Prepared for

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## Water Quality Monitoring Plan

for Newhall Ranch Specific Plan Conditions of Approval and

Clean Water Act Section 401 Water Quality Certification and Waste Discharge Requirements

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#### **1 INTRODUCTION**

Newhall Land, with assistance from Geosyntec Consultants, has prepared this Newhall Ranch Specific Plan (NRSP) Water Quality Monitoring Plan ("Monitoring Plan") to meet the following objectives: (1) evaluate surface and groundwater quality impacts of the overall development per the Newhall Ranch Specific Plan Conditions of Approval (NRSP CoA) and (2) satisfy the storm drain and receiving water quality monitoring requirements of the Clean Water Act Section 401 Water Quality Certification and Waste Discharge Requirements for the Newhall Land Resource Management and Development Plan (RMDP) and Spineflower Conservation Plan (Order No. R4-2012-0139) (WDR). Representative sites upgradient and downgradient of the NRSP area will be monitored for instream surface water and groundwater quality parameters, as specified in the NRSP CoA. Additionally, representative and rotating storm drain outfalls will be monitored as specified in the WDR. This Monitoring Plan provides the methodology and procedures for consistent and scientifically defensible water quality monitoring, including Surface Water Ambient Monitoring Program (SWAMP) compliant procedures, as applicable. A Quality Assurance Project Plan (QAPP) is included in as Appendix A.

#### 1.1 Project Setting

The approximately 12,000 acre NRSP area is located within the unincorporated portion of Los Angeles County between Interstate 5 and the Los Angeles/Ventura County line, just west of Six Flags Magic Mountain amusement park and the City of Santa Clarita (Figure 1).

The NRSP area is located within the Santa Clara River Hydrologic Basin and associated watershed, which is 1,634 square miles in area. The portion of the Santa Clara River watershed that is located generally upstream or east of the Ventura County/Los Angeles County jurisdictional line is approximately 640 square miles in size, and drains portions of the Los Padres National Forest from the north, the Angeles National Forest from the north and northeast, and the Santa Susana Mountains from the south and southeast (Figure 2). The Santa Clara River extends approximately 5.5 miles east to west across the NRSP subregion. The NRSP area comprises 2.9 percent of the Santa Clara River watershed upstream of the Los Angeles/Ventura County Line and 1.1 percent of the total Santa Clara River watershed.

The Santa Clara River (SCR) watershed drains an area in the Transverse mountain range. The SCR flows generally west from its headwaters near Acton to the Pacific Ocean near the City of Ventura, approximately 40 miles downstream of the NRSP area. The river exhibits some perennial flow in its eastern-most stretches within the Angeles National Forest then flows intermittently westward within Los Angeles County. The principal tributaries of the upper river watershed in Los Angeles County are Castaic Creek, Bouquet Canyon Creek, San Francisquito Creek, and the South Fork of the Santa Clara River. Placerita Creek is a large tributary draining the western-most end of the San Gabriel Mountains; it joins the South Fork, which flows directly

into the Santa Clara River. Castaic Creek is a south-trending creek that confluences with the Santa Clara River within the NRSP area. Castaic Lake is a state-owned reservoir located on Castaic Creek. San Francisquito Canyon Creek is an intermittent stream in the watershed adjacent to Bouquet Canyon to the southeast.

The principal sources of water contributing to the base flow of the Santa Clara River are: (a) groundwater from the Alluvial aquifer basin in Los Angeles County, which seeps into the riverbed near, and downstream of, Round Mountain (located just below the mouth of San Francisquito Creek); (b) tertiary-treated water discharged to the Santa Clara River from two existing Los Angeles County Sanitation District WRPs -- the Saugus WRP, located near Bouquet Canyon Road bridge and the Valencia WRP, located immediately downstream of I-5; and (c) in some years, DWR-released flood flows from Castaic Lake into Castaic Creek during winter and spring months. The Saugus Water Reclamation Plant, located near Bouquet Canyon Road bridge, has a permitted dry weather average design capacity of 6.5 million gallons per day (mgd) creating surface flows from the outfall to near Interstate 5. The Valencia Water Reclamation Plant outfall is located immediately downstream of the Interstate 5 bridge and has a permitted dry weather average design capacity of 21.6 mgd, creating surface flows extending through the Project area and into the far eastern portion of Ventura County.

#### **1.2 Project Permits and Approval Requirements**

The NRSP contains the land use plan, development regulations, design guidelines, and implementation program for the long-term development of the Newhall Ranch. The WDR includes specific requirements for storm drain and receiving water monitoring. The Newhall Ranch Water Reclamation Plant (WRP), operated by the Newhall Ranch Sanitation District, will be located near the Ventura/Los Angeles County line and is currently regulated under an individual National Pollutant Discharge Elimination System (NPDES) Permit (Order No. R4-2007-0046). The Newhall Ranch WRP NPDES Permit includes discharge and receiving water monitoring requirements specific to discharges of treated effluent from the WRP. The use of reclaimed water within the villages will be regulated by a future permit to be issued under the Water Recycling Requirements (WRR) program. This monitoring plan addresses only the requirements of the NRSP and the WDR. The Newhall Ranch WRP NPDES Permit requirements are provided for reference only, although neither the WDR nor the NRSP sites are required to meet these requirements.

#### **1.2.1 WDR Requirements**

The WDR includes the following specific requirements for storm drain and receiving water monitoring (pages 57-58):

**G.3.36** Representative and rotating outfall-based water quality monitoring shall be conducted to determine impacts of the NRSP over time. Water samples will be taken at least four (4) times a year to include at least twice in wet weather and once in dry weather. Parameters to be considered will include at a minimum:

- pH
- Temperature
- Dissolved oxygen
- Turbidity
- Total suspended solids (TSS)
- E. coli
- Chloride
- Ammonia as nitrogen (NH3-N)
- Nitrate as nitrogen (NO3-N)

- Nitrite as nitrogen (NO2-N)
- Total phosphorus
- Metals
- Organochlorine pesticides
- Organophosphorus pesticides
- Pyrethroid pesticides
- PAHs
- Volatile organics

Newhall Land will develop a Storm Drain monitoring plan and submit the plan to the Executive Officer for approval within 6 months of the effective date of this Order. The Storm Drain Monitoring plan will include sampling the first storm of the wet season that produces at least 0.25" of rain for the seasonal first flush.

Benthic macroinvertebrates will be assessed in the receiving waters. Newhall Land will develop a plan for the assessment of benthic macroinvertebrates and submit the plan to the Executive Officer for approval within 6 months of the effective date of this Order.

Analyses must be performed using approved USEPA methods, where applicable, or a method approved by the Executive Officer. Newhall Land shall submit results of the analyses to the Regional Board with annual reporting including comparisons to applicable water quality standards and to the estimated annual pollutant concentrations for stormwater discharges presented in the RMDP final [Environmental Impact Report] EIR. A map or drawing indicating the locations of sampling points shall be included with each submittal.

If data demonstrate exceedances of water quality standards or significant pollutant contributions contributing to exceedances of water quality standards in the receiving waters, increased monitoring may be required and the WDR may be revised to require additional or modified BMPs or effluent benchmarks or limits.

#### 1.2.2 NRSP Condition of Approval

The water quality monitoring required by the NRSP CoA is intended to assess the water quality impacts of the overall development, integrating the impacts assessed per the NPDES WRP,

WRR, and WDR programs in totality. The following excerpt from the NRSP CoA (page 4.0-101) describes the required monitoring:

**4.11-21.** The applicant, in coordination with [Los Angeles Regional Water Quality Control Board] LARWQCB staff, shall select a representative location upstream and downstream of the Newhall Ranch Specific Plan and sample surface and groundwater quality. Sampling from these two locations would begin upon approval of the first subdivision map and be provided annually to the LARWQCB and County [Los Angeles County Department of Public Works] for the purpose of monitoring water quality impacts of the Specific Plan over time. If the sampling data result in the identification of significant new or additional water quality impacts resulting from the Specific Plan, which were not previously known or identified, additional mitigation shall be required at the subdivision map level.

#### 1.2.3 Los Angeles County MS4 Permit

The 2012 Waste Discharge Requirements for Municipal Separate Storm Sewer System (MS4) Discharges within the Coastal Watersheds of Los Angeles County (Order No. R4-2012-0175, NPDES Permit No. CAS004001) ("LA MS4 Permit") includes monitoring and reporting requirements for both outfall and receiving water monitoring. These requirements may be incorporated into a future MS4-led Watershed Management Plan for the Upper Santa Clara River; if so, this NRSP Monitoring Plan may be integrated into the Coordinated Integrated Monitoring Program for the watershed.

This Monitoring Plan is consistent with the LA MS4 Permit monitoring requirements in the following ways:

- Monitoring Sites and Frequencies. The LA MS4 Permit requires receiving water mass emission site monitoring three times per year during wet weather (toxicity just two times per year) and two times per year during dry weather, stormwater outfall monitoring three times per year for all parameters except toxicity, and non-stormwater outfall monitoring as specified in the outfall monitoring plan under development by the MS4 agencies. This NRSP Monitoring Plan includes similar requirements for both receiving water monitoring and outfall monitoring. Receiving waters will be monitored during wet weather three times per year, selected representative outfalls will be monitored three times per year during wet weather and one time per year during dry weather, and the NRWRP NPDES Permit already requires receiving water monitoring two times per year during dry weather. The NRSP Monitoring Plan also includes annual bioassessments and semiannual groundwater sampling.
- Water Quality Parameters. The LA MS4 Permit requires monitoring for a variety of parameters including general constituents and other more specific pollutants (e.g.,

bacteria, total and dissolved metals, PCBs, organophosphorous pesticides, etc.). The constituent list for the NRSP Monitoring Plan is similarly comprehensive, and was developed based on the WDR and NRSP CoA requirements. The complete list of parameters to be monitored is included in Appendix B.

• Numeric Water Quality Benchmarks. The LA MS4 Permit specifies that mass emission site monitoring results be compared to numeric receiving water limitations (Basin Plan objectives and CTR criteria), stormwater outfall results compared to Municipal Action Levels, and non-stormwater outfall results compared to Non-stormwater Action Levels. The WDR monitoring results will similarly be compared to the applicable water quality standards (Basin Plan objectives and CTR criteria), as well as the estimated annual pollutant concentrations from stormwater discharges presented in the RMDP EIR and estimated outfall-specific annual pollutant concentrations based on modeled report-specific hydrologic year. These objectives are included as Appendix C.

#### 1.3 Organization

Section 2 describes the monitoring plan design, including monitoring objectives, parameters, sites, frequency, and schedule. Section 3 provides the sampling and analysis plan which describes the sampling and analysis procedures to be used, including field measurement methods and equipment, methods used for sample collection for laboratory analysis, and laboratory analytical methods. Section 4 summarizes the reporting procedures, including reporting frequency and contents, data analysis procedures, and potential mitigation actions.

#### 2 MONITORING PLAN DESIGN

#### 2.1 Monitoring Objectives

The primary objectives of this Monitoring Plan are to (1) monitor potential instream surface water and groundwater quality impacts of the NRSP over time from sources including the WRP, reclaimed water use, and stormwater discharges, and (2) monitor the water quality of storm drain outfall discharges. The first objective will be met through the routine sampling and analysis of instream surface water and groundwater quality parameters at selected sites to capture spatial (upgradient and downgradient of the NRSP) and temporal (annual, seasonal, rainfall-driven) changes in water quality. The second objective will be achieved by the routine sampling and analysis of instream surface water quality parameters at rotating storm drain outfalls within the NRSP. Outfall monitoring results will be compared to water quality objectives as described in Section 4.

#### 2.2 Monitoring Parameters

Parameters selected for monitoring include general chemistry, salts, nutrients, indicator bacteria, metals, herbicides/pesticides, polycyclic aromatic hydrocarbons (PAHs), volatile organic compounds (VOCs), and biological and habitat parameters. Monitoring parameters were selected with consideration of a number of factors including the minimum requirements of the WDR, baseline monitoring results from existing monitoring sites, knowledge of common pollutants found in urban runoff and municipal wastewater, and water quality objectives for Reach 5 of the Santa Clara River (including Basin Plan Objectives and California Toxics Rule Criteria).

SCR Reach 5 was identified as an impaired water body on California's 2002, 2006, and 2008/2010 303(d) lists. The most recent 2008/2010 303(d) Category 5 list (which records river segments where standards are not met and a Total Maximum Daily Load [TMDL] is required, but not yet completed, for at least one of the pollutants being listed for this segment) lists iron, coliform bacteria, and chloride as impairments in SCR Reach 5. TMDLs are currently in effect for nutrients (2004), chloride (2005), and coliform bacteria (2012). A TMDL for iron is required but has not yet been developed. All 303(d) listed pollutants for Reach 5, shown in Table 1, are included in the analytical suite. Monitoring parameters and analysis methods are provided in Appendix B.

Pollutants	303(d) Status (2008/2010)
Chloride	TMDL Approved (2005)
Coliform bacteria	TMDL Approved (2012)
Iron	TMDL Required
Nutrients	TMDL Approved (2004)

 Table 1: 303(d) Impairments for Santa Clara River Reach 5

Field measurements will include temperature, pH, turbidity, DO, total dissolved solids (TDS) and specific conductance. In addition, depth to groundwater will be conducted for groundwater only. Instream surface water, storm drain outfalls, and groundwater will also be monitored for general chemistry constituents, nutrients, and salts. General chemistry and inorganic compound monitoring parameters are shown in Table B1-5 in Appendix B.

Monitoring will occur for both total and dissolved concentrations for those metals shown in Table B1-6 in Appendix B. Monitoring will also be included for pesticides and herbicides. The organochlorine, organophosphorus, and pyrethroid pesticides included in the monitoring suite are listed in Tables B1-8, B1-9, and B1-10, respectively. Instream surface water and groundwater monitoring will occur for the chlorinated herbicides shown in Table B1-7 in Appendix B.

Semi-volatile organic compounds (SVOCs) in the analytical suite include PAHs and N-Nitrosodimethylamine (NDMA), as these compounds are occasionally detected in urban runoff and municipal wastewater. They have also been detected above water quality objectives during baseline monitoring conducted for the Newhall Ranch WRP NPDES Permit. These parameters will be monitored at the groundwater, storm drain outfalls and instream sites (see Table B1-11 in Appendix B).

Volatile organic compounds (VOCs) will be monitored at the groundwater, selected representative storm drain outfall, and instream sites. As required by the WDR, VOCs to be included in the analyses are listed in Table B1-12 in Appendix B.

Monitoring will also include biological parameters, including bacteria, toxicity, physical habitat, and benthic macroinvertebrate (BMI) indices. NRSP instream surface water and storm drain outfalls will be monitored for E. coli as required by the WDR. Instream surface water monitoring will also include toxicity (chronic and acute) to determine biological effects of water quality. An annual biological assessment, including a physical habitat survey and sampling of BMI community structure, will quantitatively measure and track over time the physical condition and monitor the integrity of instream biological communities upstream and downstream of the NRSP. Monitoring parameters for toxicity, bacteria, and biological assessment are found in Tables B1-2, B1-3, and B1-4, respectively.

This analytical suite is intended to be adaptive, in that pollutants can be added or removed as pollutants of concern change for regulatory or risk-based reasons. An assessment of monitoring parameters will be completed in each annual monitoring report. If it is demonstrated through monitoring that specific pollutants are not of concern because the analytes have not been measured above water quality objectives or have not been detected throughout the prior year's monitoring events (e.g., four of four events sampled), then these analytes may be removed from the analytical suite for the applicable monitoring location.

#### 2.3 Monitoring Sites

The selection of instream surface water and groundwater monitoring sites to satisfy both the NRSP CoA and the WDR requirements are discussed below. These monitoring sites are illustrated in Figure 3.

#### 2.3.1 Monitoring Sites to Satisfy Conditions of Approval

Instream surface water and groundwater monitoring sites selected to satisfy the NRSP CoA were located based on representativeness, accessibility, and the ability to meet Monitoring Plan objectives. The location name, type, and description of each selected NRSP CoA water quality monitoring location are listed in Table 2 and illustrated in Figure 3.

Location Name	Location Type	Location Description
NRSP-SW1	Surface Water	Santa Clara River above Mission Village (upstream) Approximately 34°25'59" North and 118°36'05" West
NRSP-SW2	Surface Water	Santa Clara River at Salt Creek Crossing (downstream) Approximately 34°24'07" North and 118°42'01" West
NRSP-GW1	Groundwater	Well near Hwy 126 and Interstate 5 Interchange (upgradient) Approximately 34°26'09" North and 118°36'06" West
NRSP-GW2	Groundwater	Well near Hwy 126 and LA/Ventura County line (downgradient) Approximately 34°24'20" North and 118°41'27" West

 Table 2: NRSP CoA Water Quality Monitoring Sites

Instream surface water monitoring location NRSP-SW1 is located upstream of the most upstream NRSP village, Mission Village, and is considered representative of Santa Clara River water quality upstream of the entire NRSP area. Instream surface water monitoring location NRSP-SW2 is located at the Salt Creek crossing east of the discontinued Blue Cut gaging station; this location is considered representative of water quality downgradient of the entire NRSP area. In combination, these two instream surface water sites are anticipated to characterize changes in water quality as a result of development within the NRSP boundary. Both monitoring sites are located in relatively straight reaches with relatively uniform flow, maximizing the likelihood that constituents will be well-mixed within the cross-section and that the samples will be representative.

Groundwater monitoring sites were selected by reviewing and prioritizing existing monitoring well locations based on ability to meet Monitoring Plan objectives. The criteria for groundwater well selection included:

- 1. Wells are located in areas upgradient and downgradient of the NRSP boundaries;
- 2. Wells are installed within the shallow alluvial aquifer that is in hydrologic connection with the Santa Clara River surface and groundwater system;
- 3. Wells are constructed such that the well screen is appropriately installed in the shallow groundwater in both the up- and downgradient sites; and
- 4. Water quality and water levels in the wells are representative of the shallow alluvial aquifer.

The upgradient groundwater well NRSP-GW1 was permitted and install in 2011 to fulfill the selection criteria above. The downgradient well, to be designated NRSP-GW2, will be installed near the County Line on the south bank of the Santa Clara River. If this location turns out to be unsuitable due to shallow bedrock (a common difficulty at the downgradient location), the contingency will be to collect alluvial groundwater from a temporary monitoring well installed within the riverbed itself along the Salt Creek crossing just downstream of the County line. Both of these monitoring wells will be monitored for water quality parameters.

As releases from Castaic Lake, streamflow from other tributaries, rising groundwater, and development outside of the NRSP area will contribute to surface and groundwater between the selected upgradient and downgradient monitoring sites, it is impossible to completely isolate the NRSP area for water quality impact monitoring. Given these constraints, the selected monitoring sites provide the best possible sites for characterizing water quality changes in the vicinity of the project for continued evaluation in the future.

#### 2.3.2 Monitoring Sites to Satisfy WDR Requirements

To meet the WDR requirements, one representative outfall per village will be selected. The selected outfall will be representative of the overall mixture of land uses for the village. The first outfall location constructed and selected for monitoring will be named NRWDR-SW1, the second will be named NRWDR-SW2, and so on, until each of the villages have a representative monitoring location selected. Monitoring will be conducted at one of these representative outfalls per year (as representative outfalls have been constructed) and rotated on an annual basis. A map will be included with each annual report identifying the monitoring location selected for monitoring during that year.

#### 2.4 Monitoring Frequency

Monitoring objectives also require that monitoring capture the variability of water quality parameters over time. Changes in water quality resulting from new development may occur due to stormwater runoff, dry weather runoff, infiltration of urban runoff or recycled water, or discharge of treated effluent. For this reason, the Monitoring Plan design includes instream surface water and outfall monitoring during wet and dry weather conditions and routine groundwater monitoring. Monitoring frequency for each sampling type is summarized in Table 3.

Program	Туре	Timing	Samples/Year/Location	Number of Sites
NRSP CoA	Surface water	Wet season	3	2
NRSP CoA	Groundwater	Spring and fall	2	2
WDR	Storm drain outfall	Wet season	3	1
WDR	Storm drain outfall	Dry season	1	1
WDR	Biological assessment	Summer	1	2

 Table 3: Monitoring Frequency Requirements

Three (3) wet weather events will be monitored per hydrologic year (October 1 through September 30) at the NRSP-SW and NRWDR-SW monitoring sites. Depending on site conditions, access issues, and storm timing, the same three events (or days within an event) will not necessarily be sampled for both the NRSP-SW and NRWDR-SW sites each year. Storms selected for NRWDR-SW wet weather monitoring will include the seasonal first-flush (first storm of the wet season producing at least 0.25 inches of precipitation in 24 hours, at a seventy percent probability of rainfall at least 24 hours prior to the event start time) and two additional storms each season. NRSP-SW monitoring is not required to sample the first flush event. In order to meet the requirements for monitoring, additional storms must be forecasted to be equal to or greater than the seasonal first-flush depth and separated by least 72 hours of dry weather (less than 0.1 inches of measured precipitation each day). Smaller storms are not likely to create enough runoff to meet Monitoring Plan objectives. The NRSP area receives, on average, eleven (11) events per year that meet these requirements.

As required by the WDR, dry weather monitoring of the NRWDR-SW storm drain outfall sites will be conducted one time per year during the dry season (May 1 – September 30). At least two weeks of dry weather (less than 0.1 inches of measured precipitation each day for 14 calendar days), shall precede all dry weather monitoring events. If no runoff is observed during the dry weather event, the report shall indicate as such (see Section 3.2.3 for the definition of "no runoff"). Additionally, the Newhall Ranch Sanitation District currently conducts dry weather instream sampling near the NRSP-SW downstream location for Newhall Ranch WRP NPDES monitoring. This sampling program will continue as long as the Newhall Ranch WRP NPDES Permit remains active.

Groundwater monitoring will be conducted semiannually at each of the two NRSP-GW sites in order to capture seasonal variation in pollutant concentrations in alluvial groundwater flows and any water quality changes due to the use of reclaimed water in the NRSP area. Groundwater

sampling will be conducted at the end of the wet season (approximately April) and the end of the dry season (approximately September). At least 72 hours of dry weather (less than 0.1 inches of measured precipitation each day), shall precede all groundwater sampling events.

Additionally, an instream surface water biological assessment, including an instream physical habitat survey and monitoring of benthic macroinvertebrates (BMI), will be performed once annually in early summer at the upstream NRSP-SW1 location. The downstream NRSP-SW2 bioassessment will be represented by the survey conducted at the nearby Newhall Ranch WRP NPDES monitoring site RSW-002D, as shown on Figure 3. In coordination with the Santa Clarita Valley Sanitation District, the Newhall Ranch Sanitation District will continue to conduct bioassessments on an annual basis per Newhall Ranch WRP NPDES Permit requirements. If an outfall is constructed downstream of site RSW-002D, then the biological assessment would be performed at the downstream NRSP-SW2 location.

#### 2.5 Monitoring Schedule

The NRSP CoA monitoring will begin upon approval of this Monitoring Plan. WDR monitoring will begin upon release of the Waste Discharger Identification (WDID) number for the first outfall's parcel, issued under the Construction General NPDES Permit coverage (i.e., when Notice of Termination [NOT] is issued). As discussed in Section 2.2, if it is demonstrated through this monitoring program that specific pollutants are not of concern because the analytes have not been measured above water quality objectives or have not been detected throughout the prior year's monitoring events (e.g., four of four events sampled), then these analytes may be removed from the analytical suite for the applicable monitoring location. Additionally, as the NRSP CoA monitoring program is intended to identify mitigation measures at the subdivision map level, the NRSP CoA monitoring will cease after the recordation of the final tract map. WDR monitoring will cease upon the transition of ownership of the outfall monitoring equipment, along with all the necessary property rights to operate and maintain the equipment, from Newhall Land to Los Angeles County, at which time the outfall monitoring sites will be considered part of the County's MS4 and will be monitored in accordance with the MS4 Permit Monitoring and Reporting Requirements.

#### 3 SAMPLING AND ANALYSIS PLAN

This Sampling and Analysis Plan (SAP), describes the sampling and analysis procedures, including field measurement methods and equipment, methods for sample collection, and laboratory analytical methods. This SAP along with the QAPP (Appendix A) is intended to ensure that the objectives of the Monitoring Plan are met to scientifically defensible standards.

#### 3.1 Wet Weather Surface Water Sampling

#### 3.1.1 Sampling Frequency

Wet weather instream surface water sampling will be conducted during three storm events each year at the NRSP-SW and NRWDR-SW sites. The storms selected for sampling will include the seasonal first-flush (at NRWDR-SW only) and two additional storms, when feasible and practical<sup>a</sup>, as described previously in Section 2.4. Sampling shall only occur during normal working hours.

#### **3.1.2** Wet Weather Preparation

In preparation for wet weather sampling, materials will be collected and stored prior to the beginning of a new hydrologic year (October 1). This includes sample bottles, compositing bottles, coolers, and field measurement instruments. Automated samplers will be installed (or inspected) and programed at the NRWDR-SW monitoring site. The magnitude of storm events will be predicted using weather forecasts and by watching real-time rainfall at telemetered gages. For storm prediction, the National Weather Service provides a quantitative precipitation forecast (QPF) for sites in Ventura and Los Angeles Counties. Additionally, satellite telemetered 5-minute rainfall data is available for Ventura County's Piru-L.A./Ventura County Line precipitation gage (VCWPD Gage No. 235A, located at 34°23'58.7" N, 118°42'14.5" W) approximately 0.25 mi southwest of the NRSP. If a storm is expected to meet the criteria for sampling, field staff will prepare and mobilize to collect instream surface water samples.

#### 3.1.3 Field Measurement Methods and Equipment

The downstream monitoring location (NRSP-SW2) will always be sampled prior to the upstream monitoring location (NRSP-SW1) to avoid sampling induced contamination or debris from impacting the downstream samples. Due to safety concerns relating to wading in high flows, manual flow measurements will not be made during wet weather instream surface water

<sup>&</sup>lt;sup>a</sup> Storm event timing, duration and intensity are unpredictable and uncontrollable. Therefore circumstances exist where sampling during the first flush event may not be feasible or practical. For example, if a storm event forecasted below the first flush definition, in actuality exceeds the requirement.

sampling. Instead, flow measurements from U.S. Geological Survey (USGS) Stream Gage No. 11109000, Santa Clara River near Piru (34°34'13" N, 118°44'18" W), approximately 2.5 mi downstream of the site, will be used for reporting. The automated composite samplers will be equipped with a flow gauge depth-velocity sensor to continuously measure and record flow rates at the NRWDR-SW outfall monitoring sites.

Field measurements will be taken in situ when it is safe to wade, or using a pole sampler. When using a pole sampler, two samples will be collected in glass containers and a multi-parameter water quality meter with probe will be used to measure and record temperature, pH, DO, specific conductance, and turbidity. The two samples must meet the precision quality control measures in Table 14-1 of the QAPP (Appendix A). If the two measurements do not meet the precision criteria, three additional replicates will be taken and the median of the five measurements reported. Field measurements will be made, at a minimum, on the final composite sample. Aesthetic data will also be observed and recorded. Observations will include color, odor, floating particulates or debris, foam, oil/grease, and/or the presence of breeding mosquitoes or other pests. Example field collection data sheets for both instream surface water and outfall sampling are provided in Appendix D. The invasive species protection protocols contained in the Hazard Analysis and Critical Control Points (HACCP) for the Prevention & Control of Aquatic Nuisance Species, such as allowing equipment to dry for at least 48 hours before use in the Santa Clara River after use in another waterbody, shall be implemented to ensure that all sampling equipment is free from aquatic nuisance species prior to sampling events (Newhall, 2012).

#### 3.1.4 Sample Collection Methods for Laboratory Analysis

Instream surface water samples will be analyzed for the parameters shown in the tables in Appendix B. Manual composite samples shall be collected at the instream (NRSP-SW) sites using laboratory-approved containers, depending on analyte and analytical method requirements of the laboratory. Flow-weighted or time-proportionate composite samples shall be collected at the storm drain outfall (NRWDR-SW) sites. Single grab samples will be collected for parameters not suitable for composite sampling (e.g., E. coli) in accordance with the Standard Operating Procedure (SOP) for Conducting Field Measurements and Field Collections of Water and Bed Sediment Samples in the Surface Water Ambient Monitoring Program (SWAMP) (CDFG, 2007). Parameters not suitable for composite sampling are identified in Table B2-1 of Appendix B. Appropriate sample bottles, with preservatives as necessary, will be provided by the contracted laboratory. Table B2-1 in Appendix B includes laboratory sampling guidelines, including container type, sample volume, preservative requirements, and holding time until analysis.

Quality assurance methods, such as collection of field duplicates and trip blanks, are discussed in the Monitoring Plan QAPP in Appendix A. Appropriate precautions will be taken to prevent contamination of samples, including Clean Hands/Dirty Hands sampling procedures described in the USGS Field Manual for the Collection of Water Quality Data (USGS, 2006). Relevant sections of all SOPs used in this Monitoring Plan are included in Appendix E.

Flow-weighted or time-proportionate composite sampling will occur according to the Caltrans Guidance Manual: Stormwater Monitoring Protocols (Caltrans, 2000). Manual time-proportionate composite samples will be collected at the instream surface water (NRSP-SW) sites. Table 4 recommends the minimum number of sub-samples collected over a 2 hour period, along with the time between aliquots. Each sub-sample will contain equal volume, so that the combined volume of all sub-samples is equal to the volume required by the lab to perform the associated analysis. The composite sample will be split by the analyzing laboratory.

Total Forecasted Event Precipitation	Minimum Number of Sub-Samples	Time between Sub-Samples
0.25 – 0.5"	8	15 minutes
0.5 - 1"	10	12 minutes
>1"	12	10 minutes

Table 4: Manual 7	<b>Fime-Proportionate</b>	Composite San	nnling (NRS	P-SW sites)
Table 4. Manual	i mit i ropor nonate	Composite Dan		

Automated flow-weighted or time-proportionate composite samples will be collected at the storm drain outfall (NRWDR-SW) sites. Table 5 provides recommendations for the minimum number of sub-samples collected during the storm, as well as the percent of the storm volume that should be sampled, for automated flow-weighted composite samples. As a result of holding time requirements, automated sampling will continue for a maximum of 24 hours, regardless of the percent of the storm captured. The flow rate or depth required to trigger the automated sampler for flow-weighted sampling will be calculated based on the event-specific precipitation forecasted and the geometry of the outfall infrastructure and drainage area. Prior to each storm event, the automated samplers will be programmed to pull the first sample once the calculated sub-sample volume has been measured. Subsequent sub-samples will be of equal volume, but will be collected more frequently as the storm intensity and flows increase (this results in a flow-weighted composite sample). Consistent with the MS4 Permit, automated composite sampling may also be conducted by collecting three sub-samples each hour to capture 100% of the storm or for 24 hours, whichever is less. The composite sample will be split by the analyzing laboratory.

Total Forecasted Event Precipitation	Minimum Number of Sub-Samples	Percent Capture Requirement
0.25 – 0.5"	8	80%
0.5 – 1"	10	80%
>1"	12	75%

 Table 5: Automated Flow-Weighted Composite Sampling (NRWDR-SW sites)

Due to safety concerns of wading in storm flows, instream samples will be taken from a bridge or bank station using a pole sampler when flows are high or there is substantial floating debris.

Samples may be taken by wading if conditions allow. Safety considerations are discussed below in Section 3.1.6.

Samples will be transported to the laboratory in an ice-filled cooler. Chain of custody forms will be filled out onsite and transferred to the laboratory upon sample exchange. These procedures are discussed in Section A12 of the QAPP in Appendix A, and an example chain of custody form is provided in Appendix D.

#### 3.1.5 Laboratory Analytical Methods

Analytical methods were selected from USEPA-approved methods to meet Monitoring Plan requirements, including ensuring reporting accuracy and method detection limits. These methods will be used in analysis by a USEPA-approved laboratory to be contracted before the monitoring event. Analytical methods to be used for each measured water quality parameter are shown in Appendix B Tables B1-2 through B1-12.

Whole effluent toxicity testing (NRSP-SW sites only) will be conducted by an EPA-approved bioassay laboratory. Fathead Minnow *Pimephales promelas* will be used to evaluate acute toxicity using EPA Test Method 2000.0 (USEPA, 2002a). Chronic toxicity will be assayed with *P. promelas* (larvae survival and growth) using EPA Test Method 1000.0 and *Ceriodaphnia dubia* (survival and reproduction) using EPA Test Method 1002.0 (USEPA, 2002b). Although wet-weather water quality and toxicity stressors may vary, these species were found to be most sensitive during NPDES monitoring for the Newhall Ranch WRP.

To provide consistency and comparability between events, only analytical laboratories certified by the State of California will be contracted for sampling events. Analytical data quality objectives, including accuracy, precision, percent recovery, target reporting limits, and completeness are included in Section 7 of the QAPP (Appendix A).

#### **3.1.6 Safety Considerations**

Safety is an important consideration during all field activities, but particularly during wet weather instream surface water sampling events. A site-specific Health and Safety Plan (HASP) will be prepared prior to the start of sampling and will be made available in the field (the contracted laboratory for instream biological assessment will have a separate HASP for their work). The Santa Clara River is historically subject to flash flooding, rapidly changing stage, floating debris, and an unstable channel bottom, which can quickly create safety concerns during storm event monitoring. The drainage area of this portion of the Santa Clara River is approximately 600 square miles, and peak discharge during large storms can exceed 10,000 cfs during wet years (e.g., 1998, 2005, and 2006). During average rainfall years, the annual peak typically exceeds 1,000 cfs within SCR Reach 5 (USGS, 2011).

