# **QUALITY ASSURANCE PROJECT PLAN**

## Version 2.0

LARWQCB Order No. R4-2016-0143

Prepared for:



1521 I Street Sacramento, CA 95814

November 1, 2019

Prepared by:



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# **PROJECT MANAGEMENT**

### **1.0 APPROVAL SIGNATURES**

### PROJECT ORGANIZATION:

<u>Title:</u>	Name (Affiliation):	Signature:	Date
LAILG Project Manager	Ariana Zamora McCray		
Laboratory Project Manager	Chris Samatmanakit (WECK)		
Laboratory Project Manager	Beth Maturino (ABC)		
Project Manager & QA Officer	Bryn Home (PacRL)		
Field Supervisor	Scott Jordan (PacRL)		
REGIONAL BOARD (LAR	WQCB):		
<u>Title:</u>	Name:	Signature:	Date
Executive Office	Renee Purdy		
Program Main Contact	Snejana Toneva		

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### **3.0 DISTRIBUTION LIST**

<u>Title:</u>	Name (Affiliation):	<u>Tel. No.:</u>	<u>Copies</u>
Contractor Project Manager & QA Officer	Bryn Home (PacRL)	(855) 682-1802 ext 101	1
Contractor Field Supervisor	Scott Jordan (PacRL)	(855) 682-1802 ext 102	1
Executive Officer	Renee Purdy	(213) 576-6622	original
Program Main Contact	Snejana Toneva	(213) 576-7159	1
Laboratory Project Manager	Chris Samatmanakit (Weck)	(626) 336-2139 x 141	1
Laboratory Project Manager	Beth Maturino (ABC)	(805) 643-5621 x 10	1
LAILG Administrator	Ariana Zamora McCray	(805) 668-1876	1

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#### 4.0 PROJECT TASKS/ORGANIZATION

#### 4.1 INVOLVED PARTIES AND ROLES

Nursery Growers Association (NGA) is a non-profit association chartered in the late 1950s. The purpose of NGA is to foster and encourage the growth and development of quality nursery stock and to promote all matters that pertain to the best interests of the wholesale nursery growers. NGA developed the Los Angeles Irrigated Lands Group (LAILG) for compliance with the CWIL in the Los Angeles Region. PacRL was contracted by NGA to manage the technical aspect of the LAILG.

Mrs. Ariana McCray is the Project Manager and primary contact for the LAILG. Pacific Ridgeline (PacRL) was contracted to assist the LAILG with the requirements of the CWIL. Mr. Bryn Home is the point of contact for the program and the Quality Assurance (QA) Officer, and Mr. Scott Jordan is the Field Supervisor. Table 1 presents a list of contact information for all personnel involved in the program.

NAME	ORGANIZATION AFFILIATION	TITLE	CONTACT INFORMATION
Bryn Home	Pacific Ridgeline	Project Manager / QA Officer	(855) 682-1802
Scott Jordan	Pacific Ridgeline	Field Supervisor	(855) 682-1802
Ariana Zamora McCray	NGA/LAILG	Project Manager	(805) 668-1876
Renee Purdy	LARWQCB	Executive Officer	(213) 576-6622
Snejana Toneva	LARWQCB	Supervisor	(213) 576-7159
Chris Samatmanakit	Weck Laboratories	Project Manager	(626) 336-2139 x 141
Beth Maturino	ABCLaboratories	Project Manager	(805) 643-5621 x 10

Table 1. Personnel Responsibilities

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The NGA is responsible for organizing and managing the administrative aspect of the LAILG, while PacRL manages the technical aspect of the LAILG. The NGA allowed non-NGA individuals to join the LAILG for the purposes of the Waiver program only. The NGA assisted the individual participants in completing and submitting the required enrollment forms and collects all fees associated with the program. PacRL developed the required Quality Assurance Project Plan (QAPP) and this Monitoring Reporting Plan (MRP), on behalf of the LAILG. PacRL is also responsible for field monitoring and sampling at the selected sites for the LAILG, and all additional reporting, including Water Quality Management Plans (WQMP) and Annual Monitoring Reports (AMR).

The LAILG selected Weck Laboratories, Inc. (Weck) to complete the required laboratory analytical testing, and Aquatic Bioassay and Consulting Laboratories, Inc (ABC) to complete the toxicity testing for the group. Weck and ABC are both certified by the California Environmental Laboratory Accreditation Program, and their certification numbers are 1132 and 1907, respectively. Mr. Chris Samatmanakit of Weck is the Laboratory Project Manager for this waiver program, and Alan Ching is the QA officer. Mr. Scott Johnson of ABC is the Laboratory Project Manager for this waiver program, and Michael Machuzak is the QA officer.

### 4.2 QUALITY ASSURANCE OFFICER ROLE

As the QA Officer, Mr. Home's role is to establish the QA and quality control (QC) procedures found in this QAPP as part of the sampling, field analysis, and laboratory analysis procedures. Mr. Home will perform all QA and QC procedures for the project independent of the project management and monitoring programs. Mr. Home will also work with the QA Officers for Weck and ABC, by communicating all QA and QC issues contained in this QAPP to Weck and ABC.

Mr. Home will review and assess all procedures during the duration of the CWIL against QAPP requirements. All findings will be reported to Mr. Moran, including all requests for corrective action. Mr. Home may stop all actions, including those conducted by Weck and ABC, if there are significant deviations from required practices or if there is evidence of a systematic failure.

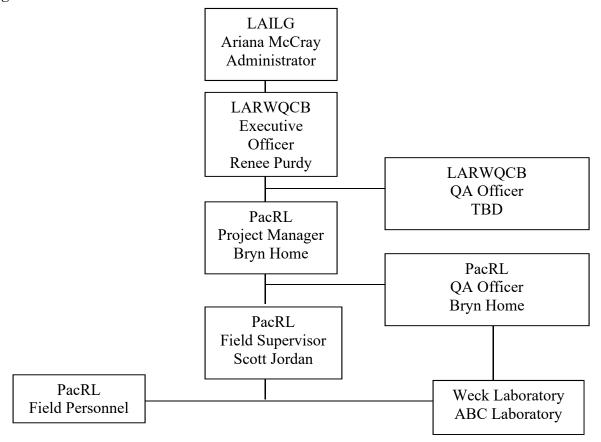
### 4.3 PERSONS RESPONSIBLE FOR QAPP UPDATE AND MAINTENANCE

Changes and updates to this QAPP may be made after a review of the evidence for change by the LAILG, and with the concurrence of both the California Regional Water Quality Control Board, Los Angeles Region (LARWQCB) Project Manager and QA Officer. LAILG's QA Officer will be responsible for making the changes, submitting drafts for review, preparing a final copy, and submitting the final for signature. Copies of previous versions of the QAPP will be discarded so as not to cause confusion.

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#### 4.4 ORGANIZATIONAL CHART AND RESPONSIBILITIES

Management Structure



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#### 5.0 PROBLEM DEFINITION AND BACKGROUND

### 5.1 PROBLEM STATEMENT

The LARWQCB is a State of California Agency that regulates water quality within the coastal watershed of Ventura and Los Angeles Counties under the authorities of the Federal Clean Water Act and State Porter Cologne Water Quality Control Act. The area under the jurisdiction of the LARWQCB is known as the Los Angeles Region.

Water quality impacts associated with agriculture can be primarily traced to discharges resulting from irrigation or stormwater. These discharges may contain pollutants that have been imported or introduced into the irrigation or stormwater; in addition, irrigation practices can mobilize and or concentrate some pollutants. In order to mitigate these potentially polluted discharges from impacting the beneficial uses of water bodies within the Los Angeles Region, the LARWQCB adopted a CWIL (Order No. R4-2005-0080) on November 3, 2005, as mandated by state law and policy. NGA developed the LAILG for compliance with the CWIL, which currently consists of Order #R4-2016-0143.

On October 7, 2010, the LARWQCB adopted a second CWIL for the Los Angeles Region (Order No. R4-2010-0186). Order R4-2010-0186 was extended for six months under Order R4-2015-0202. Order R4-2016-0134, adopted on April 14, 2016, slightly revised the program and extended water quality monitoring throughout the Los Angeles Region for an additional four years.

The LAILG has members within the Dominguez Channel LA/Long Beach Harbors Water Management Area (WMA), the Los Angeles River Watershed, the San Gabriel River Watershed, the Santa Monica Bay WMA, and the eastern portion of the Santa Clara River Watershed. All five Watersheds and WMAs have impacted waterbodies that appear on the Federal 303(d) list, and listed contaminants include constituents that could be related to agricultural uses. Annual Monitoring Reports (AMRs) submitted by the LAILG during throughout each CWIL term have reported runoff water quality that exceeded established Water Quality Benchmarks (WQBs), and could be attributing to the waterbody impacts.

Agriculture in the City of Los Angeles mostly consists of smaller parcel sizes located in urban environments, specifically under power lines. The LAILG was initially formed to assist growers of nursery stock with compliance with the CWIL, but has since expanded to include any grower in the Los Angeles Region who wishes to be part of the group. Refer to Table 2 and Table 3 for crop type and acreage information specific to the LAILG throughout each watershed.

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Table 2 LAILG Distribution and Crop Type, Irrigated acres

		Color Plon	Cutflour	General Orman	Greenhow.	Multiple	Orchand	Row	Sod	Vinevand	Unknown	IRRIGATED ACRES	% TOTAL
# Enrolled	Irrigated Acreage Mapped												
Growers	IRRIGATED ACRES	60.48	6.23	615.20	6.86	184.23	8.02	15.15	16.50	78.41	236.08	1,227.16	
6	Santa Clara River Watershed <sup>!</sup>	0.00	0.00	7.75	0.00	70.00	0.00	0.00	0.00	6.50	0.35	84.60	6.89%
31	Santa Monica Bay WMA	0.00	0.00	31.08	1.20	30.00	5.00	0.00	0.00	68.16	2.63	138.07	11.25%
142	Los Angeles River Watershed	9.30	0.28	316.24	3.91	1.89	1.00	8.00	16.50	3.75	149.83	510.70	41.62%
54	Dominguez Channel LA/Long Beach Harbor WMA	36.68	4.25	77.61	1.75	3.00	2.02	1.00	0.00	0.00	33.99	160.30	13.06%
58	San Gabriel River Watershed*	14.50	1.70	182.52	0.00	79.34	0.00	6.15	0.00	0.00	49.28	333.49	27.18%
291	% TOTAL	4.93%	0.51%	50.13%	0.56%	15.01%	0.65%	1.23%	1.34%	6.39%	19.24%		

Table 3 LAILG Distribution and Crop Type, Total Acres

		Color Plan	Cutflore	General Orneral	Greenhow	Multiple	Orchand	Row Cro	Sod	Vinevand	Unknow	TOTAL ACRES	% TOTAL
# Enrolled	Total Acreage Mapped												
Growers	TOTAL ACRES	90.89	9.80	936.27	14.30	2,241.59	14.44	20.05	16.50	204.33	243.07	3,791.24	
6	Santa Clara River Watershed!	0.00	0.00	9.50	0.00	890.00	0.00	0.00	0.00	39.00	0.35	938.85	24.76%
31	Santa Monica Bay WMA	0.00	0.00	44.97	3.89	132.50	9.92	0.00	0.00	159.53	3.38	354.19	9.34%
142	Los Angeles River Watershed	13.80	1.00	520.03	3.91	2.00	2.50	9.50	16.50	5.80	154.72	729.76	19.25%
54	Dominguez Channel LA/Long Beach Harbor WMA	57.09	5.00	110.72	6.50	4.75	2.02	1.00	0.00	0.00	35.24	222.32	5.86%
58	San Gabriel River Watershed*	20.00	3.80	251.05	0.00	1,212.34	0.00	9.55	0.00	0.00	49.38	1,546.12	40.78%
291	% TOTAL	2.40%	0.26%	24.70%	0.38%	59.13%	0.38%	0.53%	0.44%	5.39%	6.41%		

\* California State Polytechnical University of Pomona accounts for 1,200 of the total acres in the San Gabriel River Watershed

! Golden Oaks Ranch accounts for 890 of the total acres in the Santa Clara River Watershed Page 5-3 LAILG – QAPP V2.0 November 1, 2019

### 5.2 DECISIONS OR OUTCOMES

The objective of this QAPP is to identify the QA components that are necessary to implement the monitoring requirements of the CWIL. This QAPP also serves as a documented basis as to the required protocol for field monitoring, sampling, laboratory analytical accuracy and detection limits. This objective is achieved using accepted methodology to collect and analyze water and biota samples.

The objectives of the monitoring program required under the CWIL are:

- 1. To assess waters discharged from LAILG sites
- 2. To evaluate compliance with water quality benchmarks to assess if additional best management practices (BMPs) are required for sites that exceed the benchmarks established in the CWIL
- 3. To evaluate findings in conjunction with 303(d) impacted waterbodies within the Los Angeles Region
- 4. To report findings to the LARWQCB as specified in the MRP
- 5. To develop and maintain a WQMP to improve water quality

Water samples will be collected from the surface water runoff from the sites, at a point in or directly bordering the selected sites.

### 5.3 WATER QUALITY OR REGULATORY CRITERIA

The following tables present water quality benchmarks that apply to this program for the sites that are currently being sampled. They are derived from language included in Appendix 4 of the current Waiver, along with the Water Quality Control Plan Los Angeles Region (Basin Plan) objectives, California Toxics Rule benchmarks, USEPA ALB guidelines, and CCR Title 22 maximum contamination levels for municipal water (organic chemicals).

For the purpose of analysis, benchmarks are broken into four general groups: general chemistry (including nutrients), pesticides, toxicity, and field monitoring results.

**General Chemistry** 

General Chemistry water quality objectives for each site were obtained from the Water Quality Control Plan, Los Angeles Region. To choose the most appropriate water quality objectives for each site, all sites are assumed to drain through storm drains that ran perpendicularly to the closest blue line stream. The most relevant stream reach and related water quality objectives will be chosen for each site using this assumption. Table 4 outlines the location specific water quality objectives used to evaluate general chemistry results for this report.

Watershed/stream reach	Ammonia	TDS	Sulfate	Chloride	Nitrogen	TSS	Copper (µg/L)	Phosphate
Los Angeles River:								
Between Figueroa and Willow St.	a)	1,500	350	150	8	-	CCC=0.960e <sup>[(0.8545(in (hardness)))+(-1.702)]</sup>	-
Above Figueroa St.	a)	950	300	150	8	-	CCC=0.960e <sup>[(0.8545(in (hardness)))+(-1.702)]</sup>	_
Rio Hondo above Santa Ana Freeway	a)	750	300	150	8	-	CCC=0.960e <sup>[(0.8545(in (hardness)))+(-1.702)]</sup>	_
Pacoima Wash above Pacoima spreading grounds	a)	250	30	10	MUN	_	CCC=0.960e <sup>[(0.8545(in (hardness)))+(-1.702)]</sup>	
San Gabriel River:								
Between Firestone Blvd. and San Gabriel River Estuary	a)			MUN		-	CCC=0.960e <sup>[(0.8545(in (hardness)))+(-1.702)]</sup>	
Between Ramona and Firestone Blvd.	a)	750	300	150	8	-	CCC=0.960e <sup>[(0.8545(in (hardness)))+(-1.702)]</sup>	-
Between Morris Dam and Ramona Blvd.	a)	450	100	100	8	-	CCC=0.960e <sup>[(0.8545(in (hardness)))+(-1.702)]</sup>	_
Dominguez Channel	a)			MUN		-	CCC=0.960e <sup>[(0.8545(in (hardness)))+(-1.702)]</sup>	_
Santa Monica Bay	a)			MUN		-	CCC=0.960e <sup>[(0.8545(in (hardness)))+(-1.702)]</sup>	_
USEPA Municipal Drinking Water Standard	a)	500	250	400	10	-	1.3 (mg/L)	_

*Table 4 Water Quality Benchmarks, General Chemistry* 

\* All limits are recorded for milligrams per liter (mg/L)

a) Limit varies as a factor of temperature and pH. Objectives based on corresponding field readings for WARM water (One-hour average concentration), as outlined in the Water Quality Control Plan, Los Angeles Region

MUN No site specific objectives have been established. Objectives are based on USEPA guidelines for municipal drinking water standards.

No numeric benchmarks, water quality benchmarks shall be based on the surface water and groundwater basin objectives currently contained in the Water Quality Control Plan Los Angeles Region (Basin Plan) or other applicable water quality standards established for the Los Angeles Region.

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#### Pesticides

Pesticide water quality objectives were taken from the Waiver, USEPA ALB guidelines, and the California Toxics Rule. Table 5 presents pesticide benchmarks outlined in the Waiver. Table 6 presents OC pesticide levels outlined by the California Toxics Rule. The California Toxics Rule is not included in the Waiver as benchmarks for the program (except for Toxaphene), however, LAILG will be using them as general indicators for water quality exceedances for the program.

CONSTITUENT	UNITS	WATER QUALITY BENCHMARK
Chlordane	μg/L	0.00059
4,4' - DDT	μg/L	0.00059
4,4' - DDD	μg/L	0.00084
DDE	μg/L	0.00059
Dieldrin	μg/L	0.00014
Toxaphene	μg/L	0.00075
Chlorpyrifos	μg/L	0.025
Diazinon	µg/L	0.10
µg/L micrograms per liter		

Table 5 Water Quality Benchmarks, Pesticides, CWIL

Table 6 Additional Water Quality Benchmarks, Pesticides, California Toxics Rule

CONSTITUENT	UNITS	WATER QUALITY BENCHMARK Human Health (30-day Average) Drinking Water Sources (consumption of water and aquatic organisms)
Aldrin	ug/L	0.00013
alpha-BHC	ug/L	0.0039
beta-BHC	ug/L	0.014
gamma-BHC (Lindane)	ug/L	0.019
Endosulfan and derivatives	ug/L	110
Endrin	ug/L	0.76
Endrin aldehyde	ug/L	0.76
Heptachlor	ug/L	0.00021
Heptachlor epoxide	ug/L	0.0001

Table 7 presents ALB benchmarks for OP and pyrethroid pesticides. The CWIL does not directly cover benchmarks for these constituents, and does not specifically require ALB benchmarks to be considered as WQBs. The LAILG will be using them as general indicators for water quality exceedances for the program. Any pesticide that exceeded the value reported for acute invertebrates were considered a water quality exceedance for LAILG evaluation purposes. The guidelines for acute invertebrates were chosen because historically the most sensitive species in toxicity testing was Ceriodaphna dubia, a species of water flea.

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Pesticides F			Fish Invertebrates		Nonvascular Plants	Vascular Plants	Office of Water Aquatic Life Criteria			
	Footnote	CAS Number	Acute 1	Chronic 2	Acute 3	Chronic 4	Acute 5	Acute 6	Maximum Concentration (CMC)	Continuous Concentration (CCC)
OP Pesticides	.,								•	
Azinphos Methyl	9	86-50-0	0.18	0.44	0.08	0.25	—		—	_
Chlorpyrifos		2921-88-2	0.90	0.57	0.05	0.04	140	_		
Coumaphos	10	56-72-4	170	11.7	0.037	0.0337	_	166	_	_
Dichlovos (DDVP)		62-73-7	91.5	5.2	0.035	0.0058	14,000	_	0.083	0.041
Dimethoate	9	60-51-5	3100	430	21.5	0.5	20,000	>92,600	_	_
Disulfoton	9	298-04-4	19.5	3	1.95	0.01	_	_	_	_
Ethoprop		13194-48-4	150	24	22	0.8	8,400	_	_	_
Fenthion	8	55-38-9	415	7.5	2.6	0.013	400	> 2,800	_	_
Malathion		121-75-5	2.05	8.6	0.049	0.060	2,400	24,000	_	0.1
Methyl Parathion	13	298-00-0	925	< 10	0.485	0.25	15,000	18,000	_	_
Naled		300-76-5	46	2.9	0.07	0.045	25	> 1,800	_	_
Phorate	8	298-02-2	1.175	0.34	0.3	0.21	> 1,300		_	_
Pyrethroid Pesticides										
Allethrin		584-79-2	3.9	_	1.05	_	_	_	_	
Bifenthrin		82657-04-3	0.075	0.04	0.8	0.0013	_	_	_	_
Cyfluthrin		68359-37-5	0.034	0.01	0.0125	0.0074	>181	_	_	_
Cypermethrin		52315-07-8	0.195	0.14	0.21	0.069	_	_	_	_
Fenpropathrin (Danitol)		64257-84-7	1.1	0.06	0.265	0.064	_		_	_
Deltamethrin		52918-63-5	0.29	0.017	0.055	0.0041	_	_		_
Esfenvalerate	9	66230-04-4	0.035	0.035	0.025	0.017	_	_	_	_
Lambda-cyhalothrin		91465-08-6	0.039	0.031	0.0035	0.002	> 310	_		_
Pendimethalin		40487-42-1	69	6.3	140	14.5	5.2	12.5	_	—
Permethrin	16	52645-53-1	0.395	0.0515	0.0195	0.0014	68			
Prallethrin		23031-36-9	6	3	3.1	0.65	_	>1.324	_	_
Sumithrin		26002-80-2	7.9	1.1	2.2	0.47	_	_	—	_
Tefluthrin		79538-32-2	0.03	0.004	0.035	0.008	_			_

 Table 7 Additional Water Quality Benchmarks, Pesticides, Aquatic Life Benchmarks

 OPP Aquatic Life Benchmarks (µg / L) (Freshwater)

#### Limits Reported in ug/L

<sup>8</sup> Because the underlying toxicity value is a "greater-than" value (such as >265,000), this benchmark may overestimate toxicity.

<sup>9</sup> The chronic benchmark is based on the acute toxicity value (which was lower than the lowest available chronic toxicity value), and therefore may underestimate chronic

<sup>10</sup> Although the underlying acute toxicity value is greater than or equal to the chronic toxicity value, the acute benchmark is lower than the chronic benchmark because acute and chronic toxicity values were multiplied by LOC values of 0.5 and 1, respectively.

<sup>13</sup> Because the underlying toxicity value is a "less-than" value (such as <1,500), this benchmark may underestimate toxicity.

<sup>16</sup> Toxicity values and benchmarks apply to permethrin. If monitoring data represent only the *Cis* isomer of permethrin in water, comparison with benchmarks may underestimate potential toxicity.

#### Toxicity

Tests are run on 100% concentration of samples (no dilution water), which makes numerical values of TUc unable to be accurately determined. Due to the lack of TUc values, a TIE was generally run on samples that exhibited a high mortality. Chronic toxicity testing is conducted for Pimephales promelas (fathead minnow), Ceriodaphnia (water flea), and Selenastrum capricornutum (green algae). During this waiver period, Ceriodaphnia has been the most sensitive species and was the only species tested this sampling year.

For the purpose of triggering TIE procedures, significant toxicity was defined as at least 50 percent mortality or a 50 percent reduction in growth. The 50 percent threshold is consistent with the approach recommended in guidance published by the EPA for conducting TIEs, which

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recommends a minimum threshold of 50 percent mortality because the probability of completing a successful TIE decreases rapidly for samples with less than this level of toxicity.

#### Field Monitoring

For field monitoring results, the Basin Plan for the Los Angeles Region contains narrative objectives for certain chemicals, most notably: biostimulator substances, temperature, pH, turbidity, and Total Suspended Solids. Table 8 presents field monitoring and toxicity benchmarks, as outlined in the Los Angeles Basin Plan. These narrative objectives contain verbiage stating that the natural or ambient conditions of receiving waters are not to be altered by discharges, including some of the constituents listed above. This is problematic, as natural or ambient conditions have not been established in many receiving waters, and discharges from growing operations in the urban Los Angeles Region drain primarily to storm drains. The ultimate endpoint of these storm drains is not well mapped or established, and are comingled with discharges from a number of land use types. Due to the difficulty in ascertaining the impacts to receiving waters, it is assumed in this report that discharges do not affect the receiving water bodies in a large enough magnitude to alter natural or ambient conditions.

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Constituent	Narrative Objective	Applicable Benchmarks	
рН	The pH of inland surface water shall not be depressed below 6.5 or raised above 8.5 as a result of waste discharges. Ambient pH levels shall not be changed by more than 0.5 pH units from natural conditions as a result of waste discharges.	$6.5 \le pH \le 8.5$ Changes to ambient receiving water conditions are not assessed; "ambient" or "natural" conditions have not been established	
Temperature	For water designated WARM, water temperature shall not be altered by more than 5°F above natural temperature. At no time shall WARM-designated waters be raised above 80°F as a result of water discharge	WARM: $\leq 80^{\circ}$ F Changes to ambient receiving water conditions are not assessed; "ambient" or "natural" conditions have not been established	
	For waters designated as COLD, water temperature shall not be altered by more than 5°F above the natural temperature.	COLD: No numeric benchmark. Changes to ambient receiving water conditions are not assessed; "ambient" or "natural" conditions have not been established.	
	No single dissolved oxygen determination shall be less than 5 mg/L, except when natural conditions cause lesser concentrations.	$\geq$ 5 mg/L	
Dissolved Oxygen	The dissolved oxygen content of all surface waters designated as WARM shall not be depressed below 5 mg/L as a result of waste discharge.	WARM: $\geq 5 \text{ mg/L}$	
	The dissolved oxygen content of all surface waters designated as COLD and SPWN shall not be depressed below 7 mg/L as a result of waste discharge.	COLD, SPWN: $\geq$ 7 mg/L	
Turbidity	Waters shall be free of changes in turbidity that cause nuisance or adversely affect beneficial uses. Increases in natural turbidity attribute to controllable water quality factors shall not exceed the following limits: Where natural turbidity is between 0 and 50 NTU, increases shall not exceed 20%. Where natural turbidity is greater than 50 NTU, increases shall not exceed 10%.	No Numeric benchmarks. Changes to ambient receiving water conditions are not assessed; "ambient" or "natural" conditions have not been established.	
Toxicity	All waters shall be free of toxic substances in concentrations that are toxic to, or that produce detrimental physiological responses in human, plant, animal or aquatic life. There shall be no chronic toxicity in ambient waters outside mixing zones.	$\leq 1.0 \ \mathrm{TUc}^{[3]}$	
Biostimulator Substances	Waters shall not contain biostimulator substances in concentrations that promote aquatic growth to the extent that such growth causes nuisance or adversely affect beneficial uses.	No Numeric benchmarks. Nutrients listed on Table X.	
Total Suspended Solids (TSS)	Wastes shall not contain suspended material in concentrations that cause nuisance or adversely affect beneficial uses.	No numeric benchmarks.	

Table 8 Water Quality Benchmarks, Field Monitoring and Toxicity

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#### 6.0 PROJECT TASK/DESCRIPTION

### 6.1 WORK STATEMENT AND PRODUCED PRODUCTS

LAILG will collect water quality data at randomized LAILG sites throughout the year. Maps presenting the location of sampling groups is presented as Figures 2.1-2.5. Detailed information on sample sites, sample locations, and sampling schedule are presented in the MRP. In addition, and if required, receiving water quality and toxicity data from various water bodies in the Los Angeles Region will be compared to data collected for the LAILG to evaluate if the LAILG is potentially contributing to detrimental surface water impacts.

Detailed information on the measurement processes and sampling techniques used to collect data are presented in Sections 11 and 13.

#### 6.2 CONSTITUENTS TO BE MONITORED/MEASUREMENT TECHNIQUES

The constituents to be monitored, as outlined in the CWIL, are presented on Table 9.

CONSTITUENT	UNITS	FIELD/LABORATORY TEST
Flow	Cubic feet per second	Field
pН	pH units	Field
Temperature	°F	Field
Dissolved Oxygen	mg/L	Field
Turbidity	NTU	Field
Total Dissolved Solids	mg/L	Laboratory
Total Suspended Solids	mg/L	Laboratory
Hardness (as CaCO <sub>3</sub> )	mg/L	Laboratory
Chloride	mg/L	Laboratory
Ammonia	mg/L	Laboratory
Nitrate-Nitrogen	mg/L	Laboratory
Phosphate	mg/L	Laboratory
Sulfate	mg/L	Laboratory
Total Copper	ng/L	Laboratory
Organophosphate Suite <sup>1</sup>	ng/L	Laboratory
Organochlorines Suite <sup>2</sup>	ng/L	Laboratory
Toxaphene	ng/L	Laboratory
Pyrethroids	ng/L	Laboratory
Toxicity	TU <sub>c</sub> <sup>3</sup>	Laboratory
E.Coli	MPN/100ml	Laboratory
Trash	Observations	Field

*Table 9 Monitoring Constituents* 

<sup>1</sup> Organophosphate Suite: Bolstar, Chlorpyrifos, Demeton, Diazinon, Dichlorvos, Dimethoate, Disulfoton, Ethoprop, Fenchlorophos, Fensulfothion, Fenthion, Malathion, Merphos, Methyl Parathion, Mevinphos, Phorate, Tetrachlorvinphos, Tokuthion, Trichloronate.

<sup>2</sup> Organochlorine Suite: 2.4' - DDD, 2,4' - DDE, 2,4' DDT, 4,4' -DDD, 4,4' -DDE, 4,4' -DDT, Aldrin, BHCalpha, BHC-beta, BHC-delta, BHC-gamma, Chlordane-alpha, Chlordane-gamma, Dieldrin, Endosulfan sufate, Endosulfan-I, Endosulfan-II, Endrin, Endrin Aldehyde, Endrin Ketone.

<sup>3</sup> Chronic Toxic Unit is the reciprocal of the sample concentration that caused no observable effect on the test organism by the end of a chronic toxicity test.

mg/l	milligrams per liter
ng/L	nanograms per liter
°F	degrees Fahrenheit
TUc	chronic toxic unit
NTU	nephalitic turbidity units

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### 6.3 **PROJECT SCHEDULE**

The seasons, as described in the CWIL, are the wet and the dry season. The wet season is from October 15 - May 15, and the dry season is from May 15 - October 15. As required by the Waiver, a total of two sampling events will be completed each year; one event will be completed in the wet season and one event will be completed in the dry season. Wet season samples will be conducted within 24-hours of a rain event with at least 0.5 inches of rain. The first wet season sample will be collected during or after the first storm event of the wet season. Dry season samples will be collected after an irrigation event has occurred at each sampling site. The first dry season sampling event will be collected after the majority of growers have applied pesticides or fertilizers.

Samples collected from the first sampling event will include chronic toxicity tests for three test species: Pimephales promelas (fathead minnow), Ceriodaphnia dubia (water flea) and Selenastrum capricornutum (green algae). Once one toxicity sample has been collected and analyzed, the LAILG will select the most sensitive species for subsequent toxicity monitoring. Based on review of the annual monitoring reports, the Executive Officer may increase or decrease the frequency of sampling.

Table 10 shows an anticipated schedule for when monitoring will be conducted and when AMR demonstrating the monitoring results are due to the LARWQCB. AMRs will present data collected during the previous calendar year.

TASK	SCHE	DULE		
MRP	Submtted with QAPI	P, November 1, 2019		
Updated QAPP	Submtted with MRP, November 1, 2019			
Conduct Monitoring	1 Dry Season Event May 15 - Oct 15, annually	1 Wet Season Event Oct. 16 - May 14, annually		
Submit Anunal Monitoring Report	December 15th, Annually			
Water Quality	With AMR, December 15th, Annually			
Management Plan	Final WQMP, C	October 31, 2020		

Table 10 Anticipated Schedule for Monitoring and Reporting

### 6.4 GEOGRAPHICAL SETTING

In general, rivers, streams, creeks, washes, and storm drains in Los Angeles County eventually drain to the south and west to the Pacific Ocean. Sampling locations for this project are distributed throughout the portion of the Los Angeles Region within Los Angeles County, and are displayed on the regional map (Figure 2).

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### 6.5 CONSTRAINTS

Reasonable efforts will be made to collect samples during a rainfall trigger event in the wet season and during or immediately after irrigation events in the dry season. However, due to the small amounts of water generally used by growers during their irrigation practices, it is anticipated that a number of sites in the LAILG will have no runoff during the dry season. Depending on the size of the LAILG being sampled, the soil permeability, and the size of the rain event, it is possible that stormwater runoff from these sites could be minimal. Based on the LAILGs experience during the previous CWIL period, the constraints listed above generally cause runoff only to be encountered at sampling sites during rainfall periods of storm events, and extended tailwaters from sites are rarely encountered. Due to the length of this program, sampling events will not be rescheduled if there is no discernable runoff, unless there is ample evidence that a larger rain event of sufficient rainfall intensity or a larger volume irrigation event would generate enough runoff volume for sampling purposes.

Wet season sampling events will be conducted as outlined in this report, to the extent practicable. Practical restraints on wet season sampling events include, but are not limited to, the following: lab, subcontractor, and sampling site closures during weekends and holidays; sample holding times; safety of the monitoring team; and the time of storm events (day or night) Page 7-1 LAILG – QAPP V2.0 November 1, 2019

### 7.0 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

### 7.1 DATA QUALITY OBJECTIVES

The data quality objectives of this monitoring program are to supply information that characterizes, as closely as possible, the degree to which runoff water released from growing agricultural properties is impacting surface water bodies of the Los Angeles Region. This objective will be accomplished using Surface Water Ambient Monitoring Program (SWAMP) procedures for sample collection, and standard methods for laboratory analysis.

QA objectives are the detailed QC specifications for precision, accuracy (bias), sensitivity and resolution, comparability, representativeness, and completeness. The quality assurance objectives will be used as comparison criteria during data quality review to determine if the minimum requirements have been met and the data may be used as planned.

#### 7.1.1 Precision

Precision is a measurement of the degree to which two or more measurements are in agreement. Precision objectives in this program are determined through field and lab duplicates, laboratory replicates, matrix spikes and matrix spike duplicates. Precision for chemical measurements is represented by using relative percent difference (RPD) between duplicate samples.

Precision objective for toxicity measurements in this program focus on replicate reference toxicant exposures. Precision for toxicity measurements is quantified relative to the mean and standard deviation of previous reference toxicant exposures.

### 7.1.2 Accuracy (Bias)

Accuracy is the degree of agreement between an observed value and an accepted reference or true value. Bias is the quantitative measurement of the difference of the observed and accepted values. Accuracy objectives in this program are determined through field, equipment, and laboratory blanks, laboratory control standards, matrix spikes, and surrogate spikes. The accuracy of chemical measurements is defined as percent recovery.

Accuracy objectives for toxicity measurements in this program focus on reference survival, growth, or larval development. Accuracy for toxicity measurements is quantified relative to the mean and standard deviation of previous reference toxicant exposures.

### 7.1.3 Sensitivity and Resolution

Sensitivity is most commonly defined as the lowest value an instrument or method can measure a value with reasonable statistical certainty. Resolution refers to the capability of the instrument or method to recognize small differences between values. For chemical analysis, the sensitivity of

an instrument or method is reported as the Method Reporting Limit (MRL). The MRL is the is the lowest level that the instrument or method can detect to with reasonable statistical certainty.

Sensitivity and resolution objectives for this program are to keep MRLs, at a minimum, above the WQOs set in Table 3. Sensitivity and resolution for bioassessments is applied to taxonomic identifications, where organisms are identified to rank in the hierarchy of classification of biologic organisms based on need or objectives. This level of identification is referred to the standard taxonomic effort.

### 7.1.4 Comparability

Comparability is an expression of the assurance with which one measurement can be compared to another. Comparability of data will be managed by ensuring that sampling teams adhere to standardized measurement and sampling protocol, and that labs also follow standardized protocols for the preparation and analysis of samples. Detailed information on these protocols is outlined in this QAPP.

### 7.1.5 Representativeness

Representativeness expresses the degree to which data accurately represents a characteristic of a parameter variation at a sampling point. Representativeness will be managed through the selection of site locations and sampling methodology, along with adherence to Standard Operating Procedures (SOPs), use of appropriate sampling equipment, and maintaining proper sample handling and storage conditions. Sites were chosen to represent the group as a whole to characterize the type of discharges from participants in the LAILG.

### 7.1.6 Completeness

Completeness is a measure of the amount of valid data obtained from a sampling event compared to the amount that was expected to be obtained under normal conditions. Completeness is commonly expressed as a percentage of the number of reported measurements that meet the requirements outlined in this QAPP, and for this program, is set at 90 percent. Completeness criteria will be based solely on sampling data that is available to be collected.

General data quality objectives for field and laboratory analysis are presented in Table 11 through Table 14. Evaluation of collected data will be conducted to ensure that data quality objectives, as outlined in the QAPP, were achieved.

Constituent	Method	Units	Method Reporting Limit	Precision % or RPD	Accuracy, LCS* (value or %)	Accuracy, Matrix Spikes %	Completeness
Flow	Field	cubic ft/sec	na	na	+/- 10 %	na	90%
pН	Field	pH units	na	+/- 5	+/- 0.5 units	na	90%
Temperature	Field	deg F	na	+/- 5	+/- 0.5°C	na	90%
Disolved Oxygen	Field	mg/L	na	+/- 20	+/- 0.5 mg/L	na	90%
Turbidity	Field	NTU	na	+/- 10	+/- 10 %	na	90%
Electrical Conductivity	Field	S/m	na	+/- 5	+/- 5 %	na	90%
Trash	Field	observations	na	na	na	na	90%
Hardness (as CaCO3)	EPA 200.7	mg/L	0.100	30	85-115	70-130	90%
Solids, Total Dissolved	SM 2540C	mg/L	5	10	96-102	na	90%
Solids, Total Suspended	SM 2540D	mg/L	5	20	na	na	90%
Chloride	EPA 300.0	mg/L	0.50	20	90-110	76-118	90%
Ammonia-N	EPA 350.1	mg/L	0.10	15	90-110	90-110	90%
Nitrate Nitrogen	EPA 300.0	mg/L	0.11	20	90-110	84-115	90%
Toxaphene	EPA 608	ng/L	500	30	41-126 %	41-126 %	90%
o-Phosphate	EPA 365.1	mg/L	0.0020	20	90-110	90-110	90%
o-Phosphate, dissolved	EPA 365.1	mg/L	0.0020	20	90-110	90-110	90%
Phosphorus	EPA 365.1	mg/L	0.010	20	90-110	90-110	90%
Phosphorus, dissolved	EPA 365.1	mg/L	0.010	20	90-110	90-110	90%
Sulfate	EPA 300.0	mg/L	0.50	20	90-110	78-111	90%
Organophosphate Suite				See Table	X.1		
Organochlorines Suite				See Table	X.1		
Pyrethroids				See Table	X.1		
Total Copper	EPA 200.8	ug/L	0.00050	30	85-115	70-130	90%
E. Coli	SM 9223B	MPN/100 ml	na	25	na	na	90%
Toxicity	na	TUc	na	!	!	na	90%

### Table 11 Data Quality Objectives – General Chemistry

Relative Percent Difference RPD

LCS Laboratory Control Sample

MPN Most Probable Number

na not applicable/available

or control limits at +/- 3 standard deviations based on actual lab data \*

! Meet criteria as set forth in relevant toxicity test, including replicates

Constituent	Method	Units	Method Reporting Limit	Precision % or RPD	Accuracy, LCS* (value or %)	Accuracy, Matrix Spikes %	Completeness
Azinphos Methyl	EPA 525.2	ng/L	10	30	0.1-188	0.1-154	90%
Bolstar	EPA 525.2	ng/L	10	30	11-166	4-184	90%
Chlorpyrifos	EPA 525.2	ng/L	10	30	37-169	37-168	90%
Coumaphos	EPA 525.2	ng/L	10	30	0.1-225	0.1-203	90%
Demeton-o	EPA 525.2	ng/L	10	30	0.1-211	0.1-208	90%
Demeton-s	EPA 525.2	ng/L	10	30	0.1-213	0.1-207	90%
Diazinon	EPA 525.2	ng/L	10	30	43-152	36-153	90%
Dichlorvos	EPA 525.2	ng/L	10	30	46-133	42-137	90%
Dimethoate	EPA 525.2	ng/L	10	30	10-234	4-222	90%
Disulfoton	EPA 525.2	ng/L	10	30	0.1-212	12-199	90%
Ethoprop	EPA 525.2	ng/L	10	30	53-163	51-167	90%
Ethyl parathion	EPA 525.2	ng/L	10	30	7-230	5-229	90%
Fensulfothion	EPA 525.2	ng/L	10	30	0.1-265	0.1-316	90%
Fenthion	EPA 525.2	ng/L	10	30	20-177	23-169	90%
Malathion	EPA 525.2	ng/L	10	30	14-175	6-184	90%
Merphos	EPA 525.2	ng/L	10	30	28-181	3-210	90%
Methyl parathion	EPA 525.2	ng/L	10	30	0.1-252	0.1-249	90%
Mevinphos	EPA 525.2	ng/L	10	30	14-202	25-189	90%
Naled	EPA 525.2	ng/L	10	30	0.1-240	0.1-242	90%
Phorate	EPA 525.2	ng/L	10	30	26-180	31-181	90%
Ronnel	EPA 525.2	ng/L	10	30	34-154	29-153	90%
Stirophos	EPA 525.2	ng/L	10	30	0.1-188	0.1-167	90%
Tokuthion (Prothiofos)	EPA 525.2	ng/L	10	30	23-159	27-160	90%
Trichloronate	EPA 525.2	ng/L	10	30	34-153	40-150	90%

Table 12 Data Quality Objectives – Organophosphate Pesticides

RPD Relative Percent Difference

LCS Laboratory Control Sample

na not applicable/available

\* or control limits at +/- 3 standard deviations based on actual lab data

Constituent	Method	Units	Method Reporting Limit	Precision % or RPD	Accuracy, LCS* (value or %)	Accuracy, Matrix Spikes %	Completeness
2,4'-DDD	EPA 608	ng/L	5	na	na	na	90%
2,4'-DDE	EPA 608	ng/L	5	na	na	na	90%
2,4'-DDT	EPA 608	ng/L	5	na	na	na	90%
4,4'-DDD	EPA 608	ng/L	5	30	42-133	23-124	90%
4,4'-DDE	EPA 608	ng/L	5	30	33-126	30-114	90%
4,4'-DDT	EPA 608	ng/L	5	30	35-147	11-151	90%
Aldrin	EPA 608	ng/L	5	30	18-117	18-110	90%
alpha-BHC	EPA 608	ng/L	5	30	47-119	43-114	90%
alpha-Chlordane	EPA 608	ng/L	5	na	na	na	90%
Aroclor 1016	EPA 608	ng/L	100	na	na	na	90%
Aroclor 1221	EPA 608	ng/L	100	na	na	na	90%
Aroclor 1232	EPA 608	ng/L	100	na	na	na	90%
Aroclor 1242	EPA 608	ng/L	100	na	na	na	90%
Aroclor 1248	EPA 608	ng/L	100	na	na	na	90%
Aroclor 1254	EPA 608	ng/L	100	na	na	na	90%
Aroclor 1260	EPA 608	ng/L	100	na	na	na	90%
beta-BHC	EPA 608	ng/L	5	30	53-123	24-135	90%
Chlordane (tech)	EPA 608	ng/L	100	na	na	na	90%
cis-Nonachlor	EPA 608	ng/L	5	na	na	na	90%
delta-BCH	EPA 608	ng/L	5	30	51-123	37-122	90%
Dieldrin	EPA 608	ng/L	5	30	48-123	27-132	90%
Endosulfan I	EPA 608	ng/L	5	30	14-131	0.1-140	90%
Endosulfan II	EPA 608	ng/L	5	30	40-121	17-122	90%
Endosulfan sulfate	EPA 608	ng/L	5	30	44-140	37-311	90%
Endrin	EPA 608	ng/L	5	30	40-143	42-144	90%
Endrin aldehyde	EPA 608	ng/L	5	30	18-136	11-113	90%
gamma-BCH (Lindane)	EPA 608	ng/L	5	30	49-117	33-112	90%
gamma-Chlordane	EPA 608	ng/L	5	na	na	na	90%
Heptachlor	EPA 608	ng/L	5	30	31-130	28-131	90%
Heptachlor epoxide	EPA 608	ng/L	5	30	49-122	36-117	90%
Methoxychlor	EPA 608	ng/L	5	na	na	na	90%
Mirex	EPA 608	ng/L	5	na	na	na	90%
Oxychlordane	EPA 608	ng/L	5	na	na	na	90%
Toxaphene	EPA 608	ng/L	500	na	na	na	90%
trans-Nonachlor	EPA 608	ng/L	5	na	na	na	90%

Table 13 Data Quality Objectives – Organochlorines Pesticides

RPD Relative Percent Difference

LCS Laboratory Control Sample

na not applicable/available

\* or control limits at +/- 3 standard deviations based on actual lab data

Constituent	Method	Units	Method Reporting Limit	Precision % or RPD	Accuracy, LCS* (value or %)	Accuracy, Matrix Spikes %	Completeness
Allethrin	EPA 8270M	ng/L	2.0	50	50-150	na	90%
Bifenthrin	EPA 8270M	ng/L	2.0	50	50-150	na	90%
Cyfluthrin	EPA 8270M	ng/L	2.0	50	50-150	na	90%
Cypermethrin	EPA 8270M	ng/L	2.0	50	50-150	na	90%
Deltamethrin/Tralomethrin	EPA 8270M	ng/L	2.0	50	50-150	na	90%
Dichloran	EPA 8270M	ng/L	2.0	50	50-150	na	90%
Fenpopathrin	EPA 8270M	ng/L	2.0	50	50-150	na	90%
Fenvalerate/Esfenvalerate	EPA 8270M	ng/L	2.0	50	50-150	na	90%
L-Cyhalothrin	EPA 8270M	ng/L	2.0	50	50-150	na	90%
Pendimethalin	EPA 8270M	ng/L	2.0	50	50-150	na	90%
Permethrin	EPA 8270M	ng/L	2.0	50	50-150	na	90%
Prallethrin	EPA 8270M	ng/L	2.0	50	50-150	na	90%
Sumithrin (Phenothrin)	EPA 8270M	ng/L	10	50	50-150	na	90%
Tefluthrin	EPA 8270M	ng/L	2.0	50	50-150	na	90%

Table 14 Data Quality Objectives – Pyrethroid Pesticides

RPD Relative Percent Difference

LCS Laboratory Control Sample

na not applicable/available

\* or control limits at +/- 3 standard deviations based on actual lab data

### 8.0 SPECIAL TRAINING NEEDS/CERTIFICATION

### 8.1 SPECIALIZED TRAINING OR CERTIFICATIONS

No specialized training or certification is required for this project. Although no specialized training is required, field sampling employees have received training in water sampling techniques as outlined in ASTM standard D3370 and SWAMP guidelines for surface water collection. An in-house refresher course on water sampling is undertaken at least once a year.

BC and ABC adhere to the training outlined in their specific quality assurance manuals. The California Environmental Laboratory Accreditation Program has certified Weck and ABC; their certification number is 1132 and 1907, respectively.

