QUALITY ASSURANCE MANUAL

Prepared for

The Analysts, Supervisors, and Managers
of the Environmental Monitoring Division

ENVIRONMENTAL MONITORING DIVISION

BUREAU OF SANITATION

DEPARTMENT OF PUBLIC WORKS

CITY OF LOS ANGELES

12000 Vista del Mar
Playa del Rey, California 90293

December 2008

APPROVALS

The Quality Assurance Manual, Revision No. 11, documents the QA Program at EMD. Revisions, prepared by the Quality Assurance Unit, reflect changes to the December 2007 edition of the QA Manual.

The signatures of all laboratory managers below indicate that the manual is being accepted individually and collectively, and that the contents shall be implemented in the division laboratories' daily activities.

_____	_____
Lee L. Huang	Farhana Mohamed
Laboratory Manager I	Laboratory Manager I
_____	_____
Gerald McGowen	Jeffrey D. Beller
Laboratory Manager I	Laboratory Manager II

_____
Masahiro Dojiri
Laboratory Manager III

December 2008

TABLE OF CONTENTS

I. INTRODUCTION

A. VISION AND MISSION OF EMD

Vision

The vision of Environmental Monitoring Division (EMD) is to set the standard for excellence in environmental monitoring and assessment with a total commitment to continuous improvement.

Mission and Objectives

EMD is a division in the Bureau of Sanitation, Department of Public Works, City of Los Angeles. It provides technical support to the operations of the Bureau. These operations include the collection, treatment, and disposal of over 400 million gallons per day of wastewater, and the disposal and treatment of solid waste. These operations are essential to the well-being of the citizens of the City of Los Angeles.

The mission of EMD is to provide quality and cost-effective environmental data, research, and assessment in support of the Bureau's operation, compliance, and source control activities to protect public health and the environment. The following are EMD objectives in providing services to support Bureau operations:

1. Assess the impact of the Bureau of Sanitation operations on the environment to minimize or avoid adverse environmental effects. The quality of influent and effluent of the wastewater treatment plants is monitored for conventional, non-conventional, and priority pollutants. The treatment plants serviced by EMD are the Hyperion Wastewater Treatment Plant, the Donald C. Tillman Water Reclamation Plant, the Terminal Island Wastewater Treatment Plant, and the Los Angeles-Glendale Water Reclamation Plant. In addition, toxicity tests are performed on plant effluents and receiving waters. The receiving waters of Santa Monica Bay, Los Angeles Harbor, Los Angeles River, Balboa Lake, and Wildlife Lake are monitored for chemical, physical, and biological parameters to assess changes in the receiving environment that may be attributable to the discharges of the treatment plants. Non-point discharge sources, such as storm drains, are monitored microbiologically.

2. Perform legally required testing and reporting in a timely and efficient manner. The legal mandates include: the National Pollutant Discharge Elimination System (NPDES) permits issued by the US Environmental Protection Agency (EPA) and the California Regional Water Quality Control Board (RWQCB) for wastewater treatment plants; permits issued by the South Coast Air Quality Management District (SCAQMD) for both wastewater treatment plants and landfills; biosolids disposal and re-use in accordance with 40 CFR

503; tertiary and reversed osmosis-treated water use for ground water recharge in accordance with Waste Discharge Regulations; and other Federal, State, and local rules and regulations.

3. Perform laboratory tests for plant process control in optimizing operational effectiveness to minimize the impact to the environment. Routine monitoring data are utilized to identify current or potential problem areas. Special tests are designed and performed to assist in troubleshooting.

4. Perform laboratory tests to assist the Industrial Waste Management Division in the pre-treatment management of various industries in the City of Los Angeles.

5. Perform laboratory tests to assist the Watershed Protection Division monitor the quality of inland receiving waters and enforce the provisions of the Stormwater NPDES permit.

B. PURPOSE OF THE QUALITY ASSURANCE MANUAL

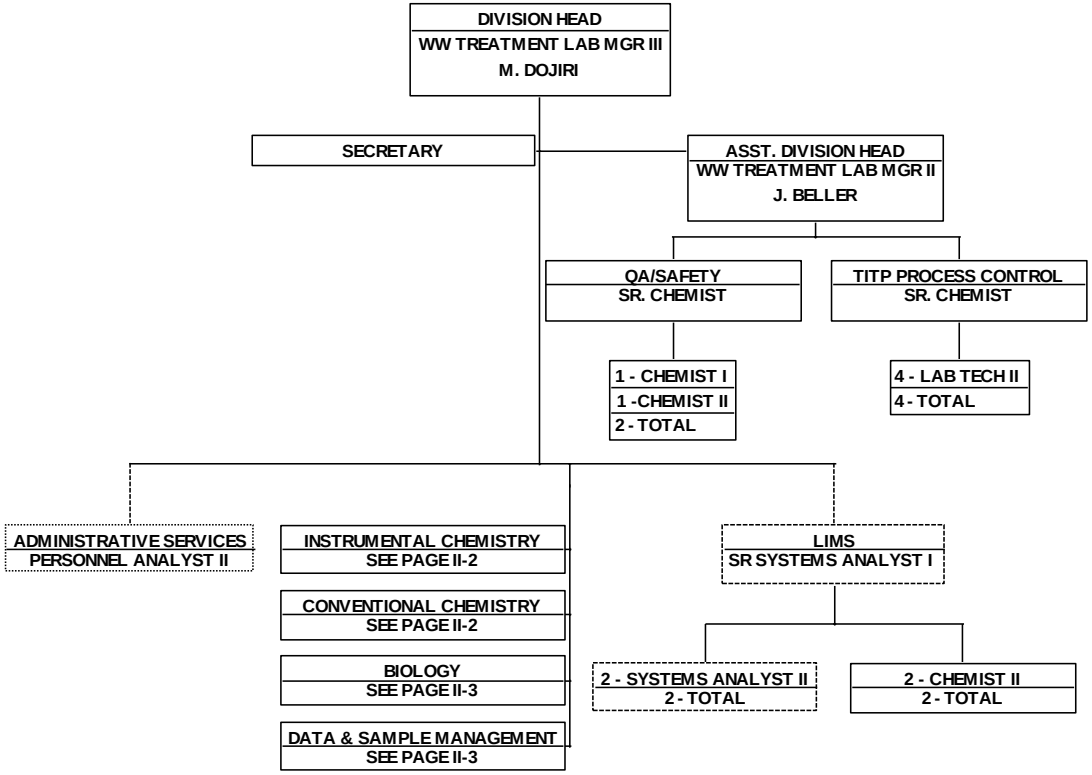
The purpose of this manual is to clearly describe the requirements of EMD' s Quality Assurance Program. A quality assurance program, based on a good quality assurance plan and applied to laboratory operations, is necessary to ensure that laboratory data meets predetermined quality assurance objectives and complies with the requirements of the US EPA, the California RWQCB, the California Department of Health Services (DHS), and the SCAQMD. A written quality assurance plan helps to ensure that consistent laboratory policies and operational procedures are followed by all laboratory personnel thereby assuring performance reliability of all sections in the laboratory. It provides a means for continuous assessment and improvement of the quality of data generated by laboratory personnel. Finally, implementation of this plan ensures that laboratory staff produce analytical results that can withstand legal scrutiny.

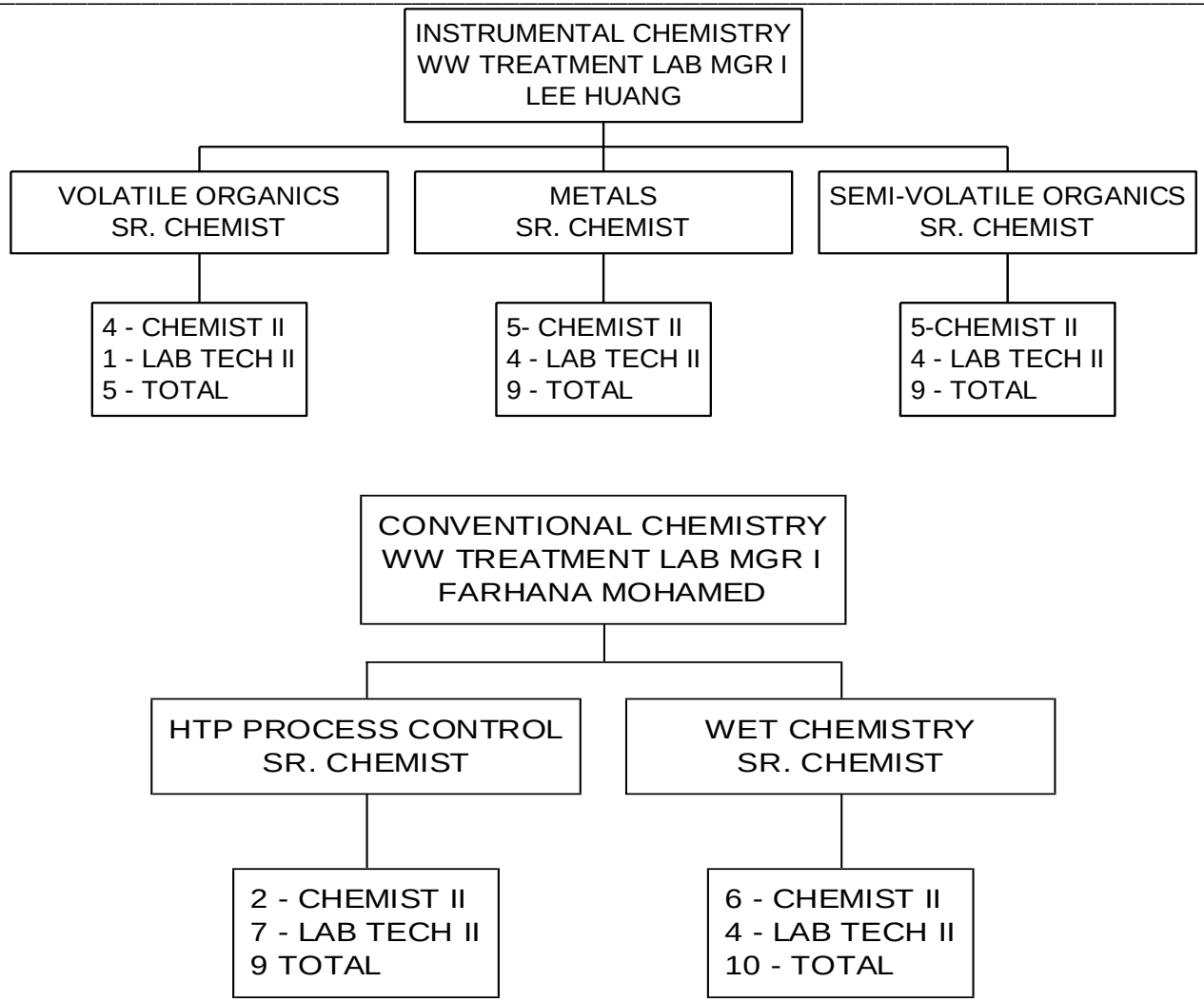
C. A STATEMENT OF THE DIVISION'S COMMITMENT TO QUALITY ASSURANCE

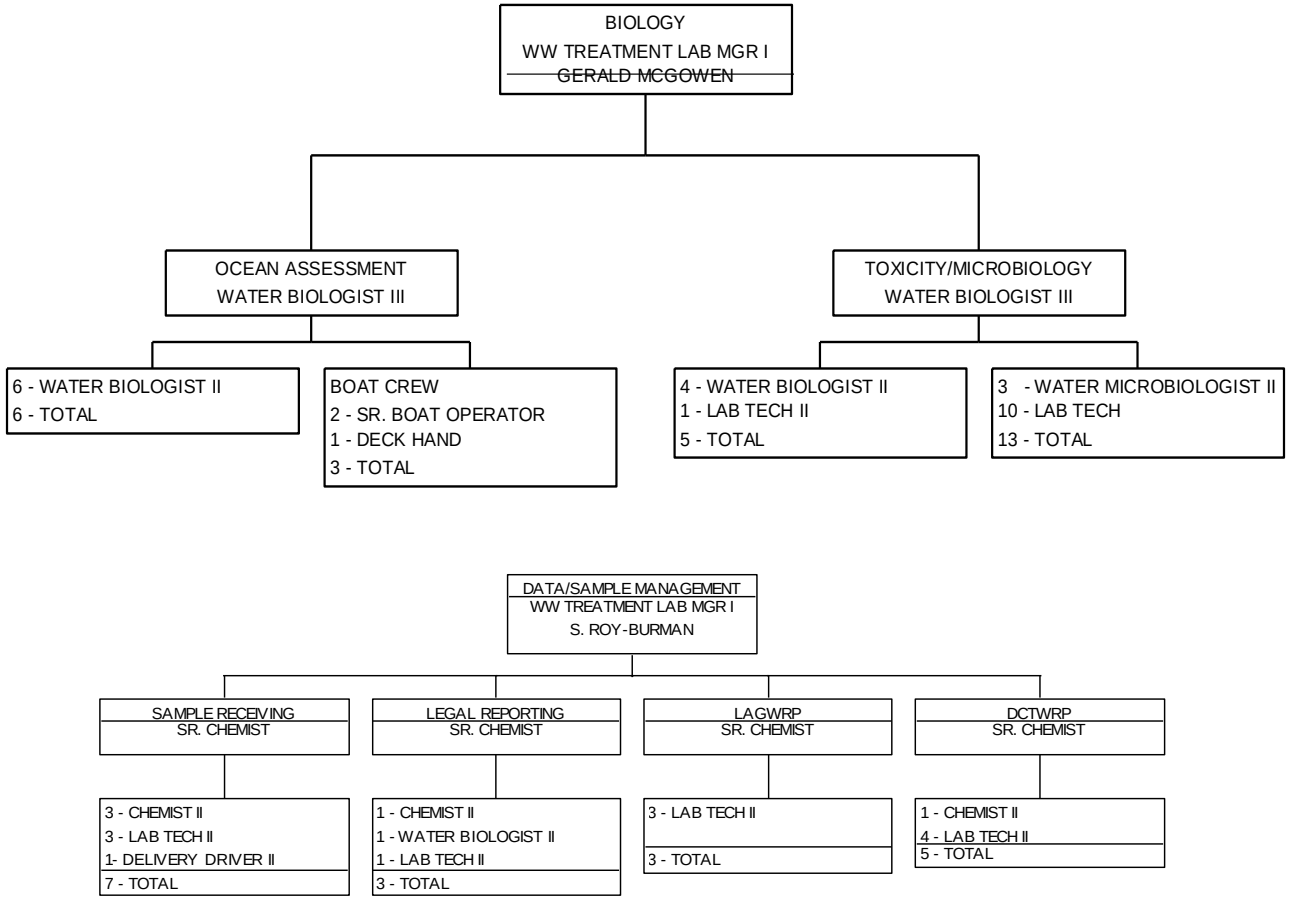
The Environmental Monitoring Division (EMD) of the Bureau of Sanitation, Department of Public Works, City of Los Angeles, provides laboratory services for the Bureau and other departments by performing legal and process control-related scientific monitoring and reporting. EMD is committed in providing quality data to its clients. EMD's policy of excellence in service is achieved through the active commitment of all levels of personnel within the division. It is the personnel within EMD who ensure the basic quality of service that EMD provides, and it is the Quality Assurance Program that assures the quality performance by our personnel. All personnel have been specifically instructed and are continuously reminded of the importance of strict adherence to the Quality Assurance Program. To this end, the Quality Assurance Officer takes the responsibility to ensure that personnel comply with the Quality Assurance Manual.

II. LABORATORY ORGANIZATION

A. ORGANIZATION CHART




INSTRUMENTAL CHEMISTRY
WW TREATMENT LAB MGR I
LEE HUANG
VOLATILE ORGANICS
SR. CHEMIST
METALS
SR. CHEMIST
SEMI-VOLATILE ORGANICS
SR. CHEMIST
4 - CHEMIST II
1 - LAB TECH II
5 - TOTAL
5- CHEMIST II
4 - LAB TECH II
9 - TOTAL
5-CHEMIST II
4 - LAB TECH II
9 - TOTAL
CONVENTIONAL CHEMISTRY
WW TREATMENT LAB MGR I
FARHANA MOHAMED
HTP PROCESS CONTROL
SR. CHEMIST
WET CHEMISTRY
SR. CHEMIST
2 - CHEMIST II
7 - LAB TECH II
9 TOTAL
6 - CHEMIST II
4 - LAB TECH II
10 - TOTAL


BIOLOGY
WW TREATMENT LAB MGR I
GERALD MCGOWEN
OCEAN ASSESSMENT
WATER BIOLOGIST III
TOXICITY/MICROBIOLOGY
WATER BIOLOGIST III
6 - WATER BIOLOGIST II
6 - TOTAL
BOAT CREW
2 - SR. BOAT OPERATOR
1 - DECK HAND
3 - TOTAL
4 - WATER BIOLOGIST II
1 - LAB TECH II
5 - TOTAL
3 - WATER MICROBIOLOGIST II
10 - LAB TECH
13 - TOTAL
DATA/SAMPLE MANAGEMENT
WW TREATMENT LAB MGR I
S. ROY-BURMAN
SAMPLE RECEIVING
SR. CHEMIST
LEGAL REPORTING
SR. CHEMIST
LAGWRP
SR. CHEMIST
DCTWRP
SR. CHEMIST
3 - CHEMIST II
3 - LAB TECH II
1- DELIVERY DRIVER II
7 - TOTAL
1 - CHEMIST II
1 - WATER BIOLOGIST II
1 - LAB TECH II
3 - TOTAL
3 - LAB TECH II
3 - TOTAL
1 - CHEMIST II
4 - LAB TECH II
5 - TOTAL

B. DESCRIPTION OF THE LABORATORIES

The Environmental Monitoring Division (EMD) maintains four laboratories, one in each of the City's Wastewater Treatment/Reclamation Plants, namely Hyperion Wastewater Treatment Plant, Terminal Island Wastewater Treatment Plant, D.C. Tillman Water Reclamation Plant, and L.A.-Glendale Water Reclamation Plant. These laboratories are individually certified by the Environmental Laboratory Accreditation Program (ELAP) of the California Department of Health Services.