During wet weather, field sampling personnel shall be familiar with swift water safety and stream rescue procedures, and U.S. Coast Guard-approved personal floatation devices and a rescue throw bag will be available in accordance with OSHA Standards. When stream conditions such as high velocities or floating debris make wading unsafe, water quality samples will be taken from the bank or bridge using a pole sampler. A common rule of thumb is to only wade when the product of depth in feet and velocity in feet per second is less than ten (10). If rapidly rising stage, large debris, or hazardous weather conditions create a situation in which the pole sampler cannot be used, it shall be at the discretion of the field team leader to cancel storm sampling.

#### **3.2** Dry Weather Storm Drain Outfall Sampling

#### 3.2.1 Sampling Frequency

Dry weather outfall sampling will be conducted annually at the same selected representative, rotating storm drain outfall monitored during wet weather. Sampling will occur during the dry season and will be preceded by at least two weeks of dry weather (less than 0.1 inches of measured precipitation each day).

#### 3.2.2 Field Measurement Methods and Equipment

Field measurements collected at storm drain outfalls shall include water temperature, pH, DO, specific conductance, and turbidity. Aesthetic observations including color, odor, floating particulates or debris, foam, oil/grease, and/or the presence of breeding mosquitoes or other pests will also be observed and recorded. An example field collection data sheet for storm drain outfall sampling is provided in Appendix D. The invasive species protection protocols contained in the Hazard Analysis and Critical Control Points (HACCP) for the Prevention & Control of Aquatic Nuisance Species, such as allowing equipment to dry for at least 48 hours before use in the Santa Clara River after use in another waterbody, shall be implemented to ensure that all sampling equipment is free from aquatic nuisance species prior to sampling events (Newhall, 2012).

#### 3.2.3 Sample Collection Methods for Laboratory Analysis

Dry weather samples will be analyzed for the parameters shown in the tables in Appendix B. Samples shall be collected as grab samples and composite samples using laboratory-approved containers, depending on analyte and analytical method requirements of the laboratory. Appropriate sample bottles, with preservatives as necessary, will be provided by the contracted laboratory. Table B2-1 in Appendix B includes laboratory sampling guidelines, including container type, sample volume, preservative requirements, and holding time until analysis.

Single grab samples will be collected for parameters not suitable for composite sampling (e.g., E. coli) in accordance with the Caltrans Guidance Manual: Stormwater Monitoring Protocols (Caltrans, 2000). Quality assurance methods, such as collection of field duplicates and trip

blanks, are discussed in the Monitoring Plan QAPP in Appendix A. Appropriate precautions will be taken to prevent contamination of samples, including Clean Hands/Dirty Hands sampling procedures described in the USGS Field Manual for the Collection of Water Quality Data (USGS, 2006).

A minimum of eight (8) equal volume aliquots will be taken over a 2 hour period, so that the total volume of all aliquots is equal to the volume required by the lab to perform the associated analysis. The composite sample will be split by the analyzing laboratory. If the flow levels are too low for collection by either the automatic samplers or manual bottle collection without contacting the bottle lip to the submerged channel or pipe surface, then zero discharge will be reported for the monitoring event and no samples will be analyzed.

Samples will be transported to the laboratory in an ice-filled cooler. Chain of custody forms will be filled out onsite and transferred to the laboratory upon sample exchange. These procedures are discussed in Section A12 of the QAPP in Appendix A, and an example chain of custody form is provided in Appendix D.

#### 3.2.4 Laboratory Analytical Methods

Analytical methods were selected from USEPA-approved methods to meet Monitoring Plan requirements, including ensuring reporting accuracy and method detection limits. These methods will be used in analysis by a USEPA-approved laboratory to be contracted before the first monitoring event. Analytical methods to be used for each measured water quality parameter are shown in Appendix B Tables B1-2 through B1-12.

To provide consistency and comparability between events, only analytical laboratories certified by the State of California will be contracted for sampling events. Analytical data quality objectives, including accuracy, precision, percent recovery, target reporting limits, and completeness are included in Section 7 of the QAPP (Appendix A).

#### **3.3** Groundwater Sampling

#### 3.3.1 Sampling Frequency

Groundwater sampling will be conducted semiannually in approximately April (end of wet season) and September (end of dry season) at NRSP-GW1 and NRSP-GW2. Groundwater quality parameters to be sampled in the field include depth to groundwater, temperature, pH, specific conductance, and TDS. Samples will also be collected for laboratory analysis.

#### **3.3.2** Field Measurement Methods and Equipment

Field protocols will be followed in order to meet data quality requirements of the Monitoring Plan, described in the QAPP (Appendix A). Equipment for field analysis includes an electronic

water level indicator, an electric pump, and multi-parameter water quality meter. Methods will follow the SOPs from USGS field methods for field-measured groundwater quality parameters (Wilde, 2008) and USEPA for low-flow purging (if necessary) (see Appendix E). A sample field data collection form is included in Appendix D.

Field procedures include water level measurement (distance to water surface from top of well), purging of the well to remove standing water and promote representative alluvial groundwater to enter the well (minimum of 3 well volumes), and collection of samples for field analysis. If available water is insufficient for a standard purge, a purge of one well volume and water for field rinsing the sampler and tubing will be utilized. In cases where purging one well volume still removes too much water, low flow sampling will be utilized (USEPA, 2010). During purging, field measurements are to be recorded at regular time intervals approximately every 3-5 minutes until results for the previous 4 measurements stabilize within the criteria in Table 6. Median value from the stabilized measurements will be reported. Purged water will be discharged far enough from the well so as not to enter or affect water quality in the well or any other surface water bodies, and to prevent muddy and slippery work conditions.

Field Measurement	Stabilization Criteria
pH (standard units)	$\pm 0.2$
Specific Conductivity (µS/cm)	$\pm 5\%$
Temperature (degrees Celsius)	$\pm 0.5$
TDS (mg/L)	$\pm 10\%$

 Table 6: Stabilization Criteria for Field Measurements (Adapted from Wilde, 2008)

#### **3.3.3** Sample Collection Methods for Laboratory Analysis

Samples will be analyzed for the parameters shown in the tables in Appendix B. Samples shall be collected using polyethylene or glass containers, with preservatives as necessary, depending on analyte and method requirements of the laboratory. A list of analyses, container types, preservatives, and holding times is included in Table B3-1 in Appendix B. The contracted laboratory will supply the sample containers for all analyses.

Groundwater sample collection will be done in accordance with USGS field methods for groundwater sampling (USGS, 2006) after well purging and completion of field measurements as described in Section 3.3.2. Groundwater samples will be collected by lowering a disposable Teflon bailer with a check valve into the well and collecting groundwater from the top of the water column. The groundwater in the bailer will be transferred to the laboratory supplied containers. Collected samples will be stored in an ice-filled cooler for transport to the laboratory. Chain of custody forms will be filled out onsite and transferred to the laboratory upon sample exchange.

#### 3.3.4 Laboratory Analytical Methods

USEPA-approved analytical methods were selected to meet the objectives of monitoring, including reporting accuracy and method detection limits. These methods will be used in analysis by a USEPA-approved laboratory to be contracted before the first monitoring event. To provide consistency and comparability between events, only analytical laboratories certified by the State of California will be contracted for sampling events. The tables in Appendix B show the analytical methods to be used for each monitoring parameter. Table B3-1 includes laboratory sampling guidelines, including container type, sample volume, preservative requirements, and holding time until analysis. Analytical data quality objectives, including precision, accuracy, representativeness, comparability, and sensitivity are discussed in Section 6 of the QAPP (Appendix A).

#### 3.4 Instream Biological Assessment

#### 3.4.1 Sampling Frequency

A biological assessment and survey of BMI community assemblage will be conducted annually, in summer, at both NRSP-SW1 and RSW-002D (as part of the nearby Newhall Ranch WRP NPDES monitoring program). If an outfall is constructed downstream of site RSW-002D, then the biological assessment would be performed at the downstream NRSP-SW2 location.

These assessments will provide quantitative metrics to assess and track over time the physical condition of the Santa Clara River and the integrity of the instream biological communities. It is assumed that these recurring instream bioassessments will also satisfy the requirements of the WDR, which specifies that "benthic macroinvertebrates will be assessed in the receiving waters".

#### 3.4.2 Field Measurement Methods and Equipment

Biological assessments will be performed using Surface Water Ambient Monitoring Program (SWAMP) Biological assessment Procedures (SWAMP, 2007). These methods involve surveys of physical habitat, including discharge, channel geometry, bank stability, substrate characteristics, canopy cover, and gradient/sinuosity. BMI assemblage will be sampled and analyzed using the Southern California Index of Biological Integrity (Ode et al., 2005). The contracted bioassay laboratory will provide internal SAPs and SOPs upon request.

#### **4 REPORTING PROCEDURES**

#### 4.1 **Report Frequency and Contents**

Data will be analyzed and reported annually, by April 1<sup>st</sup>, concurrently to the LARWQCB and the Los Angeles County Department of Public Works. The report will describe monitoring efforts over the prior hydrologic year (October 1 – September 30) and data analysis for the NRSP CoA and WDR sites. The analysis for the NRSP CoA sites will include a comparison between upgradient and downgradient monitoring, the statistical significance of those differences, and the possible sources of any water quality changes observed. It is likely that too little data will be available in the first few years to make statistically significant assessments of the results. However, as the datasets grow over several years, confidence in the analysis should increase. The analysis for the WDR sites will include a comparison between wet weather outfall monitoring results and: (1) applicable water quality standards (based on receiving water beneficial uses), (2) estimated annual pollutant concentrations for stormwater discharges presented in the RMDP Final EIS/EIR for the whole NRSP, and (3) new estimated annual pollutant concentrations for stormwater discharges specific to each monitored outfall, based on modeling analyses that are consistent with those used for the RMDP Final EIS/EIR. Dry weather outfall monitoring results will be compared with applicable water quality standards only.

#### 4.2 Data Analysis Approach

#### 4.2.1 NRSP CoA Monitoring Sites

The data analysis for the NRSP-SW and NRSP-GW monitoring sites will follow two approaches and will be evaluated separately for wet and dry weather: (1) differences in water quality and differences in changes in water quality through time will be compared between upstream and downstream monitoring sites, and (2) temporal changes in water quality will be assessed for long-term and seasonal trends at each of the monitoring sites.

First, analysis will determine if there is any statistically significant difference between monitored water quality parameters upgradient and downgradient of the NRSP. Paired non-parametric sign tests will be used to determine the significance of any measured differences using the null hypothesis that there is no difference in upstream and downstream water quality parameter concentrations. As background differences may exist between upstream and downstream water quality, analyses will also take into account the change in water quality relative to a baseline that will be established using existing data from past monitoring (i.e., NPDES monitoring for the Newhall Ranch WRP) and data collected for this Monitoring Plan prior to build-out of the NRSP. Second, water quality data will be plotted on time series charts and analyzed using statistical methods for both long-term and seasonal trends.

To evaluate water quality changes, best professional judgment will be used to consider factors such as natural and anthropogenic sources outside the NRSP (e.g., rising groundwater, releases



from Castaic Lake, streamflow from tributaries that drain areas outside the NRSP, other development, etc.), as well as sources within the NRSP. NRSP sources may include stormwater runoff, dry weather storm drain discharges, infiltration of urban runoff or recycled water, or discharge of treated effluent from the Newhall Ranch WRP. Baseline water quality information will help increase understanding of background differences and natural variability in upgradient and downgradient water quality parameter concentrations.

#### 4.2.2 WDR Storm Drain Outfall Monitoring Sites

The data analysis for the NRWDR-SW sites will require the calculation of the average annual wet weather pollutant concentrations discharged from the storm drain outfalls. This will be calculated for the entire hydrologic year using the following equation:

$$C = \sum (EMC_i x F_i) / \sum F_i$$
 For i = 1 to n

Where,

C = Average annual flow-weighted pollutant concentration

EMC<sub>i</sub> = Event Mean Concentration of pollutant for sampled event i

- $F_i$  = Average flow rate for sampled event i, found by dividing the discharge volume by the discharge duration
- n = Total number of uniquely sampled storm events

The average annual wet-weather flow-weighted pollutant concentration (C) for the NRWDR-SW sites will then be compared to the following, and reported on an annual basis:

- 1) Applicable water quality standards;
- 2) Estimated annual pollutant concentrations for stormwater discharges presented in the RMDP Final EIS/EIR; and
- 3) Estimated outfall-specific annual pollutant concentrations based on the modeled reportspecific hydrologic year.

Dry weather results will be similarly analyzed, but compared only to any applicable water quality standards.

#### 4.3 **Response Actions**

If water quality concerns are identified based on evaluation of sampling data results and it is determined that they are due to sources found within the NRSP, Newhall will propose in the Annual Report to further monitor, evaluate, and confer with RWQCB on a proposed response action.

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Prepared for

Newhall Land 25124 Springfield Court, 3rd Floor Valencia, California 91355-1088

## Appendix A

## **Quality Assurance Project Plan (QAPP)**

for the Newhall Ranch Specific Plan Water Quality Monitoring Plan Conditions of Approval and Clean Water Act Section 401 Water Quality Certification and Waste Discharge Requirements



engineers | scientists | innovators

924 Anacapa Street, Suite 550 Santa Barbara, CA 93101

Project Number LA0170

March 27, 2013



#### 1 APPROVAL SIGNATURES

### Newhall Land (Responsible Organization):

<u>Title:</u> Director Environmental	Name:	Signature:	Date:
Resources	Matt Carpenter		
Geosyntec Consult	ants (Contracted by Newhall La	nd)	
<u>Title:</u>	Name:	Signature:	Date:
Project Manager	Brandon Steets, PE		
QA Officer	Donna Bodine		
Los Angeles Regio	onal Water Quality Control Boar	d (LARWOCB)	
<u>Title:</u>	Name:	Signature:	Date:
Project Manger	LB Nye		
Los Angeles Coun			
T:41	Nama	C:	Deter
<u>11ue:</u>	<u>ivame:</u>	<u>Signature:</u>	Date:
Project Manager	TBD		



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## Geosyntec<sup>></sup>

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#### **3 DISTRIBUTION LIST**

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Title	Name (Affiliation)	Tel. No.	No Copies
Regional Board Project Manager	L.B. Nye (LARWQCB)	(213) 576-6785	Original
Public Works Project Manager	TBD (LACDPW)	TBD	1
Director, Environmental Resources	Matt Carpenter (Newhall Land)	(661) 255-4259	1
Project Manager	Brandon Steets, PE (Geosyntec)	(805) 979-9122	1
Project QA Officer	Donna Bodine (Geosyntec)	(510) 285-2785	1
Laboratory Project Manager	Brandon Gee (Weck Labs)	(626) 336-2139 x133	1


#### 4 **PROJECT/TASK ORGANIZATION**

Implementation of the Newhall Ranch Specific Plan (NRSP) Water Quality Monitoring Plan ("Monitoring Plan, "Project") requires the involvement and cooperation of staff from Newhall Land, Geosyntec Consultants, LARWQCB, and LACDPW. This section describes the roles and responsibilities of key Project personnel.

#### 4.1 Newhall Client Representative

The Client Representative for Newhall Land will be responsible for review and approval of annual monitoring reports completed by Geosyntec and subsequent submittal to LARWQCB and LACDPW. The Client Representative will also be responsible for maintaining contracts that are required for completion of Project tasks and reports, including those with the consultant and the analytical laboratory.

#### 4.2 **Project Manager**

The Project Manager (PM) is responsible for the overall direction of the technical and administrative functions within the program, as well as the day-to-day activities associated with site characterization and data analysis. The PM will be responsible for implementing and modifying all program plans and coordinating and communicating with those involved in the Project. The PM is also responsible for the management of data collection activities and project deliverables, as well as all communication with the Newhall Client representative and the LARWQCB and the LACDPW Project Managers. Although various functions will be performed by other individuals, it is the PM who will ultimately provide signature approval to all Project activities.

#### 4.3 Quality Assurance Officer

The Quality Assurance Officer (QAO) will implement this QAPP, make updates as necessary, and conduct project reviews with respect to quality assurance. The QAO will be responsible for assuring the integrity of the QAPP and coordinating all quality assurance (QA) specific activities. The QAO will (a) check that the appropriate analytical methods and sampling supplies are ordered from the laboratory, (b) be responsible for data validation and advise the PM with respect to data management and statistical evaluation of the data, and (c) be responsible for performance and/or systems audits of the laboratory, should they be required.

#### 4.4 Data Manager

The Data Manager is responsible for all data collection and laboratory coordination activities associated with the project. The Data Manager or her designee will be located at the site during field activities and will coordinate the technical field activities in accordance with approved

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plans, including the Monitoring Plan, QAPP, and Health and Safety Plan (HASP). She is responsible for verifying that the field work, including sampling operations and sampling quality control (QC), is performed within the approved guidelines. The Data Manager is responsible for implementing and maintaining overall operating standards and field QA responsibilities. Such responsibilities will include (a) calibrating and maintaining field instruments appropriately, (b) ensuring that appropriate equipment decontamination is performed, and (c) monitoring compliance with QA/QC sampling requirements (e.g. field replicate collection). She coordinates all safety and technical activities occurring at the site and conducts daily briefing sessions prior to field work. The Data Manager is responsible for communicating bottle orders, data quality, and reporting turnaround time expectations to the lab, and is responsible for leading the annual monitoring reporting and data analysis process.

#### 4.5 Laboratory Project Manager

The laboratory will provide analytical services for the scope of work detailed in the Monitoring Plan. The Laboratory Project Manager will be responsible for managing laboratory work (i.e., data processing and data processing QA), verifying that laboratory QA/QC procedures are maintained, and conducting a technical review of reports. Although various laboratory functions will be performed by different individuals, it is the Laboratory Project Manager who will provide signature approvals to laboratory-generated information and bear laboratory responsibilities.

#### 4.6 LARWQCB and LACDPW Project Managers

The LARWQCB and LACDPW Project Managers will review and approve the annual monitoring reports as specified in the Conditions of Approval for the NRSP and the WDR. Newhall Land will provide annual reports to the LARWQCB and the LACDPW.

Name	Organization	Role	Contact Information
Matt Carpenter	Newhall Land	Client Representative	(661) 255-4259
Brandon Steets, PE	Geosyntec	Project Manager	(805) 979-9122
Donna Bodine	Geosyntec	QA Officer	(510) 285-2785
Megan Otto, PE	Geosyntec	Data Manager	(310) 957-6112
L.B. Nye	LARWQCB	Regional Board Project Manager	(213) 576-6785
TBD	LACDPW	Public Works Project Manager	TBD

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#### 5 **PROBLEM DEFINITION/BACKGROUND**

#### 5.1 **Problem Statement**

The Newhall Ranch Specific Plan (NRSP) involves the development and management of approximately 12,000 acres of property owned by the Newhall Land and Farming Company (Newhall) and currently used for agriculture, grazing, and oil and natural gas extraction. The proposed development will be located in the Santa Clara River Valley between Interstate 5 and the Los Angeles/Ventura County boundary. LARWQCB Reach 5 of the Santa Clara River, previously called Reach 7 by the U.S. Environmental Protection Agency (USEPA), flows through the NRSP, which is approximately 5 miles east-west and 5.5 miles north-south. The conversion of land to residential use has the potential to alter water chemistry in the Santa Clara River and its alluvial groundwater.

Reach 5 was identified as an impaired water body on California's 2002, 2006, 2008, and 2010 303(d) lists. The most recent 2008/2010 303(d) Category 5 list (which records river segments where standards are not met and a Total Maximum Daily Load (TMDL) is required, but not yet completed, for at least one of the pollutants being listed for this segment) shows iron, coliform bacteria, and chloride as impairments in Reach 5. TMDLs are currently effective for nutrients (2004), chloride (2005), and coliform bacteria (2012). As the Santa Clara River has a history of water quality concerns, it will be important to monitor water quality and to identify and track any changes in water quality potentially attributable to NRSP implementation. Monitoring will measure any potential water quality changes that might be caused by NRSP discharges to surface or groundwater, including reclaimed water that is used within the NRSP area, treated effluent from Newhall Ranch Water Reclamation Plant (NRWRP), and stormwater and dry weather runoff from the NRSP area.

#### 5.2 Monitoring Objectives

The primary objectives of the Monitoring Plan are to (1) evaluate instream surface water and groundwater quality changes that could potentially result from development or other activities associated with the NRSP and to monitor any changes over time and (2) satisfy the requirements of the Waste Discharge Requirements (WDR). The first objective will be met through the routine sampling and analysis of instream surface water and groundwater quality parameters at selected locations to capture spatial (upstream/upgradient and downstream/downgradient from the NRSP) and temporal (annual, seasonal, rainfall-driven) changes in pollutant concentrations. The second objective will be achieved by the routine sampling and analysis of instream surface water quality parameters at representative rotating storm drain outfalls in the NRSP area.

Project design, sampling procedures, and laboratory analysis need to provide data of adequate quantity and quality to (1) identify differences in measured water quality parameters upgradient and downgradient of the NRSP, (2) quantify the statistical significance and magnitude of any changes, (3) determine likely sources or causes of any changes, (4) compare measured storm

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drain outfall water quality parameters to any applicable water quality standards, (5) compare to annual pollutant concentrations presented in the RMDP final EIR, (6) compare to outfall specific annual pollutant concentrations modeled based on the prior hydrologic year and (7) decide if additional mitigation or monitoring is necessary. This QAPP establishes procedures so that the Project provides environmental data of known, acceptable, and defensible quality.

#### 5.3 Water Quality or Regulatory Criteria

This Monitoring Plan and QAPP were developed to satisfy the Newhall Ranch Specific Plan Conditions of Approval (NRSP CoA) and Clean Water Act Section 401 Water Quality Certification and Waste Discharge Requirements (Order No. R4-2012-0139) (WDR). The water quality criteria used for the NRSP CoA component of the Project will be no significant decrease in water quality, based on statistical analysis of water quality parameter data upgradient and downgradient of the NRSP area. For the WDR component, results will be compared with the estimated annual pollutant concentrations for stormwater discharges presented in the RMDP final EIR as well as any applicable water quality standards.

#### 6 **PROJECT/TASK DESCRIPTION**

#### 6.1 Work Statement

Representative locations upgradient and downgradient of the NRSP will be monitored for surface water and groundwater quality with the goal of identifying and tracking any changes in water quality created by NRSP implementation. Representative storm drain outfalls (one per village, to be rotated) will be monitored for water quality parameters, as specified in the WDR. The project includes water quality sampling of surface water in the Santa Clara River during storm events, sampling of storm drain discharges during both wet and dry weather, semiannual sampling of alluvial groundwater, and surveys of instream physical and biological habitat. The Monitoring Plan provides the background, methodology, and protocols that will be used so that monitoring is performed in a consistent and scientifically defensible way.

Monitoring data will be analyzed and reported annually each April to the LACDPW, concurrent with submittal to the LARWQCB. The report will describe monitoring efforts and data analysis over the prior hydrologic year (October 1 – September 30). The data analysis for the NRSP CoA locations will include a comparison between upgradient and downgradient monitoring, the statistical significance of those differences, and the possible sources of any water quality changes observed. The analysis for the WDR locations will include a comparison to water quality standards, to the annual pollutant concentrations presented in the RMDP final EIR, and to outfall specific annual pollutant concentrations modeled based on the prior hydrologic year.

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#### 6.2 Constituents to be Monitored and Measurement Techniques

Water quality parameters to be analyzed, measurement techniques, and analytical methods are discussed in detail in the Monitoring Plan. The specific analytical methods are listed in the tables in Appendix B.

#### 6.3 **Project Schedule**

The NRSP CoA monitoring will begin upon approval of this Monitoring Plan. WDR monitoring will begin upon release of the Waste Discharger Identification (WDID) number for the first outfall's parcel, issued under the Construction General NPDES Permit coverage (i.e., when Notice of Termination [NOT] is issued). As the CoA/NRSP monitoring program is intended to identify mitigation measures at the subdivision map level, the NRSP CoA monitoring will cease after the recordation of the final tract map. WDR monitoring will cease upon the transition of ownership of the outfall monitoring equipment, along with all the necessary property rights to operate and maintain the equipment, from Newhall Land to Los Angeles County, at which time the outfall monitoring sites will be considered part of the County's MS4 and will be monitoring Reports will be provided annually, and concurrently, to the LARWQCB and LACDPW The project schedule is shown in Table A6-1.

	Anticipated Date of	Anticipated Date of		Deliverable
Activity	Initiation	Completion	Deliverable	Due Date
Monitoring Plan Development	10/1/2012	2/1/2013	Monitoring Plan	3/1/2013
QAPP Development	10/1/2012	2/1/2013	QAPP	3/1/2013
NRSP CoA Sampling	At approval of this Monitoring Plan	After the recordation of the final tract map	NA	NA
WDR Sampling	Upon release of the Waste Discharger Identification (WDID) number for the first outfall's parcel, issued under the Construction General NPDES Permit coverage (i.e., when Notice of Termination [NOT] is issued)	Upon ownership transfer to County	NA	NA
Annual Monitoring Report	Upon receipt of laboratory data from first sampling event	Annually following water quality monitoring	Annual Report	April of each year
Project Completion or Reassessment	NA	After NRSP CoA and WDR monitoring have ceased	NA	NA

#### Table A6-1: Project Schedule



#### 6.4 Geographical Setting

The Santa Clara River flows approximately 83 miles from the headwaters to the Pacific Ocean, drains an area of approximately 1,600 square miles, and is considered one of the few natural river systems remaining in Southern California. The NRSP area is located within the Santa Clara River Valley between Interstate 5 and the Los Angeles/Ventura County line, just west of Six Flags Magic Mountain amusement park and the City of Santa Clarita. The Santa Clara River LARWQCB Reach 5 flows through the NRSP area, which covers approximately 5 miles eastwest and 5.5 miles north-south. The area includes flat agricultural land in the valley, rolling foothills and plateaus to the north and south, and steep terrain of the Santa Susana Mountains to the south. The land contains rich biological resources, including oak savannah and woodland habitat, much of which will be preserved or restored as part of the NRSP.

#### 6.5 Constraints

It is likely that too little data will be available in the first few years to make statistically significant assessments of the results. However, as the datasets grow over several years, confidence in the analysis will increase. Both the variability in data collected at a single monitoring site and the difference in results between compared sites will impact statistical significance. As measured concentrations for water quality parameters often vary by orders of magnitude between sites, monitoring events, or during a given storm event, it is difficult to predict the number of data points needed to make statistically significant comparisons of results.

Additionally, stormwater monitoring can be subject to unforeseen circumstances, including unpredictable weather, equipment malfunctions, potential safety concerns, and seasonal rainfall variations (e.g., drought). Implementation of the Monitoring Plan will minimize the effects of these problems by providing procedures for storm tracking and for deciding if and when to sample during a given storm event. Despite all reasonable efforts to meet the wet weather sampling goals, there is always the potential that the goal of three storms per year will not be met.

#### 7 DATA QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

#### 7.1 Data Quality Objectives (DQOs)

To generate data that will meet Project objectives, it is necessary to define the types of decisions that will be made, identify the intended uses of the data, and design a data collection program. DQOs are defined as an integrated set of thought processes that define data quality requirements based on the intended use of the data. DQOs are necessary in obtaining data of known defensible quality for the intended use. The DQO process assists in determining appropriate quantitation, detection, and reporting limits by defining analytical methods, sample handling procedures, and data assessment requirements.



#### 7.1.1 **Problem to be Resolved**

Monitoring will measure any potential water quality changes that might be caused by NRSP discharges to surface or groundwater, including reclaimed water that is used within the NRSP area, treated effluent from NRWRP, and stormwater and dry weather runoff from the NRSP area and compare storm drain discharges to water quality standards. The NRSP CoA require Newhall, in coordination with LARWQCB staff, to select representative locations upstream and downstream of the NRSP and to sample surface and groundwater quality changes through time. The WDR require representative, rotating storm drains to be selected for the monitoring outfall discharges.

#### 7.1.2 Decisions to be Made

The primary objectives of the Project are to assess the impacts of the NRSP on surface water and groundwater quality over time, as well as to assess potential impacts resulting from storm drain discharges.

#### 7.1.3 Inputs to the Decision

Inputs to the decision include the type, quality, and quantity of data that will be collected in order to make decisions. Data type refers to the physical and chemical data needed for each matrix sampled. Data quality is achieved through adherence to the protocols and acceptance criteria set forth in this QAPP (i.e., precision, accuracy, representativeness, completeness, comparability, and sensitivity [PARCCS], adherence to field and laboratory Standard Operating Practices [SOPs], and data validation). Quantity refers to the amount of data necessary to make remedial decisions. PARCCS acceptance criteria are discussed in Section 7.3 of this QAPP. Input data will include the validated field measurements, as well as laboratory analytical results and supporting QA information, from samples gathered and analyzed in support of the Project.

#### 7.2 Boundaries of the Study

Surface water and groundwater samples will be collected in a biased fashion, both in areas upgradient and downgradient of the NRSP area. This design maximizes the likelihood of detection of any changes in water quality created within the NRSP area. The spatial and temporal boundaries of the study are discussed in detail in Sections 2.3 and 2.4, respectively, of the Monitoring Plan. Representative storm drain outfalls will be selected from among the constructed outfalls in the NRSP. One will be identified per village.

#### 7.2.1 Decision Rules

Paired, non-parametric sign tests, as described in Section 4.2.1 of the Monitoring Plan, will be performed on the NRSP samples to determine if statistically significant changes in water quality are created by the NRSP. Reasonable effort will be made to differentiate between the effects of stormwater runoff, discharges from the NRWRP, infiltration of reclaimed water, and other

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sources. WDR storm drain samples will be compared for exceedances, as described in Section 4.2.1 of the Monitoring Plan, of water quality standards and the pollutant concentrations presented in the RMDP final EIR.

#### 7.2.2 Acceptable Decision Errors

Since sample data from the Site can only estimate the actual conditions of the Site, decisions based on measurement data could be in error (known as decision error). Types of decision errors for the NRSP sampling include:

- Type I Error (false positive): Identifying differences in upgradient and downgradient water quality when none exist
- Type II Error (false negative): Not identifying differences in upgradient and downgradient water quality when they exist

For the purposes of the statistical analyses performed as part of the Project, the acceptable decision errors shall be set at a 90 percent confidence against Type I Error ( $\alpha = 0.10$ , or significance of 90%) and 80 percent confidence against Type II Error ( $\beta=0.2$ , or power of 80%) for the null hypothesis that there have been changes to water quality at the upgradient and downgradient monitoring locations. The acceptability of the data used for the statistical analysis will be based on the associated field and laboratory QC results as described in this QAPP.

#### 7.2.3 Sample Design

All details pertaining to the sample design and procedures are contained in Section 2 of the Monitoring Plan.

#### 7.2.4 Intended Use of Data

Data collected as part of the Project will broaden the understanding of any changes to surface and groundwater quality caused by NRSP implementation. The nature and extent of any water quality changes will be determined based upon data generated from the collection and chemical analysis of surface water, groundwater, and storm drain samples.

#### 7.3 PARCCS Review

This QAPP addresses both field and laboratory activities. QA objectives, formally known as Data Quality Indicators (DQIs), for measurement data are expressed in terms of precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS). Evaluation of DQIs provides the mechanism for ongoing review and evaluation of data quality throughout the project and ultimately will be used to define the data quality achieved for the various measurement parameters. The field QA/QC program will be accomplished through the collection of field replicates and trip blanks. The analytical QA/QC program will be assessed through the internal laboratory QC performed, including but not limited to method blanks,

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laboratory control sample (LCS) recoveries, laboratory duplicates, surrogate recoveries, and matrix spike/matrix spike duplicate (MS/MSD) recoveries and positive and negative controls. Data quality acceptance criteria are presented below.

#### 7.3.1 Precision

Precision describes the extent to which a measurement is reproducible and is expressed by calculating variability in a group of measurements. During the collection of data using field methods and/or instrumentation, precision is checked by reporting several measurements taken at one location and comparing the results. Precision will be reported as the relative percent difference (RPD) for two results and relative standard deviation (RSD) for three or more results.

In the field, precision is determined by replication of field measurements and collection of field duplicates (for a minimum of 5 percent of total project sample count). In the laboratory, analytical precision is measured through laboratory duplicates (for a minimum 5 percent of samples), matrix spike/matrix spike duplicate pairs, and LCS/LCS duplicate pairs and is evaluated by comparison to the maximum allowable relative percent difference (RPD) used by the analytical laboratory and the Project Measurement Quality Objectives (MQOs) shown in Tables A7-2 to A7-11. Precision RPD is calculated using the equation:

$$RPD(Precision) = \left|\frac{C_1 - C_2}{(C_1 + C_2)/2}\right| \times 100$$

where  $C_1$  = Sample 1 concentration, and  $C_2$  = Sample 2 concentration

Field measurement precision MQOs are shown in Table A7-1. For field duplicates, the acceptance criteria will be 30 RPD.

#### 7.3.2 Accuracy

Accuracy describes the degree of closeness of a measurement to its true (or actual) value. The accuracy of field protocols is difficult to assess quantitatively, but sampling accuracy can be maximized by the adoption of and adherence to a strict field QA program. Specifically, procedures will be performed following the SOPs discussed in Section 3 of the Monitoring Plan and shown in Table A11-1. Equipment and instrumentation will be properly calibrated and well-maintained. Through regular review of field procedures, any deficiencies will be documented and corrected in a timely manner.