### 8.2 TRAINING AND CERTIFICATION DOCUMENTATION

Documents outlining field sampling employee training are filed and available for review. Documented information consists of the type of training, the data covered, the instructor, and whether the course was completed satisfactorily. Records of lab analyst training are available from Weck and ABC upon request.

#### 8.3 TRAINING PERSONNEL

The QA Officer will supervise all refresher courses. Weck and ABC's individual QA Officers will be responsible for training lab personnel.

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#### 9.0 DOCUMENTS AND RECORDS

All documents generated by this project will be stored in raw and database form. Examples of documentation generated and kept on record include: the MPP, QAPP, field monitoring records, analytical data reports, AMRs, WQMPs and revisions, communications with stakeholders, and chain of custody (COC) documentation. Field monitoring records will be developed and maintained on a site-specific basis. Copies of records held by the laboratory will be provided to PacRL's Project Manager and maintained in the project file.

Pertinent field information will be recorded on SWAMP field log sheets, including (as applicable):

- The width, depth, and flow rate of runoff, if applicable
- The surface water condition
- Weather and environmental conditions
- Evidence of pesticide/fertilizer or sediment management
- Visible crop and cultivation practices
- Photographic documentation
- Field monitoring data
- Discussion of any problems encountered
- Discussion of any deviations from the QAPP.

An example of a field log sheet is presented in Appendix A.

At minimum, the following sample specific information from field and laboratory analytical reports will be provided for each sampling event:

- Lab name
- Sample Identification
- Date and time of sample collection
- Date of analysis, analysis method, and dilution factor
- Method Reporting Limits (MRL), matrix spikes, relative percent differences (RPD), and percent recovered (if applicable)
- Monitoring location/field descriptions
- Sample type, e.g. grab or composite type
- QC sample type and frequency
- Results of samples collected, all laboratory QC samples (calibrations, blanks, surrogates, laboratory spikes, matrix spikes, reference materials, etc.) and the identification of each analytical sample batch.

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Copies of this QAPP will be distributed to parties involved with the project, as listed in Section 3, including field sampling and laboratory personnel. Future changes or amendments to the QAPP will be distributed in the same fashion. Copies of previous versions of the QAPP will be discarded to avoid confusion.

Any SOPs for use by office or field personnel will be certified by the Project Manager and Project QA officer prior to implementation and will be periodically reviewed and updated as necessary. All pertinent SOPs will be distributed to field monitoring personnel prior to sampling events for use in the field.

The LAILG Project Manager and Project QA Officer will be responsible for the hard copy documents collected during this program. Electronic reports will be complied in a database for reference and submittal to the LARWQCB. All monitoring information and data used to complete the monitoring reports will be retained for at least ten years from the date of sampling, measurement, report, or application.

# **DATA GENERATION AND ACQUISITION**

### **10.0 SAMPLING PROCESS DESIGN**

### **10.1 OPERATIONAL GROUPING**

In the Los Angeles Region, LAILG members vary from country-wide growing operations to small, less than one-acre growers. In order to properly represent all members enrolled in the LAILG, members were broken into various groups based on operational information and land use patterns. This was done in order to separate and sample both small growers and large institutional growers to determine the potential water quality impacts between growers who are substantially different in both growing practices and corporate practices.

LAILG used grower information to evaluate the potential impact of facilities based on their operational practices, including how much material (fertilizer, pesticides, irrigation water, etc.) members used on a per acre basis. The general hypothesis of the LAILG is that larger operations, based on sales, total company size, and shipping patterns, will show more intense fertilizer and pesticide use patterns, corresponding to an increased risk of contaminants leaving the property.

In order to determine these operational practices, LAILG submitted a General Questionnaire to each grower. The questionnaire requested the following information: gross sales for an operator, company-wide; crop type; total irrigated acreage operated by company, including acreage both inside and outside LA County; shipping patterns; total fertilizer use and formulation, per parcel per year; total pesticide/herbicide/fungicide applications, per parcel per year; irrigation use per year; fertilizer application practices; and, irrigation practices.

Based on the self-reported answers, growers were grouped into Large Operations, Medium Operations, Small Operations, and Micro Operations. Growers who did not report data were put into an unknown category.

### **10.2 SAMPLING SITE METHODOLOGY**

Due to logistical issues with stormwater sampling in the Los Angeles Region during storm events (traffic, timing for sample delivery), the entire group was divided into North and South Regions for sampling purposes. Interstate 5 and the 101 freeway were chosen as the dividing line, which splits the entire group and each sub group roughly in half. Sampling during each dry and wet season event will alternate between the North and South regions yearly.

In order to evaluate differences in water quality being discharged from the various member groups, LAILG proposes to randomize sampling sites in each region for each sampling event. LAILG will collect samples from one random member in each regional grouping during each sampling event, plus an additional follow up sample from a member that previously reported a WQB exceedance in historical sampling events in the region. A total of five sites will be visited each sampling event, once during the dry season and once during the wet season of each year.

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Randomization for sampling sites will be conducted with random.org, by randomizing each grouping within each region for each sampling event. Records of the randomization will be kept on file. The top location in each group will be selected as the sampling site, and the second location in each group will be selected as the alternate site. The follow up sampling for a location that previously reported a WQB exceedance will be hand selected by LAILG. Once a site has been randomly chosen for sampling, it will be removed from the randomization list. If WQB exceedances are reported at a location, it will be added to the list for follow up sampling.

An alternate site was included in the randomization since many of the current locations have never been visited by LAILG personnel. It is anticipated that some chosen random location may not have sufficient runoff during rain events for sampling, due to topographic settings or operational practices. If a site is visited during a wet season sampling event and it is apparent that there will not be sufficient runoff for sampling during the time of the visit, the alternative location will be visited and site conditions will be noted. Included in the notes will be observations on what size storm might be required in order to produce runoff at the location.

A regional map showing member locations, regional sampling boundaries, and watersheds is presented as Figure 1. Zoomed in maps presenting member groupings and the previous information is presented as Figures 1.1 through 1.4. A list of all members in each group and each sampling region that will be used for randomization is included in the MRP.

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#### **11.0 SAMPLING METHODS**

### **11.1 FIELD PROTOCOLS**

Field sampling events will consist of a minimum two-person crew for safety purposes. Field sampling will only be conducted on weekdays during daylight hours. All field personnel involved in sample collection will be trained in SWAMP procedures, and will use proper sample collection containers and equipment. Sampling personnel will be trained on how to collect a representative sample and avoid potential sources of sample contamination. All equipment that comes into contact with samples or sampling water will be decontaminated in between sampling stations and sampling events.

In order to identify the storm events large enough to trigger wet season monitoring events, LAILG will be continually monitoring the 10-day forecast for the sampling region. Once a rain event starts, LAILG will watch rain gauges throughout greater Los Angeles to evaluate when the threshold of 0.50 inches of rain has been met. Once 0.50 inches of rain has fallen, sampling personnel will be mobilized for sample collection. If required, sampling events may be initiated on weekends as well as weekdays; however, sampling will not be performed during late nighttime hours due to safety concerns involving field crews. If 0.50 inches has fallen but the rain event has ceased, LAILG will evaluate if there is enough potential runoff to yield sufficient water for sampling based on communications with personnel in the sampling area.

Prior to departure for the sites, field personnel will confirm that all field equipment, as outlined in the field equipment checklist (Appendix A), is available and cleaned for sample and data collection. This includes the number and type of sample containers used for water collection. While on site, field log sheets (Appendix A) will be completed to the extent possible, and a COC will be filled out prior to returning to the office. All samples will be collected and stored as outlined in this document. Standard Operating Procedures (SOPs) for collection of water samples are presented in (Appendix B). Any deviations from the SOP will be documented on the field logs. Following sample collection at all sites, a COC will be completed using field log sheets, and samples will either be directly delivered to the appropriate laboratories or stored in a refrigerator until shipping or delivery. All samples that are shipped to a laboratory will be shipped in sealed coolers maintained at approximately 2-4 °C with ice or ice substitutes.

#### **11.2 SAMPLE COLLECTION**

Pools of water with no observable flow will not be sampled, and site conditions will be documented on the field log sheets and with photographs. A new pair of clean nitrile gloves are to be used at each individual sampling site. Sample containers will be pre-cleaned by Weck and ABC, and certified to be free of contamination according to the US EPA specifications for the appropriate methods.

Depending on the site topography, grab samples will be collected directly from concrete-lined drainage channels, dirt channels, and sheet flows directly leaving the sites. The samples will be collected by directly submerging the sample containers when applicable. Standing water with no

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visible flow will not be sampled. Although this is the primary method for sample collection, due to varying runoff and surface conditions this approach will not always be feasible. Site layout, configuration, and runoff conditions will influence the technique in which grab samples are collected. Complete procedures for collection of surface water samples are presented in Appendix B.

Sampling devices and sample bottles (that are not pre-sterilized and do not contain preservatives/fixing agents) will be rinsed three times with sample water prior to collecting each sample. Sterile bottles, whirl-paks, and sample bottles with preservatives will not be rinsed with sample water prior to collecting the sample. Also, sample bottles containing preservatives/fixing agents will not be used directly for sampling; a sample device will be used to collect the sample and will be transferred to the appropriate sample bottle.

### 11.3 FIELD MEASUREMENTS AND QC SAMPLES

QC samples will be collected to verify data quality. QC criteria are outlined in Section 14.

Water quality parameters, including flow rate, pH, dissolved oxygen, electrical conductivity, turbidity, and temperature will be measured prior to collecting samples for laboratory analysis. Water quality parameters will be measured with a hand-held meter. SOPs for the calculation of flow measurements are presented in Appendix C.

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#### 12.0 SAMPLE HANDLING AND CUSTODY

Sample custody will be traceable from the time of sample collection until results are reported. Table 15 describes the sample holding containers, sample preservation methods, and maximum holding times for each monitoring constituent.

PARAMETER	SAMPLE BOTTLE	TYPICAL SAMPLE VOLUME	PREFERRED/MAXIMUM HOLDING TIMES
Flow, pH, Temperature, Dissolved Oxygen, Turbidity, Conductivity	Field Measurements	NA	NA
Trash	Field Observation	NA	NA
Total Suspended Solids	Plastic Bottle	0.5 Liter	7 days at 4°C, dark
Total Dissolved Solids	Plastic Bottle	0.5 Liter	7 days at 4°C, dark
Hardness (as CaCO <sub>3</sub> )	NA	NA	10 days at 4°C, dark
Chloride	Plastic Bottle	0.25 Liter	28 days at 4°C, dark
Sulfate	Plastic Bottle	0.25 Liter	28 days at 4°C, dark
Nitrate	Plastic Bottle	0.25 Liter	28 days at 4oC, dark, preserved with H SO
Phosphate	Plastic Bottle	0.25 Liter	28 days at 4oC, dark, preserved with H SO
Ammonia	Plastic Bottle	0.25 Liter	28 days at 4oC, dark, preserved with H SO
Organochlorine Pesticides	Amber Glass	2 Liter	7 days at 4°C, dark
Organophosphorus Pesticides	Amber Glass	2 Liter	14 days at 4°C, dark
Total Copper	Plastic Bottle	0.25 Liter	180 days, dark, preserved with HNO <sub>3</sub>
Pyrethroids	Amber Glass	2 Liter	21 days at 4°C, dark
Toxicity	5 gal plastic cube	10 gal	Refrigerate at 4°C send to lab immediately

Table 15 Sampling Method Requirements

### **12.1 FIELD CUSTODY**

Sampling personnel will be responsible for the maintenance and completion of field log books that contain all the required field sheets, sampling custody information, and documentation. Detailed description of information contained in the field logs is presented in Section 9. Field logbooks will be maintained at the office, and all data will be transferred to electronic format for submittal to the LARWQCB.

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Sample containers will be pre-labeled prior to field events, to the extent possible. Sample labels are labeled with a standardized sampling code to ensure that results are easily interpreted and each sample is uniquely codes. The format for sample identifications are as follows:

LAILG-NGA-(NGA ID number)-(sequentially numbered sampling event)

- The NGA number is unique to each member and will not be repeated throughout the life of the program
- The sequentially numbered sampling event will only be recorded if a physical sample is collected for the location, and will be sequential through the life of the program.
- Quality control samples will be labeled EB for Equipment Blank, FB for Field Blank, and Dup for duplicate.

The COC and field forms will contain the following information, at a minimum:

- Site name
- Sample ID and type
- Date
- Sample time
- Sampling personnel
- Analytical and preservative requirements

Upon sample collection in the field, all sample containers will be checked for tightness and labeling, and will be placed immediately on ice. The ice chest will be maintained at approximately 2-4 °C. When the ice chest has been filled, it will be labeled and sealed to be transferred for storage. While in storage, all samples will be placed in a refrigerator maintained at 2-4 °C until the time of shipping or laboratory courier pickup.

### **12.2 CHAIN-OF-CUSTODY FORM**

Sample custody will be traceable from the time of sample collection until results are reported. When samples are transferred from one sampler to another member of the same organization or from the LAILG to Weck and/or ABC, a COC form will be used. This form will identify the site name, sample location, sample number, matrix, date and time of collection, the sampler's name, sampling equipment and sampling type, and method to preserve sample (if any). It will also indicate the date and time of transfer, and the name and signature of the sampler and sample recipient. When quality control checks are performed by Weck and/or ABC, their samples will be processed under their COC procedures with their labels and documentation procedures. Examples of COCs are included in Appendix D.

### **12.3 SAMPLE SHIPMENTS AND HANDLING**

The field sampling team will have custody of each sample until the time of return to the office or delivery to the analytical laboratory. A COC form that identifies the contents will accompany all

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sample shipments. The original COC form will accompany the shipment in a waterproof Ziploc bag, and a copy will be retained in the project file.

All shipping containers will be secured with COC seals for transportation to Weck and/or ABC. All samples will be examined to verify proper sealing and that there are no leaks or breaks. The samples will be placed on artificial ice inside an ice chest to maintain the temperature between 2-4 °C. The artificial ice packed with samples will be sealed in Ziploc bags, and will contact the samples. All samples will be packaged in bubble wrap or foam containers to minimize the chance of breaking. Prior to shipping, the ice chest will be secured shut with tape to prevent accidental spillage. Samples will be shipped to Weck and/or ABC according to Department of Transportation standards.

#### **12.4 LABORATORY CUSTODY PROCEDURES**

The laboratory will follow sample custody procedures as outlined in their quality assurance manual. Activities will be documented to ensure samples are analyzed within the proper time frame for each analytical requirement. At minimum, Weck and/or ABC will conduct the following:

- Initial sample login and verification of samples received with the COC form
- Document any discrepancies noted during login on the COC
- Initiate internal laboratory custody procedure
- Verify sample preservation
- Notify the project coordinator if any problems or discrepancies are identified
- Properly store samples, including daily refrigerator temperature monitoring and sample security.

All laboratory procedure documents and records will be stored at the analyzing lab. Sample volumes remaining after analysis will be stored for at least 30 days after analysis, and will be disposed of appropriately after this time.

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#### **13.0 ANALYTICAL METHODS**

Laboratory analytical methods and MRLs are summarized in Table 11 through Table 14.

#### **13.1 CHEMISTRY ANALYSES**

Pesticide analyses will be conducted on unfiltered (whole) sample fractions. Prior to the analyses of environmental samples, Weck will demonstrate the ability to meet the minimum performance requirements for each analytical method. Initial demonstration of laboratory capabilities includes the ability to meet the project specified quantitation limits, to the extent practical, the ability to generate acceptable precision and recoveries, and other analytical and quality control parameters as stated in this QAPP. Analytical methods used for chemistry analysis must follow a published method (EPA or Standard Method for the Examination of Water and Wastewater) and document the procedure for sample analyses in a laboratory SOP for review and approval. The Laboratory Quality Assurance Manual for general water quality constituents and pesticides is presented in Appendix E.

#### **13.2 TOXICITY TESTING**

Runoff samples will be analyzed for toxicity for three test species: fathead minnow, ceriodaphnia, and green algae. The first monitoring event will analyze for all three species, and successive monitoring events will analyze for the most susceptible species. All toxicity testing will be conducted on 100 percent sample. The ambient water toxicity test results will provide a reliable qualitative prediction of impacts in stream biota. At a minimum the toxicity testing will include the four-day static renewal procedures described in Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms (US EPA, 2002). ABC is responsible for complying with the EPA methods. Laboratory SOPs for toxicity testing are presented in Appendix F.

The results of toxicity testing will be used to trigger further investigation to determine the cause of the observed laboratory toxicity. If testing indicates the presence of significant toxicity in the sample, TIE procedures shall be initiated to investigate the cause of toxicity. For the purpose of triggering TIE procedures, significant toxicity is defined as at least 50 percent mortality or a 50 percent reduction in growth. The 50 percent threshold is consistent with the approach recommended in guidance published by US EPA for conducting TIEs, which recommends a minimum threshold of 50 percent mortality because the probability of completing a successful TIE decreases rapidly for samples with less than this level of toxicity. TIE methods will generally adhere to documented EPA procedures, as outlined in Understanding and Accounting for Method Variability in Whole Effluent Toxicity Applications Under the National Pollutant Discharge Elimination System Program (2000) and Clarification Regarding Toxicity Reduction and Identification Evaluations in the National Pollutant Discharge Elimination System Program (March 27, 2001).

Adequate sample volume will be collected so that TIE procedures can be initiated as soon as possible after toxicity is observed. This will reduce the potential for loss of toxicity due to

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extended sample storage and will therefore increase the likelihood that the toxicant will be identified.

#### **13.3 DETECTION AND REPORTING LIMITS**

Weck will routinely conduct MRL studies to document that the MRLs are less than the project specified MRL objectives. If the MRLs for any analytes exceed the project MRL objectives, the following steps will be taken:

- 1. Perform a new MRL study using concentrations sufficient to prove analyte quantitation at concentrations less than the project specified MRLs per the procedure for the Determination of the Method Detection Limit presented in Revision 1.1, 40 CFR 136, 1984.
- 2. No samples may be analyzed until the issue has been resolved. MRL studies results must be available for review during audits, data review, or as requested. Current MRL study results must be reported at the beginning of every project for review and inclusion in project files.

An MRL is developed from seven aliquots of a standard containing all analytes of interest spiked at three to five times the expected MRL, which are taken through the analytical method sample processing steps. The data is evaluated and used to calculate the MRL. If the calculated MRL is less than three times below the spiked concentrations, another MRL study must be performed using a lower concentration.

Weck will establish MRLs lower than the proposed or existing numeric water quality objectives as determined by the LARWQCB, to the extent possible by the analyzing laboratory. Occasionally samples may be diluted for analysis; this will raise the MRLs for the diluted sample, and is unavoidable on occasion. If samples have to be continually diluted to perform analysis, the analyzing laboratory will contact the Project Manager to put procedures in place to modify the existing analytical protocol. Water quality objectives, as outlined in the CWIL, are presented on Table 3 and Table 4. Weck will have documentation to support quantitation at the attainable levels.

#### **13.4 LABORATORY STANDARDS AND REAGENTS**

All stock standards and reagents used for extraction and standard solutions will be tracked through Weck. The preparation and use of all working standards will be recorded in laboratory files that document standard tractability to National Institute for Standards and Technology criteria. Records will have sufficient detail to allow determination of the identity, concentration, and viability of the standards including all dilutions performed to obtain the working standard. Date of preparation, analyte or mixture, concentration, name of preparer, lot number, and expiration date (if applicable) must be recorded on each working standard.

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#### **13.4 ALTERNATE LABORATORIES**

If laboratories are unable to perform analysis as outlined in this QAPP due to unforeseen problems, an alternate laboratory will be chosen to provide analysis. The selection of alternate laboratories will be the responsibility of the Project Manager and QA Officer.

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#### **14.0 QUALITY CONTROL**

The types of quality control assessments required in the monitoring program are discussed below. General procedures for preparation and analysis of quality control samples are provided in Weck and ABC Laboratory's Quality Assurance Manuals, which are available upon request.

#### 14.1 QUALITY ASSURANCE OBJECTIVES AND DEVELOPMENT

QA objectives are the detailed QC specifications for precision, accuracy (bias), sensitivity and resolution, comparability, representativeness, and completeness, as discussed in Section 7. The quality assurance objectives will be used as comparison criteria during data quality review by LAILG to evaluate if minimum requirements have been met and the data may be used as planned.

#### **14.2 INTERNAL QUALITY CONTROL**

Internal QC is achieved by collecting and/or analyzing a series of duplicate, blank, spike, and spike duplicate samples to ensure that analytical results are within the specified QC objectives. The QC sample results are used to quantify precision and accuracy and identify any problems or limitation in the associated sample results. The internal QC components of a sampling and analysis program will ensure that the data of known quality are produced and documented. The internal QC samples, frequency, acceptance criteria, and corrective action will meet the minimum requirements presented in the following sections.

#### **14.3 FIELD QUALITY CONTROLS**

Field QC samples are used to assess the influence of sampling procedures and equipment used in sampling. They are also used to characterize matrix heterogeneity.

For basic water quality analyses, quality control samples, prepared in the field, will consist of equipment blanks, field blanks, and field duplicates (when applicable). Field measurements will be taken in triplicate to ensure accuracy.

#### 14.3.1 Equipment Blanks

Equipment blanks will be collected at a rate of one per 10 normal samples or every other year, whichever is greater. Equipment blanks will be collected and analyzed for all analytes of interest along with the associated environmental samples. Equipment blanks will consist of distilled blank water (contaminant free) processed through the sampling equipment prior to field use using the same procedures for environmental samples. This is used to demonstrate that equipment is free of contamination, and will help to identify anomalous sources of data.

#### 14.3.2 Field Duplicates

Field duplicates will be collected at a rate of one per 10 normal samples or every other year, whichever is greater. Field duplicates will be collected at the same time as environmental samples,

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or as two grab samples collected in rapid succession, and will be analyzed along with the associated environmental samples. If the RPD of field duplicate results is greater than 25 percent and the absolute difference is greater than the Method Reporting Limit (MRL), both samples should be reanalyzed, if possible. This is used to insure the precision of sampling and analytical methodologies.

#### 14.3.3 Field Blanks

Field blanks will be collected at a rate of one per 10 normal samples or every other year, whichever is greater. Field blanks will consist of laboratory-prepared blank water (certified contaminant free) processed through the sampling equipment in the field using the same procedures for This is used to demonstrate that sampling and decontamination environmental samples. procedures do not result in contamination of the samples and will help to identify anomalous sources of data.

Table 16 Presents the field quality controls discussed above, and Table 17 present the quality controls utilized for field measurements.

LABORATORY QUALITY CONTROL	FREQUENCY	MEASUREMENT QUALITY OBJECTIVES	CORRECTIVE ACTION			
Field Duplicate	10% of total project sample count	RPD<25% (n/a if native concentration of either sample <rl)< td=""><td>Visually inspect the samples to determine if a high RPD between results could be attributed to sample heterogeneity. For duplicate results due to matrix heterogeneity, or where ambient concentrations are below the reporting limit, qualify the results and document the heterogeneity. All failures should be communicated to the project coordinator, who in turn will follow the process detailed in the method.</td></rl)<>	Visually inspect the samples to determine if a high RPD between results could be attributed to sample heterogeneity. For duplicate results due to matrix heterogeneity, or where ambient concentrations are below the reporting limit, qualify the results and document the heterogeneity. All failures should be communicated to the project coordinator, who in turn will follow the process detailed in the method.			
Field Blanks	10% of total project sample count	<rl analyte<="" for="" target="" td=""><td>Investigate the source of contamination. Potential sources of contamination include sampling equipment, protocols, and handling. The laboratory should report evidence of field contamination as soon as possible so corrective actions can be implemented. Samples collected in the presence of field contamination should be flagged.</td></rl>	Investigate the source of contamination. Potential sources of contamination include sampling equipment, protocols, and handling. The laboratory should report evidence of field contamination as soon as possible so corrective actions can be implemented. Samples collected in the presence of field contamination should be flagged.			
Equipment Blanks	10% of total project sample count	<rl analyte<="" for="" target="" td=""><td>Investigate the source of contamination. Potential sources of contamination include sampling equipment, protocols, and handling. The laboratory should report any evidence of field contamination as soon as possible so corrective actions can be implemented. Samples collected in the presence of field contamination should be flagged.</td></rl>	Investigate the source of contamination. Potential sources of contamination include sampling equipment, protocols, and handling. The laboratory should report any evidence of field contamination as soon as possible so corrective actions can be implemented. Samples collected in the presence of field contamination should be flagged.			

#### Table 16 Field Samples Quality Control

#### **Field Quality Control**

MDL Method Detection Limits GWQC General Water Quality Constituents LCS Laboratory control sample

RPD RL

Relative Percent Difference Reporting Limit

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#### Table 17 Field Samples Quality Control

#### Quality Control<sup>1</sup> for Field Measurements

LABORATORY QUALITY CONTROL	RECOMMENDED DEVICE	UNITS	RESOLUTION	INSTRUMENT ACCURACY SPECS	POINTS PER CALIBRATION	PRE-SAMPLING CALIBRATION CHECK FREQUENCY <sup>2</sup>	POST-SAMPLING CALIBRATION CHECK FREQUENCY <sup>2</sup>	ALLOWABLE DRIFT <sup>3</sup>	CORRECTIVE ACTION
pH	Electrode	pH	0.01	±0.2	2	Per manufacturer	Per manufacturer	±0.2 units	The instrument should be recalibrated
Specific Conductance (Conductivity)	Conductivity cell	$\mu S/cm^{\ast}$	1	±0.5%	Per manufacturer	Per manufacturer	Per manufacturer	±10%	following manufacturer cleaning and maintenance procedures. If measurements
Temperature	Thermistor or bulb	°C	0.1	±0.15%	Per manufacturer	Per manufacturer	Per manufacturer	±0.5%	continue to fail measurement quality
Dissolved Oxygen	Polarographic or luminescence quenching probe	mg/L	0.01	±0.2	1	Before every monitoring day onsite (re-calibrate if change of elevation is >500 m or barometric pressure > 2mmHg)	After every monitoring day (within 24 hours)	±0.5 or 10%	objectives, affected data should not be reported and the instrument should be returned to the manufacturer for maintenance. All troubleshooting and
Turbidity	Portable turbidimeter or optical probe	NTU	0.1	±1% up to 100 NTU; ±3% from 100-400 NTU; and ±5% from 400-3000 NTU	2	Per manufacturer	Per manufacturer	0.01±0.2 or 10%	corrective actions should be recorded in calibration and field data logbooks.

mg/L Milligrams per Liter μS/cm Microsiemens per Centimeter NTU Nephelometric Turbidity Unit

mS/cm for marine water

°C Degrees Celsius

1 Unless manufacturer specifies more stringent requirements

2 SWAMP requires daily pre- and post-sampling calibration checks when the manufacturer or documented procedure (e.g., standard operating procedure) do not provide calibration instructions 3 Unit or percentage, whichever is greater

3 Unit or percentage, whichever is greater

4 Electromagnetic meters should undergo periodic maintenance according to manufacturer instructions

5 Price AA meter: spin test>2 minutes; pygmy meter: spin test>45 seconds; electromagnetic meter: zero check

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#### **14.4 LABORATORY QUALITY CONTROL**

For basic water quality analysis, quality control samples prepared by Weck will typically consist of method blanks, laboratory duplicates, laboratory control samples, matrix spikes and matrix spike duplicates, and surrogate spikes added to each sample (organic analysis).

#### 14.4.1 Method Control Blanks

Method blanks will be prepared and analyzed by Weck with each batch of samples. If any analyte is detected in the blank, the blank and associated samples must be re-analyzed after the source of contamination is identified.

#### 14.4.2 Laboratory Duplicates

Laboratory duplicates will be analyzed by Weck with each batch of samples, and will consist of replicate environmental samples. If the RPD of laboratory duplicate results is greater than laboratory standards and the absolute difference is greater than the MRL, both samples should be reanalyzed, if possible. This is used to insure the precision of sample preparation and analytical methodologies.

#### 14.4.3 Laboratory Control Samples and Surrogate

Laboratory Control Samples (LCS) will be analyzed at the rate of one sample per batch. LCS will consist of a standard reference material or a laboratory fortified method blank. If recovery of any analyte is outside the acceptable range, the laboratory must locate the source of the problem, and the LCS and associated samples must be re-analyzed after the source of the problem is fixed. This is used to insure the accuracy of sample preparation and analytical methodologies.

#### 14.4.4 Matrix Spikes and Matrix Spike Duplicates

Matrix spikes and matrix spike duplicates will be analyzed at a rate of one pair per sample batch. An additional 6 liters of water will be collected from one sampling site each event in order to supply the lab with enough sample to run matrix spikes and matrix spike duplicates. Matrix spike samples consist of a laboratory fortified environmental sample that is spiked at Weck. Spike concentrations are added at approximately five to ten times the reporting limit of the target analyte. If the matrix spike recovery of any analyte is outside the acceptable range, the laboratory must locate the source of the problem, and the matrix spike and associated samples must be re-extracted and re-analyzed after the source of the problem is fixed. If the matrix spike duplicate RPD is greater than the laboratory standards and the absolute difference is greater than the MRL, the laboratory must locate the source of the problem, and the problem, and the matrix spikes and associated samples must be re-analyzed after the source of the problem is fixed. This is used to insure the performance of sample preparation and analytical methodologies.

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#### 14.4.5 Surrogate Spikes

Surrogate spikes are used to evaluate the compounds added by the laboratory to samples to prepare them for analysis. They will be utilized on a sample-specific basis, and are applicable to the analysis of all the pesticides. This will verify the accuracy of analytical measurements for organic analyses.

#### 15.0 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

#### **15.1 EQUIPMENT/MATERIALS CLEANING PROCEDURES**

Weck and ABC will clean and prepare containers used for sample collection prior to each monitoring event in accordance with SOP documents for each analysis. Sample containers will be sealed after preparation and will not be opened until the sampling event. All equipment will be inspected prior to use and after sampling for damage, and any damage will be recorded. Cleaned equipment will be used to generate the equipment blanks, as outlined in Section 14.

Proper hygene must be practiced at all times to minimize the potential for spreading pathogens or invasive species between sampling sites. All sampling equipment that comes into contact with the sample water or immediate surroundings must be decontaminated between sites.

#### **15.2 FIELD MONITORING EQUIPMENT**

Monitoring personnel will be responsible for testing, cleaning, and maintaining all water quality monitoring equipment and sample collection equipment in accordance with manufacturer specifications and standard decontamination procedures.

# **15.3 ANALYTICAL INSTRUMENT/EQUIPMENT TESTING PROCEDURES AND CORRECTIVE ACTIONS**

Analytical equipment used by the laboratory will be tested, maintained, and inspected according to the QA manual for each analyzing laboratory.

#### 16.0 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

#### 16.1 LABORATORY ANALYTICAL PROCEDURES AND CALIBRATION

Calibration frequencies and procedures for laboratory analytical equipment used by the laboratory are outlined in the QA manual. Any deficiencies in analytical equipment calibration will be dealt with accordingly and will be reported to the project QA Officer.

For this program, only linear calibration with either an average response factor or a linear regression is acceptable for organic analysis. Non-linear calibration is not allowed since using this calibration option could generate a potential for poor quantitation or biased concentration of compounds at low or high concentrations (near the high and low ends of the calibration range).

BC will construct an initial calibration curve using a minimum of five calibration points for organic analytes and three calibration points for inorganic analytes, where the low-level standard concentrations is less than or equal to the analyte quantitation limits.

#### **16.2 FIELD MONITORING EQUIPMENT**

All field monitoring equipment will be calibrated according to manufacturer specifications, or before each field monitoring event, whichever is greater. A description of calibration procedures and frequency is presented on Table 18. The rental company that supplies the meter or sampling personnel will be responsible for the calibration of the equipment. If calibration results do not meet manufacturers specifications, and subsequent recalibration is not acceptable, spare equipment will be calibrated and used for the monitoring event. All discrepancies in calibration will be noted in the field monitoring log.

Results and frequency of calibration will be recorded on a calibration log sheet (Appendix G).

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Field Monitoring Parameter	Calibration Description and Criteria	Frequency of Calibration	Responsible Person	
рН	Use standard buffering solutions, including high, low, and mid range measurements			
Temperature	Is factory set, but will be verified by a standard glass thermometer	Prior to rental, day of		
Electrical Conductivity	Will be corrected by comparing readings to known conductivity standards	sampling event, or by manufacturers specifications,	Rental Company or Sampling personnel	
Dissolved Oxygen	Will be calibrated according to manufacturer specifications	whichever is greater		
Turbidity	Use standard solution, including high, low, and mid range measurements			

Table 18 Field Equipment Calibration

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#### 17.0 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Sampling personnel will inspect all equipment and materials used during field monitoring events prior to monitoring and sampling. Any equipment or materials that show signs of damage or contamination will not be used for sampling events and will be decontaminated accordingly. A complete equipment list is presented in Appendix A. Inspection protocols for laboratory materials, equipment, and reagents are documented in the QA manual for the laboratory.

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#### **18.0 NON-DIRECT MEASUREMENTS**

Data from prior studies and other monitoring programs may be used for data evaluation purposes. Data used will be reviewed in accordance with the data quality objectives as stated in this QAPP, and will be deemed either acceptable or unacceptable by the QA Officer.

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#### **19.0 DATA MANAGEMENT**

Data will be maintained as outlined in Section 9. Copies of field logs, copies of COC forms, original preliminary and final lab reports, and electronic media reports will be kept for review by the LARWQCB. LAILG will retain original field logs. Weck and ABC will retain original COC forms. Weck and ABC will also retain copies of the preliminary and final data reports.

Field log sheets are checked and signed in the field by sampling personnel. Sampling personnel will identify any results that exceed holding times, sample identification information is incorrect, samples were inappropriately handled, or calibration information is missing or inadequate. Such data will be marked as unacceptable by sampling personnel and will not be entered into the electronic database. All acceptable data will be entered electronically into a database and stored.

Weck and ABC will report their results to the Project Manager. The Project Manager will verify sample identification information, review the COC forms, and identify the data appropriately in the database.

Concentrations of chemicals and toxicity endpoints, and all numerical biological parameters will be calculated as described in the referenced method document for each analyte or parameter, or a SOP. The data generated will be converted to a standard database format maintained by LAILG and available to the LARWQCB for review. After data entry or data transfer procedures are completed for each sample event, data will be inspected for data transcription errors, and corrected as appropriate. After the final QA checks for errors are completed, the data will be added to the final database.

# ASSESSMENT AND OVERSIGHT

#### 20.0 ASSESSMENT AND RESPONSE ACTIONS

Data will be consistently assessed and documented to evaluate whether project QA objectives have been met, to quantitatively assess data quality, and to identify potential limitations on data use. Assessment and compliance with quality control procedures will be undertaken during the data collection phase of the project. Field personnel will assess the performance of sampling procedures, and will report to the Project Manager on any corrective actions. Individual laboratories are responsible for the assessment of analytical equipment maintenance, calibration, analytical methods, and QC results presented in the Quality Assurance Manual and SOPs, and will report corrective actions to the project QA Officer.

Upon return from field sampling, field data sheets, COC documentation, sample numbers, sample holding times, QC samples, and other documents will be assessed by LAILG's QA Officer to insure proper procedures and documentation were followed. The QA Officer will review laboratory analytical data for spike and duplicate precision and accuracy, assign data flags, calculate completeness, and evaluate any indication of contamination.

If any discrepancies are encountered during assessment or during an audit, the QA Officer will address the discrepancy with the appropriate personnel. The discussion will involve whether the data collected is accurate, the possible causes leading to the deviation, how the deviation could affect data quality, and what corrective actions are to be considered. A log will be maintained outlining all discussion points and corrective actions, and will be available to the LARWQCB and the Project Manager upon request.

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#### **21.0 REPORTS TO MANAGEMENT**

In addition to information outlined in Section 9, LAILG will also generate the following reports:

#### **21.1 ANNUAL MONITORING REPORT**

The AMR will be prepared annually by the Project Manager and will include the following sections:

- 1. Introduction: title page, table of contents, description of group membership, updated membership list, and objectives of AMR.
- 2. Monitoring: location of samples collected, descriptions and photographs of sampling sites, location map of sampling sites including GPS coordinates of sampling sites, constituents monitored and frequency, objective, and analytical methods.
- 3. Results and Discussion: tabulated data, summary of toxicity exceedances, summary of data to demonstrate compliance or non-compliance, comparison of data to benchmark values in the CWIL (Order No. R4-2010-0186, Appendix 2), quality control results, data interpretation.
- 4. Quality control data interpretation and affirmation that analyses were conducted by a certified laboratory.
- 5. Perjury Statement.
- 6. Conclusion and recommendations. Including a statement of intent to prepare a WQMP if Water Quality Benchmarks are not attained.
- 7. References and Appendices including, but not limited to, documentation that education requirements have been fulfilled by each member of the LAILG, copies of field data/sample log sheets, COC forms and laboratory and field quality control samples results.

#### **21.2 WATER QUALITY MANAGEMENT PLAN**

If Annual Monitoring Reports indicate non-compliance with one or more established CWIL benchmarks, a WQMP will be implemented. The WQMP will include the following sections:

- 1. Introduction: title page, table of contents, objectives of WQMP.
- 2. Monitoring: samples collected, location, benchmarks exceeded, and analytical methods.

- 3. Results and Discussion: tabulated data; summary of data to demonstrating compliance and non-compliance; evaluation of site conditions and information to determine possible source of benchmark exceedance; evaluation of time, frequency, and flow direction of contaminants; assessment of impacts of waste discharges from irrigated lands to waters of the state, list existing and possible best management practices to mitigate problem.
- 4. Describe a revised MRP to assess efficiency of management practices.
- 5. Conclusion and recommendations.
- 6. References and Appendices, if necessary.

Table 19 outlines the timetable for report submittal to management.

Table 19 Report Submittals

Type of Report	Frequency	Projected Delivery Date	Organization Responsible for Report Preparation	Report Recipients	
Lab Analytical Data Reports	Per Sampling Event	Within thirty days of sampling event	Analytical Laboratories	LAILG/Pacific Ridgeline	
Annual Monitoring Report	Annually	December 15th	PacRL	LARWQCB	
Water Quality Management Plan	Annually, if necessary	With Annual Monitoring Report, if necessary. Final Report for	LAILG and Pacific Ridgeline	LARWQCB	
	Final on October 31, 2020	CWIL on October 31, 2020			

# DATA VALIDATION AND USABILITY

#### 22.0 DATA REVIEW, VERIFICATION, AND VALIDATION

Applicable data generated during this project will be reviewed against the data quality objectives outlined in Sections 7, 14, 15, 16, and 17 of this report. Data that does not meet all quality assurance/quality control objectives will be analyzed to assess their potential impacts on data quality. Once their impacts have been evaluated, the data will either be included or thrown out of the data set. Only data that has met data quality objectives, or data that has acceptable deviations explained, will be submitted. All data that is included, but does not meet all data quality objectives, will be flagged in the reports. When QA requirements have not been met, the samples will be reanalyzed when possible and only the results of the reanalysis will be submitted, provided they are acceptable.

The laboratory QA Officer will be responsible for verifying data generated by the laboratory meets data quality objectives. Once complete laboratory analytical results are received, LAILG's QA Officer will independently review and validate the data, including reviews of precision, accuracy, and completeness. LAILG's QA Officer is ultimately responsible, based on the review of data, to accept or qualify data.

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#### 23.0 VERIFICATION AND VALIDATION METHODS

Data will be visually checked and recorded upon initial sampling and field monitoring, and will be checked again upon return. This verification will include review of all sample numbers and collection logs, sample preparation, field logbooks, COC forms, calculation worksheets, and raw data. The QA Officer will be responsible for the review of all the data records upon return to the office.

The laboratory QA manual will be used to accept, reject, or qualify the data generated by Weck and ABC. The laboratory management will be responsible for validating the data generated by the laboratory.

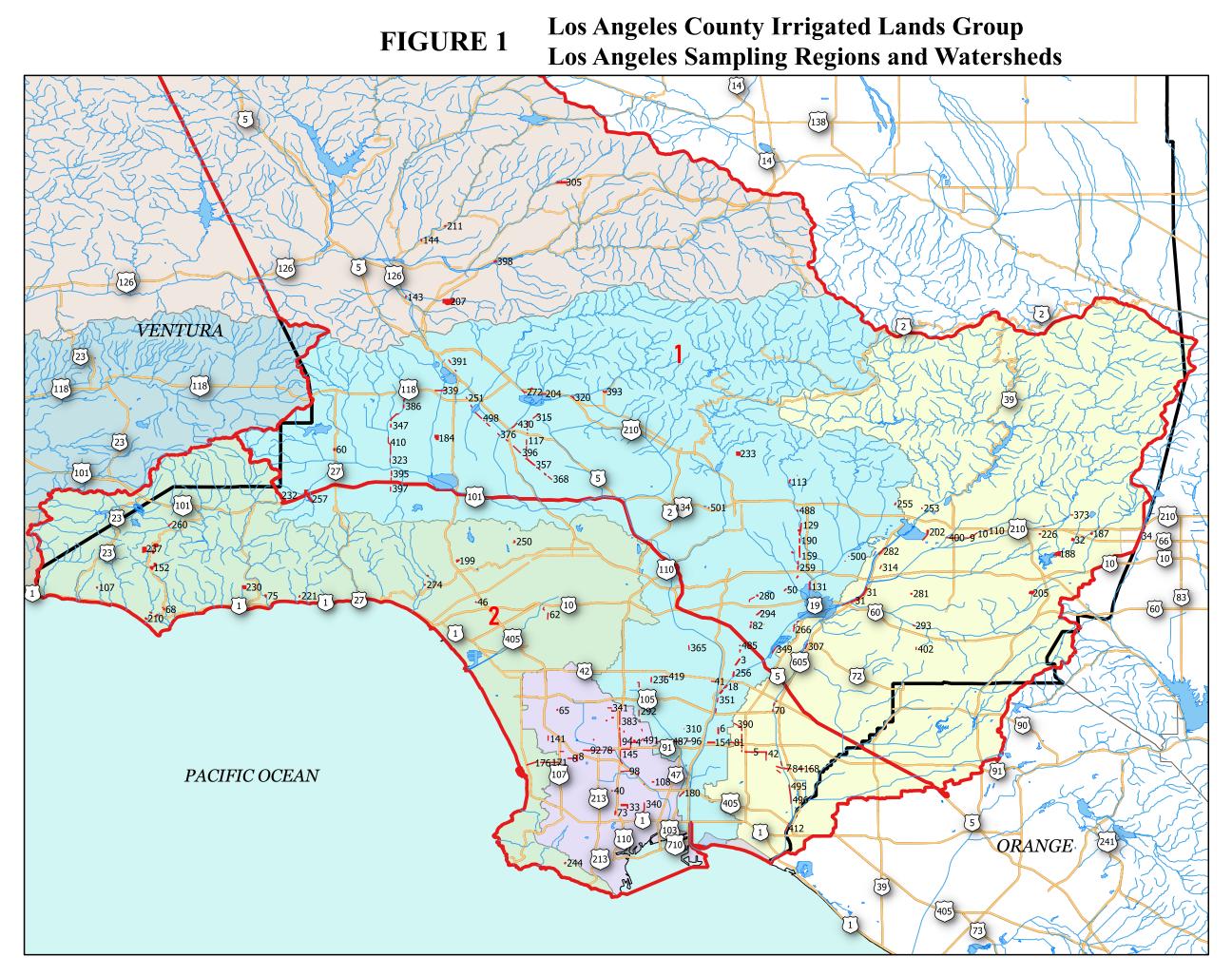
The laboratory personnel will verify that the measurement process was "in control" (i.e., all specified data quality objectives were met or acceptable deviations explained) for each batch of samples before proceeding with analysis of a subsequent batch. In addition, each laboratory will establish a system for detecting and reducing transcription and/or calculation errors prior to reporting data.

Upon receipt of laboratory analytical data, LAILG's QA Officer will be responsible for reviewing the sample analyses, documentation, data collection processes, sample preparations, and sample collection for completeness. This includes checking for data flags, correct application of dilution factors and conversion factors, and transcription errors.