1. City of Los Angeles, Environmental Monitoring Laboratory
   Hyperion Treatment Plant
   12000 Vista Del Mar
   Playa Del Rey, CA 90293
   (310) 648-5610

2. City of Los Angeles, Environmental Monitoring Laboratory
   Terminal Island Treatment Plant
   445 Ferry Street
   San Pedro, CA 90731
   (310) 732-4713

3. City of Los Angeles, Environmental Monitoring Laboratory
   Donald C. Tillman Water Reclamation Plant
   6100 Woodley Avenue
   Van Nuys, CA 91406
   (818) 778-4217

4. City of Los Angeles, Environmental Monitoring Laboratory
   L.A.-Glendale Water Reclamation Plant
   4600 Colorado Blvd.
   Los Angeles, CA 90039
   (213) 972-1307

CITY OF LOS ANGELES ENVIRONMENTAL MONITORING LABORATORY
HYPERION WASTEWATER TREATMENT PLANT
12000 Vista Del Mar
Playa Del Rey, CA 90293
(310) 648-5610

Jeffrey Beller
Laboratory Director

ELAP REGISTRATION NO: 1723
DATE OF FIRST ISSUE: JANUARY 31, 1992
RENEWAL DATE: JANUARY 31, 2008
EPA LAB I.D. NO: CA00375 (CHEMISTRY & MICROBIOLOGY)
EPA LAB I.D. NO: CA01301 (TOXICITY)
NPDES PERMIT NO: CA0109991

The laboratory is divided into four sections: Instrumental Chemistry, Conventional Chemistry, Biology, and Data and Sample Management. The Fields of Testing (FoTs) which are currently certified are listed below with their corresponding Method Numbers. A list of the instruments and equipment available in the laboratory are also included in the FoT lists.

Field of Testing 101 : Microbiology of Drinking Water

Subgroup Code	Analyte Code	Method	Analyte	Technology/ Medium
101.010	001	SM9215B	Heterotrophic Bacteria	Pour plate
101.060	002	SM9223	Total Coliform	Colilert [1]
101.060	003	SM9223	E. coli	Colilert [1]
			Enumeration in drinking water source	
101.140	001	SM9222A,B,C	Total Coliform (Enumeration)	MF/m-Endo [2]
101.150	001	SM9222D	Fecal Coliform (Enumeration)	MF/m-FC
101.160	001	SM9223	Total Coliform (Enumeration)	Colilert [3]

[1] Colilert represents both Colilert and Colilert 18 media.
[2] m-Endo represents both m-Endo and m-Endo LES media.
[3] A-1 is a single step multiple tube fermentation test for Fecal Coliforms only.

Field of Testing 107: Microbiology of Wastewater

Subgroup Code	Analyte Code	Method	Analyte	Technology/ Medium
107.010	001	SM9215B	Heterotrophic Bacteria	Pour plate
107.040	001	SM9221C,E (MTF/EC)	Fecal Coliform	MTF/EC
107.060	001	SM9222B	Total Coliform	MF/m-Endo [1]
107.080	001	SM9222D	Fecal Coliform	MF/m-FC
107.110	002	SM9230C (MF/ME)	Enterococci	MF/mE

[1] m-Endo Represents both m-Endo and m-Endo LES media.

Field of Testing 108: Inorganic Chemistry of Wastewater

SG Code	Analyte Code	Method	Analyte	Instrumentation/Sample Preparation
108.090	001	EPA 160.4	Residue, Volatile	
108.112	001	EPA 200.7	Boron	Thermo ICAP,Varian Vista-Pro
108.112	002	EPA 200.7	Calcium	Thermo ICAP,Varian Vista-Pro
108.112	003	EPA 200.7	Hardness (calc.)	Thermo ICAP,Varian Vista-Pro
108.112	004	EPA 200.7	Magnesium	Thermo ICAP,Varian Vista-Pro
108.112	005	EPA 200.7	Potassium	Thermo ICAP,Varian Vista-Pro
108.112	007	EPA 200.7	Sodium	Thermo ICAP,Varian Vista-Pro
108.120	002	EPA 300.0	Chloride	Dionex, DX 320, ICS-3000
108.120	003	EPA 300.0	Fluoride	Dionex, DX 320, ICS-3000
108.120	004	EPA 300.0	Nitrate	Dionex, DX 320, ICS-3000
108.120	005	EPA 300.0	Nitrite	Dionex, DX 320, ICS-3000
108.120	008	EPA 300.0	Sulfate	Dionex, DX 320, ICS-3000
108.183	001	EPA 335.4	Cyanide, Total	Lachat Quikchem FIA
108.200	001	EPA 350.1	Ammonia	Lachat Quikchem FIA
108.211	001	EPA 351.2	Kjeldahl Nitrogen	Lachat Quikchem FIA
108.232	002	EPA 353.2	Nitrite	Lachat Quikchem FIA
108.231	001	EPA 353.2	Nitrate calc.	Lachat Quikchem FIA
108.232	001	EPA 353.2	Nitrate-nitrite, Total	Lachat Quikchem FIA
108.360	001	EPA 420.1	Phenols, Total	Shimadzu UV 1601
108.381	001	EPA 1664A	Oil and Grease	SPEX-DEX 3000XL
108.390	001	SM2130B	Turbidity	HACH 2100AN
108.410	001	SM2320B	Alkalinity	Orion EA 920
108.430	001	SM2510B	Conductivity	Orion 115A+
108.440	001	SM2540B	Residue, Total	

SG Code	Analyte Code	Method	Analyte	Instrumentation/Sample Preparation
108.441	001	SM2540C	Residue, Filterable	
108.442	001	SM2540D	Residue, Non-filterable	
108.443	001	SM2540F	Residue, Settleable	
108.447	001	SM3120B	Boron	Thermo ICAP, Varian Vista-Pro
108.447	002	SM3120B	Calcium	Thermo ICAP, Varian Vista Pro
108.447	003	SM3120B	Hardness (calc.)	Thermo ICAP, Varian Vista Pro
108.447	004	SM3120B	Magnesium	Thermo ICAP, Varian Vista Pro
108.447	005	SM3120B	Potassium	Thermo ICAP, Varian Vista Pro
108.447	007	SM3120B	Sodium	Thermo ICAP, Varian Vista Pro
108.465	001	SM4500-Cl G	Chlorine	HACH Colorimeter
108.450	001	SM4500-Cl-B	Chloride	
108.473	001	SM4500-CN G	Cyanide, amenable	Lachat Quikchem FIA
108.490	001	SM4500-H+ B	pH	Orion EA 920, BECKMAN 71
108.491	001	SM4500-NH3 C	Ammonia	Labconco Kjeldahl
108.491	001	SM4500-NH3 C	Kjeldahl Nitrogen	Labconco Kjeldahl
108.493	001	SM4500-NH3 D	Ammonia	Orion EA 940
108.531	001	SM4500-O G	Dissolved Oxygen	Orion 083010 YSI 5010 Probes
108.540	001	SM4500-P E	Phosphate, Ortho	BECKMAN 7400 Shimadzu UV 1601
108.541	001	SM4500-P E	Phosphorus, Total	BECKMAN 7400 Shimadzu UV 1601
108.580	001	SM4500-S= D	Sulfide	Shimadzu UV 1601
108.590	001	SM5210B	Biochemical Oxygen Demand	Skalar BOD Analyzer
108.591	001	SM5210B	Carbonaceous BOD	Skalar BOD Analyzer
108.602	001	SM5220D	Chemical Oxygen Demand	Hach DR4000
108.610	001	SM5310B	Total Organic Carbon	Apollo 9000
108.611	001	SM5310C	Total Organic Carbon	Shimadzu TOC–V WS
108.620	001	SM5320B	Total Organic Halides	MITSUBISHI TOX-100
108.640	001	SM5540C	Surfactants	Shimadzu UV 1601
108.903	001	SM4500-B B	Boron	Shimadzu UV 1601
108.925	001	OIA-1677	Cyanide, amenable	Lachat Quickchem FIA
*	*	SM2540G	Total, Fixed, and Volatile Solids	

* FoT Not available
Ammonia analysis preceded by distillation
Kjeldahl Nitrogen analysis preceded by digestion and distillation

Field of Testing 109: Toxic Chemical Elements of Wastewater

SG Code	Analyte Code	Method	Analyte	Instrumentation/Sample Preparation
109.010	001	EPA 200.7	Aluminum	Thermo ICAP, Varian Vista-Pro
109.010	002	EPA 200.7	Antimony	Thermo ICAP, Varian Vista-Pro
109.010	003	EPA 200.7	Arsenic	Thermo ICAP, Varian Vista-Pro
109.010	004	EPA 200.7	Barium	Thermo ICAP, Varian Vista-Pro
109.010	005	EPA 200.7	Beryllium	Thermo ICAP, Varian Vista-Pro
109.010	007	EPA 200.7	Cadmium	Thermo ICAP, Varian Vista-Pro
109.010	009	EPA 200.7	Chromium	Thermo ICAP, Varian Vista-Pro
109.010	010	EPA 200.7	Cobalt	Thermo ICAP, Varian Vista-Pro
109.010	011	EPA 200.7	Copper	Thermo ICAP, Varian Vista-Pro
109.010	012	EPA 200.7	Iron	Thermo ICAP, Varian Vista-Pro
109.010	013	EPA 200.7	Lead	Thermo ICAP, Varian Vista-Pro
109.010	015	EPA 200.7	Manganese	Thermo ICAP, Varian Vista-Pro
109.010	016	EPA 200.7	Molybdenum	Thermo ICAP, Varian Vista-Pro
109.010	017	EPA 200.7	Nickel	Thermo ICAP, Varian Vista-Pro
109.010	019	EPA 200.7	Selenium	Thermo ICAP, Varian Vista-Pro
109.010	021	EPA 200.7	Silver	Thermo ICAP, Varian Vista-Pro
109.010	023	EPA 200.7	Thallium	Thermo ICAP, Varian Vista-Pro
109.010	024	EPA 200.7	Tin	Thermo ICAP, Varian Vista-Pro
109.010	026	EPA 200.7	Vanadium	Thermo ICAP, Varian Vista-Pro
109.010	027	EPA 200.7	Zinc	Thermo ICAP, Varian Vista-Pro
109.020	002	EPA 200.8	Antimony	Perkin Elmer ELAN 9000
109.020	003	EPA 200.8	Arsenic	Perkin Elmer ELAN 9000
109.020	004	EPA 200.8	Barium	Perkin Elmer ELAN 9000
109.020	005	EPA 200.8	Beryllium	Perkin Elmer ELAN 9000
109.020	006	EPA 200.8	Cadmium	Perkin Elmer ELAN 9000
109.020	007	EPA 200.8	Chromium	Perkin Elmer ELAN 9000
109.020	008	EPA 200.8	Cobalt	Perkin Elmer ELAN 9000
109.020	009	EPA 200.8	Copper	Perkin Elmer ELAN 9000
109.020	010	EPA 200.8	Lead	Perkin Elmer ELAN 9000
109.020	011	EPA 200.8	Manganese	Perkin Elmer ELAN 9000
109.020	012	EPA 200.8	Molybdenum	Perkin Elmer ELAN 9000
109.020	013	EPA 200.8	Nickel	Perkin Elmer ELAN 9000
109.020	014	EPA 200.8	Selenium	Perkin Elmer ELAN 9000
109.020	015	EPA 200.8	Silver	Perkin Elmer ELAN 9000
109.020	016	EPA 200.8	Thallium	Perkin Elmer ELAN 9000
109.020	017	EPA 200.8	Vanadium	Perkin Elmer ELAN 9000
109.020	018	EPA 200.8	Zinc	Perkin Elmer ELAN 9000
109.104	001	EPA 218.6	Chromium (VI)	Dionex ICS - 3000
109.400	001	SM3112B	Mercury	CETAC M-6000A, CETAC M7500
109.420	001	SM3114B	Arsenic	PE 300 FIAS, PE 400 FIAS
109.420	002	SM3114B	Selenium	PE 300 FIAS, PE 400 FIAS

SG Code	Analyte Code	Method	Analyte	Instrumentation/Sample Preparation
109.430	001	SM3120B	Aluminum	Thermo ICAP, Varian Vista-Pro
109.430	002	SM3120B	Antimony	Thermo ICAP, Varian Vista-Pro
109.430	003	SM3120B	Arsenic	Thermo ICAP, Varian Vista-Pro
109.430	004	SM3120B	Barium	Thermo ICAP, Varian Vista-Pro
109.430	005	SM3120B	Beryllium	Thermo ICAP, Varian Vista-Pro
109.430	007	SM3120B	Cadmium	Thermo ICAP, Varian Vista-Pro
109.430	009	SM3120B	Chromium	Thermo ICAP, Varian Vista-Pro
109.430	010	SM3120B	Cobalt	Thermo ICAP, Varian Vista-Pro
109.430	011	SM3120B	Copper	Thermo ICAP, Varian Vista-Pro
109.430	012	SM3120B	Iron	Thermo ICAP, Varian Vista-Pro
109.430	013	SM3120B	Lead	Thermo ICAP, Varian Vista-Pro
109.430	015	SM3120B	Manganese	Thermo ICAP, Varian Vista-Pro
109.430	016	SM3120B	Molybdenum	Thermo ICAP, Varian Vista-Pro
109.430	017	SM3120B	Nickel	Thermo ICAP, Varian Vista-Pro
109.430	019	SM3120B	Selenium	Thermo ICAP, Varian Vista-Pro
109.430	021	SM3120B	Silver	Thermo ICAP, Varian Vista-Pro
109.430	023	SM3120B	Thallium	Thermo ICAP, Varian Vista-Pro
109.430	024	SM3120B	Vanadium	Thermo ICAP, Varian Vista-Pro
109.430	025	SM3120B	Zinc	Thermo ICAP, Varian Vista-Pro
109.809	002	SM3500-Cr B	Chromium (VI)	Shimadzu UV 1601

Field of Testing 110: Volatile Organic Chemistry of Wastewater

SG Code	Analyte Code	Method	Analyte	Instrumentation
110.040	040	EPA 624	Halogenated Hydrocarbons	HP5890N/5973, Agilent 6890N/5975B
110.040	041	EPA 624	Aromatic Compounds	HP5890N/5973, Agilent 6890N/5975B
110.040	043	EPA 624	Other Volatile Organics	HP5890N/5973, Agilent 6890N/5975B

Field of Testing 111: Semi-volatile Organic Chemistry of Wastewater

SG Code	Analyte Code	Method	Analyte	Instrumentation
111.101	032	EPA 625	Polynuclear Aromatic Hydrocarbons	HP 5890/5972, 6890/5973, HP 6850/5975
111.101	034	EPA 625	Phthalates	HP 5890/5972, 6890/5973, HP 6850/5975
111.101	036	EPA 625	Other Extractables	HP 5890/5972, 6890/5973, HP 6850/5975
111.170	030	EPA 608	Organochlorine Pesticides	Varian 3800, HP6890
111.170	031	EPA 608	PCBs	Varian 3800, HP6890

Field of Testing 113: Whole Effluent Toxicity of Wastewater

Subgroup Code	Species Code	Method	Species
		Freshwater Acute	
113.010	001B	EPA 600/4-90/027F, Static Renewal	Fathead Minnow (P. promelas)
		Saltwater Acute	
113.025	009B	EPA 2006 (EPA-821-R-02-012), Static Renewal	Siverside (Menidia spp.)
113.027	012B	EPA 2007 (EPA-821-R-02-012), Static Renewal	Mysid (M. bahia)
113.028	008B	EPA-821-R-02-012, Static Renewal	Topsmelt (A. affinis)
		Freshwater Chronic	
113.040	001	EPA 1000 (EPA/600/4-91/002)	Fathead Minnow (P. promelas)
113.050	005	EPA 1002 (EPA/600/4-91/002)	Daphnid (C. dubia)
113.060	020	EPA 1003 (EPA/600/4-91/002)	Green algae (S. capricornutum)
		Saltwater Chronic	
113.120	008	EPA 600/R-95/136	Topsmelt (A. affinis)
113.120	017D	EPA 600/R-95/136, Fertilization Test	Purple sea urchin (S. purpuratus)
113.120	022	EPA 600/R-95/136	Giant kelp (M. pyrifera)
113.120	023	EPA 600/R-95/136	Red abalone (H. rufescens)

Field of Testing 114: Inorganic Chemistry of Hazardous Waste

SG Code	Analyte Code	Method	Analyte	Instrumentation/Sample Preparation
114.010	001	EPA 6010B	Antimony	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	002	EPA 6010B	Arsenic	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	003	EPA 6010B	Barium	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	004	EPA 6010B	Beryllium	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	005	EPA 6010B	Cadmium	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	006	EPA 6010B	Chromium	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	007	EPA 6010B	Cobalt	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	008	EPA 6010B	Copper	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	009	EPA 6010B	Lead	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	010	EPA 6010B	Molybdenum	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	011	EPA 6010B	Nickel	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	012	EPA 6010B	Selenium	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	013	EPA 6010B	Silver	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	014	EPA 6010B	Thallium	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	015	EPA 6010B	Vanadium	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.010	016	EPA 6010B	Zinc	THERMO ICAP,VARIAN VISTA-PRO/3050B, 3010A
114.140	001	EPA 7470A	Mercury	CETAC M-6000A, CETAC M7500
114.141	001	EPA 7471A	Mercury	CETAC M-6000A, CETAC M7500
114.240	001	EPA 9040B	pH	Orion EA 920, BECKMAN 71
114.241	001	EPA 9045C	pH	Orion EA 920, BECKMAN 71

Field of Testing 115: Extraction Test of Hazardous Waste

SG Code	Analyte Code	Method	Analyte
115.020	001	EPA 1311	Toxicity Characteristic Leaching Procedure (TCLP)
115.030	001	CCR Chapter11, Article 5, Appendix II	Waste Extraction Test (WET)

Field of Testing 116: Volatile Organic Chemistry of Hazardous Waste

SG Code	Analyte Code	Method	Analyte	Instrumentation/Sample Preparation
116.080	000	EPA 8260B	Volatile Organic Compounds	HP 5890-5970/ AGILENT 6890N-5973/5035 5g in Methanol, 5030B 5 ml
116.080	120	EPA 8260B	Oxygenates	HP 5890-5970/ AGILENT 6890N-5973/5035 5g in Methanol, 5030B 5 ml

Field of Testing 117: Semi-volatile Organic Chemistry of Hazardous Waste

SG Code	Analyte Code	Method	Analyte	Instrumentation/Sample Preparation
117.110	000	EPA 8270C	Extractable Organics	HP5890-5972, 6890-5973 / 3545(3g MethCl:Acetone), 3510C, 3520C(1L MethCl)-3620B
117.210	000	EPA 8081A	Organochlorine Pesticides	Varian 3800, HP6890 / 3545(3g MethCl:Acetone), 3510C, 3520C(1L MethCl)-3620B
117.220	000	EPA 8082	PCBs	Varian 3800, HP6890 / 3545(3g MethCl:Acetone), 3510C, 3520C(1L MethCl)-3620B

Field of Testing 119: Toxicity Bioassay of Hazardous Waste

SubgroupCode	Species Code	Method	Species
119.010	001	Polisini & Miller (CDFG 1988)	Fathead Minnow (P. promelas)

Field of Testing 120: Physical Properties of Hazardous Waste

SG Code	Analyte Code	Method	Analyte	Instrumentation
120.070	001	EPA 9040B	Corrosivity - pH Determination	Orion EA920,Beckmann 71
120.080	001	EPA 9045C	Corrosivity- pH Determination	Orion EA920,Beckmann 71

Field of Testing 126: Microbiology of Recreational Water

SG Code	Analyte Code	Method	Analyte	Technology/Medium
			Enumeration in recreational marine water	
126.020	001	SM9222A,B	Total Coliform (Enumeration)	MF/m-Endo
126.040	001	SM9222D	Fecal Coliform (Enumeration)	MF/m-FC
126.050	001	SM9223	Total Coliform and E. coli	Colilert 18
126.060	001	SM9230C	Enterococci	mE
126.080	001	IDEXX	Enterococci	Enterolert

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The Process Control Laboratory provides support to plant operations. The Fields of Testing that are currently certified, including instruments and equipment, are listed below.