In the laboratory, accuracy will be determined by measurement of a standard solution with a known concentration of analyte. Laboratory accuracy will be ascertained through the analysis of matrix spike/matrix spike duplicate (MS/MSD), laboratory control samples (LCSs), and surrogate recoveries (for organic constituents). Accuracy is reported as percent recovery (%R) and compared against laboratory performance criteria and Project MQOs. Maximum acceptable %R for accuracy is shown in Tables A7-1 to A7-11.

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%R is calculated using the equation:

% 
$$R = \frac{Spiked Sample Concentration - Sample Concentration}{Spike Concentration} \times 100 \square$$

#### 7.3.3 Representativeness

Samples must be representative of conditions at storm drain outfall locations throughout the NRSP and upgradient and downgradient of the NRSP. Representativeness is maintained by utilizing representative sampling locations, timing sampling events to be temporally representative, and employing appropriate field sampling techniques and laboratory procedures. Representative storm drain outfalls will be chosen (once constructed) based on site hydrology, accessibility, and the ability to meet the project objectives. To increase representativeness, storm drain outfall sampling locations will be rotated on an annual basis. Sampling sites that are spatially representative of upgradient and downgradient conditions were chosen based on site hydrology, accessibility, and ability to meet the objectives of the Project. Site descriptions are included in Section 2 of the Monitoring Plan. To ensure temporal representativeness, Monitoring Plan design includes surface water sampling during wet weather to capture stormwater runoff, outfall discharge sampling during wet and dry weather, and routine groundwater sampling following the end of the wet and dry seasons. This schedule will capture long-term, seasonal, and runoff-driven fluctuations in water quality constituent concentrations.

SOPs will be used for field sample collection techniques and laboratory procedures to ensure representativeness and avoid contamination of samples. Collection of field duplicates will further allow for assessing representativeness of samples. The MQO for field duplicates will be 30 RPD, as discussed in Section 7.2.1, above. Sample collection methods are described in greater detail in Section 3 of the Monitoring Plan and Section 11 of this QAPP, and field duplicates are discussed in Section 14 of this QAPP.

#### 7.3.4 Completeness

Completeness is the measurement quality criterion that assesses the proportion of data obtained that is determined to be valid based on analytical QA/QC methods. By design, the sampling sites, frequency, and water quality measurements will provide sufficient depth and quantity of information to meet Project objectives. No data gaps have been identified that might impede success of the Project. For the purposes of meeting Monitoring Plan objectives, the Project MQO will be 90 percent completeness for all measurements.

The percent completeness for each set of samples will be calculated as follows:

% Completeness = 
$$\frac{Valid Data}{Total Data Planned} \times 100$$



#### 7.3.5 Comparability

USEPA-established methods and approved protocols have been selected or specified as appropriate for this investigation. By using standard sampling and analytical procedures, data sets will be comparable. These procedures are discussed in detail in the Section 3 of the Monitoring Plan.

#### 7.3.6 Sensitivity

Sensitivity refers to the minimum magnitude at which analytical methods can resolve quantitative differences among sample concentrations. If the minimum magnitude for a particular analytical method is below an action level or risk screening criterion, then the method sensitivity is acceptable to fully evaluate the dataset with respect to the desired reference values. To allow for matrix interferences variability in instrument control, a reporting limit of 2.5-5 times the method detection limit (MDL) is typically selected. Sensitivity is measured by the method reporting limit, which expresses the lowest concentration of analyte that can be accurately detected by the method. Laboratory reporting limits shall be less than or equal to the method reporting limit MQOs are shown in Tables A7-1 through A7-11.

#### Table A7-1: Field Measurement MQOs

Parameter	Precision	Accuracy (%)	Target Reporting Range	Completeness
Depth to Groundwater (ft)	$\pm 0.01$	$\pm 0.01$	< 250.0	90
Temperature (°Celsius)	$\pm 1.0$	$\pm 0.5$	0.0 - 60.0	90
pH (standard units)	$\pm 0.2$	$\pm 0.01$	0.00 - 14.00	90
Turbidity (NTU)	$\pm 2.0$	$\pm 5\%$	1 - 100	90
Specific Conductivity ( $\mu$ S/cm)	$\pm \ 10 \ \%$	$\pm 2$ %	0 - 3999	90
Dissolved Oxygen (mg/L)	±6 %	±6 %	0-19.9	90
Total Dissolved Solids (mg/L)	$\pm 5\%$	±2 %	0 - 2000	90

#### Table A7-2: Biological Parameter MQOs

Parameter	Accuracy	Precision	Target Reporting Limit	Completeness	
Acute Toxicity	Method Performance	Method Performance	< 30% MSD (minimum significant difference)	90	
Chronic Toxicity	Criteria	Criteria	< 47% MSD (minimum significant difference)	90	
Index of Biological Integrity	Method Performance Criteria				

#### Table A7-3: Bacteria MQOs

Parameter	Accuracy	Precision	Target Reporting Limit	Completeness
E. coli	Positive Control and Reference Material 80-120% Negative Control: No growth on filter	RPD < 25%	2 MPN/100 mL	90

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Parameter	Precision (RPD)	Accuracy (%R)	Target Reporting Limit (mg/L)	Completeness
Ammonia-N	15	90-110	0.1	90
Biochemical Oxygen Demand (BOD)	20	85-115	2.0	90
Chloride	20	72-118	0.50	90
Chlorine, Total Residual	15	65-128	0.05	90
Cyanide, Total	10	90-110	5.0	90
Dissolved Oxygen (DO)*	25	80-120	1.0	90
Fluoride, Total	20	79-109	0.10	90
Methylene Blue Active Substances	20	77-118	0.05	90
(MBAS)				
Non-Ionic Surfactants as CTAS)	8	43-115	0.20	90
Nitrate-N	20	80-107	0.11	90
Nitrite-N	20	86-111	0.15	90
Nitrogen, Total	NA	NA	NA	90
Nitrogen, Total Kjeldahl	10	90-110	0.10	90
Oil and Grease	18	78-114	5.0	90
Orthophosphate-P	20	80-120	0.010	90
Perchlorate	15	80-120	0.002	90
Phosphorus, Total as P	20	71-118	0.10	90
Settleable Solids	NA	NA	0.1 (mL/L/hr)	90
Sulfate	20	84-114	0.50	90
Total Dissolved Solids (TDS)*	10	91-104	10	90
Total Hardness (as CaCO3)	30	70-130	0.10	90
Total Suspended Solids (TSS)	20	NA	5.0	90

#### Table A7-4: Inorganics and General Chemistry MQOs

 Total Suspended Solids (TSS)
 20
 NA
 5.0

 \* Samples may be taken for laboratory analysis if DO and TDS cannot be measured in the field

 NA
 National sectors analysis if DO and TDS cannot be measured in the field

#### Table A7-5: Metals MQOs

Parameter	Precision (RPD)	Accuracy (%R)	Target Reporting Limit (µg/L)	Completeness
Antimony, Total	50	50-150	0.10	90
Antimony, Dissolved	30	70-130	0.50	90
Arsenic, Total	NA	58-110	0.050	90
Arsenic, Dissolved	30	70-130	0.40	90
Barium, Total	30	70-130	0.20	90
Barium, Dissolved	30	70-130	0.50	90
Beryllium, Total	50	50-150	0.050	90
Beryllium, Dissolved	30	70-130	0.10	90
Boron, Total & Dissolved	30	70-130	1.0	90
Cadmium, Total	30	70-130	0.010	90
Cadmium, Dissolved	30	70-130	0.10	90
Chromium, Total	30	70-130	0.20	90
Chromium-3	NA	NA	NA	90
Chromium-6	10	88-112	0.30	90
Copper, Total	30	70-130	0.010	90
Copper, Dissolved	30	70-130	0.50	90
Iron, Total	30	70-130	1	90
Iron, Dissolved	30	70-130	20	90
Lead, Total	30	70-130	0.010	90
Lead, Dissolved	30	70-130	0.20	90
Mercury, Total & Dissolved	20	70-130	0.05	90
Nickel, Total	30	70-130	0.010	90
Nickel, Dissolved	30	70-130	0.8	90
Selenium, Total	30	70-130	0.10	90
Selenium, Dissolved	30	70-130	0.40	90
Silver, Total	NA	30-151	0.050	90
Silver, Dissolved	30	70-130	0.20	90
Thallium, Total	30	65-125	0.050	90
Thallium, Dissolved	30	70-130	0.20	90
Zinc, Total	NA	70-130	0.20	90
Zinc, Dissolved	30	70-130	5.0	90

#### Table A7-6: Chlorinated Herbicides MQOs

Parameter	Precision (RPD)	Accuracy (%R)	Target Reporting Limit (µg/L)	Completeness
2,4,5-T	25	53-147	0.25	90
2,4,5-TP	25	61-146	0.25	90
2,4-D	25	48-148	0.50	90
2,4-DB	25	10-206	2.5	90
3,5-Dichlorobenzoic Acid	25	26-206	1.2	90
4-Nitrophenol	25	10-174	1.2	90
Aciflurofen	25	28-190	0.50	90
Bentazon	25	34-192	2.5	90
Dalapon	25	10-202	0.50	90
DCPA	25	40-178	0.25	90
Dicamba	25	54-164	0.75	90
Dichloroprop	25	48-174	1.0	90
Dinoseb	25	10-149	0.50	90
MCPA	25	10-168	100	90
MCPP	25	17-195	100	90
Pentachlorophenol	25	61-165	0.25	90
Picloram	25	48-169	0.75	90

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Parameter	Precision (RPD)	Accuracy (%R)	Target Reporting Limit (µg/L)	Completeness
4,4'-DDD	30	31-141	0.050	90
4,4'-DDE	30	30-145	0.050	90
4,4'-DDT	30	25-160	0.050	90
Aldrin	30	42-122	0.050	90
Aroclor 1016	30	50-114	1.0	90
Aroclor 1221	NA	NA	1.0	90
Aroclor 1232	NA	NA	1.0	90
Aroclor 1242	NA	NA	1.0	90
Aroclor 1248	NA	NA	1.0	90
Aroclor 1254	NA	NA	1.0	90
Aroclor 1260	30	8-127	1.0	90
BHC-alpha	30	37-134	0.050	90
BHC-beta	30	17-147	0.050	90
BHC-delta	30	19-140	0.050	90
BHC-gamma (Lindane)	30	32-127	0.050	90
Chlordane	30	45-119	0.50	90
Dieldrin	30	36-146	0.050	90
Endosulfan sulfate	30	26-144	0.050	90
Endosulfan I	30	45-153	0.050	90
Endosulfan II	30	2-202	0.050	90
Endrin	30	30-147	0.050	90
Endrin aldehyde	30	30-180	0.050	90
Heptachlor	30	34-111	0.050	90
Heptachlor epoxide	30	37-142	0.050	90
Methoxychlor	NA	NA	0.050	90
Toxaphene	30	41-126	2.0	90

#### Table A7-7: Organochloride Pesticides MQOs

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#### Table A7-8: Organophosphorus Pesticides MQOs

Parameter	Precision (RPD)	Accuracy (% R)	Target Reporting Limit (µg/L)	Completeness
Azinphos methyl	25	18-159	0.10	90
Bolstar (Sulprofos)	25	35-171	0.10	90
Chlorpyrifos	25	36-157	0.10	90
Coumaphos	25	25-199	0.10	90
Demeton-o	25	22-179	0.10	90
Dematon-s	25	32-173	0.10	90
Diazinon	25	33-172	0.10	90
Dichlorvos	25	11-197	0.10	90
Dimethoate	25	NA	0.25	90
Disulfoton	25	46-155	0.10	90
Ethoprop (Ethoprofos)	25	54-148	0.10	90
Ethyl parathion	25	36-169	0.25	90
Fenchlorophos (Ronnel)	25	30-166	0.10	90
Fensulfothion	25	32-236	0.10	90
Fenthion	25	50-154	0.10	90
Malathion	25	7-180	0.25	90
Merphos	25	40-188	0.10	90
Methyl parathion	25	43-169	0.10	90
Mevinphos (Phosdrin)	25	18-186	0.10	90
Phorate	25	46-160	0.10	90
Tetrachlorvinphos (Stirophos)	25	28-180	0.10	90
Thionazin	25	NA	0.25	90
Tokuthion	25	34-164	0.10	90
Trichloronate	25	41-155	0.10	90

#### Table A7-9: Pyrethroid Pesticides MQOs

Parameter	Precision	Accuracy Recovery (%)	Target Reporting Limit (ng/L)	Completeness
Allethrin	NA	48-156	2.0	90
Bifenthrin	NA	25-191	2.0	90
Cyfluthrin	NA	37-169	2.0	90
Cypermethrin	NA	29-178	2.0	90
Danitol (Fenpropathrin)	NA	NA	2.0	90
Deltamethrin/Tralomethrin	NA	30-200	2.0	90
Dichloran	NA	53-164	2.0	90
Esfenvalerate	NA	31-178	2.0	90
Fenvalerate	NA	31-178	2.0	90
L-Cyhalothrin	NA	38-182	2.0	90
Pendimethalin	NA	52-145	2.0	90
Permethrin	NA	59-148	5.0	90
Prallethrin	NA	54-148	2.0	90
Sumithrin	NA	56-146	10	90
Tefluthrin	NA	38-195	2.0	90

#### Table A7-10: Semivolatile Organics MQOs

Parameter	Precision (RPD)	Accuracy Recovery (%)	Target Reporting Limit (µg/L)	Completeness
N-Nitrosodimethylamine (NDMA)	NA	50-150	0.002	90
1-Methylnaphthalene	30	50-150	0.10	90
2-Methylnaphtalene	30	50-150	0.10	90
Acenaphthene	30	47-145	0.10	90
Acenaphthylene	30	33-145	0.10	90
Anthracene	30	27-133	0.10	90
Benzo[a]anthracene	30	33-143	0.10	90
Benzo[a]pyrene	30	17-163	0.10	90
Benzo[b]fluoranthene	30	24-159	0.10	90
Benzo[g,h,i]perylene	30	0.1-219	0.10	90
Benzo[k]fluoranthene	30	11-162	0.10	90
Chrysene	30	17-168	0.10	90
Dibenz(a,h)anthracene	30	0.1-227	0.10	90
Fluoranthene	30	26-137	0.10	90
Fluorene	30	59-121	0.10	90
Indeno(1,2,3-cd)pyrene	30	0.1-171	0.10	90
Naphthalene	30	21-133	0.10	90
Phenanthrene	30	54-120	0.10	90
Pyrene	30	52-115	0.10	90

#### Table A7-11: Volatile Organics MQOs

Parameter	Precision (RPD)	Accuracy (% R)	Target R.L. (µg/L)	Completeness
1,1,1-Trichloroethane	30	52-162	1.0	90
1,1,2,2,-Tetrachloroethane	30	46-157	1.0	90
1,1,2-Trichloroethane	30	52-150	1.0	90
1,1-Dichloroethane	30	59-155	1.0	90
1,1-Dichloroethene	30	0.1-234	1.0	90
1,2-Dichloroethane	30	49-155	1.0	90
1,2-Dichloropropane	30	0.1-210	1.0	90
2-Butanone	NA	NA	5.0	90
2-Chloroethyl vinyl ether	30	0.1-305	5.0	90
2-Hexanone	NA	NA	5.0	90
4-Methyl-2-pentanone	NA	NA	5.0	90
Acetone	NA	NA	5.0	90
Acrolein	NA	NA	5.0	90
Acrylonitrile	NA	NA	2.0	90
Benzene	30	37-151	1.0	90
Bromodichloromethane	30	35-155	1.0	90
Bromoform	30	45-169	1.0	90
Bromomethane	30	0.1-242	1.0	90
Carbon Disulfide	NA	NA	1.0	90
Carbon tetrachloride	30	70-140	1.0	90
Chlorobenzene	30	37-160	1.0	90
Chloroethane	30	14-230	1.0	90
Chloroform	30	51-138	1.0	90
Chloromethane	NA	NA	1.0	90
cis-1,3-Dichloropropene	30	0.1-227	1.0	90
Dibromochloromethane	30	53-149	1.0	90
Ethylbenzene	30	37-162	1.0	90
m-Dichlorobenzene	30	59-156	1.0	90
Methyl tert-butyl ether (MTBE)	30	70-130	2.0	90
Methylene chloride	30	0.1-221	1.0	90
o-Dichlorobenzene	30	18-190	1.0	90
p-Dichlorobenzene	30	18-190	1.0	90
Tetrachloroethene	30	64-148	1.0	90
Toluene	30	47-150	1.0	90
trans-1,2-Dichloroethene	30	54-156	1.0	90
trans-1,3-Dichloroethene	30	17-183	1.0	90
Trichloroethene	30	71-157	1.0	90
Vinyl chloride	30	0.1-251	1.0	90



#### 8 SPECIAL TRAINING NEEDS/CERTIFICATION

#### 8.1 Specialized Training or Certifications

All field sampling personnel will have taken a minimum of 8 hours of coursework in first aid/CPR or wilderness/remote area first aid (equivalent to courses offered by the American Red Cross), be experienced with environmental sampling techniques, and be familiar with swift water safety and rescue procedures. Additional training, such as confined space entry, 24-hour hazwoper, fall protection, traffic control, and hazard communication, will be required if deemed necessary based on site conditions. Sampling personnel will be required to review the Project Health and Safety Plan. The analytical laboratory selected to perform chemical analysis will be certified by the USEPA and the California Department of Public Health's Environmental Laboratory certified by the California Department of Public Health's Environmental Laboratory Accreditation Program.

#### 8.2 Training and Certification Documentation

Copies of required training documentation for Project personnel will be kept on file. Contracted laboratories will maintain documentation of certification and will provide to Project representatives on request.

#### 9 DOCUMENTS AND RECORDS

The Data Manager will collect and maintain all documents and records associated with field documentation and laboratory analysis. The QAPP will be maintained by the Data Manager, and the most recent version will be redistributed to those persons listed in Table A3-1 after any revision.

#### 9.1 Field Documentation

Data will be collected on standardized field data sheets, examples of which are included in Appendix D. Field Data Sheets will include date, time, sampling site, names of field personnel, and collected field data. On return to the office, field data sheets will be scanned and transcribed electronically into a spreadsheet (such as Excel) format. All field data sheets and photographic documentation will be kept in a project folder on a computer server for reference by all Project personnel. Electronic data kept on the server will be backed up at least weekly and will be stored as described in Table A9-1.

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#### 9.2 Analytical Data Records

The analytical laboratory will provide reports (electronic and hard-copy) that include a letter of transmittal, a case narrative, chain of custody information, and analytical results for all field and quality control samples. Additionally, electronic spreadsheets of laboratory results will be provided for ease of analysis. Reports will be reviewed for completeness and errors by the Data Manager and QA/QC will be conducted by the QAO. Any concerns resulting from these reviews will be remedied with the laboratory, and the final reports will be stored as described in Table A9-1: Record Retention and Archival Information.

Document	Retention	<b>Responsible for Archival</b>
Field Records	15 years	Data Manager
Analytical Records	15 years	Data Manager
QAPP	15 years	Data Manager

#### Table A9-1: Record Retention and Archival Information

#### **10 SAMPLING PROCESS DESIGN**

Sampling process design is described in detail in Section 2 of the Monitoring Plan.

15 years

#### 11 SAMPLING METHODS

Reports

Sampling methods for wet weather surface water sampling, wet and dry weather outfall sampling, groundwater sampling, and biological assessment are described in detail Section 3 of the Monitoring Plan. A number of standard operating procedures for field sampling are cited in sample collection methods. These SOPs are summarized in Table A11-1, and relevant excerpts are included in Appendix E. The contracted laboratory for biological assessment and BMI sampling, Aquatic Bioassay and Consulting Laboratories, Inc., has their own SAP and SOPs, which are not provided here but can be made available upon request.

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SOP	Source	Citation
Clean Hand/Dirty Hands Sampling	USGS Field Manual for the Collection of Water Quality Data	USGS, 2006
Procedures	US EPA Method 1669. Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels	US EPA, 1996
Collection of Groundwater Samples	Field Measurements: USGS Techniques of Water- Resources Investigations, Book 9 Ch. A4	USGS, 2006
Collection of Low Flow Groundwater Samples	Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells	US EPA, 2010
Field Measurements of Surface Water / Field Collection Procedures for [Surface] Water Samples	CDFG Standard Operating Procedures for Conducting Field Measurements	CDFG, 2007
Equipment Installation and Maintenance / [Storm Drain Outfall] Sample Collection	Caltrans Guidance Manual: Storm Water Monitoring Protocols	Caltrans, 2000
Biological Assessment	SWAMP Standard Operating Procedures for Collecting BMI Samples and Associated Physical and Chemical Data	Ode, 2007

#### Table A11-1: Standard Operating Procedures (SOPs) for Field Sampling

#### 12 SAMPLE HANDLING AND CUSTODY

Sample handling and custody, including sample collection and identification, documentation, field datasheets, sample containers, sample packing, and sample shipping are described below.

#### **12.1** Sample Handling and Custody Protocols

The following sample handling and custody protocols will be used to prevent sample contamination:

- 1. One member of the sampling team will take custody of all collected samples for laboratory analysis.
- 2. Collected samples will be stored in an ice-filled cooler at approximately 4° C for storage and transport.
- 3. As some samples must be analyzed within 6 hours to prevent degradation, samples will be transferred as efficiently as possible to the laboratory using standard chain of custody

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documentation. Samples will be field filtered, as necessary, to satisfy short hold times that could not be met otherwise.

4. Sample storage, preservation, and holding time before analysis will be done in accordance with Table B2-1 in Appendix B.

#### 12.2 Sample Custody Roles and Responsibilities

The persons responsible for sample custody, and a brief description of their duties, are as follows:

- 1. Laboratory Sample Custodian or Commercial Supplier: Verifies that the sample containers are certified clean; arranges for container shipment to field sampling personnel or the contractor's equipment shop;
- 2. Equipment Manager: Receives and stores sample containers that are shipped from a laboratory or a commercial supplier; relinquishes sample containers to field sampling personnel; initiates chain of custody (COC) from sample containers in storage;
- 3. Field Staff: Receive sample containers from laboratory, inspect sample containers for physical integrity; retain shipping invoice or packing list from shipping courier as documentation of transfer of sample containers; collect and preserve samples; complete the COC, retain sample containers and samples under custody until sample shipment; relinquish samples to shipping courier or to lab representative.
- 4. Project Manager: Verifies reported laboratory analyses to the sample COC form; assures that COC documentation is incorporated into the project file.

#### **12.3** Chain of Custody Record (COC)

The field COC record is used to record the custody of all samples collected and sent to the laboratory for analysis. The COC also serves as a sample logging reference for the analytical laboratories' sample custodian.

The following information must be supplied in the indicated spaces on the field COC record:

- 1. Project name and number
- 2. Signatures of all samplers and/or the sampling team leader in the designated signature block
- 3. Sampling station number, date, and time of sample collection, grab or composite sample designation, sample preservation type, and a brief description of the type of sample and the sampling location must be included on each line (each line shall contain only those samples collected at a specific location).
- 4. Sampling team leader's name shall be recorded in the right or left margin of the COC when samples collected by more than one sampling team are included on the same form.
- 5. Total number of sample containers must be listed in the indicated space for each sample. The total number of individual sample containers must also be listed for each type of

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analysis under the indicated media or miscellaneous columns. Note that it is impossible to have more than one media type per sample. The type of container and required analyses should be circled as indicated on the COC.

- 6. The field investigator and subsequent transferee(s) must document the transfer of the samples listed on the COC in the spaces provided at the bottom of the Record. Both the person relinquishing the samples and the person receiving them must sign the form; the date and time that this occurred must be documented in the proper space on the Record. Usually, the last person receiving the samples or evidence should be a laboratory sample custodian.
- 7. Any person relinquishing the samples to a commercial carrier (i.e. Federal Express) shall note the name of the carrier on the COC in the "relinquished to" space with the date and time. The remarks column at the bottom of the Record is used to record air bill numbers or registered or certified mail serial numbers.

The COC record is a serialized document. Once the COC is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for COC should be evaluated upon its inclusion of all of the above information in a legible format. An example of a COC form for Weck Laboratories is provided in Appendix D.

#### **13** ANALYTICAL METHODS

Field and laboratory analytical methods will be USEPA-approved and are discussed in Section 3 of the Monitoring Plan and displayed in tabular form in Appendix B.

#### 14 QUALITY CONTROL

The following measures will be taken to ensure the quality of data collected during field measurements, field sampling, and laboratory analysis.

#### 14.1 Field Measurement Quality Control

Field equipment will be calibrated as described in Section 16 to ensure accuracy of field data collection. Additionally, field measurements will be duplicated in the field and must agree by the precision MQOs discussed in Section 7 and shown below in Table A14-1. If the two measurements do not meet the precision criteria, three additional replicates will be taken and the median of the five measurements reported on the field data sheet.

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Field Measurement	Replicates	Acceptance Limits
Temperature	2	± 1.0° C
рН	2	$\pm 0.2$ standard units
Turbidity	2	$\pm 5\%$
Sepcific Conductivity	2	$\pm$ 10 %
Dissolved Oxygen	2	$\pm$ 6 %
Total Dissolved Solids	2	$\pm 5\%$
Depth to Groundwater	2	$\pm 0.01$ ft

#### Table A14-1: Field Measurement Quality Control Measures

#### 14.2 Field Sampling Quality Control

Sources of contamination in the field include dirty sampling equipment, airborne contaminants, and contaminants introduced by field personnel (e.g., dirty hands/gloves, sunscreen, and insect repellant). Quality control in the field consists of prevention and testing of field duplicates and trip blanks. Adherence to SOPs, including CDFG SOP for Conducting Field Measurements (CDFG, 2007), Caltrans Guidance Manual: Storm Water Monitoring Protocols (Caltrans, 2000) and USGS Field Manual for the Collection of Water Quality Data (USGS, 2006) will minimize contamination. Additionally, sample quality will be checked by analyzing field duplicates.

#### 14.2.1 Trip Blanks

Trip Blank - One trip blank for every cooler containing liquid samples for VOC analyses (prepared and supplied by the laboratory with appropriately preserved laboratory certified clean water), will be transported to the monitoring site, handled like a sample, and submitted to the laboratory for VOC analysis. Control limits for trip blanks will be no VOCs detected in the sample.

If contamination of the trip blanks and associated samples is known or suspected, the laboratory should qualify the affected data, and notify the project coordinator, who in turn will follow the process detailed in the method.

#### 14.2.2 Field Duplicates

Blind field duplicate samples will be collected to test sampling precision, and will represent at a minimum 5 percent of the total project sample count. The QAO and/or Data Manager will choose analyte(s) and sampling locations for the field duplicate prior to the sampling event. Analytes may be chosen randomly or as a quality check for specific constituents of interest. Field duplicates will be taken from the same sampling container to minimize differences between the samples. Control limits for field duplicates will be equal to the Precision MQOs shown in Tables A7-1 to A7-11.

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For duplicates with a heterogeneous matrix or ambient levels below the reporting limit, failed results may be qualified. All failures should be communicated to the project coordinator, who in turn will follow the process detailed in the method.

#### 14.3 Analytical Laboratory Quality Control

#### 14.3.1 Method Blanks

A method (or preparation) blank is prepared at the frequency specified by the referenced method (typically one per analytical batch). The purpose of the method blank is to check that contaminants are not introduced by the glassware, reagents, standards, personnel, during sample preparation and/or analysis. An instrument blank is also analyzed during each calibration shift to verify that contaminants are not being introduced by components of the instrumentation or analytical laboratory.

Various, other routine blank checks are in place to verify that new lots of glassware, reagents and standards, decontaminated glassware, sample storage areas (including refrigerators), and water purification systems are contaminant-free. Monitoring parameters should not be detected above the RL in the method blanks. If this occurs, the sample analysis must be halted, the source of the contamination investigated, the samples along with a new method blank prepared and/or re-extracted, and the sample batch and fresh method blank reanalyzed. If reanalysis is not possible due to sample volume, flag associated samples as estimated.

#### **14.3.2** Laboratory Surrogates

Surrogate standards are added to each sample intended for organics analysis in accordance with the particular method being used. Surrogate recoveries must meet method acceptance criteria before the analytical data will be released. In some instances, the sample matrix may produce interferences that adversely affect recoveries. Surrogate recovery interferences must be confirmed by preparation and reanalysis of the sample.

#### 14.3.3 Laboratory Control Samples

In addition, a laboratory control sample (LCS) consists of a clean matrix fortified with known concentrations of standard solutions containing target analytes of interest. The recovery of these standards is quantitatively measured during analysis, and historical records maintained on the percent recovery for each sample. One LCS is analyzed for each sample extraction/analytical batch (a batch is a group of 20 samples or less) as applicable to the method. The control limits for LCSs are the MQOs for accuracy shown in Tables A7-1 to A7-11

#### 14.3.4 Laboratory (Matrix) Duplicates

Laboratory precision will be measured by duplicating an analysis by splitting the same field sample and using the same sample extraction/preparation procedure and analysis for both

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aliquots. The control limits for laboratory replicates are the MQOs for Precision shown in Tables A7-1 to A7-11. For duplicates with a heterogeneous matrix or ambient levels below the reporting limit, failed results may be qualified. Other failures should be reanalyzed as sample volume allows. A matrix spike duplicate may not be analyzed in place of a laboratory duplicate.

#### 14.3.5 Matrix Spikes and Matrix Spike Duplicates

A matrix spike (MS) is an environmental sample to which known concentrations of analytes have been added. The MS is taken through the entire analytical procedure and the recoveries of the analytes are calculated. Results are expressed as percent recovery. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis.

A matrix spike duplicate (MSD) is one of the aliquots of an environmental sample that is then either collected in separate containers (as the MS/MSD samples) or divided into two separate aliquots once received by the laboratory, each of which is spiked with a known concentrations of analytes. The two spiked aliquots are processed separately and the results compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as RPD and percent recovery.

One MS/MSD set will be analyzed for every 20 investigative samples. The MS/MSD will be site-specific and, therefore, field personnel will be responsible for collecting additional sample volumes to account for the MS/MSD samples. The sample to be used for the MS/MSD analysis shall be identified on the COC, to ensure that a project sample is used (instead of a non-project sample that is part of the analytical batch). Results will be compared to the Recovery MQOs shown in Tables A7-1 to A7-11.

If matrix interference is suspected and reference material recoveries are acceptable, the matrix spike duplicate result must be qualified.

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

Field measurement and sampling equipment shall include an electronic multiparameter water quality meter, a velocity meter, automated storm drain samplers and flow measurement device, and a well pump for groundwater. Equipment will be maintained in accordance with manufacturer specifications, including battery replacement, sensor/electrode cleaning, membrane replacement, and other recommended maintenance. Additionally, all instruments will be inspected before and after each sampling event for signs of damage or potential malfunction.

Contracted laboratories are responsible for testing and maintaining laboratory equipment according to manufacturer and method specifications. Laboratories will provide equipment maintenance records to Project staff on request.

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#### 16 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

Field measurement equipment will be calibrated at the frequency recommended by the manufacturer and if accuracy or precision issues are found. Mechanical current meters for measuring stream velocity will be tested prior to each use by ensuring parts spin freely when manually spun above water. Electronic sensors on water quality meters will be cleaned before and after each use and will be calibrated in-house or sent to the manufacturer for repair or calibration, as required.

Laboratory instruments will be calibrated at the manufacturer-recommended frequency by the contracted laboratory. Calibration information will be provided to Project staff on request.

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

Supplies, including sample collection bottles received from the laboratory, will be inspected on receipt for completeness and quality. If any supplies are missing or damaged, the supplier will be contacted and the supplies will be replaced. Supply inventory will be taken before each sampling event to ensure that all necessary materials are available. The contracted laboratory is responsible for inspection and maintenance of laboratory and analysis supplies.

#### **18 NON-DIRECT MEASUREMENTS**

The only non-direct measurement used in the Project is wet-weather streamflow, which will be estimated from the USGS stream gage approximately 2.5 miles downstream of the Project site. It will be explicit on all field forms and in reports that this is an estimate rather than a measured value of streamflow at the time of sampling.

#### **19 DATA MANAGEMENT**

All Project data, if not initially in electronic form (e.g. field data sheets), will be digitized within 7 days of the sampling event. All electronic data, including field data, laboratory data, and quality information will be stored on a computer server that is shared with Project personnel at the Los Angeles office of Geosyntec Consultants. This server is backed up on an off-site server at least every 7 days.

Prior to analysis, field and analytical data will be transcribed or otherwise entered into spreadsheets, saved uniquely by sample date, for analysis and inclusion in annual reports. The Data Manager is responsible for ensuring that all data management requirements are met.

#### 20 ASSESSMENT AND RESPONSE ACTIONS

The Project QAO will annually review sampling, data acquisition, laboratory analysis, and data analysis procedures for the purpose of meeting the quality objectives as described in this QAPP. Reviews will consist of (1) confirming SOPs are being followed during field sampling based on inquiries to field staff and/or the Data Manager, (2) verification of COC documentation, (3) review of analytical data as they relate to MQOs, and (4) review of data report before submission to LARWQCB and LACDPW.

If the annual review finds that any part of the QAPP is not being applied, the QAO will discuss the appropriate actions to take with responsible Project staff and/or the Project Manager. Actions may include determining the cause of the discrepancy, quantifying or qualifying the extent of the quality issues, discussing data quality impacts of the discrepancy to the Project, correcting the problem if possible, and developing a plan to avoid similar issues in the future. If a deviation from the QAPP is discovered, the annual report to the RWQCB and LACDPW will include a discussion of it and its implications to meeting project objectives.