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#### **24.0 REFERENCES**

- United States Environmental Protection Agency (USEPA). 1983. *Methods for Chemical Analysis of Water and Wastes (third edition)*. EPA-600/4-79-020.
- USEPA. 1988. *Methods for Determination of Organic Compounds in Drinking Water*. EPA-600/4-88/039.
- USEPA. 1991. Methods for Aquatic Toxicity Identification Evaluations: Phase 1 Toxicity Characterization Procedures (second edition). EPA-600/6-91/003.
- USEPA. 1992. Toxicity Identification Evaluation: Characterization of Chronically Toxic Effluents Phase I. EPA-600/6-91-005.
- USEPA. 1993a. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms (fourth edition). EPA/600/4-90/027F.
- USEPA. 1993b. Methods for Aquatic Toxicity Identification Evaluations: Phase II Toxicity Identification Procedures for Samples Exhibiting Acute and Chronic Toxicity. EPA/600/R-92/080.
- USEPA. 1996. Marine Toxicity Identification Evaluation (TIE): Phase I Guidance Document. EPA-600/R-96/054.
- USEPA. 2000. Water Quality Standards; Establishment of Numeric Criteria for Priority Toxic Pollutants for the State of California. 40-CFR Part 131.
- USEPA. 2001. Laboratory Documentation Requirements for Data Evaluation. R9QA/004.1.
- USEPA. 2000. Understanding and Accounting for Method Variability in Whole Effluent Toxicity Applications Under the National Pollutant Discharge Elimination System Program and Clarification Regarding Toxicity Reduction and Identification Evaluations in the National Pollutant Discharge Elimination System Program.
- USEPA. 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms (fourth edition). Office of Water, Washington, D.C.
- USEPA. 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms
- "Swamp Field Methods Course." Water Board Training Academy. CD-ROM. November 1, 2005.
- LARWQCB. 2005. Conditional Waiver of Waste Discharge Requirements for Discharges from Irrigated Lands Within the Los Angeles Region. Order No. R4-2005-0080.California Department of Fish and Game. 2005. Controlling the Spread of New Zealand Mud Snails on Wading Gear. Office of Spill Prevention and Response, Administrative Report 2005-02

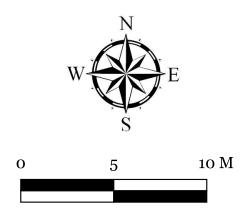


# Legend

- Enrolled Grower and Number
- Sampling Region 1
- Sampling Region 2
- LA County Boundary
  - CA State Road and Numner
- Water Bodies
- Flowlines
- LA Cities

### Watersheds

- Callegus Creek
- Dominguez Channel LA LB Harbor
- Los Angels River
- Misc. Ventura Coastal Stream
- Santa Clara River
- San Gabriel River
- Santa Monica Bay
- 📃 Ventura River

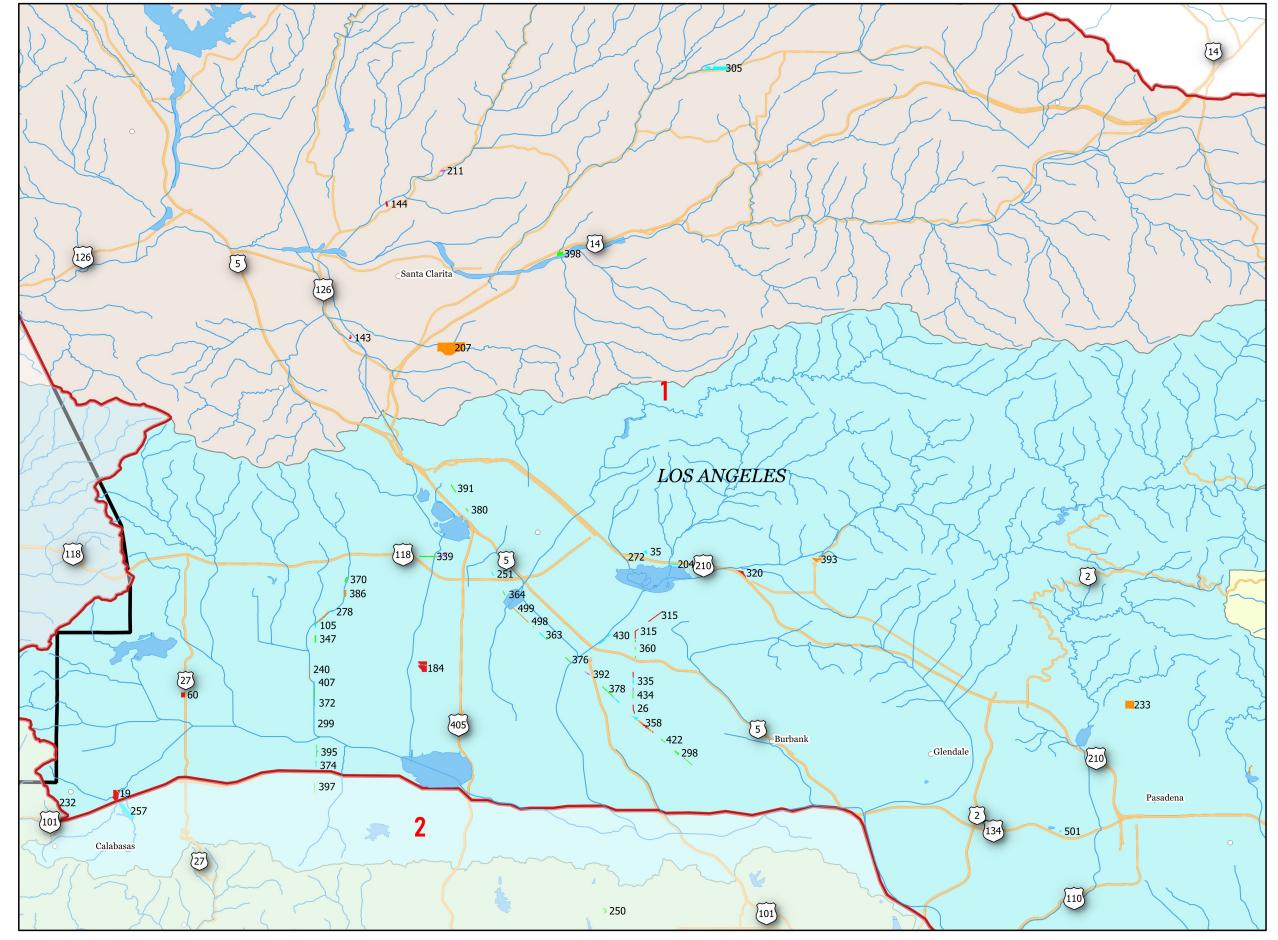


Scale: 1 Inch = 5 Miles



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# Legend

- Sampling Region and Number
  - CA State Roads and Numbers
- LA Cities
- LA County Boundary
- Streams

### Enrolled Growers by Group

- Unknown (No Data)
- Micro Growers
- Small Growers
- Medium Growers
- Large Growers

### Watersheds

- Dominguez Channel LA LBHarbor
- Los Angels River
- Santa Clara River
- San Gabriel River
- Santa Monica Bay
- Ventura River
  - Misc. Ventura Coastal Stream
  - Callegus Creek

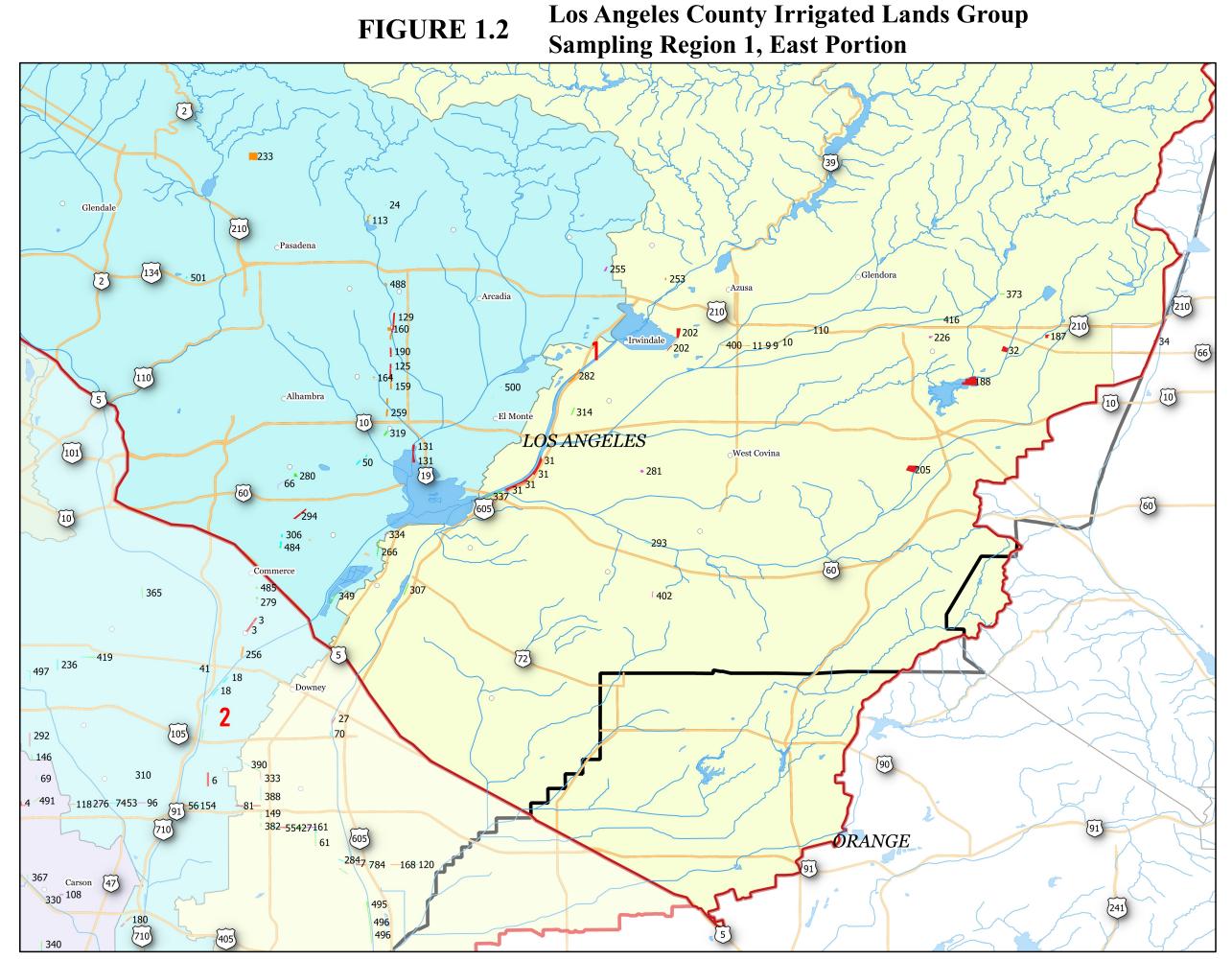




Scale: 1 Inch = 2.5 Miles



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# Legend

- Sampling Region and Number
- LA Cities
- Streams
- CA State Roads and Numbers
- LA County Boundary

Enrolled Growers by Group

- Unknown (No Data)
- Micro Growers
- Small Growers
- Medium Growers
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  - Dominguez Channel LA LBHarbor
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- Santa Clara River
- San Gabriel River
- Santa Monica Bay
- Ventura River
  - Misc. Ventura Coastal Stream
  - Callegus Creek

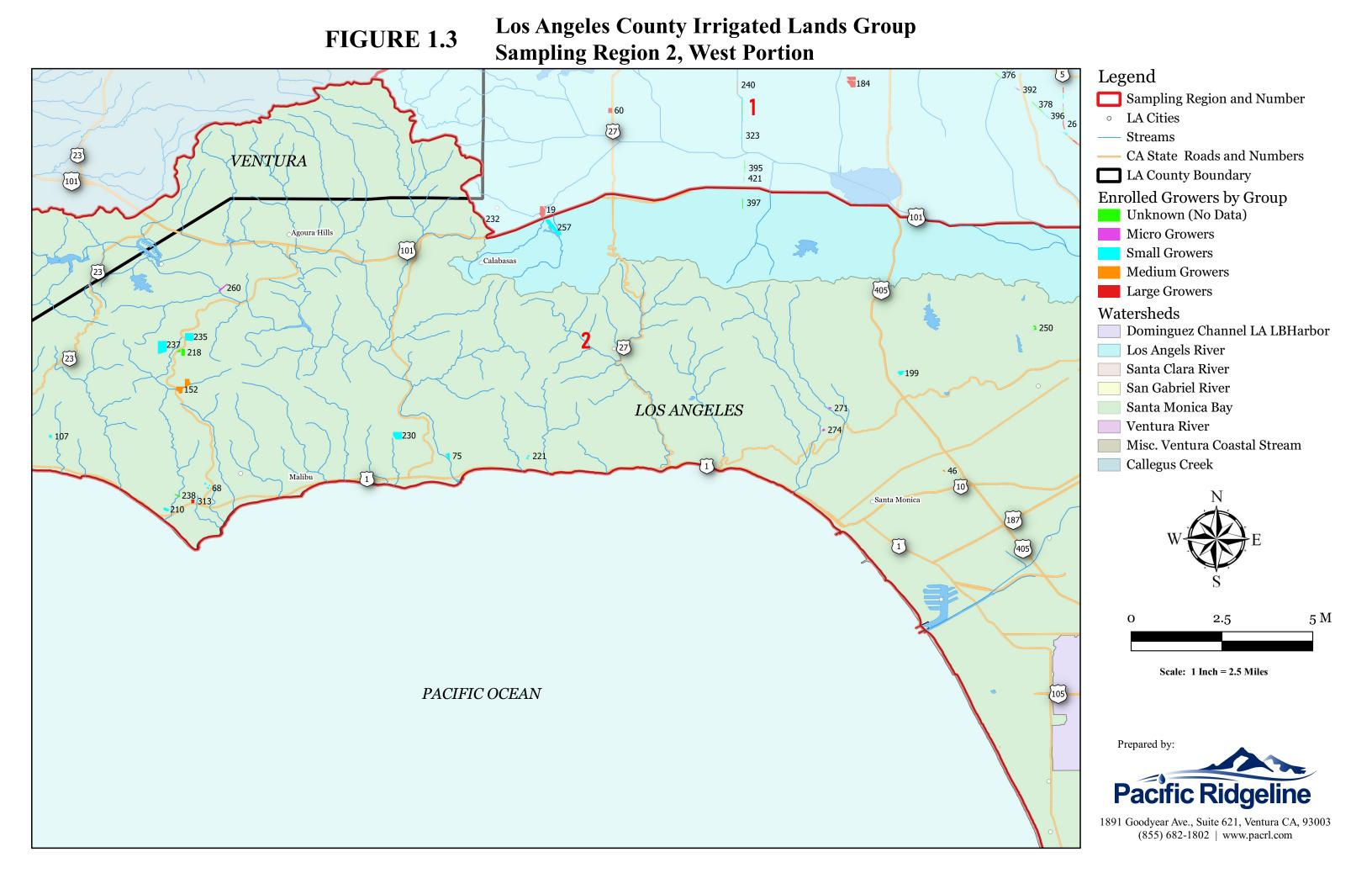


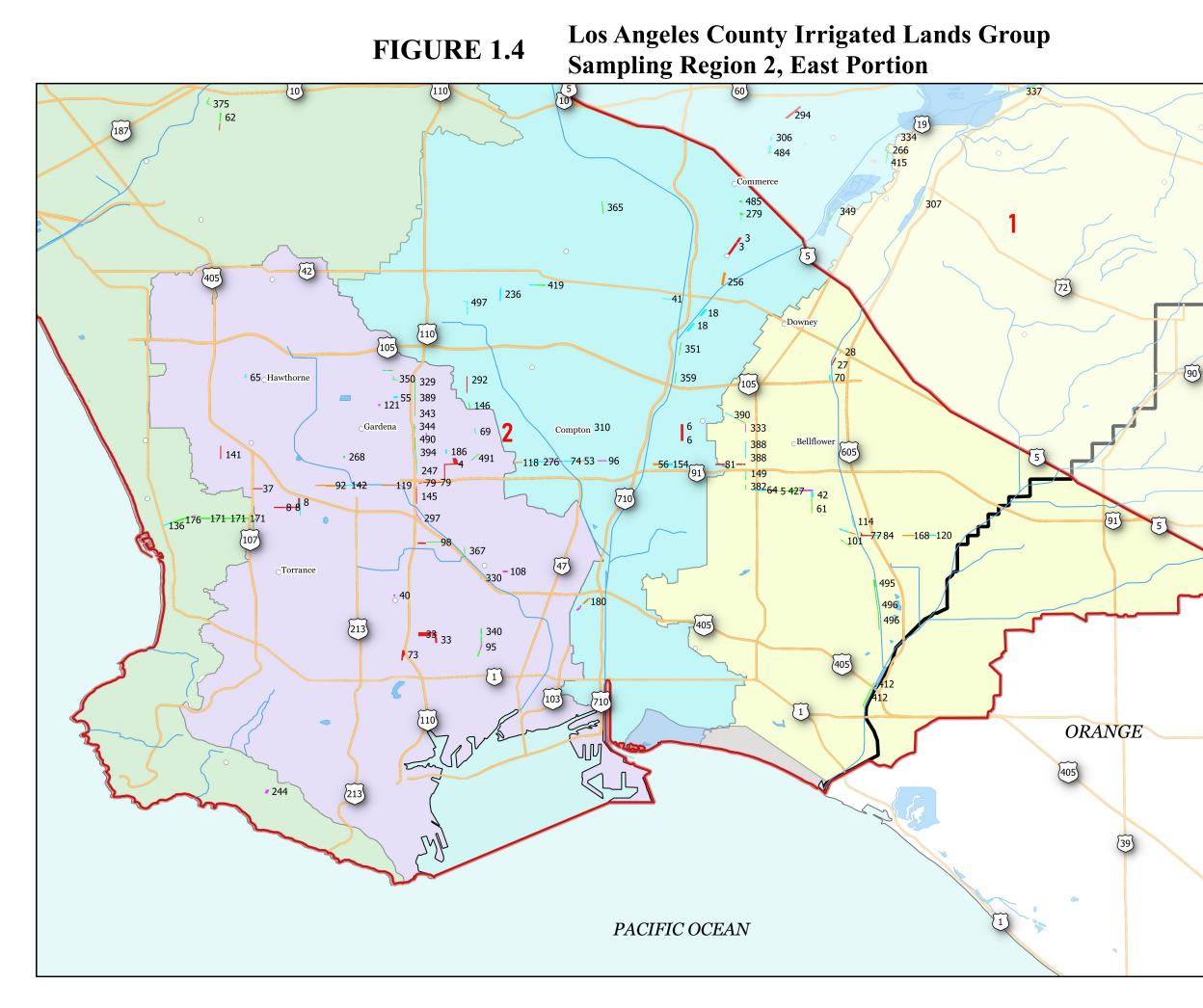


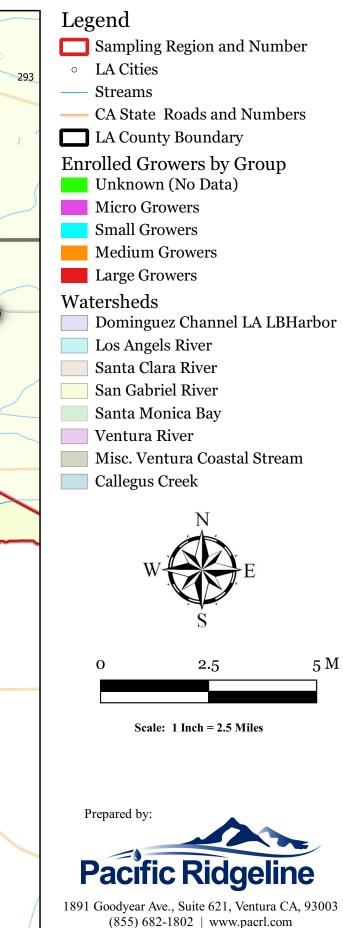
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## **APPENDIX A**

# FIELD LOG SHEETS

#### FIELD EQUIPMENT CHECKLIST

#### SITE: \_\_\_\_\_

# CLIENT CONTACT/PHONE NUMBER:

#### REGULATOR/PHONE NUMBER:

- \_\_\_\_ Copies of MRP, QAPP and NOI
- \_\_\_\_ Proper number of sample container, including extras. Also bubble wrap and zip locks for containers.
- \_\_\_\_ Proper labels, including extras
- \_\_\_\_\_ Field Log Sheets/Field Folders
- \_\_\_\_ COC Forms
- \_\_\_\_\_ Safety equipment and first aid kit, including rain gear
- \_\_\_\_ Field monitoring equipment
- \_\_\_\_ Nitrile gloves and Tyveks
- **\_\_\_\_** Coolers and ice or artificial ice
- \_\_\_\_ Camera, phone, and watch
- \_\_\_\_ Extra pens
- \_\_\_\_ Decontamination equipment
- \_\_\_\_ Trash bags
- \_\_\_\_ Extra deionized water
- \_\_\_\_ Dipping pole and clean secondary container
- \_\_\_\_ Sheet flow sampling device
- \_\_\_\_ Peristaltic pump, plus extra batteries
- \_\_\_\_ Clean sample tubing
- <u>Measuring tape/measuring stick</u>
- \_\_\_\_ other: \_\_\_\_\_

	LOS ANGE	ELES REGI	ON CONDIT	IONAL WAI	VER FOR II	RRIGATE	D LANDS		
			FIEL	D DATA SHE	ЕТ				
Site Name:				Sampling					
Address:				Event:	Dry	Wet	DATE:		
				Weather	Clear	Partly	Cloudy	Light	Raining
				Conditions:		Cloudy	\$7' 1	Precipitation	
				Crop Type:	Tree Farm	Greenhouse	Vinyard	Sod Farm	Row Crops
					Color Plants	General Ornar	nentals		
			Gener	ral Observation					
Pesticide Type:	Motorized	Hand	Granular	Dust	Type of	Drip	Hand	Sprinkler	Other:
Application Time:	Spray	Spray	Application	Application	(Circle all that apply		Tunu	Sprinder	C LINCT
Fertilizer Type:	Topdress	Slow	Irrigation	Mixed	(Circle an that apply	)			
Application Time:		Release	Water						
			Runo	off Observation	S				
Water color:	Clear Light Bro	wn Brown	Other:		Stream Widt	h:		(inches)	
Observations	Debris Trash	Debris Trash Algea Other:			Stream Depth: (inches)				
Water Odor:	Yes No	If Yes,	describe:						
Sample Number	Location	Time (12 Hour)	Type (Circle one)	Velocity (feet per sec)	Temperatu (°C)	re pH	<b>E.C.</b> (uS/cm)	Dissolved Oxygen (mg/L)	Turbidity (NTU)
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		AM PM	Pump Grab Bucket						
		AM PM	Pump Grab Bucket						
		AM PM	Pump Grab Bucket						
		AM PM	Pump Grab Bucket						
Picture Numbers:		_							
Sampling Personnel:									
	(prin	t)			(sign)			(Organization)	

<b>PW ENVIRONMENTAL</b>	1	FIELD REF	ООРТ		D
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Site:	Sampling Event:	Wet	Dry	Date:	
Site Address:	Weather Condition		Partly	Cloudy	
	Visit Duration:	Arrival Time:	Cloudy	AM	Precipitation Project Manager: Zack Moran
	Visit Duration.	Annvai Time.		PM	
		Departure Time:		AM PM	QA Officer: Bryn Home
Nearest Cross Street:	Site Contact:				Prepared By:
Field notes (describe activities, site conditions, run	off pattern, etc.):				
		-			
Site Visitors (inspectors, regulators, utilities):	Agency/Company:	Title:			Phone Number:
This report presents opinion formed as a result of activity observa irrespective of the presence of PW's representative. PW's work d project, unless PW directed site activities or directed by contract	oes not include supervision or d				
Attachments:		-			
Distribution:		-		Signed:	

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### **APPENDIX B**

### STANDARD OPERATING PROCEDURE FOR WATER SAMPLE COLLECTION

#### STANDARD OPERATING PROCEDURES SURFACE WATER SAMPLING

#### **1.0 PREPARATION**

Prior to mobilization for each sampling site, project personnel will prepare the necessary field equipment, verify proper bottle orders, and contact all necessary personnel. Due to the variability of the first wet season sampling event, the following items will be prepared two weeks prior to the start of the season:

- Order all necessary sample containers for each sampling event, and double check sample containers.
- Prepare sample labels.
- Prepare, calibrate, and decontaminate all equipment.
- Prepare and review all field logbooks, including the field log sheets.
- Review and confirm equipment checklist (Appendix A of this QAPP).
- Review SWAMP and SOP for sample collection, storage, and data collection with the sampling crew.
- Pre-select sites for each sampler, and review site specific concerns and site locations.

On the day of sampling, the following will be completed:

- Final confirmation of all equipment and laboratory sample containers.
- Verify all equipment is operating properly.
- Coordinate sample pickup with Weck and ABC.
- Conduct a final review with the sampling team of quality control procedures and specific site issues.

#### 2.0 LABORATORY INTERACTION

Prior to each sampling season, orders will be placed with Weck and ABC for all sampling containers required for the first sampling event, along with all trip blanks, duplicates, and extra containers. All containers will be verified to have the proper preservatives for the analytical methods, if applicable, and are the proper sizes.

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#### 3.0 SAMPLE CONTAINER LABELING

All sample containers will be labeled prior to each monitoring event, to the extent possible. Extra labels will be provided in case field observations dictate a change in the sampling. All labels will contain the following information: group name, site name, sample ID, date, time, sampling personnel, preservatives (if applicable), the laboratory conducting the analysis, and the analytical requirements.

#### 4.0 SAMPLE COLLECTION

All samples will be collected in pre-cleaned sample containers supplied by the laboratory. All secondary containers and sampling equipment will be pre cleaned. Clean nitrile gloves will be used for sample collection, and will be changed as appropriate. Field personnel will adhere to the following protocol to minimize the potential for cross contamination:

- No eating or drinking during sample collection
- Nitrile gloves will be changed for each site
- Tobacco products are prohibited near the samples and sample locations
- Personnel will avoid coughing and sneezing near open sample containers
- Samples are not to be collected near a running vehicle.

The sampling crew will also take the necessary precautions to avoid rainwater or surface drip into the sampling containers, to the extent practical.

Grab samples will be collected at the point of the greatest flow, in the center of the runoff stream, to the extent possible. The preferred method is to directly submerge the sample container, however, site-specific conditions and runoff patterns will dictate the type of grab sample collected. Grab samples will be collected directly into the appropriate containers, as outlined in the QAPP. The expected types of grab sample techniques are as follows.

a. Direct Submersion

Labeled containers will be opened, submerged approximately mid stream, be filled, and then the lid will be secured. Clean nitrile gloves will be worn at all times. The sample will be immediately put on ice, and subsequent samples will be used following the same procedure. After all samples have been collected, the COC form and the field log will be completed, and the samples will be delivered to the laboratory.

b. Intermediate Container

In the case where direct submersion is not practical, an intermediate sample container will be used. The intermediate sample container will be pre-cleaned prior to sample collection, and will be decontaminated in between sampling events. Clean nitrile gloves will be worn at all times. The container could consist of a bucket, a dipping stick, or something that consists of the same composition as the sample containers, depending on site-specific conditions. The intermediate container will be filled through direct submersion, and the sample will be transferred to the appropriate pre-labeled sample containers as quickly as possible. Each sample container will be placed on ice after the containers have been filled. After all samples have been collected, the COC form and the field log will be completed, and the samples will be delivered to the laboratory. In the case of sheet flow from the runoff of a site, direct submersion of the intermediate container will not be possible. In this case, a collection device will be used to collect water to directly fill the sample containers.

#### c. Pumping

In the case where direct submersion or intermediate container sampling is not applicable, a peristaltic pump will be used. A peristaltic pump will not be used to collect samples analyzed for ammonia. The peristaltic pump will use new sample tubing, and will be properly cleaned between sampling events. Clean nitrile gloves will be worn at all times. Precautions will be used to insure that the sample tubing does not come into contact with any surfaces that are known not to be clean. One end of the tubing will be placed approximately mid-stream, in a place that will not collect suspended or settled solids. The other end of the tubing will be placed directly above the sample container to collect the appropriate amount of water. Each sample container will be placed on ice after the containers have been filled. After all samples have been collected, the COC form and the field log will be completed, and the samples will be delivered to the laboratory.

#### **5.0 DIRECT MEASUREMENTS**

Field measurements and observations will be made at each site after the appropriate amount of samples has been collected. Measurements of pH, temperature, dissolved oxygen, conductivity, and turbidity will be conducted using hand held monitoring devices that have been calibrated prior to the measurements. Flow will be monitored as outlined in Appendix C. Field observations will consist of stream odor, color, suspended materials, and other applicable information as deemed by field personnel. An example of a field monitoring log is presented in Appendix A. Measurements will be taken at approximately mid-stream when possible. All measurements and observations will be entered into a field log sheet for each site, and will be transferred to an electronic database.

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#### 6.0 FIELD PROTOCOL

Field teams will consist of one to two persons each, and will mobilize for sampling events when weather conditions are appropriate. Sampling will be completed in daylight hours. Before departing, the number and types of sample containers will be verified. The technician will mobilize to the site, and record general information on the field log sheet. After locating the proper sampling location, the appropriate number of samples and duplicates will be collected. After all samples have been collected, the COC form will be completed and direct field measurements will be collected and recorded on the field log. Field observations will also be recorded on the field log sheet. The technician will then proceed to the next designated sampling site. After all sampling sites have been completed, the samples will be delivered to the appropriate laboratory.

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### **APPENDIX C**

### STANDARD OPERATING PROCEDURES FOR FLOW MEASUREMENTD

#### STANDARD OPERATING PROCEDURES FLOW MEASUREMENT

Due to the nature of surface water runoff, it is expected that all samples will be collected from flows that are less than 1.5 feet in depth. Flow measurements will be calculated using either a volumetric method or the float method, depending on site conditions.

#### **VOLUMETRIC METHOD**

If the surface runoff is free-falling, the volumetric method will be used. The entire flowing stream of water will be collected in a container of known volume, and the amount of time it takes the container to fill will be recorded with a stopwatch. This procedure will be repeated at least three times, and the times will be averaged to insure that the discharge is representative of actual site conditions. The estimated flow rate (Q) will be calculated by:

Q = Volume of Container / Average Time to Fill Container

#### FLOAT METHOD

If the flow is not able to be entirely collected in a container, a float and a stopwatch will be used. The float will consist of free-flowing debris in the body of water, or an object that is equivalent (plant matter, etc.). Both the width of the water body and the depth of the runoff will be measured with a tape measure. The average velocity of the flowing water will be measured by recording the time it takes the float to travel a set distance (ideally 10 feet) in at least three separate events, then averaging the time. All data will be recorded on the field log. The estimated flow rate (Q) will be calculated by:

Q = (f) x (Cross sectional area of stream) x (Average Velocity)

\*Note – f is the coefficient of friction to account for friction effects of the stream bottom. Depending on the bottom surface of the stream, the value generally ranges from 0.60-0.90. Pre-established coefficients will be used, depending on the surface (concrete, dirt, rocky, etc.).

In the event that surface flow is observed but no uniform channel exists, a flow channel will be constructed using weighted rubber tubing. The entire flowing stream of water will be diverted into the constructed flow channel and the flow will be calculated using the float method described above.

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### **APPENDIX D**

# **CHAIN OF CUSTODY FORMS**

				Weck Laboratories, Inc.	ories, I	Ŀ		σ	A	z	Ы	CU	ST	CHAIN OF CUSTODY RECORD	REC	ORD
14859 East Clark Avenue: Industry : C/ Tel 626-336-2139 ◆ Fax 626-336-2634	enue:In Fax 62(	ndustry : ( 6-336-2634	: CA 91 2634 • W	A 91745 A www.wecklabs.com	vices - Since	1964		STP	STANDARD	RD				Page_	e 1	of 1
CLIENT NAME:				PROJECT:		μ		AN	ALYSE	ES RE	ANALYSES REQUESTED	TED		SPI	SPECIAL HANDLING	NDLING
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CHAIN OF CUSTODY RECORD

Aquatic Bioassay and Consulting Laboratories 29 N. Olive Street Ventura, CA 93001 Phone: (805) 643-5621 Fax: (805) 643-5621

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# **APPENDIX E**

# LABORATORY QUALITY ASSURANCE MANUAL WECK LABORATORIES, INC.



Weck Laboratories, Inc.

Environmental and Analytical Services - Since 1964

# **Quality Assurance Program Manual**

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#### 1 INTRODUCTION

Weck Laboratories is an independent testing laboratory specializing in environmental analytical services. The company was founded in 1964 and it is organized as a California corporation.

The purpose of the Weck Laboratories Quality Assurance Program is to operate under standardized QA procedures, to provide guidance to all personnel and it is designed to continually monitor the reliability of test results, ensuring that they fall within acceptable limits, and provide guidelines for the implementation of corrective action when necessary.

This Quality Assurance Manual is a summary document that outlines the policies and operational procedures and the laboratory management system associated with work carried out at its permanent facility in the City of Industry, California, as well as at sites away from its permanent facilities, or in associated temporary or mobile facilities. It is intended to ensure the high quality of analytical services that the Laboratory is committed to provide to its clients. This Manual contains references to other supporting documents also related to the Quality Assurance Program, such as SOPs, QC acceptance limits, MDL studies, Performance Evaluation Results and Policy documents.

The QA Manual and its supporting documents are reviewed annually to ensure that they reflect current laboratory practices and are in agreement with current regulations.

All policies and procedures have been structured in accordance with the NELAC standards and applicable requirements, regulations, guidance, and technical standards from the USEPA and State regulatory agencies. This manual, which also incorporates the requirements of ISO 17025, has been prepared in accordance with the guidance documents listed in section 19.

If more stringent standards or requirements than the specified in this Manual are included in a mandated test method or by regulation, such requirements must be met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed.

This Quality Manual, SOPs and related documentation describe the quality system for Weck Laboratories, Inc.

#### 1.1 Mission Statement

Weck Laboratories provides qualitative and quantitative data for use in critical decisions relating to the protection of the public and the environment. The data used for such purposes must be scientifically valid, defensible and of known and documented quality. All environmental testing activities are carried out in such a way as to meet the requirements of the current NELAC Standard and to satisfy the needs of the client, the regulatory authorities or organizations providing recognition.

It is our goal to provide our clients with the best possible services, in terms of quality of laboratory work, honesty in our procedures and reporting, efficiency in our turnaround time and reasonable prices for our services and at the same time satisfy the needs of the regulatory authorities and organizations providing recognition.

Top management of the laboratory is totally committed to the attainment of the best possible quality of data and instructs and educates the staff on this company policy.

All the necessary resources and materials shall be provided to the personnel of the laboratory in order to meet and/or improve the quality requirements of NELAC and consequently of ISO 9001 and 9002, of the analytical methods performed at the lab and any special requirements from clients.

#### 1.2 Services provided

The services provided by this facility are the following:

- Organic chemical analyses
- Inorganic chemical analyses
- Trace metal analyses
- Microbiological analysis limited to total coliform, fecal coliform and standard plate count.
- Physical analyses
- Field services (sampling and simple field determinations)

The technical and service requirements for all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. This includes a review of facilities and instrumentation, staffing, and any special QC or reporting requirements to ensure that analyses can be performed within the expected schedule. All measurements are made using published reference methods or methods developed by Weck Laboratories. Competence with all methods is demonstrated according to the procedure described in Appendix 9 prior to use.

#### **1.3 Proficiency Testing**

Weck Laboratories, Inc. analyzes Proficiency Testing samples at a frequency established by the current regulations, typically two times per year, from an approved PT provider that meets the requirements specified in chapter 2 of the current NELAC standard. The specific analytes and matrices analyzed are based on the current scope of the laboratory services and are documented in a laboratory SOP on PT samples analyses.

The goal for PT results is obtaining 100% of all analytes within acceptable limits. When there are results out of the acceptance range, corrective action is initiated to prevent the error from reoccurring. A report with the documentation of the corrective action is also filed.

#### 1.4 Ethics policy

Weck Laboratories, Inc. has developed a proactive program for prevention and detection of improper, unethical or illegal actions. A main component of this program is the periodic training and communications that the employees receive from management about the ethics policy and the utmost importance of an honest and ethical behavior in all activities performed at the laboratory.

Proper ethical conduct in the laboratory is strictly enforced. The Company's Code of Ethics (Appendix 2) is presented to current and prospective employees in both the QA manual and the Employee Handbook.

The Data Integrity Plan, which includes the description of the data integrity procedures, serves to combine the elements currently in place and document further procedures to ensure our compliance with requirements in the NELAC standard and from other regulatory agencies.

These procedures include the following elements:

- data Integrity training
- signed data integrity documentation for all laboratory employees
- in-depth, periodic monitoring of data integrity
- data integrity procedure documentation.

The data integrity procedures are signed and dated by senior management. These procedures and the associated implementation records are properly maintained and made available for assessor review. The data integrity procedures are annually reviewed and updated if necessary by management.

The Data Integrity Plan also provides a mechanism for confidential reporting of data integrity issues in the laboratory. A primary element of the mechanism is to assure confidentiality and a receptive environment in which all employees may privately discuss ethical issues or report items of ethical concern. In instances of ethical concern, the mechanism also includes a process whereby laboratory management is to be informed of the need for any further detailed investigation.

Each employee is required to understand and sign a Data Integrity Agreement, contained in the Data Integrity Plan document. The Laboratory Ethics seminar that is presented as a refresher to current employees on an annual basis and as part of the hiring process for new employees include elements describing examples of improper and illegal actions, how to identify appropriate and inappropriate laboratory and instrument manipulation practices, guidance for manual integration practices and consequences of unethical or improper behavior.

Punishment for improper, illegal or unethical activities range from suspension to termination, depending on the degree and nature of the unethical activity.

Employees are required and encouraged to bring up to management any improper activities they detect or are suspicious of. Any incident reported is immediately investigated by the management and the person or persons involved are subject to disciplinary actions.

The Management shall also monitor the program for detecting improper, unethical or illegal action by performing internal proficiency testing (single or double blind), reviewing of analytical data postanalysis, performing electronic data audits using special software as Mint Miner® and providing an open door policy for employees to report any suspicious activity without fears.

In order to assist the laboratory technical personnel in performing their duties without detrimental influences, it is the policy of the Company that the laboratory be impartial and that it and its personnel are free from any undue commercial, financial and other pressures which might influence or adversely affect their normal performance having an impact on the quality of the work they produce or their technical judgment. By this policy all laboratory personnel dedicated to technical activities should not be influenced by, or involved in any financial or commercial matter while performing laboratory work. If any employee feels that he or she might be under any kind of pressure as described above, the Laboratory Director must be notified immediately. Additionally, the Laboratory will not engage in any activities that may endanger the trust in its independence of judgment and integrity in relation to its environmental testing.

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#### 2 QUALITY POLICY

#### 2.1 QA objectives for measuring data

The objective of the Quality Assurance Program is to monitor the reliability of the analytical data produced by the Laboratory and to implement effectively the quality control procedures and operations defined for each analysis. The purposes of this program are:

- Provide data that is scientifically valid, defensible, and of known and documented quality in accordance with standards developed by the National Environmental Laboratory Accreditation Conference (NELAC) and any applicable state or EPA regulations or requirements.
- Ensure that analytical results fall between acceptable control limits.
- Provide mechanisms for corrective action when necessary.
- Establish standardized practices to provide consistency in the generation of data.
- Define the quality of each analytical system in terms of accuracy, precision and sensitivity.
- Identify in the early stages possible problems that may affect data quality.

#### 2.2 Resources

The resources of Weck Laboratories are instrumental in implementing this policy. Highly trained personnel, including chemists and related scientists continue their education by attending seminars and technical meetings; instrumentation that is continuously upgraded to maintain the state-of-the-art in analytical instruments; and a facility currently consisting of 22,000 sq. ft. of laboratory area distributed in a manner that minimizes laboratory contamination.

#### **3 DESCRIPTION OF THE QAP MANUAL**

#### 3.1 Terminology

°C: Degrees Celsius.

AA: Atomic Absorption.

Accreditation body: Authoritative body that performs accreditation.

Aliquot: A discrete, measured, representative portion of a sample taken for analysis.

**Analyte**: The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together.

ANSI/ASQC: American National Standards Institute/American Society for Quality Control.

ASQC: American Society for Quality Control.

ASTM: American Society for Testing and Materials.

**Assessment**: The evaluation process used to measure the performance of effectiveness of a system and its elements against specific criteria. It includes any of the following: audit, performance evaluation, peer review, inspection, or surveillance.

Atomization: A process in which a sample is converted to free atoms.

**Audit**: A documented investigative evaluation used to determine the degree of compliance with established procedures and guidelines, applied to specific analytical processes.

BFB: Bromofluorobenzene.

BNA: Base, neutral and acid.

**BOD**: Biochemical Oxygen Demand.

**BS**: Blank Spike, equivalent to LFB and LCS.

BTEX: Benzene, toluene, ethyl benzene and xylene.

**CA**: Corrective Action, the measures taken to correct a situation that is out of the control limits set by QC procedures.

**CAL**: Calibration standard, a solution prepared from the dilution of stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

**Calibration Range**: The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.

CARB: California Air Resources Board.

CAS: Chemical Abstract Service.

CATC: Cyanide amenable to chlorination.

**CCC**: Calibration check compound.

CFR: Code of Federal Regulations.

**Chain of Custody**: An unbroken trail of accountability that verifies the physical security of samples, data and records.

CI: Chemical ionization.

**Client**: Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.

CLP: Contract Laboratory Program.

**COC**: Chain of Custody.

**COD**: Chemical oxygen demand.

Congener: A member of a class of related chemical compounds (e.g., PCBs, PCDDs).

**Consensus Standard**: A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.

**Continuing calibration verification (CCV)**: The verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as wells as to linear and non-linear calibration models.

**CRDL**: Contract Required Detection Limit.

CV: Coefficient of variation.

CVAA: Cold Vapor Atomic Absorption Spectroscopy.

**DBPs**: Disinfection by-products.

**Definitive Data**: Analytical data of known quality, concentration, and level of uncertainty. The levels of quality and uncertainty of the analytical data are consistent with the requirements for the decision to be made. Suitable for final decision-making.

**Detection Limit (DL)**: The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

**DFTPP**: Decafluorotriphenylphosphine.

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**Digestion**: A process in which a sample is treated (usually in conjunction with heat) to convert the sample to a more easily measured form.

**Dissolved**: The concentration of analyte in an aqueous sample that will pass through a 0.45  $\mu$ m membrane filter assembly prior to sample acidification.

**DLR**: Detection Limit for Reporting purposes, established by the California Department of Health Services for potable water analysis.

**DOC**: Demonstration of capability.

**DOE**: Department of Energy.

**DOT**: Department of Transportation.

**DOD**: Department of Defense.

**DQIs**: Data Quality Indicators.

**DQOs:** Data Quality Objectives.

**DRO**: Diesel-range organics.

**Duplicate**: The analysis or measurement of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analysis are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

**ECD**: Electron capture detector.

EDD: Electronic data deliverable.

El: Electron impact ionization.

ELAP: Environmental Laboratory Accreditation Program.

Eluent: A solvent used to carry the components of a mixture through a stationary phase.

**Elute**: To extract; specifically, to remove (adsorbed material) from an adsorbent by means of a solvent. **Elution**: A process in which solutes are washed through a stationary phase by a movement of a mobile phase.

**Environmental Data**: Any measurement or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.

Environmental Monitoring: The process of measuring or collecting environmental data.

**EPA**: United States Environmental Protection Agency.

**False Negative**: An analyte incorrectly reported as absent from the sample, resulting in potential risks from their presence.

**False Positive**: An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern.

FIA: Flow-injection analysis.

FID: Flame-ionization detector.

**Finding**: An assessment conclusion referenced to a NELAC Standard and supported by objective evidence that identifies a deviation from a NELAC requirement. An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative and is normally accompanied by specific examples of the observed condition and may be linked to a specific requirement.

FPD: Fame photometric detector.

GC/MS: Gas chromatography/mass spectrometry.

**GFAA**: Graphite Furnace Atomic Absorption Spectroscopy.

GPC: Gel-permeation chromatography.

**GRO**: Gasoline-range organics.

HAAs: Haloacetic acids.

HAN: Haloacetonitrile.

HDPE: High Density Polyethylene.

**Holding Times**: The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. The time elapsed from the time of sampling to the time of extraction or analysis, or from extraction to analysis, as appropriate.

**Homologue**: One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, CH<sub>3</sub>OH (methanol),

C<sub>2</sub>H<sub>5</sub>OH (ethanol), C<sub>3</sub>H<sub>7</sub>OH (propanol), C<sub>4</sub>H<sub>9</sub>OH (butanol), etc., form a homologous series.

HPLC: High Performance Liquid Chromatography.

**HRGC**: High Resolution Gas Chromatography.

HRMS: High Resolution Mass Spectrometry.

IC: Ion Chromatography.

IC/MS/MS: Ion Chromatography-Tandem Mass Spectrometry.

**ICP**: Inductively Coupled Plasma spectrometry.

ICP-MS: Inductively coupled plasma-mass spectrometer.

**ICV:** Initial calibration verification.

**ICS**: Interference check sample.

**IDL**: Instrument Detection Limit.

**IEC**: Interelement correction factor.

**Interference, spectral**: Occurs when particulate matter from the atomization scatters the incident radiation from the source or when the absorption or emission of an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.

**IPC**: Instrument Performance Check Solution - A solution of the method analyte, used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

ISE: Ion-selective electrode.

**ISO/IEC**: International Standards Organization/International Electrotechnical Commission.

**Isomer**: One of two or more compounds, radicals, or ions that contain the same number of atoms of the same elements but differ in structural arrangement and properties. For example, hexane ( $C_6H_{14}$ ) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.

LCL: Lower Control Limit.

LCS: Laboratory control sample (equivalent to LFB).

LC/MS/MS: Liquid Chromatography-Tandem Mass Spectrometry.

**LD1 and LD2**: Laboratory Duplicates - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicate precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures. **LDR**: Linear Dynamic Range - The concentration range over which the instrument response to an

analyte is linear.

**LFB**: Laboratory Fortified Blank - An aliquot of LRB to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

**LFM**: Laboratory Fortified Sample Matrix (LFM) – Also known as Matrix Spike. An aliquot of an environmental sample to which a known quantity of the method analyte is added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of the analyte in the sample matrix must be determined in a separate aliquot and the measured value in the LFM corrected for background concentration.

LIMS: Laboratory information management system.

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**Limit of Detection (LOD)**: An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent. The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. **Limits of Quantitation (LOQ)**: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The lowest concentration that produces a quantitative result within specified limits of precision and bias. The LOQ is set at or above the concentration of the lowest initial calibration standard. Also known as Practical Quantitation

above the concentration of the lowest initial calibration standard. Also known as Practical Quantitation Limit or PQL and Method Reporting Limit or MRL.

LLE: Liquid-liquid extraction.

**LRB**: Laboratory Reagent Blank - An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The LRB is used to determine if the method analyte or other interferences are present in the laboratory environment, reagents, or apparatus.

LWL: Lower Warning Limit.

**Management**: Those individuals directly responsible and accountable for planning, implementing, and assessing work.

**Management System**: System to establish policy and objectives and to achieve those objectives. **Matrix Spike (MS)**: Also known as spiked sample or fortified sample, it is a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

**Matrix Spike Duplicate (MSD)**: Also known as fortified sample duplicate, a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

**Method Detection Limit**: One way to establish a Limit of Detection, defined as the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

**Method of Standard Additions (MSA)**: A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is often called spiking the sample.)

MSDS: Material Safety Data Sheet.

MS/MS: Multistage mass spectrometry or tandem mass spectrometry.

NELAC: National Environmental Laboratory Accreditation Conference.

NELAP: National Environmental Laboratory Accreditation Program.

NIOSH: National Institute for Occupational Safety and Health.

NIST: National Institute for Standards and Technology.

**Nonconformance**: An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.

**NPD**: Nitrogen-phosphorus detector.

NPDES: National Pollutant Discharge Elimination System.

**OCP**: Organochlorine pesticides.

**OSHA**: Occupational Safety and Health Administration.

PAH: Polynuclear Aromatic Hydrocarbons (or PNA).

**PBMS**: Performance Based Measurement System.

PCBs: Polychlorinated biphenyls.

PCDD: Polychlorinated dibenzo-p-dioxins.

PCDF: Polychlorinated dibenzofurans.

**PID**: Photoionization detection.

**PQL**: Practical Quantitation Limit.

**PT**: Proficiency Testing.

**RF**: Response Factor. **QA**: Quality Assurance.

**QAP**: Quality Assurance Program.

**Quality Assurance (Project) Plan (QAPP)**: A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

QC: Quality Control.

**QCS**: Quality Control Sample - A solution of the method analyte of known concentration, which is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of the calibration standards. It is used to check either laboratory or instrument performance.

**Quantitation Range**: The range of values in a calibration curve between the LOQ and the highest successfully analyzed initial calibration standard. The quantitation range lies within the calibration range. **Reporting Limit (RL)**: A client-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.

**Retention Time (RT)**: The time between sample injection and the appearance of a solute peak at the detector.

**RPD**: Relative percent difference.

**RSD**: Relative standard deviation.

**Sample**: Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.

Sampling and Analysis Plan (SAP): See Quality Assurance Project Plan.