Field of Testing 101 : Microbiology of Drinking Water

SG Code	Analyte Code	Method	Analyte	Technology/ Medium
101.060	002	SM9223	Total Coliform	Colilert [1]
101.060	003	SM9223	E. coli	Colilert [1]
			Enumeration in drinking water source	
101.160	001	SM9223	Total Coliform (Enumeration)	Colilert [1]

[1] Colilert represents both Colilert and Colilert 18 media.

Field of Testing 108: Inorganic Chemistry of Wastewater

SG Code	Analyte Code	Method	Analyte	Instrumentation/ Sample Preparation
108.090	001	EPA 160.4	Residue, Volatile	Mettler-Toledo AB204-S
108.390	001	SM2130B	Turbidity	HACH 2100N
108.410	001	SM2320B	Alkalinity	Orion 720A
108.440	001	SM2540B	Residue, Total	Mettler AB204-S
108.441	001	SM2540C	Residue, Filterable	Mettler AB204-S
108.442	001	SM2540D	Residue, Non-filterable	Mettler AB204-S
108.443	001	SM2540F	Residue, Settleable	
108.450	001	SM4500-Cl- B	Chloride	
108.460	001	SM4500-Cl B	Chlorine	
108.465	001	SM4500-Cl G	Chlorine	Thermo Orion AQ4000

SG Code	Analyte Code	Method	Analyte	Instrumentation/ Sample Preparation
108.470	001	SM4500-CN C	Cyanide, Manual Distillation	
108.472	001	SM4500-CN E	Cyanide, Total	Beckman DU64
108.490	001	SM4500-H+ B	pH	Orion 720A
108.500	001	SM4500-NH3 C	Ammonia	LABCONCO
108.501	001	SM4500-NH3 C	Kjeldahl Nitrogen	LABCONCO
108.531	001	SM4500-O G	Dissolved Oxygen	YSI 58
108.590	001	SM5210B	Biochemical Oxygen Demand	
108.591	001	SM5210B	Carbonaceous BOD	

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The Process Control Laboratory provides support to plant operations. The Fields of Testing (FoTs) that are currently certified, including instruments and equipment, are listed below.

Field of Testing 101 : Microbiology of Drinking Water

SG Code	Analyte Code	Method	Analyte	Technology/ Medium
101.060	002	SM9223	Total Coliform	Colilert
101.060	003	SM9223	E. coli	Colilert
			Enumeration in drinking water source	
101.140	001	SM9222A,B,C	Total Coliform (Enumeration)	MF/m-Endo [1]
101.150	001	SM9222D	Fecal Coliform (Enumeration)	MF/m-FC
101.160	001	SM9223	Total Coliform (Enumeration)	Colilert

[1] m-Endo represents both m-Endo and m-Endo LES media.

Field of Testing 107: Microbiology of Wastewater

SG Code	Analyte Code	Method	Analyte	Technology/ Medium
107.060	001	SM9222B	Total Coliform	MF/m-Endo [1]
107.080	001	SM9222D	Fecal Coliform	MF/m-FC
107.245	001	SM9223	E. Coli	Colilert

[1] m-Endo Represents both m-Endo and m-Endo LES media.

Field of Testing 108: Inorganic Chemistry of Wastewater

SG Code	Analyte Code	Method	Analyte	Instrumentation/Sample Preparation
108.090	001	EPA 160.4	Residue, Volatile	Thermolyne 48000
108.390	001	SM2130B	Turbidity	HACH 2100N
108.410	001	SM2320B	Alkalinity	Corning 250,Orion 720A+
108.421	001	SM2340C	Hardness	
108.430	001	SM2510B	Conductivity	Orion 162A,150A+
108.440	001	SM2540B	Residue, Total	Yamato DKN 600
108.441	001	SM2540C	Residue, Filterable	Yamato DKN 600
108.442	001	SM2540D	Residue, Non-filterable	Yamato DKN 600
108.443	001	SM2540F	Residue, Settleable	
108.465	001	SM4500-Cl G	Chlorine	HACH 46700-00
108.490	001	SM4500-H+ B	pH	Corning 250 Orion 720A+, Toledo
108.491	001	SM4500-NH3 C	Ammonia	Labconco
108.491	001	SM4500-NH3 C	Kjeldahl Nitrogen	Labconco
108.531	001	SM4500-O G	Dissolved Oxygen	YSI Model 58
108.540	001	SM4500-P E	Phosphate, Ortho	Shimadzu 160U, Shimadzu 1601
108.541	001	SM4500-P E	Phosphorus, Total	Shimadzu 160U, Shimadzu 1601
108.590	001	SM5210B	Biochemical Oxygen Demand	YSI 58 DO Meter
108.602	001	SM5220D	Chemical Oxygen Demand	Hach 5000

Field of Testing 126: Microbiology of Recreational Water

SG Code	Analyte Code	Method	Analyte	Technology/Medium
			Enumeration in recreational marine water	
126.020	001	SM9222A,B	Total Coliform (Enumeration)	MF/m-Endo
126.040	001	SM9222D	Fecal Coliform (Enumeration)	MF/m-FC
126.050	001	SM9223	Total Coliform and E. Coli	Colilert 18

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Field of Testing 101 : Microbiology of Drinking Water

SG Code	Analyte Code	Method	Analyte	Technology/ Medium
101.060	002	SM9223	Total Coliform	Colilert
101.060	003	SM9223	E. coli	Colilert
			Enumeration in drinking water source	
101.140	001	SM9222A,B,C	Total Coliform (Enumeration)	MF/m-Endo [1]
101.150	001	SM9222D	Fecal Coliform (Enumeration)	MF/m-FC
101.160	001	SM9223	Total Coliform (Enumeration)	Colilert

[1] m-Endo represents both m-Endo and m-Endo LES media.

Field of Testing 107: Microbiology of Wastewater

SG Code	Analyte Code	Method	Analyte	Technology/ Medium
107.060	001	SM9222B	Total Coliform	MF/m-Endo [1]
107.080	001	SM9222D	Fecal Coliform	MF/m-FC
107.245	001	SM9223	E. Coli	

[1] m-Endo Represents both m-Endo and m-Endo LES media.

Field of Testing 108: Inorganic Chemistry of Wastewater

SG Code	Analyte Code	Method	Analyte	Instrumentation/Sample Preparation
108.410	001	SM2320B	Alkalinity	Orion 720A+, Beckman 72
108.442	001	SM2540D	Residue, Non-filterable	Thelco
108.443	001	SM2540F	Residue, Settleable	
108.465	001	SM4500-Cl G	Chlorine	HACH DR 2500
108.490	001	SM4500-H+ B	pH	Orion 720A+, Beckman 72
108.531	001	SM4500-O G	Dissolved Oxygen	Orion EA 940
108.590	001	SM5210B	Biochemical Oxygen Demand	Orion EA 940
108.602	001	SM5220D	Chemical Oxygen Demand	

Field of Testing 126: Microbiology of Recreational Water

SG Code	Analyte Code	Method	Analyte	Technology/Medium
			Enumeration in recreational marine water	
126.020	001	SM9222A,B	Total Coliform (Enumeration)	MF/m-Endo
126.040	001	SM9222D	Fecal Coliform (Enumeration)	MF/m-FC
126.050	001	SM9223	Total Coliform and E. Coli	Colilert 18

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Section B: Wastewater Parameters

Microbiology of Wastewater

Description	Internal Code	Reference	Method
Fecal Coliform by MTF	14.20	C 9221E	SM 9221 E

Section C: Hazardous Waste Parameters

Metals Sample Preparation in Hazardous Waste

Description	Internal Code	Reference	Method
Sludges and Soil	27.9	F 3050B	EPA 3050B

Metals of Hazardous Waste

Description	Internal Code	Reference	Method
Arsenic	9.2	F 6010B	EPA 6010B
Cadmium	9.5	F 6010B	EPA 6010B
Chromium, Total	9.6	F 6010B	EPA 6010B
Copper	9.8	F 6010B	EPA 6010B
Lead	9.9	F 6010B	EPA 6010B
Mercury	9.10	F7471A	EPA 7471A
Molybdenum	9.11	F 6010B	EPA 6010B
Nickel	9.12	F 6010B	EPA 6010B
Selenium	9.13	F 6010B	EPA 6010B
Zinc	9.17	F 6010B	EPA 6010B

Microbiology of Hazardous waste

Description	Internal Code	Reference	Method
Salmonella in Sludge	WW	C 9260D	SM 9260D
Total, Fixed and Volatile Solids in Solids, and Semisolid Samples in Sludges	WW	C 2540G	SM 2540G

III. PERSONNEL RESPONSIBILITIES

All laboratory personnel in the division are responsible for the implementation of the Quality Assurance Program. Quality assurance starts with sample collection and extends to the Division Manager who, through the Quality Assurance Officer, oversees the management of the program. It is only through the combined efforts of technical staff and management that quality assurance is achievable.

A. DIVISION MANAGER

1. Assumes final responsibility and authority of the Quality Assurance Program.

2. Assures proper implementation of the Quality Assurance Program by reviewing the activities of the Quality Assurance Officer and laboratory managers.

3. Reviews and approves the Quality Assurance Manual.

B. QUALITY ASSURANCE OFFICER

1. Reports to the Division Manager (or designee) directly and is independent of all laboratory operations.

2. Monitors and coordinates division-wide quality assurance activities to meet requirements of regulatory agencies and the division's quality assurance policy; conducts system audits and inspections; and submits reports, including recommended corrective actions and follow-through.

3. Submits performance evaluation and blind samples when needed. Evaluates test results statistically and issues performance reports. Discusses with laboratory managers appropriate/required corrective actions and reports to the Division Manager.

4. Reviews and updates the Quality Assurance Manual for the Division Manager's approval.

5. Interprets quality assurance requirements. Recommends changes in quality assurance protocols to the Division Manager for approval.

6. Ensures proper documentation and record-keeping; ensures maintenance of records including control charts, calibration records, performance evaluations, and system audits.

7. Trains the QA staff to perform quality assurance functions.

8. Disseminates QA information, as well as the importance and relevancy of analyses, to laboratory personnel.

9. Keeps abreast of new practices and procedures in quality assurance; recommends changes to the Division Manager to ensure a dynamic and efficient operation.

10. Provides annual review and assessment of QA/QC staff activities for EMD management.

11. Coordinates communication with regulatory agencies.

12. Reviews requests from laboratory managers for compliance with regulatory requirements and record-keeping to formally bring new equipment on-line. Provides Performance Package for new equipment.

13. Reviews regulatory requirements and record-keeping data packages for compliance when submitted by laboratory managers to change testing methodology. Provides Performance Package for new method.

14. Acts as the official source on the status of QA/QC-related information by closely communicating with laboratory managers and unit supervisors.

C. LABORATORY MANAGERS

1. Review quality control programs of each unit; provide a plan for improvement; and implement the improvement plan.

2. Ensure that unit supervisors adhere to guidelines listed under "UNIT SUPERVISORS" in this chapter of the Quality Assurance Manual.

3. Review with unit supervisors the unit's quality control performance and confer with the QA Officer to provide assessments and recommendations to unit supervisors.

4. Receive corrective action recommendations from the QA Officer and work with unit supervisors and the QA Officer to resolve the problems.

5. Review unit supervisor's evaluation of methodologies and provide input.

6. Work with the QA Officer to provide policy direction and interpretation on quality assurance matters to personnel in the section.

7. Periodically report to the Division Manager and the QA Officer on the status of their section's quality control practices and performances.

8. In conjunction with the QA Officer, maintain communication with regulatory agencies.

9. Evaluate for completeness of data packages submitted by unit supervisors to bring new equipment on-line and submit these to the QA Officer for compliance checking and record-keeping.

10. Evaluate for completeness of data packages submitted by unit supervisors to change testing methodology and submit these to the QA Officer for compliance checking and record keeping.

11. Inform the QA Officer of all QA/QC program changes.

D. QUALITY ASSURANCE STAFF MEMBERS

1. Assist the QA Officer in developing, revising, and coordinating quality assurance/control programs.

2. Assist in the management, documentation, and record keeping of the established QA Program.

3. Prepare an annual report of QA activities and submit to QA Officer for approval.

4. Review and monitor QC data and inform the QA Officer of long-term trends.

5. Periodically assist the QA Officer to inspect laboratories and evaluate compliance with Good Laboratory Practices (GLP) criteria.

6. Assist the QA Officer in certification programs and related performance evaluation program management.

E. UNIT SUPERVISORS

1. Ensure that all analysts adhere to guidelines listed under "ANALYSTS" in this chapter of the Quality Assurance Manual.

2. Ensure that all staff members are trained to perform each assigned analytical task with good analytical technique.

3. Ensure that unit Standard Operating Procedures (SOP's) have been written and are available to the analysts; review SOP's and recommend modifications to laboratory managers.

4. Ensure that analyses are conducted according to the protocols described in the QA Manual and approved SOPs.

5. Ensure proper collection, receiving, preservation, handling, and disposal of samples.

6. Provide day-to-day supervision of unit laboratory operations to ensure that GLP and all safety rules are met.

7. Review and evaluate unit QA/QC activities; take corrective action when necessary.

8. Review data for correctness, proper documentation, and compliance with QC criteria established for each method; validate data.

9. Periodically evaluate all methods currently being used in the unit to assess the need for method modifications.

10. Implement specific changes required by corrective action recommendations to assist laboratory managers in resolving QA/QC problems.

11. Promote staff understanding of each analytical methodology: its theory, purpose for each step, and key factors affecting the results of the analysis.

12. Implement specific changes in QA requirements received from the QA Officer or laboratory manager.

13. Conduct new equipment start-up evaluation procedures, prepare data packages, and submit to laboratory managers for review.

14. Conduct new methodology evaluations and parallel studies, prepare data packages, and submit to laboratory managers for review.

15. Inform laboratory managers of QA/QC program changes that have been implemented.

F. ANALYSTS

1. Have a clear understanding of, and strictly adhere to, the guidelines outlined in the Quality Assurance Manual.

2. Understand the theory behind each analytical method to be performed.

3. Perform each analysis according to approved protocols and SOP's.

4. Follow QC requirements for each analysis to provide clear and traceable documentation; review QC results.

5. Notify the supervisor of all QC problems, equipment malfunctions, and safety hazards.

6. Develop the ability to identify potential analytical problems, communicate them to the supervisor, and suggest corrective actions.

7. Respond to and/or implement corrective action.

8. Subscribe to good laboratory practices, including general housekeeping, and observe all safety rules.

IV. QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT OF DATA

The quality assurance objectives for measurement of data are unique to the particular program for which the data are collected and utilized. They describe the overall uncertainty that the data user is willing to accept in order to make decisions for environmental or other concerns. This uncertainty describes the data quality that is needed, which are usually expressed in terms of precision, bias, representativeness, comparability, and completeness. Prior to starting, the project should define the data quality objectives and how they will be attained in order for all laboratory and field personnel involved to make informed decisions during the course of the project.

The laboratory staff at EMD use approved and recognized test methods, and complies with their QC requirements. Quality control samples are measured and precision and accuracy are assessed, and need to be within the method prescribed limits. Internal acceptance criteria are established by analyzing laboratory control samples on a routine basis. Therefore, EMD can attest to the quality of the measured data being provided to the client. If its personnel conduct the sampling, EMD will also be able to attest to the integrity of the sampling process.

V. SAMPLE MANAGEMENT

This section covers two sample management elements defined by the Environmental Laboratory Accreditation Program (ELAP): 1) sampling procedures when the laboratory performs the sampling and 2) custody, holding time, and disposal of samples.

A. SAMPLING PROCEDURES

Laboratory personnel follow each unit's sampling plan to collect samples. Samples are usually site-specific for a predetermined location, routinely scheduled, or dictated by emergency response procedures. Only trained laboratory staff are assigned to collect samples using proper sampling procedures, appropriate sampling equipment, required containers, and proper preservation techniques. For those samples collected by Wastewater Treatment Operators, EMD staff works closely with operators to ensure proper sampling procedures are followed.

