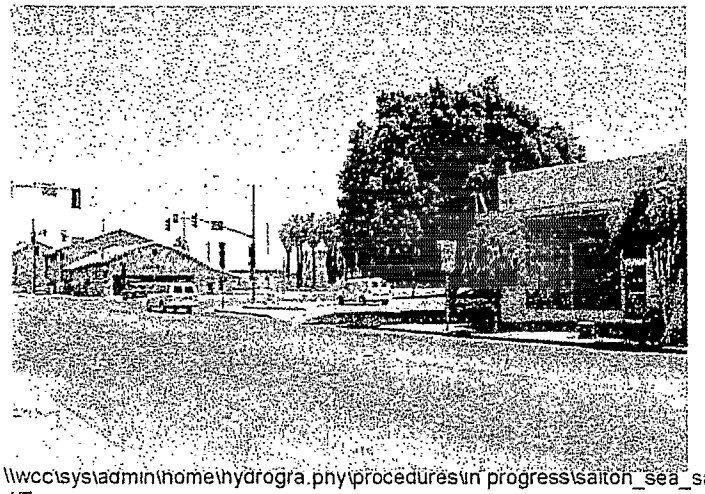


Figure 9 – ATS Laboratory in Brawley. Located near the southeast corner of 8th and Main Street.

OFFICE PROCEDURE

Step 6 *Assign Laboratory Numbers to Samples*

A three-ring binder, labeled **Silt/TDS Log Sheets,** with *Laboratory Numbers Index* (form IID-442C R2 8-65), located at WCC's Hydrography Unit, contains a sequential list of Lab numbers. Follow format of previous entries. Write corresponding numbers onto slips. Make copies of slips. Send originals to Water Department, Engineering Services, Technical Resources and Planning Unit.

Step 7 *File copies*

File copies of slips in file cabinet, located in Hydrography Unit's Data Technician office area. Place in folder labeled with corresponding numerical sequence. Once ATS Laboratory submits a report of the chemical breakdown, attach copy of relative slips to this report and file. Duty completed.