**Second-source calibration verification (ICV)**: A standard obtained or prepared from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration.

SCAQMD: South Coast Air Quality Management District.

SI: International System of Units.

**Signal to Noise Ratio**: The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude.

**SIM**: Selected-ion monitoring.

SOC: Synthetic organic chemical.

SOP: Standard Operating Procedure.

SPCC: System Performance Check Compounds.

**SPE**: Solid-phase extraction.

**SPME**: Solid-phase microextraction.

SRM: Standard Reference Material.

**Standard**: (Chemical) Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material

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produced by the US National Institute of Standards and Technology (NIST) and characterized for absolute content, independent of analytical test method.

SUR: Surrogate compound.

**SVOA**: Semivolatile organics analysis.

Target Analytes: Analytes specifically named by a client (also called project-specific analytes).

**TCD**: Thermal conductivity detector.

TCDD: Tetrachlorodibenzodioxin.

TCDF: Tetrachlorodibenzofuran.

TCLP: Toxicity Characteristic Leaching Procedure.

TDS: Total dissolved solids (total filterable residue).

**TEM**: Transmission electron microscopy.

TIC: Tentatively identified compounds.

TKN: Total Kjeldahl Nitrogen.

**TOC**: Total Organic Carbon.

TOX: Total Organic Halides.

**TPH**: Total petroleum hydrocarbon.

**TRPH**: Total recoverable petroleum hydrocarbon.

**TSS**: Total suspended solids (total non-filterable residue).

**Tuning**: A check and/or adjustment of instrument performance for mass spectrometry as required by the method.

UCL: Upper Control Limit.

UV: Ultraviolet.

UV/VIS: Ultraviolet/visible-light.

**UWL**: Upper Warning Limit.

**VOA**: Volatile Organic Analyte.

**VOCs**: Volatile organic compound(s).

WET: Waste Extraction Test (California leaching test).

**WET**: Whole effluent toxicity.

**Work Cell**: A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented.

WP: Water Pollution Performance Evaluation Samples.

WS: Water Supply Performance Evaluation Samples.

ZHE: Zero-headspace extraction.

Other terminology commonly used can be found in the glossary section of the NELAC standards.

#### 3.2 Scope

The purpose of the Quality Assurance Program (QAP) described in this manual is to ensure the integrity of the data produced by the laboratory. The QAP encompasses all aspects of the analytical process. The management of Weck Laboratories, Inc. is committed to provide analytical and environmental services of the highest possible quality in order to satisfy the requirements of the regulatory agencies and to meet or exceed our clients' expectations.

This commitment is transmitted to all levels of our organization. Employees and associates are encouraged to constantly improve the quality of their work.

#### **3.3** Fields of Testing

The analytical activities that will be described in this manual are divided into the following main groups:

- Environmental testing involving analysis of drinking water, wastewater, soil and hazardous waste. The analysis of environmental samples follows primarily the methodology approved by the California Department of Health Services under the Environmental Laboratory Accreditation Program and other regulatory agencies.
- Industrial Hygiene analysis of metals and organics in air filters and sorbent tubes following primarily NIOSH published methods.
- Analysis of air samples follows the methodology of the California Air Resources Board, the SCAQMD and other agencies.

#### 3.4 Management of the QAP Manual

The Quality Assurance Program is constantly monitored, reviewed and evaluated. The Quality Assurance Officer is the primary person in charge of updating, revising and distributing this QAP Manual. The Laboratory Director and Technical Directors also have input in the upgrade of the Manual. The revision process takes place when needed if there is a change in some of the processes described, and it is also reviewed and re-approved yearly, if no changes are needed. After the revision is completed, the manual is approved for release by the QA Officer and by the Management. After it is submitted, some time is allowed for training of the personnel in the changes introduced if any. The Dates of submittal and the effective date are in the cover page of the document.

#### 4 DESCRIPTION OF THE LABORATORY

#### 4.1 Identification

Dr. Friedrich J. Weck founded Weck Laboratories, Inc. in 1964 as a consulting and contract laboratory dedicated to independent analytical testing and research activities. Over the years the Laboratory's primary activity shifted to environmental analytical chemistry.

The company is a California Corporation established in 1981. The address of the Laboratory facility is 14859 East Clark Avenue, City of Industry, California, 91745, located north of the 60 Freeway, Seventh Avenue exit.

#### 4.2 Fields of Activity

Weck Laboratories offers a full range of environmental testing, including drinking water, wastewater, groundwater, soil, hazardous waste, ambient air and industrial hygiene testing. The types of analyses performed include both organic & inorganic chemical, physical and bacteriological tests, distributed between two buildings located at the facility.

#### 4.3 Organizational Structure

The different positions within the laboratory have job descriptions that are maintained in the Human Resources department. The organization chart of Weck Laboratories, Inc. can be found in Appendix 3.

#### 5 STAFF

#### 5.1 Management Personnel

The managerial and technical personnel have the authority and resources needed to carry out their duties and to identify the occurrence of departures from the quality system or from the procedures for performing environmental tests and/or calibrations, and to initiate actions to prevent or minimize such departures.

Technical management has overall responsibility for the technical operations and for the provision of the resources needed to ensure the required quality of laboratory operations. Deputies are appointed for key managerial personnel, including the technical director(s) and QA Officer, to perform their duties in case of prolonged absences.

The following laboratory management staff is considered key staff:

- President/CEO Laboratory Director
- Technical Directors
- Section Supervisors
- Quality Assurance Officer
- IT Manager
- Administration Manager
- Client Service Manager
- Project Managers

The reporting relationship between key personnel and other staff is detailed in the Organization Chart (Appendix 3) and Job descriptions of positions found in the Personnel Records.

The following are the responsibilities and activities within the QAP in which the key and management personnel are engaged:

#### Laboratory Management

- Defining the minimal level of experience and skills necessary for all positions in the laboratory
- Ensuring that all technical laboratory personnel have demonstrated capability in the activities for which they are responsible
- Ensuring that the training of its personnel is kept up-to-date
- Documenting all analytical and operational activities
- Supervising all personnel
- Ensuring that all sample acceptance criteria are verified and that samples are logged into the sample tracking system and properly labeled and stored
- · Performing with the other management staff an annual Management System Review
- Documenting the quality of all data reported by the laboratory
- Ensuring that the laboratory has the appropriate resources and facilities to perform requested work
- Ensuring that corrective actions relating to findings from the internal audit are completed; and

- Nominating deputies when the Technical Directors or QA Officer are absent
- Developing a proactive program for prevention and detection of improper, unethical or illegal actions and operating in accordance with the Laboratory's documented ethics policy
- Ensuring that only those outside support services and supplies that are of adequate quality to sustain confidence in the laboratory's tests are used
- Commitment to meet customer requirements and whenever possible exceed their expectations
- Commitment to operate in accordance with statutory and regulatory requirements

#### QA Officer

The QA Officer is responsible for the Quality System of the laboratory and its implementation. He or she has direct access to the highest level of management (President/Laboratory Director) and to the Technical Directors to resolve any dispute involving data quality.

The specific functions and characteristics of the QA Officer are the following:

- Serve as the focal point for QA/QC and be responsible for the oversight and/or review of quality control data
- Have functions independent from laboratory day-to-day operations for which he or she has quality assurance oversight
- Be able to evaluate data objectively and perform assessments without any outside influence
- Have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system as defined under NELAC
- Have a general knowledge of the analytical tests methods for which data review is performed
- Arrange for or conduct internal audits on the entire technical operation annually
- Notify laboratory management of deficiencies and non-compliance items in the quality system and monitor corrective action
- Be responsible for implementing, maintaining, and improving the quality system
- Ensuring that all personnel understand their contributions to the quality system
- Ensuring communication takes place at all levels within the laboratory regarding the effectiveness of the quality system
- Evaluating the effectiveness of training
- Using available tools, such as audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and management reviews in efforts to monitor trends and continually improve the quality system
- The QA Officer has sufficient authority to stop work as deemed necessary in the event of serious QA/QC issues.

#### Technical Directors

The full time individuals who have overall responsibility for the technical operation of the laboratory. There are four technical directors for the specific areas of the laboratory: Chemical Organic Analyses, Chemical Inorganic Analyses, Microbiological Analyses and Radiochemistry. The daily activities and responsibilities of the Technical Directors are the following:

- Certifying that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited
- Monitoring standards of performance in quality control and quality assurance.
- Monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data
- Ensuring that sufficient number of qualified personnel are employed to supervise and perform the work of the laboratory
- Providing educational direction to laboratory staff
- Exercising day-to-day supervision of laboratory operations for the corresponding department

The Technical Directors of Weck Laboratories meet the requirements specified in Section 4.1.1.1 of the NELAC Standards.

Resumes of management personnel are in Appendix 1.

#### 5.2 Personnel Qualifications

The technical staff is responsible for sample analysis and identification of corrective actions. The staff reports directly to the Laboratory Director or Lab Manager. All personnel are responsible for complying with all quality assurance/quality control (QA/QC) requirements that pertain to their organizational/ technical function. As documented in the employee records, each employee has the experience and education to adequately demonstrate knowledge for their particular function and the general knowledge of laboratory operations, analytical test methods, QA/QC procedures and records management.

The laboratory management shall ensure the competence of all who operate specific equipment, perform environmental tests, evaluate results, and sign test reports and calibration certificates. When using staff that are undergoing training, appropriate supervision shall be provided. Personnel performing specific tasks shall be qualified on the basis of appropriate education, training, experience and/or demonstrated skills, as required.

#### 5.3 Personnel Training

Each employee is required to read, understand, and to use the current versions of the established Standard Operating Procedures and Analytical Method Protocols, which relates to his/her job responsibilities. The Training records show evidence of the revisions of the SOPs the employees have reviewed. Each employee demonstrates initial proficiency by following the procedure described in Appendix 9 of this manual, and demonstrates continued proficiency on a yearly basis by acceptable performance on Laboratory Control Samples (LCS), successful analysis of blind samples or by analyzing in parallel a sample analyzed by a trained or re-trained analyst. The training records of the analysts are organized by analyst and kept with personnel files. They include initial and continuing training, continuing education, participation in technical conferences or seminars and internal training activities.

Initial training for new employees is performed by experienced personnel with management guidance and includes the observation of the QC procedures described in this manual.

The company has a policy that encourages all technical personnel to participate in technical seminars and meetings involving innovative analytical technologies, new instrumentation and software applied to environmental testing. Records of this participation are maintained in the personnel files. The management of the laboratory shall formulate the goals with respect to the education, training and skills of the laboratory personnel.

The personnel performing analytical and related tasks at the laboratory must be employed by, or under contract to, the laboratory. Where contracted and additional technical and key support personnel are used, the laboratory shall ensure that such personnel are supervised and competent and that they work in accordance with the laboratory's quality system.

The laboratory shall maintain current job descriptions for all personnel who manage, perform, or verify work affecting the quality of the environmental tests. The job descriptions shall include the following:

- Duties relative to scheduling and performing tests and evaluating results;
- Duties relative to the development, validation, and approval of new methods or method modifications;
- Required experience, qualifications, and training
- Managerial duties.

The management shall authorize specific personnel to perform particular types of sampling, environmental test, to issue test reports and calibration certificates, to give opinions and interpretations and to operate particular types of equipment. The laboratory shall maintain records of the relevant authorization(s), competence, educational and professional qualifications, training, skills and experience of all technical personnel, including contracted personnel. This information shall be readily available and shall include the date on which authorization and/or competence is confirmed.

Records on the relevant qualifications, training, skills and experience of the technical personnel shall be maintained by the laboratory, including records on demonstrated proficiency for each laboratory test method.

#### 6 LABORATORY CAPABILITIES AND ACCREDITATIONS

Weck Laboratories, Inc. analyzes water, soil, hazardous waste and air samples. The following are the type of analysis performed:

- Drinking Water and Groundwater
  - Sampling: Production wells and monitoring wells
  - Inorganic: Trace metals, physical parameters, wet chemistry
  - Organic: Volatile, semi-volatile, pesticides, herbicides
  - Bacteriological: Total and fecal coliforms, Heterotrophic Plate Count
- Waste Water

- Sampling: Composite samplers, grabs.
- Inorganic: Metals, physical parameters, wet chemistry
- Organic: Volatile, semi-volatile, pesticides, herbicides
- Bacteriological: Total and fecal coliforms, Heterotrophic Plate Count
- Hazardous Waste and Soil
  - Characteristics: Physical properties, leaching tests
  - Organic: Volatile, semi-volatile, pesticides, herbicides
  - Inorganic: Metals, wet chemistry
- Industrial Hygiene
  - Indoor Air Analysis: Air filters (metals)
  - Sorbent tubes (organics)

The different analytical techniques and methods performed at the laboratory are described in the laboratory specific SOPs.

The Laboratory is accredited by various regulatory agencies to perform environmental testing. Current accreditations are listed in appendix 11.

The instrumental analytical capabilities of Weck Laboratories, Inc. include the following:

#### • Sampling and field equipment

24 hours composite samplers for water. Flow measurement instruments Water quality kits Encore samplers for soil Immunoassay determinations

#### • Inorganic analysis:

ICP-AES ICP-MS ICP-MS Flow Injection Analysis (hydride generation) Cold Vapor Atomic Absorption Cold Vapor Atomic Fluorescence Cold Vapor Atomic Florescence with Gold Amalgamation UV-visible spectrometry Ion Chromatography IC/MS/MS Ion Selective Electrodes

• Organic Analysis

Purge and Trap equipment for direct purging of soils Purge and Trap for water Automated SPME GC/MS for volatile organics GC/MS for semi volatile organics GC/MS/MS (tandem Mass spectrometry) GC/MS with Chemical Ionization positive ion and negative ion GC with FID,NPD,ECD,PID,TCD LC/MS/MS for UCMR 2, EDC/PPCPs & Perchlorate HPLC with post-column derivatization and UV-Visible and Fluorescence detectors. TOX TOC Infrared analysis

A complete list of laboratory instrumentation is in Appendix 4.

#### 7 QUALITY ASSURANCE OBJECTIVES

The overall QA objective of Weck Laboratories, Inc. is to develop and implement procedures for laboratory analysis, chain-of-custody, and reporting that will provide results, which are of known and documented quality. Data Quality Indicators (DQIs) are used as qualitative and quantitative descriptors in interpreting the degree of acceptability or utility of data. The principal DQIs are precision, bias (accuracy), representativeness, comparability, completeness and detection limits. The DQIs are used as quantitative goals for the quality of data generated in the analytical measurement process. This section summarizes how specific QA objectives are achieved. The specific application of these various activities are contained in the method SOPs.

#### 7.1 Precision

Precision is a measure of the degree to which two or more measurements are in agreement.

Precision is assessed through the calculation of relative percent differences (RPD) and relative standard deviations (RSD) for replicate samples. For analyses that have detectable levels of analytes (for example inorganic analyses), laboratory precision is usually assessed through the analysis of a sample/sample duplicate pair and field duplicate pairs. For analyses that frequently show no detectable levels of analytes (e.g., organic analyses), the precision is usually determined through the analysis of matrix spike/matrix spike duplicates (MS/MSD) and field duplicate samples.

#### 7.2 Accuracy

Accuracy (Bias) is the degree of agreement between an observed value and an accepted reference or true value.

Accuracy is assessed by the analysis of blanks and through the adherence to all sample handling, preservation and holding times. Laboratory accuracy is further assessed through the analysis of MS/MSD, external quality control check samples, laboratory control samples (LCS and LCSD) and surrogate compounds spikes.

#### 7.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point process condition, or an environmental condition within a defined spatial and/or temporal boundary.

Representativeness is ensured by using the proper sampling techniques, proper analytical procedures, appropriate methods; meeting sample holding times and analyzing field duplicate samples.

#### 7.4 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

Laboratory completeness is a measure of the amount of valid measurement obtained from all the measurement taken in the project. The laboratory completeness objective is that the generation of valid data for all samples be greater than 95 percent.

#### 7.5 Comparability

Comparability is an expression of the confidence with which one data can be compared to another.

Comparability is achieved by the use of routine analytical methods, achieving holding times, reporting results in common units, use of consistent detection levels, and consistent rules for reporting data.

#### 7.6 Detection Limits

Method Detection Limits (MDLs) are determined for all analytes as specified in the NELAC standards. From these, Reporting Limits (RLs) are obtained. See section 12.2 for more detailed information.

#### 8 SAMPLING

Most samples processed at the laboratory are collected by clients or their representatives. When required, Weck Laboratories can provide technical assistance for sample collection and handling and can prepare appropriate sample containers with preservatives.

Weck Laboratories field personnel conduct sampling of wastewater and potable water for projects that require this service. Our personnel do not perform industrial hygiene sampling.

In order to assure the quality of the entire analytical process, Weck Laboratories works closely with field personnel employed by the client to meet general QA criteria and if available specific criteria as per the QAPP.

When performing sampling activities related to environmental testing, the laboratory sampling personnel follows the corresponding SOPs. Copies of the SOPs are kept at the field for reference.

The procedures to obtain subsamples, such as obtaining sample aliquots, are documented in each analytical SOP that requires it.

Where the client requires deviations, additions or exclusions from the documented sampling procedure, these are recorded in detail in the case narrative of the work order and reported with the analytical report. They are also communicated to the appropriate personnel.

In the instances that the laboratory does not perform the sampling and whenever possible all sampling information, such as name of sampler, company that employs the sampler, sampling procedure, etc. is recorded in the sampling section of each work order and reported to the client. All other pertinent sampling information and relevant data for operations relating to sampling that forms part of the environmental testing that is undertaken is also recorded and reported with the analytical report.

#### 9 SAMPLE HANDLING

This section summarizes policies and practices for sample handling. Further details are contained in the corresponding SOPs.

#### 9.1 Sample Tracking

Weck Laboratories, Inc. uniquely identifies each sample to be tested, to ensure that there can be no confusion regarding identity. The sample identification system includes identification for all samples, sub-samples and subsequent extracts and/or digestates. A unique identification (ID) code is placed on each sample container.

#### 9.2 Review of Requests, Tenders and Contracts

When a request, tender or contract is received by the Laboratory, the Management or designated staff member will review and ensure that the requirements, including the methods to be used, are adequately defined, documented and understood and that the laboratory has the capability and resources to meet the requirements. The purpose of this review of capability is to establish that the laboratory possesses the necessary physical, personnel and information resources, and that the laboratory's personnel have the skills and expertise necessary for the performance of the tests in question. The review may encompass results of earlier participation in interlaboratory comparisons or proficiency testing and/or the running of trial environmental test or calibration programs using samples or items of known value in order to determine uncertainties of measurement, detection limits of confidence limits, or other essential quality control requirements. The current accreditation status of the laboratory is also reviewed. The laboratory then informs the client of the results of this review if it indicates any potential conflict, deficiency, lack of appropriate accreditation status, or inability on the laboratory's part to complete the client's work. Another item to review is whether or not the appropriate test method is selected and capable of meeting the clients' requirements.

The management or designated staff will discuss and resolve any differences between the request or tender and the contract before any work commences in order to assure that each contract is acceptable both to the laboratory and the client. A contract may be any written or oral agreement to provide a client with environmental testing or other laboratory services.

Records of reviews, including any significant changes, shall be maintained. Records shall also be maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract.

For review of routine and other simple tasks, the date and the identification (e. g. the initials) of the person in the laboratory responsible for carrying out the contracted work are considered adequate.

For repetitive routine tasks, the review need be made only at the initial enquiry stage or on granting of the contract for on-going routine work performed under a general agreement with the client, provided that the client's requirements remain unchanged. For new, complex or advanced environmental testing, a more comprehensive record should be maintained.

The review shall also cover any work that is subcontracted by the laboratory.

The client shall be informed of any deviation from the contract. If a contract needs to be amended after work has commenced, the same contract review process shall be repeated and any amendments shall be communicated to all affected personnel.

If there is any suspension of accreditation, revocation of accreditation, or voluntary withdrawal of accreditation during the time the contract is in effect, this must be reported to the client.

#### 9.3 Sample Acceptance Policy

The following are the requirements for sample acceptance. Data from any samples, which do not meet the policy here specified, are noted in the laboratory report defining the nature and substance of the variation:

- Proper, full, and complete documentation, including the sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample. This information must be fully documented in the chain of custody record. See Appendix 5.
- Unique identification of samples using durable labels completed in indelible ink on all sample containers.
- Use of appropriate sample containers and preservatives as per table in Appendix 6.
- All samples have adequate holding time to be analyzed (Appendix 6).
- If no previous special arrangements were made, parameters that are "field" analysis (i.e. pH, residual chlorine, etc.) will be analyzed within 24 hours from arrival at the laboratory. Samples that arrive at the laboratory after 4 PM on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday).
- Adequate sample size for all analysis requested.
- Special instructions and additional information required to perform the analysis properly (i.e., time, flow rate, etc.).
- Procedures that are used when samples show signs of damage or contamination.
- Samples received at the required temperature (usually  $\leq 6$  °C, but above freezing) or with evidence of chilling process started (received "on ice") if they were collected the same day as received at the lab.

If any of the above requirements are not met, the client is notified immediately, and the irregularity is documented:

- If the client acknowledges the irregularity and instructs the laboratory to continue with analysis this is documented and samples accepted.
- If the client does not acknowledge the irregularity the samples are rejected.
- If the irregularity is noted in samples submitted for bacteriological analysis for compliance purposes, the samples are rejected without exception.

When a request for a new project is received involving multiple samples or tests that have a short holding time the Management is notified. The Management staff with the assistance of the appropriate technical personnel evaluates the project and calculates the resources needed to complete it within the turn around time required and the holding times, taking into consideration the volume of work in house and/or expected.

If it is determined that the new project will not affect the proper completion of jobs already in house and that the laboratory has the resources (personnel, equipment and facilities) necessary to accommodate the new project, this is accepted.

If the Management or any of the technical staff involved thinks that the new job will create problems in terms of reduced quality of work, completion out of specified or required time, or any other detrimental situation, the new project is not accepted and the client notified. If there are alternatives, such as postponement, modification of sampling schedules or partial subcontracting to another lab in order to accommodate the project, this is proposed to the client.

#### 9.4 Sample Receipt Protocol

Upon receipt, the condition of the sample, including any abnormalities or departures from standard condition is recorded. All samples, which require thermal preservation, are considered acceptable if the arrival temperature is within the acceptable range. Samples that are hand delivered to the laboratory immediately after collection may not meet these criteria. In these cases, the samples will be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice. The temperature at which the samples are received is measured and recorded in the documents and in the LIMS.

Where applicable, Weck Laboratories, Inc. verifies chemical preservation using readily available techniques, such as pH or free chlorine, prior to or during sample preparation or analysis. The results of all checks are recorded.

When there is any doubt as to the sample's suitability for testing or if the sample does not meet any of the above criteria or if irregularities are noted, the client is notified immediately, and the irregularity is documented. If the client acknowledges the irregularity and instructs the laboratory to continue with analysis this is also documented. If the client does not acknowledge the irregularity the samples are rejected. If the irregularity is noted in samples submitted for bacteriological analysis for compliance purposes, the samples are rejected without exception.

The sample identification number is affixed to all sample containers and worksheets are prepared for the different types of analyses requested. When there are different containers or sub-samples belonging to one sample for multiple tests, the fraction name is indicated on the sample bottle by a suffix letter or other means. Alternatively, pre-labeled bottles containing the required tests are also provided.

#### 9.5 Storage conditions

Samples that require thermal preservation are stored under refrigeration, as specified in the corresponding SOP or analytical method, which is typically just above the freezing temperature to 6 °C. Samples are stored in a manner that prevents cross contamination, normally they are separated based on matrix, analysis and level of known contamination. Other samples are kept in specific areas while they are being tested. Evidence samples are stored in secured and controlled access areas.

#### 9.6 Custody of Samples and Documentation

The Chain-of-Custody procedures begin when the sample is collected. At that time, a COC form is prepared, containing all the information about the sample (project name, sample identification, date and time of collection, name of person performing the sampling, matrix type, tests requested, number of containers, field measurements, and all other pertinent information).

The person who does the sampling must sign the COC record. The relinquishing and receiving parties must also sign the COC, indicating the date and time this operation was performed.

If the client submits the sample to the laboratory, a copy of the COC form is given to the client as evidence of receipt, while the other two copies are kept at the laboratory.

For samples received in sealed ice chests by commercial freight companies (UPS, FedEx), copies of shipping papers are attached to the COC form for future reference. The person receiving the sample also makes a notation of the type of shipment on the COC.

Access to all samples and sub-samples is controlled. The laboratory area is maintained secured and is restricted to authorized personnel only.

When full Legal/Evidentiary Chain of Custody protocols are required, COC records are used to establish an intact, continuous record of the physical possession, storage and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates, The COC records account for all time periods associated with the samples. The COC records identify all individuals who physically handled individual samples. The COC forms remain with the samples during transport or shipment. If shipping containers and/or individual sample containers are submitted with sample custody seals, and any seals are not intact, the lab shall note this on the chain of custody. Other documents pertaining to the transport of the samples, such as receipts from common carriers are kept as part of the documentation. When evidentiary samples, subsamples, digestates or extracts are transferred to another party they are subject to the requirements of legal chain of custody. These samples are kept in a locked area or refrigerator with the key in possession of the designated sample custodian.

#### 9.7 Sample disposal

Samples are retained for thirty days from report date unless otherwise instructed by the client or if the samples are part of litigation or have been received under legal/evidentiary requirements, in which case the disposal of the physical sample is accomplished with the concurrence of the affected legal authority. After the retention period samples are either returned to the client or properly disposed of according to federal and state laws and regulations.

#### 10 CALIBRATION PROCEDURES AND FREQUENCY

#### 10.1 Measurement Traceability

#### 10.1.1 General

Whenever applicable, calibration of analytical support equipment and instruments and the overall program of calibration and/or verification is designed and operated so as to ensure that measurements are traceable to national standards of measurement.

All equipment used for environmental tests and/or calibrations, including equipment for subsidiary measurements (e.g., for environmental conditions) having a significant effect on the accuracy or validity of the result of the environmental test or sampling shall be calibrated before being put into service and on a continuing basis. The calibration of such equipment is performed according to the established program and procedure. This includes balances, thermometers, and control standards. The program also includes a system for selecting, using, calibrating, checking, controlling and maintaining measurement standards, reference materials used as measurement standards, and measuring and test equipment used to perform environmental tests.

#### 10.1.2 Specific Requirements

The calibration of equipment shall be designed and operated so as to ensure that calibrations and measurements made by the laboratory are traceable to the International System of Units (SI). The traceability is established for measuring instruments to the SI by means of an unbroken chain of calibrations or comparisons linking them to relevant primary standards of the SI units of measurement. The link to SI units may be achieved by reference to national measurement standards. National measurement standards may be primary standards, which are primary realizations of the SI units or agreed representations of SI units based on fundamental physical constants, or they may be secondary standards which are standards calibrated by another national metrology institute. When using external calibration services, traceability of measurement shall be assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability.

There are certain calibrations that currently cannot be strictly made in SI units. In these cases calibration shall provide confidence in measurements by establishing traceability to appropriate measurement standards such as the use of certified reference materials provided by a competent supplier to give a reliable physical or chemical characterization of a material and the use of specified methods and/or consensus standards that are clearly described and agreed by all parties concerned. Participation in a suitable program of interlaboratory comparisons is required where possible.

The requirements above specified do not apply when it has been established that the associated contribution from the calibration contributes little to the total uncertainty of the test result. When this situation arises, the laboratory shall ensure that the equipment used can provide the uncertainty of measurement needed.

Where traceability of measurements to SI units is not possible and/or not relevant, the same requirements for traceability to, for example, certified reference materials, agreed methods and/or consensus standards, are required.

- The overall program of calibration and/or verification and validation of equipment shall be designed and operated so as to ensure that measurements made by the laboratory are traceable to national standards of measurement.
- Calibration certificates shall indicate the traceability to national standards of measurement and shall provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. The laboratory shall maintain records of all such certifications.
- Where traceability to national standards of measurement is not applicable, the laboratory shall
  provide satisfactory evidence of correlation of results, for example by participation in a suitable
  program of interlaboratory comparisons, proficiency testing, or independent analysis.

Calibration certificates obtained by the laboratory shall indicate the traceability to national standards of measurement and shall provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. The laboratory shall maintain records of all such certifications.

Where traceability to national standards of measurement is not applicable, the laboratory shall provide satisfactory evidence of correlation of results, for example by participation in a suitable program of interlaboratory comparisons, proficiency testing, or independent analysis, if any is available.

#### 10.2 Reference Standards and Reference Materials

Reference standards of measurement (such as Class S or equivalent weights or traceable thermometers) are used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated. Reference standards are subjected to in-service checks between calibrations and verifications. Reference standards shall be calibrated before and after any adjustment.

Where traceability of measurements to SI units is not possible or not relevant, the same requirements for traceability to, for example, certified reference materials, agreed methods and/or consensus standards, are required. The laboratory shall provide satisfactory evidence of correlation of results, for example by participation in a suitable program of interlaboratory comparisons, proficiency testing, or independent analysis.

Reference materials that require re-certification are submitted promptly to a qualified certification body can provide traceability to national standards of measurement.

Reference materials shall, where commercially available, be traceable to SI units of measurement, or to certified reference materials. Where possible, traceability shall be to national or international standards of measurement or to national or international standard reference materials. Internal reference materials shall be checked as far as is technically and economically practicable.

Checks needed to maintain confidence in the status of reference, primary, transfer or working standards and reference materials are carried out according to defined procedures and schedules recommended by the manufacturer or maintenance organization.

The procedures employed for safe handling, transport, storage and use of reference standards and reference materials in order to prevent contamination or deterioration and in order to protect their integrity, are the ones recommended by the manufacturer or other organization involved in the maintenance of such materials/standards.

#### **10.3** General Requirements

Each calibration is dated and labeled with or traceable to the method, instrument, analysis date, and each analyte name, concentration and response (or response factor). Sufficient information is recorded to permit reconstruction of the calibration. Acceptance criteria for calibrations comply with method requirements or are established and documented.

#### 10.4 Analytical Support Equipment

Analytical support equipment includes but it is not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All such support equipment is:

- Maintained in proper working order. The records of all activities including service calls are kept.
- Calibrated or verified annually using NIST traceable references when available, over the entire range of use. The results of such calibration must be within the specifications required in the application for which the equipment is used, if not, the equipment is either removed from service until repaired or a correction factor is applied to it, if applicable.

Raw data records shall be retained to document equipment performance.

Prior to use on each working day, balances, ovens, refrigerators, freezers, incubators and water baths are verified for the expected use range using NIST traceable references (where possible). The acceptability for use or continued use is according to the needs of the analysis or application for which the equipment is being used. Mechanical volumetric dispensing devices (except Class A glassware and microsyringes) are checked for accuracy quarterly.

For chemical tests the temperature, cycle time, and pressure of each run of autoclaves is documented by the use of appropriate chemical indicators or temperature recorders and pressure gauges. For biological tests that employ autoclave sterilization see SOP MIS031.

#### 10.4.1 Balances and reference weights

Laboratory balances are serviced and calibrated once a year by a third party specialist, Watson Bros. Weck Laboratories has a contract with Watson Bros., by which they automatically come for balance inspection and calibration every year. The calibration or service is performed more frequently if a problem is suspected or observed by visual inspection. Class S reference weights are not used beyond one year from most recent calibration date.

#### 10.4.2 Thermometers

All thermometers are checked annually against a NIST traceable reference thermometer, which is submitted for certification on annual basis.

#### 10.4.3 Monitoring of Temperature

All refrigerators and freezers used for storage of samples and standards or reagents are monitored for temperature daily. The incubators used for bacteriological analysis are monitored twice a day for temperatures and the incubator for BOD is monitored daily. The temperatures are entered in charts posted on each unit that also include the initials of the person performing the checks and the acceptance ranges. When a temperature is out of compliance in any refrigerator, freezer or incubator, immediate action is taken to correct the problem.

Some support instruments such as ovens and water bath for fecal coliforms are not in use every day, so temperature is checked only for the days they are actually in operation.

#### 10.5 Initial Instrument Calibration (ICAL) and Continuing Calibration Verification (CCV)

All instruments are calibrated in accordance with the respective SOPs and/or method of analysis. The typical calibration procedure consists of an initial calibration, performed by running a series of standards and calculating the response by using either the response factors or by linear or polynomial regression analysis. This is followed by a calibration verification. All calibration procedures are thoroughly documented.

When an initial instrument calibration is not performed on the day of analysis, it is verified by analyzing CCVs standards using the following criteria, unless something different is specified in the corresponding SOPs or QAPP:

- The concentration of the CCV standard shall be from the low-calibration standard to the midpoint of the calibration range;
- The source of the CCV standard should be the same as the source for the initial calibration standard(s); and
- The baseline for evaluating the CCV is the initial calibration curve, except for the evaluation of retention times in organic chromatographic methods, which may be based on comparison with the retention times in the initial CCV.

When the method specifies that CCVs shall be run at specific sample intervals, the count of these samples shall be of field samples only.

When a CCV fails to fall within acceptance limits then CCVs and all samples analyzed since last successful calibration verification are re-analyzed. If reanalysis is not possible, the client is notified prior to reporting data associated with a noncompliant CCV and if data are reported, appropriate qualifiers are used and if further clarification is needed this is explained in the case narrative. The exception to this is when a CCV fails with high bias, but the field samples remain not detected.

In all cases, the validity of the standards used in the initial calibration is verified using an independently prepared calibration verification solution. For all chemical determinations in which standards are involved for calibration, it is the policy of the company to use a secondary reference material (second source) obtained from a second manufacturer or lot if the lot can be demonstrated from the manufacturer

as prepared independently from other lots. Traceability shall be to a national standard, when commercially available. If not commercially available, it can be prepared in-house. This secondary reference can be an LCS or other standard run to verify the integrity of the primary standard. Ideally, the secondary reference will be prepared identically to the calibration standards (i.e. if the calibration standard is directly injected without preparation, then directly injecting the reference standard removes any biases present by any field sample preparation steps).

When project-specific or method-specific requirements do not exist:

- The initial calibration verification shall be successfully completed prior to analyzing any samples;
- The use of a standard from a second lot is acceptable when only one manufacturer of the standard exists (note: manufacturer refers to the producer of the standard, not the vendor); and
- The concentration of the second source standard shall be at or near the midpoint of the calibration range. Acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.

Specific analyses' calibrations are checked more frequently. Some instruments, such as TOX analyzers have built-in calibration features. The internal calibration of these instruments is monitored daily for accuracy.

Some calibration curves for spectrophotometric methods are very stable over a long period of time, however it is the policy of the Laboratory to perform a new initial calibration curve even if the continuing calibration check meets specified criterion, in any of the following events:.

- At least every three years
- When the instrument is moved to a different location
- If any maintenance that can affect the calibration has been performed
- If the analysts judges it necessary for special projects or different range of calibration

Spectrophotometers are also subject to wavelength calibration which it shall be performed at least annually, according to the procedure described by the manufacturer in the instrument manual or other documentation.

All results are calculated based on the response curve from the initial calibration and generally not quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method, or program. The results are bracketed by calibration standards which cover the entire quantitation range for each analyte. Any data reported below the lower-limit of quantitation is considered to have an increased quantitative uncertainty and consequently it is reported using defined qualifiers or flags or explained in the case narrative. The highest calibration standard is the highest concentration for which quantitative data are to be reported. Any data reported above this highest standard is considered to have an increased quantitative uncertainty and it is reported as an estimated value using the defined data qualifiers or explained in the case narrative, unless the sample can be diluted and re-run within the limits of the initial calibration curve.

The following is the criteria used for the acceptance of an initial calibration, unless specified differently in the analytical methods:

- Use the average response factor (RF) if the percent relative standard deviation (%RSD) of the points is less than 20%. In this case, linearity through the origin is assumed.
- If the %RSD is greater than 20%, linearity through the origin cannot be assumed and a linear regression, a weighed linear regression or a non-linear regression can be used. The acceptance criteria for linear regression are a coefficient of correlation (r) equal or greater than 0.99 and for non-linear regression the coefficient of determination (COD) must be equal or greater than 0.98. In both cases, the curve is not to be forced through the origin nor is the origin used as another point. The sample results must be within the first and last standards.
- The number of data points to construct the initial calibration curve shall be obtained from the analytical method employed. If no criteria are specified, the laboratory shall construct initial calibration curves using a minimum of five calibration points for organic analytes and three calibration points for inorganic analytes and IH samples. All reported target analytes and surrogates (if applicable) shall be included in the initial calibration. Reported results for all target analytes shall be quantified using a multipoint calibration curve; surrogates are calibrated according to each analytical method requirements, unless there are project specific requirements in which case these are followed. It is not permitted to exclude calibration points unless there is technical justification for it.
- The lowest standard shall be at or below the reporting limit for the method and at or below the regulatory limit/decision level if known by the laboratory.
- The lowest calibration standard must be above the detection limit. Noted exceptions: for turbidity analysis and for instrument technology (such as ICP or ICP/MS) with validated techniques from manufacturers or methods employing standardization with a zero point and a single point calibration standard:
  - Prior to the analysis of samples the zero point and single point calibration must be analyzed and the linear range of the instrument must be established by analyzing a series of standards, one of which must be at the lowest quantitation level.
  - Zero point and single point calibration standard must be analyzed with each analytical batch.
  - A standard corresponding to the lowest quantitation level must be analyzed with each analytical batch and must meet established acceptance criteria.
  - The linearity is verified at a frequency established by the method and/or the manufacturer.
  - If a sample within an analytical batch produces results above its associated single point standard then one of the following should occur:
    - analyze reference material at or above the sample value that meets established acceptance criteria for validating the linearity; dilute the sample such that the result falls below the single point calibration concentration (when sufficient sample volume permits);
    - Report the data with an appropriate data qualifier and/or explain in the case narrative.
    - For metals analysis with a single-point calibration, a sample result may be reported up to 90% of the linear dynamic range (LDR). All samples exceeding this value must be diluted to within the LDR.

If the initial calibration fails, the analysis procedure is stopped and evaluated. For example, a second standard may be analyzed and evaluated or a new initial calibration curve may be established and verified. In all cases, the initial calibration must be acceptable before analyzing samples. If samples can

not be reanalyzed, data associated with an unacceptable initial instrument calibration must be reported with appropriate data qualifiers.

When an initial calibration is not performed on the day of the analysis, a calibration verification check standard is analyzed at the beginning and at the end of each batch. An exception to this policy is for internal standard methods (e.g., most organic methods). For these analyses, the calibration check is only analyzed at the beginning of the analytical sequence or analytical batch. The concentration of this calibration check is specified in each method SOP and whenever possible is varied within the established calibration range.

Sufficient raw data records are retained electronically as printouts to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor, or unique equations or coefficients used to convert instrument responses into concentrations. Continuing calibration verification records explicitly connect the continuing verification data to the initial instrument calibration by listing in the quantification report the initial calibration file that was used for the calculation.

When intermediate checks are needed to maintain confidence in the calibration status of the equipment, these checks shall be carried out according to each Standard Operating Procedure for the analytical method.

Where calibrations give rise to a set of correction factors, the laboratory shall have procedures to ensure that copies (e.g., in computer software) are correctly updated.

If the continuing instrument calibration verification results obtained are outside established acceptance criteria, corrective actions are performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, the following options are available:

- Demonstrate performance after corrective action with two consecutive successful calibration verifications
- Perform a new initial instrument calibration.

If acceptable performance has not been demonstrated, sample analyses shall not occur until a new initial calibration curve is established and verified. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

- When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported.
- When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level or if the samples are not for regulatory compliance and accurate values are not required by the customer.

## 11 TEST METHODS AND STANDARD OPERATING PROCEDURES

The methods and procedures used at the laboratory are the appropriate ones for all environmental tests within its scope. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement uncertainty as well as statistical techniques for analysis of environmental test and/or calibration data.

The methods used at the laboratory, including methods for sampling, must meet the needs of the client and are appropriate for the environmental tests it undertakes. These analytical procedures currently in use are based on the methodology approved by the EPA, the California Department of Health Services, the AIHA, and other regulatory agencies.

In some cases, Weck Laboratories can perform analyses that are not specifically described in the guidelines cited above. In these cases, the following approach is taken:

- Review other sources of test methods such as AOAC, ASTM, Pesticide Manual, etc., to find a suitable method for the matrix and analyte in question.
- Produce a modification of a standard test procedure for similar parameter or matrix
- Develop a special method in house suitable for the particular problem

For these special situations the analytical procedure is discussed with the client and performed upon the client's approval. Whenever possible, the same QA/QC guidelines as for standard methods are used, but the laboratory may deviate from these guidelines if necessary.

The Laboratory in some instances must deviate from prescribed environmental test methods; if this occurs the deviation is documented, technically justified, authorized, and accepted by the client.

The Laboratory maintains Standard Operating Procedures (e.g., SOPs, Laboratory Method Manual) that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods. The SOPs provide all information needed to perform the different analytical tasks in accordance with regulatory requirements and in a consistent and controlled manner following the guidelines described in this QAP manual. All technical SOPs (e.g., sample preparation, analytical procedures, sample storage, sample receipt, etc.) are reviewed for accuracy and adequacy annually and whenever method procedures change, and updated as appropriate. Copies of all SOPs, both electronic and paper, are accessible to all personnel. Each SOP has an alphanumeric code that indicates the section it belongs, the number that identifies it, the revision number, the effective date and the signature of the QA Officer, Technical Director or Laboratory Director.

If other documents besides laboratory generated SOPs (i.e. equipment manuals, copies of published methods, etc.) are used as Standard Operating Procedures, they must be written in a way that they can be used as written and any changes, including the use of a selected option must be documented and included in the laboratory's SOP manual. For DoD related work, where published methods are specified as required for a project, requirements contained within that method shall be followed and any modifications to existing method requirements will require project-specific approval by DoD personnel.

SOPs are written in a standardized format and with standardize contents, as indicated in SOP MIS048.

A current list of the Standard Operating Procedures in use is in Appendix 7.

## 11.1 Test Methods

## 11.1.1 Source of Methods

The sources of Methods used at the laboratory are the following:

- Methods published in international, regional or national standards are preferably used, ensuring that the latest valid edition of a standard is used unless it is not appropriate or possible to do so. When necessary, the standard shall be supplemented with additional details to ensure consistent application.
- When the use of specific methods for a sample analysis are mandated or requested, only those methods shall be used.
- When the client does not specify the method to be used or where methods are employed that are not required, as in the Performance Based Measurement System approach, the methods shall be fully documented and validated, and be available to the client and other recipients of the relevant reports. The laboratory shall select appropriate methods that have been published either in international, regional or national standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer of the equipment. In some cases Laboratory-developed methods or methods adopted by the laboratory might be used if they are appropriate for the intended use and if they are validated. The client shall be informed as to the method chosen.
- The client is informed when the method proposed by the client is considered to be inappropriate or out of date.

The Laboratory in some instances will develop methods for its own use; in this case this is considered a planned activity and will be assigned to qualified personnel equipped with adequate resources. Plans shall be updated as development proceeds and effective communication amongst all personnel involved shall be ensured.

When it is necessary to use methods not covered by standard methods, these shall be subject to agreement with the client and shall include a clear specification of the client's requirements and the purpose of the environmental test and/or calibration. The method developed shall have been validated appropriately before use.

For multi-analyte methods, the laboratory uses a standard set of target analytes but those target analytes identified by the client on a project specific basis will be analyzed. If project-specific information is not available, then the standard list of analytes or the list published in the method will be used.

Most methods in use at the laboratory are described in the following publications:

- Tests Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, current edition,
- Methods for Chemical Analysis of Water and Wastewater, EPA-600/4-79-020.
- Standard Methods for the Examination of Water and Wastewater, current approved edition, APHA, AWWA, WPCF.
- Criteria for Identification of Hazardous and Extremely Hazardous Wastes, California Code of Regulations Title 22.
- Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater EPA-600/4-82-057.
- Recommended Methods of Analysis for the Organic components required for AB1803, 5th Edition Revised April 1986.

- Draft Method for Total Petroleum Hydrocarbons and Total Organic Lead, LUFT Methods, California Department of Health Services.
- Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water EPA 500 series.
- NIOSH Manual of Analytical Methods, US Department of Health and Human Services.
- Laboratory Methods of Analysis for Enforcement samples, SCAQMD, 1986.
- Stationary Source Test Methods, Air Resources Board, 1990.
- OSHA Analytical Methods Manual, 2nd Ed., U.S. Dept. of Labor, 1990.

Reference methods for all analytical procedures are kept in the Laboratory Office. Copies of specific methods are also in the corresponding sectors where the analyses are performed.

### **11.1.2 Validation of Methods**

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

The laboratory shall validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use. The validation shall be as extensive as is necessary to meet the needs of the given application or field of application using quality control procedures and acceptance criteria that are consistent with those of similar standard methods or technology. At a minimum, quality control procedures must address:

- Calibration;
- Interferences/contamination;
- Analyte identification;
- Selectivity;
- Sensitivity;
- Precision and Bias.

The laboratory shall record the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use.

The range and accuracy of the values obtainable from validated methods (e. g. the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), as assessed for the intended use, shall be relevant to the clients' needs and in most cases it requires prior approval from the client.

The minimum requirements for method validation are the ones specified in Appendix C.3 of NELAC chapter 5.

#### **11.1.3 SOPs for Sample Management**

These SOPs describe the receipt, handling, scheduling, and storage of samples.

<u>Sample receipt and handling</u> – These procedures describe the precautions to be used in opening sample shipment containers and how to verify that chain of custody has been maintained, examine samples for damage, check for proper preservatives and temperatures, and log samples into the laboratory sample streams.

<u>Sample scheduling</u> – These procedures describe the sample scheduling in the laboratory and includes procedures used to ensure that holding time requirements are met.

<u>Sample storage</u> – These procedures describe the storage conditions for all samples, verification and documentation of daily storage condition, and how to ensure that custody of the samples is maintained while in the laboratory.

### 11.1.4 SOPs for Reagent/Standard Preparation

These SOPs describe how to prepare standards and reagents. Information concerning specific grades of materials used in reagent and standard preparation, appropriate glassware and containers for preparation and storage, and labeling and record keeping for stocks and dilutions is included.

### 11.1.5 SOPs for General Laboratory Techniques

These SOPs describe all essentials of laboratory operations that are not addressed elsewhere. These techniques include glassware cleaning procedures, operation of analytical balances, pipetting techniques, and use of volumetric glassware, among others.

Procedures for test methods describing how the analyses are actually performed in the laboratory are specified in method SOPs. These SOPs for sample preparation, cleanup and analysis are based on publications listed in Section 11.1 above or on internally developed methods validated according to EPA's Performance-Based Measurement System.