Selection of containers, application of preservatives, and holding times specifications are based on EPA guidelines. Refer to Appendix A for specific requirements. This section also includes the sample size ordinarily required for analyses.

General guidelines for sample collection by laboratory staff are as follows:

1. Care must be taken to prevent contamination of the sample by using appropriately cleaned sample jars. For routine process operations, jars are pre-assigned to a specific site and specific parameters.

2. Samples must be uniquely identified. At a minimum, they should be labeled with sample date, sample time, sampling point, and the name of the sampler. This information, as well as other pertinent information such as sample type, preservative added, and analyses should be recorded on the chain of custody.

3. Once received, samples are logged into the Laboratory Information Management System (LIMS) as soon as possible, assigned a unique number, and properly stored.

4. Sample preparation steps done prior to analysis, such as sieving, blending, filtration, grinding, compositing, mixing, sub-sampling, and preservation are described in individual test SOP's.

B. CUSTODY, HOLDING, AND DISPOSAL

1. CHAIN-OF-CUSTODY

Samples submitted to EMD at HTP for analysis are delivered to the Harry Pregerson Technical Support Building (HPB) Room 550, EMD's sample receiving area. A chain-of-custody (COC) must accompany each sample submitted to EMD. If a COC has not been filled out prior to delivery of the sample, a form will be provided to the delivery person prior to EMD acceptance of said sample. The COC will be reviewed to make sure that all of the needed information has been supplied. The Chain-of-Custody Form being used at EMD is shown in Figure 2.

The purpose of the chain-of-custody is to establish detailed written and legal documentation of all transactions in which samples are transferred from the custody of one individual to another. The custody procedure is also used whenever samples are submitted to a laboratory within the division or to a contract laboratory. The chain-of-custody begins at the sample collection site and includes couriers or messengers who handle the sample in transit. It follows the sample until its ultimate disposal. It is a form of proof used to establish the authenticity and integrity of the sample, since there is always the possibility that the chain-of-custody will be used in litigation.

According to EPA's National Enforcement Investigation Center, a sample is under custody if one of the following situations is applicable:

a. It is in your possession, or

b. It is in your view, after being in your possession, or

c. It is in your possession and you locked it up, or

d. It is in a designated secure area, or

e. It is in your possession and you document the transfer of custody to the receiving party.

2. HOLDING TIME

Analyses of samples must meet EPA holding time requirements for each parameter. The holding times and/or sample preservation are crucial to some analyses (e.g., BOD, cyanide, sulfide, indicator bacteria, and oil and grease). If

the sample requires preservation and it was not performed in the field, EMD personnel will preserve the sample in accordance with the analysis requested of that sample. Some tests, e.g., toxicity, require cooling to 4^{o}C during transport.

For tests with such a requirement, temperature must be measured at the time of arrival at the laboratory and recorded on the chain-of-custody. All samples must be stored under the right conditions (e.g., refrigeration may be required) and free from contamination. See Appendix A for specific requirements.

2. SAMPLE LOGGING AND HANDLING

The sample information will be entered into the EMD Laboratory Information Management System (LIMS) and a unique laboratory registration number will be generated for that sample. The sample will then be split into different aliquots as dictated by the requested analyses and sent to the appropriate operating laboratories for testing within the holding time specified per analysis. A portion of the sample will be retained in the sample receiving area (under proper storage conditions) for archiving purposes. If the sample is split into different aliquots, each aliquot will be uniquely identified.

3. DISPOSAL

After the analyses are completed, the sample will be retained as legal evidence or legally disposed of as determined by the chemical or biological analysis of the sample. Analyzed samples and standards used in analyses are disposed of according to EMD's Chemical Hygiene Plan.

VI. ANALYTICAL PROCEDURES

A. ANALYTICAL PROCEDURES

1. ANALYSES

   a. Analyses performed at EMD laboratories are generally driven by regulatory concerns and plant operations' requirements. There are many different analytical methods applicable to environmental analyses. Our methods are generally based on those specified by EPA, Federal and State regulatory agencies, or professional organizations.

   b. EMD laboratories also use methods that are adopted from scientific literature, developed internally, or acquired with the purchase of instruments or reagent kits. Results from these methods are for in-house (internal) information only. Occasionally, we are required by permits to perform certain types of analyses where official methods are not yet available. Under these circumstances, data from the best methods available are used to fulfill the regulatory requirements.

   c. The choice of methodology depends on regulatory requirements, the intended use of the generated values by the data user, sample matrix, availability of equipment (e.g., GC, GC/MS, ICP, ICP/MS), quantitative sensitivity, sample size, turn-around time, accuracy, precision, and cost.

2. REFERENCES

   Typical methodology references available at EMD are listed below:

   a. "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, revised March 1983.

   b. "Test Methods for Evaluating Solid Waste" (SW 846), 3rd edition (Nov. 1986) to Update III (Dec. 1996), Office of Solid Waste and Emergency Response, U.S. EPA.

   c. "Standard Methods for the Examination of Water and Wastewater", several editions, APHA, AWWA, WPCF, Washington, DC. Official use of a particular edition for legal reporting requires EPA sanction. As

of the revision date of this manual, the 18th, 19th, and 20th editions were approved for use.

d. "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act", 40 CFR, Part 136.

e. "Determination of Inorganic Anions in Water by Ion Chromatography", Method 300.0, EPA, version 2.1, rev. Aug. 1993.

f. "Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma – Mass Spectrometry", Method 200.8, EPA, version 5.4, May 1994.

g. "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma – Atomic Emission Spectrometry", Method 200.7, EPA, version 4.4, May 1994.

h. "Annual Book of ASTM Standards", Volumes 11.01, 11.02, 14.01, and 14.02, ASTM, Philadelphia, PA, 1990 and 1997.

i. "Official Methods of Analysis", 13th edition, AOAC, Arlington, VA., 1980.

j. "Microbiological Methods for Monitoring the Environment, Water, and Wastes", EPA-600/8-78-017.

k. "Static Acute Bioassay Procedures for Hazardous Waste Samples", Polisini and Miller (CDFG), 1998, Title 22, CCR 66261.24.

l. "Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms", EPA/600/4-85/013.

m. "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms", EPA/600/4-90/027F

n. "Short-term Methods of Estimating the Chronic Toxicity of Effluents and Receiving Water to Freshwater Organisms", EPA-600-4-91-002.

o. "Short-term Methods of Estimating the Chronic Toxicity of Effluents and Receiving Water to Marine and Estuarine Organisms", EPA/600/4-91-003.

p. "Procedures Manual for Conducting Toxicity Tests Developed by the Marine Bioassay Project", State of California Water Resources Board, 90-10WQ, Anderson, B.S. et al., 1990.

q. "Short-term Methods of Estimating the Chronic Toxicity of Effluents and Receiving Water to West Coast Marine and Estuarine Organisms", EPA/600R/95/136.

r. "Methods for Aquatic Toxicity Identification Evaluation, Phase I Characterization Procedures", EPA 600/6-91/003.

s. "Methods for Aquatic Toxicity Identification Evaluation, Phase II Toxicity Identification Procedures", EPA 600/3-88/035.

t. "Methods for Aquatic Toxicity Identification Evaluation, Phase III Toxicity Confirmation Procedures", EPA 600/3-88/036.

B. STANDARD OPERATING PROCEDURES (SOPs)

Routine analyses are defined in Standard Operating Procedures (SOPs) which are detailed descriptions of how to use and what to expect from a method. They contain method-specific QC criteria (i.e., instrument calibration, reagent blank, method blank, calibration standards, etc.), and QC requirements such as duplicate analysis, spike recoveries, holding time, etc. EMD follows a standardized SOP format; its content and application are presented in Appendix D.

C. ANALYSES PERFORMED IN EMD LABORATORIES

Analyses performed in the laboratories are grouped under the following general classifications:

1. WET CHEMICAL METHODS

a. Colorimetric Analyses - Tests include cyanide, phenols, nitrogen (nitrate, nitrite), MBAS, hexavalent chromium, dissolved sulfide, boron, residual chlorine, and phosphate, etc.

b. Titrimetric Analyses - Hardness, alkalinity, chloride, ammonia, and organic nitrogen, and biological oxygen demand (BOD) are typical analyses.

c. Gravimetric Analyses - Oil and grease, solids, moisture, sulfate, etc.

d. Miscellaneous Tests - A meter or probe is used to measure conductivity, turbidity, temperature, DO, ammonia, and pH.

e. Inorganic Analyses (Anions and Cations) - Ion chromatograph (IC) is used primarily to analyze for anions such as chloride, sulfate, fluoride, nitrate, and nitrite.

2. INSTRUMENTAL ANALYSES

Analyses in this group require complex, sophisticated instrumentation and the training of analysts in instrument operation. Analytes fall into two major categories: organic and inorganic (metals). There are common elements associated with the selection of an instrument that include application, sensitivity, initial and continuing instrument calibration, dynamic linear range, detection limits, matrix effects, and instrument limitations.

a. Organic Analyses

Instruments used for organic analyses are Gas Chromatograph (GC) or Gas Chromatograph/Mass Spectrometer (GC/MS). Gas chromatographs are equipped with detectors specific to the analytes of interest. Gas chromatographic identification requires a secondary column to confirm measurements made by the primary column. Individual organic compounds are generally grouped into these categories:

* Herbicides (chlorinated)

* Pesticides (organochlorine) and PCBs

* Semivolatile Organic Compounds

* Volatile Organic Compounds

b. Inorganic Analyses - Trace Metals

Instruments for metal analysis include an atomic absorption spectrophotometer equipped with a cold vapor generator/analyzer or a Hydride Generator (HGAA), Inductively-Coupled Plasma (ICP), and Inductively-Coupled Plasma Mass Spectroscopy (ICP/MS).

3. MICROBIOLOGICAL ANALYSES

The analysis performed depends upon the matrix of the sample and/or the bacterial organisms of interest.

a. Membrane Filtration

* Total coliform
* Fecal coliform
* Enterococcus

b. Multiple-tube Fermentation

* Fecal coliform
* Salmonella

c. Chromogenic Substrate

* Total coliform
* E.coli
* Enterococcus

d. Heterotrophic Plate Count

e. Double-agar Overlay

* Bacteriophage

4. BIOASSESSMENT ANALYSES

a. Toxicity Testing

* Acute toxicity tests

* Hazardous waste toxicity tests

* Marine chronic toxicity tests

* Freshwater chronic toxicity tests

b. Ocean Assessment

* Water quality monitoring is performed using a Conductivity-Temperature-Depth (CTD) profiler to measure salinity, temperature, transmissivity, density, dissolved oxygen, and pH, and chlorophyll.

* Benthic sorting and taxonomic identification are done using dissecting and compound microscopes. Identifications are confirmed using peer-reviewed published literature, as needed. Species name, abundance, biomass, and locality of collection are recorded.

* Taxonomic identifications of trawled organisms are performed using peer-reviewed published literature, as needed. Species name, abundance, standard length for fish, biomass, and locality of collection are recorded.

* Rig-fishing data include species name, number collected, weight, standard length, and locality of collection.

D. START-UP TEST (INITIAL DEMONSTRATION OF TEST PROFICIENCY), ALTERNATE TEST PROCEDURES, METHOD MODIFICATION, AMENDMENT TO AN ELAP-CERTIFIED FIELD OF TESTING

There are similarities and differences in the requirements for a start-up test, obtaining approval for alternate test methods, modifications to a method, and amendment to an ELAP-certified field of testing. For this reason, it is recommended that the plan of action be discussed with and concurred by the QA Officer before initiating the project.

Furthermore, when all the required validation tests are completed, laboratory managers must submit to the QA Officer a formal request to officially adopt new start-ups or modifications. The QA Officer shall review the data package and submit it to the designated laboratory director for final approval. Upon the laboratory director's approval, the QA Officer shall record the date of approval and notify laboratories for implementation.

1. START-UP TEST (INITIAL DEMONSTRATION OF TEST PROFICIENCY)

   EPA, as well as ELAP, requires the laboratory to perform a start-up test prior to using a promulgated/approved method for routine analysis. The start-up test must be documented. This requirement is applicable to chemical analyses, as well as microbiological analyses, bioassay analyses, and any other non-chemical procedures.

2. ALTERNATE TEST PROCEDURES

   EPA defines an alternate test procedure as "one that differs from a method previously approved for determining the constituent of interest in National Pollutant Discharge Elimination System (NPDES) monitoring". EPA has established criteria that must be met before an alternate test procedure can be approved for use.

3. METHOD MODIFICATION

   a. The EPA Office of Water allows limited flexibility within the promulgated wastewater methods to improve method performance and has guidelines for method modification.

   b. The Office of Solid Waste has defined general measures for modifying SW-846.

4. AMENDMENT TO ELAP-CERTIFIED FIELD OF TESTING

   The Environmental Laboratory Accreditation Program of the California Department of Health Services allows the addition of one or more subgroups from a certified field of testing provided certain requirements are met.

VII. CALIBRATION PROCEDURES AND FREQUENCY

All analytical systems/instruments are calibrated at the time of use, or as often as each method requires, with standards traceable to the National Institute of Standards and Technology (NIST), EPA, or other certified standard sources. Each instrument is calibrated within its dynamic linear range bracketing the concentration of the target analyte, and for spectrophotometers, within the optimum performance range. Some instruments may require final calibration at the end of a test analysis. Calibration processes should comply with method-specific requirements and must be documented.

A. REAGENTS AND SOLVENTS

1. Reagents are analytical grade or better, properly stored, and discarded after the expiration date.

2. All analytical reagents/solvents received in the laboratory are labeled with the following information:

   a. Date received

   b. Date opened

   c. Expiration date

B. STANDARD SOLUTIONS

Calibration standard solutions are prepared from neat compounds/solutions, concentrates, or raw material of documented purity. The most appropriate measuring devices and techniques are used in preparing calibration standards. Prepared reagents are properly stored. All calibration standards are cross referenced with standards or check samples from a different source or lot number and are ultimately traceable to an NIST-certified source.

1. STANDARD PREPARATION LOG

   A standard preparation log is maintained by each lab unit. A logbook entry includes the following information:

a. Source of the standard

b. Lot number

c. Cross check

d. Dilutions, final concentrations (units)

e. Preparer

f. Date prepared

g. Expiration date

2. LABELING

Prepared solutions are labeled with the following information:

a. Identity of the solution

b. Concentration (with units of measurement)

c. Date prepared

d. Expiration date

e. Preparer's identity

3. EXPIRATION DATE

The expiration date of the prepared solution is established based on the information below:

a. Analyte concentration of the solution

b. Stability of chemical/solution under specified conditions

c. Manufacturer's recommendation

d. Method-specific requirement

d. In no case is the expiration date established by the laboratory later than the expiration date certified by the manufacturer.

C. VOLUMETRIC ANALYSIS

The frequency of re-standardizing a titrant against a primary standard is based on method- specific requirements and the stability (shelf-life) of the solution.

D. STANDARD CURVE

1. The standard curve is constructed with the minimum of standards specified in the method and may include a reagent blank. The range of the standards encompasses the entire linear range or the range of interest.

2. All samples quantified must be within the calibration curve. Calculation by extrapolation is not acceptable to EPA or ELAP. Subsequent sample dilution or the construction of a new curve is required.

3. Some manufacturers suggest single point calibration, excluding the blank, for daily use of their instruments (e.g., TOC Analyzer, ICP, and ICP/MS). In this case, a reagent blank and a high standard may be used provided low and mid-range solutions are run as unknown samples and the results are within the acceptance limits.

E. INSTRUMENT CALIBRATION

1. Instruments are calibrated before use with documentation to support the calibration process. Documentation includes name of person who performed the calibration, date, the specific test or instrument, and linearity such as correlation coefficient. State of the art instruments are capable of drawing the calibration curve.

2. Each instrument is calibrated with standard solutions specific to the analysis and appropriate to the instrument.
   Generally, the frequency of calibration, as well as the concentration and number of standards, are based on instrument manufacturer's guidelines, analytical method, or requirements of clients.

3. Initial instrument calibration is performed with three to six calibration points, depending on method-specific requirements. The calibration curve is verified

with at least one standard during the course of sample analysis. The linear range of the standards should be determined and should bracket the expected concentration of the samples with the lowest standard near the detection limit.

4. Specific calibration procedures for each analytical method are described in the SOP for that particular method.

VIII. ACQUISITION, REDUCTION, VALIDATION, AND REPORTING OF DATA

The analyst who generates the data has the initial and primary responsibility for the completeness and correctness of the data. The data are then checked by the unit supervisor (or designee).

A. ACQUISITION

Both raw and calculated data are acquired in the laboratory by manual or electronic (direct computer) acquisition. Acquired data are properly and securely stored for the duration specified by regulatory agencies and the customer.

Guidelines for documentation and recording of information are as follows:

1. MANUAL DATA ENTRY

   a. Hand-written on Worksheet

      * Data are entered directly into the notebook or worksheet with non-erasable ink.

      * Data entries are initialed and dated by the analyst making the entry. If the entry is more than one page, each page is initialed and dated.

      * Mistakes are corrected by drawing a single line through the entry, entering the correct value, and initialing and dating the correction. The use of correction fluid is not acceptable.

      * Blank pages or substantial portions of pages with no entries are marked with a large "X" to indicate that they were intentionally left blank.

   b. Manual Entry into Computer

      The program/software used to generate results is prepared internally. A designated staff member of the Information & Control Systems Division (ICSD) at Hyperion has the responsibility of preparing the program and maintaining the supporting documents.

2. ELECTRONIC DATA ACQUISITION (spectra, chromatogram, or hard-copy read-outs from instruments)

   a. Print-outs contain the laboratory sample ID/number, analyst initials, date, time, etc.

   b. Clear identification on the print-out for each peak of interest.

B. REDUCTION

Data reduction, where applicable, is described in specific SOP's. It involves reporting values with the appropriate significant figures in the concentration units established by the regulatory agency or the data user.