#### 21 **REPORTS TO MANAGEMENT**

Data will be analyzed and reported annually each April to the LACDPW, concurrent with submittal to the LARWQCB. The report will describe monitoring efforts over the prior hydrologic year (October 1 – September 30) and data analysis, including a comparison between upgradient and downgradient monitoring, the statistical significance of those differences, and the possible sources of any water quality changes observed, as well as storm drain water quality comparison to water quality data. Data reports will be reviewed and approved by the Data Manager and the Project Manager before submission to Newhall Land, the LARWQCB, and LACDPW. Transmission of the annual report will include electronic files of field measurements as well as chemistry laboratory results in either of the following formats:

- A) SWAMP comparable format presented in a flat file (spreadsheets, tab-delimited format, comma separated value, etc.); or
- B) Using the latest Standardized Data transfer Formats (SDTF) from the Southern California Municipal Stormwater Monitoring Coalition (SMC).

#### 22 DATA REVIEW, VERIFICATION, AND VALIDATION

All Project data will be reviewed by the Data Manager and QAO for validation, and all reports will be reviewed by the Data Manager and Project Manager. Data quality will be verified in writing to the appropriate Project staff. Any issues with data quality or reporting will be noted and corrected, if possible. All changes to original data require agreement of the Project QAO, Data Manager, and Laboratory Project Manager, as well as written documentation of the change.

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Data that does not meet the quality objectives will be qualified with an identifying code in all reports. A list of validation qualifiers for analytical data, in accordance with US EPA guidelines, is included in Table A22-1.

Qualifier	Explanation
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
J+	The analyte was positively identified; however, the associated numerical value is likely to be higher that the concentration of the analyte in the sample due to positive bias of associated QC or calibration data or attributable to matrix interference.
J-	The analyte was positively identified; however, the associated numerical value is likely to be lower that the concentration of the analyte in the sample due to negative bias of associated QC or calibration data or attributable to matrix interference.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

#### 23 RECONCILIATION WITH USER REQUIREMENTS

Data that satisfies the quality objectives outlined in this QAPP will be analyzed and reported as described in Section 4 of the Monitoring Plan.

Prepared for

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## **Appendix B**

## **Monitoring Parameters and Laboratory Analyses**

for the Newhall Ranch Specific Plan Water Quality Monitoring Plan Conditions of Approval and Clean Water Act Section 401 Water Quality Certification and Waste Discharge Requirements

Prepared by



engineers | scientists | innovators

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Project Number LA0170

March 27, 2013



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### 1 FIELD MEASUREMENTS AND LABORATORY ANAYTICAL METHODS <u>PROGRAM KEY</u>

SW = Surface water monitoring  $\rightarrow$  NRSP-SW locations

SD = Outfall monitoring  $\rightarrow$  NRWDR-SW locations

 $GW = Groundwater monitoring \rightarrow NRSP-GW locations$ 

Parameter	Program	Units	Analysis Method
Estimated Flow	SW and SD	ft <sup>3</sup> /s	Current Meter
Depth to Groundwater	GW Only	ft	Water Level Indicator
Water Temperature	SW, SD and GW	degrees Celsius (°C)	Probe/Meter
pH	SW, SD and GW	standard units (s.u.)	Probe/Meter
Turbidity	SW and SD	NTU	Probe/Meter
Specific Conductivity	SW, SD, and GW	µS/cm	Probe/Meter
Dissolved Oxygen	SW, SD and GW	mg/L	Probe/Meter
Total Dissolved Solids (TDS)	SW and GW	mg/L	Probe/Meter

#### **Table B1-1: Field Measurements**

**Table B1-2: Toxicity** 

Parameter	Program	Units	Analysis Method
Acute Toxicity	SW Only	% Survival	EPA 821-R-02-012
Chronic Toxicity	SW Only	toxicity units (TUc)	EPA 821-R-02-013

#### Table B1-3: Bacteria

Parameter	Program	Units	Analysis Method
E. coli	SW and SD	MPN/100mL	SM 9221 F

#### **Table B1-4: Bioassessment**

Parameter	Program	Units	Analysis Method
Physical Habitat Survey	SW Only	Score (0-200)	SWAMP 2007
Benthic Macroinvertebrate Index	SW Only	Score (0-100)	SoCal-IBI



Parameter	Program	Units	Analysis Method
Ammonia-N	SW, SD and GW	mg/L	EPA 350.1
Biochemical Oxygen Demand (BOD)	SW Only	mg/L	SM 5210 B
Chloride	SW, SD and GW	mg/L	EPA 300.0
Chlorine, Total Residual	SW and GW	mg/L	SM 4500 Cl G
Cyanide, Total	SW and GW	mg/L	EPA 335.4
Dissolved Oxygen (DO)*	SW, SD and GW	mg/L	SM4500 O-G
Fluoride, Total	SW and GW	mg/L	EPA 300.0
MBAS	SW Only	mg/L	SM 5540 C
NID as CTAS	SW Only	mg/L	SM 5540 D
Nitrate-N	SW, SD and GW	mg/L	EPA 300.0
Nitrite-N	SW, SD and GW	mg/L	EPA 300.0
Nitrogen, Total	SW and GW	mg/L	Calculation
Oil and Grease	SW Only	mg/L	EPA 1664 A
Orthophosphate-P	SW Only	mg/L	EPA 365.3
Perchlorate	GW Only	μg/L	EPA 314.0
Phosphorus, Total as P	SW, SD and GW	mg/L	EPA 365.3
Settleable Solids	SW Only	mg/L	SM 2540 F
Sulfate	SW and GW	mg/L	EPA 300.0
Total Dissolved Solids (TDS)*	SW and GW	mg/L	SM 2540 C
Total Hardness (as CaCO3)	SW, SD and GW	mg/L	EPA 200.7
Total Suspended Solids (TSS)	SW and SD	mg/L	SM 2540 D

#### **Table B1-5: Inorganics and General Chemistry**

\* Samples may be taken for laboratory analysis if DO and TDS cannot be measured in the field


Table B1-6: Metals

Parameter	Program	Units	Analysis Method
Antimony, Total	SW, SD and GW	µg/L	EPA 1640
Antimony, Dissolved	SW, SD and GW	μg/L	EPA 200.8
Arsenic, Total	SW, SD and GW	μg/L	EPA 1640
Arsenic, Dissolved	SW, SD and GW	μg/L	EPA 200.8
Barium, Total	SW, SD and GW	μg/L	EPA 1640
Barium, Dissolved	SW, SD and GW	μg/L	EPA 200.8
Beryllium, Total	SW, SD and GW	μg/L	EPA 1640
Beryllium, Dissolved	SW, SD and GW	μg/L	EPA 200.8
Boron, Total & Dissolved	SW, SD and GW	μg/L	EPA 200.8
Cadmium, Total	SW, SD and GW	μg/L	EPA 1640
Cadmium, Dissolved	SW, SD and GW	μg/L	EPA 200.8
Chromium, Total	SW, SD and GW	μg/L	EPA 200.8
Chromium-3	SW, SD and GW	μg/L	Calculation
Chromium-6	SW, SD and GW	μg/L	EPA 218.6
Copper, Total	SW, SD and GW	μg/L	EPA 1640
Copper, Dissolved	SW, SD and GW	μg/L	EPA 200.8
Iron, Total	SW, SD and GW	μg/L	EPA 1640
Iron, Dissolved	SW, SD and GW	μg/L	EPA 200.8
Lead, Total	SW, SD and GW	μg/L	EPA 1640
Lead, Dissolved	SW, SD and GW	μg/L	EPA 200.8
Mercury, Total & Dissolved	SW, SD and GW	μg/L	EPA 245.1
Nickel, Total	SW, SD and GW	μg/L	EPA 1640
Nickel, Dissolved	SW, SD and GW	μg/L	EPA 200.8
Selenium, Total	SW, SD and GW	μg/L	EPA 1640
Selenium, Dissolved	SW, SD and GW	μg/L	EPA 200.8
Silver, Total	SW, SD and GW	μg/L	EPA 1640
Silver, Dissolved	SW, SD and GW	μg/L	EPA 200.8
Thallium, Total	SW, SD and GW	μg/L	EPA 1640
Thallium, Dissolved	SW, SD and GW	μg/L	EPA 200.8
Zinc, Total	SW, SD and GW	μg/L	EPA 1640
Zinc, Dissolved	SW, SD and GW	μg/L	EPA 200.8



Parameter	Program	Units	Analysis Method
2,4,5-T	SW and GW	µg/L	EPA 8151A
2,4,5-TP	SW and GW	µg/L	EPA 8151A
2,4-D	SW and GW	µg/L	EPA 8151A
2,4-DB	SW and GW	µg/L	EPA 8151A
3,5-Dichlorobenzoic Acid	SW and GW	µg/L	EPA 8151A
4-Nitrophenol	SW and GW	µg/L	EPA 8151A
Aciflurofen	SW and GW	µg/L	EPA 8151A
Bentazon	SW and GW	µg/L	EPA 8151A
Dalapon	SW and GW	µg/L	EPA 8151A
DCPA	SW and GW	µg/L	EPA 8151A
Dicamba	SW and GW	µg/L	EPA 8151A
Dichloroprop	SW and GW	µg/L	EPA 8151A
Dinoseb	SW and GW	µg/L	EPA 8151A
MCPA	SW and GW	µg/L	EPA 8151A
MCPP	SW and GW	µg/L	EPA 8151A
Pentachlorophenol	SW and GW	µg/L	EPA 8151A
Picloram	SW and GW	µg/L	EPA 8151A

### **Table B1-7: Chlorinated Herbicides**

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Parameter	Program	Units	Analysis Method
4,4'-DDD	SW, SD and GW	μg/L	EPA 608
4,4'-DDE	SW, SD and GW	μg/L	EPA 608
4,4'-DDT	SW, SD and GW	μg/L	EPA 608
Aldrin	SW, SD and GW	μg/L	EPA 608
Aroclor 1016	SW, SD and GW	μg/L	EPA 608
Aroclor 1221	SW, SD and GW	μg/L	EPA 608
Aroclor 1232	SW, SD and GW	μg/L	EPA 608
Aroclor 1242	SW, SD and GW	μg/L	EPA 608
Aroclor 1248	SW, SD and GW	μg/L	EPA 608
Aroclor 1254	SW, SD and GW	μg/L	EPA 608
Aroclor 1260	SW, SD and GW	μg/L	EPA 608
BHC-alpha	SW, SD and GW	μg/L	EPA 608
BHC-beta	SW, SD and GW	μg/L	EPA 608
BHC-delta	SW, SD and GW	μg/L	EPA 608
BHC-gamma (Lindane)	SW, SD and GW	μg/L	EPA 608
Chlordane	SW, SD and GW	μg/L	EPA 608
Dieldrin	SW, SD and GW	μg/L	EPA 608
Endosulfan sulfate	SW, SD and GW	μg/L	EPA 608
Endosulfan I	SW, SD and GW	μg/L	EPA 608
Endosulfan II	SW, SD and GW	μg/L	EPA 608
Endrin	SW, SD and GW	μg/L	EPA 608
Endrin aldehyde	SW, SD and GW	μg/L	EPA 608
Heptachlor	SW, SD and GW	μg/L	EPA 608
Heptachlor epoxide	SW, SD and GW	μg/L	EPA 608
Methyozychlor	SW, SD and GW	μg/L	EPA 608
Toxaphene	SW, SD and GW	μg/L	EPA 608

### **Table B1-8: Organochlorine Pesticides and PCBs**



Parameter	Program	Units	Analysis Method
Azinphos methyl	SW, SD and GW	μg/L	EPA 8141A
Bolstar (Sulprofos)	SW, SD and GW	μg/L	EPA 8141A
Chlorpyrifos	SW, SD and GW	μg/L	EPA 8141A
Coumaphos	SW, SD and GW	μg/L	EPA 8141A
Demeton-o	SW, SD and GW	μg/L	EPA 8141A
Demeton-s	SW, SD and GW	μg/L	EPA 8141A
Diazinon	SW, SD and GW	μg/L	EPA 8141A
Dichlorvos	SW, SD and GW	μg/L	EPA 8141A
Dimethoate	SW, SD and GW	μg/L	EPA 8141A
Disulfoton	SW, SD and GW	μg/L	EPA 8141A
Ethoprop (Ethoprofos)	SW, SD and GW	μg/L	EPA 8141A
Ethyl parathion	SW, SD and GW	μg/L	EPA 8141A
Fenchlorophos (Ronnel)	SW, SD and GW	μg/L	EPA 8141A
Fensulfothion	SW, SD and GW	μg/L	EPA 8141A
Fenthion	SW, SD and GW	μg/L	EPA 8141A
Malathion	SW, SD and GW	μg/L	EPA 8141A
Merphos	SW, SD and GW	μg/L	EPA 8141A
Methyl Parathion	SW, SD and GW	μg/L	EPA 8141A
Mevinphos (Phosdrin)	SW, SD and GW	μg/L	EPA 8141A
Naled	SW, SD and GW	μg/L	EPA 8141A
Phorate	SW, SD and GW	μg/L	EPA 8141A
Tetrachlorvinphos (Stirophos)	SW, SD and GW	μg/L	EPA 8141A
Trhionazin	SW, SD and GW	μg/L	EPA 8141A
Tokuthion	SW, SD and GW	μg/L	EPA 8141A
Trichloronate	SW, SD and GW	μg/L	EPA 8141A

### Table B1-9: Organophosphorus Pesticides



Parameter	Program	Units	Analysis Method
Allethrin	SW, SD and GW	ng/L	GC/MS NCI-SIM
Bifenthrin	SW, SD and GW	ng/L	GC/MS NCI-SIM
Cyfluthrin	SW, SD and GW	ng/L	GC/MS NCI-SIM
Cypermethrin	SW, SD and GW	ng/L	GC/MS NCI-SIM
Deltamethrin/Tralomethrin	SW, SD and GW	ng/L	GC/MS NCI-SIM
Danitol (Fenpropathrin)	SW, SD and GW	ng/L	GC/MS NCI-SIM
Deltamethrin	SW, SD and GW	ng/L	GC/MS NCI-SIM
Dichloran	SW, SD and GW	ng/L	GC/MS NCI-SIM
Esfenvalerate	SW, SD and GW	ng/L	GC/MS NCI-SIM
Fenvalerate	SW, SD and GW	ng/L	GC/MS NCI-SIM
L-Cyhalothrin	SW, SD and GW	ng/L	GC/MS NCI-SIM
Pendimethalin	SW, SD and GW	ng/L	GC/MS NCI-SIM
Permethrin	SW, SD and GW	ng/L	GC/MS NCI-SIM
Prallethrin	SW, SD and GW	ng/L	GC/MS NCI-SIM
Sumithrin	SW, SD and GW	ng/L	GC/MS NCI-SIM
Tefluthrin	SW, SD and GW	ng/L	GC/MS NCI-SIM

### Table B1-10: Pyrethroid Pesticides

### Table B1-11: Semivolatile Organic Compounds (including PAHs)

Parameter	Program	Units	Analysis Method
N-Nitrosodimethylamine (NDMA)	SW and GW	ng/L	EPA 1625M
1-Methylnaphthalene	SW and GW	μg/L	EPA 8270C-SIM
2-Methylnaphthalene	SW and GW	μg/L	EPA 8270C-SIM
Acenaphtene	SW, SD and GW	μg/L	EPA 8270C-SIM
Acenaphthylene	SW, SD and GW	μg/L	EPA 8270C-SIM
Anthracene	SW, SD and GW	μg/L	EPA 8270C-SIM
Benzo[a]anthracene	SW, SD and GW	μg/L	EPA 8270C-SIM
Benzo[a]pyrene	SW, SD and GW	μg/L	EPA 8270C-SIM
Benzo[b]fluoranthene	SW, SD and GW	μg/L	EPA 8270C-SIM
Benzo[g,h,i]perylene	SW, SD and GW	μg/L	EPA 8270C-SIM
Benzo[k]fluoranthene	SW, SD and GW	μg/L	EPA 8270C-SIM
Chrysene	SW, SD and GW	μg/L	EPA 8270C-SIM
Dibenzo(a,h)anthracene	SW, SD and GW	μg/L	EPA 8270C-SIM
Fluoranthene	SW, SD and GW	μg/L	EPA 8270C-SIM
Fluorene	SW, SD and GW	μg/L	EPA 8270C-SIM
Indeno(1,2,3-cd)pyrene	SW, SD and GW	μg/L	EPA 8270C-SIM
Naphthalene	SW, SD and GW	μg/L	EPA 8270C-SIM
Phenanthrene	SW, SD and GW	μg/L	EPA 8270C-SIM
Pyrene	SW, SD and GW	μg/L	EPA 8270C-SIM



Parameter	Program	Units	Analysis Method
1,1,1-Trichloroethane	SW, SD and GW	µg/L	EPA 624
1,1,2,2,-Tetrachloroethane	SW, SD and GW	μg/L	EPA 624
1,1,2-Trichloroethane	SW, SD and GW	μg/L	EPA 624
1,1-Dichloroethane	SW, SD and GW	μg/L	EPA 624
1,1-Dichloroethene	SW, SD and GW	μg/L	EPA 624
1,2-Dichloroethane	SW, SD and GW	μg/L	EPA 624
1,2-Dichloropropane	SW, SD and GW	μg/L	EPA 624
2-Butanone	SW, SD and GW	μg/L	EPA 624
2-Chloroethyl vinyl ether	SW, SD and GW	μg/L	EPA 624
2-Hexanone	SW, SD and GW	μg/L	EPA 624
4-Methyl-2-pentanone	SW, SD and GW	μg/L	EPA 624
Acetone	SW, SD and GW	μg/L	EPA 624
Acrolein	SW, SD and GW	μg/L	EPA 624
Acrylonitrile	SW, SD and GW	μg/L	EPA 624
Benzene	SW, SD and GW	μg/L	EPA 624
Bromodichloromethane	SW, SD and GW	μg/L	EPA 624
Bromoform	SW, SD and GW	µg/L	EPA 624
Bromomethane	SW, SD and GW	µg/L	EPA 624
Carbon Disulfide	SW, SD and GW	µg/L	EPA 624
Carbon tetrachloride	SW, SD and GW	µg/L	EPA 624
Chlorobenzene	SW, SD and GW	µg/L	EPA 624
Chloroethane	SW, SD and GW	µg/L	EPA 624
Chloroform	SW, SD and GW	µg/L	EPA 624
Chloromethane	SW, SD and GW	µg/L	EPA 624
cis-1,3-Dichloropropene	SW, SD and GW	µg/L	EPA 624
Dibromochloromethane	SW, SD and GW	μg/L	EPA 624
Ethylbenzene	SW, SD and GW	μg/L	EPA 624
m-Dichlorobenzene	SW, SD and GW	μg/L	EPA 624
Methyl tert-butyl ether (MTBE)	SW, SD and GW	μg/L	EPA 524.2
Methylene chloride	SW, SD and GW	μg/L	EPA 624
o-Dichlorobenzene	SW, SD and GW	μg/L	EPA 624
p-Dichlorobenzene	SW, SD and GW	μg/L	EPA 624
Tetrachloroethene	SW, SD and GW	μg/L	EPA 624
Toluene	SW, SD and GW	μg/L	EPA 624
trans-1,2-Dichloroethene	SW, SD and GW	μg/L	EPA 624
trans-1,3-Dichloroethene	SW, SD and GW	µg/L	EPA 624
Trichloroethene	SW, SD and GW	µg/L	EPA 624
Vinyl chloride	SW, SD and GW	μg/L	EPA 624

### Table B1-12: Volatile Organic Compounds

### 2 SURFACE WATER AND STORM DRAIN SAMPLE COLLECTION REQUIREMENTS

Parameter	Bottle Type	Bottle Size	Preservative	Holding Time (days)	Analysis Method
Acute Toxicity*	Polyethylene	1 gal	< 6°C	1.5	EPA 821-R-02-012
Ammonia-N*	Polyethylene	250 mL	< 6°C, H2SO4	28	EPA 350.1
Bacteria – Coliform*	Sterile Polyethylene	125 mL	< 6°C	0.25	SM 9221B/F
Biochemical Oxygen Demand (BOD)	Polyethylene	1 L	< 6°C	2	SM 5210 B
Boron	Polyethylene	250 mL	HNO3	180	EPA 200.8
Chloride	Polyethylene	250 mL	< 6°C	28	EPA 300.0
Chlorinated Herbicides	Amber Glass	1 L	< 6°C	7	EPA 8151A
Chlorine, Total Residual	Polyethylene	250 mL	< 6°C	0.01	SM 4500 Cl G
Chromium, Total	Polyethylene	250 mL	HNO3	180	EPA 200.8
Chromium-6	Polyethylene	250 mL	< 6°C, (NH4)2SO4	1	EPA 218.6
Chronic Toxicity	Cubitainer	10 L	< 6°C	1.5	EPA 821-R-02-013
Cyanide, Total	Polyethylene	500 mL	< 6°C, NaOH	14	EPA 335.4
Dissolved Oxygen (DO)	Polyethylene	250 mL	< 6°C	0.01	SM 4500 O-G
Fluoride, Total	Polyethylene	250 mL	< 6°C	28	EPA 300.0
MBAS	Polyethylene	500 mL	< 6°C	2	SM 5540 C
Mercury	Polyethylene	250 mL	HNO3	28	EPA 245.1
Metals, Total	Polyethylene	1000 mL	HNO3	180	EPA 1640
Metals, Dissolved	Polyethylene	250 mL	HNO3	180	EPA 200.8
NID as CTAS	Polyethylene	1 L	< 6°C	2	SM 5540 D
Nitrate-N	Polyethylene	250 mL	< 6°C	2	EPA 300.0
Nitrite-N	Polyethylene	250 mL	< 6°C	2	EPA 300.0
Oil and Grease*	Glass Wide Mouth	1 L	< 6°C, HCl	28	EPA 1664 A
Organochlorine Pesticides*	Amber Glass	1 L	< 6°C	7	EPA 608
Organophosphorus Pesticides*	Amber Glass	1 L	< 6°C	7	EPA 8141A
Orthophosphate-P	Polyethylene	250 mL	< 6°C, Filtered	0.01	EPA 365.3
Phosphorus, Total as P	Polyethylene	250 mL	< 6°C, H2SO4	28	EPA 365.3
Pyrethroid Pesticides	Amber Glass	1 L	< 6°C	7	GC/MS NCI-SIM
Semivolatile Organic Compounds*	Amber Glass	1 L	< 6°C	7	EPA 8270 SIM

# Table B2-1: Surface Water & Outfall Sample container, preservative, & holding requirements

# 

Parameter	Bottle Type	Bottle Size	Preservative	Holding Time (days)	Analysis Method
Settleable Solids	Polyethylene	1 L	< 6°C	2	SM 2540 F
Sulfate	Polyethylene	250 mL	< 6°C	28	EPA 300.0
Total Dissolved Solids (TDS)	Polyethylene	500 mL	< 6°C	7	SM 2540 C
Total Hardness (as CaCO3)	Polyethylene	300 mL	< 6°C, HNO3	180	EPA 200.7
Total Suspended Solids (TSS)	Polyethylene	500 mL	< 6°C	7	SM 2540 D
Volatile Organic Compounds*	Glass	40 mL	< 6°C, HCl	14	EPA 624

\* Samples will be collected as grab samples as specified in the Caltrans Guidance Manual: Storm Water Monitoring Protocols, some parameters not presently noted as such may be collected as grab samples at a later time

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### **3** GROUNDWATER SAMPLE COLLECTION REQUIREMENTS

Parameter	Bottle Type	Bottle Size	Preservative	Holding Time (days)	Analysis Method
Ammonia-N	Polyethylene	250 mL	< 6°C, H2SO4	28	EPA 350.1
Boron	Polyethylene	250 mL	HNO3	180	EPA 200.8
Chloride	Polyethylene	250 mL	< 6°C	28	EPA 300.0
Chlorinated Herbicides	Amber Glass	1 L	< 6°C	7	EPA 8151A
Chlorine, Total Residual	Polyethylene	250 mL	< 6°C	0.01	SM 4500 Cl G
Chromium, Total	Polyethylene	250 mL	HNO3	180	EPA 200.8
Chromium-6	Polyethylene	250 mL	< 6°C, (NH4)2SO4	1	EPA 218.6
Cyanide, Total	Polyethylene	500 mL	< 6°C, NaOH	14	EPA 335.4
Dissolved Oxygen (DO)	Polyethylene	250 mL	< 6°C	0.01	SM 4500 O-G
Fluoride, Total	Polyethylene	250 mL	< 6°C	28	EPA 300.0
Mercury	Polyethylene	250 mL	HNO3	28	EPA 245.1
Metals, Total	Polyethylene	1000 mL	HNO3	180	EPA 1640
Metals, Dissolved	Polyethylene	250 mL	HNO3	180	EPA 200.8
Nitrate-N	Polyethylene	250 mL	< 6°C	2	EPA 300.0
Nitrite-N	Polyethylene	250 mL	< 6°C	2	EPA 300.0
Organochlorine Pesticides	Amber Glass	1 L	< 6°C	7	EPA 608
Organophosphorus Pesticides	Amber Glass	1 L	< 6°C	7	EPA 8141A
Phosphorus, Total as P	Polyethylene	250 mL	< 6°C, H2SO4	28	EPA 365.3
Pyrethroid Pesticides	Amber Glass	1 L	< 6°C	7	GC/MS NCI-SIM
Perchlorate	Polyethylene	300 mL	< 6°C	28	EPA 314.0
Semivolatile Organic Compounds	Amber Glass	1 L	< 6°C	7	EPA 8270 SIM
Sulfate	Polyethylene	250 mL	< 6°C	28	EPA 300.0
Total Dissolved Solids (TDS)	Polyethylene	500 mL	< 6°C	7	SM 2540 C
Total Hardness (as CaCO3)	Polyethylene	300 mL	< 6°C, HNO3	180	EPA 200.7
Volatile Organic Compounds	Glass	40 mL	< 6°C, HCl	14	EPA 624

### Table B3-1: Groundwater Sample container, preservative, and holding requirements

Prepared for

Newhall Land 25124 Springfield Court, 3rd Floor Valencia, California 91355-1088

# Appendix C

### **Monitoring Parameters Water Quality Objectives**

for the Newhall Ranch Specific Plan Water Quality Monitoring Plan Conditions of Approval and Clean Water Act Section 401 Water Quality Certification and Waste Discharge Requirements



engineers | scientists | innovators

924 Anacapa Street, Suite 4A Santa Barbara, CA 93101

Project Number LA0170

March 27, 2013

		Los Angeles Basin	California Toxic Rule -Freshwater	
		Diam <sup>3</sup>	<b>Criterion Maximum</b>	Criterion Continuous
Parameter	Units	Plan	Concentration	Concentration
1,1,1-Trichloroethane	μg/L			
1,1,2,2,-Tetrachloroethane	μg/L			
1,1,2-Trichloroethane	μg/L			
1,1-Dichloroethane	μg/L			
1,1-Dichloroethene	μg/L			
1,2-Dichloroethane	μg/L			
1,2-Dichloropropane	μg/L			
2-Butanone	μg/L			
2-Chloroethyl vinyl ether	μg/L			
2-Hexanone	μg/L			
4,4'-DDD	μg/L			
4,4'-DDE	μg/L			
4,4'-DDT	μg/L		1.1	0.001
4-Methyl-2-pentanone	μg/L			
Acenaphtene	μg/L			
Acenaphthylene	μg/L			
Acetone	μg/L			
Acrolein	μg/L			
Acrylonitrile	μg/L			
Aldrin	μg/L		3	
Allethrin	ng/L			
Ammonia-N	μg/L	1.2		
Anthracene	μg/L			
Antimony, Dissolved	μg/L			
Antimony, Total	μg/L			
Aroclor 1016	μg/L	0.014		0.014
Aroclor 1221	μg/L	0.014		0.014
Aroclor 1232	μg/L	0.014		0.014
Aroclor 1242	μg/L	0.014		0.014
Aroclor 1248	μg/L	0.014		0.014
Aroclor 1254	μg/L	0.014		0.014
Aroclor 1260	μg/L	0.014		0.014
Arsenic, Dissolved	μg/L		340	150
Arsenic, Total	μg/L		340	150
Azinphos methyl	μg/L			
Barium, Dissolved	μg/L			
Barium, Total	μg/L			
Benzene	μg/L			
Benzo[a]anthracene	μg/L			
Benzo[a]pyrene	μg/L			
Benzo[b]fluoranthene	μg/L			
Benzo[g,h,i]perylene	μg/L			
Benzo[k]fluoranthene	μg/L			
Beryllium, Dissolved	μg/L			
Beryllium, Total	μg/L			
BHC-alpha	μg/L			
BHC-beta	μg/L			
BHC-delta	μg/L			
BHC-gamma (Lindane)	μg/L		0.95	

		Los Angeles Basin	California Toxic Rule -Freshwater	
		Dian <sup>3</sup>	<b>Criterion Maximum</b>	<b>Criterion Continuous</b>
Parameter	Units	Fidii	Concentration	Concentration
Bifenthrin	ng/L			
Bolstar (Sulprofos)	μg/L			
Boron, Total	mg/L	1.5		
Bromodichloromethane	μg/L			
Bromoform	μg/L			
Bromomethane	μg/L			
Cadmium, Dissolved <sup>1</sup>	μg/L		12	4.6
Cadmium, Total <sup>1</sup>	μg/L		13	5.3
Carbon Disulfide	μg/L			
Carbon tetrachloride	μg/L			
Chlordane	μg/L		2.4	0.0043
Chloride	mg/L	100		
Chlorobenzene	μg/L			
Chloroethane	μg/L			
Chloroform	μg/L			
Chloromethane	μg/L			
Chlorpyrifos	μg/L			
Chromium, Total	μg/L			
Chromium-3 <sup>1</sup>	μg/L		3800	460
Chromium-6	μg/L		16	11
Chrysene	μg/L			
cis-1,3-Dichloropropene	μg/L			
Copper, Dissolved <sup>1</sup>	μg/L		35	21
Copper, Total <sup>1</sup>	μg/L		13	9
Coumaphos	μg/L			
Cyfluthrin	ng/L			
Cypermethrin	ng/L			
Danitol (Fenpropathrin)	ng/L			
Deltamethrin	ng/L			
Demeton-o	μg/L			
Demeton-s	μg/L			
Diazinon	μg/L			
Dibenzo(a,h)anthracene	μg/L			
Dibromochloromethane	μg/L			
Dichloran	ng/L			
Dichlorvos	μg/L			
Dieldrin	μg/L		0.24	0.056
Dimethoate	μg/L			
Dissolved Oxygen <sup>2</sup>	mg/L	5		
Disulfoton	μg/L			
E. coli	MPN/100mL	235		
Endosulfan I	μg/L		0.22	0.056
Endosulfan II	μg/L		0.22	0.056
Endosulfan sulfate	μg/L			
Endrin	μg/L		0.086	0.036
Endrin aldehyde	μg/L			
Esfenvalerate	ng/L			
Ethoprop (Ethoprofos)	μg/L			

		Los Angeles Basin	California Toxic	Rule -Freshwater
		Plan <sup>3</sup>	<b>Criterion Maximum</b>	<b>Criterion Continuous</b>
Parameter	Units	Fiall	Concentration	Concentration
Ethyl parathion	μg/L			
Ethylbenzene	μg/L			
Fenchlorophos (Ronnel)	μg/L			
Fensulfothion	μg/L			
Fenthion	μg/L			
Fenvalerate	ng/L			
Fluoranthene	μg/L			
Fluorene	μg/L			
Heptachlor	μg/L		0.52	0.0038
Heptachlor epoxide	μg/L		0.52	0.0038
Indeno(1,2,3-cd)pyrene	μg/L			
Iron, Dissolved	μg/L			
Iron, Total	μg/L			
L-Cyhalothrin	ng/L			
Lead, Dissolved <sup>1</sup>	μg/L		180	7.1
Lead. Total <sup>1</sup>	ug/L		280	11
Malathion	<u>بع، -</u> ارم/ا			
m-Dichlorobenzene	μ <u>β</u> / Ε			
Mercury Total	μ <u>σ</u> /Γ			
Merchos	μ <u>σ</u> /Γ			
Methyl Parathion	μ <u>σ</u> /Ι			
Methyl tert-butyl ether (MTBE)	μ <u>σ</u> /Ι			
Methylene chloride	μ <u>σ</u> /Ι			
Methyozychlor	μ <u>σ/</u> ι			
Mevinphos (Phosdrin)	<u>لعم</u> ر ا			
Naled	ug/L			
Naphthalene	ug/L			
Nickel Dissolved <sup>1</sup>	ug/L		1100	120
Nickel Total <sup>1</sup>	но/I		1100	120
Nitrate-N	mg/l	10		
Nitrite-N	mg/l	1		
o-Dichlorobenzene	116/L			
p-Dichlorobenzene	<u>لعمار الم</u>			
Pendimethalin	ng/i			
Permethrin	ng/L			
	8/ -	6.5 to 8.5		
рΗ	pH Units	and 0.5 units		
		change from		
Phenanthrene	μg/L			
Phorate	μg/L			
Phosphorus, Total as P	mg/L			
Prallethrin	ng/L			
Pyrene	μg/L			
Selenium, Dissolved	μg/L			
Selenium, Total	μg/L			5
Silver, Dissolved <sup>1</sup>	μg/L		18	
Silver. Total <sup>1</sup>	με/Γ		21	
Specific Conductivity	us/cm			
	μ3/ τη			L