The elements included or referenced in the SOPs, when applicable are the following:

- 11.1.1 Identification of the test method
- 11.1.2 Applicable matrix or matrices
- 11.1.3 Method detection limit
- 11.1.4 Scope and application, including components to be analyzed
- 11.1.5 Summary of the method
- 11.1.6 Definitions
- 11.1.7 Interferences
- 11.1.8 Safety
- 11.1.9 Equipment and supplies
- 11.1.10 Reagents and standards
- 11.1.11 Sample collection, preservation and handling
- 11.1.12 Quality control
- 11.1.13 Calibration and Standardization
- 11.1.14 Procedure
- 11.1.15 Calculations
- 11.1.16 Method Performance
- 11.1.17 Pollution prevention
- 11.1.18 Data assessment and acceptance criteria for quality control measures

- 11.1.19 Corrective actions for out-of-control data
- 11.1.20 Contingencies for handling out-of-control or unacceptable data
- 11.1.21 Waste management
- 11.1.22 References
- 11.1.23 Tables, Diagrams, flowcharts and data verification checklists.

### 11.1.6 SOPs for Equipment Calibration and Maintenance

These SOPs describe how to ensure that laboratory equipment and instrumentation are in working order. These procedures include calibration procedures and schedules, maintenance procedures and schedules, maintenance logs, services agreements for all equipment, and spare parts available in-house. Calibration and maintenance of laboratory equipment and instrumentation are in accordance with manufacturers' specifications or applicable test specifications.

# 12 QUALITY CONTROL DETERMINATIONS

## 12.1 General

The quality control procedures are used for monitoring the validity of environmental tests undertaken. The resulting data is recorded in a computerized database contained within the LIMS system which permits the monitoring of trends and the application of statistical techniques for the reviewing of the results. This monitoring includes among other parameters the use of certified reference materials and/or internal quality control using secondary reference material, participation in interlaboratory comparisons and proficiency-testing programs, replicate tests using the same or different methods, retesting of retained samples and correlation of results for different characteristics of a sample (for example, total phosphate should be greater than or equal to orthophosphate).

Quality control samples are processed in the same manner as field samples. They are analyzed and reported with their associated field samples. If QC results are outside method-specified or project-specified criteria, a corrective action is implemented to correct the problem and prevent incorrect results from being reported, or if no error is encountered to report the samples with appropriate qualifiers. For additional guidance on batch-specific QC samples, refer to the Quality Assurance Matrix contained in the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP).

#### 12.2 Essential QC determinations

The data acquired from QC determinations are used to estimate the quality of analytical data, to determine the need for corrective action in response to deficiencies, and to interpret results after corrective action procedures are implemented. Each method SOP includes a QC section, which addresses the minimum QC requirements for the procedure. The internal QC checks may differ slightly for each individual procedure but in general are described below. The acceptance limits and corrective actions for these QC checks are described in Section 15 and 16 of this manual.

The quality control protocols specified in each analytical method and method SOP are followed, as well as the essential standards outlined in Appendix D of NELAC Chapter 5 or mandated methods or regulations (whichever are more stringent). When it is not apparent which is more stringent the QC in the mandated method or regulations is to be followed.

All quality control measures are assessed and evaluated on an on-going basis, and quality control acceptance criteria is used to determine the usability of the data. The procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist have been established (See Section 9.3, Sample Acceptance Policy)

## 12.2.1 Blanks – Negative Controls

Method Blanks or LRBs are performed at a frequency of one per preparation batch of samples per matrix type. The result of this analysis is one of the QC measures to be used to assess batch acceptance.

The method blank is used to assess the preparation batch for possible contamination during the preparation and processing steps. The method blank is processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure.

The method blank is analyzed at a minimum of 1 per preparation batch or one every 20 environmental samples, whichever is more frequent. The method blank shall consist of a matrix that is similar to the associated samples and is known to be free of the analytes of interest.

Blanks and negative controls are used in microbiological analysis on regular basis. They consist of blanks, sterility checks and known negative cultures. The detailed description is contained in the corresponding SOP.

Blanks are prepared and analyzed in the following situations, or whenever there is a need to obtain further information:

- A blank is extracted for every batch and type of matrix for analysis of semi-volatile organics by GC, GC/MS or HPLC.
- A blank is carried through all the digestion procedures for analysis of metals by AA, ICP or ICP-MS for every batch of samples and type of matrix for each instrument used.
- A blank is carried through the leaching procedures (TCLP, EP TOX, and WET) using the same extraction fluid, bottles and agitators as the samples.
- System/Reagent blanks are analyzed at the beginning of the day prior to calibration, after a high level standard, after changing matrix and after samples that are known or suspected to be very concentrated.
- Reagent blanks are analyzed for all wet chemistry determinations involving titrations or colorimetry and their value are subtracted from the reading of the samples, if appropriate.
- Blanks for mobility procedures (TCLP, ZHE, EP TOX, and WET) are analyzed by the appropriate method.
- Additional field and trip blanks are prepared and analyzed where required or whenever requested by the client

Sometimes the blanks may show detectable amounts of target analytes. In these cases the source of the contamination must be investigated and measures taken to correct, minimize or eliminate the problem if:

• The blank contamination is at or above the reporting limit and exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch or

- The blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.
- The blank contamination otherwise affects the sample results as per the test method requirements or the individual project data quality objectives.
- For DoD samples, in addition to the above, the method blank will be considered contaminated for a particular target analyte if it concentrations exceeds <sup>1</sup>/<sub>2</sub> the reporting limit unless is a common laboratory contaminant such as acetone, methylene chloride, MTBE, zinc and aluminum, among others.

If the method blank is contaminated as described above, then the affected samples shall be reprocessed in a subsequent preparation batch, except when sample results are below the detection limit or LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

#### 12.2.2 Reproducibility and Recovery Determinations – Positive Controls

For the determination of accuracy and precision of the analytical methods, the techniques of fortified blanks, matrix spike/ matrix spike duplicate, sample duplicates and surrogate spiking are used on a regular basis. The frequency is dictated by each analytical method or Standard Operating Procedure (minimum 1 per batch of 20 samples). The results obtained are compared with current acceptance limits (Appendix 8) and recorded in the LIMS. For methods that do not specify the acceptance criterion, this is statistically obtained from data generated at the lab.

For microbiological determination of total and fecal coliforms positive checks are included with each batch analyzed. A more detailed description is included in the corresponding SOP.

### 12.2.2.1 Duplicates

Matrix duplicates are defined as replicate aliquots of the same sample taken through the entire analytical procedure. The results from this analysis indicate the precision of the results for the specific sample using the selected method. The matrix duplicate provides a usable measure of precision only when target analytes are found in the sample chosen for duplication and it is performed on replicate aliquots of actual samples, usually of unknown composition.

The frequency of the analysis of matrix duplicates may be determined as part of a systematic planning process (e.g., Data Quality Objectives) or as specified by the mandated test method. Duplicate analysis is also performed when unusual or suspicious results are obtained or when a higher degree of confidence in the analytical result is desired.

The routine analysis of field duplicates is often impractical (many analytes are frequently not detected) or not possible (not enough sample provided), so the evaluation of precision for most methods is accomplished by comparing the results obtained for matrix spike and matrix spike duplicate determinations (Section 12.1.2.3), rather than analysis of field duplicate samples. This is preferred since in many cases samples with frequent "not detected" results yield no useful information for statistical determinations of precision.

The results from matrix duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD) or another statistical treatment (e.g., absolute differences). The calculation of the RPD is detailed in Section 12.2.2.5.

Results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, internal criteria developed at the laboratory is used, which consists on using a minimum of 20 data points and calculating the maximum acceptable RPD based on 3 standard deviations of the historical values. For matrix duplicates results outside of established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.

# 12.2.2.2 Laboratory Control Sample (LCS)

Laboratory Control Samples are also known as LFBs or Blank Spikes and are defined as a quality system matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS are compared to established criteria and, if found to be outside of these criteria, indicates that the analytical system is "out of control". Any affected samples associated with an out of control LCS shall be reprocessed for re-analysis or the results reported with appropriate data qualifying codes. Note: Samples that are not detected (ND) may be reported with an LCS that failed with high bias, but any qualifier may only be used for two consecutive batches before the problem must be corrected.

At least one LCS is analyzed per preparation batch. Exceptions would be for those analytes for which no spiking solutions are available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. In those instances for which no separate preparation method is used (example: volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

The LCS is a quality system matrix, known to be free of analytes of interest, spiked with known and verified concentrations of analytes. The matrix spike (Sect. 12.2.2.3) may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. Alternatively the LCS may consist of a media containing known and verified concentrations of analytes or as Certified Reference Material (CRM). All analyte concentrations shall be within the calibration range of the methods.

The components to be spiked shall be as specified by the mandated test method or other regulatory requirement or as requested by the client. In the absence of specified spiking components the laboratory shall spike per the following:

- For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.
- For those test methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected should be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2-year period.

a) For methods that include 1-10 targets, spike all components.

- b) For methods that include 11-20 targets, spike at least 10 compounds or 80% of the total, whichever is greater.
- c) For methods with more than 20 targets, spike at least 16 components.

The results of the individual batch LCS are calculated in percent recovery as specified in Sect.12.2.2.5. The individual LCS is compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, internal criteria are generated based on recoveries of past LCSs. To determine these criteria, at least 30 data points generated under the same analytical process are used and the upper and lower acceptance limits are calculated as the "Mean + 3 SD" and "Mean – 3 SD" respectively, where SD is the standard deviation. These statistically derived limits must:

- Meet the limits specified by the project or as stated in the method, if available;
- Should be updated on an annual basis, or as stated in the method, and re-established after major changes in the analytical process (e.g., new instrumentation);
- Should not exclude failed LCS recovery data and statistical outliers from the calculation, unless there is a documented and scientifically valid reason.

Control charts generated from the LIMS are used to detect trends and prevent out-of-control conditions. Control limits are continually monitored for shifts in mean recovery, changes in standard deviation, and development of trends.

A LCS that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with a LCS determined to be "out of control" should be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.

If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. This may not indicate that the system is out of control, therefore corrective action may not be necessary. Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A ME is defined as being beyond the LCS control limit (3 standard deviations), but within the ME limits. ME limit is 4 standard deviations around the mean. The number of allowable marginal exceedances is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes. It will not apply to target analyte to exceed its LCS control limits, even marginally and if this happens the batch is considered not acceptable.

The number of allowable marginal exceedances is as follows:

- 1) >90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit;
- 2) 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit;
- 3) 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit;
- 4) 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit;
- 5) 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit;
- 6) <11 analytes in LCS, no analytes allowed in ME of the LCS control limit;

Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly (i.e. 2 out of 3 consecutive LCS), it is an indication of a systemic problem. The source of the error must be located and corrective action taken.

The procedure to monitor the application of marginal exceedance allowance to the LCS to ensure random behavior consist of establishing a data base with all exceedances and compare the analytes affected on quarterly basis to verify is not the same analyte having the problem.

## 12.2.2.3 Matrix Spikes and Matrix Spike Duplicates

The procedure to determine the effect of the sample matrix on method performance is by analyzing with each preparation batch matrix spikes, matrix spikes duplicates sample duplicates and surrogates, which are designed as data quality indicators for a specific sample using the designated test method. These controls alone are not used to judge laboratory performance.

Matrix specific QC samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. The information from these controls is sample/matrix specific and would not normally be used to determine the validity of the entire batch.

The frequency of the analysis of matrix specific samples is determined as part of a systematic planning process (e.g., Data Quality Objectives) or as specified by the required mandated test method or SOP and it is at a minimum, one per batch of 20 samples or less, per matrix type.

The components to be spiked are the ones specified by the mandated test method or laboratory SOP. Matrix spikes are not performed for analytes for which spiking solutions are not available such as, solids determinations (total suspended, total dissolved, total volatile), pH, color, odor, temperature, dissolved oxygen, BOD, COD or turbidity.

The selected sample(s) for spiking are to be rotated among client samples, as much as possible, so that various matrix problems may be noted and/or addressed. The spiked samples are then analyzed as the other samples in the batch and the recoveries calculated and compared with acceptance limits. Results are recorded in the LIMS, where the analysts or QA Officer can track and manage the results for QC samples. For industrial hygiene samples, unused sample collection media is used for spiking. Samples that are labeled equipment blanks, field blanks or trip blanks must not be used for matrix spiking. All efforts shall be made to obtain additional sample aliquots for matrix spiking; when bottles are prepared in house, additional containers are provided for matrix spikes. If the sample containers are prepared by the client or provided by a third party, good communication should be established with all parties involved in order to obtain enough sample aliquots to perform matrix spiking for all test methods required. If, in spite of all efforts made, there are no extra samples received for matrix spiking, a pair of LCS/ LCS duplicate is analyzed for assessing accuracy and precision.

Any permit specified analytes, as specified by regulation or client requested analytes shall also be included. If there are no specified components, the laboratory shall spike per the following:

• For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

- For those test methods that have extremely long lists of analytes, a representative number may be chosen using the following criteria for choosing the number of analytes to be spiked, but alternating them in order to ensure that all targeted components are included in the spike mixture over a 2 year period.
- For methods that include 1-10 targets, spike all components;
- For methods that include 11-20 targets, spike at least 10 components or 80% of the total, whichever is greater;
- For methods with more than 20 targets, spike at least 16 components.

Some project may require MS/MSD to be performed on their samples (i.e. DoD) in which case these are used for the entire batch if it also contains samples from other clients.

The requirements for MS/MSD are not applicable to all methods (e.g., asbestos, certain air-testing samples, classic chemistry, and industrial hygiene samples). If adequate sample material is not available, then the lack of MS/MSDs shall be noted in the case narrative. Additional MS/MSDs may be required on a project-specific basis.

The results from matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R) and relative percent difference (RPD). The calculations are performed as specified in Sect.12.2.2.5. Results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory established internal criteria determined as described in Sect. 12.2.2.2 for LCSs.

Some projects may have specific criteria such as DoD that require that the results of all MS/MSDs must be evaluated using the same acceptance criteria used for the LCS.

Poor performance in a matrix spike generally indicates a problem with the sample composition, and not the laboratory analysis and is reported to the client whose sample was used for the spike with the appropriate data qualifiers or in the case narrative to assist in data assessment.

## 12.2.2.4 Surrogates

For GC and GC/MS analysis, surrogate standards are added to all samples, blanks and QC samples, prior to sample preparation/extraction, for all organic chromatography test methods except when the matrix precludes its use or when a surrogate is not available. Surrogates are compounds that are very similar in their chemical and chromatographic characteristics as the target compounds but are not present in environmental samples, or at least they are not part of the target compounds list.

Results from recoveries of surrogate standards are compared with acceptance values, which may be mandated by the method, specified in the project by the client or lab generated. Acceptance limits generated at the laboratory are established based on a minimum of 30 valid data points by calculating the mean and standard deviation, the upper limit is set at "mean + 3SD" and the lower limit at "Mean – 3SD".

Surrogates outside the acceptance criteria are evaluated for the effect indicated for the individual sample results. A corrective action is initiated which is guided by the data quality objectives or other site specific

requirements. Results reported from analyses with surrogate recoveries outside the acceptance criteria include appropriate data qualifiers.

#### 12.2.2.5 Equations used for calculations

The following equations are used in the calculation of recovery and RPD:

From duplicate sample:

$$RPD = \frac{S_a - S_b}{((S_a + S_b) \div 2)} x100\%$$
  
Where:  $S_a =$  First sub-sample analyzed  
 $S_b =$  Second sub-sample analyzed

From MS/MSD analysis:

$$RPD = \frac{R_a - R_b}{((R_a + R_b) \div 2)} x100\%$$

Where:

 $R_a$  = Amount of analyte found in Matrix Spike.  $R_b$  = Amount of analyte found in Matrix Spike Duplicate

Recovery of matrix spikes:

Re cov ery = 
$$\frac{SSR - SR}{CA} x100\%$$
  
Where: SSR = Results of spiked sample  
SR = Results of sample (unspiked)  
CA = Concentration of spike added

Surrogate recoveries:

% Re cov ery = 
$$\frac{ConcentrationFound}{ConcentrationAdded} x100\%$$
  
Where: Concentration found = Result obtained after analysis  
Concentration added = Amount of surrogate spiked

#### 12.2.2.6 Quality Control Charts

Quality Control charts can be generated at any time from data stored in the LIMS for recoveries of matrix spikes, LCSs, surrogates and RPD and they are a valuable tool to monitor in real time the performance of the analytical method, providing a graph with the mean and upper and lower warning and acceptance limits (2 and 3 standard deviation respectively).

#### 12.2.3 External References and Control Samples

External Reference Samples or QCS are obtained from various sources are analyzed on a regular basis, minimum quarterly. Reference samples simulating matrix and analytes of interest are purchased from Environmental Resource Associates, Inc. or other NIST approved vendors, and analyzed for drinking water, wastewater, hazardous waste and priority pollutants.

Interlaboratory comparisons are run whenever possible, as well as intralaboratory comparisons by analyzing an analyte by different analytical methods.

## 12.3 Method Detection Limit and Reporting Limits

In general the laboratory utilizes a test method that provides a Limit of Detection (LOD) that is appropriate and relevant for the intended use of the data. LODs are determined by the protocol in the mandated test method or applicable regulation, e.g., Method Detection Limit (MDL) and all sample-processing steps of the analytical method are included. If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

The MDL is defined as the minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

For analytes for which spiking is a viable option, detection limits are determined by a Method Detection Limit (MDL) study for each common matrix (water and soil/solid) by the procedure described in 40CFR Part 136, Appendix B. This procedure consists of spiking seven or more aliquots of the matrix with each compound of interest, at a concentration between 3 and 5 times the estimated MDL. These spiked samples are subject to the entire analytical process and analyzed. The MDL is calculated as follows:

MDL = S x t

Where:

S = Standard deviation of the seven replicates.t = Student's "t" value for 99% confidence for the corresponding number of degrees of freedom. For 7 replicates this number is 3.14.

The method detection limit is initially determined for the compounds of interest in each method and in each matrix (aqueous or soil/solid). Laboratory pure reagent water and Ottawa sand are used as matrices for aqueous and soil/solid matrix respectively.

The detection limit is initially determined for the compounds of interest in each test method in a matrix in which there are neither target analytes nor interferences at a concentration that would impact the results. Detection limits are repeated each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis. The MDL studies are documented in spreadsheets created for that purpose. The documentation includes the matrix type, date of analysis, analyst name or initials, instrument used, values obtained and calculations. The raw data and supporting documents are retained, either attached to the spreadsheet used for calculation or filed by date with the general raw data.

The validity of the LOD shall be confirmed by qualitative identification of the analyte(s) in a QC sample in each quality system matrix containing the analyte at no more than 2-3X the LOD for single analyte

tests and 1-4X the LOD for multiple analyte tests. This verification must be annually performed (for NELAC work, quarterly for DoD work) on every instrument that is to be used for analysis of samples and reporting of data.

A LOD study is not required for any component for which spiking solutions or quality control samples are not available such as temperature, or, when test results are not to be reported to the LOD (versus the limit of quantitation or working range of instrument calibration), according to Appendices D.1.2, D.4.5, D.5.4, and D.6.6 of NELAC chapter 5, 2003. Where an LOD study is not performed, the laboratory may not report a value below the Limit of Quantitation.

The Limit of Quantitation (LOQ) is often referenced as Reporting Level (RL) or Practical Quantitation Limit (PQL). The LOQ is normally set at 10 times the standard deviation. This is equivalent to multiply the MDL (obtained for 7 replicates) by 3.18 and rounding to the nearest 1, 2 or 5. In other cases, for certain methods the reporting limit is obtained by multiplying the MDL by another factor (between 2 and 10). The reporting limit for each analyte in each method is referenced in the corresponding SOP. Some projects may require special LOQs, different of those specified in the SOPs; this can be done providing that the new LOQ is supported by the Limit of Detection or MDL, the concentration level is included in the calibration, and is confirmed for each analyte of concern by analyzing a standard at the LOQ level or near and obtaining a recovery between 50 and 150% of the true value.

Certain projects require reporting all detected analytes, even below the reporting limit; in this case, when an analyte is detected but it is below the PQL, it is reported with a "J" flag indicating that the concentration is only estimated.

The LOQ must be set within the calibration range prior to sample analysis and at a minimum, it must be verified annually (for NELAC work) or quarterly (for DoD work).

The laboratory procedure for establishing the LOQ must empirically demonstrate precision and bias at the LOQ. The LOQ and associated precision and bias must meet client requirements and must be reported. If the method is modified, precision and bias at the new LOQ must be demonstrated and reported

Unless the analytical method specifies otherwise, the LOQ is confirmed for each analyte of concern by analyzing a standard at the LOQ level or near and obtaining a recovery between 50 and 150% of the true value. This confirmation is not performed for any component or property for which spiking solutions or quality control samples are not commercially available or otherwise inappropriate (e.g., pH).

In certain cases the recovery of each analyte must be within the established test method acceptance criteria or client data quality objectives for accuracy.

In some cases project-specific reporting limits are used, when the DQOs mandate a different reporting limit than the RLs used routinely by Weck Laboratories.

For potable water analysis, the Detection Limit for Reporting purposes (DLRs) is used instead of the actual MDLs or RLs. For this matrix the calculated MDL must not be greater than the DLR. DLRs are verified on regular basis by including the lowest calibration point at or below the DLR.

## 12.4 Selectivity

Absolute retention time and relative retention time aid in the identification of components in chromatographic analyses and to evaluate the effectiveness of a column to separate constituents. Acceptance criteria for retention time windows are documented in the corresponding method SOP or in the SOP ORG074.

A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. The confirmation is documented in the bench sheets and/or the LIMS.

When reporting data for methods that require analyte confirmation using a secondary column or detector, project-specific reporting requirements shall be followed. If project-specific requirements have not been specified, the reporting requirements in the method are followed. If the method does not include reporting requirements, the results from the primary column or detector are reported, unless there is a scientifically valid and documented reason for not doing so.

Results that are unconfirmed, or for which confirmation was not performed, shall be identified in the test report, using appropriate data qualifier flags, and explained in the narrative. The laboratory shall use method-specified acceptance criteria for analyte confirmation. If method-specific criteria do not exist, the analyte confirmation is performed as specified in SOP MIS052.

Other procedures for evaluating selectivity are described in the analytical methods, which may include mass spectral tuning, ICP inter-element interference checks, sample blanks, spectrochemical absorption or fluorescence profiles, co-precipitation evaluations, and electrode response factors. Acceptance criteria for mass spectral tuning are contained in the corresponding SOPs.

### 12.5 Demonstration of Method Capability

Prior to acceptance and use of any method, satisfactory initial demonstration of method performance is required. The initial demonstration of method performance is performed each time there is a significant change in instrument type, personnel or test method and includes verification of method sensitivity, precision, and bias in each quality system matrix of concern. "Change" refers to any change in personnel, instrument, test method, or sample matrix that potentially affects the precision and bias, sensitivity, or selectivity of the output (e.g., a change in the detector, column type, matrix, or other components of the sample analytical system, or a method revision). The process is described in Appendix 9. A Certification Statement is completed for each analyst documenting that this activity has been performed (Appendix 9). The associated records supporting the activity are also retained at the laboratory and they are available to reproduce the analytical results summarized in the Certification Statement.

The demonstration of method capability consists of performing the analysis on a clean quality system matrix, which has been spiked with the compounds of interest or purchased from a certified vendor. For analysis that require the use of a specialized "work cell" (a group consisting of analysts with specifically defined tasks that together perform the test method), the group as a unit performs the IDC. The supporting documentation is also kept at the laboratory.

When a work cell is employed, and the members of the cell change, the new employee works with experienced analysts in the specialty area and this new work cell demonstrates acceptable performance through acceptable continuing performance checks, such as laboratory control samples. This continued performance check is documented and the four preparation batches following the change in personnel is monitored to ensure that none of the batches result in the failure of any batch acceptance criteria (method blank and laboratory control sample). If there is a failure, the demonstration of capability is repeated. When the entire work cell is changed or replaced, the new work cell repeats the demonstration of capability (Appendix 9).

When a work cell(s) is employed the performance of the group (work cell) is linked to the training records of the individual members of the work cell. Each member of the work cell must demonstrate proficiency in his/her area(s) of responsibility. A work cell may not be defined as a group of analysts who perform the same step in the same process (for example, extractions for Method 8270) represented by one analyst who has demonstrated proficiency for that step.

A continuing demonstration of capability (DOC) is also performed for methods used. The continuing DOC, as the initial DOC, includes verification of method sensitivity, precision, and bias in each quality system matrix of concern by performing a quarterly Limit of Detection (LOD) verification to verify method sensitivity and a Limit of Quantitation (LOQ) verification quarterly (for DoD work) or annually (for NELAC work), to verify precision and bias at the LOQ. LCS and other QC samples are used to verify precision and bias of the quantitation range.

For test methods that have been in use by the laboratory before July 1999, and there have been no significant changes in instrument type, personnel or test method, the continuing demonstration of method performance and the analyst's documentation of continued proficiency is considered acceptable. Records are kept on file to demonstrate that a demonstration of capability is not required.

For new methods that need to be implemented, a validation procedure is documented before they are used in the laboratory. Appropriate method validation techniques include the following:

- Testing of reference standards or reference materials;
- Comparison of results to those achieved using other validated, standard methods
- Interlaboratory comparisons.

When the above techniques are not feasible, the following options are used:

- Systematic assessment of factors that could influence the result; and/or
- Assessment of the precision and bias of the result based on the science of the method and practical experience.

## 12.6 Performance and Proficiency Testing Programs

The following are the proficiency testing programs in which the laboratory currently participates on regular basis:

- Drinking water analysis: WS Studies
- Wastewater analysis: WP studies
- Hazardous waste and soil

- Bacteriological Performance Evaluation Study.
- Radiochemistry

The Proficiency Testing samples are purchased from NIST approved vendors, as per NELAC regulations.

For DoD related work, PT samples are obtained from a Proficiency Testing Oversight Body (PTOB)/Proficiency Testing Provider Accreditor (PTPA)-approved PT Provider.

The PT samples are analyzed and the results returned electronically to the PT Provider by the closing date of the study, which is no later than 45 calendar days from study opening. All PT samples are handled (i.e., managed, analyzed, and reported) by the laboratory management and individual analysts in the same manner as real environmental samples utilizing the same staff, methods as used for routine analysis of that analyte, procedures, equipment, facilities, and frequency of analysis. When analyzing a PT sample, the same calibration, laboratory quality control and acceptance criteria, sequence of analytical steps, number of replicates and other procedures are employed as used when analyzing routine samples.

In addition to the required PT studies, the laboratory participates in other special PT programs managed by government agencies or private entities.

### 12.7 Additional Quality Control Checks

The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

Glassware shall be cleaned to meet the sensitivity of the test method. The cleaning and storage procedures that are not specified by the test method are documented in the method SOPs or in SOP MIS028 for cleaning protocols.

Whenever possible, additional QC checks are performed such as running a sample using different techniques and different standards (EPA Method 602 & EPA Method 624), correlations between COD, BOD and TOC; TDS & Specific Conductivity, balance between cations and anions on water analysis, etc.

#### 12.8 Estimation of Uncertainty of Measurement

A procedure to estimate the uncertainty of measurement for all analytical methods used at the laboratory has been established.

In certain cases the nature of the test method may preclude rigorous, metrologically and statistically valid, calculation of uncertainty of measurement. In these cases the laboratory shall attempt to identify all the components of uncertainty and make a reasonable estimation, and shall ensure that the form of reporting of the result does not give a wrong impression of the uncertainty. Reasonable estimation shall be based on knowledge of the performance of the method and on the measurement scope and shall make use of, for example, previous experience and validation data.

The need of estimating uncertainty will be considered satisfied where a well-recognized test method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form

of presentation of calculated results and the test method and reporting instructions are followed appropriately.

When estimating the uncertainty of measurement, all uncertainty components which are of importance in the given situation shall be taken into account using appropriate methods of analysis.

The estimation of uncertainty will be performed only on the portion of measurement that is under the control of the laboratory. The test reports shall include a statement of the estimated uncertainty of measurement only when required by client instruction. If a specific project requires measurement uncertainty to be reported, the laboratory shall report the estimated uncertainty based on project-specific procedures or, if not available, any other scientifically valid and documented procedures. The estimated measurement uncertainty can be expressed as a range  $(\pm)$  around the reported analytical results at a specified confidence level. In-house, statistically-derived LCS control limits based on historical LCS recovery data may be reported as an estimate of the minimum laboratory contribution to measurement uncertainty at a 99% confidence level.

## 13 DATA REDUCTION, VERIFICATION AND REPORTING

### 13.1 Laboratory worksheets - Raw data documentation

Upon acceptable receipt of samples by the laboratory, sample worksheets are generated for the required testing. These worksheets are distributed to the respective laboratory departments. A paperless system has been implemented for some departments, in which case paper worksheets are not generated at this stage but analysts can obtain information about pending samples and holding times from the LIMS.

The data that are being obtained, such as weights, extraction volumes, calculations, etc. are recorded in the worksheets or in the LIMS. "Bench sheets" are generated either from the data entered in the LIMS or manually for all raw data being produced.

After raw data is entered in the corresponding worksheets and run logs, it is initialed by the analyst and saved chronologically for future review. All electronic raw data is stored in magnetic tapes or CDs.

#### 13.2 Data Reduction and Review

Some instruments have a computerized data reduction and calculation, such as GC/MS, HPLC, GC and ICP. The protocols to perform these tasks are described in the corresponding SOPs and the computer programs used for data reduction are validated before use and checked periodically by manual calculations.

Internal data review consists of a tiered or sequential system of verification, consisting of at least three tiers, with each check performed by a different person. The three tiers include a 100% review of the entire data package and completion of corresponding Data Review Checklist the analyst, then a 100% verification review by a technically qualified person, such as a supervisor or another chemist, experienced in that particular method or procedure, who checks for proper integration of peaks, identification of compounds, QC, etc. The third review is mainly an administrative one, to check for accuracy and completeness, typically performed by the Project Manager in charge of that project. The procedures used for performing the data review are detailed in the SOP MIS018.

If a discrepancy is noted in any stage of the reviewing process, the package is returned to the primary analyst for corrective action. For analyses that do not have automatic data reduction, the analyst performs the necessary calculations to obtain the final result, and then the results are reviewed as indicated above.

All information used in the calculations (e.g., raw data, calibration files, tuning records, results of standard additions, interference check results, sample response, and blank or background correction protocols) as well as sample preparation information (e.g., weight or volume of sample used, percent dry weight for solids, extract volume, dilution factor used) are recorded in order to enable reconstruction of the final result.

As described in Section 16, the results of the quality control sample analysis are reviewed, and evaluated before data are reported.

After the results are entered into the LIMS, the third tier is completed and if no discrepancies are encountered they are released for reporting.

If electronic audit trail functions are available, they must be in use at all times, and associated data must be accessible. If the instrument does not have an audit trail, the integrity of the data is documented as described in SOP MIS043 Implementation of the Business Ethics and Data Integrity Policy.

### 13.3 Report Format and Contents

After the data is entered in the LIMS and approved, a report or "Certificate of Analysis" is generated from the information contained in the LIMS database. The certificate of analysis, containing the results of each test, or series of tests, is then submitted with all supporting documentation to the Project Manager for signature. Other authorized signatory personnel include the Lab Technical Director, QA Officer or Lab Manager. The signature could be either in the form of "wet signature" or "electronic signature" which is stored in the LIMS database.

The analytical report, of which the Chain of Custody Document is part, contains the following information, at a minimum:

- Header with complete laboratory information.
- Unique identification of each page and an indication of the total number of pages included in the report
- Client's information (Company name, address, contact person, etc.)
- Project name or number
- Lab ID number assigned to the sample (unique identification number).
- Description and unambiguous identification of the sample(s) including the client identification code.
- Sample login information (date, time and initials of person that received the sample)
- Sampling information (date, time, name of sampler)
- If the laboratory collected the sample, reference to sampling procedure.
- Analysis performed.
- Results obtained with reporting units
- Date of preparation and analysis

- Time of preparation and/or analysis for tests with holding times of equal or less than 72 hours when required to demonstrate that the test was performed within holding times (the time of preparation/analysis can be entered in the case narrative section of the report).
- Name of method used for preparation and analysis
- Minimum Reporting Level or PQL
- Identification of results for any sample that did not meet sample acceptance requirements.
- Signature of authorized person (Lab Manager, Lab Director, etc.)
- Any additional information that is important to be reported.
- Any deviations from, additions to, or exclusion from SOPs; any conditions that may have affected the quality of results and any failures (such as failed quality control), including the use and definitions of data qualifiers (appendix 12).
- Measurements, examinations and derived results, supported by tables, graphs, sketches and photographs as appropriate, and any failures identified; identification of whether data are calculated on dry weight basis; identification of the reporting units such as ug/l or mg/kg
- Clear identification of all test data provided by outside sources, such as subcontracted laboratories, clients, etc.
- Clear identification of numerical results with values below the RL (J qualifier).

Exceptions to this standard approach for reporting are allowed with the approval of the QA Manager and should be documented; for DoD related work, both date and time of preparation and analysis are considered essential information, regardless of the length of the holding time, and shall be included as part of the laboratory report. If the time of the sample collection is not provided, the laboratory must assume the most conservative time of day (i.e., earliest).

Any result not obtained in accordance with the approved method and the lab QA Plan by use of proper lab technique, must be documented as such in the case narrative section of the Certificate of Analysis.

Material amendments to a test report after issue are made only in the form of a further document, or data transfer including the statement "Supplement to Certificate of Analysis, identification number".

Clients are notified promptly, in writing, of any event such as the identification of defective measuring or test equipment that cast doubt on the validity of results given in any test report or amendment to a report.

Test results are certified to meet all requirements of the NELAC standards, or reasons are provided if they do not. After signed, the Certificates of Analysis are sent to the client by US mail. In some cases the report is submitted by facsimile, electronically or electromagnetically. In this last case, all reasonable steps are taken to preserve confidentiality and the data is only sent to fax numbers or email addresses properly authorized by the client. Hard copies are submitted by US Mail.

## 13.4 Records

Records provide the direct evidence and support for the necessary technical interpretations, judgments, and discussions concerning laboratory results. These records, particularly those that are anticipated to be used as evidentiary data, provide the historical evidence needed for later reviews and analyses. Records must be legible, identifiable, and retrievable, and protected against damage, deterioration or loss. All records referenced in this section are retained for a minimum of ten years.

The laboratory has established and maintain procedures to control all documents that form part of its quality system (internally generated or from external sources), such as regulations, standards, other normative documents, environmental test and/or calibration methods, as well as drawings, software, specifications, instructions and manuals. Documents include policy statements, procedures, specifications, calibration tables, charts, textbooks, posters, notices, memoranda, software, drawings, plans, etc. These may be on various media, whether hard copy or electronic, and they may be digital, analog, photographic or written.

A procedure has been established to review and approve for use by authorized personnel prior to issue, all documents issued to personnel in the laboratory as part of the quality system. The procedure also establishes a document control system and the policy to be followed with invalid and/or obsolete documents.

Laboratory records generally consist of bound notebooks with pre-numbered pages, official laboratory worksheets, personnel qualifications and training forms, facilities, Corrective Action reports, PT records, equipment maintenance and calibration forms, chain-of-custody forms, sample analysis request forms, and analytical change request forms. All records are recorded in indelible ink and retained for ten years. Records that are stored or generated by computers have hard copy or write protected backup copies. Electronic records are supported by the hardware and software necessary for their retrieval.

Any documentation changes are corrected by drawing a single line through the change so that it remains legible and is initialed by the responsible individual, along with the date of change and reason. The correction is written adjacent to the error. Strip-chart recorder or computer printouts are signed by the person who performed the instrumental analysis. If corrections need to be made in computerized data, a system parallel to the corrections for handwritten data is used.

In the event the Laboratory is sold, all past records shall be transferred to the custody of the new legal owner or operator of the Laboratory.

This management however shall maintain responsibility and accountability for laboratory work performed prior to the transfer. A written statement to this effect shall be provided. The new owner/operator shall be accountable and liable for all work performed after the transfer date and he/she shall provide a written statement to that effect.

In the case the laboratory goes out of business, the present management shall maintain custody of all records and make them available to clients for a period of ten years.

Laboratory records include the following:

#### 13.4.1 Standard Operating Procedures

SOPs are controlled documents. They are reviewed on regular basis and if there are any revisions, these are distributed to all affected individuals to ensure implementation of changes. All revisions of SOPs are archived for historical reference, per regulatory or client requirements.

#### 13.4.2 Equipment Maintenance Documentation

Documents detailing the receipt and specification of analytical equipment are retained. A history of the maintenance record of each system serves as an indication of the adequacy of maintenance schedules and parts inventory. As appropriate, the maintenance guidelines of the equipment manufacturer are followed. When maintenance is necessary, it is documented in either standard forms or in logbooks.

## 13.4.3 Calibration Records and Traceability of Standards/Reagents

The frequency, conditions, standards, reagents and records reflecting the calibration history of a measurement system are recorded. These include but are not limited to the source of standards and reagents, receipt, preparation and use.

The overall program of calibration and/or verification and validation of equipment is designed and operated so as to ensure that measurements made by the laboratory are traceable to national standards of measurement.

Calibration certificates indicate the traceability to national standards of measurement and provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. The laboratory maintains records of all such certifications. Where traceability to national standards of measurement is not applicable, the laboratory will provide evidence of correlation of results by participation in a suitable program of interlaboratory comparisons, proficiency testing, independent analysis or other suitable means.

### **13.4.4 Sample Management**

A record of all procedures to which a sample is subjected while in the possession of the laboratory is maintained, including the personnel involved in each activity. These include records pertaining to:

- Sample preservation including appropriateness of sample container and compliance with holding time requirements.
- Sample identification, receipt, acceptance or rejection and log-in
- Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records.
- Disposal of hazardous samples including the date of sample or sub-sample disposal and name of responsible person.
- Automated sample handling systems

## 13.4.5 Original Data

The raw data and calculated results for all samples is maintained in laboratory notebooks, logs, bench sheets, files or other sample tracking or data entry forms. Instrumental output is stored in a computer file and/or a hard copy report. These records include:

- Laboratory sample ID code
- Date of analysis
- Instrumentation identification and instrument operating conditions/parameters
- Analysis type and sample preparation information, including sample aliquots processed, cleanup, and separation protocols.
- All manual, automated, or statistical calculations

- Confirmatory analysis data, when required to be performed
- Review history of sample data
- Analyst's or operator's initials/signature
- All data generated, except those that are generated by an automated data collection system, are recorded directly, promptly and legibly in permanent ink.
- Date of analysis and extraction as well as time if the Hold Time is 72 hours or less.

### 13.4.6 QC Data

The raw data and calculated results for all QC samples and standards are maintained in the manner described in 13.4.5. Documentation allows correlation of sample results with associated QC data. Documentation also includes the source and lot numbers of standards for traceability. QC samples include, but are not limited to, control samples, method blanks, matrix spikes and matrix spike duplicates.

#### 13.4.7 Correspondence

Correspondence pertinent to a project is kept and placed in the project files.

### 13.4.8 Deviations

When a deviation from a documented policy occurs, including SOPs, analytical methods, QA/QC criteria, etc., the laboratory notifies the client of this in the Certificate of Analysis under the case narrative section or in a supplemental report indicating the deviation and the reasons for it.

All deviations from SOPs are reviewed and approved by the QA Officer or Technical Director.

When mistakes occur in records, each mistake is crossed out, leaving it legible, and the correct value and initials of person making the correction are entered alongside.

When corrections are due to reasons other than transcription errors, the reason for the correction is documented.

## 13.4.9 Final Reports

Copies of final reports are kept in each client's file, along with supporting documentation.

#### 13.4.10 Administrative Records

The following are maintained:

- Personnel qualifications, experience and training records
- Initial and continuing demonstration of proficiency for each analyst
- A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record.

## 13.5 Document Control System

The laboratory has established and maintains procedures to control all documents that form part of its quality system (internally generated or from external sources).

A document control system is used to ensure that all personnel have access to current policies and procedures at all times. Documents, which are managed by this system, include this Quality Manual, all SOPs, policy statements, procedures, specifications, calibration tables, charts, textbooks, posters, notices, memoranda, software, drawings, plans, etc. The system consists of a document review, revision and approval system, and document control and distribution. The documents may be on various media, whether hard copy or electronic, and they may be digital, analog, photographic or written.

All quality documents (this manual, SOPs, policies, etc.) are reviewed and approved by the QA Officer, the Technical Directors and the Laboratory Director. Such documents are revised whenever the activity described changes significantly. All documents are reviewed at least every 5 years, with the exception of the QA Manual, which is reviewed annually.

All QA/QC documents are controlled by the QA Officer. Controlled copies are made available to all affected individuals in the laboratory. The QA Officer maintains a distribution list for controlled copies and ensures that any revisions are available.

More detailed procedures related to Document Control are specified in the corresponding SOP (MIS045).

### 13.6 Confidentiality

All analytical reports, results, electronic records and transmission of results are kept in confidence to the customer who requested the analyses and only released to third parties with written permission from a properly authorized representative of the client. This information includes, but is not limited to COCs, Certificates of Analysis, raw data, bench sheets, electronic information and sample results. In addition no information pertaining to clients is posted in public areas where the access is not restricted.

Access to laboratory records and LIMS data is limited to authorized laboratory personnel except with the permission of the QA Officer or Laboratory Director. NELAP-related records are made available to authorized accrediting authority personnel.

#### 13.7 Service to the Client

The laboratory shall afford clients or their representatives' cooperation to clarify the client's request and to monitor the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other clients.

The laboratory shall maintain and document timely communication with the client for the purposes of seeking feedback, both positive and negative, and clarifying customer requests. Feedback shall be used and analyzed to improve the quality system, testing activities, and service to the client.

The following are specific situations for which immediate clarification or feedback is required from the client:

- The client has specified incorrect, obsolete, or improper methods;
- Methods require modification to ensure achievement of project-specific objectives contained in planning documents (e.g., difficult matrix, poor-performing analyte);

- Project-planning documents (e.g., Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP)) are missing or requirements in the documents (e.g., action levels, detection and quantification capabilities) require clarification; or
- The laboratory has encountered problems with sampling or analysis that may impact results (e.g., improper preservation of sample).

#### 14 PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

### 14.1 Internal Laboratory Audits

Annual internal audits are performed to verify that laboratory operations continue to comply with the requirements of the quality system and the corresponding NELAC Standard. The internal audit program shall address all elements of the quality system, including all of the environmental testing activities. The quality assurance officer plans and organizes internal audits as required by a predetermined schedule and requested by management, ensuring that all areas of the laboratory are reviewed over the course of one year. Such audits are performed by the Quality Assurance Officer or personnel designated by the QA officer, who are trained and qualified in the specific quality system element or technical area under review and wherever resources permit, independent of the activity to be audited. Technical personnel are not allowed to audit their own activities unless it can be thoroughly demonstrated that an effective audit will be carried out.

Where the audit findings cast doubt on the correctness or validity of the laboratory's results, an immediate corrective action is initiated and any client must be notified in writing within 30 days of the finding if investigations show that the laboratory results may have been affected.

The laboratory shall notify clients promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in test report or test certificate or amendment to a report or certificate.

The internal system audits include an examination of laboratory documentation and records on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc. Specific records that are subject to review are detailed in the corresponding SOP for performing audits and data review (SOP MIS014).

#### 14.2 Management Review

At least once per year, laboratory executive management conducts a review of the quality system and environmental testing activities to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The management review is a separate activity from the internal audit. The review takes account of the following:

- The suitability of policies and procedures;
- Reports from managerial and supervisory personnel;
- The outcome of recent internal audits;
- Corrective and preventive actions;
- Assessments by external bodies;
- The results of interlaboratory comparisons or proficiency tests;

- Changes in the volume and type of the work;
- Client feedback;
- Complaints;
- Other relevant factors, such as quality control activities, resources and staff training.

The managerial review is performed according to specified procedures detailed in the corresponding SOP and the records of review findings and actions are kept at the laboratory.

The area of activity audited, the audit findings and corrective actions that arise from them shall be recorded. The laboratory management shall ensure that these actions are discharged within the agreed time frame as indicated in this QA manual and/or in the corresponding SOPs. Follow-up audit activities shall verify and record the implementation and effectiveness of the corrective action taken.

The laboratory, as part of their overall internal auditing program, shall insure that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery of potential issues shall be handled in a confidential manner until such time as a follow up evaluation, full investigation, or other appropriate actions have been completed and the issues clarified. All investigations that result in finding of inappropriate activity shall be documented and shall include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients. All documentation of these investigation and actions taken shall be maintained for 10 years.

### 14.3 Other Audits

The Laboratory is also subject to external audits performed by regulatory agencies and clients. The State regulatory agency under which the laboratory is accredited under NELAC performs a bi-annual quality systems audit. The QA Manager and other relevant management personnel ensure that all the items identified in NELAC Chapter 5 Quality Systems are available for on-site inspection at the time they are requested in order to facilitate the audit process.

Audits performed by clients are non-routine and could be part of the evaluation process in selecting a laboratory for a particular project. For these audits, the management personnel can make available all items requested that are relevant to the evaluation of the Quality System and specific QA/QC practices without releasing information that could be considered confidential or pertaining to other clients data.

## 15 FACILITIES, EQUIPMENT AND REAGENTS

#### 15.1 Facilities

The Laboratory is segregated into different areas for operations that are not compatible with each other. This separation prevents contamination of low levels of common laboratory solvents in the volatile organics analyses and maintains culture handling or incubation areas segregated from other areas. The access to the volatile organics laboratory and microbiology laboratory is restricted to appropriate personnel only; signs to that effect are posted on the entry doors of these areas.

It is the policy of the company to assure that the facilities housing the laboratory and the workspaces are adequate to perform the analyses for which it is accredited. These include physical space, energy sources, lighting and environmental conditions, sufficient storage space, workbenches, ventilation, utilities, access

and entryways to the laboratory, sample receipt area(s), sample storage area(s), chemical and waste storage area(s); and data handling and storage area(s). For microbiology, floors and work surfaces shall be non-absorbent and easy to clean and disinfect. Work surfaces shall be adequately sealed and shall be clean and free from dust accumulation. Plants, food, and drink shall be prohibited from the laboratory work area. The company will procure to improve the condition of the facilities whenever possible and make plans for future expansions or improvements.

The laboratory, as per Standard Operating Procedures, monitors, control and records environmental conditions as required by the relevant specifications, methods and procedures or where they influence the quality of the results, for example monitoring biological sterility and other environmental effects, as appropriate to the technical activities concerned. Environmental tests shall be stopped when the environmental conditions jeopardize the results of the environmental tests and/or calibrations.