1. USE OF SIGNIFICANT FIGURES

   The following three guidelines should be taken into consideration when determining the number of significant figures to include in reporting results:

   a. Method constraints – Some methods constrain the number of significant figures that can be reported. Analysis of bacteria by methods that generate a Most Probable Number (MPN) is one example of this.

   b. Accuracy of the MDL – Data should not be reported that is more accurate than the MDL.

   c. In most, if not all cases, data should contain no more than three significant figures.

2. RULES OF ROUNDING OFF

   When results need to be rounded off in order to conform to the number of significant figures necessary to be reported for the result, the following guidelines should be used:

   a. When a number to be rounded off is followed by the digits 6, 7, 8, or 9, increase the number to be rounded off by 1, e.g., 23.7 when rounded off to the nearest digit becomes 24.

b. When a number to be rounded off is followed by the digits 0, 1, 2, 3, or 4, retain the number to be rounded off, e.g., 23.4 when rounded off to the nearest digit becomes 23.

c. When a number to be rounded off is followed by the digit 5, retain the number to be rounded off if the number to be rounded off is even, but increase the number to be rounded off by one if the number to be rounded off is odd, e.g., 22.5 when rounded off to the nearest digit becomes 22, while 23.5 when rounded off to the nearest digit becomes 24.

C. REVIEW AND VALIDATION

1. REVIEW

a. Data review is the process of comparing results to all available information, such as sample preparation and QC sample data, to evaluate the validity of the results. It supports the contention that the data are

* reasonable (experience with similar situations, common sense), and
* capable of supporting a defensible decision.

b. The analyst and the unit supervisor (or designee) are responsible for reviewing the data relative to the following:

* Instrument calibration
* Standard preparation
* Method blanks and QC samples
* Raw data
* Calculations
* Transcription

2. VALIDATION

a. Data validation is the systematic procedure of reviewing data against a set of criteria to provide assurance of its validity before reporting the data. It is accomplished through routine examination of data collection, flow procedures, and QC sample results. It uses QC criteria to reject or accept specific data.

b. Validation includes the following:

* Dated and signed entries by analysts on the worksheets and log books used for all samples.

* Use of QC criteria to reject or accept specific data.

* Checking of LIMS data entry and reporting

c. Validation Guidelines include the following:

* Calibration requirements as defined in the method.

* Documented traceability of instrument and spiking standards.

* Documentation of methods used and QC applied.

* Maintenance performed on instruments.

* Documentation of sample preservation, transport, and storage.

* Review of QC sample data.

* Second analyst review before submitting the data to the Unit Supervisor (or designee) for approval, and wherever applicable, the QA Officer or Lab Director.

c. Data validation is performed, signed, and dated by the analyst, the unit supervisor (or designee), and where applicable, the laboratory manager.

D. REPORTING

1. The format and content of a data report depends on the project needs, client or customer requirements, or the specific government agency's established reporting format. The reporting format conforms to the requirements of the data user.
2. Data prepared for external release are checked and approved by the unit supervisor (or designee). The final report is signed by the unit supervisor and/or laboratory manager before distribution and may include the following:

    a. Sample ID used by the laboratory and the client (if available).

    b. Sample matrix type, description, and method number.

    c. The chemical/physical/biological parameters analyzed with the reported values and units of measurement.

    d. Minimum levels or reporting limits used for the analytes.

    e. Method detection limits of the analytes.

    f. Data for all parameters reported with consistent number of significant figures.

    f. Results of QC samples, if appropriate.

    g. Footnotes referenced to specific data, if required, to explain reported values.

    h. Discussion on non-compliance data.

    i. Report transmittal letter or memorandum identifying the person sending the report and the person(s) receiving the data.

3. Where required by the regulatory agency, the following reporting protocol will be used:

    a. Sample results greater than or equal to the minimum level (ML) will be reported as the measured concentration.

    b. Sample results less than the ML but greater than or equal to MDL will be reported as "Detected but not Quantified" or DNQ. The estimated

concentration of the sample will be reported next to DNQ with the words "Estimated Concentration" or "Est. Conc.".

c. Sample results less than MDL shall be reported as "Not Detected" or ND.

IX. INTERNAL QUALITY CONTROL CHECKS

The laboratories monitor data quality with internal QC checks. These checks are method-specific. Regulatory agencies have additional requirements for meeting acceptable data quality criteria. The QC checks are used to ensure that the data was generated correctly and is reliable.

The generation of data is checked by method-specific requirements described in the Standard Operating Procedure for each test. The reliability of data is addressed through Statistical Process Control (SPC). The unit supervisor has the responsibility to ensure that internal QC checks are documented and followed.

A. TYPICAL METHOD-SPECIFIC CHECKS

1. GENERAL APPLICATIONS

a. Sampling

* Procedures - Samples are collected according to each unit's sampling plan.

* Preparation - Each unit ensures that adequate sample volume/ weight and appropriate digestion/extraction techniques are used.

* Sample Storage - EPA guidelines on holding time are observed.

b. Blanks

Where applicable, blanks are included in each batch of samples collected and/or analyzed.

* Method (Reagent) blanks are analyzed with each batch of samples and are carried through the analytical scheme like a regular sample. Blank corrections are not applied to analytical data unless stated in the method.
* Calibration blanks are prepared with standards to create a calibration curve. The blank may provide the "zero point" for the curve.

* Travel/Field Blanks may be included during sample collection and analysis to comply with project or customer requirements. They undergo the complete analytical measurement process.

* Sample blanks are run only when necessary. They are used when certain sample characteristics, such as color or turbidity, may interfere with the analysis.

c. Initial Demonstration of Laboratory Proficiency

The analyst must demonstrate test proficiency after being trained. Test proficiency is also required prior to the use of a new method by the laboratory, with a newly purchased instrument, or after a prolonged instrument down-time requiring major repair.

d. Laboratory Control Sample

A laboratory control sample is included in each batch of samples. The sample is subjected to the same preparation/extraction procedure and measurement process as the routine samples. It is required to be prepared from a different source or another lot from the same source.

2. CHEMICAL ANALYSES

a. Calibration with Standards (Calibration Curve)

Initial calibration with the appropriate number of standards is performed and a calibration curve is generated at the start of an analysis. Sample results are usually quantified based on the initial calibration curve.

b. Continuing (verification) calibration may be required during the course of some analyses to verify instrument stability. The result must be within specifications before the analysis can proceed without recalibration.

c. System Monitoring (Surrogate) Compound Performance

A surrogate is a compound similar in chemical behavior to the analyte of interest, but it is not present in the sample. It is used as an indicator of sample-specific preparation efficiency and accuracy. The amount of surrogate recovered, expressed as Percent Recovery (% Recovery),

indicates possible preparation or extraction problems due to sample matrix.

d. Matrix-Spike Analysis

Samples are spiked based on method requirements. Matrix-spike analysis provides a measure of accuracy for the target analytes/compounds in the specific matrix of the sample. A known amount of analyte is added to one of the samples in the batch and is processed through the entire analytical procedure. The results are calculated as % Recovery.

e. Sample (Matrix) Duplicate/Matrix-Spike Duplicate

Where applicable, duplicate analysis is included in each sample batch at the frequency specified in the method. Duplicate analysis may be performed on a sample or a matrix-spiked sample. It provides a measure of analytical precision.

When the analyte concentration is suspected to be below the method detection limit, duplicate spikes are substituted for sample duplicates. The degree of agreement is expressed as Relative Percent Difference (RPD).

f. Minimum Level (ML)

In some cases the State Water Resources Control Board (SWRCB) requires the use of MLs as reporting limits for NPDES-related data. ML represents the lowest calibration standard used in analysis after the application of a method-specific factor. The SWRCB established ML requirements for some organic and inorganic analytes that need to be met for reporting effluent data. See Appendix E, Table 1 for this list.

g. Reporting Limit (RL)

Reporting Limit is the lowest concentration that can be analyzed within specified accuracy and precision. Generally it is established as 5 to 10 times the MDL. Reporting limit is verified by spiking a solid or liquid sample at the reporting level in duplicate.

h. Method Detection Limits (MDL)

Method detection limits have been established for most analyses. MDL is defined by EPA as "the minimum concentration of a substance that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte".

It is determined by multiplying the standard deviation, from a minimum of 7 replicate analyses, by the Student t-value at the desired 99% confidence level.

"The Procedure for the Determination of Method Detection Limits" - Revision 1.11 is published in CFR 40, Part 136, Appendix B. See Appendix B of this manual for the procedure. ELAP's "Procedure to Determine Method Detection Limits" is in Appendix C of this manual.

i. Mass Tuning Checks

EPA requires tuning of mass analyzers with specific standards to verify instrument sensitivity for every 12-hour working shift.

j. Internal Standard (Organic Analysis)/Post-Digestion Spike (Inorganic Analysis) Checks

This procedure is practiced when necessary to determine instrument measurement integrity and capability, and overcome matrix interference. Standard solutions of known concentrations are added after sample preparation or extraction and just before instrument measurement.

k. Method of Standard Addition (Interference Check) Analysis

The use of this procedure is recommended to verify the absence of matrix effects. Usually, when a new matrix type is to be analyzed, the amount of interference is determined. Its use is also suggested when the analyte of interest is at a very high or low level or suspected to be absent.

A sample is fortified with a series of known amounts of an analyte in increasing concentrations. The fortified samples are analyzed and may be calculated as % Recovery.

3. MICROBIOLOGICAL ANALYSES

Microbiological SOP's comply with the special quality control criteria regulated by California Department of Health Services that includes the following:

a. Sterility QC checks

b. Laboratory environment QC checks

c. Analyst intracomparison checks

d. Incubation time checks

e. Specific equipment temperature requirements (e.g., incubators, water baths, autoclaves)

f. Duplicate analyses on 10% of samples as a measure of analytical precision

g. Positive and negative bacterial control culture checks

h. Monitoring of deionized water for media preparation

4. BIOASSESSMENT ANALYSES (OCEAN ASSESSMENTS)

The Standard Operating Procedures for Ocean Assessments (Benthic Ecology/Taxonomy and Water Quality) follows protocols described in the most current edition of the Field Operations Manual for Marine Water Column, Benthic, and Trawl Monitoring in Southern California.

* Benthic and Trawling Program

  ** Sorting efficiency
  Ten percent of each sorter's samples are resorted.

  ** Identification efficiency
  Ten percent of each identifier's samples are reidentified.

* Water Quality Program

** Scheduled CTD instrument calibration and preventive maintenance

** Specific acclimation time and depth for instrument packages

** Data review for outlier removal

5. Toxicity Testing

The Standard Operating Procedures for Toxicity Testing include special guidelines provided by the California Department of Fish & Game and/or EPA.

* Specific test organisms, sample dilutions/volumes including controls

* Water quality test parameters

* Reference Toxicant/Parallel Test

* Statistical analyses parameters

* Precise requirements for specific organisms

* Acclimation period/temperature for test specimens

* Mortality rate of test control specimens

* Test Acceptability Criteria (TAC) for all methods.

*

B. STATISTICAL PROCESS CONTROL

Control charts/tables are established for precision (sample and spike duplicate analyses) as RPD and for accuracy (spiked analysis and laboratory control samples) as % Recovery. A minimum of 15 analyses is recommended before a statistical mean and standard deviation are calculated and a control chart is constructed.

A chart is constructed with the mean as the center line. Lines above or below the center line signify the warning or control limits, depending upon the confidence limits chosen. Warning limits are established at ± 2 standard deviations from the mean (95%

confidence interval). Upper limits correspond to ± 3 standard deviations from the mean (99% confidence interval).

Control charts are evaluated by the laboratory staff for trends. Statistical values based on laboratory results are also compared to published data established by EPA or other regulatory agencies, where applicable.

1. PRECISION CONTROL CHARTS/TABLES

   a. Laboratory Control (QC Check) Samples

      A standard of known concentration is analyzed with each batch of samples. The calculated results for the parameter of interest may be plotted and statistically evaluated. Statistical values for the mean and standard deviation may be compared to the manufacturer's certified values for precision of the control sample.

   b. Duplicate Analysis - Range (R) Charts

      Running duplicate analyses is one way of establishing control charts for precision. To construct the chart, the relative percent difference (RPD) is calculated and plotted with zero (no difference between control samples) as the expected value.

      Warning and control limits are calculated based on a distribution table of "Factors for Use in Duplicate Range Charts and Other Sets of Replicates ". Warning limits (95% confidence limit) are calculated by multiplying the mean range or mean RPD by a factor of 2.512. Upper limits (99% confidence limit) are calculated by multiplying the mean range or mean RPD by a factor of 3.267.

2. ACCURACY (BIAS) CONTROL CHARTS

   Laboratory control samples analyzed and calculated as % Recovery and spike sample results are used to measure accuracy or bias of a measurement.

   a. Laboratory Control (QC check) Samples

      When charts are plotted with % Recovery values, the results represent accuracy.

   b. Spiked Samples

The percent recovery is calculated and plotted on the chart. The lines on the spiked-sample chart correspond to mean recovery and the 95 and 99 percent confidence limits, which are calculated by multiplying the standard deviation by 2 and 3, respectively.

3. IMPORTANCE OF CONTROL CHARTS

Control charts are used to monitor the system in its day-to-day operations. These charts not only indicate serious immediate problems, but can also act as early warning signs by indicating potentially bad trends.

Out-of-control situations are investigated. An analytical system is considered out-of-control when any one of the following occurs:

a. One or more points are outside the control limits.

b. Two or more consecutive points are outside the warning limits.

c. Seven or more consecutive points are on the same side of the mean.

d. Cyclic (non-random) patterns are observed.

e. Six or more consecutive points are in the same direction.

X. PERFORMANCE AND SYSTEM AUDITS

An audit is a periodic check to ensure that the laboratory operates according to the policies and procedures described in the Quality Assurance Manual, complies with good laboratory practices, and meets the requirements of regulatory agencies. It may be either a system or performance audit.

A. SYSTEM AUDIT

A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff, and procedures in place to generate acceptable data. It is an on-site inspection of the laboratory's system of operations. It may be an internal or external audit. Internal inspections may be performed by quality assurance personnel. External audits are generally laboratory certification-related activities.

1. INTERNAL

   Periodically, the QA Officer (or designee) audits the laboratories and reports the results to the Division Manager, laboratory director, laboratory managers, and unit supervisors.

2. EXTERNAL

   EMD laboratories are site visited every two years by auditors from the Environmental Laboratory Accreditation Program (ELAP) of the California Department of Health Services (CA DHS). Accreditation is by scientific discipline or field-of-testing. Non-compliances with good laboratory practices are identified and reported as deficiencies and are subject to corrective action before accreditation is renewed.

B. PERFORMANCE AUDIT

A performance audit is a review to evaluate the laboratory's analytical activities as well as the data produced by analysts. It verifies the ability of the laboratory to correctly identify and quantitate compounds in unknown samples submitted by the auditing entity. The purpose of these audits is to determine the laboratory's capability to generate scientifically sound data.

1. INTERNAL

Periodically, the QA staff submits unknown samples to the laboratories. These samples are usually from the inventory of previous Performance Evaluation (PE) samples from EPA or National Institute of Science and Technology (NIST)-accredited providers. Analysis of these samples is also a corrective action requirement for external performance evaluation (PE) results evaluated as "unacceptable". EMD staff may also participate in inter-laboratory comparison studies.

2. EXTERNAL

All laboratory units at EMD participate in mandatory QA Performance Evaluation (PE) Study Programs.

a. Discharge Monitoring Report (DMR) QA Study is NPDES permit-related. It consists of chemistry and whole effluent toxicity analyses. The PE samples are obtained from commercial providers that have been accredited by the National Institute of Standards and Technology (NIST). These samples are submitted for analysis on an annual basis to confirm the analytical capabilities of the laboratories used by permittees. EMD, as the provider of laboratory services to plant operations, performs testings for all the parameters listed in the permit as defined in the DMR program.

b. Water Pollution (WP) Study Program serves a dual purpose. It satisfies EPA's wastewater testing laboratory requirements and meets one of ELAP's laboratory certification criteria. Test samples are analyzed for parameters listed under each field of testing on EMD certifications and are specified in the WP Program following certified procedures. A laboratory can participate in a WP Study twice a year.

c. Hazardous Waste (HW) Study is also an ELAP requirement for certification. Test samples of solid or non-aqueous matrices are analyzed for organic and inorganic constituents using all methods that are listed in EMD hazardous waste fields-of-testing certification. Like the WP Study, the laboratory can participate in up to two study series per year.

d. Microbiology Performance Evaluation (PE) Study, Drinking Water/Source Water-Wastewater Enumeration is also required for ELAP certification. Like all the other PE programs, the samples are acquired from NIST-

approved vendors and analyses are done for certified analytes and methods. The laboratory can also participate in up to two study series per year.

3. ELAP requires that analysis of Performance Evaluation (PE) samples must be rotated among all trained analysts.

XI. PREVENTIVE MAINTENANCE

Preventive maintenance is the process of taking positive actions to minimize the occurrence of equipment and instrument failure. It ensures that equipment and instruments are calibrated and operated with the reliability required for quality results.

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument. Designated laboratory personnel are trained in routine maintenance procedures for major instruments. When repairs are necessary, they are performed by either trained staff or trained service engineers through commercial service contracts.

Complete or abbreviated operating instructions are kept with each instrument. Each laboratory unit has detailed SOP's or manuals describing preventive maintenance procedures and frequencies for routine inspection, cleaning, testing, calibration, and/or standardization after instrument failure. Information documenting the preventive maintenance and repairs performed on each analytical instrument is also maintained. Documentation may include date, description of maintenance (scheduled maintenance or instrument malfunction/failure), actual findings, probable cause, name of person who performed the service, and calibration or standardization procedures that were performed with acceptable results or that were within performance criteria.