		Los Angeles Basin	California Toxic	Rule -Freshwater
Parameter	Units	Plan <sup>3</sup>	Criterion Maximum Concentration	Criterion Continuous Concentration
Sumithrin	ng/L			
Tefluthrin	μg/L			
Tetrachloroethene	μg/L			
Tetrachlorvinphos (Stirophos)	μg/L			
Thallium, Dissolved	μg/L			
Thallium, Total	μg/L			
Tokuthion	μg/L			
Toluene	μg/L			
Total Hardness (as CaCO3)	mg/L			
Total Suspended Solids (TSS)	mg/L			
Toxaphene	μg/L		0.73	0.0002
Tralomethrin	ng/L			
trans-1,2-Dichloroethene	μg/L			
trans-1,3-Dichloroethene	μg/L			
Trhionazin	μg/L			
Trichloroethene	μg/L			
Trichloronate	μg/L			
Turbidity	NTU	Natural Turbidity (Max Increase) 0 to 50 (20%) > 50 (10%)		
Vinyl chloride	μg/L			
Water Temperature	degrees Celsius (°C)			
Zinc, Dissolved <sup>1</sup>	μg/L		270	270
Zinc, Total <sup>1</sup>	μg/L		270	270

1. Concentrations shown for CTR Freshwater Metals objectives were determined using an average receiving water hardness of 263 mg/L, based on NPDES WRP sampling at station RSW-001U from 2008 to 2012. Actual objectives will be determined based on the sample specific hardness

2. Water quality objective is a minimum value

3. The strictest basin plan objective for inland surface waters with beneficial uses applicable to Santa Clara River reach 5 (IND, PROC, AGR, GWR, FRSH, REC1, REC2, WARM, WILD, RARE, WET) and TMDLs for bacteria, nutrients, and chloride

Prepared for

Newhall Land 25124 Springfield Court, 3rd Floor Valencia, California 91355-1088

## **Appendix D**

### Field Data and Chain of Custody Forms

for the Newhall Ranch Specific Plan Water Quality Monitoring Plan Conditions of Approval and Clean Water Act Section 401 Water Quality Certification and Waste Discharge Requirements



engineers | scientists | innovators

924 Anacapa Street, Suite 4A Santa Barbara, CA 93101

Project Number LA0170

March 27, 2013

### Instream Surface Water Quality Monitoring Field Data Collection Form



Sample Location:		STREAM FL	OW:		
Staff:		_ Discharge at T	ime of Collect	ion:c	fs () Measured () From Gage
Project Name:	Project #:	_ If measured, at	tach stream di	scharge measure	ement sheet.
Date:	Time:	If estimated from	om stream gag	e, time of Gage	Reading:
FIELD OBSERVATIONS:		Gage ID:			
Weather Conditions:	Air Temperature:	-			
Oil, grease, scum, solids, or sludge	deposits present?	INSTRUMEN	T DATA:		
Significant turbidity?	Foam present?	Meter Type:			_ Serial #:
Algal blooms present?	Odors present?	Calibration Da	te:		Calibration By:
Comments and Observations:		Calibration:	PH:	4.0 std.	=
		-			
SAMPLING INFORMATION:					
Sample Method:					
Time Start:	Time End:	_			
Laboratory:		_			
Tests Performed:					
Field Blank: C	Cooler Temperature:				

Instream Surface Water Quality Monitoring Field Data Collection Form

### Geosyntec consultants

#### FIELD MEASUREMENTS:

Sample			Ter (°	mp. C)						р (s.	H u.)					J	Гurb (N7	idity (U)	Ÿ		
1																					
2																					
(3)																					
(4)																					
(5)																					
Sample Median																					
Composite Median																					

Sample			Т (р	DS pm)					Sp	ecifi	c Co (µS/	ondu (cm)	ictivi	ity					D (mg	O g/L)			
1																							
2																							
(3)																							
(4)																							
(5)																							
Sample Median																							
Composite Median																							

### Storm Drain Water Quality Monitoring Field Data Collection Form



<b>AUTOMATED</b>	<b>EQUIPMENT:</b>
------------------	-------------------

Sample Location:		AUTOMATED EQUIPMENT:	
Staff:		Automated flow sampler functioning prop	perly:
Project Name:	Project #:	Automated composite sampler functioning	g properly:
Date:	Time:	Approximate sample bottle capacity rema	ining (%):
FIELD OBSERVATIONS:			
Weather Conditions:	Air Temperature:	<b>INSTRUMENT DATA:</b>	
Oil, grease, scum, solids, or sludge d	leposits present?	Meter Type:	Serial #:
Significant turbidity?	Foam present?	Calibration Date:	Calibration By:
Algal blooms present?	Odors present?	Calibration:	_
Comments and Observations:		F11. 4.0 Stu	. –
		-	

#### **SAMPLING INFORMATION:**

Sample Method: \_\_\_\_\_

Time Start:\_\_\_\_\_ Time End: \_\_\_\_\_

Laboratory: \_\_\_\_\_

Tests Performed:

Field Blank: \_\_\_\_\_ Cooler Temperature: \_\_\_\_\_

### Storm Drain Water Quality Monitoring Field Data Collection Form

# Geosyntec Consultants

### FIELD MEASUREMENTS:

Sample			Ten (°C	n <b>p.</b> C)						р (s.	H u.)					5	Furl (N	bidit ГU)	у		
1																					
2																					
(3)																					
(4)																					
(5)																					
Sample Median																					
Composite Median																					

Sample		Sp	ecifi	ic Co (µS/	ondu /cm)	ıctiv	ity					D (mg	0 g/L)			
1																
2																
(3)																
(4)																
(5)																
Sample Median																
Composite Median																

### PURGE & SAMPLE FIELD REPORT

Sample Point ID:	Field Rep.:
Project Name:	Project #:
Facility:	Location:
Sample Matrix:	

### **PURGE INFORMATION:**

Dedicated: Y / N
Pump Inlet Depth:
One Casing Vol.:
Total Vol. Purged:
Well Purged to Dryness: Y / N
GW Level After Purge:
Date/Time Completed:

\_\_\_\_\_

### **PURGE DATA:**

Time	Purge Rate ( )	Total Volume Removed	Temp. (°C)	рН	Conduct. (mS/cm)	Turb. (NTU)	DO (mg/L)	<b>Sal.</b> ( )	ORP (mV)

# Geosyntec <sup>></sup>

### consultants

### **SAMPLING INFORMATION:**

Sample Method:			Dedicated:	Y	/	Ν
Water Level at Samplin	g:					
Sampling Frequency: A	Annual ( )	Semi-Annual ()	Quarterly ()			
Ν	fonthly ()	Initial ()	Other ( ):			_

### **SAMPLING DATA:**

Date/ Time	Sample Rate ( )	Temp. (°C)	рН	Conduct. (mS/cm)	Turb. (NTU)	DO (mg/L)	<b>Sal.</b> ( )	ORP (mV)

**Pump Parameters:** 

Refill Time (sec):	Discharge Time (sec):	
Pressure Setting (psi):	Others ():	
Sample Characteristics:		
Comment and Observations:		

### **INSTRUMENT DATA:**

Meter Type:		Serial #:
Calibration Dat	te:	Calibration By:
Calibration:	Turbidity:	0 NTU std. = NTU
	PH:	4.0 std. =
	Conductivity:	$4.49 \text{ mS/cm} = \ \text{mS/cm}$

### SAMPLE COLLECTION DATA:

Laboratory:			
Tests Performed:			

Trip Blank Date: \_\_\_\_\_ Field Blank Date: \_\_\_\_\_

### **GENERAL INFORMAITON:**

Weather Conditions at Time of Sampling: General Comments and Observations:

14859 East Clark	Weck Laboratories, Inc. Analytical Laboratory Services - Since 1964								CHAIN OF CUSTODY RECORD								ORD	
Tel 626-336-213	39 ♦ Fax 62	26-336-263	4 🔶 v	ww.wecklabs.co	m											Page	1	_Of1
CLIENT NAME: ADDRESS:				PROJECT:					ANA	ALYSE	SRE	QUES	TED				L HAN Same D 24 Hour 48-72 H	NDLING Pay Rush 150% Rush 100% our Rush 75%
				FAX: EMAIL:													4 - 5 Day Rush Ex 10 - 15 F	y Rush 30% «tractions 50% Business Days
PROJECT MANAGE	R			SAMPLER												Charges will	QA/QC I apply fo	Data Package or weekends/holidays
ID# (For lab Use Only)	DATE SAMPLED	TIME SAMPLED	SMPL TYPE	SAMPLE IDENTIFIC	ATION/SITE LOCATION	# OF CONT.										Method of S COMMENTS	nipment	:
												+	-					
RELINQUISHED	BY		DAT	L E / TIME	RECEIVED	) BY							<b>S</b> Actua	AMP al Tem	LE C(	ONDITION: re:		SAMPLE TYPE CODE AQ=Aqueous NA= Non Aqueous SL = Sludge
RELINQUISHED	BY		DAT	E / TIME	RECEIVED BY Prese Evider Conta				ived C erved ence S ainer A	d On Ice         Y / N         DW = Drin           ed         Y / N         WW = War           e Seals Present         Y / N         RW = Rair           er Attacked         Y / N         GW = Gro		DW = Drinking Water WW = Waste Water RW = Rain Water GW = Ground Water						
RELINQUISHED	BY		DAT	E/TIME	RECEIVE	BY							Prese	erved a	at Lab		Y / N	SO = Soil SW = Solid Waste OL = Oil OT = Other Matrix
PRESCHEDULED R OVER UNSCHEDUL Client agrees to Term	USH ANALYSE ED RUSH REC	S WILL TAKE UESTS at:		RITY SPECI/ wecklabs.com	AL REQUIREMENTS /	BILLIN	IG INF	ORM	ATION	I								COC version 042707

Prepared for

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### **Appendix E**

### **Standard Operating Procedures—Relevant Excerpts**

for the Newhall Ranch Specific Plan Water Quality Monitoring Plan Conditions of Approval and Clean Water Act Section 401 Water Quality Certification and Waste Discharge Requirements

Prepared by

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engineers | scientists | innovators

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Project Number LA0170

March 27, 2013

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### USGS Field Manual for the Collection of Water Quality Data

SOP for Clean Hand/Dirty Hands Sampling Procedures 
 Table 4-3.
 Clean Hands/Dirty Hands techniques for water-quality sampling

- Clean Hands/Dirty Hands techniques require two or more people working together.
- At the field site, one person is designated as Clean Hands (*CH*) and a second person as Dirty Hands (*DH*). Although specific tasks are assigned at the start to *CH* or *DH*, some tasks overlap and can be handled by either, as long as the prescribed care is taken to prevent contaminating the sample.
- *CH* and *DH* wear appropriate disposable, powderless gloves during the entire sampling operation and change gloves frequently, usually with each change in task. (Wearing multiple layers of gloves allows rapid glove changes.) Gloves must be appropriate to withstand any acid, solvent, or other chemical substance that will be used or contacted.
- *CH* takes care of all operations involving equipment that contacts the sample; for example, *CH* 
  - Handles the surface-water sampler bottle
  - Handles the discharge end of the surface-water or ground-water sample tubing
  - Handles the inner protective bag on the churn splitter
  - Transfers sample to churn or cone splitter
  - Prepares a clean work space (inside vehicle)
  - Sets up processing and preservation chambers
  - Places equipment inside chambers (for example, sample bottles, filtration and preservation equipment)
  - Works exclusively inside chambers during collection/processing and preservation
  - Changes chamber covers, as needed
  - Sets up field-cleaning equipment and cleans equipment
- *DH* takes care of all operations involving contact with potential sources of contamination; for example, *DH* 
  - Works exclusively exterior to processing and preservation chambers
  - Prepares and operates sampling equipment, including pumps and discrete samplers, peristaltic pump switch, pump controller, manifold system
  - Operates cranes, tripods, drill rigs, vehicles, or other support equipment
  - Handles the compressor or other power supply for samplers
  - Handles tools such as hammers, wrenches, keys, locks, and sample-flow manifolds
  - Handles single or multiparameter instruments for field measurements
  - Handles the churn carrier, including outer protective bags
  - Handles stream-gaging or water-level equipment
  - Sets up and calibrates field-measurement instruments
  - Measures and records water levels and field measurements

**Use Clean Hands/Dirty Hands** (*CH/DH*) **sampling procedures.** *CH/DH* procedures were developed for collecting (and processing) samples vulnerable to contamination. *CH/DH* procedures separate field-duty chores and dedicate one individual (designated as Clean Hands or *CH*) to tasks related to direct contact with sample-wetted equipment and sample containers (table 4-3). Implementation of this protocol requires hands-on training and field-team coordination.<sup>4</sup>

- ► **Requirement:** *CH/DH* procedures are required when collecting samples for analysis of metals and other inorganic trace elements (hereafter referred to collectively as trace elements), as follows:
  - For trace elements with ambient concentrations at or near 1 μg/L.
  - For iron, aluminum, or manganese with ambient concentrations to about 200 μg/L.
- ► **Recommendation:** *CH/DH* procedures are recommended when collecting samples for analysis of most trace elements with concentrations to about 100 µg/L.
- Recommendation: CH/DH procedures are recommended when collecting samples for analysis of trace-organic compounds and major inorganic elements, particularly when the target analyte could be subject to contamination from field or laboratory procedures at a level that could exceed data-quality requirements.

<sup>&</sup>lt;sup>4</sup>A detailed description of Clean Hands/ Dirty Hands techniques for surface-water sampling can be found in Hor owitz and others (1994). Clean Hands/Dirty Hands techniques have been incorporated in the procedures for ground-water sampling (refer to section 4.2), equipment cleaning (NFM 3), and sample processing (NFM 5).

#### 24-COLLECTION OF WATER SAMPLES

**Minimize atmospheric contamination.** Water bodies that are isolated from the atmosphere or that have dissolved-oxygen concentrations that are substantially less than that of air can be found in surface-water systems (deeper sections of stratified lakes and reservoirs, for example), but are more common in ground-water systems. For such sites, exposure of the sample to the atmosphere can increase dissolved-oxygen concentrations, causing reduced metal ions to oxidize and precipitate as a hydroxide.

Collection of environmental samples from water bodies for which concentrations of dissolved gases differ substantially from atmospheric concentrations might require special field equipment or procedures. Equipment and procedures should be selected that minimize contact with the atmosphere or minimize the effect of pressure changes from the source of the sample to the point of field measurement or sample processing. Sampling methods and equipment for preventing contact of anoxic and suboxic water samples with atmospheric gases are described in section 4.2.2.C.

**TECHNICAL NOTE:** Exposure of anoxic or suboxic samples to atmospheric oxygen can cause reduced metal ions to oxidize and precipitate as a hydroxide (for example, oxidation of iron species from ferrous (Fe<sup>+2</sup>) to ferric (Fe<sup>+3</sup>) iron). Precipitation of an iron (or other metal) hydroxide can occur either before or during sample filtration, thereby lowering concentrations of soluble iron and coprecipitating metals in the sample. Examples of nonmetal analytes for which atmospheric exposure can compromise sample integrity include volatile organic compounds (VOCs), pH, alkalinity, sulfide, chlorofluorocarbons (CFCs), and some bacteria species.

### US EPA Method 1669. Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels

SOP for Clean Hand/Dirty Hands Sampling Procedures

### Method 1669

### Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels

**July 1996** 

U.S. Environmental Protection Agency Office of Water Engineering and Analysis Division (4303) 401 M Street S.W. Washington, D.C. 20460

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Shier Berman, National Research Council, Ottawa, Ontario, Canada; Nicholas Bloom, Frontier Geosciences Inc, Seattle, Washington; Eric Crecelius, Battelle Marine Sciences Laboratory, Sequim, Washington; Russell Flegal, University of California/Santa Cruz, California; Gary Gill, Texas A&M University at Galveston, Texas; Carlton Hunt and Dion Lewis, Battelle Ocean Sciences, Duxbury, Massachusetts; Carl Watras, Wisconsin Department of Natural Resources, Boulder Junction, Wisconsin

Additional support was provided by Ted Martin of the EPA Office of Research and Development's Environmental Monitoring Systems Laboratory in Cincinnati, Ohio and by Arthur Horowitz of the U.S. Geological Survey.

This version of the method was prepared after observations of sampling teams from the University of California at Santa Cruz, the Wisconsin Department of Natural Resources, the U.S. Geological Survey, and Battelle Ocean Sciences. The assistance of personnel demonstrating the sampling techniques used by these institutions is gratefully acknowledged.

### Disclaimer

This sampling method has been reviewed and approved for publication by the Analytical Methods Staff within the Engineering and Analysis Division of the U.S. Environmental Protection Agency. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

### **Further Information**

For further information, contact:

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### Introduction

This sampling method was designed to support water quality monitoring programs authorized under the Clean Water Act. Section 304(a) of the Clean Water Act requires EPA to publish water quality criteria that reflect the latest scientific knowledge concerning the physical fate (e.g., concentration and dispersal) of pollutants, the effects of pollutants on ecological and human health, and the effect of pollutants on biological community diversity, productivity, and stability.

Section 303 of the Clean Water Act requires states to set a water quality standard for each body of water within its boundaries. A state water quality standard consists of a designated use or uses of a waterbody or a segment of a waterbody, the water quality criteria that are necessary to protect the designated use or uses, and an antidegradation policy. These water quality standards serve two purposes: (1) they establish the water quality goals for a specific waterbody, and (2) they are the basis for establishing water quality-based treatment controls and strategies beyond the technology-based controls required by Sections 301(b) and 306 of the Clean Water Act.

In defining water quality standards, the state may use narrative criteria, numeric criteria, or both. However, the 1987 amendments to the Clean Water Act required states to adopt numeric criteria for toxic pollutants (designated in Section 307(a) of the Act) based on EPA Section 304(a) criteria or other scientific data, when the discharge or presence of those toxic pollutants could reasonably be expected to interfere with designated uses.

In some cases, these water quality criteria are as much as 280 times lower than those achievable using existing EPA methods and required to support technology-based permits. Therefore, this sampling method, and the analytical methods referenced in Table 1 of this document, were developed by EPA to specifically address state needs for measuring toxic metals at water quality criteria levels, when such measurements are necessary to protect designated uses in state water quality standards. The latest criteria published by EPA are those listed in the National Toxics Rule (57 FR 60848) and the Stay of Federal Water Quality Criteria for Metals (60 FR 22228). These rules include water quality criteria for 13 metals, and it is these criteria on which this sampling method and the referenced analytical methods are based.

In developing these methods, EPA found that one of the greatest difficulties in measuring pollutants at these levels was precluding sample contamination during collection, transport, and analysis. The degree of difficulty, however, is highly dependent on the metal and site-specific conditions. This method, therefore, is designed to provide the level of protection necessary to preclude contamination in nearly all situations. It is also designed to provide the procedures necessary to produce reliable results at the lowest possible water quality criteria published by EPA. In recognition of the variety of situations to which this method may be applied, and in recognition of continuing technological advances, the method is performance-based. Alternative procedures may be used, so long as those procedures are demonstrated to yield reliable results.

Requests for additional copies of this method should be directed to:

U.S. EPA NCEPI 11029 Kenwood Road Cincinnati, OH 45242 513/489–8190 Note: This document is intended as guidance only. Use of the terms "must," "may," and "should" are included to mean that EPA believes that these procedures must, may, or should be followed in order to produce the desired results when using this guidance. In addition, the guidance is intended to be performance-based, in that the use of less stringent procedures may be used so long as neither samples nor blanks are contaminated when following those modified procedures. Because the only way to measure the performance of the modified procedures is through the collection and analysis of uncontaminated blank samples in accordance with this guidance and the referenced methods, it is highly recommended that any modifications be thoroughly evaluated and demonstrated to be effective before field samples are collected.

### Method 1669

### Sampling Ambient Water for Determination of Metals at EPA Water Quality Criteria Levels

### **1.0** Scope and Application

- 1.1 This method is for the collection and filtration of ambient water samples for subsequent determination of total and dissolved metals at the levels listed in Table 1. It is designed to support the implementation of water quality monitoring and permitting programs administered under the Clean Water Act.
- 1.2 This method is applicable to the metals listed below and other metals, metals species, and elements amenable to determination at trace levels.

Analyte	Symbol	Chemical Abstract Services Registry Number (CASRN)
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Cadmium	(Cd)	7440-43-9
Chromium (III)	$\mathrm{Cr}^{+3}$	16065-83-1
Chromium (VI)	$\mathrm{Cr}^{+6}$	18540-29-9
Copper	(Cu)	7440-50-8
Lead	(Pb)	7439-92-1
Mercury	(Hg)	7439-97-6
Nickel	(Ni)	7440-02-0
Selenium	(Se)	7782-49-2
Silver	(Ag)	7440-22-4
Thallium	(TI)	7440-28-0
Zinc	(Zn)	7440-66-6

- 1.3 This method is accompanied by the 1600 series methods listed in Table 1. These methods include the sample handling, analysis, and quality control procedures necessary for reliable determination of trace metals in aqueous samples.
- 1.4 This method is not intended for determination of metals at concentrations normally found in treated and untreated discharges from industrial facilities. Existing regulations (40 *CFR* Parts 400-500) typically limit concentrations in industrial discharges to the mid to high part-per-billion (ppb) range, whereas ambient metals concentrations are normally in the low part-per-trillion (ppt) to low ppb range. This guidance is therefore directed at the collection of samples to be measured at or near the levels listed in Table 1. Actual concentration ranges to which this guidance is applicable will be dependent on the sample matrix, dilution levels, and other laboratory operating conditions.
- 1.5 The ease of contaminating ambient water samples with the metal(s) of interest and interfering substances cannot be overemphasized. This method includes sampling techniques that should maximize the ability of the sampling team to collect samples reliably and eliminate sample contamination. These techniques are given in Section 8.0 and are based on findings of researchers performing trace metals analyses (References 1-9).

- 1.6 Clean and Ultraclean—The terms "clean" and "ultraclean" have been used in other Agency guidance to describe the techniques needed to reduce or eliminate contamination in trace metals determinations. These terms are not used in this sampling method due to a lack of exact definitions. However, the information provided in this method is consistent with summary guidance on clean and ultraclean techniques (Reference 10).
- 1.7 This sampling method follows the EPA Environmental Methods Management Council's "Format for Method Documentation" (Reference 11).
- 1.8 Method 1669 is "performance-based"; i.e., an alternate sampling procedure or technique may be used, so long as neither samples nor blanks are contaminated when following the alternate procedures. Because the only way to measure the performance of the alternate procedures is through the collection and analysis of uncontaminated blank samples in accordance with this guidance and the methods referenced in Table 1, it is highly recommended that any modifications be thoroughly evaluated and demonstrated to be effective before field samples are collected. Section 9.2 provides additional details on the tests and documentation required to support equivalent performance.
- 1.9 For dissolved metal determinations, samples must be filtered through a 0.45 μm capsule filter at the field site. The filtering procedures are described in this method. The filtered samples may be preserved in the field or transported to the laboratory for preservation. Procedures for field preservation are detailed in this sampling method; procedures for laboratory preservation are provided in the methods referenced in Table 1. Preservation requirements are summarized in Table 2.
- 1.10 The procedures in this method are for use only by personnel thoroughly trained in the collection of samples for determination of metals at ambient water quality control levels.

### 2.0 Summary of Method

- 2.1 Before samples are collected, all sampling equipment and sample containers are cleaned in a laboratory or cleaning facility using detergent, mineral acids, and reagent water as described in the methods referenced in Table 1. The laboratory or cleaning facility is responsible for generating an acceptable equipment blank to demonstrate that the sampling equipment and containers are free from trace metals contamination before they are shipped to the field sampling team. An acceptable blank is one that is free from contamination below the minimum level (ML) specified in the referenced analytical method (Section 9.3).
- 2.2 After cleaning, sample containers are filled with weak acid solution, individually doublebagged, and shipped to the sampling site. All sampling equipment is also bagged for storage or shipment.

**NOTE:** EPA has found that, in some cases, it may be possible to empty the weak acid solution from the bottle immediately prior to transport to the field site. In this case, the bottle should be refilled with reagent water (Section 7.1).

2.3 The laboratory or cleaning facility must prepare a large carboy or other appropriate clean container filled with reagent water (Section 7.1) for use with collection of field blanks during sampling activities. The reagent-water-filled container should be shipped to the field site and handled as all other sample containers and sampling equipment. At least one field blank should be processed per site, or one per every ten samples, whichever is more frequent (Section 9.4). If samples are to be collected for determination of trivalent

chromium, the sampling team processes additional QC aliquots are processed as described in Section 9.6.

- 2.4 Upon arrival at the sampling site, one member of the two-person sampling team is designated as "dirty hands"; the second member is designated as "clean hands." All operations involving contact with the sample bottle and transfer of the sample from the sample collection device to the sample bottle are handled by the individual designated as "clean hands." "Dirty hands" is responsible for preparation of the sampler (except the sample container itself), operation of any machinery, and for all other activities that do not involve direct contact with the sample.
- 2.5 All sampling equipment and sample containers used for metals determinations at or near the levels listed in Table 1 must be nonmetallic and free from any material that may contain metals.
- 2.6 Sampling personnel are required to wear clean, nontalc gloves at all times when handling sampling equipment and sample containers.
- 2.7 In addition to processing field blanks at each site, a field duplicate must be collected at each sampling site, or one field duplicate per every 10 samples, whichever is more frequent (Section 9.5). Section 9.0 gives a complete description of quality control requirements.
- 2.8 Sampling
  - 2.8.1 Whenever possible, samples are collected facing upstream and upwind to minimize introduction of contamination.
  - 2.8.2 Samples may be collected while working from a boat or while on land.
  - 2.8.3 Surface samples are collected using a grab sampling technique. The principle of the grab technique is to fill a sample bottle by rapid immersion in water and capping to minimize exposure to airborne particulate matter.
  - 2.8.4 Subsurface samples are collected by suction of the sample into an immersed sample bottle or by pumping the sample to the surface.
- 2.9 Samples for dissolved metals are filtered through a 0.45  $\mu$ m capsule filter at the field site. After filtering, the samples are double-bagged and iced immediately. Sample containers are shipped to the analytical laboratory. The sampling equipment is shipped to the laboratory or cleaning facility for recleaning.
- 2.10 Acid preservation of samples is performed in the field or in the laboratory. Field preservation is necessary for determinations of trivalent chromium. It has also been shown that field preservation can increase sample holding times for hexavalent chromium to 30 days; therefore it is recommended that preservation of samples for hexavalent chromium be performed in the field. For other metals, however, the sampling team may prefer to utilize laboratory preservation of samples to expedite field operations and to minimize the potential for sample contamination.
- 2.11 Sampling activities must be documented through paper or computerized sample tracking systems.

### 3.0 Definitions

- 3.1 Apparatus—Throughout this method, the sample containers, sampling devices, instrumentation, and all other materials and devices used in sample collection, sample processing, and sample analysis activities will be referred to collectively as the Apparatus.
- 3.2 Definitions of other terms are given in the Glossary (Section 15.0) at the end of this method.

### 4.0 Contamination and Interferences

- 4.1 Contamination Problems in Trace Metals Analysis
  - 4.1.1 Preventing ambient water samples from becoming contaminated during the sampling and analytical process is the greatest challenge faced in trace metals determinations. In recent years, it has been shown that much of the historical trace metals data collected in ambient water are erroneously high because the concentrations reflect contamination from sampling and analysis rather than ambient levels (Reference 12). Therefore, it is imperative that extreme care be taken to avoid contamination when collecting and analyzing ambient water samples for trace metals.
  - 4.1.2 There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination during sampling include metallic or metal-containing sampling equipment, containers, labware (e.g. talc gloves that contain high levels of zinc), reagents, and deionized water; improperly cleaned and stored equipment, labware, and reagents; and atmospheric inputs such as dirt and dust from automobile exhaust, cigarette smoke, nearby roads, bridges, wires, and poles. Even human contact can be a source of trace metals contamination. For example, it has been demonstrated that dental work (e.g., mercury amalgam fillings) in the mouths of laboratory personnel can contaminate samples that are directly exposed to exhalation (Reference 3).
- 4.2 Contamination Control

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- 4.2.1 Philosophy—The philosophy behind contamination control is to ensure that any object or substance that contacts the sample is nonmetallic and free from any material that may contain metals of concern.
  - 4.2.1.1 The integrity of the results produced cannot be compromised by contamination of samples. Requirements and suggestions for controlling sample contamination are given in this sampling method and in the analytical methods referenced in Table 1.
  - 4.2.1.2 Substances in a sample or in the surrounding environment cannot be allowed to contaminate the Apparatus used to collect samples for trace metals measurements. Requirements and suggestions for protecting the Apparatus are given in this sampling method and in the methods referenced in Table 1.
  - 4.2.1.3 While contamination control is essential, personnel health and safety remain the highest priority. Requirements and suggestions for personnel safety are
given in Section 5 of this sampling method and in the methods referenced in Table 1.

- 4.2.2 Avoiding contamination—The best way to control contamination is to completely avoid exposure of the sample and Apparatus to contamination in the first place. Avoiding exposure means performing operations in an area known to be free from contamination. Two of the most important factors in avoiding/reducing sample contamination are (1) an awareness of potential sources of contamination and (2) strict attention to work being performed. Therefore, it is imperative that the procedures described in this method be carried out by well trained, experienced personnel. Documentation of training should be kept on file and readily available for review.
  - 4.2.2.1 Minimize exposure—The Apparatus that will contact samples or blanks should only be opened or exposed in a clean room, clean bench, glove box, or clean plastic bag, so that exposure to atmospheric inputs is minimized. When not being used, the Apparatus should be covered with clean plastic wrap, stored in the clean bench or in a plastic box or glove box, or bagged in clean, colorless zip-type bags. Minimizing the time between cleaning and use will also reduce contamination.
  - 4.2.2.2 Wear gloves—Sampling personnel must wear clean, nontalc gloves (Section 6.7) during all operations involving handling of the Apparatus, samples, and blanks. Only clean gloves may touch the Apparatus. If another object or substance is touched, the glove(s) must be changed before again handling the Apparatus. If it is even suspected that gloves have become contaminated, work must be halted, the contaminated gloves removed, and a new pair of clean gloves put on. Wearing multiple layers of clean gloves will allow the old pair to be quickly stripped with minimal disruption to the work activity.
  - 4.2.2.3 Use metal-free Apparatus—All Apparatus used for metals determinations at the levels listed in Table 1 must be nonmetallic and free of material that may contain metals. When it is not possible to obtain equipment that is completely free of the metal(s) of interest, the sample should not come into direct contact with the equipment.
    - 4.2.2.3.1 Construction materials—Only the following materials should come in contact with samples: fluoropolymer (FEP, PTFE), conventional or linear polyethylene, polycarbonate, polysulfone, polypropylene, or ultrapure quartz. PTFE is less desirable than FEP because the sintered material in PTFE may contain contaminants and is susceptible to serious memory effects (Reference 6). Fluoropolymer or glass containers should be used for samples that will be analyzed for mercury because mercury vapors can diffuse in or out of other materials, resulting either in contamination or low-biased results (Reference 3). Metal must not be used under any circumstance. Regardless of construction. all materials that will directly or indirectly contact the sample must be cleaned using the procedures described in the

referenced analytical methods (see Table 1) and must be known to be clean and metal-free before proceeding.

- 4.2.2.3.2 The following materials have been found to contain trace metals and must not be used to hold liquids that come in contact with the sample or must not contact the sample, unless these materials have been shown to be free of the metals of interest at the desired level: Pyrex, Kimax, methacrylate, polyvinylchloride, nylon, and Vycor (Reference 6). In addition, highly colored plastics, paper cap liners, pigments used to mark increments on plastics, and rubber all contain trace levels of metals and must be avoided (Reference 13).
- 4.2.2.3.3 Serialization—Serial numbers should be indelibly marked or etched on each piece of Apparatus so that contamination can be traced, and logbooks should be maintained to track the sample from the container through the sampling process to shipment to the laboratory. Chain-of-custody procedures may also be used if warranted so that contamination can be traced to particular handling procedures or lab personnel.
- 4.2.2.3.4 The Apparatus should be clean when the sampling team receives it. If there are any indications that the Apparatus is not clean (e.g., a ripped storage bag), an assessment of the likelihood of contamination must be made. Sampling must not proceed if it is possible that the Apparatus is contaminated. If the Apparatus is contaminated, it must be returned to the laboratory or cleaning facility for proper cleaning before any sampling activity resumes.
- 4.2.2.3.5 Details for recleaning the Apparatus between collection of individual samples are provided in Section 10.0.
- 4.2.2.4 Avoid sources of contamination—Avoid contamination by being aware of potential sources and routes of contamination.
  - 4.2.2.4.1 Contamination by carryover—Contamination may occur when a sample containing low concentrations of metals is processed immediately after a sample containing relatively high concentrations of these metals. At sites where more than one sample will be collected, the sample known or expected to contain the lowest concentration of metals should be collected first with the sample containing the highest levels collected last (Section 8.1.4). This will help minimize carryover of metals from high- concentration samples to low- concentration samples. If the sampling team does not have prior knowledge of the waterbody, or when necessary, the sample collection system should be rinsed with dilute acid and reagent water between samples and followed by collection of a field blank (Section 10.3).