In order to prevent cross-contamination, samples suspected of containing high concentrations of target analytes shall be isolated from other samples. Samples or extracts designated for volatile organics analysis are stored in separate refrigerators located in volatile organics area, completely segregated from all other samples and extracts. Samples suspected of containing high concentrations of volatile organics are further isolated from other volatile organics samples and samples for volatile organic analysis in potable water are kept in designated refrigerator.

When the project requires it, travel blanks, used as storage blanks, are kept with the samples until the moment of analysis to determine whether or not cross-contamination occurred. The procedures for evaluation of storage blanks, as well as other considerations for incompatible activities as detailed in the SOP MIS036.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality.

### 15.2 Equipment and Equipment Maintenance

The Laboratory is furnished with all items of sampling, measurement and test equipment required for the correct performance of the environmental tests (including sampling, preparation of samples, processing and analysis of environmental data). If the laboratory needs to use equipment outside its permanent control, this equipment must meet the requirements of other lab equipment according to this QA Manual.

The Laboratory acquires only equipment and its software required for testing and sampling that is capable of achieving the accuracy required and that complies with specifications relevant to the environmental tests concerned.

Before being placed into service, equipment (including that used for sampling) is calibrated and/or checked to establish that it meets the laboratory's specification requirements and complies with the relevant standard specifications.

Records are maintained for all major equipment, including documentation of all routine and non-routine maintenance activities.

The records include:

• The name of the equipment

- The manufacturer's name, type identification, and serial number or other unique identification of the equipment and its software.
- Date received and date placed in service (if available)
- Current location, where appropriate.
- If available, condition when received (e.g., new, used, reconditioned)
- Dates and results of calibrations, if appropriate
- Details of routine and non-routine maintenance carried out to date and planned for the future
- History of any damage, malfunction, modification or repair

When purchasing new laboratory equipment and accessories, only reputable brands will be considered and always the instruments that have the best quality shall be considered, regardless of the difference in price with a similar instrument, considered of an inferior quality.

Instruments and equipment are maintained in optimum condition. Frequent inspections, routine preventative maintenance, prompt service, etc. ensure optimal performance.

It is the policy of the company to provide analytical instruments and software adequate to meet the method requirements and the quality control operations specified in both NELAC and the individual methods. Older instruments shall be replaced with newer ones as technology improves and efforts shall be made to provide a greater degree of automation and security in analytical instruments. A list of major instruments and reference materials is in Appendix 4.

Equipment shall be operated by authorized personnel. Up-to-date instructions on the use and maintenance of equipment (including any relevant manuals provided by the manufacturer of the equipment) shall be readily available for use by the appropriate laboratory personnel.

Service contracts or agreements with the manufacturer or instrument Maintenance Company are maintained for the following instruments:

- ICP and/or ICP-MS instruments for metal analysis
- GC/MS units for volatile organics
- Purge and Trap systems and autosamplers
- GC/MS units for semi-volatile organics

The analyst in charge of each particular instrument performs preventive maintenance for all other analytical instruments.

All maintenance and repairs are thoroughly documented in logbooks, with information pertaining to the description of the problem or routine maintenance, date of occurrence and name of person that performed the maintenance operation.

A routine preventive maintenance program is used to minimize the occurrence of instrument failure and other system malfunctions. Designated employees regularly perform routine scheduled maintenance and repair of instruments. They also check that equipment complies with the specifications, design a plan for maintenance, where appropriate, and verify that the maintenance is carried out to date. All laboratory instruments are maintained according with manufacturer's specifications.

Any item of the equipment which has been subjected to overloading or mishandling, or which gives suspect results, or has been shown by verification or otherwise to be defective, is taken out of service, isolated to prevent its use or clearly labeled as being out of service until it has been repaired and shown by calibration, verification or test to perform satisfactorily. The laboratory will examine the effect of this defect or departure from specified limits on previous tests and shall institute the "Control of nonconforming work" or Corrective Action procedures.

The equipment and its software used for testing, calibration and sampling used at the laboratory is capable of achieving the accuracy required and comply with specifications relevant to the environmental tests concerned. Calibration programs are established for key quantities or values of the instruments where these properties have a significant effect on the results. All new analytical and sampling equipment is calibrated or checked to establish that it meets the laboratory's specification requirements and complies with the relevant standard specifications before being placed into service. All pieces of equipment are calibrated or checked before use.

Whenever practicable, all equipment under the control of the laboratory and requiring calibration shall be labeled, coded or otherwise identified to indicate the status of calibration, including the date when last calibrated and the date or expiration criteria when recalibration is due.

When, for whatever reason, equipment goes outside the direct control of the laboratory, the laboratory shall ensure that the function and calibration status of the equipment are checked and shown to be satisfactory before the equipment is returned to service.

Test and calibration equipment, including both hardware and software, shall be safeguarded from adjustments which would invalidate the test and/or calibration results.

Glassware is cleaned to meet the sensitivity of the method. Any cleaning and storage procedures that are not specified by the method are documented in laboratory records or SOPs.

## 15.3 Reagents and Chemicals

The reagents and chemicals used in the laboratory are obtained from reputable suppliers that have proven consistency over the years. Purity specifications are chosen based on the analysis and this is always verified by the analysis of solvent blanks and check standards. In methods where the purity of reagents is not specified, analytical reagent grade are used. Reagents of lesser purity than those specified by the test method are not used. Upon receipt of reagents, the labels on the container are checked to verify that the purity of the reagents meets the requirements of the particular test method. Such information is documented in the corresponding section of the LIMS.

The following are some of the reagents used:

- Solvents used for Gas Chromatography and GC/MS are "organic residue analysis" grade.
- Methanol used for volatile organics by GC or GC/MS is "Purge and Trap" grade.
- All inorganic chemicals are "reagent grade" or better, depending of the requirement.
- Nitric acid used for preparation of standards for ICP/MS analysis is "trace metals".

The quality (e.g., purity) specifications for all standards and reagents (including water) are documented in SOP MIS004.

The quality of reagent water sources used for microbiological analyses is monitored for trace metals, TKN, TOC and bacteria content. The results are documented in the corresponding logbook kept at the Microbiological Lab. On daily basis, the quality of reagent water is monitored by performing method blanks and system blanks for all tests that require water and the results documented with the analytical batch. If the reagent water does not meet method specific requirements a corrective action procedure is initiated.

The concentration of titrants is verified in accordance with written laboratory procedures (SOPs) and documented in the Standardization log book kept in the Wet Chemistry section of the Laboratory.

### 15.4 Analytical Standards and Reference Materials

In general the Laboratory uses reference materials that are traceable, when possible to SI units of measurement, or to certified reference materials. Where possible, traceability shall be to national or international standards of measurement or to national or international standard reference materials. Internal reference materials are checked as far as is technically and economically practicable.

Most of the standards used are purchased as certified solutions from qualified vendors. These stock standards are traceable to NIST, the corresponding documentation, including certificate of analysis or purity, date of receipt, recommended storage conditions, expiration date, lot numbers, etc., is maintained in laboratory files.

All standard containers, both original and of daughter standards, are labeled with an expiration date.

All analytical standards received at the laboratory are inspected for appearance and expiration date, if any. They are recorded in the LIMS, which assigns a unique identification number to assure traceability. The identification number is referenced when a dilution of the stock is made or when a reagent solution is prepared.

All reference materials after they have been properly inspected and logged in, are handled, transported, stored and used, according to the manufacturer's instructions in order to prevent contamination or deterioration and to protect their integrity.

Analytical standards prepared in the laboratory are prepared from certified stock solutions or pure product. Quality Control Standards (QCS) are prepared or obtained from a separate source other than the working standards.

The management does not reject any request from technical personnel to obtain a reference material or any type of instrument or chemical that he or she considers essential for the normal operation of the laboratory.

## 15.5 Computers and Electronic Data Related Requirements

Where computers or automated equipment are used for the acquisition, processing, recording, reporting, storage or retrieval of test data the following are taken into consideration:

- Computer software developed by the user is documented in sufficient detail and is suitably validated as being adequate for use;
- Procedures are established and implemented for protecting the data; including, but not limited to, integrity and confidentiality of data entry or collection, data storage, data transmission and data processing;
- Computers and automated equipment are maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of environmental test data.
- Establishment and implementation of appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.
- Commercial off-the-shelf software (e. g. word processing, database and statistical programs) in general use within their designed application range is considered to be sufficiently validated, however, laboratory software configuration or modifications must be validated.
- All aspects of electronic data management shall be addressed. At a minimum, a sample data set shall be used to test and verify the operation of all automated data reduction processes (including data capture, manipulation, transfer, and reporting). This shall be done any time new software (including commercially available software) is installed or programming code is modified or manipulated.

## 16 SPECIFIC ROUTINE PROCEDURES USED TO EVALUATE DATA QUALITY

Quality control acceptance criteria are used to determine the validity of the data based on the analysis of internal quality control check (QC) samples (see section 11). The specific QC samples and acceptance criteria are found in the laboratory SOPs. Typically, acceptance criteria are taken from published EPA methods. Where no EPA criteria exist, laboratory generated acceptance criteria are established. Acceptance criteria for bias are based on historical mean recovery plus or minus three standard deviation units, and acceptance criteria for precision range from zero (no difference between duplicate control samples) to the historical mean relative percent difference plus three standard deviation units. Analytical data generated with QC samples that fall within prescribed acceptance criteria indicate the laboratory was in control. Data generated with QC samples that fall outside the established acceptance criteria indicate the laboratory was "out of control" for the failing tests. These data are considered suspect and the corresponding samples are reanalyzed or reported with qualifiers.

Many published EPA methods do not contain recommended acceptance criteria for QC sample results. In these situations, Weck Laboratories, Inc. uses 70 - 130 % as interim acceptance criteria for recoveries of spiked analytes, until in-house limits are developed. In-house limits are based on a 95% confidence interval and should include all historical data points (minimum of 20 data points).

#### 16.1 Laboratory Control Samples

A Laboratory Control Sample is analyzed with each batch of samples to verify that the accuracy of the analytical process is within the expected performance of the method.

The results of the LCS are compared to acceptance criteria to determine usability of the data. Data generated with LCS samples that fall outside the established acceptance criteria are judged to be out-of-

control. These data are considered suspect and the corresponding samples are reanalyzed or reported with qualifiers.

LCS samples are prepared in each corresponding matrix (reagent water for aqueous and Ottawa sand for soil/solid), which must be free of the target analytes to be analyzed.

## 16.2 Matrix Spikes/Matrix Spike Duplicates

Results from MS/MSD analyses are primarily designed to assess data quality in a given matrix, and not laboratory performance. In general, if the LCS results are within acceptance criteria, performance problems with MS/MSD results may either be related to the specific sample matrix or to an inappropriate choice of extraction, cleanup, or determinative methods. If any individual percent recovery in the matrix spike (or matrix spike duplicate) falls outside the designated acceptance criteria, Weck Laboratories, Inc. will determine if the poor recovery is related to a matrix effect or a laboratory performance problem. A matrix effect is indicated if the LCS data are within acceptance criteria but the matrix spike data exceed the acceptance criteria.

#### 16.3 Surrogates Recoveries

Surrogates are exclusively used in organic analysis. Surrogate recovery data from individual samples are compared to surrogate recovery acceptance criteria in the methods. As for MS/MSD results, surrogate recoveries are used primarily to evaluate data quality and not laboratory performance.

### 16.4 Method Blanks

Method blank analyses are used to assess acceptance of sample results. The source of contamination is investigated and measures taken to correct, minimize or eliminate the problem in the situations detailed in Section 12.1.1.

Any sample associated with the contaminated blank is reprocessed for analysis or the results reported with appropriate qualifying codes.

## 17 NON-COMFORMING WORK, CORRECTIVE ACTION AND PREVENTIVE ACTION

#### 17.1 Control of Nonconforming Environmental Testing Work

A policy has been established to handle situations when any aspect of the Laboratory's environmental testing work, or the results of this work, do not conform to its own procedures or the agreed requirements of the client. The procedures to be implemented when this situation occurs are detailed in the corresponding SOP (MIS044).

## **17.2** Corrective Action

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out of control QC performance that can affect data quality. To the extent possible, samples are reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with

the failed quality control measure are reported with the appropriate data qualifier(s). Sample results may also be qualified when holding times are not met, improper sample containers and/or preservatives are used or when other deviations from laboratory standard practices and procedures occur.

Corrective action in the laboratory may occur prior to, during and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low or high pH readings, and potentially high concentration samples may be identified during sample login or just prior to analysis. The SOPs specify conditions during and after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, and automatic reinjection/reanalysis when certain QC criteria are not met.

Any QC sample result outside of acceptance limits requires corrective action. Once the problem has been identified and addressed, corrective action may include the reanalysis of samples, or appropriately qualifying the results.

The analyst will identify the need for corrective action. The Technical Director will approve the required corrective action to be implemented by the laboratory staff. The QA Officer will ensure implementation and documentation of the corrective action.

Corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both a corrective action log (Appendix 10), signed by the personnel involved, and the narrative in the data report.

Where a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the quality of the laboratory's tests, the laboratory shall ensure that those areas of activity and responsibility involved are promptly audited in accordance with internal audit procedures established under this QA Manual. All complaints received at the laboratory from clients or other parties shall be treated according to the corresponding standard operating procedure for its resolution. Records of the compliant and subsequent actions are maintained for future review.

There are some cases in which the QC checks do not fail but the analyst or supervisor discovers that an unexpected or contradictory result has been obtained. These situations are considered also as "Out-Of-Control" and an investigation is carried out.

The investigations/corrective action procedures include but are not limited to:

- Identification of the individuals responsible for assessing each QC data type
- Identification of the individuals responsible for initiating and/or recommending corrective actions
- Definition of how the analyst should treat the data set if the associated QC measurements are unacceptable
- Investigate the probable cause of irregularity and determine the root cause(s) of the problem.
- Review the sample's documented history.
- Review the documentation for errors.
- Scrutinize the sample preparation (digestion, extraction, dilutions, cleanup, etc.)
- Verify standards with reference materials.
- Re-analyze the sample if possible.
- Investigate alternate methodologies.

- If the event is determined to be matrix dependent the data is reported with a qualifier.
- Definition of how out-of-control situations and subsequent corrective actions are to be documented
- Definitions of how management, including the QA Officer, review corrective action reports

Where corrective action is needed, the laboratory shall identify potential corrective actions. It shall select and implement the action(s) most likely to eliminate the problem and to prevent recurrence.

Corrective actions shall be to a degree appropriate to the magnitude and the risk of the problem. The laboratory shall document and implement any required changes resulting from corrective action investigations.

The laboratory shall monitor the results to ensure that the corrective actions taken have been effective.

Where the identification of nonconformances or departures casts doubts on the laboratory's compliance with its own policies and procedures, or on its compliance with the NELAC Standard, the laboratory shall ensure that the appropriate areas of activity are audited in accordance with Section 14.1 of this Manual, Internal Laboratory Audits as soon as possible.

## 17.3 Preventive Action

Preventive action is a pro-active process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

Needed improvements and potential sources of nonconformances, either technical or concerning the quality system, shall be identified. If preventive action is required, action plans shall be developed, implemented and monitored to reduce the likelihood of the occurrence of such nonconformances and to take advantage of the opportunities for improvement.

Procedures for preventive actions shall include the initiation of such actions and application of controls to ensure that they are effective.

## 18 SUBCONTRACTING AND SUPPORT SERVICES AND SUPPLIES

#### 18.1 Subcontracted Laboratory Services

A subcontracted laboratory will be used only if Weck Laboratories does not have the capability of performing the requested test, because of unforeseen reasons (e. g. workload, need for further expertise or temporary incapacity) or if the client specifically requests a particular analysis to be subcontracted. Weck Laboratories advises the client in writing or by other means of its intention to subcontract any portion of the testing to another party, and when appropriate, gain the approval of the client, preferably in writing.

When subcontracting any part of the testing, this work will be placed with a laboratory accredited under NELAP for the tests to be performed or with a laboratory that meets applicable statutory and regulatory requirements for performing the tests and submitting the results of tests performed.

For DoD related work, only subcontracted laboratories accredited by DoD or its designated representatives will be used. Subcontracted laboratories must receive project-specific approval from the DoD client before any samples are analyzed.

The corresponding records demonstrating that the above requirements are met are retained (e.g., copies of the subcontracted lab certifications, communications with the client, etc.).

When subcontracted laboratories are used, this is indicated in the Certificate of Analysis and a copy of the subcontractor's report is kept in file in case the client requests it at a later time. Subcontracted work performed by non-NELAP accredited laboratories is also clearly identified in the final report.

Weck Laboratories is responsible to the client for the subcontractor's work, except in the case where the client or a regulatory authority specifies which subcontractor is to be used.

A register of all subcontractors that are routinely used by the laboratory is kept on file, along with evidence of certifications.

#### 18.2 Outside Support Services and Supplies

Weck Laboratories, Inc. only uses those outside support services and supplies that are of adequate quality to sustain confidence in the laboratory's tests. Records of all suppliers for support services or supplies required for tests are maintained. Services and supplies that may affect the quality of environmental tests include, but are not limited to, balance calibration, solvents, standards, and sample containers; their records include the following, where applicable:

- Date of receipt;
- Expiration date;
- Source;
- Lot or serial number;
- Calibration and verification records
- Certifications.

Specific procedures to evaluate, select and monitor suppliers of materials and services as well as required documentation is detailed in the corresponding SOP (MIS042)

#### **19 REFERENCES**

- 19.1 NELAC 2003 Standard
- 19.2 Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans,
- 19.3 QAMS-005/80, December 29, 1980, Office of Monitoring Systems and Quality Assurance, ORD, USEPA, Washington, DC 20460
- 19.4 RCRA QAPP Instructions, USEPA Region 5, Revision: April 1998
- 19.5 ASTM D-5283-92. Generation of Environmental Data Related to Waste Management Activities: Quality Assurance and Quality Control Planning and Implementation.
- 19.6 American National Standards Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs (ANSI/ASQC E-4), 1994.

- 19.7 EPA 2185 Good Automated Laboratory Practices, 1995
- 19.8 ISO/IEC Guide 25: 1990. General Requirements for the Competence of Calibration and Testing Laboratories.
- 19.9 QA/R-2: EPA Requirements for Quality Management Plans, August 1994.
- 19.10 QA/G-4: Guidance for the Data Quality Objectives Process EPA/600/R-96/055, September 1994.
- 19.11 A/R-5: EPA Requirements for Quality Assurance Project Plans Draft November 1997
- 19.12 QA/G-5: Guidance on Quality Assurance Project Plans EPA/600/R-98/018, February 1998.
- 19.13 A/G-6: Guidance for the Preparation of Standard Operating Procedures for Quality Related Operations EPA/600/R-96/027, November 1995.
- 19.14 A/G-9: Guidance for the Data Quality Assessment: Practical Methods for Data Analysis EPA/600/R-96/084, January 1998.
- 19.15 Manual for the Certification of Laboratories Analyzing Drinking Water EPA/570/9-90/008.
- 19.16 ISO. 2005. General requirements for the competence of testing and calibration laboratories. ISO 17025
- 19.17 DoD Quality Systems Manual for Environmental Laboratories, Version 4, dated 3/19/09.

### **Appendix Detail**

Appendix 1	Resumes of Key Personnel
Appendix 2	Code of Ethics
Appendix 3	Organization Chart
Appendix 4	List of Major Equipment
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Appendix 6	Sample Collection and Holding Times
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Appendix 8	Acceptance Limits for QC Determinations
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Appendix 10	<b>Corrective Action Report Form</b>
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#### APPENDIX 1 RESUMES OF KEY PERSONNEL

Name	Position .
Alfredo Pierri	President/CEO – Laboratory Director
David Cerna	QA Officer
Joe Chau	Technical Director Inorganics
Alan Ching	Technical Director Organics
Hai-Van Nguyen	Technical Director Microbiology - Senior Project manager

#### **ALFREDO E. PIERRI**

<u>Title</u>

President, Laboratory Director

#### **Education**

M.S. (equiv.) - University of Buenos Aires, Argentina, 1978. Organic Chemistry

 University of California, Los Angeles Certificate in Hazardous Materials Control and Management, 1991 - 1993

#### **Affiliations**

American Chemical Society, member American Water Works Association, member Water Environment Federation, member American Council of Independent Laboratories (ACIL), member The NELAC Institute, member

#### **Professional Experience**

Jan/1987 to Present	Weck Laboratories, Inc., City of Industry, CA Full Service Environmental Testing laboratory
Sep/1984 to Dec/1986	SCS Engineers, Long Beach, CA Environmental Testing laboratory owned by Large Environmental Engineering Firm
Jul/1979 to Aug/1984	Argentina Atomic Energy Commission, Buenos Aires, Argentina Government Agency – Research and Development

Mr. Pierri has extensive experience in analytical chemistry. Most of his work in this field has been in the application and development of instrumental methods of analysis for organic analytes using GC, GC/MS, HPLC, IR and UV-Visible spectrometry. He has also worked in Spectrometric techniques for metals analysis such as Atomic Absorption with flame and graphite furnace and Inductively Coupled Plasma with Optical Emission and Mass Spectrometry.

Since 1984 he has been working exclusively in the environmental field obtaining in 1993 the certification as Registered Environmental Assessor (REA-04975) from the California Environmental Protection Agency.

As Laboratory Director, Mr. Pierri is responsible for all laboratory operations including the supervision of the overall performance of the laboratory, revision of analytical reports and Quality Assurance Program, provision of technical assistance and direction to laboratory personnel and consulting with clients about technical and regulatory issues.

Mr. Pierri is well acquainted in all aspects of environmental regulations at Federal and State level, providing consulting services and guidance to clients in regulatory compliance and chemical treatment issues as well as understanding and interpreting analytical data.

Other relevant experience and projects in which Mr. Pierri has participated are as follows:

- For over 22 years provided Project Management for large environmental monitoring projects for wastewater treatment plants, desalination plants, groundwater studies, potable water compliance monitoring and unregulated contaminants studies managed by the EPA such as ICR, UCMR 1 and UCMR 2. These projects required dealing with significant technical issues, regulatory compliance and innovative analytical methods.
- Characterization of wastes to be classified as hazardous as per State of California and Federal Regulations.
- Developing of analytical methods for emerging contaminants in water using GC/MS, LC/MS and other analytical techniques and writing the operating procedures.
- Identification and selection of new laboratory equipment for the laboratory
- Determination of contamination in soil and groundwater due to leaking underground storage tanks.
- Design and implementation of a Quality Assurance Program based on NELAC requirements for the laboratory, writing of the QA manual and training of laboratory personnel.
- Developing and implementation of an Ethics Training Program for the Laboratory, writing the documentation and training course for laboratory employees.
- Interpretation of analytical data and compliance with regulations for drinking water for different potable water purveyors in Southern California.
- Compliance for wastewater discharges with local regulatory agencies and NPDES permits.
- Consulting services to industrial clients on pre-treatment of effluents in order to minimize organic matter and solids and reduce costs in taxes imposed by POTWs.
- Identification of unknown materials by chemical and physical methods.
- Implementation of a LIMS and use of personal computers for data acquisition, handling, and reporting.
- Teaching of Analytical Organic Chemistry at University Level for MS program.

#### **Participation in Seminars and Conferences**

Over the years, Mr. Pierri has participated in innumerable conferences and technical meeting involving environmental testing, environmental policy and remediation.

He has been speaker in several conferences and technical meetings related to environmental monitoring in general and emergent contaminants in particular.

#### **DAVID CERNA**

#### Title

QA Manager

#### Education

B.S. - California Polytechnic University, Pomona, 1997 Chemistry

#### **Professional Experience**

May/1997 to Present	Weck Laboratories, Inc., City of Industry, CA
	Full Service Environmental Testing laboratory

Mr. Cerna has hands on experience for the analysis of environmental samples by different techniques, including TOC, TOX, Ion chromatography, Liquid Chromatography, GC/MS and sample extraction and preparation for organic analysis by Liquid-Liquid, Solid Phase, sonication and other techniques. As Group Leader for the IC/HPLC section he was instrumental in developing analytical methods, selecting and setting up new analytical instrumentation and providing training to lab personnel. Mr. Cerna has also been a data reviewer for analytical batches in the organic department including QA/QC

Mr. Cerna has also been a data reviewer for analytical batches in the organic department including QA/QC and data accuracy.

As QA Manager, Mr. Cerna is responsible for monitoring and upgrading the QA program for the laboratory, performing internal audits and interacting with State and client auditors. Other responsibilities include providing training to analysts for QA/QC issues and verifying that SOPs are in compliance with current laboratory practices.

Other relevant experience and projects in which Mr. Cerna has participated are as follows:

- Review data packages generated by IC or HPLC for different methods.
- Write SOPs for laboratory procedures.
- Development of analytical methods for trace level contaminants in water by LC/MS/MS and IC
- HPLC and IC troubleshooting and maintenance
- Analysis of water, wastewater, soil and hazardous waste samples by GC/MS for volatile organics
- Analysis of environmental samples by HPLC using different detectors and post-column derivatization systems.

#### **Participation in Seminars and Conferences**

Mr. Cerna has participated in many technical seminars for IC, HPLC and LC/MS. He has also attended training classes and conferences relevant to his current position as QA Manager.

#### JOE CHAU

#### Title

Technical Director Inorganic

#### Education

B.S	California Polytechnic University, Pomona, CA, 1988
	Electrical Engineering

- B.S California Polytechnic University, Pomona, CA. 1993 Chemistry, Industrial Option
  - University of California, Irvine Certificate in Hazardous Materials Control and Management, 1991

#### **Professional Experience**

Sep/1989 to Present	Weck Laboratories, Inc., City of Industry, CA Full Service Environmental Testing laborator		
Sep/1988 to Sep/1989	Lights of America, Walnut, CA Electrical Engineering		

Mr. Chau has extensive experience in environmental analysis, especially for inorganic and physical parameters.

He has been working as analytical chemist for inorganic and wet chemistry determinations, metal analyses by Flame and Graphite furnace AA, ICP, ICP-MS and Cold vapor AA and AF.

Mr. Chau has been instrumental in developing analytical methods for trace metal analyses in a variety of matrices, including brines and sea water. He has also developed for the laboratory especially methods for physical parameters, metal speciation and non-routine determinations.

As lab supervisor, Mr. Chau has provided guidance, technical advice and training to bench chemists and other lab personnel and has managed lab operations to improve logistics such as sample receiving and project management

Mr. Chau is an expert in spectroscopic analysis and provides advice to clients about technical and QA/QC issues.

Other relevant experience and projects in which Mr. Chau has participated are as follows:

- Coordination of monitoring projects that requires large number of analysis on short turnaround time for metals.
- Supervision of lab personnel for the Inorganic Section

- Development of analytical procedures for the determination of environmental samples by ICP-MS in particularly difficult matrices
- Develop of methods by atomic fluorescence and amalgamation for ultra trace level analysis of mercury.
- Design of a clean room and develop protocols for its operation for analysis of trace metals in ambient waters and ultra trace levels of mercury
- Maintenance and troubleshooting of spectroscopy instrumentation.
- Design and improvement of sample digestion procedures for metal analysis to reduce contamination and improve recoveries.
- Development of analytical methods for speciation analysis of metals, including the use of hyphenated analytical techniques.

#### **Participation in Seminars and Conferences**

During his time at Weck Laboratories, Mr. Chau has participated in many technical and user meetings provided by spectroscopy equipment manufacturers, such as Perkin Elmer, Thermo and Agilent. He routinely participates in technical conferences about environmental analysis, where technical issues, new techniques and regulatory subjects are discussed; they include NEMC, NELAC and Pittcon, among others.

#### ALAN CHING

#### Title

Technical Director Organic

#### **Education**

B.S	Chu Hai College, Hong Kong, 1985
	Chemistry
	Shangai University of Technology, China
	Analytical Chemistry Courses 1978 - 1981

M.S. - California Polytechnic University, Pomona Analytical Chemistry, 1997

#### **Professional Experience**

Oct/1990 to Present	Weck Laboratories, Inc., City of Industry, CA Full Service Environmental Testing laboratory
Jan/1985 to Jun/1989	Dinippon Ink and Chemical, Sheng Zheng, China Chemical Manufacturing Company

Mr. Ching' primary experience is in the organic analysis field although he has performed as bench chemist inorganic and metal analyses as well. At Weck Labs, he has hands on experience in GC, GC/MS, HPLC and organic extractions.

Mr. Ching has developed many analytical procedures for volatile organic compounds, pesticides, herbicide and semivolatile organic analysis.

As lab supervisor, Mr. Ching has provided training and technical advice to bench chemists in the organic section.

Mr. Ching has also served as QA Manager being instrumental in developing the QA/QC program, obtaining accreditation under NELAC for the laboratory, writing the QA Manual and monitoring its implementation. Mr. Ching also provides technical support to clients in the areas of Quality Assurance, analytical chemistry and regulatory compliance.

Other relevant experience and projects in which Mr. Ching has participated are as follows:

- Project Management for ICR, UCMR 1 and UCMR 2 analysis, including method development, interaction with Utilities and reporting to the EPA.
- Analysis of environmental samples for metals, and other elements by atomic absorption and ICP spectrometry using flame, hydride generation, cold vapor and graphite furnace.
- Hazardous waste characterization by different analytical techniques.

- Maintenance and troubleshooting of GC, GC/MS and HPLC instrumentation.
- Separation and detection of four different arsenic compounds using ion exchange chromatography and UV detection. (Master's degree project).
- Development of new methods for UCMR testing and other emergent contaminants
- Developing a comprehensive QA/QC program for the Laboratory in compliance with NELAC and ISO 17025.

#### **Participation in Seminars and Conferences**

Mr. Ching regularly attends many technical meeting regarding technical and regulatory issues. He has participated in NELAC conferences and other meeting related to Quality Assurance and regulatory compliance issues.

#### HAI-VAN NGUYEN

#### <u>Title</u>

Senior Project Manager - Technical Director Microbiology

#### **Education**

B.S. - California Polytechnic University, Pomona, CA, 2000 Biology, Minor in Chemistry

> University of California, Irvine, CA, 2008 Environmental management Certificate Program

#### **Professional Experience**

Apr/2000 to Present	Weck Laboratories, Inc., City of Industry, CA
	Full Service Environmental Testing laboratory

Ms. Nguyen has extensive experience in the environmental laboratory. She has been a bench chemist for inorganic, bacteriological testing, HPLC, GC and GC/MS, which has given her a well rounded view of the operation of the environmental laboratory in all its aspects. Other important tasks completed include assisting the QA Manager in preparing SOPs and updating the program.

As Technical Director for Microbiology she oversees the department and provides training to analysts. Ms. Nguyen is also very well versed in compliance regulations for potable water and wastewater programs, as well as interpretation of analytical data.

In her position as Senior Project Manager, she has managed many large environmental projects for potable water, wastewater and groundwater investigations, proving consulting to clients and interacting with regulatory agencies.

Other relevant experience and projects in which Ms. Nguyen has participated are as follows:

- Managing testing projects for large clients.
- Assisting the QA Manger in supervising and designing QA/QC operations.
- Writing and upgrading of SOPs.
- Evaluation and reviewing analytical data for inorganic analysis, HPLC, GC, GC/MS and wet chemistry methods.
- Reviewing analytical data for microbiological determinations and providing technical support to analysts.

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#### **Participation in Seminars and Conferences**

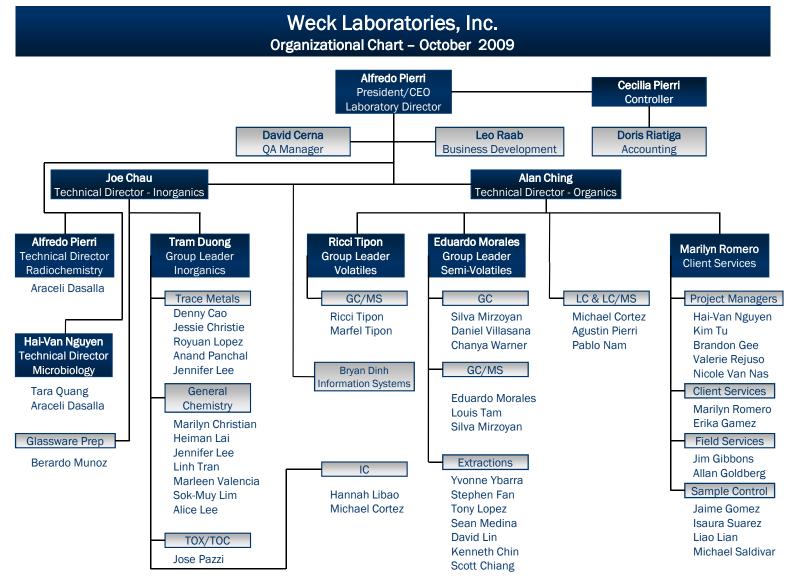
Ms. Nguyen regularly participates in technical seminars and meeting regarding regulatory compliance issues.

# **CODE OF ETHICS**

Weck Laboratories, Inc. is committed to ensuring the integrity of our data and meeting the quality needs of our clients. We pledge to manage our business according to the following principals:

- To produce results that are technically sound and legally defensible;
- To assert competency only for work for which adequate equipment and personnel are available;
- To present services in a confidential, honest, and forthright manner;
- To have a clear understanding with the client as to the extent and kind of services to be rendered;
- To provide employees with guidelines and an understanding of the ethical and quality standards required in this industry;
- To operate facilities in a manner that protects the environment and the health and safety of employees and the public;
- To obey all pertinent federal, state, and local laws and regulations;
- To continually improve product and service quality;
- To treat employees equitably, acknowledge their scientific contributions, and provide them with opportunities for professional growth and development;
- To recognize and respond to community concerns; and
- To deal openly, honestly, and fairly in all business and financial matters with employees, clients and the public.

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#### List of Major Equipment as of July 2009

Туре	Section	Number	Instrument Description	<b>Tests Performed</b>
LC/MS/MS	LC/MS	1	ABI 4000 Q trap Triple quad with +ESI, -ESI, APCI,MS/MS and linear Ion Trap capabilities	PPCPs, Endocrine disruptors, Emergent chemicals
LC/MS/MS	LC/MS	1	LC/MS/MS Varian 1200L Triple quad with positive and negative ESI, APCI and MS/MS capabilities	EPA 535, EPA 331, EPA 332, Emergent Chemicals
GC/MS	Semivolatile Organics	1	GC/MS/MS system, Varian 4000 with EI, CI and MS/MS capabilities	EPA 521, Nitrosamines
GC/MS	Semivolatile Organics	1	GC/MS/MS system, Varian 4000 with EI, CI and MS/MS capabilities and Combi-Pal robotic autosampler	Special tests, low level pesticides; EDCs, EPA 521 backup
GC/MS	Semivolatile Organics	1	GC/MS system, Agilent 7890/5975 Turbo with EI and PTV injection capabilities	EPA 525.2, 548.1, 527, 529
GC/MS	Semivolatile Organics	1	GC/MS system, Agilent 6890/5973N Turbo with EI and PCI capabilities	EPA 625, 8270 and 1,4-Dioxane
GC/MS	Semivolatile Organics	1	GC/MS system, ThermoFinnigan DSQ II with EI, PCI,NCI and PTV capabilities	EPA 527, PCB congeners, low level pesticides, Pyretroids
GC/MS	Volatile Organics	1	GC/MS system, Agilent 6890/5973 with Tekmar Solatek autosampler and Tekmar 3100 Purge & Trap	EPA 524.2, Low level 123TCP
GC/MS	Volatile Organics	1	GC/MS system, Agilent 6890/5973 with Archon autosampler and Tekmar 3000 Purge and Trap	EPA 524.2
GC/MS	Volatile Organics	1	GC/MS system, Agilent 6890/5973 with Archon autosampler and Tekmar 3100 Purge and Trap	EPA 8260 and 624
GC/MS	Volatile Organics	1	GC/MS system, Hewlett-Packard 5890 series II/5972 MSD with Aquatek 70 autosampler and Tekmar 3000 Purge and Trap	EPA 524.2
GC/MS	Volatile Organics	1	GC/MS system, Hewlett-Packard 5890 series II/5972 MSD with Archon autosampler and O-I Eclipse Purge and Trap	EPA 8260 and 624
GC	Semivolatile Organics	2	Gas chromatograph Agilent model 6890 with autosampler and dual ECD detectors	EPA 551.1, EPA 508, 515.3

Туре	Section	Number	Instrument Description	<b>Tests Performed</b>
GC	Semivolatile Organics	1	Gas chromatographs Agilent 6890 with autosampler FID and ECD	EPA 8015 TPH, Alcohols
GC	Semivolatile Organics	1	Gas chromatographs Varian 3800 with autosampler and dual ECDs and TSD detectors	EPA 504.1, EPA 552.2
GC	Semivolatile Organics	1	Gas chromatograph Hewlett Packard model 5890A with autosampler and ECD and NPD detector.	EPA 507, Backup instrument for EPA 508, 504 or 515.3
GC	Semivolatile Organics	1	Gas chromatograph Hewlett Packard model 5890A with autosampler and FID and TCD detectors.	Backup instrument for EPA 8015 TPH and alcohols
GC	Volatile Organics	1	Gas Chromatograph, Hewlett-Packard 5890A with FID/PID in series with Tekmar 2016 autosampler and Tekmar 2000 Purge and Trap	EPA 8021 BTEX
HPLC	IC/HPLC	1	Liquid Chromatograph system Dionex DX500 with gradient pump, post-column reaction systems, and fluorescence and UV-VIS detectors.	EPA 531.1 and 547
HPLC	IC/HPLC	1	Liquid Chromatograph system Dionex DX500 with gradient pump and UV-VIS detector	EPA 549.2, 8315 and 8330
HPLC	IC/HPLC	1	Liquid Chromatograph system Shimadzu with dual pumps, UV-VIS detector and autosampler Model SIL 10AD-vp	Backup for EPA 549.2, 8315 and 8330
IC	IC/HPLC	1	Ion chromatograph DIONEX DX-120 with isocratic pump and conductivity detector	EPA 300.0
IC	IC/HPLC	1	Ion Chromatograph Dionex with gradient pump, post-column derivatization and UV-Vis detector dedicated for hexavalent chromium.	EPA 218.6, EPA 7199
IC	IC/HPLC	1	Ion Chromatograph Dionex ICS-2000 with eluent generator and conductivity detector dedicated to perchlorate analysis	EPA 314.0
IC	IC/HPLC	1	Ion Chromatograph Dionex DX-500 with gradient pump and conductivity detector	EPA 314.0
IC	IC/HPLC	1	Ion Chromatograph system Dionex DX- 600 with gradient pump, post column derivatization, conductivity and Photodiode array detectors.	EPA 300.1 and 326 low levels Bromide, chlorite, chlorate and bromate

Туре	Section	Number	Instrument Description	Tests Performed
ICP-MS	Metals	1	ICP-MS Spectrometer Agilent 7500ce	EPA 200.8, EPA 6020, EPA 1638, EPA 1640
ICP-MS	Metals	1	ICP-MS Spectrometer Perkin Elmer model ELAN DRC-II with Apex Duo Fast autosampler option with Preconcentration column On-line. Also option with hydride generation On-line.	EPA 200.8, EPA 1638, EPA 1640, Modified 200.8 for sea water and brines; hydride analysis
ICP	Metals	1	ICP Spectrometer Perkin Elmer model Optima DV-5300 with FAST autosampler	EPA 200.7, EPA 6010
CVAA	Metals	1	Mercury analyzer CETAC model M- 6000 with autosampler	EPA 245.1; EPA 7470; EPA 7471
CVAF	Metals	1	Low Level Mercury Analyzer Leeman Labs model Hydra AF Gold +	EPA 1631; EPA 245.7 and methyl mercury
HPLC	Metals	1	Dionex HPLC system DCX500	Connected to ICP- MS for Metal Speciation
Automated SPE	Sample Prep	1	Solid phase extraction system Horizon Technologies 4790 consisting in 8 automated extractors	Various EPA 500's series methods and UCMR
Automated SPE	Sample Prep	3	Caliper Autotrace automated cartridge solid phase extractor with 6 positions	PPCP/EDC; Various EPA 500's series and UCMR
Continuous L-L	Sample Prep	3	Continuous accelerated liquid-liquid extractor/concentrator Corning from Organomation of 8 position each.	Various
Concentrator	Sample Prep	1	Automated solvent blow-down apparatus Horizon model Dry-Vap with 6 positions	Various
Concentrator	Sample Prep	1	Turbo Vap solvent blow-down apparatus with 50 positions	Various
Automated ASE	Sample Prep	1	Accelerated Solvent Extraction system Dionex model ASE 200 for soils/sediments	EPA 8000's series in soil/sediment
Automated SPE	Sample Prep	1	Automated solid phase extractor for Oil and Grease with 3 positions Horizon Technologies Model 3000 XL	EPA 1664
L-L	Sample Prep	1	Separatory funnel shaker 4-positions from Glas-Col	Various
Digester	Sample Prep	2	Block digesters for trace metal sample preparation	EPA 200.7; 200.8; 245.1; 6010; 6020; 7470 and 7471
Digester	Sample Prep	2	Block digesters for TKN and total phosphorus sample preparation	Various
Shaker/Extractor	Sample Prep	2	TCLP rotary extractors for leaching procedures with glassware	Various

Туре	Section	Number	Instrument Description	<b>Tests Performed</b>
Shaker/Extractor	Sample Prep	2	Zero Headspace apparatus for TCLP extractions for Volatiles	EPA 8260-TCLP
Titrator/ISE/pH/EC	General Chemistry	1	Automated Titration-ISE instrument Man-Tech Associates, model PC Titrate with autosampler	SM2320B; SM2310B, pH, SM5210
Autoanalyzer	General Chemistry	1	Lachat model 8500 + FIAS auto analyzer with four simultaneous channels for NO3-N, NO2-N, TKN, TP, OP, Cyanide and NH3	EPA 353.2, 351.2; 365.1; 335.2 and 350.1
Autoanalyzer	General Chemistry	1	Seal Analytical model AQ2+ discrete spectrophotometric wet chemistry analysis (NO3, NO2, TKN, TP, OP, Phenols, Cyanide and NH3	EPA 353.2; 351.2; 365.1; 335.2; 350.1 and 420.4
Proportional Counter	Radiochemistry	2	Gas flow Alpha + Beta Counter Protean model MPC 9604 for radiological analyses.	EPA 900.0, SM7110C EPA 903.0, EPA 904
Liquid Scintillation	Radiochemistry	1	Beckman Liquid Scintillation apparatus model LS6500	Radon, Tritium, EPA 903.1
TOC	General Chemistry	1	Total organic carbon (TOC) Tekmar- Dorhman Phoenix 8000 with autosampler.	SM5310C
ТОХ	General Chemistry	1	Total organic halides (TOX) Mitsubishi TX-10.	SM5320B, EPA 9020
UV-VIS	General Chemistry	1	UV-Visible Spectrophotometer Milton Roy Genesis 5.	Various
UV-VIS	/-VIS General Chemistry		UV-Visible Spectrophotometer Hach model DR4000U	Various
ISE/pH	-		Ion Selective electrode system Accumet 150 for pH, conductivity and ISE measurements	EPA 150.1, SM2510B,
Trucks	Field	3	Pickup trucks for field sampling Toyota Tacoma, models 2009, 2006 and 1998	Field work
Samplers	Field	9	Composite water sampling equipment ISCO, different models.	Wastewater sampling
Software	IT	1	Laboratory Information Management System (LIMS) "Element" from Promium running on SQL database.	Supports all methods
Software	IT	1	Element Web program to allow clients to review projects on real time through the Laboratories' web page.	Supports all methods
Software	IT	1	Element Data tool program to transfer analytical data directly from instruments into the LIMS.	Supports all methods
Software	IT	1	Agilent Chem Station software latest revision for control and data processing of Agilent GC and GC/MS instruments.	Supports organic methods
Software	IT	1	Varian Star Chromatography software for control and data processing of Varian GC and GC/MS instruments.	Supports organic methods

Туре	Section	Number	Instrument Description	Tests Performed
Software	IT	1	Dionex Peak Net Software for control and data processing of Dionex HPLC and IC instruments	Supports inorganic methods
Software	IT	1	Tal Technologies Wedge software for data acquisition of all RS232 devices (balances, pH meter, turbidimeter etc.) and other vendor specific software for data acquisition and processing of all other instruments.	Various

14859 East Clark Avenue : Industry : CA	Weck L	Weck Laboratories, Inc.		CHAIN OF CUSTODY RECORD	RD
161 020-330-2139 + Fax 020-330-2034	Industry : CA 91745 626-336-2634 • www.wecklabs.com	Jonatory Strvicca - Since 1964		Page 1 O	of 1
CLIENT NAME:	PROJECT:		ANALYSES REQUESTED	SPECIA	L HANDLING Same Day Rush 150%
ADRESS:	PHONE: FAX: EMAIL:			24 Hour Rush 100%           1         48-72 Hour Rush 30%           1         4 - 5 Day Rush 30%           1         Rush Extractions 50%	24 Hour Rush 100% 48-72 Hour Rush 75% 4 - 5 Day Rush 30% Rush Extractions 50%
PROJECT MANAGER	SAMPLER			10 - 15 Business Days     OA/OC Data Package     OA/OC Data Package	10 - 15 Business Days QA/QC Data Package
ID#         DATE         TIME         S           (For lab Use Only)         SAMPLED         SAMPLED         T	SMPL SAMPLE IDENTIFICATION/SITE LOCATION	N/SITE LOCATION # 0F CONT.		Consiges win appy for w Method of Shipment: COMMENTS	weekening
	DATE / TIME	RECEIVED BY	-	SAMPLE CONDITION: A Adual Temperature: N	SAMPLE TYPE CODE AQ=Aqueous NA= Non Aqueous
RELINQUISHED BY	DATE / TIME	RECEIVED BY		Y / N Y / N Y / N Y / N Y / N	SL = Sludge DW = Dmking Water WW = Waste Water RW = Rain Water GW = Crouod Water
RELINQUISHED BY	DATE / TIME	RECEIVED BY		Z / X	SO = Solid Waste SW = Solid Waste OL = Oil OT = Other Matrix