XII. ASSESSMENT OF PRECISION AND ACCURACY

Data quality may be assessed in terms of precision, accuracy, representativeness, comparability, and completeness. The first two are assessed in quantitative terms, while the latter three are generally expressed as qualitative characteristics. Moreover, the latter three are usually determined outside of the laboratory operations and with limited involvement of laboratory staff. These measures are not included in this section. The internal quality control measures (i.e., precision and accuracy) that are performed in the laboratory to evaluate data quality are described in this section. Precision and accuracy data are documented and assessed through quality control charts as discussed in Chapter VIII, INTERNAL QUALITY CONTROL CHECKS.

A. PRECISION

Precision is the agreement among a set of replicate measurements without knowledge of the true value. It is the degree to which a measurement is reproducible. Precision, expressed as Relative Percent Difference (RPD), is determined for each laboratory unit by analyzing a number of duplicate pairs, or matrix-spiked duplicate samples. It can also be expressed as Relative Standard Deviation (RSD) when replicates of the same sample are analyzed, or laboratory control samples are routinely analyzed.

B. ACCURACY

Accuracy is a measurement of how close the result is to the true value. Each laboratory unit establishes its accuracy of measurement by analyzing QC check samples (spiked samples, standard reference materials from a reliable source, etc.). The results of the QC samples are correlated to documented, certified values. Results of spiked samples are calculated as Percent Recovery. Actual Percent Recovery is compared to established reference data. The degree of closeness of the QC check sample contributes to the general assurance that the accuracy of the data is within acceptable limits.

XIII. CORRECTIVE ACTION

Laboratory events and data that fall outside established acceptance criteria may require investigation or corrective action. The corrective action implemented depends on the type of analysis, the extent of the error, and whether the error can be determined and corrected. The purpose of the corrective action is to resolve the problem and to restore the system to proper operation. Investigative steps and corrective actions implemented are documented.

A. CORRECTIVE ACTION PROCEDURES

1. The initial corrective action procedures may be handled at the bench level. The unit supervisor is immediately notified of the deviation. The analyst reviews the sample preparation or extraction procedure for possible errors and checks the instrument calibration, calibration and spike solutions, instrument sensitivity, etc.

2. If the error cannot be resolved by the analyst, the unit supervisor has the responsibility of resolving the problem with assistance, if needed, from the laboratory manager and/or the QA Officer.

3. The corrective action adopted may be determined by the analyst, the unit supervisor, the laboratory manager, the QA Officer, or through a consensus. If needed, the final decision for corrective action rests on the laboratory manager after consultation with the QA Officer.

4. The unit supervisor shall maintain an accurate and up-to-date record of corrective actions taken in the unit. A corrective action report form (Figure 1) is available for use.

5. The laboratory manager shall periodically review corrective action records and plan for system improvement by involving analysts, unit supervisors, and QA personnel.

B. GENERAL GUIDELINES FOR INITIATING A CORRECTIVE ACTION

1. Identify/define the problem.

2. Assign responsibility for investigating the problem.

3. Investigate and determine the causes.

4. Develop corrective action to eliminate the problem.

5. Measure the effectiveness of the corrective action.

6. Analyst, unit supervisor, laboratory manager, and the QA Manager meet to review and evaluate the process, if necessary.

7. Document the process by filling out the Corrective Action Report Form.

XIV. QUALITY ASSURANCE REPORTS

The QA Officer keeps the Division Manager, laboratory managers, laboratory directors, and unit supervisors abreast of quality assurance issues in laboratory operations through meetings, memos, and reports. Typical reports may include internal system audit findings with recommended corrective actions, annual assessment of the Quality Assurance Program, and summary of laboratory proficiency in external Performance Evaluation QA Study Programs. Additional information is also provided through ongoing discussions and dialogues with laboratory staff and the QA Officer.

The internal system audit report issued by the QA Officer is a summary of a specific laboratory operation. Included are a summary of deficiencies, corrective actions required, and recommendations. A copy of this report is sent from the QA Officer to the unit supervisor, laboratory manager, laboratory directors, and the Division Manager.

A division-wide annual assessment of the Quality Assurance Program is a progress report of the QA Program. This progress report is sent by the QA Officer to all unit supervisors, laboratory managers, laboratory director, and the Division Manager.

In addition, laboratory managers periodically perform operational system assessments based on information collected with the assistance of unit supervisors and staff. Such reports focus on systematic improvement of the quality of laboratory operations. These reports are submitted by laboratory managers to the Division Manager.

Laboratory proficiency in external Performance Evaluation QA Study Programs include test results, acceptable ranges, corrective actions required, if any, and recommendations. A copy of this report is sent from the QA Officer to the unit supervisors, laboratory managers, and laboratory directors.

XV. QUALITY ASSURANCE MANUAL MANAGEMENT

A. RESPONSIBILITY FOR MANUAL MANAGEMENT

The QA Officer has the responsibility for reviewing, updating, and distributing the QA Manual. The QA Manual is distributed to all EMD laboratory staff after each revision. A distribution list is maintained and the revised pages are archived by the QA Officer. The QA Manual is a dynamic document that will be reviewed annually to comply with ELAP certification regulation and revised to meet new conditions and requirements.

B. GENERAL PROCEDURE FOR REVISION

1. Any laboratory personnel may suggest revisions to the manual.

2. Suggestions, brought to the attention of the QA Officer, should include reasons for the revision and a proposal for the revised statements.

3. The QA Officer will review the merits of the proposed revision. If necessary, the QA Officer may discuss the proposal with the laboratory director or manager, unit supervisor, analysts, and any other personnel affected by the changes, including the person proposing the revision. As a result of the discussion, the suggestion may either be adopted, modified, or rejected.

4. When the manual is revised, the QA Officer will have the affected pages rewritten, reproduced, and distributed so that all copies of the QA Manual are revised.

APPENDIX A

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES*

PARAMETER NAME	CONTAINER [1]	PRESERVATION[2,3]	HOLDING TIME [4]	MINIMUM SAMPLE SIZE (mL)/g
Aquatic Toxicity Tests				
Toxicity, acute and chronic 10000 or 20000	P, G	Cool, 4ºC[16]	36 hours	
Bacterial Tests:				
Coliforms, Total and Fecal	P, G (Sterile)	Cool, 4ºC, 0.008% $Na_2S_2O_3$ [5]	6 hours	100
Fecal streptococci/Enterococci	P, G (Sterile)	Cool, 4ºC, 0.008% $Na_2S_2O_3$ [5]	6 hours	100
Salmonella	P, G (Sterile)	Cool, 4ºC	6 hours	500
Inorganic tests:				
Alkalinity	P, G	Cool, 4ºC	14 days	200
Ammonia	P, G	Cool, 4ºC, H_2SO_4 to pH < 2	28 days	500/10
Biochemical Oxygen Demand	P, G	Cool, 4ºC	48 hours	500
Boron	P, PFTE, or Quartz	HNO_3 to pH< 2	6 months	50/250
Biochemical Oxygen Demand, carb.	P, G	Cool, 4ºC	48 hours	500
Bromide	P, G	None required	28 days	100
Chemical Oxygen Demand	P, G	Cool, 4ºC, H_2SO_4 to pH < 2	28 days	250
Chloride	P, G	None required	28 days	50/250
Chlorine, Total Residual	P, G	None required	Analyze Immediately	100
Cyanide, total & amenable to chlorination	P, G	Cool, 4ºC, NaOH to pH > 12, 0.6g ascorbic acid [5]	14 days [6]	250/10
Fluoride	P	None required	28 days	50/250
Hardness	P, G	HNO_3 to pH< 2, H_2SO_4 to pH < 2	6 months	150
Hydrogen ion (pH)	P, G	None required	Analyze Immediately	50/50
Kjeldahl & organic nitrogen	P, G	Cool, 4ºC, H_2SO_4 to pH < 2	28 days	500/10
Nitrate	P, G	Cool, to 4ºC	48 hours	50/250
Nitrate-nitrite	P, G	Cool, to 4ºC, H_2SO_4 to pH < 2	28 days	50/250
Nitrite	P, G	Cool, to 4ºC	48 hours	50/250
Oil & Grease	G	Cool, to 4ºC, HCl or H_2SO_4 to pH < 2	28 days	1000/250
Organic carbon	P, G	Cool, to 4ºC, HCl or H_2SO_4 or H_3PO_4 to pH < 2	28 days	100/10
Orthophosphate	P, G	Filter immediately, Cool to 4ºC	48 hours	100
Oxygen, Dissolved Probe	Glass Bottle & Top	None required	Analyze Immediately	500
Oxygen, Winkler	Glass Bottle & Top	Fix on-site and store in dark	8 hours	500
Phenols	G only	Cool, 4ºC, H_2SO_4 to pH < 2,	28 days	500
Phosphorous, (Elemental)	G	Cool, 4ºC	48 hours	100
Phosphorus, Total	P, G	Cool, 4ºC, H_2SO_4 to pH < 2	28 days	100/10
Residue, Total	P, G	Cool, 4ºC	7 days	500
Residue, Filterable	P, G	Cool, 4ºC	7 days	500
Residue, Nonfilterable (TSS)	P, G	Cool, 4ºC	7 days	1000
Residue, Settleable	P, G	Cool, 4ºC	48 hours	2000
Residue, Volatile	P, G	Cool, 4ºC	7 days	500
Silica	P, PFTE, or Quartz	Cool, 4ºC	28 days	200
Specific Conductance	P, G	Cool, 4ºC	28 days	500
Sulfate	P, G	Cool, 4ºC	28 days	50/250
Sulfide	P, G	Cool, 4ºC add Zn acetate + NaOH to pH > 9	7 days	500
Sulfite	P, G	None required	Analyze immediately	250
Surfactants	P, G	Cool, 4ºC	48 hours	250
Temperature	P, G	None required	Analyze Immediately	
Turbidity	P, G	Cool, 4ºC	48 hours	125

PARAMETER NAME	CONTAINER [1]	PRESERVATION [2,3]	HOLDING TIME [4]	MIN. SAMPLE SIZE (mL)/g[@]
Metals				
Chromium VI	P, G	Cool, 4°C	24 hours	250
Hg[17]	P, G	HNO_3 to pH < 2,	28 days	1000/10
Al, Sb, As, Ba, Be, Cd, Ca, Cr(total)	P, G	HNO_3 to pH < 2,	6 months	1000/10
Co, Cu, Au, Ir, Fe, Pb, Mg, Mn, Mo, Ni, Os, Pd, Pt, K, Rh, Ru, Se, Ag, Na, Tl, Sn, Ti, V, Zn)[7]				
Organic Tests[8]				
Purgeable Halacarbons[†]	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	14 days	40/10
Purgeable Aromatic Hydrocarbons (Benzene, Ethylbenzene, Toluene)	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5], HCl to pH 2[9]	14 days	40/10
Acrolein and acrylonitrile	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5], adjust pH 4-5[10]	14 days	40/10
Nitrosoamines [‡,11,14], Nitroaromatics & Isophorone[‡,11], PAHs[‡,11]	G, teflon-lined cap	Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction	1000/50
Phenols[§11], Haloethers[§11], TCDD[§11]	G, teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction	1000/50
Benzidines [11] (Benzidine, 3,3' -Dichlorobenzidine)	G, teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction[13]	1000/50
Chlorinated Hydrocarbons[#,11], Phenoxy Acid, Herbicides, Phthalate Esters[#,11], PCBs[#,11], Acrylonitrile	G, teflon-lined cap	Cool, 4°C,	7 days until extraction, 40 days after extraction	1000/50
Pesticides (§136.3 Table ID)[11]	G, teflon-lined cap	Cool, 4°C, pH 5 – 9[15]	7 days until extraction, 40 days after extraction	1000/50
Radiological Tests				
Alpha, Beta, Radium	P, G	HNO_3 to pH < 2	6 months	2000

* 40 CFR Ch 1 (11-19-2002 ed), Part 136.3, Table II.

1 Polyethylene (P) or Glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic), except for samples collected for trace-level mercury (see footnote 17).

2 Sample preservation should be performed immediately upon collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed, except for samples collected for trace-level mercury (see footnote 17).

3 When any sample is to be shipped common carrier or sent through the US Mails, it must comply with the Dept. of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of this table, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric Acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric Acid (HNO_3) in water solutions at a concentration of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium Hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 1230 or less).

4 Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. (See footnote 17 for samples collected for trace level mercury). Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that for the specific types of samples under study, the analytes are stable for a longer time, and has received a variance from the Regional Administrator §136.3 (e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists that this is necessary to maintain sample

stability. See § 136.3(e) for details. The term " analyze immediately" usually means within 15 minutes or less of sample collection.

5 Should be used only in the presence of chlorine.

6 Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot is obtained. The sample is filtered and then NaOH is added to pH 12.

7 Samples should be filtered immediately on-site before adding preservative for dissolved metals, except for samples collected for trace-level mercury (see footnote 17)..

8 Guidance applies to samples to be analyzed by GC, LC, or CG/MS for specific compounds.

9 Samples receiving no pH adjustment must be analyzed within 7 days of sampling.

10 The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

11 When the extractable analysis of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

12. If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 0.2 to prevent rearrangement to benzidine.

13. Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

14. For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7 - 10 with NaOH within 24 hours of sampling.

15. The pH adjustment may be performed upon receipt at the laboratory and maybe omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

16. Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the 4C temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature can not be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

17. Samples collected for the determination of trace level mercury (100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. Samples collected for dissolved trace level mercury should be filtered in the laboratory. However, if circumstances prevent overnight shipment, samples should be filtered in a designated clean area in t he field in accordance with procedures given in Method 1669. Samples that have been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

† Benzyl chloride, Bromodichloromethane, Bromoform, Bromomethane, Carbon tetrachloride, Chlorobenzene, Chloroethane, 2-Chloroehtyl vinyl ether, Chloroform, Chloromethane, Dibromochloromethane, 1,2-Dichlorobenzene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, Dichlorodifluoromethane, 1,1-Dichloroethane, 1,2-Dichloroethane, 1,1-Dichloroethene, trans-1,2-Dichloroethene, 1,2-Dichloropropane, cis-1,3-Dichloropropene, trans-1,3-Dichloropropene, Epichlorohydrin, Methylene chloride, 1,1,2,2-Tetrachloroethane, Tetrachloroethene, 1,1,1-Trichloroethane, 1,1,2-Trichloroethane, Trichloroethene, Trichlorofluoromethane, Vinyl chloride.

‡ Nitrosamines: N-Nitrosodimethylamine, N-Nitroso-di-n-propylamine, N-Nitrosodiphenylamine.
Nitroaromatics and isophorone: 2,4-Dinitrotoluene, 2,6-Dinitrotoluene, Isophorone, Nitrobenzene.
PAHs (Polynuclear Aromatic Hydrocarbons): Acenaphthene, Acenaphthylene, Anthracene, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,I)perylene, Benzo(k)fluoranthene, Chrysene, Dibenzo(a,h)anthracene, Fluoranthene, Fluorene, Indeno(1,2,3-cd) pyrene, Naphthalene, Phenanthrene, Pyrene.

§ Phenols: 4-Chloro-3-methylphenol, 2-Chlorophenol, 2,4-Dichlorophenol, 2,4-Dimethylphenol, 2,3-Dinitrophenol, 2-Methyl-4,6-dinitrophenol, 2-Nitrophenol, 4-Nitrophenol, Pentachlorophenol, Phenol, 2,4,6-Trichlorophenol.
Haloethers: Bis(2-chloroethoxy) methane, Bis(2-chlroethyl) ether, 4-Bromophenylphenyl ether, 4-Chlorophenylphenyl ether, 2,2-Oxybis(1-chloropropane).

Chlorinated hydrocarbons: 2-Chloronaphthalene, 1,2-Dichlorobenzene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, Hexachlorobenzene, Hexachlorobutadiene, Hexachlrocyclopentadiene, Hexachloroethane, 1,2,4-Trichlorobenzene.
Phthalate esters: Benzyl butyl phthalate, Bis(2-ethylhexyl) phthalate, Diethyl phthalate, Dimethyl phthalate, Di-n-butyl phthalate, Di-n-octyl phthalate.

PCBs: PCB-1016, PCB-1221, PCB-1232, PCB-1242, PCB-1248, PCB-1254, PCB-1260.

APPENDIX B

PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT - REVISION 1.11, (40 CFR Ch.1 (7-1-93 Edition) Part 136, Appendix B]

A. SCOPE AND APPLICATION

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well-defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample. This MDL procedure is designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure is device or instrument independent.

B. PROCEDURE

1. MAKE AN ESTIMATE OF THE DETECTION LIMIT USING ONE OF THE FOLLOWING:

   a. The concentration value that corresponds to an instrument signal/noise in the range of *2.5* to *5*.

   b. The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

   c. The region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.

   d. Instrument limitations
   It is recognized that the experience of the analyst is important to this process. However, the analyst must include the limitations of the instrument being used in the initial estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference-free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each

analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

3a. If the MDL is to be determined in reagent (blank) water, prepare a laboratory standard (analyte in reagent water) at a concentration that is at least equal to or in the same concentration range as the estimated method detection limit. (Recommended between one and five times the estimated method detection limit.) Proceed to Step 4.

3b. If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 4.

If the measured level of the analyte is less than the estimated detection limit, add a known amount of the analyte to bring it to a concentration which is between one and five times the estimated detection limit.

If the measured level of analyte is greater than five times the estimated detection limit, there are two options:

1) Obtain another sample with a lower level of analyte in the same matrix, i if possible.

2) The sample may be used for determining the method detection limit if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL; hence, the MDL determined under these circumstances may not truly reflect method variances at lower analyte concentrations.

4a. Take a minimum of seven aliquots of the sample to be used to calculate the method detection limit and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

4b. It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding with 4a. This will prevent repeating this entire procedure when the costs of analyses are high and insure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL, even though the level of analyte is less than five times the calculated method detection limit. To insure that the estimate of the method detection limit is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each through the entire method, including blank measurements as describe above in 4a. Evaluate these data.

1) If these measurements indicate the sample is in a desirable range for determination of the MDL, take five additional aliquots and proceed. Use all seven measurements for calculation of the MDL.

2) If these measurements indicate the sample is not in correct range, re-estimate the MDL, obtain a new sample as in 3 and repeat either 4a or 4b.