- 4.2.2.4.2 Contamination by samples—Significant contamination of the Apparatus may result when untreated effluents, inprocess waters, landfill leachates, and other samples containing mid- to high-level concentrations of inorganic substances are processed. As stated in Section 1.0, this sampling method is not intended for application to these samples, and samples containing high concentrations of metals must not be collected, processed, or shipped at the same time as samples being collected for trace metals determinations.
- 4.2.2.4.3 Contamination by indirect contact—Apparatus that may not directly contact samples may still be a source of contamination. For example, clean tubing placed in a dirty plastic bag may pick up contamination from the bag and subsequently transfer the contamination to the sample. Therefore, it is imperative that every piece of the Apparatus that is directly or indirectly used in the collection of ambient water samples be cleaned as specified in the analytical method(s) referenced in Table 1.
- 4.2.2.4.4 particulate Contamination bv airborne matter—Less obvious substances capable of contaminating samples include airborne particles. Samples may be contaminated by airborne dust, dirt, particulate matter, or vapors from automobile exhaust; cigarette smoke; nearby corroded or rusted bridges, pipes, poles, or wires; nearby roads; and even human breath (Section 4.1.2). Whenever possible, the sampling activity should occur as far as possible from sources of airborne contamination (Section 8.1.3). Areas where nearby soil is bare and subject to wind erosion should be avoided.
- 4.3 Interferences—Interferences resulting from samples will vary considerably from source to source, depending on the diversity of the site being sampled. If a sample is suspected of containing substances that may interfere in the determination of trace metals, sufficient sample should be collected to allow the laboratory to identify and overcome interference problems.

### 5.0 Safety

5.1 The toxicity or carcinogenicity of the chemicals used in this method has not been precisely determined; however, these chemicals should be treated as a potential health hazard. Exposure should be reduced to the lowest possible level. Sampling teams are responsible for maintaining a current awareness file of OSHA regulations for the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets should also be made available to all personnel involved in sampling. It is also suggested that the organization responsible perform personal hygiene monitoring of each sampling team member who uses this method and that the results of this monitoring be made available to the member.

- 5.2 Operating in and around waterbodies carries the inherent risk of drowning. Life jackets must be worn when operating from a boat, when sampling in more than a few feet of water, or when sampling in swift currents.
- 5.3 Collecting samples in cold weather, especially around cold water bodies, carries the risk of hypothermia, and collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should wear adequate clothing for protection in cold weather and should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

### 6.0 Apparatus and Materials

**NOTE:** Brand names, suppliers, and part numbers are for illustration only and no endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here. Meeting the performance requirements of this method is the responsibility of the sampling team and laboratory.

- 6.1 All sampling equipment and sample containers must be precleaned in a laboratory or cleaning facility, as described in the methods referenced in Table 1, before they are shipped to the field site. Performance criteria for equipment cleaning is described in the referenced methods. To minimize difficulties in sampling, the equipment should be packaged and arranged to minimize field preparation.
- 6.2 Materials such as gloves (Section 6.7), storage bags (Section 6.8), and plastic wrap (Section 6.9), may be used new without additional cleaning unless the results of the equipment blank pinpoint any of these materials as a source of contamination. In this case, either a different supplier must be obtained or the materials must be cleaned.
- 6.3 Sample Bottles—Fluoropolymer (FEP, PTFE), conventional or linear polyethylene, polycarbonate, or polypropylene; 500 mL or 1 L with lids. If mercury is a target analyte, fluoropolymer or glass bottles should be used. Refer to the methods referenced in Table 1 for bottle cleaning procedures.
  - 6.3.1 Cleaned sample bottles should be filled with 0.1% HCl (v/v). In some cases, it may be possible to empty the weak acid solution from the sample bottle immediately prior to transport to the field site. In this case, the bottle should be refilled with reagent water (Section 7.1).
  - 6.3.2 Whenever possible, sampling devices should be cleaned and prepared for field use in a class 100 clean room. Preparation of the devices in the field should be done within the glove bag (Section 6.6). Regardless of design, sampling devices must be constructed of nonmetallic material (Section 4.2.2.3.1) and free from material that contains metals. Fluoropolymer or other material shown not to adsorb or contribute mercury must be used if mercury is a target analyte; otherwise, polyethylene, polycarbonate, or polypropylene are acceptable. Commercially available sampling devices may be used provided that any metallic or metalcontaining parts are replaced with parts constructed of nonmetallic material.
- 6.4 Surface Sampling Devices—Surface samples are collected using a grab sampling technique. Samples may be collected manually by direct submersion of the bottle into the water or by using a grab sampling device. Examples of grab samplers are shown in Figures 1 and 2 and may be used at sites where depth profiling is neither practical nor necessary.

- 6.4.1 The grab sampler in Figure 1 consists of a heavy fluoropolymer collar fastened to the end of a 2-m-long polyethylene pole, which serves to remove the sampling personnel from the immediate vicinity of the sampling point. The collar holds the sample bottle. A fluoropolymer closing mechanism, threaded onto the bottle, enables the sampler to open and close the bottle under water, thereby avoiding surface microlayer contamination (Reference 14). Polyethylene, polycarbonate, and polypropylene are also acceptable construction materials unless mercury is a target analyte. Assembly of the cleaned sampling device is as follows (refer to Figure 1):
  - 6.4.1.1 Thread the pull cord (with the closing mechanism attached) through the guides and secure the pull ring with a simple knot. Screw a sample bottle onto the closing device and insert the bottle into the collar. Cock the closing plate so that the plate is pushed away from the operator.
  - 6.4.1.2 The cleaned and assembled sampling device should be stored in a double layer of large, clean zip-type polyethylene bags or wrapped in two layers of clean polyethylene wrap if it will not be used immediately.
- 6.4.2 An alternate grab sampler design is shown in Figure 2. This grab sampler is used for discrete water samples and is constructed so that a capped clean bottle can be submerged, the cap removed, sample collected, and bottle recapped at a selected depth. This device eliminates sample contact with conventional samplers (e.g., Niskin bottles), thereby reducing the risk of extraneous contamination. Because a fresh bottle is used for each sample, carryover from previous samples is eliminated (Reference 15).
- 6.5 Subsurface Sampling Devices—Subsurface sample collection may be appropriate in lakes and sluggish deep river environments or where depth profiling is determined to be necessary. Subsurface samples are collected by pumping the sample into a sample bottle. Examples of subsurface collection systems include the jar system device shown in Figure 3 and described in Section 6.5.1 or the continuous-flow apparatus shown in Figure 4 and described in Section 6.5.2.
  - 6.5.1 Jar sampler (Reference 14)—The jar sampler (Figure 3) is comprised of a heavy fluoropolymer 1-L jar with a fluoropolymer lid equipped with two 1/4 in. fluoropolymer fittings. Sample enters the jar through a short length of fluoropolymer tubing inserted into one fitting. Sample is pulled into the jar by pumping on fluoropolymer tubing attached to the other fitting. A thick fluoropolymer plate supports the jar and provides attachment points for a fluoropolymer safety line and fluoropolymer torpedo counterweight.
    - 6.5.1.1 Advantages of the jar sampler for depth sampling are (1) all wetted surfaces are fluoropolymer and can be rigorously cleaned; (2) the sample is collected into a sample jar from which the sample is readily recovered, and the jar can be easily recleaned; (3) the suction device (a peristaltic or rotary vacuum pump, Section 6.15) is located in the boat, isolated from the sampling jar; (4) the sampling jar can be continuously flushed with sample, at sampling depth, to equilibrate the system; and (5) the sample does not travel through long lengths of tubing that are more difficult to clean and keep clean (Reference 14). In addition, the device is designed to eliminate atmospheric contact with the sample during collection.

- 6.5.1.2 To assemble the cleaned jar sampler, screw the torpedo weight onto the machined bolt attached to the support plate of the jar sampler. Attach a section of the 1/4 in. o.d. tubing to the jar by inserting the tubing into the fitting on the lid and pushing down into the jar until approximately 8 cm from the bottom. Tighten the fitting nut securely. Attach the solid safety line to the jar sampler using a bowline knot to the loop affixed to the support plate.
- 6.5.1.3 For the tubing connecting the pump to the sampler, tubing lengths of up to 12 m have been used successfully (Reference 14).
- 6.5.2 Continuous-flow sampler (References 16-17)—This sampling system, shown in Figure 4, consists of a peristaltic or submersible pump and one or more lengths of precleaned fluoropolymer or styrene/ethylene/butylene/ silicone (SEBS) tubing. A filter is added to the sampling train when sampling for dissolved metals.
  - 6.5.2.1 Advantages of this sampling system include (1) all wetted surfaces are fluoropolymer or SEBS and can be readily cleaned; (2) the suction device is located in the boat, isolated from the sample bottle; (3) the sample does not travel through long lengths of tubing that are difficult to clean and keep clean; and (4) in-line filtration is possible, minimizing field handling requirements for dissolved metals samples.
  - 6.5.2.2 The sampling team assembles the system in the field as described in Section 8.2.8. System components include an optional polyethylene pole to remove sampling personnel from the immediate vicinity of the sampling point and the pump, tubing, filter, and filter holder listed in Sections 6.14 and 6.15.
- 6.6 Field-Portable Glove Bag—I2R, Model R-37-37H (nontalc), or equivalent. Alternately, a portable glove box may be constructed with a nonmetallic (PVC pipe or other suitable material) frame and a frame cover made of an inexpensive, disposable, nonmetallic material (e.g., a thin-walled polyethylene bag) (Reference 7).
- 6.7 Gloves—Clean, nontalc polyethylene, latex, vinyl, or PVC; various lengths. Shoulderlength gloves are needed if samples are to be collected by direct submersion of the sample bottle into the water or when sampling for mercury.
  - 6.7.1 Gloves, shoulder-length polyethylene—Associated Bag Co., Milwaukee, WI, 66-3-301, or equivalent.
  - 6.7.2 Gloves, PVC—Fisher Scientific Part No. 11-394-100B, or equivalent.
- 6.8 Storage Bags—Clean, zip-type, nonvented, colorless polyethylene (various sizes).
- 6.9 Plastic Wrap—Clean, colorless polyethylene.
- 6.10 Cooler—Clean, nonmetallic, with white interior for shipping samples.
- 6.11 Ice or Chemical Refrigerant Packs—To keep samples chilled in the cooler during shipment.
- 6.12 Wind Suit—Pamida, or equivalent.

**NOTE:** This equipment is necessary only for collection of metals, such as mercury, that are known to have elevated atmospheric concentrations.

- 6.12.1 An unlined, long-sleeved wind suit consisting of pants and jacket and constructed of nylon or other synthetic fiber is worn when sampling for mercury to prevent mercury adsorbed onto cotton or other clothing materials from contaminating samples.
- 6.12.2 Washing and drying—The wind suit is washed by itself or with other wind suits only in a home or commercial washing machine and dried in a clothes dryer. The clothes dryer must be thoroughly vacuumed, including the lint filter, to remove all traces of lint before drying. After drying, the wind suit is folded and stored in a clean polyethylene bag for shipment to the sample site.

#### 6.13 Boat

- 6.13.1 For most situations (e.g., most metals under most conditions), the use of an existing, available boat is acceptable. A flat-bottom, Boston Whaler-type boat is preferred because sampling materials can be stored with reduced chance of tipping.
  - 6.13.1.1 Immediately before use, the boat should be washed with water from the sampling site away from any sampling points to remove any dust or dirt accumulation.
  - 6.13.1.2 Samples should be collected upstream of boat movement.
- 6.13.2 For mercury, and for situations in which the presence of contaminants cannot otherwise be controlled below detectable levels, the following equipment and precautions may be necessary:
  - 6.13.2.1 A metal-free (e.g., fiberglass) boat, along with wooden or fiberglass oars. Gasoline- or diesel-fueled boat motors should be avoided when possible because the exhaust can be a source of contamination. If the body of water is large enough to require use of a boat motor, the engine should be shut off at a distance far enough from the sampling point to avoid contamination, and the sampling team should manually propel the boat to the sampling point. Samples should be collected upstream of boat movement.
  - 6.13.2.2 Before first use, the boat should be cleaned and stored in an area that minimizes exposure to dust and atmospheric particles. For example, cleaned boats should not be stored in an area that would allow exposure to automobile exhaust or industrial pollution.
  - 6.13.2.3 The boat should be frequently visually inspected for possible contamination.
  - 6.13.2.4 After sampling, the boat should be returned to the laboratory or cleaning facility, cleaned as necessary, and stored away from any sources of contamination until next use.

- 6.14 Filtration Apparatus—Required when collecting samples for dissolved metals determinations.
  - 6.14.1 Filter—0.45 μm, 15 mm diameter or larger, tortuous-path capsule filters (Reference 18), Gelman Supor 12175, or equivalent.
  - 6.14.2 Filter holder—For mounting filter to the gunwale of the boat. Rod or pipe made from plastic material and mounted with plastic clamps.

**NOTE:** A filter holder may not be required if one or a few samples are to be collected. For these cases, it may only be necessary to attach the filter to the outlet of the tubing connected to the pump.

6.15 Pump and Pump Apparatus—Required for use with the jar sampling system (Section 6.5.1) or the continuous-flow system (Section 6.5.2). Peristaltic pump; 115 V a.c., 12 V d.c., internal battery, variable-speed, single-head, Cole-Parmer, portable, "Masterflex L/S," Catalog No. H-07570-10 drive with Quick Load pump head, Catalog No. H-07021-24, or equivalent.

**NOTE:** Equivalent pumps may include rotary vacuum, submersible, or other pumps free from metals and suitable to meet the site-specific depth sampling needs.

- 6.15.1 Cleaning—Peristaltic pump modules do not require cleaning. However, nearly all peristaltic pumps contain a metal head and metal controls. Touching the head or controls necessitates changing of gloves before touching the Apparatus. If a submersible pump is used, a large volume of sample should be pumped to clean the stainless steel shaft (hidden behind the impeller) that comes in contact with the sample. Pumps with metal impellers should not be used.
- 6.15.2 Tubing—For use with peristaltic pump. SEBS resin, approximately 3/8 in. i.d. by approximately 3 ft, Cole-Parmer size 18, Cat. No. G-06464-18, or approximately 1/4 in. i.d., Cole-Parmer size 17, Catalog No. G-06464-17, or equivalent. Tubing is cleaned by soaking in 5-10% HCl solution for 8-24 hours, rinsing with reagent water in a clean bench in a clean room, and drying in the clean bench by purging with mercury-free air or nitrogen. After drying, the tubing is double-bagged in clear polyethylene bags, serialized with a unique number, and stored until use.
- 6.15.3 Tubing—For connection to peristaltic pump tubing. Fluoropolymer, 3/8 or 1/4 in. o.d., in lengths as required to reach the point of sampling. If sampling will be at some depth from the end of a boom extended from a boat, sufficient tubing to extend to the end of the boom and to the depth will be required. Cleaning of the fluoropolymer can be the same as cleaning the tubing for the rotary vacuum pump (Section 6.15.1.2). If necessary, more aggressive cleaning (e.g., concentrated nitric acid) may be used.
- 6.15.4 Batteries to operate submersible pump—12 V, 2.6 amp, gel cell, YUASA NP2.6-12, or equivalent. A 2 amp fuse connected at the positive battery terminal is strongly recommended to prevent short circuits from overheating the battery. A 12 V, lead-acid automobile or marine battery may be more suitable for extensive pumping.

- 6.15.5 Tubing connectors—Appropriately sized PVC, clear polyethylene, or fluoropolymer "barbed" straight connectors cleaned as the tubing above. Used to connect multiple lengths of tubing.
- 6.16 Carboy—For collection and storage of dilute waste acids used to store bottles.
- 6.17 Apparatus—For field preservation of aliquots for trivalent chromium determinations.
  - 6.17.1 Fluoropolymer forceps—1 L fluoropolymer jar, and 30 mL fluoropolymer vials with screw-caps (one vial per sample and blank). It is recommended that 1 mL of ultrapure nitric acid (Section 7.3) be added to each vial prior to transport to the field to simplify field handling activities (See Section 8.4.4.6).
  - 6.17.2 Filters—0.4 μm, 47 mm polycarbonate Nuclepore (or equivalent). Filters are cleaned as follows. Fill a 1 L fluoropolymer jar approximately two-thirds full with 1 N nitric acid. Using fluoropolymer forceps, place individual filters in the fluoropolymer jar. Allow the filters to soak for 48 hours. Discard the acid, and rinse five times with reagent water. Fill the jar with reagent water, and soak the filters for 24 hours. Remove the filters when ready for use, and using fluoropolymer forceps, place them on the filter apparatus (Section 6.17.3).
  - 6.17.3 Vacuum filtration apparatus—Millipore 47 mm size, or equivalent, vacuum pump and power source (and extension cords, if necessary) to operate the pump.
  - 6.17.4 Eppendorf auto pipet and colorless pipet tips (100-1000 μL)
  - 6.17.5 Wrist-action shaker—Burrel or equivalent.
  - 6.17.6 Fluoropolymer wash bottles—One filled with reagent water (Section 7.1) and one filled with high- purity 10% HCl (Section 7.4.4), for use in rinsing forceps and pipet tips.

### 7.0 Reagents and Standards

- 7.1 Reagent Water—Water in which the analytes of interest and potentially interfering substances are not detected at the Method Detection Limit (MDL) of the analytical method used for analysis of samples. Prepared by distillation, deionization, reverse osmosis, anodic/cathodic stripping voltammetry, or other techniques that remove the metal(s) and potential interferent(s). A large carboy or other appropriate container filled with reagent water must be available for the collection of field blanks.
- 7.2 Nitric Acid—Dilute, trace-metal grade, shipped with sampling kit for cleaning equipment between samples.
- 7.3 Sodium Hydroxide—Concentrated, 50% solution for use when field-preserving samples for hexavalent chromium determinations (Section 8.4.5).
- 7.4 Reagents—For field-processing aliquots for trivalent chromium determinations
  - 7.4.1 Nitric Acid, Ultrapure—For use when field-preserving samples for trivalent chromium determinations (Sections 6.17 and 8.4.4).

- 7.4.2 Ammonium Iron (II) Sulfate Solution (0.01M)—Used to prepare the chromium (III) extraction solution (Section 7.4.3) necessary for field preservation of samples for trivalent chromium (Section 8.4.4). Prepare the ammonium iron (II) sulfate solution by adding 3.92 g ammonium iron (II) sulfate (ultrapure grade) to a 1 L volumetric flask. Bring to volume with reagent water. Store in a clean polyethylene bottle.
- 7.4.3 Chromium (III) extraction solution—For use when field-preserving samples for trivalent chromium determinations (Section 8.4.4). Prepare this solution by adding 100 mL of ammonium iron (II) sulfate solution (Section 7.4.2) to a 125 mL polyethylene bottle. Adjust pH to 8 with approximately 2 mL of ammonium hydroxide solution. Cap and shake on a wrist-action shaker for 24 hours. This iron (III) hydroxide solution is stable for 30 days.
- 7.4.4 Hydrochloric acid—High-purity, 10% solution, shipped with sampling kit in fluoropolymer wash bottles for cleaning trivalent chromium sample preservation equipment between samples.
- 7.4.5 Chromium stock standard solution (1000  $\mu$ g/mL)—Prepared by adding 3.1 g anhydrous chromium chloride to a 1 L flask and diluting to volume with 1% hydrochloric acid. Store in polyethylene bottle. A commercially available standard solution may be substituted.
- 7.4.6 Standard chromium spike solution (1000  $\mu$ g/L)—Used to spike sample aliquots for matrix spike/matrix spike duplicate (MS/MSD) analysis and to prepare ongoing precision and recovery standards. Prepared by spiking 1 mL of the chromium stock standard solution (Section 7.4.5) into a 1 L flask. Dilute to volume with 1% HCl. Store in a polyethylene bottle.
- 7.4.7 Ongoing precision and recovery (OPR) standard (25  $\mu$ g/L)—Prepared by spiking 2.5 mL of the standard chromium spike solution (Section 7.4.6) into a 100 mL flask. Dilute to volume with 1% HCl. One OPR is required for every 10 samples.

### 8.0 Sample Collection, Filtration, and Handling

- 8.1 Site Selection
  - 8.1.1 Selection of a representative site for surface water sampling is based on many factors including: study objectives, water use, point source discharges, non-point source discharges, tributaries, changes in stream characteristics, types of stream bed, stream depth, turbulence, and the presence of structures (bridges, dams, etc.). When collecting samples to determine ambient levels of trace metals, the presence of potential sources of metal contamination are of extreme importance in site selection.
  - 8.1.2 Ideally, the selected sampling site will exhibit a high degree of cross-sectional homogeneity. It may be possible to use previously collected data to identify locations for samples that are well mixed or are vertically or horizontally stratified. Since mixing is principally governed by turbulence and water velocity, the selection of a site immediately downstream of a riffle area will ensure good vertical mixing. Horizontal mixing occurs in constrictions in the channel. In the absence of turbulent areas, the selection of a site that is clear of immediate point sources, such

as industrial effluents, is preferred for the collection of ambient water samples (Reference 19).

- 8.1.3 To minimize contamination from trace metals in the atmosphere, ambient water samples should be collected from sites that are as far as possible (e.g., at least several hundred feet) from any metal supports, bridges, wires or poles. Similarly, samples should be collected as far as possible from regularly or heavily traveled roads. If it is not possible to avoid collection near roadways, it is advisable to study traffic patterns and plan sampling events during lowest traffic flow (Reference 7).
- 8.1.4 The sampling activity should be planned to collect samples known or suspected to contain the lowest concentrations of trace metals first, finishing with the samples known or suspected to contain the highest concentrations. For example, if samples are collected from a flowing river or stream near an industrial or municipal discharge, the upstream sample should be collected first, the downstream sample collected second, and the sample nearest the discharge collected last. If the concentrations of pollutants is not known and cannot be estimated, it is necessary to use precleaned sampling equipment at each sampling location.
- 8.2 Sample Collection Procedure—Before collecting ambient water samples, consideration should be given to the type of sample to be collected, the amount of sample needed, and the devices to be used (grab, surface, or subsurface samplers). Sufficient sample volume should be collected to allow for necessary quality control analyses, such as matrix spike/matrix spike duplicate analyses.
  - 8.2.1 Four sampling procedures are described:
    - 8.2.1.1 Section 8.2.5 describes a procedure for collecting samples directly into the sample container. This procedure is the simplest and provides the least potential for contamination because it requires the least amount of equipment and handling.
    - 8.2.1.2 Section 8.2.6 describes a procedure for using a grab sampling device to collect samples.
    - 8.2.1.3 Section 8.2.7 describes a procedure for depth sampling with a jar sampler. The size of sample container used is dependent on the amount of sample needed by the analytical laboratory.
    - 8.2.1.4 Section 8.2.8 describes a procedure for continuous-flow sampling using a submersible or peristaltic pump.
  - 8.2.2 The sampling team should ideally approach the site from down current and downwind to prevent contamination of the sample by particles sloughing off the boat or equipment. If it is not possible to approach from both, the site should be approached from down current if sampling from a boat or approached from downwind if sampling on foot. When sampling from a boat, the bow of the boat should be oriented into the current (the boat will be pointed upstream). All sampling activity should occur from the bow.

If the samples are being collected from a boat, it is recommended that the sampling team create a stable workstation by arranging the cooler or shipping container as a work table on the upwind side of the boat, covering this worktable and the upwind gunnel with plastic wrap or a plastic tablecloth, and draping the wrap or cloth over the gunnel. If necessary, duct tape is used to hold the wrap or cloth in place.

8.2.3 All operations involving contact with the sample bottle and with transfer of the sample from the sample collection device to the sample bottle (if the sample is not directly collected in the bottle) are handled by the individual designated as "clean hands." "Dirty hands" is responsible for all activities that do not involve direct contact with the sample.

Although the duties of "clean hands" and "dirty hands" would appear to be a logical separation of responsibilities, in fact, the completion of the entire protocol may require a good deal of coordination and practice. For example, "dirty hands" must open the box or cooler containing the sample bottle and unzip the outer bag; clean hands must reach into the outer bag, open the inner bag, remove the bottle, collect the sample, replace the bottle lid, put the bottle back into the inner bag, and zip the inner bag. "Dirty hands" must close the outer bag and place it in a cooler.

To minimize unnecessary confusion, it is recommended that a third team member be available to complete the necessary sample documentation (e.g., to document sampling location, time, sample number, etc). Otherwise, "dirty hands" must perform the sample documentation activity (Reference 7).

- 8.2.4 Extreme care must be taken during all sampling operations to minimize exposure of the sample to human, atmospheric, and other sources of contamination. Care must be taken to avoid breathing directly on the sample, and whenever possible, the sample bottle should be opened, filled, and closed while submerged.
- 8.2.5 Manual collection of surface samples directly into the sample bottle.
  - 8.2.5.1 At the site, all sampling personnel must put on clean gloves (Section 6.7) before commencing sample collection activity, with "clean hands" donning shoulder-length gloves. If samples are to be analyzed for mercury, the sampling team must also put their precleaned wind suits on at this time. Note that "clean hands" should put on the shoulder-length polyethylene gloves (Section 6.7.1) and both "clean hands" and "dirty hands" should put on the PVC gloves (Section 6.7.2).
  - 8.2.5.2 "Dirty hands" must open the cooler or storage container, remove the double-bagged sample bottle from storage, and unzip the outer bag.
  - 8.2.5.3 Next, "clean hands" opens the inside bag containing the sample bottle, removes the bottle, and reseals the inside bag. "Dirty hands" then reseals the outer bag.
  - 8.2.5.4 "Clean hands" unscrews the cap and, while holding the cap upside down, discards the dilute acid solution from the bottle into a carboy for wastes (Section 6.16) or discards the reagent water directly into the water body.

- 8.2.5.5 "Clean hands" then submerges the sample bottle, and allows the bottle to partially fill with sample. "Clean hands" screws the cap on the bottle, shakes the bottle several times, and empties the rinsate away from the site. After two more rinsings, "clean hands" holds the bottle under water and allows bottle to fill with sample. After the bottle has filled (i.e., when no more bubbles appear), and while the bottle is still inverted so that the mouth of the bottle is underwater, "clean hands" replaces the cap of the bottle. In this way, the sample has never contacted the air.
- 8.2.5.6 Once the bottle lid has been replaced, "dirty hands" reopens the outer plastic bag, and "clean hands" opens the inside bag, places the bottle inside it, and zips the inner bag.
- 8.2.5.7 "Dirty hands" zips the outer bag.
- 8.2.5.8 Documentation—After each sample is collected, the sample number is documented in the sampling log, and any unusual observations concerning the sample and the sampling are documented.
- 8.2.5.9 If the sample is to be analyzed for dissolved metals, it is filtered in accordance with the procedure described in Section 8.3.
- 8.2.6 Sample collection with grab sampling device—The following steps detail sample collection using the grab sampling device shown in Figure 1 and described in Section 6.4.1. The procedure is indicative of the "clean hands/dirty hands" technique that must be used with alternative grab sampling devices such as that shown in Figure 2 and described in Section 6.4.2.
  - 8.2.6.1 The sampling team puts on gloves (and wind suits, if applicable). Ideally, a sample bottle will have been preattached to the sampling device in the class 100 clean room at the laboratory. If it is necessary to attach a bottle to the device in the field, "clean hands" performs this operation, described in Section 6.4.2, inside the field-portable glove bag (Section 6.6).
  - 8.2.6.2 "Dirty hands" removes the sampling device from its storage container and opens the outer polyethylene bag.
  - 8.2.6.3 "Clean hands" opens the inside polyethylene bag and removes the sampling device.
  - 8.2.6.4 "Clean hands" changes gloves.
  - 8.2.6.5 "Dirty hands" submerges the sampling device to the desired depth and pulls the fluoropolymer pull cord to bring the seal plate into the middle position so that water can enter the bottle.
  - 8.2.6.6 When the bottle is full (i.e., when no more bubbles appear), "dirty hands" pulls the fluoropolymer cord to the final stop position to seal off the sample and removes the sampling device from the water.
  - 8.2.6.7 "Dirty hands" returns the sampling device to its large inner plastic bag, "clean hands" pulls the bottle out of the collar, unscrews the bottle from the

sealing device, and caps the bottle. "Clean hands" and "dirty hands" then return the bottle to its double-bagged storage as described in Sections 8.2.5.6 through 8.2.5.7.

- 8.2.6.8 Closing mechanism—"Clean hands" removes the closing mechanism from the body of the grab sampler, rinses the device with reagent water (Section 7.1), places it inside a new clean plastic bag, zips the bag, and places the bag inside an outer bag held by "dirty hands." "Dirty hands" zips the outer bag and places the double-bagged closing mechanism in the equipment storage box.
- 8.2.6.9 Sampling device—"Clean hands" seals the large inside bag containing the collar, pole, and cord and places the bag into a large outer bag held by "dirty hands." "Dirty hands" seals the outside bag and places the double-bagged sampling device into the equipment storage box.
- 8.2.6.10 Documentation—After each sample is collected, the sample number is documented in the sampling log, and any unusual observations concerning the sample and the sampling are documented.
- 8.2.6.11 If the sample is to be analyzed for dissolved metals, it is filtered in accordance with the procedures described in Section 8.3.
- 8.2.7 Depth sampling using a jar sampling device (Figure 3 and Section 6.5.1)
  - 8.2.7.1 The sampling team puts on gloves (and wind suits, if applicable) and handles bottles as with manual collection (Sections 8.2.5.1 through 8.2.5.4 and 8.2.5.6 through 8.2.5.7).
  - 8.2.7.2 "Dirty hands" removes the jar sampling device from its storage container and opens the outer polyethylene bag.
  - 8.2.7.3 "Clean hands" opens the inside polyethylene bag and removes the jar sampling apparatus. Ideally, the sampling device will have been preassembled in a class 100 clean room at the laboratory. If, however, it is necessary to assemble the device in the field, "clean hands" must perform this operation, described in Section 6.5.2, inside a field-portable glove bag (Section 6.6).
  - 8.2.7.4 While "dirty hands" is holding the jar sampling apparatus, "clean hands" connects the pump to the to the 1/4 in. o.d. flush line.
  - 8.2.7.5 "Dirty hands" lowers the weighted sampler to the desired depth.
  - 8.2.7.6 "Dirty hands" turns on the pump allowing a large volume (>2 L) of water to pass through the system.
  - 8.2.7.7 After stopping the pump, "dirty hands" pulls up the line, tubing, and device and places them into either a field-portable glove bag or a large, clean plastic bag as they emerge.

- 8.2.7.8 Both "clean hands" and "dirty hands" change gloves.
- 8.2.7.9 Using the technique described in Sections 8.2.5.2 through 8.2.5.4, the sampling team removes a sample bottle from storage, and "clean hands" places the bottle into the glove bag.
- 8.2.7.10 "Clean hands" tips the sampling jar and dispenses the sample through the short length of fluoropolymer tubing into the sample bottle.
- 8.2.7.11 Once the bottle is filled, "clean hands" replaces the cap of the bottle, returns the bottle to the inside polyethylene bag, and zips the bag. "Clean hands" returns the zipped bag to the outside polyethylene bag held by "dirty hands."
- 8.2.7.12 "Dirty hands" zips the outside bag. If the sample is to be analyzed for dissolved metals, it is filtered as described in Section 8.3.
- 8.2.7.13 Documentation—After each sample is collected, the sample number is documented in the sampling log, and any unusual observations concerning the sample and the sampling are documented.
- 8.2.8 Continuous-flow sampling (Figure 4 and Section 6.5.2)—The continuous-flow sampling system uses peristaltic pump (Section 6.15) to pump sample to the boat or to shore through the SEBS-resin or PTFE tubing.
  - 8.2.8.1 Before putting on wind suits or gloves, the sampling team removes the bags containing the pump (Section 6.15), SEBS-resin tubing (Section 6.15.2), batteries (Section 6.15.4), gloves (Section 6.7), plastic wrap (Section 6.9), wind suits (Section 6.12), and, if samples are to be filtered, the filtration apparatus (Section 6.14) from the coolers or storage containers in which they are packed.
  - 8.2.8.2 "Clean hands" and "dirty hands" put on the wind suits and PVC gloves (Section 6.7.2).
  - 8.2.8.3 "Dirty hands" removes the pump from its storage bag, and opens the bag containing the SEBS-resin tubing.
  - 8.2.8.4 "Clean hands" installs the tubing while "dirty hands" holds the pump. "Clean hands" immerses the inlet end of the tubing in the sample stream.
  - 8.2.8.5 Both "clean hands" and "dirty hands" change gloves. "Clean hands" also puts on shoulder length polyethylene gloves (Section 6.7.1).
  - 8.2.8.6 "Dirty hands" turns the pump on and allows the pump to run for 5-10 minutes or longer to purge the pump and tubing.
  - 8.2.8.7 If the sample is to be filtered, "clean hands" installs the filter at the end of the tubing, and "dirty hands" sets up the filter holder on the gunwale as shown in Figure 4.

**NOTE:** The filtration apparatus is not attached until immediately before sampling to prevent buildup of particulates from clogging the filter.