### Chain of Custody Form

APPENDIX 6 Sample Collection and Holding Times

					Preservative				
Test Name	Matrix	Bottle Type	Bottle size	Unchlorinated Water (Raw)	Chlorinated Water (Treated)	Soil/Solid	Holding Time until start of analysis	Analytical Technique	Analytical Method
1,2,3-TCP	Water	Glass	2 x 40 ml	None	Ascorbic		14 days	GC/MS Isot. Dil.	EPA 524.2SIM
1,4-Dioxane	Water	Amber Glass	2 x 1 L (*)	None	None		14 days	GC/MS Isot. Dil.	EPA 8270M
Alcohols	Water	Glass	1 x 40 ml	None	None		14 days	Dir. Inj./FID	EPA 8015B
Aldehydes	Water	Glass	2 x 40 ml	CuSO4	NH4Cl/CuSO4		7 Days	GC/ECD	EPA 556
Aldehydes	Water	Glass	1 L (*)	None	Thiosulfate		3 days	HPLC-UV	EPA 8315
Aldehydes(1)	Soil/Solid	Glass	4 oz			None	3 days	HPLC-UV	EPA 8315
Alkalinity, Total	Water	Poly	250 ml		None		14 Days	Titration	SM2320B
Anions by IC (F-,Cl- ,SO4=)	Water	Poly	250 ml	None	None		28 days	IC	EPA 300.0
Anions by IC (NO2- ,NO3-,PO4≡)	Water	Poly	250 ml	None	None		48 hours	IC	EPA 300.0
Arsenic speciation	Water	Poly	250 ml	EDTA/acetic acid	EDTA/acetic acid		14 Days	Resin-ICP/MS	EPA 200.8
Asbestos-Sub	Water	Poly	1 L	None	None		48 Hours	TEM	EPA 100.1/.2- Sub
Bacteria-Coliform - solid/sludge/soil	Soil/solid	Glass-Sterile	4 oz			None	N/A	MTF	SM 9221B
Bacteria-Coliform - Wastewater	Water	Poly-Sterile	125 ml	Thiosulfate	Thiosulfate		6 hours	MTF	SM 9221B
Bacteria-Coliform - Drinking Water	Water	Poly-Sterile	125 ml	Thiosulfate	Thiosulfate		24 Hours	Colilert P/A or enumeration	SM 9223B
Bacteria- Enterococcus - Wastewater	Water	Poly-Sterile	125 ml	Thiosulfate	Thiosulfate		24 Hours	Enumeration Quantitray	Enterolert
Bacteria- Heterotrophic Plate Count	Water	Poly-Sterile	125 ml	Thiosulfate	Thiosulfate		24 Hours	Pour Plate Method	SM 9215B
BOD	Water	Poly	1 L	None	None		48 Hours	DO Probe	SM 5210B
BOD, Carbonaceous	Water	Poly	1 L	None	None		48 Hours	DO Probe	SM 5210
Bromate	Water	Poly	250 ml	EDA	EDA		28 Days	IC	EPA 300.1
Bromate- Low Level	Water	Poly	250 ml	EDA	EDA		28 Days	IC	EPA 326
Bromide	Water	Poly	250 ml	None	None		28 Days	IC	EPA 300.0
Bromide-Low Level	Water	Poly	250 ml	None	None		28 Days	IC	EPA 300.1
Carbamates	Water	Glass	1 x 40 ml	MCAA	MCAA/thiosulf.		28 Days	HPLC	EPA 531.1
COD	Water	Poly	250 ml	H2SO4	H2SO4		28 Days	Colorimetric	EPA 410.4
Chloral Hydrate	Water	Glass	2 x 60 ml	Sulfite/buffer	Sulfite/buffer		14 days	GC/ECD	EPA 551.1
Chlorate	Water	Poly	250 ml	EDA	EDA		28 Days	IC	EPA 300.1

## Weck laboratories, Inc. - Sampling Guidelines

Chloride	Water	Poly	250 ml	None	None	28 Days	IC	EPA 300.0
Chlorine Dioxide	Water	Glass	250 ml	None	None	24 Hours	Colorimetric	SM 4500CLO2D
Chlorine Residual	Water	Glass	250 ml	None	None	24 Hours	Colorimetric	SM 4500CL-G
Chlorite	Water	Amber Glass	125 ml	EDA	EDA	14 Days	IC	EPA 300.1
Chlorophyll-a	Water	Amber Poly	2 x 1L	None		48 Hours	Spectrophotometric	SM 10200H
Chromium, Hexavalent	Water	Poly	250 ml	None	None	24 Hours	Spectrophotometric	SM3500CR- D/7196
Chromium, Hexavalent	Soil/solid	Glass	4 oz	None	None	30 days	Spectrophotometric	EPA 3060/7196
Chromium, Hexavalent (low level)	Water	Poly	250 ml	None	None	24 Hours	IC	EPA 218.6
Chromium, Hexavalent (low level)	Soil/solid	Glass	4 oz	None	None	30 days	IC	EPA 3060/7199
Color	Water	Glass	500 ml	None	None	48 Hours	Visual	SM2120B
Conductivity (Specific Conductance)	Water	Poly	250 ml	None	None	28 Days	Electrometric	SM2510B
Cyanide	Water	Poly	500 ml	NaOH	NaOH/ascorbic	14 Days	FIA-Colorimetric	EPA 335.2/335.4
Dioxin-Sub	Water	Glass	2 x 1 L	None	None	1 year	GC/ MS	EPA 1613/8290
Diquat/Paraquat	Water	Amber poly	1L	None	Thiosulfate	7 Days	HPLC	EPA 549.2
Disinfection by- products	Water	Glass	2 x 60 ml	Sulfite/buffer	Sulfite/buffer	14 days	GC/ECD	EPA 551.1
Diuron	Water	Amber Glass	1 L (*)	None	None	7 days	HPLC/UV	EPA 632
Diuron-UCMR	Water	Amber Glass	1 L (*)	CuSO4/Trizma	CuSO4/Trizma	14 days	HPLC/UV	EPA 532
EDB and DBCP	Water	Glass	2 x 40ml	None	Thiosulfate	14 Days	GC/ECD	EPA 504.1
Endothall	Water	Amber Glass	250 ml	None	None	7 days	GCMS	EPA 548.1
Ethanol	Water	Glass	1 x 40 ml	None	None	14 Days	Dir. Inj./FID	EPA 8015B
Explosives	Water	Amber Glass	1 L (*)	None	Thiosulfate	7 days	HPLC/UV	EPA 8330
Fluoride	Water	Poly	250 ml	None	None	28 Days	IC	EPA 300.0
General Minerals (excluding metals)	Water	Poly	1 L	None	None	Various	Wet Chem methods	various
General Minerals (metals only)	Water	Poly	250 ml	HNO3	HNO3	6 Months	ICP-AES	EPA 200.7
General Physical (Color, Odor, Turbidity	Water	Glass	500 ml	None	None	24 Hours	Wet Chem methods	various
Glyphosate	Water	Glass	1 x 40 ml	None	Thiosulfate	14 Days	HPLC	EPA 547
HAAs	Water	Amber Glass	250 ml (*)	NH4CI	NH4CI	14 days	GC/ECD	EPA 552.2

HAAs-Formation Potential	Water	Amber Glass	1L	None	None	14 days	GC/ECD	SM 5710B/EPA 552.2
Herbicides-DW	Water	Amber Glass	250 ml (*)	None	Thiosulfate	14 days	GC/ECD	EPA 515.3
Herbicides-GW	Water	Amber Glass	2 x 1 L (*)	None	Thiosulfate	7 Days	GC/ECD	EPA 8151
Mercury	Water	Glass jar	250 ml	HNO3	HNO3	28 Days	Cold Vapor AAS	EPA 245.1/7470
Methanol	Water	Glass	1 x 40 ml	None	None	14 Days	Dir. Inj./FID	EPA 8015B
Mercury in soil/solid/sludge	Soil/Solid	Glass jar	4 oz.	None	None	28 Days	Cold Vapor AAS	SW 7471
Metals (2)	Water	Poly	250 ml	HNO3	HNO3	6 Months	ICP/MS or ICP- AES	EPA 200.8/200.7
NDMA	Water	Amber Glass	2 x 1 L (*)	None	Ascorbic	7 days	GC/MS/CI SIM	EPA1625M
Nitrate	Water	Poly	250 ml	None	None	48 Hours	IC or FIA	EPA 300.0/353.2
Nitrite	Water	Poly	250 ml	None	None	48 Hours	IC or FIA	EPA 300.0/353.2
Nitrite+Nitrate as N	Water	Poly	250 ml	H2SO4	H2SO4	28 Days	FIA-Colorimetric	EPA353.2
Nitrogen, Total Kjeldahl (TKN)	Water	Poly	250 ml	H2SO4	H2SO4	28 Days	FIA-Colorimetric	EPA 351.2
Nitrogen-Ammonia	Water	Poly	250 ml	H2SO4	H2SO4	28 Days	FIA-Colorimetric	EPA 350.1
Nitrogen-Ammonia in ww with distillation	Water	Poly	250 ml	H2SO4	H2SO4	28 Days	FIA-Colorimetric	EPA 350.1
Nitrosamines	Water	Amber Glass	2 x 1 L (*)	None	Ascorbic	14 days	GC/MS/CI SIM	EPA 521
Odor	Water	Glass	500 ml	None	None	24 Hours	Odor	SM 2150B
Oil and Grease	Water	Glass	1 L	HCL	HCL	28 Days	Gravimetric	EPA1664
Organotins (tributyltin)	Water	Glass	1 L (*)	None	None	7 Days	GC/MS	GC/MS
Oxygen, Dissolved	Water	Glass	BOD bottle	None	None	24 Hours	O2 Probe	SM 4500-OG
PBDEs	Water	Amber Glass	2 x 1 L (*)	None	None	14 days	GC/MS SIM	EPA 1614M
Perchlorate	Water	Poly	250 ml	None	None	28 Days	IC	EPA 314
Perchlorate - Low Level by LC/MS/MS	Water	Poly Sterile	125 ml	Sterile field filtration	Sterile field filtration	28 Days	LC/MS/MS	EPA 331/332
Perchlorate in soils	Soil	Glass jar	4 oz	None	None	28 Days	IC	EPA 314M
Pesticides- Organophosphorus	Water	Amber Glass	2 x 1 L (*)	None	Thiosulfate	7 Days	GC/NPD	EPA8141
Pesticides, Chlorinated (DW)	Water	Amber Glass	2 x 1 L (*)	None	Thiosulfate	7 days	GC/ECD	EPA 508
Pesticides, Chlorinated WW/GW	Water	Amber Glass	2 x 1 L (*)	None	Thiosulfate	7 Days	GC/ECD	EPA 608/8081
PCBs - GW	Water	Amber Glass	2 x 1 L (*)	None	Thiosulfate	7 Days	GC/ECD	EPA 8082
Pesticides, N/P -DW	Water	Amber Glass	2 x 1 L (*)	None	Thiosulfate	14 days	GC/ NPD	EPA 507/8141
рН	Water	Poly	250 ml	None	None	3 Days	Electrometric	SM4500H

Phenolics	Water	Amber Glass	500 ml	H2SO4	H2SO4	28 Days	Spectrophotometric	EPA 420.1
Phosphate, Ortho	Water	Poly	250 ml	None	None	48 hours	FIA-Colorimetric	EPA 365.1
Phosphate, Total	Water	Poly	250 ml	H2SO4	H2SO4	28 Days	FIA-Colorimetric	EPA 365.1
Polynuclear Aromatics (PNAs) Low level	Water	Amber Glass	2 x 1L	None	Thiosulfate	7 Days	HPLC or GC/MS	EPA 610/8310 or EPA 8270SIM
Radiological-Gross Alpha	Water	Poly	1 L	HNO3	HNO3	6 Months	GPC	EPA 900.0
Radiological-Gross Alpha high TDS	Water	Poly	1 L	HNO3	HNO3	6 Months	Coprecipitation- GPC	SM7110C
Radiological-Gross Beta	Water	Poly	1 L	HNO3	HNO3	6 Months	GPC	EPA 900.0
Radiological-Radium 226-Sub	Water	Poly	2 x 1 L	HNO3	HNO3	6 Months		EPA 903.1 Sub
Radiological-Radium 228-Sub	Water	A-Poly	1 L	HNO3	HNO3	6 Months		RA-05 Sub
Radiological-Radon 222-Sub	Water	Glass	2 x 60 ml	None	None	4 Days	LSC	EPA 913.0
Radiological- Strontium 90-Sub	Water	Poly	1 L	HNO3	HNO3	6 Months		EPA 905.0 sub
Radiological-Tritium- Sub	Water	Amber Glass	125 ml	None	None	6 Months	LSC	EPA 906.0 sub
Radiological- Uranium-Sub	Water	Poly	250 ml	HNO3	HNO3	6 Months	ICP-MS	EPA 200.8
Semivolatile Organics (BNA) - GW or WW	Water	Amber Glass	2 x 1L	None	Thiosulfate	7 Days	GC/MS	EPA 625/8270C
Silica by ICP	Water	Poly	250 ml	None	None	28 Days	ICP	EPA 200.7
SOCs - Drinking Water	Water	Amber Glass	2 x 1 L	HCL	Sulfite/HCL	14 days	GC/MS	EPA 525.2
SOCs - Special Analytes	Water	Amber Glass	2 x 1 L	HCL	Asc., EDTA, Diazol. Urea, Buffer	14 days	GCMS	EPA 526
SOCs - Phenolics	Water	Amber Glass	2 x 1 L	HCL	Sulfite/HCL	14 days	GCMS	EPA 528
Solids, Settleable	Water	Poly	1 L	None	None	48 Hours	Gravimetric	EPA 160.5
Solids, TDS	Water	Poly	500 ml	None	None	7 Days	Gravimetric	SM2540C
Solids, Total	Water	Poly	500 ml	None	None	7 Days	Gravimetric	SM2540B
Solids, TSS	Water	Poly	500 ml	None	None	7 Days	Gravimetric	EPA 160.2
Solids, TVS	Water	Poly	500 ml	None	None	7 Days	Gravimetric	EPA 160.4
Solids, VSS	Water	Poly	500 ml	None	None	7 Days	Gravimetric	SM 2540E
Sulfate	Water	Poly	250 ml	None	None	28 Days	IC	EPA 300.0
Sulfide, Dissolved	Water	Poly	250 ml	NAOH	NAOH	24 hours	Colorimetric	SM4500S2D
Surfactants (MBAS)	Water	Poly	500 ml	None	None	48 Hours	Colorimetric	SM5540C
t-Butyl Alcohol	Water	Glass	2 x 40 ml	none	None	14 Days	GC/MS	EPA 524.2

THMs	Water	Amber Glass	2 x 40 ml	Thiosulfate	Thiosulfate	14 Days	GC/MS	EPA 524.2
THMs-Formation Potential	Water	Amber Glass	1L	None	None	14 Days	GC/MS	SM5710/EPA 524.2
Total Organic Carbon	Water	Amber Glass	250 ml	H3PO4	H3PO4	28 Days	UV-Persulfate	SM5310C
Total Organic Halides	Water	Amber Glass	500 ml	H2SO4	Sulfite/H2SO4	14 Days	Pyrolysis/ Coulometric	SM5320B/EPA 9020
Turbidity	Water	Poly	250 ml	None	None	48 Hours	Nephelometric	EPA 180.1
UCMR2-PBDEs	Water	Amber Glass	2 x 1 L	Ascorbic, EDTA, Citrate	Ascorbic, EDTA, Citrate	14 days	GCMS	EPA 527
UCMR2-Explosives	Water	Amber Glass	2 x 1 L	CuSO4/Trizma Buffer	CuSO4/Trizma Buffer	14 days	GCMS	EPA 529
UCMR2-Perchlorate	Water	Poly-Sterile	125 ml	Sterile Field Filtration	Sterile Field Filtration	28 days	LC/MS/MS	EPA 331/332
UCMR2-Acetanilide Degradates	Water	Amber Glass	2 x 500 ml	NH4CI	NH4CI	14 days	LC/MS/MS	EPA 535
UCMR2-Acetamide Pesticides	Water	Amber Glass	2 x 1 L	Sulfite/HCL	Sulfite/HCL	14 days	GCMS	EPA 525.2
UCMR2- Nitrosamines	Water	Amber Glass	1 x 1 L	Thiosulfate	Thiosulfate	14 days	GCMS	EPA 521
UV254	Water	Amber Glass	250 ml	None	None	2 Days	Spectrophotometric	SM 5910B
Volatile Organics- DW	Water	Glass	3 x 40 ml	HCL	Ascorbic/HCL	14 Days	GC/MS	EPA 524.2
Volatile Organics- Aromatics only	Water	Glass	2 x 40 ml	HCL	Thiosulfate/HCL	14 Days	P&T/PID	EPA 602
Volatile Organics- WW/GW	Water	Glass	2 x 40 ml	HCL	Thiosulfate/HCL	14 Days	GC/MS	EPA 624/8260B
Gasoline -TPH	Water	Glass	2 x 40 ml	HCL	Thiosulfate/HCL	14 Days	P&T/FID	EPA 8015B
Diesel/Oil-TPH	Water	Amber Glass	1 L (*)	HCL	Thiosulfate/HCL	14 Days	GC/FID	EPA 8015B

Notes:

(1): Formaldehyde and acetaldehyde only
(2): Al,Sb,As,Ba,Be,B,Cd,Ca,Na,Mg,K,Cr,Co,Cu,Fe,Pb,Li,Mn,Mo,Ni,Se,Ag,Sr,Tl,Ti,V,Zn
(\*): Needs extra bottles for QA/QC for certain projects.

List of SOPs as of July 2009

### SOP's LIST AND INDEX Administration - Miscellaneous and administrative SOPs

File Name	Rev No	Rev Date	Method	Title
MIS001	18	Jun-09	General	Sample Receiving, Log in, Storage and Disposal
MIS002	5	Mar-09	Sampling	Industrial Wastewater Sampling Instructions
MIS003	3	Jul-05	General	Back Up Procedures for Data Files
MIS004	5	Apr-08	General	Chemicals, Standards and consumable materials, Receipt, Storage and Preparation of Solutions
MIS005	3	Jul-09	General	Procedures for Start Up and Shut Down the File Servers
MIS006				Discontinued
MIS007	2	Mar-08	General	Sample Container Management
MIS008	3	Mar-08	General	Laboratory Hazardous Waste Management
MIS009	3	Feb-08	General	Handling of Foreign Soil
MIS010	2	Mar-08	Sampling	Sampling Instructions for Protected Groundwater Supplies and Water Supplies with Treatment
MIS011	4	Mar-08	General	Preparation, Approval, Distribution, & Revision of standard Operating Procedures
MIS012	2	Mar-08	General	Significant Figures and Rounding
MIS012	2	Mar-08	General	Generation and Utilization of Control Charts
MIS014	5	Mar-09	General	Performing Internal Audits
MIS015	4	Jun-09	General	Handling and Analysis of Proficiency Testing (PT) Samples
MIS016	3	Apr-08	General	Corrective Action Procedures
MIS017	3	Apr-08	General	Maintenance, Utilization and Review of Laboratory Logbooks
MIS018	5	May-09	General	Internal Laboratory Data Verification and Review
MIS019	3	Apr-08	General	Resolution of Customer Complaints
MIS020	3	Apr-08	General	Calibration and Verification of Analytical Balances
MIS021	3	Apr-08	General	Calibration and Maintenance of Mechanical Pipettes
MIS022	2	Oct-03	General	LIMS Security Systems
MIS024	2	Apr-08	General	DI Water Quality Checks
MIS025	3	Apr-08	General	Control of Data and Manual Data Entry
MIS026	3	May-09	General	Taking Representative Samples and Sub-samples in the Laboratory.
MIS028	4	Mar-09	General	Standard Cleaning Protocols for Containers and Labware
MIS029	3	Apr-08	General	Calibration and Verification of Thermometers
MIS030	4	Apr-08	General	Performing Managerial Reviews
MIS031	5	Mar-09	General	Calibration and Verification of Lab Support Equipment
MIS032	3	Mar-09	General	Calculation of Method Detection Limits (MDL) and Reporting Limits (RL)
MIS033	2	Apr-08	General	Rejection/acceptance Criteria for Special Analyses
MIS034	4	Mar-09	General	Performing Initial Demonstration of Capability (IDC)
MIS035	4	Apr-08	General	Procedures for Initiation of Employment for a new Associate
MIS036	2	Apr-08	General	Use of Areas of Incompatible Activities
MIS037	3	Nov-06	General	Computers and Electronic Data Requirements
MIS038	2	Apr-08	General	Chain of Custody Procedures for Legal and Evidentiary Custody of Samples
MIS039	2	Apr-08	General	Proper Raw Data Handling and Manual Integration Procedures
MIS040	2	Oct-03	General	Archival System for Instrument Raw Data
MIS041	2	Apr-08	General	Procedures for Subcontracting Client Samples
MIS042	4	Mar-09	General	Outside Support Services and Supplies
MIS043	3	Apr-08	General	Implementation of the Business Ethics and Data Integrity Policy
MIS044	3	Mar-09	General	Control of Nonconforming Environmental Testing
MIS045	4	Mar-09	General	Control of Records and Documents
MIS046	3	Mar-09	General	Training of Laboratory Personnel

MIS047	3	Mar-09	General	Estimating the Uncertainty of Measurements
MIS048	3	Apr-08	General	Development and Maintenance of Test Method SOPs
MIS049	2	Apr-08	General	Health and Safety Training Procedures
MIS050	1	Oct-08	General	Disaster Procedures
MIS051	1	Jun-09	General	Sample Disposal
MIS052	1	Jul-09	General	Acceptance criteria for analyte confirmation

### SOP's LIST AND INDEX Inorganic Department - Metals SOPs

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01	6	07	1311	Toxicity Characteristic Leaching Procedure (TCLP)
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	3	S	IVIOU	Acia Digestion of Sediments, Sludges, Solis and Wipes, EPA Method 5050 Modified.
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11	5	08	245.1	Analysis of Hg in water by manual cold vapor technique EPA method 245.1
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17	8	08	6010	Analysis of Trace Metal in Water and Solid Matrices by ICP-AES, EPA Method 6010
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MIC		8		b-09	SM9223	Analysis of Total Coliform and E. Coli in Water by P/A Colilert <sup>™</sup> and Enumeration by the Quanti-Tray® method, SM9223
MIC		6	Fe	b-09	SM9215B /SimPlate	Analysis of Heterotrophic Plate Count by Pour Plate and SimPlate Methods, SM 9215B
MIC		7		or-09	SM9221	Analysis of Total and Fecal Coliform in Water by Multiple Tube Fermentation Technique, SM9221
MIC MIC		5 2		ıy-09 ıl-09	QAQC QAQC	Quality Assurance for Microbiological Tests           Using New Methods or Test Kits for Microbiological Determinations
MIC		3		il-09 il-09	QAQC	Verification of Support Equipment Used for Microbiological Determinations
MIC		2		or-09	Enterolert	Bacteriological Analysis of Ambient Water Samples for Enterococci by Enterolert Presence/Absence and Quanti-Tray® Method
MIC		1		or-09	Disposal	Disposal of Material Used for Microbiological Determinations

### SOP's LIST AND INDEX Radio Chemistry Department - RadChem SOPs

File	Rev.	Rev	Method	Title
Name	No	Date		
RAD001	2	Nov-07	900.0	Determination of Gross Alpha and Gross Beta Radioactivity in Drinking Water, EPA Method 900.0
RAD002	1	Jul-05	SM7110C	Determination of Gross Alpha Radioactivity in Water by Coprecipitation, SM 7110C
RAD003	2	Apr-08	903.0	Determination of Alpha-emitting Radium Isotopes in Water, EPA Method 903.0
RAD004	1	Oct-05	All	Quality Control for Radiochemical Analysis
RAD005	1	Apr-06	All	The Procedure for Monitoring Radiation Measurement Instrumentation for Radioactive Contamination
RAD006	1	Apr-06	All	The Procedure for Handling, Storing and Establishing Expiration Dates for Reference Standards
RAD007	1	Jul-06	RA-05	Radiochemical Determination of Radium-228 in Water Samples, EPA Method Ra-05
RAD008	2	May-08	904	Radiochemical Determination of Radium-228 in Water Samples, EPA Method 904.0
RAD009	1	Sep-07	200.8	Spectrometric Determination of Uranium in Water Samples for Radiological Compliance, EPA Method 200.8
RAD010	1	Aug-08	SM7500Rn	Radiochemical Determination of Radon-222 in water samples, SM7500-Rn

### SOP's LIST AND INDEX Inorganic Department - Wet Chemistry SOPs

File	Rev	Rev	Method	Title
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WET001	10	Sep-07	300	Analysis of Anions in Water by Ion Chromatography, EPA 300.0
WET002	1	Sep-02	9056	Analysis of Anions in Solid and Liquid Matrices by Ion Chromatography, EPA Method 9056
WET003	11	Oct-08	SM4500CN C,D,E	Analysis of Total Cyanide in Water - Manual Colorimetric/Titimetric, SM4500CN-C,D,E
WET004	8	Oct-08	SM5210B	Biological Oxygen Demand (BOD) Test, SM 5210B
WET005	2	Oct-08	D240	Heat of combustion
WET006	3	Oct-08	418.1	Analysis of Total Recoverable Petroleum Hydrocarbons in Soil, EPA 418.1M
WET007	2	Oct-08	5050	Parr Bomb Preparation Method for Solid Waste analysis, EPA Method 5050
WET008	3	Oct-08	SM5540D	Non-ionic Surfactants as CTAS (Cobalt Thiocyanate Active Substances) SM 5540D
WET009	7	Oct-08	SM2120B	Analysis of Color in Water, SM 2120B
WET010	2	Oct-08	SM4500CN M	Analysis of Thiocyanate in Wastewater by SM 4500-CN M
WET013	3	Oct-08	140.1	Analysis of Odor in Drinking Water, EPA Method 140.1/SM 2150
WET015	3	Oct-08	E203	Analysis of Water Content by Karl Fisher Titration ASTM Method E203
WET018	4	Oct-08	SM4500CN G	Analysis of Cyanide Amenable to Chlorination in Water - Manual Colorimetric, SM 4500CN-G
WET019	5	Mar-08	420.1	Analysis of Low Level Total Recoverable Phenolics in Water by chloroform Extraction and Manual Spectrophotometry, EPA 420.1
WET021	7	Oct-08	1010	Ignitability by Pensky Marten Closed Cup Method, EPA Method 1010
WET022	4	Nov-08	SM2320B	Determination of Alkalinity by the Titrimetric Method, SM 2320B

WET024	5	Dec-08	SM2310B	Analysis of Acidity as CaCO3, SM 2310B
WET027	3	Dec-08	3060	Alkaline Digestion for Analysis of Hexavalent Chromium in Solid Matrices, EPA Method 3060
WET027 WET028	5	Jan-08	SM4500 H B	pH (Electrometric), SM 4500-H+ B
	-	Dec-08	SM3500 Cr	
WET029	4		D SM4500 S2	Analysis of Hexavalent Chromium in Water - Manual Colorimetric, SM 3500-Cr D
WET032	4	Dec-08	D	Analysis of Dissolved Sulfide - Methylene Blue Method, SM 4500-S= D)
WET033	4	Dec-08	9030/9034	Analysis of Acid-Soluble and Acid-Insoluble Sulfides, EPA Method 9030A
WET038	4	Dec-08 Jan-08	SM4500CI G	Analysis of Total Residual Chorine by Colorimetry with DPD, SM 4500Cl G
WET039 WET041	<u>6</u> 7	May-08	SM2510B SM2540C	Determination of Specific Conductance, SM 2510B Filterable Residue (TDS) by Gravimetric analysis, SM 2540C
WET041 WET042	7	Dec-08	SM2540C	Determination of Non-filterable Residue (TSS) by Gravimetry, SM 2540D
WE1012		200.00	010120400	Determination of Methylene Blue Active Substances (MBAS) by Spectrophotometry,
WET043	4	Jan-09	SM5540C	SM 5540C
WET044	2	Dec-08	253B	Analysis of Thiosulfate and Sulfite by Iodometric Titration, LACSD Procedure 253B
WET046	3	Dec-08	SM2540B	Determination of Total Residue (TS) by Gravimetry, SM 2540B
WET047	4	Jun-08	160.4	Determination of Volatile Residue (VS) by Gravimetry, EPA Method 160.4
WET048	4	Dec-08	SM2540F	Determination of Settleable Residue (SS) by Volumetric Imhoff Cone, SM 2540F
WET050	5	Jan-08	410.4	Determination of Chemical Oxygen Demand in Water by Colorimetry, EPA Method 410.4
				Determination of Oil & Grease (HEM and SGT-HEM) by Solid Phase Extraction and
WET055	7	Dec-08	1664	Gravimetry, EPA Method 1664A
WET056	5	May-09	180.1	Determination of Turbidity by Nephelometric Method, EPA Method 180.1
WET059	3	Dec-08	FMC	Analysis of Hydrogen Peroxide by FMC Method
WET062	3	Dec-08	9065M	Analysis of Total Recoverable Phenolics in Solid Matrices, EPA Method 9065 Modified
WET064	3	Dec-08	9045C	Determination of pH in Soil and Solid Matrices, EPA Method 9045C
WET065	3	May-09	9040B	Determination of pH in Liquid Waste and Multiphase Waste, EPA Method 9040B
WET069	2	May-09	SM2340B	Determination of Hardness by Calculation, SM 2340B
WET070	•	D	SM4500CIO2	
WET070	3	Dec-08	D	Analysis of Chlorine Dioxide by Colorimetric Method with DPD, SM 4500-CIO2 D
WET072	3	Dec-08	SM4500 O G	Determination of Dissolved Oxygen by Membrane Electrode Method, SM 4500-O G
WET073	3	Dec-08	SM4500SO3 B	Analysis of Sulfite by Iodometric Method, SM4500SO3= B
WEIGIG	0	200.00	В	Distillation and Analysis of Total and Amenable Cyanide in Waste and Solid Matrices
WET074	3	Dec-08	9010/9014	,EPA Method 9010B/9014
WET075	2	Dec-08	CCR ch10	Determination of Ignitability in Waste, CCR Chapter 10, Article 3
WET077	2	Dec-08	CCR ch10	Determination of Corrosivity in Waste, CCR Chapter 10, Article 3
WET078	2	Dec-08	SM5910	Determination of UV Absorbing Constituents (UV-254), SM 5910
	~	Dec 00	7400	Analysis of Haussiant Observing by Manual Osserting by the EDA M. H. 174004
WET079	2	Dec-08	7196	Analysis of Hexavalent Chromium by Manual Spectrophotometric, EPA Method 7196A
WET080	4	Dec-08	365.3	Analysis of Total Phosphorus and Ortho Phosphate in Water by Manual Colorimetric Method, EPA Method 365.3
WET081	2	Dec-08	ASTM2382	Determination of Heat of combustion, ASTM Method 2382
WET084	2	Jan-09	353.2	Analysis of Nitrate and Nitrite in Water by Automated Colorimetry, EPA Method 353.2
WET086	2	Jan-09	350.1	Analysis of Ammonia in Water by Automated Colorimetry, EPA Method 350.1
	~			Analysis of Total Phosphorus in Water by Acid Persulfate Digestion and Automated
WET087	2	May-09	365.1	Colorimetry, EPA Method 365.1
WET088	2	May-09	365 1	Analysis of Orthophosphate in Water by Automated Colorimetry, EPA Method 365.1
VVE1000	2	way-09	365.1	Analysis of Orthophosphale in Waler by Automated Coloninetry, EFA Method 363.1

WET089	3	Jan-09	351.2	Analysis of Total Kjeldahl Nitrogen (TKN) in Water by Heating Block Digestion and Automated Colorimetry, EPA Method 351.2
WET091	2	Jan-09	335.4	Analysis of Total Cyanide in Water by Midi-Distillation and Automated Colorimetry, EPA Method 335.4
WET093	2	Jan-09	SM10200H	Analysis of Chlorophyll-a and Pheophytin-a, SM 10200-H
WET094	1	Sep-05	SM5710B	Determination of Trihalomethane Formation Potential (THMFP), SM 5710B
WET095	2	Jan-09	415.3	Determination of TOC and SUVA in Drinking Water, EPA Method 415.3
WET096	2	Jan-09	D6646-03	Analysis of the Accelerated Hydrogen Sulfide Breakthrough Capacity of Granular and Pelletized Activated Carbon, ASTM D6646-03
WET097	2	Jan-09	D2862	Standard Test Method for Particle Size distribution of Granular Activated Carbon, ASTM D2862-82
WET098	2	Jan-09	D2867	Standard Test Method for Moisture in Activated Carbon, ASTM D2867-83
WET099	2	Jan-09	D2866	Standard Test Method for Total Ash in Activated Carbon, ASTM D2866-83
WET100	2	Jan-09	D3802	Standard Test Method for Ball-Pan Hardness of Activated Carbon, ASTM D3802-79
WET101	2	Jan-09	D5029	Standard Test Methods for Water Solubles in Activated Carbon, ASTM D5029-98
WET102	2	Jan-09	D5832	Standard Test Methods for Volatile Matter Content of Activated Carbon, ASTM D5832- 98
WET103	2	Jan-09	USFilter	Standard Test Methods for Contact pH Test Method
WET104	2	Jan-09	D93	Standard Method for Test for Flash Point by Pensky-Martens Closed Cup Tester, ASTM D93-73
WET105	1	Sep-07	420.4	Determination of Total Recoverable Phenolics in Water by Semi-Automated Colorimetry, EPA Method 420.4

### SOP's LIST AND INDEX Organic Department - Organics SOPs

File	Rev.	Rev	Method	Title
Name	No	Date		
ORG003	7	Apr-05	SM5310C	Total Organic Carbon (TOC) and Dissolved Organic Carbon (DOC), SM 5310C
ORG004	9	Mar-02	SM5320B	Determination of Total Organic Halides (TOX) in Water by Adsorption-Pyrolysis- Titrimetric Method, SM 5320B
ORG005	7	Mar-08	8315	Analysis of Ketones and Aldehydes by HPLC, EPA Method 8315
ORG006	7	Apr-08	8318	Analysis of N-Methylcarbamates by HPLC, EPA Method 8318
ORG007	1	Sep-92	9076	Analysis of Total Halogens and Total Extractable Organic Halides in Solid matrices, EPA Method 9076
ORG008	4	Sep-01	551.1	Analysis of Chlorination Disinfection Byproducts (DBPs) in Drinking water by Liquid- Liquid Extraction and GC/ECD, EPA Method 551.1
ORG009	10	Apr-01	8260	Determination of Volatile Organic Compounds in Groundwater and Soil by GC/MS, EPA 8260B
ORG011	5	Jun-09	8330A	Analysis of Explosive Residues by HPLC
ORG012	4	Dec-04	508A	Screening for Polychlorinated Biphenyls by Perchlorination and Gas Chromatography - EPA Method 508A
ORG013	5	Sep-01	8015	Analysis of Volatile Petroleum Hydrocarbons (VPH, C6 to C10) in Soil and Water samples by P&T and GC/FID, EPA Method 8015
ORG014	4	Sep-01	8021	Determination of Aromatic and Halogenated Volatiles by GC/PID and GC/ELCD, EPA Method 8021A
ORG015	6	Mar-02	8141	Analysis of Organophosphorus Pesticides in Water and Solid Matrices by GC/NPD, EPA Method 8141A
ORG016	7	Mar-02	8081	Analysis of Organochlorine Pesticides in Water and Solid Matrices by GC/ECD, EPA Method 8081A
ORG017	5	Apr-01	549.2	Analysis of Diquat and Paraquat by Solid Phase Extraction and HPLC-UV, EPA Method 549.2

ORG020	6	Apr-08	547	Analysis of Glyphosate by HPLC-Fluorescence, EPA Method 547	
				Analysis of Organochlorine Pesticides and PCBs in Drinking Water by LL Extraction	
ORG022	4	Mar-01	508	and GC-ECD, EPA Method 508	
ORG023	5	Mar-02	8015B	Analysis of Diesel Range Organics in soil and water samples by GC-FID, EPA Method 8015	
ORG024	1	Dec-93	547M	Analysis of Glyphosate in Soil by Extraction and HPLC-Fluorescence, EPA Method 547 Modified	
ORG025	2	Jul-94	24	Determination of Volatile Organic Content (VOC) in Paints and Related Coatings, EPA Method 24	
ORG026	9	Jan-02	524.2	Determination of Volatile Organic Compounds in Water by GC/MS, EPA Method 524.2	
ORG027	1	Feb-94	509	Analysis of Ethylene Thiourea in Drinking Water, EPA Method 509	
ORG028	6	Apr-08	531.1	Analysis of N-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Post Column Derivatization, EPA Method 531.1	
ORG029	5	Jun-02	8151	Analysis of Chlorinated Acid Herbicides in Water and Solid Matrices by GC-ECD, EPA Method 8151	
UKGUZ9	5	Juii-02	0151	Analysis of EDB, DBCP and 123TCP in Water by Microextraction and GC/ECD, EPA	
ORG030	5	Sep-01	504.1	504.1	
ORG032	1	Mar-94	N1003	Analysis of Halogenated Hydrocarbons in Charcoal Tubes, NIOSH Method 1003	
ORG033	5	Apr-08	632	Analysis of Diuron by HPLC-UV, EPA Method 632	
ORG034	1	Jun-94	OSHA57	Analysis of 4,4-Methylenedianiline (MDA) in Air Filters, OSHA Method 57	
0110004	- 1	Juli 34	001//07	Analysis of Semi-Volatile Organic Compounds in Water and Solid Matrices by GC/MS,	
ORG036	10	Feb-01	8270	EPA Method 8270C	
ORG037	5	Mar-01	548.1	Analysis of Endothall in Drinking Water by Solid Phase Extraction and GC/MS, EPA Method 548.1	
ORG038	2	Mar-02	508.1	Analysis of Chlorinated Pesticides and PCBs in Water by Solid Phase Extraction and GC-ECD, EPA Method 508.1	
ORG039	8	Apr-04	525.2	Analysis of Semi-volatile Organic Compounds in Drinking Water by Solid Phase Extraction and GC/MS, EPA Method 525.2	
ORG040	5	Feb-01	625	Analysis of Semivolatile Organics in Wastewater by LL Extraction and GC/MS, EPA Method 625	
ORG041	3	Apr-00	601/602	Analysis of Purgeable Halocarbons and Aromatics in Waste Water by GC-ELCD and GC-PID, EPA Method 601/602	
ORG042	10	Sep-08	314	Analysis of Perchlorate in Water and Solid Matrices by Ion Chromatography, EPA Method 314.0	
ORG043	3	May-02	8270M	Determination of 1,4 Dioxane in Water and Soil by L-L Extraction and Isotopic Dilution GC/MS, EPA Method 8270M	
ORG045	4	Feb-02	3600	Cleanup Procedures for Organic Analyses, EPA Method 3600	
000040	2	Eab 00	2500	Sample Preparation and Extraction for Hazardous Waste Samples, EPA Method	
ORG046	3	Feb-02	3500	3500B	
ORG047	3	Feb-02	3510	Separatory Funnel Liquid-Liquid Extraction, EPA Method 3510B	
ORG048 ORG049	3	Feb-02	3550	Ultrasonic Extraction, EPA Method 3550B	
	2	Feb-02	3580 5030	Waste Dilution Procedure, EPA Method 3580A	
ORG050	3	Mar-02		Purge-and-Trap Extraction Procedure, EPA 5030B	
ORG054	1	Jun-98	8031	Determination of Acrylonitrile by Gas Chromatography, EPA Method 8031	
ORG056	2	Feb-02	3520	Continuous Liquid-Liquid Extraction Procedure, EPA Method 3520C	
ORG057	2	Feb-02	3540	Soxlet Extraction Procedure, EPA Method 3540C Analysis of Polychlorinated Biphenyl's (PCBs) in Liquid and Solid Matrices by GC-ECD, EPA Method 8082	
ORG058 ORG059	5	Mar-02 Jul-99	8082	Determination of Volatile Organic Compounds Specific to the Pharmaceutical Industry by Isotope Dilution GC/MS, EPA Method 1666	
ORG060	3	Feb-01	624	Analysis of Volatile Organic Compounds in Wastewater by GC/MS, EPA Method 624	
ORG062	6	Nov-03	9020B	Determination of Total Organic Halides in Water by Adsorption-Pyrolysis-Titrimetric Method, EPA Method 9020B	

ORG063	3	Jul-02	9020M	Determination of Total Halogens and Total Extractable Organic Halides in Solid and Oil Matrices, EPA Method 9020B Modified			
ORG064	3	Mar-02	608	Analysis of Organochlorine Pesticides and PCBs in Wastewater by GC-ECD, EPA Method 608.			
ORG065	10	Dec-03	1625M	Determination of Ultra Low Levels of N-Nitrosodimethylamine (NDMA) by Continuous L-L Extraction and Isotopic Dilution GC/MS. EPA Method 1625C Mod			
ORG066	2	Feb-03	8270sim	Determination of Low Levels of Polynuclear Aromatic Compound in Water and Solid Matrices by GC/MS SIM Mode, EPA Method 8270 Modified			
ORG067	3	Mar-02	5035	Determination of Volatile Organic Compounds in Soil by Closed-System Purge and Trap and GC/MS, EPA 5035/8260			
ORG069	6	May-08	7199	Analysis of Hexavalent Chromium by Ion Chromatography, EPA Method 7199			
ORG071	2	Mar-02	8015B	Analysis of Alcohols by GC-FID, EPA Method 8015B			
ORG072	2	Mar-02	515.3	Analysis of Chlorinated Acid Herbicides in Water by Microextraction and GC-ECD, EPA Method 515.3			
ORG073	3	Sep-01	505	Analysis of Chlorinated Pesticides and PCBs in Drinking Water by Microextraction and GC-ECD, EPA Method 505			
ORG074	2	Jul-02	General	Establishing Retention Times Windows for Organic Analyses by GC and GC/MS			
ORG075	2	Mar-01	552.2	Analysis of Haloacetic Acids by Microextraction and GC-ECD, EPA 552.2			
ORG076	2	Mar-02		Instrument Maintenance for Organic Analysis			
ORG077	4	May-08	218.6	Analysis of Hexavalent Chromium in Water by Ion Chromatography, EPA 218.6			
ORG078	1	Apr-01	524.2M	Analysis of tert-butyl alcohol (TBA) in drinking water by EPA 524.2M			
ORG079			luft	Analysis of TPH and BTEX by GC/MS LUFT Method			
ORG080	1	Jan-02	528	Analysis of Phenols in Drinking Water by SPE and GC/MS, EPA Method 528			
ORG081	1	Jan-02	526	Analysis of Selected SVOA in Drinking Water by SPE and GC/MS, EPA Method 526			
ORG082	1	Apr-02	TCP-E	Analysis of Low Levels of 1,2,3-Trichloropropane by L-L extraction and GC/MS SIM mode, SRL Method			
		•		Analysis of Low Levels of 1,2,3-Trichloropropane by Purge and Trap and GC/MS SIM			
ORG083	1	May-02	TCP-PT	mode, SRL Method			
ORG085	2	Aug-07	556	Analysis of Aldehydes by Microextraction and GC-ECD, EPA Method 556			
ORG086	1	Jul-02	3535	Solid Phase Extraction Procedures - Manual and Automated, EPA Method 3535			
ORG087	2	May-08	300.1	Analysis of Low Levels of Oxyhalides by Ion chromatography, EPA Method 300.1			
ORG088	2	May-08	532	Analysis of Diuron and Linuron in Water by SPE and HPLC-UV, EPA Method 532			
ORG089	1	Feb-04	1624	Analysis of Acrolein and Acrylonitrile in Water by EPA 1624			
ORG090	1	Mar-04	8270SIM	Analysis of Low Level Phenols in Water and Solid by GC/MS in SIM Mode, EPA Method 8270 Modified			
ORG091	3	Jun-08	326	Analysis of Low Level Chlorite, Chlorate and Bromate by Ion Chromatography and Post-column derivatization, EPA Method 326			
ORG092	2	Jan-08	OSHA 20M	Analysis of Hydrazine by HPLC, OSHA Method 20M (Modified)			
ORG094	2	May-09	8316	Analysis of Acrylamide by HPLC, EPA Method 8316			
ORG095	1	Sep-05	1614M	Analysis of PBDEs by isotopic dilution GC/MS-EI, EPA Method 1614 Modified			
ORG096	1	Nov-06	orgtin	Determination of Low Levels Organotins by GC-MS.			
ORG097	2	Jun-08	332	Analysis of Low Levels organouns by CO-MS, EPA Method 332.0			
ORG098	1	Aug-06	8310	Analysis of Polynuclear Aromatic Hydrocarbons by HPLC, EPA Method 8310			
ORG099	2	Oct-08	331	Analysis of Low Level Perchlorate by LC-MS/MS, EPA Method 331.0			
ORG100	1	Mar-06	535	Analysis of Chloroacetanilide/acetamide Herbicides by LC/MS, EPA Method 535			
ORG101	1	Mar-06	521	Analysis of Nitrosamines by SPE-GC/MS/MS EPA Method 521			
ORG102	2	Mar-08	527	Analysis of Pesticides and Flame Retardants by SPE-GC/MS EPA Method 527			
ORG102	2	Nov-08	529	Analysis of Explosives by SPE-GC/MS EPA Method 529			
ORG104	1	May-06	300M	Analysis of Iodide by Ion Chromatography, EPA Method 300 Modified			
ORG105		Apr-06	LCMS	Tuning the Varian 1200L LC/MS			

ORG106	1	Aug-06	610	Analysis of Polynuclear Aromatic Hydrocarbons by HPLC, EPA Method 610			
			DOD-				
ORG107	1	Oct-06	CIO4	Analysis of Low Level Perchlorate in Water and Soil by LC-MS/MS, DoD Method			
ORG108	1	Jan-07	556M	Analysis of Aldehydes in Solid/Soil by GC-ECD, EPA Method 556 Modified			
ORG109	1	Sep-07	1671	Analysis of Triethanolamine by Direct Injection and GC-FID			
ORG110	1	Dec-07	D7065	Analysis of Alkyl Phenols and Alkyl Phenol Ethoxylates by L-L extraction and GC/MS full scan and SIM, ASTM Method D7065			
				Analysis of Pharmaceuticals, Personal Care Products and Endocrine Disruptive			
ORG111	2	Mar-09	LCMS	Compounds LC-MS/MS.			
ORG113	1	May-08	632M	Determination of Diuron in solid matrices			
ORG114	1	Jun-08	IC/MS/MS	Analysis of 4-Chlorobenzenesulfonic acid (pCBSA) by IC/MS/MS			
ORG115	1	Jun-08	525.2	Determination of organophosphorous pesticides in drinking water by liquid-solid extraction and capillary column GC/MS, via EPA Method 525.2			
ORG116	1	Aug-08	8316M	Analysis of Acrylamide by LC/MS/MS			
ORG117	1	Nov-08	GCMS CI	Analysis of Pyrethroid Pesticides in Water and Soil/Sediment by Extraction and GC/MS in NCI mode and SIM			
ORG118	1	Apr-09	537	Analysis of Perfluorinated Compounds in Water by LC-MS/MS			
ORG119	1	Apr-09	607M	Analysis of NDMA and DMN and Bromacil by EPA Method 607 modified			
ORG120	1	May-09	SM6040D	Analysis of MIB and Geosmin by on line SPME and GC/MS/MS, SM6040D			

### APPENDIX 8 Acceptance Limits for QC Determinations

The Acceptance Limits for QC determinations are in some cases mandatory limits and in other cases the limits are updated periodically from past results. This process is performed though the LIMS. For current acceptance limits please refer to the LIMS.