5. Calculate the variance (S^2) and standard deviation (S) of the replicate measurements, as follows:

$$S^2 = 1/(n - 1) *[\sum X_i^2 - (\sum X_i)^2 /n]$$

$$S = (S^2)^{1/2}$$

Where: X_i; i = 1 to n, are the analytical results in the final method reporting units obtained from n sample aliquots and $\sum$ refers to the sum of the X values from i = 1 to n.

6a. Compute the MDL as follows:

$$MDL = t(n-1,1-a=.99)\ (S)$$

Where: MDL = the method detection limit.

$t_{(n-i,\ 1\ -\ \alpha\ =\ 0.99)}$ = the Student's t-value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table on page B-5.

S = standard deviation of the replicate analyses

6b. The 95% confidence interval estimates for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over the degrees of freedom distribution *(X^2/df).*

LCL = 0.64 MDL
UCL = 2.20 MDL

where: LCL and UCL are the lower and upper 95% confidence limits, respectively, based on seven aliquots.

7. OPTIONAL ITERATIVE PROCEDURE TO VERIFY THE REASONABLENESS OF THE ESTIMATE OF THE MDL AND SUBSEQUENT MDL DETERMINATIONS

a. If this is the initial attempt to compute MDL based on the estimate of MDL formulated in Step 1, take the MDL as calculated in Step 6, spike the matrix at this calculated MDL, and proceed through the procedure starting with Step 4.

b. If this is the second or later iteration of the MDL calculation, use S^2 from the current MDL calculation and S2 from the previous MDL calculation to compute the F-ratio. The F-ratio is calculated by substituting the larger S^2 into the numerator S^2_A and the other into the denominator S^2_B. The computed F-ratio is then compared with the F-ratio found in the table which is *3.05* as follows:

If $S^2_A / S^2_B < 3.05$, then compute the pooled standard deviation by the following equation:

$$S_{pooled} = \sqrt{\left[\frac{6S_A^2 + 6S_B^2}{12}\right]}$$

If $S^2_A / S^2_B > 3.05$, re-spike at the most recent calculated MDL and process the samples through the procedure starting with Step 4. If the most recent calculated MDL does not permit qualitative identification when samples are spiked at that level, report the MDL as a concentration between the current and previous MDL which permits qualitative identification.

Use the S_{pooled} as calculated in 7b to compute the final MDL according to the

following equation MDL = 2.681 (S_{pooled})

where: 2.681 is equal to $t_{(12, 1 - \alpha)} = 0.99$

d. The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared distribution.

LCL = 0.72
UCL = 1.65

Where LCL and UCL are the lower and upper 95% confidence limits, respectively, based on 14 aliquots.

TABLE OF STUDENT'S t VALUES AT THE 99% CONFIDENCE LEVEL

NUMBER OF REPLICATES	Degrees of freedom (n-1)	t(n-1, 0.99)
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.588
26	25	2.485
31	30	2.457
61	60	2.390
∞	∞	2.326

C. OUTLIERS IN DATA SET

An outlier in a data set can result from analyst error, malfunction of an instrument, inconsistent use of the SOP, unusual losses in sample preparation, and/or contamination. If outliers occur too often, this could indicate that there may be deficiencies in the application or the analytical method used. These can be corrected to improve the measurement process. [One should always search diligently for causes of outliers before data are rejected. Whenever an outlier is suspected, the analyst should look for a reason for its occurrence]. This will help the laboratory to improve quality control procedures.

EMD will apply Grubbs Test for determination of outliers and data rejection while determining MDL and also when multiple measurements are done to achieve confidence on an analysis. If more than seven aliquots are prepared and analyzed, results from all aliquots must be used in the MDL determination unless the presence of an outlier is determined.

D. GRUBBS TEST FOR OUTLYING OBSERVATIONS:

This test is useful for making statistical decisions on the identification of outliers. The procedure for using it is as follows:

1. Arrange the data in the order of increasing numerical value.

   $X_1 < X_2 < X_3 < \ldots\ldots < X_{n-1} < X_n$

2. Decide whether the smallest, X_1, or the largest, X_n, is suspected to be an outlier.

3. Calculate the standard deviation (s) and Mean ($\bar{X}$) of the data set using all data.

4. Calculate appropriate value of T as follows:

   $$T_1 = (\bar{X} - X_1) / s \qquad \text{or} \qquad T_n = (X_n - \bar{X})/s$$

5. Choose the larger of T_1 and T_n value.

6. Refer to the Table of Critical Values for T in Grubbs test.

7. If either T_1 or T_n value is higher than the tabulated T value, the tested data to be an outlier and hence is rejected.

TABLE: CRITICAL VALUES FOR T IN THE GRUBBS TEST

Number of Data Points (n)	Critical Values of T
7	2.020
8	2.126
9	2.215
10	2.290
11	2.355
12	2.412
13	2.462
14	2.507
15	2.549

E. REPORTING

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method allows options that affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with the MDL value. Report the mean analyte level with the MDL and indicate if the MDL procedure was iterated. If a laboratory standard or a sample that contained a known amount of analyte was used for this determination, also report the mean recovery. If the level of analyte in the sample was below the determined MDL or exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

APPENDIX C

PROCEDURE TO DETERMINE METHOD DETECTION LIMIT (MDL) (PROVIDED BY ELAP)

A. DEFINITION

The Method Detection Limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing analyte.

B. DETERMINATION

1. Make an estimate of the detection limit using one of the following methods:

   a. A concentration value that corresponds to an instrument signal/noise ratio in the range of *2.5* to 5.0.

   b. A low concentration value showing a break in the slope of the calibration curve.

2. Prepare reagent (blank) water that is as free of analyte as possible.

3a. If the MDL is to be determined in reagent water (blank), prepare a laboratory standard (analyte in reagent water) at a concentration which is between 1 and *5* times the estimated MDL. Proceed to step 4.

3b. If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of 1 to *5* times the estimated MDL, proceed to step

4. Take a minimum of seven aliquots and process each through the entire analytical method.

5. Calculate the Standard Deviation of the replicate measurements.

$$S^2 = 1/(n - 1) *[\sum X_i{}^2 – (\sum X_i)^2/n]$$

$$S = (S^2)^{1/2}$$

Where: Xi (i = 1 to 7) are the analytical results from the seven sample aliquots.

$$MDL = S \times (3.143)$$

6. A lower concentration of the analyte will not result in a significantly lower calculated MDL.

Example of the MDL Calculation:

Run #1	X_i	X_i^2
True Value = 1.5 mg/L		
1.	1.2310	1.515
2.	1.3620	1.855
3.	1.5192	2.292
4.	1.5776	2.488
5.	1.6621	2.762
6.	1.7226	2.962
7.	1.7383	3.021
	$\sum X_i = 10.807$	$\sum X_i^2 = 16.895$

$(\sum X_i)^2 = 116.79$
$(\sum X_i)^2/n = 16.68$

$S^2 = 0.215/6$

$S = 0.189$

MDL = (3.143) x S
MDL = 0.594 mg/L
MDL = 0.6 mg/L

Reference Glaser, J.A., Forest, D.L., McKee, G.d. Quave, S.A. and Budde, W.L., "Trace Analysis for Waste Waters," Environmental Science and Technology, 15, 1426, 1981.

APPENDIX D

STANDARD OPERATING PROCEDURES CONTENT AND FORMAT

The laboratories of EMD develop and maintain written documents that clearly and completely delineate the exact steps followed in performing every test method and procedure used in the laboratory. EMD managers, laboratory directors, the QA/QC Officer, and the Division Manager in conjunction with laboratory staff develop and maintain Standard Operation Procedures (SOPs)

A reference to a book of standard analytical test methods, guides, and practices published by a reputable organization such as the EPA, Standard Methods, ASTM, etc. is not sufficient to guarantee acceptable results. A significant part of the variability or results generated by different laboratories analyzing the same samples and citing the same general reference is due to differences in the way the analytical test methods and procedures are actually performed in each laboratory. These differences are often caused by the slight changes or adjustments allowed by the general reference, but that can affect the final results. Therefore, the importance of SOPs lies in their impact on maintaining uniformity of test method performance and the utility of data generated by our laboratories.

SOPs contain the step-by-step description of how every test method and procedure is performed in the laboratory to help guarantee uniform performance among different analysts using them. Those SOPs become the cornerstone of a laboratory's (EMD's) credibility.

EMD SOPs are written, practiced, and maintained within the technical allowances of the mandated procedures (e.g., EPA Method 624) on which they are based. The final version of the SOP is annotated with an effective date, revision number, and total number of pages. The effective date is the date when it was first approved to be used to produce actual data. The final version and all subsequent revisions are approved and signed by the laboratory manager, the quality assurance officer or their designees before being distributed to all analysts for bench use.

A master copy of all of EMD's SOPs are kept in a database by the QA/QC Unit. Therefore, whenever a procedure or test method is modified, a Revised Version will be issued by the QA/QC Unit and the master copy will be updated and archived along with the original version.

A very important factor in the use of SOPs is that the actual performance of each analyst while performing a test method or procedure is audited by the responsible supervisor or the quality assurance officer on a periodic basis, ideally not less than once per year.

The format to be used for EMD's SOPs is as follows:

Format for EMD SOPs:

1) Title

Name of the Laboratory
Name of the Test
Method Number
EMD SOP #
Revision Number and Effective Date
Name and Signature of the Laboratory Manager
Name and Signature of the Quality Assurance Manager
Total # of Pages and Pages Revised

EMD follows an SOP numbering system based on each unit, as follows:

1000 – Microbiology
2000 – Toxicity and Ocean Assessments
    2100 – Toxicity
    2200 – Ocean Assessments
3000 – LAG, DCTWRP, and TITP
    3100 – LAG
    3200 – DCTWRP
    3300 – TITP
4000 – Wet Chemistry
5000 – HTP Process Control
6000 – Metals
7000 – Organics
    7100 – Volatiles
    7200 – Semi-Volatiles
    7300 – Air
8000 – Sample Receiving

Normally, each unit assigns SOP numbers in chronological order followed by letters to reflect revisions numbers.

2) Scope and Application

Type of sample matrices and the analytical range to which this method can be applied

3) Summary of the Test Method

A short description highlighting the definitive chemical and procedural elements of the test method.

4) Interferences

A general overview of the kinds of matrices that can cause unacceptable performance and the general mechanisms for compensating for them.

5) Sample Collection, Preservation, and Handling

A description of the proper sampling procedure needed for this SOP. The description of the procedure must include: the type of sample container needed, types of preservatives to add, conditions under which the sample is to be transported, etc. Itemize any concerns relating to proper handling of samples after they are received for analysis. Specifically indicate holding times, storage procedures, and preservation procedures (applied at or prior to receipt).

6) Apparatus

Instruments and labware used in the method or procedure.

7) Chemicals and Reagents

A listing of required chemicals, purity, and grade; instructions for reagent makeup; standardizing, storing, and disposing of reagents; and reagent and chemical shelf life.

8) Safety

Identify at each point in the test method where safety precautions are to be observed.

9) Procedure

A detailed description of each step considered essential to the reproducibility and accuracy of the test method as actually carried out in the laboratory. Include calibration procedures. Re-analysis sample preparation steps should be specified.

10) Calculation

A description of the mathematical steps required to complete the analysis. Include sample calculations and the number of significant figures to report. (Also include a sample of the analytical bench form.)

11) Data Management

Specific instruction on how and where data should be reported, and on what, how, and where data should be stored.

12) Quality Assurance and Quality Control

Itemize desirable and mandatory quality assurance procedures specific to this test method, especially equipment and reagent checks, calibrations, and other system checks that should be done routinely. This section should be referenced in the procedure section.

Specify statistical quality control parameters: batch size, reference materials, QC frequencies, and data handling. This section should be referenced in the procedure section.

13) Lowest Reporting Level

The concentration below which all results are reported as "less than", based on the specified procedure and sample size.

14) Precision and Bias Statement

A tabulation of these statistics as determined using this test method in your laboratory, by matrix and concentration level.

15) References

A listing of published documents supporting the specifics of this test method.

16) Appendices

Copies of documents, tables, or graphs that would be useful to have appended to the test method.

The standardized EMD cover page format is presented below:

City of Los Angeles
ENVIRONMENTAL MONITORING DIVISION
Name of Laboratory Unit
STANDARD OPERATING PROCEDURE for

Name of Analyte

(EPA Method XXX.X)SOP# XXXX

Effective Date:
_____
Version No.: _____
Total Number of pages: _____
Pages Revised: _____

APPROVAL:

Laboratory Manager:
Signature: _____
Quality Assurance Manager

Signature: _____
Quality Assurance Officer:
Signature: _____

TABLE OF CONTENTS

Page

APPENDIX E

TERMINOLOGY

Accreditation - A formal process by which a laboratory is evaluated by an authoritative body, with respect to established criteria, for its competence to perform a specified kind(s) of measurement

Accreditation Criteria - For laboratory accreditation, a set of requirements used by an accrediting body that a testing laboratory must meet to be accredited.

Accuracy - The degree of agreement of a measured value with the true or expected value of the quantity of concern. It is the closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy is a measure of the combination of a random component and of a common systematic error (or bias) component.

Analyte - The specific component measured in a chemical analysis; also called analyte.

Assessment (of a laboratory) - The on-site examination of a laboratory's compliance with accreditation criteria.

Assessor (of a laboratory) - An individual who carries out some or all functions related to laboratory assessment.

Assignable cause - A cause believed to be responsible for an identifiable change of precision or accuracy of a measurement process.

Audit - A systematic and independent examination to determine whether quality activities and related results comply with planned arrangements and whether these arrangements are implemented effectively and are suitable to achieve objectives.

Batch - Environmental samples, which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. Preparation Batch is composed of one to 20 environmental samples of the same NELAP- defined matrix, meeting the "Batch" criteria and with a maximum of 24 hours between the start of processing of the first and last sample in the batch. Analytical Batch is composed of prepared environmental samples (extracts, digestates, or concentrates), which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Bias - A systematic error inherent in a method or caused by some artifact or idiosyncracy of the measurement system. Temperature effects and extraction inefficiencies are examples of systematic error. Blanks, contamination, mechanical losses, and calibration errors are some artifacts of a measurement system. Or it is a deviation due to matrix effects of the measured value from a known spiked sample. It may be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount, of contaminant spiked into a sample.

Blank - A synthetic sample that does not contain the analyte of interest. Or, it is a material made up to contain all the components of a product other than the analyte. It is the measured value obtained when a specified component of a sample is not present during the measurement.

Blind Sample - A sample submitted for analysis whose composition is known to the submitter, but unknown to the analyst. It is a way to test the proficiency of a measurement process.

Calibration - Comparison of a measurement standard or instrument with another standard or instrument to report or eliminate by adjustment any variation (deviation) in the accuracy of the item being compared.

Certification - A process by which a third party gives written assurance (certificate of conformity) that a product, process, or service conforms to specified requirements.

Central Line - The long-term expected value of a variable displayed on a control chart.

Certified Reference Material (CRM) - A reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other document that a certifying body issues.

Certified Value - The value that appears in a certificate as the best estimate of the value for a property of a reference material.

Chain-of-Custody - A detailed written and legal document of all transactions in which the samples are transferred from the custody of one individual to another.

Check Standard - In physical calibration, an artifact measured periodically, the results of which typically are plotted on a control chart to evaluate the measurement process.

Coefficient of Variation - The standard deviation divided by the value of the parameter measured.

Composite Sample - A sample composed of two or more increments selected to represent a population of interest. A mixture of grab samples collected at the same sampling point at different times.

Confidence Interval - That range of values, calculated from an estimate of the mean and the standard deviation, which is expected to include the population mean with a stated level of confidence. Confidence intervals in the same context may also be calculated for standard deviations, lines, slopes, and points.

Control Chart - A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a

state of statistical control.

Control Limit - The limits shown on a control chart beyond which it is highly improbable that a point could lie while the system remains in a state of statistical control.

Control Sample - A material of known composition analyzed with test samples to monitor the performance of the system.

Corrective Action - Action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence. It may involve changes, such as in procedures and systems, to achieve quality improvement.

Data Quality Objectives (DQO's) - A statement of the overall level of uncertainty that a decision-maker is willing to accept in results derived from environmental data. It is usually expressed in terms of objectives for precision, bias, and detection limit.

Deficiency - A departure from, or noncompliance with, specified accreditation criteria. It describes a situation which exists, but does not comply with requirements.

Detection Limit - The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

Double Blind - A sample whose composition is known to the submitter, but neither its composition nor its identification as a check sample is known to the analyst.

Duplicate Measurement - A second measurement made on the same (or identical) sample of material to assist in the evaluation of measurement variance.

Duplicate Sample - A second sample randomly selected from a population of interest to assist in the evaluation of sample variance.

Environmental Laboratory Accreditation Program (ELAP) - The California Department of Health Services' accrediting agency for laboratories that perform analyses of drinking water, wastewater, hazardous waste, contaminated soils or sediments, or any combination of these for regulatory purposes.

Error - Difference between the true or expected value and the measured value of a quantity or parameter.

Field Blank - independent samples that are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. Duplicates are useful in documenting the precision of the sampling process.

Grab Sample - A sample collected at a specific time and place.

Good Laboratory Practice (GLP) - An acceptable way to perform some basic operation or activity in a laboratory that is known or believed to influence the quality of its outputs. GLP's ordinarily are independent of the measurement techniques used.

Homogeneity - The degree to which a property or substance is randomly distributed throughout a material.

Implementation - A process for introducing a new system into the mainstream of a company or laboratory and the individual work places and jobs.

Informational Value - Value of a property not certified, but provided because it is believed to be reliable and to provide information important to the certified material.

Intercalibration - The process, procedures, and activities used to ensure that the several laboratories engaged in a monitoring program can produce comparable data. When comparable data outputs are achieved and this situation is maintained, the laboratories are said to be intercalibrated.

Laboratory Control Sample - A known matrix spiked with compound(s) representative of the target analytes. It is used to document laboratory performance.