- 8.2.8.8 The sample is collected by rinsing the sample bottle and cap three times and collecting the sample from the flowing stream.
- 8.2.8.9 Documentation—After each sample is collected, the sample number is documented in the sampling log, and any unusual observations concerning the sample and the sampling are documented.
- 8.3 Sample Filtration—The filtration procedure described below is used for samples collected using the manual (Section 8.2.5), grab (Section 8.2.6), or jar (Section 8.2.7) collection systems (Reference 7). In-line filtration using the continuous-flow approach is described in Section 8.2.8.7. Because of the risk of contamination, it is recommended that samples for mercury be shipped unfiltered by overnight courier and filtered when received at the laboratory.
  - 8.3.1 Set up the filtration system inside the glove bag, using the shortest piece of pump tubing as is practicable. Place the peristaltic pump immediately outside of the glove bag and poke a small hole in the glove bag for passage of the tubing. Also, attach a short length of tubing to the outlet of the capsule filter.
  - 8.3.2 "Clean hands" removes the water sample from the inner storage bag using the technique described in Sections 8.2.5.2 through 8.2.5.4 and places the sample inside the glove bag. "Clean hands" also places two clean empty sample bottles, a bottle containing reagent water, and a bottle for waste in the glove bag.
  - 8.3.3 "Clean hands" removes the lid of the reagent water bottle and places the end of the pump tubing in the bottle.
  - 8.3.4 "Dirty hands" starts the pump and passes approximately 200 mL of reagent water through the tubing and filter into the waste bottle. "Clean hands" then moves the outlet tubing to a clean bottle and collects the remaining reagent water as a blank. "Dirty hands" stops the pump.
  - 8.3.5 "Clean hands" removes the lid of the sample bottle and places the intake end of the tubing in the bottle.
  - 8.3.6 "Dirty hands" starts the pump and passes approximately 50 mL through the tubing and filter into the remaining clean sample bottle and then stops the pump. "Clean hands" uses the filtrate to rinse the bottle, discards the waste sample, and returns the outlet tube to the sample bottle.
  - 8.3.7 "Dirty hands" starts the pump and the remaining sample is processed through the filter and collected in the sample bottle. If preservation is required, the sample is acidified at this point (Section 8.4).
  - 8.3.8 "Clean hands" replaces the lid on the bottle, returns the bottle to the inside bag, and zips the bag. "Clean hands" then places the zipped bag into the outer bag held by "dirty hands."

8.3.9 "Dirty hands" zips the outer bag, and places the double-bagged sample bottle into a clean, ice-filled cooler for immediate shipment to the laboratory.

**NOTE:** It is not advisable to reclean and reuse filters. The difficulty and risk associated with failing to properly clean these devices far outweighs the cost of purchasing a new filter.

#### 8.4 Preservation

- 8.4.1 Field preservation is not necessary for dissolved metals, except for trivalent and hexavalent chromium, provided that the sample is preserved in the laboratory and allowed to stand for at least two days to allow the metals adsorbed to the container walls to redissolve. Field preservation is advised for hexavalent chromium in order to provide sample stability for up to 30 days. Mercury samples should be shipped by overnight courier and preserved when received at the laboratory.
- 8.4.2 If field preservation is required, preservation must be performed in the glove bag or in a designated clean area, with gloved hands, as rapidly as possible to preclude particulates from contaminating the sample. For preservation of trivalent chromium, the glove bag or designated clean area must be large enough to accommodate the vacuum filtration apparatus (Section 6.17.3), and an area should be available for setting up the wrist-action shaker (Section 6.17.5). It is also advisable to set up a work area that contains a "clean" cooler for storage of clean equipment, a "dirty" cooler for storage of "dirty" equipment, and a third cooler to store samples for shipment to the laboratory.
- 8.4.3 Preservation of aliquots for metals other than trivalent and hexavalent chromium—Using a disposable, precleaned, plastic pipet, add 5 mL of a 10% solution of ultrapure nitric acid in reagent water per liter of sample. This will be sufficient to preserve a neutral sample to pH < 2.
- 8.4.4 Preservation of aliquots for trivalent chromium (References 8-9).
  - 8.4.4.1 Decant 100 mL of the sample into a clean polyethylene bottle.
  - 8.4.4.2 Clean an Eppendorf pipet by pipeting 1 mL of 10% HCl (Section (7.4.4) followed by 1 mL of reagent water into an acid waste container. Use the rinsed pipet to add 1 mL of chromium (III) extraction solution (Section 7.4.3) to each sample and blank.
  - 8.4.4.3 Cap each bottle tightly, place in a clean polyethylene bag, and shake on a wrist action shaker (Section 6.17.5) for one hour.
  - 8.4.4.4 Vacuum-filter the precipitate through a 0.4  $\mu$ m pretreated filter membrane (Section 6.17.2), using fluoropolymer forceps (Section 6.17.1) to handle the membrane, and a 47 mm vacuum filtration apparatus with a precleaned filter holder (Section 6.17.3). After all sample has filtered, rinse the inside of the filter holder with approximately 15 mL of reagent water.
  - 8.4.4.5 Using the fluoropolymer forceps, fold the membrane in half and then in quarters, taking care to avoid touching the side containing the filtrate to any surface. (Folding is done while the membrane is sitting on the filter holder and allows easy placement of the membrane into the sample vial). Transfer

the filter to a 30 mL fluoropolymer vial. If the fluoropolymer vial was not pre-equipped with the ultrapure nitric acid (Section 7.4.1), rinse the pipet by drawing and discharging 1 mL of 10% HCl followed by 1 mL of reagent water into a waste container, and add 1 mL of ultrapure nitric acid to the sample vial.

- 8.4.4.6 Cap the vial and double-bag it for shipment to the laboratory.
- 8.4.4.7 Repeat Steps 8.4.4.4-8.4.4.6 for each sample, rinsing the fluoropolymer forceps and the pipet with 10% high-purity HCl followed by reagent water between samples.
- 8.4.5 Preservation of aliquots for hexavalent chromium (Reference 20).
  - 8.4.5.1 Decant 125 mL of sample into a clean polyethylene bottle.
  - 8.4.5.2 Prepare an Eppendorf pipet by pipeting 1 mL of 10% HCl (Section 7.4.4) followed by 1 mL of reagent water into an acid waste container. Use the rinsed pipet to add 1 mL NaOH to each 125 mL sample and blank aliquot.
  - 8.4.5.3 Cap the vial(s) and double-bag for shipment to the laboratory.

#### 9.0 Quality Assurance/Quality Control

- 9.1 The sampling team shall employ a strict quality assurance/ quality control (QA/QC) program. The minimum requirements of this program include the collection of equipment blanks, field blanks, and field replicates. It is also desirable to include blind QC samples as part of the program. If samples will be processed for trivalent chromium determinations, the sampling team shall also prepare method blank, OPR, and MS/MSD samples as described in Section 9.6.
- 9.2 The sampling team is permitted to modify the sampling techniques described in this method to improve performance or reduce sampling costs, provided that reliable analyses of samples are obtained and that samples and blanks are not contaminated. Each time a modification is made to the procedures, the sampling team is required to demonstrate that the modification does not result in contamination of field and equipment blanks. The requirements for modification are given in Sections 9.3 and 9.4. Because the acceptability of a modification is based on the results obtained with the modification, the sampling team must work with an analytical laboratory capable of making trace metals determinations to demonstrate equivalence.
- 9.3 Equipment Blanks
  - 9.3.1 Before using any sampling equipment at a given site, the laboratory or equipment cleaning contractor is required to generate equipment blanks to demonstrate that the equipment is free from contamination. Two types of equipment blanks are required: bottle blanks and sampling equipment blanks.
  - 9.3.2 Equipment blanks must be run on all equipment that will be used in the field. If, for example, samples are to be collected using both a grab sampling device and the jar sampling device, then an equipment blank must be run on both pieces of equipment.

- 9.3.3 Equipment blanks are generated in the laboratory or at the equipment cleaning contractor's facility by processing reagent water through the equipment using the same procedures that are used in the field (Section 8.0). Therefore, the "clean hands/dirty hands" technique used during field sampling should be followed when preparing equipment blanks at the laboratory or cleaning facility. In addition, training programs must require must require sampling personnel to collect a clean equipment blank before performing on-site field activities.
- 9.3.4 Detailed procedures for collecting equipment blanks are given in the analytical methods referenced in Table 1.
- 9.3.5 The equipment blank must be analyzed using the procedures detailed in the referenced analytical method (see Table 1). If any metal(s) of interest or any potentially interfering substance is detected in the equipment blank at the minimum level specified in the referenced method, the source of contamination/interference must be identified and removed. The equipment must be demonstrated to be free from the metal(s) of interest before the equipment may be used in the field.

#### 9.4 Field Blank

- 9.4.1 To demonstrate that sample contamination has not occurred during field sampling and sample processing, at least one field blank must be generated for every 10 samples that are collected at a given site. Field blanks are collected before sample collection.
- 9.4.2 Field blanks are generated by filling a large carboy or other appropriate container with reagent water (Section 7.1) in the laboratory, transporting the filled container to the sampling site, processing the water through each of the sample processing steps and equipment (e.g., tubing, sampling devices, filters, etc.) that will be used in the field, collecting the field blank in one of the sample bottles, and shipping the bottle to the laboratory for analysis in accordance with the method(s) referenced in Table 1. For example, manual grab sampler field blanks are collected by directly submerging a sample bottle into the water, filling the bottle, and capping. Subsurface sampler field blanks are collected by immersing the tubing into the water and pumping water into a sample container.
- 9.4.3 Filter the field blanks using the procedures described in Section 8.3.
- 9.4.4 If it is necessary to acid clean the sampling equipment between samples (Section 10.0), a field blank should be collected after the cleaning procedures but before the next sample is collected.
- 9.4.5 If trivalent chromium aliquots are processed, a separate field blank must be collected and processed through the sample preparation steps given in Sections 8.4.4.1 through 8.4.4.6.
- 9.5 Field Duplicate
  - 9.5.1 To assess the precision of the field sampling and analytical processes, at least one field duplicate sample must be collected for every 10 samples that are collected at a given site.

- 9.5.2 The field duplicate is collected either by splitting a larger volume into two aliquots in the glove box, by using a sampler with dual inlets that allows simultaneous collection of two samples, or by collecting two samples in rapid succession.
- 9.5.3 Field duplicates for dissolved metals determinations must be processed using the procedures in Section 8.3. Field duplicates for trivalent chromium must be processed through the sample preparation steps given in Sections 8.4.4.1 through 8.4.4.6.
- 9.6 Additional QC for Collection of Trivalent Chromium Aliquots
  - 9.6.1 Method blank—The sampling team must prepare one method blank for every ten or fewer field samples. Each method blank is prepared using the steps in Sections 8.4.4.1 through 8.4.4.6 on a 100 mL aliquot of reagent water (Section 7.1). Do not use the procedures in Section 8.3 to process the method blank through the 0.45  $\mu$ m filter (Section 6.14.1), even if samples are being collected for dissolved metals determinations.
  - 9.6.2 Ongoing precision and recovery (OPR)—The sampling team must prepare one OPR for every ten or fewer field samples. The OPR is prepared using the steps in Sections 8.4.4.1 through 8.4.4.6 on the OPR standard (Section 7.4.7). Do not use the procedures in Section 8.3 to process the OPR through the 0.45 μm filter (Section 6.14.1), even if samples are being collected for dissolved metals determinations.
  - 9.6.3 MS/MSD—The sampling team must prepare one MS and one MSD for every ten or fewer field samples.
    - 9.6.3.1 If, through historical data, the background concentration of the sample can be estimated, the MS and MSD samples should be spiked at a level of one to five times the background concentration.
    - 9.6.3.2 For samples in which the background concentration is unknown, the MS and MSD samples should be spiked at a concentration of  $25 \ \mu g/L$ .
    - 9.6.3.3 Prepare the matrix spike sample by spiking a 100-mL aliquot of sample with 2.5 mL of the standard chromium spike solution (Section 7.4.6), and processing the MS through the steps in Sections 8.4.4.1 through 8.4.4.6.
    - 9.6.3.4 Prepare the matrix spike duplicate sample by spiking a second 100-mL aliquot of the same sample with 2.5 mL of the standard chromium spike solution, and processing the MSD through the steps in Sections 8.4.4.1 through 8.4.4.6.
    - 9.6.3.5 If field samples are collected for dissolved metals determinations, it is necessary to process an MS and an MSD through the 0.45  $\mu m$  filter as described in Section 8.3.

#### **10.0** Recleaning the Apparatus Between Samples

10.1 Sampling activity should be planned so that samples known or suspected to contain the lowest concentrations of trace metals are collected first with the samples known or

suspected to contain the highest concentrations of trace metals collected last. In this manner, cleaning of the sampling equipment between samples in unnecessary. If it is not possible to plan sampling activity in this manner, dedicated sampling equipment should be provided for each sampling event.

- 10.2 If samples are collected from adjacent sites (e.g., immediately upstream or downstream), rinsing of the sampling Apparatus with water that is to be sampled should be sufficient.
- 10.3 If it is necessary to cross a gradient (i.e., going from a high-concentration sample to a lowconcentration sample), such as might occur when collecting at a second site, the following procedure may be used to clean the sampling equipment between samples:
  - 10.3.1 In the glove bag, and using the "clean hands/dirty hands" procedure in Section 8.2.5, process the dilute nitric acid solution (Section 7.2) through the Apparatus.
  - 10.3.2 Dump the spent dilute acid in the waste carboy or in the waterbody away from the sampling point.
  - 10.3.3 Process 1 L of reagent water through the Apparatus to rinse the equipment and discard the spent water.
  - 10.3.4 Collect a field blank as described in Section 9.4.
  - 10.3.5 Rinse the Apparatus with copious amounts of the ambient water sample and proceed with sample collection.
- 10.4 Procedures for recleaning trivalent chromium preservation equipment between samples are described in Section 8.4.4.

#### **11.0 Method Performance**

Samples were collected in the Great Lakes during September–October 1994 using the procedures in this sampling method.

#### **12.0 Pollution Prevention**

- 12.1 The only materials used in this method that could be considered pollutants are the acids used in the cleaning of the Apparatus, the boat, and related materials. These acids are used in dilute solutions in small amounts and pose little threat to the environment when managed properly.
- 12.2 Cleaning solutions containing acids should be prepared in volumes consistent with use to minimize the disposal of excessive volumes of acid.
- 12.3 To the extent possible, the Apparatus used to collect samples should be cleaned and reused to minimize the generation of solid waste.

#### **13.0 Waste Management**

13.1 It is the sampling team's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the discharge regulations, hazardous

waste identification rules, and land disposal restrictions; and to protect the air, water, and land by minimizing and controlling all releases from field operations.

13.2 For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* and *Less is Better—Laboratory Chemical Management for Waste Reduction,* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW, Washington, DC 20036.

### 14.0 References

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#### **15.0 Glossary of Definitions and Purposes**

These definitions and purposes are specific to this sampling method but have been conformed to common usage as much as possible.

- 15.1 Ambient Water—Waters in the natural environment (e.g., rivers, lakes, streams, and other receiving waters), as opposed to effluent discharges.
- 15.2 Apparatus—The sample container and other containers, filters, filter holders, labware, tubing, pipets, and other materials and devices used for sample collection or sample preparation, and that will contact samples, blanks, or analytical standards.
- 15.3 Equipment Blank—An aliquot of reagent water that is subjected in the laboratory to all aspects of sample collection and analysis, including contact with all sampling devices and apparatus. The purpose of the equipment blank is to determine if the sampling devices and apparatus for sample collection have been adequately cleaned before they are shipped to the field site. An acceptable equipment blank must be achieved before the sampling devices and Apparatus are used for sample collection.
- 15.4 Field Blank—An aliquot of reagent water that is placed in a sample container in the laboratory, shipped to the field, and treated as a sample in all respects, including contact with the sampling devices and exposure to sampling site conditions, filtration, storage,

preservation, and all analytical procedures. The purpose of the field blank is to determine whether the field or sample transporting procedures and environments have contaminated the sample.

- 15.5 Field Duplicates (FD1 and FD2)—Two identical aliquots of a sample collected in separate sample bottles at the same time and place under identical circumstances using a duel inlet sampler or by splitting a larger aliquot and treated exactly the same throughout field and laboratory procedures. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.
- 15.6 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)—Aliquots of an environmental sample to which known quantities of the analytes are added in the laboratory. The MS and MSD are analyzed exactly like a sample. Their purpose is to quantify the bias and precision caused by the sample matrix. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS and MSD corrected for background concentrations.
- 15.7 May—This action, activity, or procedural step is optional.
- 15.8 May Not—This action, activity, or procedural step is prohibited.
- 15.9 Minimum Level (ML)—The lowest level at which the entire analytical system gives a recognizable signal and acceptable calibration point (Reference 21).
- 15.10 Must—This action, activity, or procedural step is required.
- 15.11 Reagent Water—Water demonstrated to be free from the metal(s) of interest and potentially interfering substances at the MDL for that metal in the referenced method or additional method.
- 15.12 Should—This action, activity, or procedural step is suggested but not required.
- 15.13 Trace-Metal Grade—Reagents that have been demonstrated to be free from the metal(s) of interest at the method detection limit (MDL) of the analytical method to be used for determination of this metal(s).

The term "trace-metal grade" has been used in place of "reagent grade" or "reagent" because acids and other materials labeled "reagent grade" have been shown to contain concentrations of metals that will interfere in the determination of trace metals at levels listed in Table 1.

Method	Technique	Metal	<b>MDL (µg/L)</b> <sup>1</sup>	<b>ML (μg/L</b> ) <sup>2</sup>
1631	Oxidation/Purge & Trap/CVAFS	Mercury	0.0002	0.0005
1632	Hydride AA	Arsenic	0.003	0.01
1636	Ion Chromatography	Hexavalent Chromium	0.23	0.5
1637	CC/STGFAA	Cadmium Lead	0.0075 0.036	0.02 0.1
1638	ICP/MS	Antimony Cadmium Copper Lead Nickel Selenium Silver Thallium Zinc	0.0097 0.013 0.087 0.015 0.33 0.45 0.029 0.0079 0.14	$\begin{array}{c} 0.02\\ 0.1\\ 0.2\\ 0.05\\ 1\\ 1\\ 0.1\\ 0.02\\ 0.5 \end{array}$
1639	STGFAA	Antimony Cadmium Trivalent Chromium Nickel Selenium Zinc	1.9 0.023 0.10 0.65 0.83 0.14	5 0.05 0.2 2 2 0.5
1640	CC/ICP/MS	Cadmium Copper Lead Nickel	0.0024 0.024 0.0081 0.029	0.01 0.1 0.02 0.1

# TABLE 1. ANALYTICAL METHODS, METALS, AND CONCENTRATION LEVELSAPPLICABLE TO METHOD 1669

<sup>1</sup>Method Detection Limit as determined by 40 *CFR* Part 136, Appendix B.

<sup>2</sup> Minimum Level (ML) calculated by multiplying laboratory-determined MDL by 3.18 and rounding result to nearest multiple of 1, 2, 5, 10, 20, 50, etc., in accordance with procedures used by EAD and described in the EPA *Draft National Guidance for the Permitting, Monitoring, and Enforcement of Water Quality-Based Effluent Limitations Set Below Analytical Detection/Quantitation Levels*, March 22, 1994.

Metal	Preservation Requirements	Acceptable Containers
Antimony Arsenic Cadmium Copper Lead Nickel Selenium Silver Thallium Zinc	Add 5 mL of 10% HN0 <sub>3</sub> to 1-L sample; preserve on-site or immediately upon laboratory receipt.	500 mL or 1 L fluoropolymer, conventional or linear polyethylene, polycarbonate, or polypropylene containers with lid
Chromium (III)	Add 1 mL chromium (III) extraction solution to 100 mL aliquot, vacuum filter through $0.4 \mu m$ membrane, add 1 mL 10% HN0 <sub>3</sub> ; preserve on-site immediately after collection.	500 mL or 1 L fluoropolymer, conventional or linear polyethylene, polycarbonate, or polypropylene containers with lid
Chromium (IV)	Add 50% NaOH; preserve immediately after sample collection.	500 mL or 1 L fluoropolymer, conventional or linear polyethylene, polycarbonate, or polypropylene containers with lid
Mercury	Total: Add 0.5% high-purity HCl or 0.5% BrCl to pH < 2; Total & Methyl: Add 0.5% high-purity HCL; preserve on- site or immediately upon laboratory receipt	Fluoropolymer or borosilicate glass bottles with fluoropolymer or fluoropolymer-lined caps

 TABLE 2. ANALYTES, PRESERVATION REQUIREMENTS, AND CONTAINERS







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# Figure 4 - Sample Pumping System

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SOP for Collection of Groundwater Samples

# WELL PURGING 4.2.3

Well purging removes standing water from the borehole. The purpose of purging is to reduce chemical and biochemical artifacts caused by the materials and practices used for well installation, well construction, and well development, and by reactions occurring within an open borehole or annular space between a well casing and borehole wall.<sup>15</sup> Purging also serves to condition the sampling equipment with well water. The purging process forms a continuum with that of sample withdrawal. Sample withdrawal is the process by which sample water is transported for collection and processing, after the well has been purged.

## Standard purge procedure 4.2.3.A

As a rule of thumb, the standard USGS purge procedure removes three or more well volumes of standing water while monitoring the water level and the stabilization of routine field measurements as a function of time, pumping rate, and the volume of water being removed (figs. 4-11 and 4-12). Routine field measurements include pH, temperature, specific electrical conductance, dissolved oxygen, and turbidity. Inherent in the purge procedure is an assumption that stabilization of field properties indicates that the discharge water represents ambient formation water. Field personnel should examine this assumption for each well, using their knowledge of the well and aquifer hydraulics. Review of the purging history, including physical and chemical data monitored, can save time and help determine how the well should be purged.

<sup>&</sup>lt;sup>15</sup>Passive sampling methods may not require purging of the well prior to sample collection (Vroblesky, 2001; Powell and Puls, 1993; and Ronen and others, 1987).

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- When calculating a purge volume for a cased well:
  - Include an estimate for the volume of water stored in the annular space between the casing and borehole wall, using knowledge of the borehole diameter. It is mandatory to evacuate at least one borehole volume (that is, casing volume plus that of the annular space), whether that space has been backfilled with formation materials or with a gravel pack.
  - Make the calculation of casing volume using the height of the water column to the bottom of the well, instead of the water column height to the top of the screen.
- The number of well volumes to be evacuated relies on confirming the time over which field measurements stabilize, using knowledge of the well and aquifer hydraulics.
  - To the extent practical, field personnel should apply an understanding of the borehole and aquifer hydraulics for the well to determine when the water being withdrawn from the borehole will likely be dominated by formation water (Shapiro, 2002; Claassen, 1982).
  - Values for field properties are recorded sequentially and at regular time intervals. The frequency of these measurements depends on the purging rate, which in turn is a function of well depth and diameter, and aquifer transmissivity. Fieldproperty stabilization should be plotted as a function of a logarithmic time scale rather than a linear time scale, to best determine the point at which the contribution of aquifer water dominates pump discharge (see Shapiro, 2002). Fieldmeasurement procedures are detailed in NFM 6.
- Purging should not cause substantial drawdown in monitor or supply wells when pumping at a rate of at least 1gal (3.75 L) per minute. Ideally, drawdown will be at a steady state, with the water level remaining above the top of the open or screened interval.
- Use of a borehole packer system or well liner is recommended for wells in fractured or low-yield media, to isolate zones of highest hydraulic conductivity or of particular interest. Transducers should be installed above and below the packers to monitor head differences.

Well volume = $V = 0.0408 HD^2 = $ gallons,	Well	Gallons per
where	casing	foot of
V is volume of water in the well, in gallons,	diameter (D)	casing
<b>D</b> is inside diameter of well, in inches, and	(in inches)	
<b>H</b> is height of water column, in feet		
<b>8 1 1 1 1 1 1 1 1 1 1</b>	1.0	0.04
	1.5	.09
Purge volume = $(n)(V)$ = gallons,	2.0	.16
where	3.0	.37
<i>n</i> is number of well volumes to be removed	4.0	.65
during purging	4.5	.83
aanne parene	5.0	1.02
	6.0	1.47
Q = estimated pumping rate = gallons	8.0	2.61
per minute	10.0	4.08
	12.0	5.88
Approximate purge time = $(purge volume)/Q =$	24.0	23.50
minutes	36.0	52.90

#### **Explanation:**

Well volume: Volume of water in a borehole or cased well.

Well volumes: For cased wells, the actual number of well volumes should account for evacuation of at least one volume of water stored in the annular space between the casing and borehole wall. This can be estimated from knowledge of the drilled well diameter.

Approximate purge time: Actual purge time depends also on field-measurement stabilization (use fig. 4-12).

Figure 4-11. Estimation of purge volume and purge time.

E-47

	D	ate:		Bv:				
SITE ID _			STAT	TION NAME				
IEIGHT (	OF WATER	COLUMN	ſ	D	EPTH OF	WELL		
PUMP IN	TAKE (ft or	m below N	IP): Start	En	d			
WELL-PU	RGING ME	THOD AN	ID PUMP T	YPE (describ	be):			
TIME	WATER LEVEL below *MP LS	DRAW- DOWN	TEMPER- ATURE	CONDUC- TIVITY	рН	DISSOLVED OXYGEN	TURBID- ITY	APPROX PUMPINO RATE
HR:MIN	*ft or m	*ft or m	°Celsius	µS/cm	standard units	mg/L	**	*gpm or L/min
*Circle th meter; μS L/min, lite **Select t	e unit used; /cm, microsi ers per minut he appropria	MP, measuremens per te. tte turbidity	ring point; I centimeter a unit from h	.S, land surfa tt 25°C; mg/I ttp://water.us	ce; HR:MI L, miligram sgs.gov/ow	N, hour and mins per liter; gpn q/turbidity_cod	inutes; ft, fe n, gallons p les.xls.	eet; m, er minute;
*Circle th meter; μS, L/min, lite **Select t <b>Well volu</b> <i>V</i> = volu in feet; <i>i</i> Well volun	e unit used; /cm, microsi ers per minut he appropria $\mathbf{ne} = V = 0.0$ une of water n = number ne is 0.16 ga	MP, measuremens per te. te turbidity $408 HD^2 =$ in well, in of well volu	ring point; I centimeter a v unit from h : gallon gallons; D umes to purg pot for a 2-ir	S, land surfa tt 25°C; mg/I tttp://water.us ns. P <b>urge vo</b> = inside well ge. n. casing dian	cce; HR:MI L, miligram sgs.gov/ow <b>Jume =</b> ( <i>n</i> ) diameter, i neter.	N, hour and mi is per liter; gpn q/turbidity_cod (V) = gall n inches; H = h	inutes; ft, fe n, gallons p les.xls. lons. neight of wa	eet; m, er minute; ater column
*Circle th meter; μS, L/min, lite **Select t <b>Well volu</b> <i>V</i> = volu in feet; <i>i</i> Well volun	e unit used; /cm, microsi ers per minut he appropria $\mathbf{ne} = V = 0.0$ une of water n = number of ne is 0.16 ga	MP, measuremens per te. tte turbidity $M08 HD^2 =$ r in well, in of well volu llons per for EASUREM	ring point; I centimeter a cunit from h cunit from cunit cunit from cunit from cunit cunit from cunit from cunit cunit from cunit from cunit from cunit cunit from cunit fr	S, land surfa tt 25°C; mg/I tttp://water.us ns. P <b>urge vo</b> = inside well ge. n. casing dian	cce; HR:MI , miligram sgs.gov/ow <b>Jume</b> = ( <i>n</i> ) diameter, i neter.	N, hour and mi is per liter; gpn q/turbidity_cod (V) = gali n inches; H = h	inutes; ft, fe n, gallons p les.xls. lons. height of wa	eet; m, er minute; ater column
*Circle th meter; µS/ L/min, lite **Select t Well volun in feet; / Well volun	e unit used; /cm, microsi ers per minut he appropria $\mathbf{ne} = V = 0.0$ ume of water n = number of ne is 0.16 ga	MP, measur emens per te. tte turbidity 0408 HD <sup>2</sup> = in well, in of well volu illons per fo	ring point; I centimeter a cunit from h cunit from cunit from cunit from cunit from cunit from h cunit from h cunit from cunit from cunit from cunit from cunit from cunit from cunit from cunit from cunit from cunit from c	LS, land surfa tt 25°C; mg/I tttp://water.us ns. Purge vo = inside well ge. 1. casing dian	cce; HR:MI , miligram sgs.gov/ow <b>Jume</b> = ( <i>n</i> ) diameter, i neter. <b>S</b>	N, hour and mi is per liter; gpn q/turbidity_cod (V) = gall n inches; H = h	inutes; ft, fe n, gallons p les.xls. lons. aeight of wa	eet; m, er minute; ater column
*Circle th meter; μS/ L/min, lite **Select t <b>Well volun</b> <i>V</i> = volu in feet; <i>i</i> Well volun pH Tempera	e unit used; /cm, microsi ers per minut he appropria $\mathbf{ne} = V = 0.0$ une of water n = number of ne is 0.16 ga FIELD M ture (T) (in o	MP, measu emens per te. tte turbidity 1408 HD <sup>2</sup> = i n well, in of well volu llons per fo	ring point; I centimeter a v unit from h : gallon gallons; D : ames to purş oot for a 2-ir ENT	S, land surfa at 25°C; mg/I ttp://water.us ns. Purge vo = inside well ge. 1. casing dian $\pm 0.1$ $\pm 0.2$ $\pm 0.5$	cce; HR:MI , miligram sgs.gov/ow <b>lume</b> = (n) diameter, i neter. t standard u 2°C (therm °C (liquid	N, hour and mi as per liter; gpn q/turbidity_cod (V) = gall n inches; H = h STABILITY CRI units istor thermome -in-glass therm	inutes; ft, fe n, gallons p les.xls. lons. leight of wa <b>TERIA<sup>1</sup></b> ter) ometer)	eet; m, er minute; ater column
*Circle th meter; µS, L/min, lite **Select t Well volur V = volu in feet; i Well volun pH Tempera	e unit used; /cm, microsi ers per minut he appropria $\mathbf{ne} = V = 0.0$ une of water n = number of ne is 0.16 ga FIELD M ture (T) (in of electrical co	MP, measur emens per te. tte turbidity 9408 HD <sup>2</sup> = r in well, in of well volu 110ns per fo EASUREM degrees Cel	<pre>ing point; I centimeter a unit from h gallon gallons; D umes to purg oot for a 2-ir ENT (SC)</pre>	S, land surfa ttp://water.us ns. Purge vo = inside well ge. n. casing dian $\pm 0.1$ $\pm 0.2$ $\pm 0.5$ $\pm 5\%$ $\pm 3\%$	L, miligram L, miligram L, miligram L, miligram L, miligram L L L L L L L L	N, hour and mi is per liter; gpn $q/turbidity_cod$ (V) = galln inches; $H = hSTABILITY CRIunitsistor thermome-in-glass therm100 µS/cm100 µS/cm$	inutes; ft, fe n, gallons p les.xls. lons. height of wa <b>TERIA<sup>1</sup></b> ter) ometer)	eet; m, er minute; ater column
*Circle th meter; µS/ L/min, lite **Select t <b>Well volun</b> <i>V</i> = volu in feet; <i>i</i> Well volun pH Tempera Specific Dissolve	e unit used; /cm, microsi ers per minut he appropria $\mathbf{ne} = V = 0.0$ ume of water n = number ne is 0.16 ga FIELD M ture (T) (in c electrical co d-oxygen co	MP, measuremens per te. te turbidity 408 HD <sup>2</sup> = r in well, in of well volu llons per for EASUREM degrees Cel nductance of ncentration	<pre>ing point; I centimeter a unit from h gallou gallons; D umes to purg out for a 2-ir ENT sius) (SC) (SC) (DO)</pre>	S, land surfa ttp://water.us as. Purge vo = inside well ge. a. casing dian $\pm 0.1$ $\pm 0.2$ $\pm 0.5$ $\pm 5\%$ $\pm 3\%$ $\pm 0.3$	cce; HR:MI L, miligram sgs.gov/ow <b>lume</b> = (n) diameter, i neter. standard u $2^{\circ}C$ (therm $2^{\circ}C$ (therm) (	N, hour and mi is per liter; gpn $q/turbidity_cod(V) = galln inches; H = hSTABILITY CRIunitsistor thermome-in-glass therm100 \muS/cm$	inutes; ft, fe 1, gallons p les.xls. lons. leight of wa <b>TERIA<sup>1</sup></b> ter) ometer)	eet; m, er minute; ater column

<sup>2</sup>Select appropriate TBY unit from http://water.usgs.gov/owq/turbidity\_codes.xls

Figure 4-12. Example of a field log for well purging.

## Exceptions to the Standard Purge 4.2.3.B Procedure

Site characteristics, well characteristics, or study objectives could require modification of the standard purge procedure by changing the number of well volumes removed or by changing or adding types of field measurements and analyses. **Any modification to the standard well-purging procedure must be documented.** When standard purge volumes cannot be removed, (1) sufficient water must be withdrawn from the well to evacuate at least one borehole volume and to field rinse the sampler and sample tubing—alternatively, flush the pump and tubing system with the equivalent of three tubing volumes of DIW and purge the DIW from the tubing with clean nitrogen gas; and (2) field measurements should be determined before collecting samples, if possible. A lesser purge volume or other procedures may be modified, for example, when:

- A supply well to be sampled is being pumped continuously or daily at regular intervals and long enough to have removed three casing volumes of water—go directly to monitoring field properties.
- ► The sample-collection interval is sealed with packers (the interval to be sampled should be purged of three volumes).
- ► Water-level recovery from drawdown to approximately 90 percent of the original volume in the wellcannot be achieved within a reasonable timeframe (not to exceed 24 hours; see the previous discussion on low-yield wells).
- ► The study will customize the protocol for field-determined properties or constituent analyses to address specific study objectives; however, the routine suite of field-measurement values should be determined.