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#### **APPENDIX 9**

#### **DEMONSTRATION OF CAPABILITY**

A demonstration of capability (DOC) must be made prior to using any test method, and at any time there is a change in instrument type, personnel or test method.

All demonstrations shall be documented through the use of the form in this appendix.

The following steps are performed.

- A quality control sample shall be obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.
- b) The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration approximately 10 times the method-stated or laboratory-calculated method detection limit.
- c) At least four aliquots shall be prepared and analyzed according to the test method either concurrently or over a period of days.
- d) Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations of the population sample for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory must assess performance against established and documented criteria.
- e) The calculated mean and standard deviation are compared to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if they are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.
- f) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to 1) or 2) below.
  - 1) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.
  - 2) Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, confirms a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

#### CERTIFICATION STATEMENT

The following certification statement shall be used to document the completion of each demonstration of capability. A copy of the certification statement shall be retained in the personnel records of each affected employee.



### Training Record (Method and Technique) and Demonstration of Capability Statement

> Matrix:	Date:					
Method:	□ SOP:					
I have read, understand, and agree to use the	I have read, understand, and agree to use the latest version of the test method and SOP.					
Analyst's Signature	Date					
$\supset$ Training courses or workshops on equipments	, analytical techniques and lab procedures:					
	biking using syringes and volumetric flasks. On-site training for d hardware of GC/MS#1, 8(Agilent 5890,6890)provided by by Full Spectrum and Tekmar.					
Analyst's Signature	Date					
Technical Director's Name and Signature	Date					
➢ IDOC Certification Statement:						
	(achment)					
	achment)					
<ul> <li>Proficiency Demonstrated by: (See attained as a comparison of a blind sample.</li> <li>b. Another demonstration of capability.</li> </ul>	achment)					
<ul> <li>Proficiency Demonstrated by: (See attained as a comparison of a blind sample.</li> <li>Another demonstration of capability.</li> <li>Acceptable at least 4 consecutive LCS.</li> </ul>						
<ul> <li>Proficiency Demonstrated by: (See attained and the second s</li></ul>	tachment) r trained analyst with statistically indistinguishable results					
<ul> <li>Proficiency Demonstrated by: (See attained and the second s</li></ul>	r trained analyst with statistically indistinguishable results					
<ul> <li>Proficiency Demonstrated by: (See attain a</li></ul>	r trained analyst with statistically indistinguishable results which is in use at this facility for the analyses of samples under the					
<ul> <li>Proficiency Demonstrated by: (See attain aAcceptable performance of a blind sample.</li> <li>bAnother demonstration of capability.</li> <li>cAcceptable at least 4 consecutive LCS.</li> <li>dAnalysis of authentic sample analyzed by another</li> <li>the undersigned, CERTIFY that:</li> <li>1 The Analyst identified above, using the cited test method(s), we National Environmental Laboratory accreditation Program</li> </ul>	r trained analyst with statistically indistinguishable results which is in use at this facility for the analyses of samples under the ram, have met the Demonstration of Capability					
<ul> <li>Proficiency Demonstrated by: (See attain aAcceptable performance of a blind sample.</li> <li>bAnother demonstration of capability.</li> <li>cAcceptable at least 4 consecutive LCS.</li> <li>dAnalysis of authentic sample analyzed by another</li> <li>the undersigned, CERTIFY that:</li> <li>1 The Analyst identified above, using the cited test method(s), w</li> </ul>	r trained analyst with statistically indistinguishable results which is in use at this facility for the analyses of samples under the ram, have met the Demonstration of Capability on this certification.					
<ul> <li>Proficiency Demonstrated by: (See attain aAcceptable performance of a blind sample.</li> <li>bAnother demonstration of capability.</li> <li>cAcceptable at least 4 consecutive LCS.</li> <li>dAnalysis of authentic sample analyzed by another</li> <li>the undersigned, CERTIFY that:</li> <li>1 The Analyst identified above, using the cited test method(s), was national Environmental Laboratory accreditation Progration 2 The test method(s) was performed by the analyst(s) identified above.</li> </ul>	r trained analyst with statistically indistinguishable results which is in use at this facility for the analyses of samples under the ram, have met the Demonstration of Capability on this certification. s are available for all personnel on-site					
<ul> <li>Proficiency Demonstrated by: (See attal a</li></ul>	r trained analyst with statistically indistinguishable results which is in use at this facility for the analyses of samples under the ram, have met the Demonstration of Capability on this certification. s are available for all personnel on-site					
<ul> <li>Proficiency Demonstrated by: (See attal.</li> <li>Acceptable performance of a blind sample.</li> <li>Another demonstration of capability.</li> <li>Acceptable at least 4 consecutive LCS.</li> <li>Analysis of authentic sample analyzed by another</li> </ul> the undersigned, CERTIFY that: <ol> <li>The Analyst identified above, using the cited test method(s), we National Environmental Laboratory accreditation Progr</li> <li>The test method(s) was performed by the analyst(s) identified of the associated with the demonstration capability are true,</li> <li>A copy of the test method(s) and the laboratory-specific SOPs</li> <li>The data associated with the demonstration capability are true,</li> <li>All raw data (including a copy of this certification form) neces</li> </ol>	r trained analyst with statistically indistinguishable results which is in use at this facility for the analyses of samples under the ram, have met the Demonstration of Capability on this certification. s are available for all personnel on-site , accurate, complete and self-explanatory (*)					
<ul> <li>Proficiency Demonstrated by: (See attal.</li> <li>Acceptable performance of a blind sample.</li> <li>Another demonstration of capability.</li> <li>Acceptable at least 4 consecutive LCS.</li> <li>Analysis of authentic sample analyzed by another</li> </ul> the undersigned, CERTIFY that: <ol> <li>The Analyst identified above, using the cited test method(s), we National Environmental Laboratory accreditation Progr</li> <li>The test method(s) was performed by the analyst(s) identified of the associated with the demonstration capability are true,</li> <li>A copy of the test method(s) and the laboratory-specific SOPs</li> <li>The data associated with the demonstration capability are true,</li> <li>All raw data (including a copy of this certification form) neces</li> </ol>	r trained analyst with statistically indistinguishable results which is in use at this facility for the analyses of samples under the ram, have met the Demonstration of Capability on this certification. s are available for all personnel on-site , accurate, complete and self-explanatory (*) ssary to reconstruct and validate these analyses have been retained at					
<ul> <li>Proficiency Demonstrated by: (See attal.</li> <li>Acceptable performance of a blind sample.</li> <li>Another demonstration of capability.</li> <li>Acceptable at least 4 consecutive LCS.</li> <li>Analysis of authentic sample analyzed by another</li> </ul> the undersigned, CERTIFY that: <ol> <li>The Analyst identified above, using the cited test method(s), we National Environmental Laboratory accreditation Progr</li> <li>The test method(s) was performed by the analyst(s) identified of the associated with the demonstration capability are true,</li> <li>A copy of the test method(s) and the laboratory-specific SOPs</li> <li>The data associated with the demonstration capability are true,</li> <li>All raw data (including a copy of this certification form) neces</li> </ol>	r trained analyst with statistically indistinguishable results which is in use at this facility for the analyses of samples under the ram, have met the Demonstration of Capability on this certification. s are available for all personnel on-site , accurate, complete and self-explanatory (*) ssary to reconstruct and validate these analyses have been retained at					
<ul> <li>Proficiency Demonstrated by: (See attal.</li> <li>aAcceptable performance of a blind sample.</li> <li>bAnother demonstration of capability.</li> <li>cAcceptable at least 4 consecutive LCS.</li> <li>dAnalysis of authentic sample analyzed by another</li> <li>the undersigned, CERTIFY that:</li> <li>1 The Analyst identified above, using the cited test method(s), we National Environmental Laboratory accreditation Progr</li> <li>2 The test method(s) was performed by the analyst(s) identified 4 A copy of the test method(s) and the laboratory-specific SOPs</li> <li>4 The data associated with the demonstration capability are true,</li> <li>5 All raw data (including a copy of this certification form) neces the facility, and that the associated information is well of the set of</li></ul>	r trained analyst with statistically indistinguishable results which is in use at this facility for the analyses of samples under the ram, have met the Demonstration of Capability on this certification. s are available for all personnel on-site e, accurate, complete and self-explanatory (*) ssary to reconstruct and validate these analyses have been retained at organized and available for review by authorized assessors					

Notes: The demonstration of Capability is performed as per Section 12.5 of Quality Assurance Manual
\*: True: Consistent with supporting data; Accurate: Based on good laboratory practices consistent with sound scientific principles/practices;
Complete: Includes results of all supporting performance testing; Self-Explanatory: Data properly labeled and stored so that the results are clear and
require no additional explanation.

	JRANCE ION REPORT
Date: Name of Analyst:	
Sample ID Number(s) Involved:	
Corrective action to be implemented (1):	
	YES - NO YES - NO
Were samples reported with qualifiers:	
Were samples reanalyzed and acceptable QC obtained: Were samples reported with qualifiers: Approval of corrective action by Technical Director: Signed:	YES - NO
Were samples reported with qualifiers: Approval of corrective action by Technical Director: Signed: Technical Director	
Were samples reported with qualifiers: Approval of corrective action by Technical Director: Signed: Technical Director	YES - NO
Were samples reported with qualifiers:	YES - NO
Were samples reported with qualifiers: Approval of corrective action by Technical Director: Signed: Technical Director	<b>YES - NO</b> Date:
Were samples reported with qualifiers: Approval of corrective action by Technical Director: Signed: Technical Director Comments by TD: Verification of Implementation of corrective action by Q	YES - NO Date:
Were samples reported with qualifiers: Approval of corrective action by Technical Director: Signed: Technical Director Comments by TD:	<b>YES - NO</b> Date:

problem, probable cause of problem and how to prevent from happening again.

#### **APPENDIX 11**

#### Laboratory Accreditations

- NELAC #04229CA
- State of California ELAP #1132
- USEPA UCMR 2 certification
- State of Nevada Division of Environmental Protection Certificate No. CA211-2004-41
- State of Hawaii
- State of New Jersey, certificate # CA015
- Guam Environmental Protection Agency, Certificate # 09-007r
- Los Angeles County Sanitation Districts Industrial Wastewater Testing Number 10143
- South Coast Air Quality Management District Ambient air testing Certificate number 93LA107

### APPENDIX 12 Flags used for Data Qualifiers

Qualifier	Description
	The recommended holding time for this analysis is only 15 minutes. The sample was analyzed as soon as it
*	was possible but it was received and analyzed past holding time.
**	The recommended holding time for field filtering is only 15 minutes. The sample was filtered as soon as possible but it was filtered past holding time. However, the sample was analyzed within holding time.
<	<
>	>
>1000	> 1000
>1500	>/= 1500
_ <fis< td=""><td>&lt; 0.588</td></fis<>	< 0.588
_ <fl< td=""><td>No free liquids</td></fl<>	No free liquids
_ <fp65< td=""><td>&lt; 65</td></fp65<>	< 65
_>=1.6M	>/= 1,600,000
_>=1600	>/= 1600
_>=160K	>/= 160,000
_>=160M	>/= 160,000,000
_>=16K	>/= 16000
_>=16M	>/= 16,000,000
_>=23	>/= 23
_>=230	>/= 230
_>=3.2M	>/= 3,200,000
_>=5700	>/= 5700
_>=57K	>/= 57000
_>2419.6	>2419.6
_>FB	>750
_>fis	> 750
_>FP200	> 200
_0.000	0.000
_100	100 % Survival
_A	Absent
_C	Canceled
_ext	Extracted
_ND	ND
_No Reac	No reaction
_None Vis	None Visible
_P	Present
_pH<2	<2
_seeA	See Attached
_Sub	SUB
_t<2.78	t < 2.78
A-01	[Custom Value]
A-02	[Custom Value]
AS-1	None Detected
В	Blank contamination. The analyte was found in the associated blank as well as in the sample.
B-01	This analyte was found in the method blank, which was possibly contaminated in the lab during preparation. The reporting limit was raised due to the contamination.
B-04	Analyte was found in the travel blank, which was possibly contaminated in the lab during preparation. The batch was accepted since this analyte was not detected for all the samples in the batch.

Qualifier	Description
B-06	This analyte was found in the method blank, which was possibly contaminated during sample preparation. The batch was accepted since this analyte was either not detected or more than 10 times of the blank value for all the samples in the batch.
B-07	This analyte was found in the method blank at levels above the MDL but below the reporting limit.
B-08	Analyte is found in the method blank, which was possibly contaminated during sample preparation.
B-field	No field blank was either received or specified in this batch. Therefore, samples were analyzed without field blank.
BOD-01	The sample dilutions set-up for the BOD analysis did not meet the oxygen depletion criterion of at least 2 mg/l, therefore the reported result is an estimated value only.
BOD-02	The sample dilutions set up for the BOD analysis did not meet the criterion of a residual dissolved oxygen of at least 1 mg/l, therefore the reported result is an estimated value only.
BR	Analyte was found in the method blank, which was possibly contaminated in the lab during preparation. The reporting limit was raised to account for the contamination.
BS-01	The recovery of this analyte in the BS/LCS was over the control limit due to a possible contamination. The batch was accepted based on another acceptable BS and/or MS and MSD that meet the BS criteria.
BS-03	The recovery of this analyte in the BS/LCS was outside the control limits. The sample result was accepted based on another acceptable BS/LCS and/or MS and MSD that meet BS criteria.
BS-04	The recovery of this analyte in LCS or LCSD was outside control limit. Sample was accepted based on the remaining LCS, LCSD or LCS-LL.
BS-H	The recovery of this analyte in the BS/LCS was over the control limit. Sample result is suspect.
BS-L	The recovery of this analyte in the BS/LCS was below the control limit. Sample result is suspect.
CN-1	See case narrative for an explanation of results.
CN-2	See Case Narrative
COD_Cl	COD result is analyzed with chloride correction.
DryWt	The result is in dry weight basis.
E	The concentration indicated for this analyte is an estimated value above the calibration range of the instrument. This value is considered an estimate (CLP E-flag).
E-01	The concentration indicated for this analyte is an estimated value above the calibration range.
FILT	The sample was filtered prior to analysis.
GB-Ad	Adjusted Gross Beta equal to total Gross Beta activity minus Potassium-40 activity
HC-02	Hydrocarbon pattern present in the requested fuel quantitation range but does not resemble the pattern of the requested fuel.
I-03	Low internal standard recovery possibly due to matrix interference or leak in system. The result is suspect.
I-05	Low internal standard recovery possibly due to matrix interference. The result is suspect.
J	Detected but below the Reporting Limit; therefore, result is an estimated concentration.
J-01	No J value detected.
K-40	Potassium-40 calculated based on the concentration of total potassium in mg/L multiplied by the factor 0.82 to convert to activity in pCi/L.
М	Sample result is matrix suspect.
M-01	Result is not valid due to high sample background
M-02	Due to the nature of matrix interferences, sample was diluted prior to extraction. The reporting limits were raised due to the dilution.
M-03	Due to insufficient sample volume, sample was diluted prior to extraction. The reporting limits were raised due to the dilution.
M-04	Due to the nature of matrix interferences, sample extract was diluted prior to analysis. The reporting limits were raised due to the dilution.
M-05	Due to the nature of matrix interferences, sample was diluted prior to analysis. The reporting limits were raised due to the dilution.

Qualifier	Description
M-06	Due to the high concentration of analyte in the sample, sample extract was diluted prior to analysis. The reporting limit was raised due to this dilution.
M-07	Due to high concentration of solid particles in the sample, a smaller volume was used for analysis. The reporting limit was raised due to this dilution.
MIC-2	Result is suspect due to QC failure.
MS-01	The spike recovery for this QC sample is outside of established control limits possibly due to sample matrix interference.
MS-02	The RPD and/or percent recovery for this QC spike sample cannot be accurately calculated due to the high concentration of analyte inherent in the sample.
MS-03	Multiple analyses indicate the percent recovery is out of acceptance limits due to a possible matrix effect.
MS-04	Visual evaluation of the sample indicates the RPD or QC spike is above the control limit due to a non-homogeneous sample matrix.
MS-05	The spike recovery and/or RPD were outside acceptance limits for the MS and/or MSD due to possible matrix interference. The LCS and/or LCSD were within acceptance limits showing that the laboratory is in control and the data is acceptable.
MS-06	Due to noted non-homogeneity of the QC sample matrix, the MS/MSD did not provide reliable results for accuracy and precision. Sample results for the QC batch were accepted based on LCS/LCSD percent recoveries and RPD values.
MS-07	The spike recovery was outside acceptance limits for the MS and/or MSD. The batch was accepted based on acceptable LCS recovery.
MS-08	Due to the nature of matrix interferences, sample was diluted prior to analysis. The MS/MSD could not be quantitated due to the dilution. The batch was accepted based on acceptable LCS recovery.
MS-09	The recoveries of MS/MSD are not valid due to high sample background
MS-10	Due to insufficient sample, LCS/LCSD were analyzed in place of MS/MSD.
MS-11	The QC limits for MS/MSD are not applicable due to positive sample background.
MS-4X	The spike recovery was outside of QC acceptance limits for the MS and/or MSD due to analyte concentration at 4 times or greater the spike concentration. The QC batch was accepted based on LCS and/or LCSD recoveries within the acceptance limits.
MS-BG	The spike recovery was outside of QC acceptance limits for the MS and/or MSD due to sample background. The QC batch was accepted based on LCS and/or LCSD recoveries within the acceptance limits.
O-02	This result was analyzed outside of the EPA recommended holding time.
O-04	This analysis was performed outside the EPA recommended holding time.
O-05	The extraction for this analyte was performed outside of the EPA recommended holding time.
O-07	Sample date and/or time not provided by client. Therefore, default date and/or time has been entered. The analysis may be outside of recommended holding time.
O-08	The original extraction and/or analysis of this sample yielded QC recoveries outside acceptance criteria. It was re-extracted/re-analyzed after the recommended maximum hold time.
O-09	This sample was received with the EPA recommended holding time expired.
O-10	The original analysis of this sample yielded QC recoveries outside acceptance criteria. It was re-analyzed after the recommended maximum hold time.
<b>O-11</b>	The sample was originally analyzed within holding time. However, it required a dilution and the re- analysis was performed after the recommended holding time had expired.
O-12	The sample was originally analyzed within holding time. However, it was reanalyzed without dilution that exceeded the recommended holding time.
O-14	This analysis was requested by the client after the holding time was exceeded.
O-15	The sample was received with the recommended holding time nearly expired. It was analyzed as soon as possible but the maximum holding time was slightly exceeded.

Qualifier	Description
O-21	This sample was analyzed 1 hour past the EPA recommended holding time.
O-22	This sample was analyzed 2 hours past the EPA recommended holding time.
O-25	This sample was received unpreserved and with the recommended holding time for preservation of 48 hours expired.
P-01	Low recovery due to preservative. Sample data accepted based on passing LCS result.
P-2	Sample received without proper preservation and was preserved at the lab upon receiving.
P-5	Due to the nature of the sample matrix a 1:10 dilution was necessary to perform a corrosivity measurement.
Q	One or more quality control criteria failed.
Q-01	The recovery of this analyte in QC sample was outside control limits. Sample was justified as ND based on the low level standard at or below the reporting limit.
Q-02	Low recovery of this analyte in the QC sample. The analysis of the low level standard produced acceptable recovery indicating that the sample result might be accurately reported as Not Detected.
Q-08	High bias in the QC sample does not affect sample result since analyte was not detected.
Q-09	This analyte bias high in QC sample. A fresh spiking solution is going to be prepared.
Q-10	This analyte has high bias in QC sample, the result is suspect.
Q-11	This analyte is low in QC sample, the result is suspect.
Q-12	The RPD result exceeded the QC control limits possibly due to a possible matrix effect; however, both percent recoveries were acceptable. Sample results for the QC batch were accepted based on the percent recoveries and/or other acceptable QC data.
Q-H-1	High bias, data was accepted since sample was not detected.
Q-L-03	This analyte is low in QC sample. Sample data is accepted based on acceptable CCVs.
Q-R-01	Analyses are not controlled on RPD values from sample concentrations less than the reporting limit. QC batch accepted based on LCS and/or LCSD QC results.
QR-03	The RPD value for the sample duplicate or MS/MSD was outside of QC acceptance limits due to matrix interference. QC batch accepted based on LCS and/or LCSD recovery and/or RPD values.
QR-04	The RPD value for the MS/MSD was outside of QC acceptance limits however both recoveries were acceptable. The QC batch was accepted based on acceptable results for the recoveries and RPD for the LCS and LCSD.
R-01	The Reporting Limit for this analyte has been raised to account for matrix interference.
R-02	Elevated Reporting Limits due to limited sample volume.
R-03	The RPD is not applicable for result below the reporting limit (either ND or J value).
R-04	Due to foaming, the sample was diluted prior to analysis. The reporting limits were raised due to the dilution.
R-05	The sample was diluted due to the presence of high levels of non-target analytes resulting in elevated reporting limits.
R-06	Sample was diluted prior to extraction due to high sample concentration, reporting limit was raised due to the dilution.
RAD-1	Gross Alpha: DLR (Detection Limits for Purposes of Reporting) = 3 pCi/L, and MCL (Maximum contaminant Level) = 15 pCi/L.
RAD-2	Gross Beta: DLR (Detection Limits for Purposes of Reporting) = 4 pCi/L, and MCL (Maximum contaminant Level) = 50 pCi/L.
RAD-3	The elevated counting error and MDA was caused by smaller sample aliquot used for analysis due to matrix effect (high TDS).
S-01	The surrogate recovery could not be calculated due to sample dilution required from high analyte concentration and/or matrix interferences.
S-02	The surrogate recovery for this sample cannot be accurately quantified due to interference from coeluting organic compounds present in the sample extract.

Qualifier	Description
S-03	High surrogate recovery for this sample is possibly due to a sample matrix effect. The data was accepted since all target analytes were not detected.
S-04	The surrogate recovery for this sample is outside of established control limits due to possible sample matrix effect.
S-05	Surrogate recovery was below acceptance limit possibly due to matrix effect. Sample data was justified as acceptable since all target analytes were still not-detected or below the reporting limits when adjusted accordingly to surrogate recovery.
S-06	The recovery of this surrogate is outside control limits due to sample dilution required from high analyte concentration and/or matrix interference's.
S-07	Surrogate recovery out of acceptance limits for this sample is possibly due to sample matrix effect, confirmed by re-extracting and/or re-analyzing the sample.
S-08	No surrogate recovery, possibly surrogate spiking was missed.
S-09	Wrong amount spiked, quantification is not accurate
S-10	Surrogate recovery outside method QC limits due to extraction related problems
S-AC	Acid surrogate recovery outside of control limits due to a possible matrix effect. The data was accepted based on valid recovery of remaining two acid surrogates.
S-BLK	Surrogate recovery outside of control limits for Method Blank. The data was accepted since all target analytes were not detected
S-BN	Base/Neutral surrogate recovery outside of control limits due to a possible matrix effect. The data was accepted based on valid recovery of remaining two base/neutral surrogates.
S-BS	Surrogate recovery outside of control limits for LCS. The data was accepted based on valid recovery of the target analytes.
S-DUP	Duplicate analysis confirmed surrogate failure due to matrix effects.
S-GC	Surrogate recovery outside of control limits due to a possible matrix effect. The data was accepted based on valid recovery of the remaining surrogate.
S-HI	High surrogate recovery was confirmed as a matrix effect by a second analysis.
S-LOW	Low surrogate recovery confirmed as a matrix effect by a second analysis.
S-MS	Surrogate recovery outside of control limits for MS/MSD. The data was accepted based on valid recovery of the target analytes.
S-MS1	Surrogate recovery outside of acceptance window confirmed as matrix effect by analysis of MS/MSD on this sample.
S_ABC	Analysis subcontracted to Aquatic Bioassay & Consulting Laboratories, Inc., non NELAP certified, but is ELAP certified (ELAP Certificate 1907)
S_AIR	Analysis subcontracted to Air Technology Laboratories, Inc., NELAP Certificate # E87847
S_BIO	Analysis subcontracted to Biovir Laboratories, NELAC Certificate #05234CA, ELAP Certificate #1795.
S_CAL	Analysis subcontracted to Caltest Analytical Laboratory, NELAP Certificate 01103CA, ELAP Certificate 1664
TIC	Tentatively Identified Compound using mass spectrometry. The reported concentration is relative concentration based on the nearest internal standard. If the library search produces no matches at, or above 85%, the compound is reported as unknown.
U-01	The sample was received without the proper preservation.
U-02	The sample was received at the lab without proper preservation. However, the sample was then preserved at the lab.
S_CEL	Analysis subcontracted to Calscience Environmental Laboratories, NELAP Certificate 03220CA, ELAP Certificate 1230.
S_COL	Analysis subcontracted to Columbia Analytical Services, NELAP Accredited.
S_CRG	Analysis subcontracted to CRG Marine Laboratories Inc Non-NELAP certified, ELAP Certificate 2261.

Qualifier	Description
S_EMS	Analysis subcontracted to EMS Laboratories, non NELAP certified, but is ELAP certified (ELAP Certificate 1119)
S_EMSL	Analysis subcontracted to EMSL Analytical, Inc., non NELAP certified, but is ELAP certified (ELAP Certificate 1620).
S_FAL	Analysis subcontracted to Frontier Analytical Laboratory, NELAP Certificate 02113CA
S_FGL	Analysis subcontracted to FGL Laboratories, NELAC Certificate 01110CA
S_MAX	Analysis subcontracted to Maxxam Analytics INC., NELAP Certificate 02106A
S_NCL	Analysis subcontracted to North Coast Laboratories, ELAP Certificate 1247
S_PAR	Analysis subcontracted to Paradigm Analytical, NELAP Certificate E87634, ELAP Certificate 2451.
S_PTS	Analysis subcontracted to PTS Laboratories, Inc.
S_RSE	Analysis subcontracted to Radiation Safety Engineering, Inc., Nevada certified.
SeeAtt	See Attachment
T-AgBaH	The sample was treated with Silver, Barium and H+ cartridges to minimize chloride and sulfates interferences prior to analysis.
Т-	The sample was treated with Silver, Barium, H+, and Organics cartridges to minimize chloride, sulfates,
AgBaHRP	and organic interferences prior to analysis.
T-AgH	The sample was treated with silver, and H+ cartridges to minimize chloride interferences prior to analysis.
T-BaH	The sample was treated with Ba and H cartridges to reduce sulfates background interferences.

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## **APPENDIX F**

## STANDARD OPERATING PROCEDURES FOR TOXICITY TESTING AQUATIC BIOASSAY AND CONSULTING LABORATORIES, INC

# STANDARD OPERATING PROCEDURES FOR THE CHRONIC FATHEAD MINNOW LARVAE TOXICITY TEST (EPA 4<sup>th</sup> Ed. Methodology)

#### ENDPOINT DESCRIPTION

Twenty-four hour old fathead minnow larvae (*Pimephales promelas*) are exposed in a static renewal system to various test solutions for seven days. The endpoints are survival and growth (increase in weight) of the larvae compared to the controls.

#### DILUTION WATER

Water used for this test is moderately hard, reconstituted fresh water.

### EFFLUENT CONCENTRATIONS

Test solutions are prepared at test initiation and every 24 hours for seven days. Five concentrations and a control, each with four replicate test chambers, are used.

The chambers are 250 ml borosilicate glass crystallizing dishes. The larvae are contained within 200-micron Nytex screens cemented around a petri dish with silicone sealant. Each cylinder fits inside the dish, the liquid is poured in and the fish are added. All dishes are labeled.

Glassware cleaning procedure:

- 1. Wash in warm, soapy water.
- 2. Rinse with tap water.
- 3. Rinse with reagent grade acetone.
- 4. Rinse with D.I. water.
- 5. Soak in 3N HCL for 24 hours.
- 6. Rinse with D.I. water.
- 7. Rinse with 2N HNO3.
- 8. Rinse with D.I. water.
- 9. Soak in D.I. water for 24 hours.
- 10. Rinse with D.I. water.
- 11. Air dry.

Effluent samples arrive on ice and must be placed in a water bath until temperatures reach 25 deg C before set-up. Various sizes of graduated cylinders are used to prepare solutions. A total volume of 1,250 ml is needed for each concentration: four replicates and one 250ml sample for measuring chemical parameters. Effluent concentrations are typically set at 100%, 56%, 32%, 18% and 10% but if higher toxicity is suspected, concentrations may be at lower ranges as long as the 56% difference between dilutions is maintained.

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#### STANDARD TOXICANT CONCENTRATIONS

A reference toxicant test is run in conjunction with each effluent test conducted. Copper chloride is used as the standard. Four replicates of six concentrations are prepared at 0, 38, 75, 100, 150 and 300ppb. One gallon of each concentration is prepared at the beginning of the test and renewals are made daily.

#### SHIPPING OF TEST ORGANISMS

Newly hatched larvae are shipped from Aquatox in Hot Springs, Arkansan and arrive at Aquatic Bioassay the following day. The conditions of the organisms are checked, and the tests begin the day of arrival to ensure that 24-hour old larvae are used.

#### CHEMICAL PARAMETERS

Aeration is used only when D.O. concentrations fall below 40% saturation. If this becomes necessary, chambers are aerated at a rate not to exceed 100 bubbles per minute. At the beginning of the test and every 24 hours thereafter, the following measurements are recorded: temperature, pH, and conductivity. Dissolved oxygen is measured at the beginning and end of each 24 hour exposure period in one test chamber at all test concentrations and the control. Hardness and alkalinity measurements are made daily on the control and highest concentration as well.

#### INITIATION OF THE TEST

After concentrations are prepared and chemical measurements are recorded, 10 animals are carefully transferred into each Nytex cylinder using disposable transfer pipets. Containers are randomly placed on racks in a temperature-controlled room at 25 + 1 deg C with a photoperiod of 16 hours light and 8 hours dark. A submerged thermograph continuously records temperatures during the testing period.

#### FEEDING

The fish in each chamber are fed approximately 700-1000 newly hatched (<24 hours old) brine shrimp twice daily, once in the morning and then after renewal of the test solutions. The larvae are not fed during the final 12h of the test. All brine shrimp nauplii are rinsed with D.I. water and concentrated before use. The amount of food provided is sufficient to ensure the presence of a small amount of uneaten food at the next feeding. The suitability of each new food supply is determined in a side-by-side test using two treatments with four replicates per treatment. One treatment is fed the new food and the other is fed food already known to be suitable.

#### TEST SOLUTION RENEWAL

Test solutions are renewed daily and prepared in clean 1000 ml beakers. Each Nytex cylinder is carefully lifted from the old solution and transferred into the new solution, taking care not to disturb the larvae. The effluent, which has been stored in the refrigerator, is warmed in a water

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bath to 25 deg C before mixing solutions. Before transferring larvae, the bottom of each petri dish is cleaned of all debris by siphoning with a transfer pipet. Numbers of live larvae is recorded and all dead animals are removed.

#### TERMINATION OF TEST

After the 7-Day exposure period, the test is terminated. The number of surviving larvae are recorded and then transferred into labeled vials containing 70% ethanol for subsequent weight determination. Immediately before drying, the larvae are rinsed in D.I. water. They are then placed in clean, tared aluminum weigh boats and dried at 100 deg C for a minimum of 6 hours. Immediately after removal from the oven, boats are placed in a desiccator overnight to completely cool before weighing. All weights are measured to the nearest 0.001 mg. The average dry weight is determined for each replicate by dividing the final dry weight by the number of original larvae in the test chamber.

#### ANALYSIS

A review of concentration-response relationships as well as a comparison of the percent minimum significant difference (PMSD) measured in the test with the PMSD bound variability is conducted on all multi-concentration tests following guidelines in EPA821-B-00-004, July 2000, *Method Guidance and Recommendations for Whole Effluent Toxicity (Wet) Testing (40 CFR Part 136).* The flowcharts for statistical analysis of survival and growth (biomass) as described in the EPA manual are followed to obtain NOEC estimates.

#### TEST ACCEPTABILITY

- 1) Control survival must be greater than 80%.
- 2) Average dry weight must be greater than 0.25 mg.

#### REFERENCE

USEPA. 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. EPA-821-R-02-013.

EPA-821-B-00-004, July 2000, Method Guidance and Recommendations for Whole Effluent Toxicity (Wet) Testing (40 CFR Part 136).

# STANDARD OPERATING PROCEDURES FOR *Ceriodaphnia* SURVIVAL AND REPRODUCTION TOXICITY TEST (EPA 4<sup>th</sup> Ed. Methodology)

### ENDPOINT DESCRIPTION

Less than 24-hour-old *Ceriodaphnia* are exposed to different concentrations in a static renewal system until 60% of the surviving organisms have three broods of offspring. Control organisms usually produce three broods during a seven- eight day period. The endpoints are survival and reproduction.

### DILUTION WATER AND CULTURE MEDIA

Control and dilution water used for this test is moderately hard-reconstituted fresh water.

### EFFLUENT CONCENTRATIONS

Test solutions are prepared at test initiation and every 24 hours for seven-eight days. Five concentrations and a control, each with ten replicate test chambers, are used. 30 ml disposable plastic cups are used as testing chambers. The cups are not washed prior to use but glassware used to make effluent dilutions are cleaned by the following method:

- 1. Wash in warm, soapy water.
- 2. Rinse with tap water.
- 3. Rinse with reagent grade acetone.
- 4. Rinse with D.I. water.
- 5. Soak in 3N HCL for 24 hours.
- 6. Rinse with D.I. water.
- 7. Rinse with 2N HNO3.
- 8. Rinse with D.I. water.
- 9. Soak in D.I. water for 24 hours.
- 10. Rinse with D.I. water.
- 11. Air dry.

Effluent samples typically arrive on ice and must be warmed in a water bath until temperatures reach 25 deg C.

Various sizes of graduated cylinders are used to prepare solutions. A total volume of 500 ml is needed for each concentration: ten replicates and one 250 ml sample for measuring chemical parameters. Effluent concentrations are typically set at 100%, 56%, 32%, 18%, and 10%, but if higher toxicity is suspected, concentrations are set at lower ranges provided there is a 56% difference between dilutions.

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#### STANDARD TOXICANT CONCENTRATIONS

A reference toxicant test is run in conjunction with each effluent test conducted. Copper chloride is used as the standard. Ten replicates of five concentrations are prepared at 5.0, 2.5, 1.25, and 0.75 ppb. One gallon of each concentration is prepared at the beginning of the test and renewals are made daily.

#### TEST ORGANISMS

A culture brood stock of *Ceriodaphnia* is kept on an ongoing basis to ensure adequate supply of neonates. The brood board consists of sixty cups, each containing 15 ml of culture media. One neonate is placed in each cup in the board initiation day and its survival and young are monitored for a period of two weeks. The organisms are fed daily and are transferred to a fresh medium three times weekly. On transfer days, the adult is transferred to fresh medium and the young are counted and discarded (or used in a test). Neonates less than 24h old, and all within 8h of the same age are used for tests. After two weeks, a new board is started using neonates from adults, which produce at least eight young in their third brood. Cultures usually produce at least 15 young per adult in three broods (7 days or less). A mass culture is also maintained in case a population crash occurs in the brood board. Neonates from this culture are used only to start a new brood board and are not directly used for the test. Mass cultures are fed daily and transferred to fresh media weekly. The population is culled periodically to about 50 individuals.

#### FOOD PREPARATION

*Ceriodaphnia* are fed a combination of yeast, cerophyll, Tetramin® brand fish food, and green algae (*Selenastrum*). The yeast, cerophyll, and Tetramin® mixture is prepared in the following manner:

One week prior to making food, 5.0 grams of Tetramin® is added to one liter of deionized water and mixed in a blender. The slurry is poured into an Imhoff cone, covered and aerated for seven days at ambient laboratory temperatures. Any water lost during this digestion procedure is replaced. At the end of the digestion period, the mixture is poured into a flask and allowed to settle for one hour. The supernatant is then filtered through a nytex 100 mesh screen into another 1 liter beaker. The filtered supernatant is combined with the cerophyll and yeast. Fresh, dry "Fleischmans" brand yeast (5.0 grams) is dissolved into one liter of deionized water on a stir plate. The suspension is not allowed to settle and is immediately combined with equal parts of cerophyll and Tetramin®. Excess suspension is discarded. 5.0 grams of cerophyll is placed in a blender with one liter of deionized water and mixed for five minutes. This mixture is filtered through a 110 mesh nytex screen. Equal portions of the three types of prepared food are mixed, and aliquots are poured into 125 ml plastic beakers and frozen until needed. Thawed food is kept in the refrigerator for up to two weeks and fed to the *Ceriodaphnia* daily.

Following food preparation and before aliquots are poured, a suspended solids analysis is performed and the mixture is either concentrated of diluted to obtain a result of 1800 mg/L. The suspended solids are monitored in the following manner:

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- 1. Two pans are oven dried and weighed.
- 2. The combined YCTF is shaken to get a uniform sample.
- 3. 5.0 ml are dispensed in each of the two pans.
- 4. Pans are dried for at least four hours then allowed to cool in the desiccator.
- 5. Pans are weighed again.
- 6. The weights are converted to mg/l by:

Difference in wt. of pans  $X \frac{1000}{1000}$ 

0.005

7. The dilution factor is obtained by:

mg/L TSSthis result is multiplied by volume of YCTF to set the1800 mg/Lfinal volume after dilution.

8. If a large dilution factor was used, the procedure is repeated after dilution to confirm the TSS.

9. The acceptable solids level is between 1700 and 1900 mg/L.

Algae are prepared from an ongoing stock culture maintained in the laboratory. The algae used for the *Selenastrum* toxicity test is inoculated into fresh media weekly. Theremainder of algae is placed in the refrigerator, allowed to settle, and then concentrated. When algae are needed for feeding, a portion of the concentrate is diluted to 3.0 to  $3.5 \times 10$  (7) cells/ml. The density is obtained by hemacytometer counts. Once the final cell density is obtained, the bottle is labeled and recorded in a log book. This concentrate is used for one month. The suitability of each new food supply is determined in a side-by-side test using two treatments with four replicates per treatment. One treatment is fed the new food and the other is fed food already known to be suitable.

#### FEEDING

Cultures are fed daily. 0.1 ml YCTF and 0.1 ml algae are delivered to each cup.

#### INITIATION OF THE TEST

After effluent concentrations are prepared, the following chemical measurements are recorded: dissolved oxygen, pH and conductivity. These measurements are made at the beginning and end of each 24-hour exposure period in at least one test chamber at each test concentration and the control.

Conductivity, alkalinity and hardness are measured and recorded in each new replenishment sample and the control prior to each 24 hour exposure period. Submerged thermographs continuously record temperatures ( $25 + 1 \deg C$ ), and a photoperiod of 16 hours light and 8 hours dark is maintained throughout the testing period. Neonates which are less than 24 hours old and within 8 hours of the same age are selected from individual brood boards. Ten board animals with eight or more young are selected for setting up the test. The ten brood cups are placed in a row. Each concentration of effluent has ten cups. One neonate from the same female is placed in each concentration of effluent. This blocking procedure allows the performance of each female

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to be tracked. If the female produces one weak offspring or male, the likelihood of producing all weak offspring or males is greater. By using this technique, poor performance of young from a given female can be omitted from all concentrations (See USEPA 2002.)

#### TEST SOLUTION RENEWAL

Test solutions are renewed daily and prepared in clean 500 ml beakers. A minimum of three effluent samples are received from the client for use on days 1, 3 and 5. The identity of each sample and the time of first use are recorded on the bench sheets. Samples are stored at 4 deg C. The test organisms are transferred to fresh solutions using disposable transfer pipets. Care is taken to release the animals beneath the surface of the water so that no air is trapped under the carapace. The number of live young and the adult mortality is reported. The young are discarded after recording.

### TERMINATION OF THE TEST

Tests are finished when at least 60% of surviving control females have produced a third brood (usually seven days).

#### ANALYSIS

A review of concentration-response relationships as well as a comparison of the percent minimum significant difference (PMSD) measured in the test with the PMSD bound variability is conducted on all multi-concentration tests following guidelines in EPA821-B-00-004, July 2000, *Method Guidance and Recommendations for Whole Effluent Toxicity (Wet) Testing (40 CFR Part 136).* 

Toxcalc, a computer program is used to analyze data. The flow charts for statistical analysis of survival and growth as described in the EPA manual are followed to obtain NOEC estimates.

#### TEST ACCEPTABILITY

- 1) Control survival must be greater than 80%.
- 2) Reproduction in controls must average 15 or more young per surviving female.
- 3) 60% of surviving control females must produce three broods.

#### REFERENCE

USEPA. 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. EPA-821-R-02-013.

EPA-821-B-00-004, July 2000, Method Guidance and Recommendations for Whole Effluent Toxicity (Wet) Testing (40 CFR Part 136).

# STANDARD OPERATING PROCEDURES FOR Selenastrum capricornutum TOXICITY TEST (EPA 4<sup>th</sup> Ed. Methodology)

#### ENDPOINT DESCRIPTION

*Selenastrum* are exposed to various effluent concentrations for 96 hours. The endpoint is change in cell density.

### DILUTION WATER

Stock solutions are prepared by combining reagent grade chemicals with deionized water, mixing, and storing at 4 deg C in the dark for at least one month before use.

Culture medium is prepared by adding 1 ml of each stock solution including EDTA, in a oneliter volumetric flask and bringing to volume with deionized water. The medium is autoclaved and Na2SiO3 is added once it has cooled. The pH is adjusted to 7.5 by adding 0.1 N NaOH or HCl. Culture media are stored at room temperature until used.

#### ALGAL CULTURES

Stock cultures are initiated weekly by transferring 1 ml of seven to ten day old algae to a 500 ml culture flask containing a fresh medium. Stocks are examined microscopically for contaminating microorganisms at each transfer.

#### SAMPLE PREPARATION

Effluent samples are filtered through a 0.45 um filter to remove contaminating organisms which may be present in the effluent.

One ml of each stock solution including EDTA is added to one liter of effluent to eliminate false negative results caused by low nutrient effluents. By doing this, all test treatments will contain, as a minimum, the concentration of nutrients in the controls.

#### EFFLUENT CONCENTRATION

Test chambers used are 125 ml erlenmeyer flasks. Five concentrations and a control, each with four replicates, are prepared. Chambers and glassware are dry-air sterilized before concentrations are mixed.

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Glassware cleaning procedure:

1. Wash in warm, soapy water.

- 2. Rinse with tap water.
- 3. Rinse with reagent grade acetone.
- 4. Rinse with D.I. water.
- 5. Soak in 3N HCL for 24 hours.
- 6. Rinse with D.I. water.
- 7. Rinse with 2N HNO3.
- 8. Rinse with D.I. water.
- 9. Soak in D.I. water for 24 hours.
- 10. Rinse with D.I. water.

11. Air dry.

Effluent samples usually arrive on ice and must be placed on a hotplate until temperatures reach 25 deg C.

Various sizes of graduated cylinders are used to prepare solutions. A total volume of 500 ml is needed for each concentration: three replicates and one 125 ml sample for measuring chemical parameters. Effluent concentrations are typically set at 100%, 56%, 32%, 18%, and 10%, but if higher toxicity is suspected, concentrations may be set at lower ranges provided there is a 56% difference between dilutions.

### STANDARD TOXICANT CONCENTRATIONS

A reference toxicant test with the same number of concentrations as effluent tests, a minimum of 5 and a control, is run in conjunction with each effluent test. *Zinc chloride* is used as the standard. Typically, 5 concentrations of Zinc Chloride, 32, 56, 100, 180, 320ug/L and a control, are tested.

#### CHEMICAL PARAMETERS

Aeration Is used only when the D.O. concentrations fall below 40% saturation. If this becomes necessary, aeration proceeds at a rate not to exceed 100 bubbles per minute.

At the beginning of the test the following measurements are recorded in all test concentrations and the control: dissolved oxygen, temperature, pH, hardness, alkalinity and residual chlorine. Thereafter daily measurements of conductivity, temperature and pH are recorded. Calibrated thermographs continuously record temperatures throughout the test.

#### INITIATION OF THE TEST

The algal inoculum is prepared no more than 2-3 hours before test initiation. *Selenastrum* from a 4 to 7 day stock culture is used. 100,000 cells per container is required.

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The volume of stock culture required is obtained by: vol. =  $\frac{\text{no. flasks x test vol per flask x 10,000 cells/ml}}{\text{cell density (cells/ml) in stock culture}}$ 

This concentrate is diluted to obtain a final density of 1,250,000 cell/ml. The test begins when algae is added to the flasks. The 1,250,000 cell/ml inoculum is mixed well and 1 ml is added to each test solution.

### INCUBATION

Test flasks are incubated under continuous cool white fluorescent lighting (400 + 40 ft-c) at 25 + 1 deg C and shaken twice daily by hand. The flasks are randomly rotated daily to minimize possible spatial differences in illumination and temperature.

### TERMINATION OF THE TEST

After 96 hours, the test is terminated. The algal growth is measured by Coulter Counter calibrated against the hemacytometer. Two replicate counts are made for each flask. The flask is shaken vigorously and 2 ml are placed in a vial containing 10 ml of 1% saline, stirred and run through the Coulter Counter.

#### ANALYSIS

A review of concentration-response relationships as well as a comparison of the percent minimum significant difference (PMSD) measured in the test with the PMSD bound variability is conducted on all multi-concentration tests following guidelines in EPA821-B-00-004, July 2000, *Method Guidance and Recommendations for Whole Effluent Toxicity (Wet) Testing (40 CFR Part 136).* 

Toxcalc, a computer program, is used to analyze data. The flowchart for statistical analysis of growth as described in the EPA manual is followed to obtain NOEC estimates.

#### TEST ACCEPTABILITY

- 1) Mean cell density of at least 1 X 10(6) cells/mL in the controls.
- 2) Control variability cannot vary more than 20% among replicates.

#### REFERENCE

USEPA. 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. EPA-821-R-02-013.

EPA-821-B-00-004, July 2000, Method Guidance and Recommendations for Whole Effluent Toxicity (Wet) Testing (40 CFR Part 136).

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## **APPENDIX G**

## **EXAMPLE CALIBRATION LOG SHEET**

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## EQUIPMENT CALIBRATION LOG LOS ANGELES IRRIGATED LANDS GROUP

Date (1/1/11)	Time (24hr)	Instruments (HORIBA U10)	Calibrated (X)	Name