Laboratory Information Management System (LIMS) - A database tailored to the analytical laboratory so that it can handle data generated by the analysis of samples and integrates sample information with results obtained from analytical instruments, reducing administrative tasks and increasing the production of final reports. It provides tracking, database query, integrated graphics, data archiving, audit trails, and report formatting.

Limit of Linearity (LOL) - The upper limit of concentration or amount of substance for which incremental additions produce constant increments of response.

Matrix- The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement definitions, the following matrix distinctions shall be used:

- Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluent, industrial waste, and Toxicity Characteristic Leaching Procedure (TCLP) or other extracts.
- Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.
- Saline/Estuarine: An aqueous sample from an ocean or estuary, or other salt water sources such as the Great Salt Lake.

- Non-aqueous Liquid: Any organic liquid with <15% settleable solids.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
- Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.
- Air: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

Matrix Duplicate - An intralaboratory split sample that is used to document the precision of a method in a given sample matrix.

Matrix Spike - An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

Matrix Spike Duplicate - Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

Method - A series of measurement techniques and the order in which they are used.

Method Blank - An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

Method Detection Limit - The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero; determined by analysis of a sample in a given matrix type containing the analyte.

Method Validation - A process of determining and evaluating the attributes or figures of merit of a method.

Minimum Level (ML) – The concentration at which the entire analytical system must give a recognizable signal and acceptable calibration point. The ML is the concentration in a sample that is equivalent to the concentration of the lowest calibration standard analyzed by a specific analytical procedure, assuming that all the method-specific sample weights, volume and processing steps have been followed.

Organic-Free Water - Water in which an interferent is not observed at the method detection limit of the compounds of interest.

Outlier - An individual measurement, from a set of replicates, that differs so markedly from the other replicates as to raise the question of measurement error.

Performance Audit - A process to evaluate the proficiency of an analyst or laboratory by evaluation of the results obtained on known test materials.

Population - A generic term denoting any finite or infinite collection of individual things, objects, or events; in the broadest concept, an aggregate determined by some property that distinguishes things that do not belong.

Precision - The agreement among a set of replicate measurements without knowledge of the true value. Usually expressed by standard deviation, coefficient of variation, or range. It is a degree of mutual agreement characteristic of independent measurements as a result of repeated application of the process under specified conditions.

Preventive Maintenance - Positive action taken to limit failure of equipment or eliminate the causes of a potential instrument/equipment malfunction in order to ensure fewer and shorter equipment breakdowns and increase measurement system reliability.

Primary Standard - A substance having one or more values that can be accepted (within specific limits) without question for use in standardizing or measuring the same property in another material.

Procedure - A set of systematic instructions for using a method of measurement or sampling or of the steps or operations associated with them.

Proficiency Testing - Determination of the laboratory calibration or testing performance by means of interlaboratory comparisons.

Project - Single or multiple data collection activities that are related through the same planning sequence.

Protocol - A procedure specified to be used when performing a measurement or related operation as a condition to obtain results that could be acceptable to the specifier.

Protocol for a Specific Purpose (PSP) - Detailed instructions for the performance of all aspects of a specific measurement program; sometimes referred to as the project QA plan.

Quality - An estimation of acceptability or suitability for a given purpose of an object, item, or tangible or intangible thing. It is the totality of characteristics of an entity that bears on its ability to satisfy stated and implied needs.

Quality Assessment - The overall system of activities whose purpose is to provide assurance that the quality control activities are done effectively. It involves a continuing evaluation of the performance of the production system and the quality of the products produced.

Quality Assurance - A system of activities whose purpose is to provide to the producer or user

of a product or service the assurance that it meets defined standards of quality. It is all the planned and systematic actions implemented within the quality system, and demonstrated as needed, to provide adequate confidence that an entity will fulfill requirements for quality. It consists of two separate, but related activities, quality control and quality assessment.

Quality Assurance Plan - A document setting out the specific quality practices, resources and sequence of activities relevant to a particular product, project, or contract. It usually makes reference to the parts of the quality assurance manual applicable to a specific case.

Quality Assurance Project Plan - An orderly assemblage of detailed procedures designed to produce data of sufficient quality to meet the data quality objectives for a specific data collection activity.

Quality Assurance Program - The decisions and actions required to attain and maintain the quality of performance and output that meet quality standards.

Quality Assurance Program Manual - A document stating the quality assurance policy and describing the quality system of an organization or a laboratory. It informs laboratory management and staff, regulatory agencies, and data users about the program.

Quality Audit - A systematic and independent examination to determine whether quality activities and related results comply with planned arrangements and whether these arrangements are implemented effectively and are suitable to achieve objectives. It is carried out by staff not having direct responsibility in the areas being audited but, preferably, working in cooperation with the relevant personnel.

Quality Control - The overall system of activities whose purpose is to control the quality of a product or service so that it meets the needs of users. The aim is to provide quality that is satisfactory, adequate, dependable, and economical. It involves operational techniques and activities aimed both at monitoring a process and at eliminating causes of unsatisfactory performance in order to achieve economic effectiveness. Some quality control and quality assurance actions are interrelated.

Quality Policy - Overall intentions and direction of an organization with regard to quality, as formally expressed by top management.

Quality System - Comprehensive organizational structure, procedures, processes, and resources needed to implement quality management in order to meet quality objectives.

Random Sample - A sample selected from a population by a randomization process.

Reference Material - A substance of known composition, usable as an analytical standard, for equipment calibration or for assessment of accuracy of a test procedure.

Reference Method - A method which has been specified as capable, by virtue of recognized accuracy, of providing primary reference data.

Relative Standard Deviation - The coefficient of variation expressed as a percentage.

Replicate - A counterpart of another, usually referring to an analytical sample or a measurement. It is the general term for one of several identical samples or measurements; whereas duplicate is the specific term for two samples or measurements.

R**eproducibility** - The demonstration that a method can be performed by multiple operators in multiple laboratories.

Routine Method - A method used in recurring analytical problems.

Sample - A portion of a population or lot. It may consist of an individual or groups of individuals. It may refer to objects, materials, or measurements, conceivably as part of a larger group that could have been considered.

Secondary Standard - A substance for which one or more values has been determined by comparison with a primary standard.

Selectivity - The ability of methodology or instrumentation to respond to a desired substance or constituent and not to others.

Sensitivity - The ability of a procedure or instrument to discriminate between samples having different concentrations or containing different amounts of an analyte.

Significant Figures - Digits known to be accurate plus one that is uncertain. It indicates the accuracy limitations of an analytical measurement due to the chemical nature of the procedure, instrumentation, and/or methodology.

Special Cause - A cause of variance or bias that is external (not inherent) to the measurement system.

Specificity - The ability of a procedure or instrument **to** respond only to the analyte, without interference from other materials that may or may not be present.

Split Sample - A replicate portion or subsample of a total sample obtained in such a manner that it is not believed to differ significantly from other portions of the same sample. It is an aliquot of sample taken from the same container and analyzed independently. These are usually taken after mixing or compositing and are used to document intra- or interlaboratory precision.

Standard - A substance or material with properties believed to be known with sufficient accuracy to permit its use to evaluate the same property of another. In chemical measurements, it often describes a solution or substance commonly prepared by the analyst to establish a calibration curve or the analytical response function of an instrument.

Standard Addition - A method in which small increments of the substance under measurement are added to the sample being tested to establish a response function, or to determine by extrapolation the amount of the constituent originally present in the test sample. It is the practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences.

Standard Curve - A plot of concentrations of known analyte standards versus the instrument response to the analyte.

Standardization - The process whereby the value of a potential standard is fixed by measurement with respect to a standard(s) of known value.

Standard Method - A method (or procedure) of test developed by a standards writing organization, based on consensus opinion or other criteria, and often evaluated for its reliability by a collaborative testing procedure.

Standard Operations Procedure (SOP) - A procedure adopted for repetitive use when performing a specific measurement or sampling operation. It may be a standard method or one developed by the user.

Standard Reference Material - A reference material distributed and certified by the National Institute for Standards Technology (NIST), formerly the National Bureau of Standards (NBS).

Statistical Process Control (SPC) - The application of statistical techniques for measuring and analyzing the variation in processes.

Subsample - A portion taken from a sample. A laboratory sample may be a subsample of a gross sample; similarly, a test portion may be a subsample of a laboratory sample.

Surrogate - An organic compound that is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but that is not present in the sample.

Technique - A physical or chemical operation (or instrument) applied within a test procedure, such as titration, high-pressure liquid chromatography, or atomic absorption spectroscopy, to determine the composition (analysis) of materials.

Tolerance Interval - Range of values calculated from an estimate of the mean and standard deviation within which a specified percentage of individual values of a population (measurements or sample) are expected to lie with a stated level of confidence.

Traceability - The ability to trace the origin of materials, parts, calculation process, data, and/or primary standards by means of recorded identifications or documentation. It is also the ability to trace the source of uncertainty of a measurement or a measured value.

Training - Formal or informal instruction designed to provide competence of a specific nature.

Uncertainty - The range of values within which the true value is estimated to lie. It is a best estimate of possible inaccuracy due to both random and systematic error.

Validation - The process by which a sample, measurement method, or a piece of data is deemed useful for a specified purpose. It is the process of assuring that a procedure or technique provides acceptable results for a particular purpose.

Variance - The value approached by the average of the sum of the squares of deviations of individual measurements from the limiting mean.

Warning Limits - The limits shown on a control chart within which most of the test results are expected to lie (within a *95%* probability) while most of the system remains in a state of statistical control.

TABLE 1

SWRCB MINIMUM LEVELS IN PPB (ug/L)

Table 1a - VOLATILE SUBSTANCES*	**GC**	**GCMS**
1,1 Dichloroethane	0.5	1
1,1 Dichloroethene	0.5	2
1,1,1 Trichloroethane	0.5	2
1,1,2 Trichloroethane	0.5	2
1,1,2,2 Tetrachloroethane	0.5	1
1,2 Dichlorobenzene (volatile)	0.5	2
1,2 Dichloroethane	0.5	2
1,2 Dichloropropane	0.5	1
1,3 Dichlorobenzene (volatile)	0.5	2
1,3 Dichloropropene (volatile)	0.5	2
1,4 Dichlorobenzene (volatile)	0.5	2
Acrolein	2.0	5
Acrylonitrile	2.0	2
Benzene	0.5	2
Bromoform	0.5	2
Bromomethane	1.0	2
Carbon Tetrachloride	0.5	2
Chlorobenzene	0.5	2
Chlorodibromo-methane	0.5	2
Chloroethane	0.5	2
Chloroform	0.5	2
Chloromethane	0.5	2
Dichlorobromo-methane	0.5	2
Dichloromethane	0.5	2
Ethylbenzene	0.5	2
Tetrachloroethene	0.5	2
Toluene	0.5	2
trans-1,2 Dichloroethylene	0.5	1
Trichloroethene	0.5	2
Vinyl Chloride	0.5	2

*The normal method-specific factor for these substances is 1, therefore, the lowest standard concentration in the calibration curve is equal to the above ML value for each substance.

Table 1b - SEMI-VOLATILE SUBSTANCES*	**GC**	**GCMS**	**LC**	**COLOR**
1,2 Benzanthracene	10	5		
1,2 Dichlorobenzene (semivolatile)	2	2		
1,2 Diphenylhydrazine		1		
1,2,4 Trichlorobenzene	1	5		
1,3 Dichlorobenzene (semivolatile)	2	1		
1,4 Dichlorobenzene (semivolatile)	2	1		
2 Chlorophenol	2	5		
2,4 Dichlorophenol	1	5		
2,4 Dimethylphenol	1	2		
2,4 Dinitrophenol	5	5		
2,4 Dinitrotoluene	10	5		
2,4,6 Trichlorophenol	10	10		
2,6 Dinitrotoluene		5		
2- Nitrophenol		10		
2-Chloroethyl vinyl ether	1	1		
2-Chloronaphthalene		10		
3,3’ Dichlorobenzidine		5		
3,4 Benzofluoranthene		10	10	
4 Chloro-3-methylphenol	5	1		
4,6 Dinitro-2-methylphenol	10	5		
4- Nitrophenol	5	10		
4-Bromophenyl phenyl ether	10	5		
4-Chlorophenyl phenyl ether		5		
Acenaphthene	1	1	0.5	
Acenaphthylene		10	0.2	
Anthracene		10	2	
Benzidine		5		
Benzo(a) pyrene(3,4 Benzopyrene)		10	2	
Benzo(g,h,i)perylene		5	0.1	
Benzo(k)fluoranthene		10	2	
bis 2-(1-Chloroethoxyl) methane		5		
bis(2-chloroethyl) ether	10	1		
bis(2-Chloroisopropyl) ether	10	2		
bis(2-Ethylhexyl) phthalate	10	5		
Butyl benzyl phthalate	10	10		
Chrysene		10	5	

* With the exception of phenol by colorimetric technique, the normal method-specific factor for these substances is 1000, therefore, the lowest standard concentration in the calibration curve is equal to the above ML value for each substance multiplied by 1000.

Table Ib - SEMI-VOLATILE SUBSTANCES*	**GC**	**GCMS**	**LC**	**COLOR**
di-n-Butyl phthalate		10		
di-n-Octyl phthalate		10		
Dibenzo(a,h)-anthracene		10	0.1	
Diethyl phthalate	10	2		
Dimethyl phthalate	10	2		
Fluoranthene	10	1	0.05	
Fluorene		10	0.1	
Hexachloro-cyclopentadiene	5	5		
Hexachlorobenzene	5	1		
Hexachlorobutadiene	5	1		
Hexachloroethane	5	1		
Indeno(1,2,3,cd)-pyrene		10	0.05	
Isophorone	10	1		
N-Nitroso diphenyl amine	10	1		
N-Nitroso-dimethyl amine	10	5		
N-Nitroso -di n-propyl amine	10	5		
Naphthalene	10	1	0.2	
Nitrobenzene	10	1		
Pentachlorophenol	1	5		
Phenanthrene		5	0.05	
Phenol **	1	1		50
Pyrene		10	0.05	

* With the exception of phenol by colorimetric technique, the normal method-specific factor for these substances is 1000, therefore, the lowest standard concentration in the calibration curve is equal to the above ML value for each substance multiplied by 1000.
** Phenol by colorimetric technique has a factor of 1.

Table 1c – INORGANICS***

	FAA	GFAA	ICP	ICPMS	SPGFAA	HYDRIDE	CVAA	COLOR	DCP
Antimony	10	5	50	0.5	5	0.5			1000
Arsenic		2	10	2	2	1		20	1000
Beryllium	20	0.5	2	0.5	1				1000
Cadmium	10	0.5	10	0.25	0.5				1000
Chromium (total)	50	2	10	0.5	1				1000
Chromium VI	5							10	
Copper	25	5	10	0.5	2				1000

***The normal method-specific factor for these substances is 1, therefore, the lowest standard concentration in the calibration curve is equal to the above ML value for each substance.

Table 1c – INORGANICS*

	FAA	GFAA	ICP	ICPMS	SPGFAA	HYDRIDE	CVAA	COLOR	DCP
Cyanide								5	
Lead	20	5	5	0.5	2				10,000
Mercury				0.5			0.2		
Nickel	50	5	20	1	5				1000
Selenium		5	10	2	5	1			1000
Silver	10	1	10	0.25	2				1000
Thallium	10	2	10	1	5				1000
Zinc	20		20	1	10				1000

* The normal method-specific factor for these substances is 1, therefore, the lowest standard concentration in the calibration curve is equal to the above ML value for each substance.

Table Id - PESTICIDES – PCBs**	**GC**
4,4’-DDD	0.05
4,4’-DDE	0.05
4,4’-DDT	0.01
a-Endosulfan	0.02
a-Hexachloro-cyclohexane	0.01
Aldrin	0.005
b-Endosulfan	0.01
b-Hexachloro-cyclohexane	0.005
Chlordane	0.1
d-Hexachloro-cyclohexane	0.005
Dieldrin	0.01

Endosulfan Sulfate	0.05
Endrin	0.01
Endrin Aldehyde	0.01
Heptachlor	0.01
Heptachlor Epoxide	0.01
Lindane(g-Hexachloro-cyclohexane)	0.02
PCB 1016	0.5
PCB 1221	0.5
PCB 1232	0.5
PCB 1242	0.5
PCB 1248	0.5
PCB 1254	0.5
PCB 1260	0.5
Toxaphene	0.5

** The normal method-specific factor for these substances is 100, therefore, the lowest standard concentration in the calibration curve is equal to the above ML value for each substance multiplied by 100.

Techniques:

GC - Gas Chromatography
GCMS - Gas Chromatography/Mass Spectrometry
HRGCMS - High Resolution Gas Chromatography/Mass Spectrometry (i.e., EPA 1613, 1624, or 1625)
LC - High Pressure Liquid Chromatography
FAA - Flame Atomic Absorption
GFAA - Graphite Furnace Atomic Absorption
HYDRIDE - Gaseous Hydride Atomic Absorption
CVAA - Cold Vapor Atomic Absorption
ICP - Inductively Coupled Plasma
ICPMS - Inductively Coupled Plasma/Mass Spectrometry
SPGFAA - Stabilized Platform Graphite Furnace Atomic Absorption (i.e., EPA 200.9)
DCP - Direct Current Plasma
COLOR – Colorimetric

FIGURE 1

CITY OF LOS ANGELES
ENVIRONMENTAL MONITORING DIVISION

CORRECTIVE ACTION REPORT (CAR) FORM

Originator: _____Date: _____CAR No.: _____

Description of the problem:

During what phase of operation was the problem identified?

Pertinent Information/Data:

Operations/Data affected:

Corrective Action Plan:

Personnel Responsible for Corrective Action (Initiator): _____
Estimated Corrective Action Completion Date: _____

Approval of the Corrective Action Plan:

_____ _____
Initiator (date) Lab Supervisor (date)

CAR No.: _____

Results of the Corrective Action:

Data Corrected:

Date Corrected: _____

Acceptance of the Corrective Action:

_____ _____

Lab Manager (date) QA Officer or Lab Director(date)

Note: Lab retains the original record and provides a copy to QAU

FIGURE 1- 2

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