**TECHNICAL NOTE:** Target or indicator analytes may be added to the purge criteria to address study objectives. The analysis can be performed onsite using portable analytical equipment or a mobile laboratory. The acceptable variability in analyte measurements to define stabilization and minimum number of readings is defined by the study (ASTM International, 2005).
- One or more field measurement keeps drifting, and sampling at that well cannot be avoided—NFM 6 provides suggestions for poor field-measurement stabilization, including extending the purge time and purge volume. Field personnel must make a decision based on their understanding of study objectives whether to extend purge time. Such decisions should be documented in field notes.
- Use of low-flow purging techniques is a stipulated study requirement: for a detailed description of the low-flow purge technique, refer to ASTM standard procedure D6452-99 (ASTM International, 2005).

**TECHNICAL NOTE: Low-flow purging** procedures are designed to minimize the volume of purge water and disturbance of the water column and maximize the contribution of formation water from a given interval of interest (Puls and Barcelona, 1996; Unwin and Huis, 1983). Minimizing purge volume is especially useful when regulating authorities mandate containment of purge water.

Low-flow purging is based on the theory that water moving through the well intake is representative of formation water surrounding the intake, and assumes that pumping at a low flow rate isolates the column of standing water so that only formation water is drawn into the intake. The typical flow rates for this method are on the order of 0.1 to 0.5 L/min; however, in formations of coarse-grained materials the flow rate may be as high as 1 L/min (ASTM International, 2005).

Select a low-flow purge-and-sampling technique with caution and with an understanding of aquifer and well hydraulics. The assumption should not be made that water withdrawn using a low-flow procedure represents ambient aquifer water at the targeted (intake) interval (Varljen and others, 2006), because the conductivity of well-bore flow within the specified interval is greater than that of the aquifer (Shapiro, 2002). Even where well-bore flow does not occur, aquifer heterogeneity over the length of the specified interval results in water being drawn preferentially through zones of highest permeability.

## STEPS FOR SAMPLING AT WELLS 4.2.4

Develop a systematic agenda well in advance of the field effort that follows the sampling plan and quality-assurance protocols. Offsite preparations in addition to the steps needed to carry out onsite activities need to be included in planning for field work. Review the requirements and recommendations for site inventory (reconnaissance) and site file setup (section 4.2.1)

**Field-trip preparations.** Adequate time must be scheduled to plan sampling activities, review data requirements, and make field-trip preparations. Prepare a checklist of equipment and supplies that will be needed, and order what is needed well before the field effort (fig. 4-13). Refer to NFM 2, Section 2.4, for lists of equipment and supplies commonly used for ground-water field activities. Review electronic and paper site files and make sure that they are kept up to date.

Before selecting and implementing purging methods, review table 4-8 to determine how maintaining sample integrity applies to the study and site.

- Consider whether modifications of standard USGS methods might be needed to address issues specific to the field site or program or study objectives. Document any deviation from the standard protocols.
- Review the types of quality-control (QC) samples planned for the study. Certain types of blank samples are required for all USGS studies. Review the most recent analyses of blank samples collected through the equipment to be used for sampling before field work begins.
- Determine if water level and well yield are sufficient to produce a representative sample.
- Decide how to determine or constrain the interval(s) from which the sample shouldbe collected. Consider whether packers will be used and whether screen lengths are sufficiently short or long to meet the sampling objective. Determine the major sources of flow contribution to the well, if sampling in fractured or anisotropic formation materials.

Before leaving for the field site, review reconnaissance notes from the site inventory (table 4-6), and determine the number and types of environmental and QC samples to be collected (Appendix A4-C).

- Prepare the field forms that will be needed (for example, waterlevel, purging, field-measurement, analytical services request, and chain-of-custody forms). Fill out as much information as possible, including the equipment to be used and numbers and types of samples to be collected.
- Check equipment requirements (NFM 2). When assembling the equipment, test that equipment is in good working condition. Take backup equipment, as appropriate.
  - Organic-compound samples. Use fluorocarbon polymer (Teflon), glass, or metal for equipment components that will be in contact with samples to be analyzed for organic compounds. Exception: if collecting CFC samples, do not use Teflon sampler components or Teflon tubing (NFM 5).
  - Inorganic-constituent samples. Use fluorocarbon polymer or other relatively inert and uncolored plastics or glass for any equipment components that will be in contact with samples to be analyzed for inorganic constituents. Do not use metal or rubber components for trace-element sampling. Stainless-steel sheathed pumps are generally acceptable, but can leach low concentrations of chromium, molybdenum, nickel, and vanadium to the sample. Collect an equipment blank to be analyzed before sampling begins, to demonstrate the acceptability of the data to be collected.
- Set up a clean workspace (usually in the water-quality field vehicle) and thesample-processing and -preærvation chambers. Place the filter unit and other necessary supplies for sample collection and processing into the processing chamber. The generator and gas tanks must not be stored or transported in the water-quality field vehicle.

# Plan ahead. Take adequate time for site recon, and to prepare sampling plans, order supplies, test equipment, and get the training needed.

1	Checklist for ground-water site setup and well-sampling preparations <sup>1</sup>
	Antibacksiphon device (one-way or check valve)
	Chemical reagents (for sample preservation and field analyses) and ice
	Deionized water and blank water
	Disposable, powderless, laboratory-grade gloves
	Equipment cleaning, decontamination, and disinfectant supplies
	Field forms (for example, ground-water-quality, water-level, and chain-of- custody forms) - electronic or paper; indelible ballpoint pen (black or blue ink)
	Field manual, sampling and quality-control plan(s)
	Filtration units and supplies
	Flow-regulating valve (needle valve or pinch clamps)
	Flow-splitting valve(s) for manifold system
	Flowthrough cell or chamber and field-measurement instrument(s) (single parameter or multiparameter); standard and buffer solutions; Kimwipes (see NFM 6)
	Keys (for locked facilities)
	Microbiota sampling supplies (see NFM 7)
	Photoionization detector (PID or sniffer)
	Sample processing and preservation chambers in which samples are bottled and treated, respectively, and associated supplies
	Safety equipment
	Sample containers (precleaned)
	Sampling device(s) (precleaned, portable equipment or other, as appropriate) and power supply (if needed); spare batteries
	Sample tubing (precleaned, several lengths)
	Shipping containers and supplies
	Stopwatch and calibrated bucket to measure pumping rate
	Tarp or plastic sheeting to place around well
	Threaded fittings, male/female, such as hose-type connectors (precleaned)
	Tools (such as wrenches to remove well cap)
	Tubing to direct waste discharge offsite or into sample container
	Water-level measurement equipment
<sup>1</sup> See I sampl	NFM 2.4 for more detailed examples of equipment and supply checklists for ing.
Figur	e 4-13. Example of checklist of equipment and supplies to prepare for

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sampling ground water at wells.

**Steps for sampling.** The standard USGS procedure for collecting ground-water samples consists of the following six basic steps and the activities needed to carry them out. The procedures needed for supply wells differ somewhat from those used for monitor wells. Steps 1 through 4 are detailed in this section. Steps 5 and 6 are described in NFM 5 ("Processing of Water Samples") and NFM 3 ("Cleaning of Equipment for Water Sampling"), respectively.

## Step 1. Implement safety precautions and site preparations

Act with common sense. Be aware of existing and impending environmental conditions and hazards. Field personnel must be familiar with the guidance and protocols provided in NFM 9, "Safety in Field Activities." Organized and orderly procedures for setting up a site for sampling should be routine and helps to prevent mistakes that could compromise personnel safety as well as sample integrity.

## Step 2. Measure water level

Procedures for water-level measurement can differ for supply wells and monitor wells. Detailed procedures for various methods of measuring water levels are documented by the U.S. Geological Survey (1980, p. 2-8), and additional information can be obtained from the USGS Office of Ground Water (<u>http://water.usgs.gov/ogw</u>). Refer to Appendix A4-B for a summary of water-level-measurement methods.

- Procedures and equipment for water-level measurement can differ, depending on the type, construction, and design of a well.
- Clean well tapes after each use at a well as described in NFM 3.3.8. Document in field notes if oil is floating on the water table. Review equipment-cleaning and sample-collection strategies and revise as needed if oil is present, to prevent contamination of samples. A dual-phase sonde can be used to determine the thickness of the oil layer, as well as the depth to water.
- Record discrete water-level measurements on field forms and in GWSI (USGS Office of Water Quality Technical Memorandum 2006.01).

## Step 3. Purge the well and monitor field measurements

As discussed in Section 4.2.3, purging the well of standing water is generally required to ensure that the sample water will be withdrawn directly from the aquifer. Exceptions to the well-purging protocol may apply more commonly to water-supply wells, although exceptions for some monitor wells also have been described in the previous section. Regardless of the purge procedure followed, enough water must be withdrawn from the well to field rinse sampling equipment and to make measurements of field properties (field measurements). Purging and field-measurement information must be recorded, either on electronic or paper field forms (fig. 4-12). Specific guidance for use of field-measurement instruments is described in detail in NFM 6.

## Step 4. Withdraw the sample

As a rule, pumping is the preferred method for withdrawal of groundwater samples. In this case, purging and sample withdrawal form a continuous process. Field measurements are monitored during purging with sample collection following immediately after final field measurements have been recorded. Equipment is selected that channels flow in-line to a field-measurement chamber and then, without stopping, to a sample collection/processing chamber; the sample is never exposed to the atmosphere during this process (fig. 4-10).

Depending on field conditions and study objectives, samples may be withdrawn using a thief-type sampler. Lower and raise the sampler smoothly at a constant rate, keeping the suspension line clean and off the ground. A bailer or other thief-type sampler generally is not recommended for trace-element or volatile organic compound (VOC) sampling. Bailing can mobilize particulates and, unless designed for VOC sampling, can allow VOCs to escape.

- Measurements at a monitoring well
  - The standard purging procedure usually is appropriate (section 4.2.3.A). Exceptions to the standard purging procedure are described in section 4.2.3.B.
  - Either a downhole or a flowthrough-chamber system can be used for field measurements (NFM 6). If samples will be collected, use the flowthrough chamber instead of the downhole system in order to avoid bias of chemical analyses from sample contact with downhole instruments.

• Measurements at a supply well

- The standard purging procedure may not be appropriate (see section 4.2.3.B).
- Identify well-construction materials and any equipment permanently installed in the well (such as a pump) that can affect the logistics and quality of the field measurement or sample.
- Use a flowthrough-chamber type of field-measurement system (NFM 6).
- Connect the field-measurement system to the wellhead at a point before the sample would pass through holding tanks, backflow pressure tanks, flow meters, or chemical treatment systems.

If more than one well will be sampled during a field trip, each site and (or) a field vehicle must be set up for onsite cleaning of the sampling equipment. Field personnel should design the most efficient field-cleaning system, appropriate for the sites to be sampled and in accordance with the equipment-cleaning guidelines described in NFM 3.

## Step 5. Process the sample

Sample processing involves, in part, sample filtration, sample collection into appropriate containers, and sample preservation. Standard USGS procedures for sample processing are described in general and according to analyte type in NFM 5.

## Step 6. Clean the equipment

Standard USGS procedures for cleaning (or decontamination) and QC of specific types of equipment used for collecting and processing organic and inorganic analytes are detailed in NFM 3. Field personnel should design the most efficient field-cleaning system, appropriate for the sites to be sampled and in accordance with wastewater disposal regulations.

## Practice safe sampling.

► A bailer is not recommended for purging. The plunging action of the bailer can release orstir up particulates that are not ambient in ground-water flow, resulting in biased measurements and analyses.

## Steps for sampling at monitoring wells



## Step 1. Monitor-well sampling: safety and site preparations.

- a. Upon arrival, set out safety equipment such as traffic cones and signs, as needed. Park vehicle in a position to prevent sample contamination from vehicle and traffic emissions and prevailing wind.
  - Check well-identification number (this should be indelibly marked on the well casing) and compare it with the well file and field notes (section 4.2.1).
  - Assign CH/DH tasks.
  - If a gasoline-powered generator is used, locate it downwind of sample collection or elsewhere to avoid sample contamination from fumes.
  - Prepare an area to be used for field cleaning of equipment (DH)
- b. Describe well and site conditions on field forms, as appropriate (DH).
- c. Check site for hazardous conditions (NFM 9) (DH).
  - Test for toxic fumes if the well is in an enclosed structure or if there is reason to suspect the presence of organic vapors.
  - Examine the area for evidence of animal infestation and other potential safety hazards.
- d. Spread a clean plastic sheeting (polypropylene tarp, for example) on the ground around the well tokeep sampling equipment, the well tape, and sample tubing clean (DH). Take care not to trample on the sheeting. E-57

- e. Set up equipment and instruments for field measurements and ground-water withdrawal (*DH*). Locate a power supply source, if needed.
  - Set up the pump and generator (if needed) in a location to avoid sample contamination from generator fumes.
  - Calibrate field-measurement instruments (*DH*). (Refer to NFM 6 for calibration information and instructions.)
  - Wearing disposable gloves, set up the sample-processing and -preservation chambers (usually in the water-quality field vehicle). Keep sample tubing as short as is practical and shaded from direct sunlight (to minimize changes in the temperature of the sample). *Change gloves*. Place the filter unit and other supplies that will be needed for the first sample into their respective chambers (*CH*).
- f. Remove the well cap. Verify clear access downhole by lowering a section of blank pipe through the depth interval to be sampled and raising it slowly. Take care not to drop the pipe or otherwise stir up particulates in the process of lowering and raising the pipe (*DH*).
  - i. Connect the antibacksiphon valve in-line between pump and manifold (the antibacksiphon valve is a standard component of some submersible pumps).
  - ii. Use connectors and sample tubing that will not contaminate the sample with respect to target analytes.
    - Use only precleaned sample-contacting connectors and tubing.
    - At contaminated sites, sample-contacting equipment either should be dedicated for that site or should be disposable.
  - iii. From the manifold, connect the appropriate tubing to the flowthrough chamber, the sample-processing chamber, and the waste outlet.
    - Select transparent, nonporous sample tubing and tubing to the flowthrough chamber for field measurements to be able to check for bubbles or sediment entrained in the sample flow.
    - Tubing that transfers sample to the processing chambermust be clean and of noncontaminating material. Keep the discharge end of the sample tubing sealed until use.
    - Flowthrough-chamber tubing can be of any material if used only in connection with field measurements.
    - Tubing used solely to discharge purged water to waste can be of any material (garden hose, for example), but must be long enough to transport waster away from the work area.

## Step 2. Measure water level (DH).

Procedures and equipment for water-level measurement depend on well type and construction and the presence of nonaqueous liquid phases. Important considerations and method limitations are described in Appendix A4-B-3, 4, and 5. Each well must have a designated measuring point that is indicated permanently on the well (Appendix A4-B-1).

- a. Put on gloves before chalking a steel tape. Using a weighted steel or electric tape in a nonpumping well, record two or more consecutive water-level measurements to the nearest 0.01 ft (for wells of < 200 ft to water), starting at the permanent measuring (reference) point. Repeat the measurement until precision is within 0.02 ft (U.S. Geological Survey, 1980).
  - Do not allow the well tape to contact the ground before inserting it into the well. After measuring the water level, clean the tape (NFM 3.3.8) to avoid cross contaminating the next well.
  - Do not use lead weights, but weight the tape with stainless steel or another relatively noncontaminating material.
  - At wells deeper than 200 ft, calculate the compensation factor to account for stretching of the tape.
- b. Record water-level measurement on field forms and in GWSI (USGS Office of Water Quality Technical Memorandum No. 2006.01). Note any deviations from standard water-level measuring procedures on field forms (fig. 4-12). It is useful also to record water-level data into QWDATA.
- c. Set up a system to measure water levels throughout purging. Electrical tapes or submersible pressure transducers are recommended—repeated measurements with a steel tape can be cumbersome and can generate turbidity in the water column. If a packer system is used, installpressure transducers above and below the packer.
- d. Clean the tape after each use to avoid cross contamination of wells (NFM 3.3.8).

**RULE OF THUMB:** The initial water-column height should be greater than 4 ft plus the length of the sampling device.

## Step 3. Purge the well and monitor field measurements (DH).

Purge monitor wells, preferably using a variable-speed pump (see the TECHNICAL NOTES listed at the end of step 6). Operate the pump in a manner that avoids or minimizes turbidity. **Do not use a bailer for purging** unless the well characteristics or other constraints exclude alternatives and the turbidity during and afterbailing is at the background level. **Recommendation:** Measure water levels throughout purging to document drawdown and the location of the water level with respect to the screened/open interval and the pump intake.

- Use the same pumping equipment for purging that will be used to collect samples, if possible.
- Avoid refueling or changing equipment, and do not stop the pump during the final phase of purging and sample collection. Be aware of study objectives and potential sources of contamination. For example, avoid fueling the generator on the same day that samples are collected for VOC analysis. Do not transport a generator or gas tanks in the water-quality field vehicle.
- Adjust the flow rate at the pump if using a variable-speed pump. If a constant-speed pump is used, adjust the flow rate using a needle valve.
  - Pump at a rate that does not substantially lower the water level. Ideally, well yield should be sufficient so that the water level is maintained above the screened or open interval.
  - Flow should not be halted or the flow rate changed suddenly during the final phases of purging and sampling.
- a. Calculate the well volume. For a cased well, the depth to the bottom of the well and the inside casing diameter must be known:

## V=0.0408 x HD2

where,

V is volume, in gallons H is height of water column  $D^2$  is the inside well diameter squared, in inches

Note that for a cased well, the volume of water stored within the annular space between the well screen and borehole well also should be evacuated at least once.

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- b. Lower a submersible pump, followed by a water-level sensor, to the desired location of the pump intake. (The pump position is fixed if the monitoring well has a permanently installed sampling system.) Move the equipment slowly and smoothly through the water column to avoid stirring up particulates. The intake can be either lowered continually while purging to the final depth desired or placed immediately at its final position. Note that the final pump intake position always is at the point of sample collection.
  - Position the pump intake about 3 ft (about 0.9 m) below static water surface and a minimum distance above the top of the screened/open interval of 7 to 10 times the well diameter (for example, 14 to 20 in. for a 2-in. well diameter), if the sample is to represent the entire screened or open interval of aquifer. The location of the intake might be different if the study objective requires collecting the sample from a point within the screened/open interval or from wells in which packers are installed.
  - Place water-level sensor (electric tapes) a maximum of 1 ft (about 0.3 m) below the water surface.
- c. Position the pump intake.
  - If final intake position is above the screened or open interval, do not exceed 1 ft (about 0.3 m) of drawdown.
  - If final intake position is within the screened or open interval, do not exceed 0.5 ft (about 0.15 m) of drawdown. The final pumping rate should be as slow as necessary to avoid causing turbidity.
- d. Start the pump, channeling initial discharge to waste. Discharge the initial well water through the waste line until sediment is cleared from the flow.
  - Gradually increase and (or) adjust the pumping rate to limit drawdown to between 0.5 and 1 ft (about 0.15 to 0.3 m), if possible.
  - If using a variable-speed pump, adjust the rate of flow at the pump. If using a constant-speed pump, control the flow rate using a needle valve (fig. 4-10).

- Do not use a three-way valve or flow-splitting valve to adjust flow rate. It is necessary to keep the two- or three-way valves either completely open or completely closed (partially open three-way valves can create a vacuum or air bubbles, and can draw in contaminating water).
- Contain and dispose of purge waters according to Federal, State, or local regulations. Do not discharge purge water from one well into another without proper authorization. Discharge purge water far enough away from the well or well cluster so as notto enter or affect water quality in the well, and to prevent muddy and slippery work conditions.
- e. When the water runs clear, divert flow through the manifold to the flowthrough chamber (unless a downhole instrument is being used for field measurements.
  - The flow should be a smooth, solid stream of water with no air or gas bubbles and without pump cavitation during field measurements and sample withdrawal. Adjust the pumping rate to eliminate air or gas bubbles or cavitation, but do not halt or suddenly change the flow rate.
  - Record the start time of purging, the pumping rate(s), water level(s), and final location of the pump intake (fig. 4-12). If water is flowing through more than one conduit (such as valve and manifold lines), calculate the flow rate by summing the flow rate through each conduit.
  - Begin monitoring field measurements (refer to NFM 6 for instructions) once flow to field-measurement instruments is constant (see instructions above).
  - Do not move the pump or change the rate of pumping during field measurements or sample collection after setting the intake at its final depth location.

- f. Purge a minimum of three well volumes or the purge volume dictated by study objectives. (Check exceptions to the three-well-volume procedure described in section 4.2.3.B).
  - Record water levels and field measurements at regular time intervals (fig. 4-12; NFM 6). Routine field measurements for USGS studies include water temperature, conductivity, pH, dissolved-oxygen concentration, and turbidity. Check for special instructions regarding field-measurement or field-analysis requirements based on study objectives.
  - As the final well volume (commonly the third well volume) is purged, check the field-measurement data against the measurement-stability criteria (fig. 4-12). Record at least five sets of field measurements determined at regularly spaced intervals, which indicate that measurement values are relatively constant (have "stabilized") or that stabilization cannot be achieved in the given time interval (NFM 6).

## Step 4. Withdraw the sample (*CH*). Pumped samples—

Maintain the same rate of pumping throughout sample collection as the rate used during withdrawal of the final purge volume.

- a. Put ondisposable gloves. Check that the sample tubing is properly secured within the sample-processing chamber.
- b. Direct sample flow through the sample tubing to the processing chamber and channel two tubing volumes of the water to waste. Use the needle valve at the maniford (fig. 4-10) to adjust sample flow as appropriate for the target analysis.
  - Depending on the site-specific logistics, a second needle valve can be installed after the outlet end of the maniford and close to the sample-processing chamber.
  - The flow should be smooth and non-turbulent. Avoid splashing or pooling water inside the chamber while processing sample and filling sample bottles.
  - If samples will be collected for organic carbon analysis through equipment and tubing that previously was methanol-rinsed, flush at least five tubing volumes of sample water through the tubing (or collect the organiccarbon sample using a separate, non-methanol-rinsed sampler) before proceeding to step 5.

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## Remember, flow should be constant and uninterrupted while purging and sampling.

**RULE OF THUMB:** When using a pump, the rate of flow for filling sample bottles should not exceed

- 500 mL/min for bottles 250 mL or greater in volume,

or

- 150 mL/min for 40-mL VOC vials.

## Nonpumped samples—

- a. Field rinse the sampler (typically, a bailer) and sampler emptying device (and compositing device, if used) three times before collecting the sample. Deploy the sampler so as to minimize disturbance to the water column and aquifer materials.
  - i. Use a reel to keep sampler line clean and untangled.
  - ii. Lower sampler smoothly, entering water with as little disturbance as possible.
  - iii. Allow sampler to fill, then withdraw sampler smoothly.
  - iv. Shake water in sampler vigorously to rinse all interior surfaces.
  - v. Attach sample-delivery tube or bottom-emptying device to sampler and drain the rinse water through the sampler.
  - vi. Repeat rinse procedure at least twice.
- b. Repeat steps (a) i-iii to withdraw ground water for the sample.

**TECHNICAL NOTE:** When a device is lowered and raised through the water column, the disturbance to the water column can result in outgassing or degassing of ambient dissolved gases and an increase in concentrations of suspended particulates. Repeated movement of the device through the water column exacerbates these effects and can result in substantial modification of the ambient water composition and chemistry.

c. Set up the bailer in an enclosed or protected space.

**Step 5. Process/collect the sample**  $\rightarrow$  Refer to NFM 5, *Processing of Water Samples*, for instructions regarding the field rinse of sample bottles, sample filtration, and the collection and preservation of whole-water and filtered samples.

**Step 6. Clean equipment**  $\rightarrow$  Refer to NFM 3, *Cleaning of Equipment for Water Sampling*. Sampling equipment must be cleaned as instructed in NFM 3 before leaving the field site.

At contaminated sites, use sample tubing that is disposable or dedicated to that site in order to minimize the risk of cross contamination between wells. Wear gloves while cleaning and handling sampling equipment.

- Rinse sampling equipment with deionized water before the equipment dries.
- Clean equipment to be used at another well during the same field trip after rinsing it and before moving to the next site.
- Collect field blanks to assess equipment-cleaning procedures directly after the sampling equipment has been cleaned in the field or after moving to the next site and before sampling, as dictated by the data-quality requirements of the study (section 4.3).

## APPENDIX A4-B Instructions Related to Measuring Water Levels at Wells and a Sample USGS Ground-Water-Quality Field Form

All USGS personnel who sample or make water-level or water-quality measurements at wells must comply with requirements and be familiar with the guidelines provided by the USGS Office of Ground Water. Guidelines established by the Office of Ground Water related to measurement of well depth and water level have been adapted for water-quality work and are summarized in this appendix.

A4-B-1. Establishing a permanent measuring point on wells at which water level will be measured	APP.B3
A4-B-2. Well-depth measurement Figure B1. Example of a USGS field form for ground-water- level measurements	APP.B5 APP.B7
A4-B-3. Water-level measurement by (a) Steel-tape procedure (b) Electric-tape procedure	APP.B8 APP.B9 APP.B13
Figure B2. Example of a water-level measurement using a graduated steel tape	APP.B12
A4-B-4. Water-level measurement by the air-line method Figure B3. Typical installation for measuring water level by the air-line method	APP.B17 APP.B20
A4-B-5. Water-level measurement at flowing wells using low-pressure and high-pressure methods	APP.B21
A4-B-6. Sample of the U.S. Geological Survey Ground- Water Quality Notes field form	APP.B25

## Appendix A4-B

Equipment and Supplies All sections of appendix A4-B – common supplies				
A4-B-1 – Es	tablishing a permanent measuring point on wells			
A4-B-Z - W	GWSL site schedule. Form 0, 1004, A			
All sections	GwSi site schedule, Foilii 9-1904-A			
	and/or handheld or field computer for data entry			
	Pens, ballpoint with non-enasable blue or black ink, for writing on field forms and in equipment log books			
	Field folder and well file			
	Two wrenches with adjustable jaws and other tools for removing the well cap			
	Clean rag			
	Key(s) for opening locks			
	Equipment-cleaning supplies (NFM 3). Tape-cleaning supplies: refer to NFM 3.3.8 for soap-and-water wash guidance and disinfection. If disinfecting, use either (a) commercially available hypochlorite wipes; or (b) prepare a dilute chlorine solution adding 1 mL of common household bleach to 900 mL of water (0.005-percent solution)			
A4-B-1	Establishing a permanent measuring point (MP)			
	Steel tape, graduated in feet, tenths, and hundredths of feet; calibrated for making field measurements			
	Reference steel tape, graduated in feet, tenths, and hundredths of feet; designated for calibration of field steel and electric tapes			
	Calibration and maintenance log book for each steel tape			
	Spray paint (bright color) or casing-notching tool			
A4-B-2	Well-depth measurement with steel tape			
	Steel tape, graduated in feet, tenths, and hundredths of feet; calibrated for making measurements. A black tape is better than a chromium- plated tape. If a chromium-plated tape has to be used, paint the back of the tape with a flat black paint to make it easier to read the wetted chalk mark			
	Reference steel tape, graduated in feet, tenths, and hundredths of feet; designated for calibration of field steel and electric tapes			
	Steel-tape calibration and maintenance log book (one for each steel tape)			
	Weight (stainless steel, iron, or other noncontaminating material) – not lead			
	Strong ring and wire, for attaching weight to end of tape. Wire should be strong enough to hold weight securely, but not as strong as the tape, so that if the weight becomes lodged in the well the tape can still be pulled free			
	Carpenters' chalk (blue)			

# Appendix A4-B-1 Establishing a permanent measuring point on wells at which water level will be measured $^{\rm 1}$

A permanent measuring point (MP) from which all water levels for a given well are measured must be established for each well at which USGS data are collected. The MP should be established when a monitor well is installed or an existing well is inventoried. The accuracy with which the MP is established depends on the accuracy of the water-level measurement being made. For water level measured in hundredths of a foot, the MP is to be established to an accuracy of 0.01 foot. This guidance assumes that:

- ► All water-level measurements from a given well must be referenced to the same datum to ensure data comparability.
- ► Land-surface datum (LSD) at the well was established by the person who made the initial water-level measurement at the well. LSD is an arbitrary plane chosen to be approximately equivalent to the average altitude of the ground around thewell. Because LSD around a well may change over time, the distance between the MP and LSD should be checked every 3 to 5 years, or more frequently because of land development or other changes.
- Measuring points can change from time to time, especially on privately-owned wells. Such changes must be documented and dated in field notes and in the data base(s) into which the waterlevel data are entered.

## To establish a permanent measuring point:

1. Establish the location of the MP at a specific point within the top of the casing. The MP is measured in reference to LSD. If possible, position the MP at a point on the casing where a leveling rod could be set on it directly over the well and the measuring tape can hang freely when it is in contact with the MP. Locate the MP at the most convenient place from which to measure the water level.

<sup>&</sup>lt;sup>1</sup>From the USGS Office of Ground Water, Ground-Water Procedure Document 3.

- 2. Clearly mark the MP, either with an arrow sprayed with bright-colored paint or with a notch cut into the top of the casing. The MP must be as permanent as possible and be clearly visible and easily located. Location of the MP must be described in the well file.
- 3. Measure the height of the MP in feet above or below LSD. For USGS studies, record the following information into GWSI (figure B1):
  - Height and detailed description of the MP. Note that values for measuring point below land surface should be preceded by a minus sign (-).
  - Date the MP was established.
- 4. For most water-quality studies, the LSD and MP should be surveyed in.
- 5. Establish at least one clearly displayed reference mark (RM) in a location near the well; for example, a lag bolt set into a nearby telephone pole. The RM is an arbitrary datum established by permanent marks and is used to check the MP or to re-establish an MP should the original MP be destroyed or need to be changed.
- 6. Clearly locate the MP and RM on a detailed site sketch that goes into the well folder; the sketch commonly is made on the back of the paper GWSI form. If possible, photograph the site, including the RM and MP locations; draw an arrow to the RM and MP on the photograph(s) using an indelible marker, and place the photos in the well file.

## Appendix A4-B-2 Well-depth measurement<sup>2</sup>

This method uses a graduated steel tape to measure the total depth of a well below land-surface datum. Select a graduated steel tape that is accurate to 0.01 foot. The steel tape should be calibrated against a reference steel tape. A reference steel tape is one that is maintained in the office and designated solely for tape calibration.

- If the well casing is angled, instead of vertical, the well depth will have to be corrected.
- When measuring wells of depth greater than 200 feet (deep wells), expansion and stretch of the steel tape must be considered and accounted for (see Garber and Koopman, 1968).
- Use of a steel tape is **not** recommended for measuring the depth of pumping wells.
- A weight usually is attached to the end of a steel tape to allow it to hang plumb. The weight should not be constructed of lead or other material that potentially could contaminate water in the well.
- Well obstructions could cause errors in the measurement if the steel tape cannot hang plumb.

## To measure well depth:

- 1. Using a clean, calibrated steel tape, measure from the zero point on the tape to the bottom of the weight. Record this number as the length of the weight interval.
- 2. Lower the weight and tape into the well until the weight reaches the bottom of the well and the tape slackens.
- Partially withdraw the tape from the well until the weight is standing in a vertical position, but still touching the bottom of the well. A slight jerking motion will be felt as the weight moves from the horizontal to the vertical position.
- 4. Repeat step 3 several times by lowering and withdrawing the tape to obtain a consistent reading.
- 5. Record the tape reading held at the measuring point (MP).

<sup>&</sup>lt;sup>2</sup>From the USGS Office of Ground Water, Ground-Water Procedure Document 11.

#### APP.B6—COLLECTION OF WATER SAMPLES

- 6. Withdraw the tape from the well 1 to 2 feet, so that the weight will hang freely above the bottom of the well. Repeat steps 2-4 until two consistent depth readings are obtained.
- 7. Calculate total well depth below land-surface datum (LSD) as follows:

a. Tape reading held at the MP	84.3 feet
b. Length of the weight interval	<u>+ 1.2 feet</u>
c. Sum of a + b	85.5 feet
d. MP correction	<u>- 3.5 feet</u>
e. Total well depth below LSD	82.0 feet

- 8. After completing the well-depth measurement, clean the exposed portion of the tape using the procedures described in NFM 3.3.8. To prevent microbial cross-contamination of other wells, disinfect the tape using commercially available hypochlorite wipes or a dilute (0.005-percent) chlorine solution.
- 9. Record depth data to the nearest 0.01 foot. USGS well-depth data should be recorded in GWSI and on the Ground-Water Level Notes (fig. B1) and other field forms that are kept in the field folder.

Low Stress (low flow) Purging and Sampling Procedures

SOP for Collection of Low Flow Groundwater Samples

EQASOP-GW 001 Region 1 Low-Stress (Low-Flow) SOP Revision Number: 3 Date: July 30, 1996 Revised: January 19, 2010 Page 1 of 30

## U.S. ENVIRONMENTAL PROTECTION AGENCY REGION I

## LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUNDWATER SAMPLES FROM MONITORING WELLS

Quality Assurance Unit U.S. Environmental Protection Agency – Region 1 11 Technology Drive North Chelmsford, MA 01863

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## **Revision Page**

Date	Rev	Summary of changes	Sections
	#	· .	
7/30/96	2	Finalized	
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## **USE OF TERMS**

<u>Equipment blank</u>: The equipment blank shall include the pump and the pump's tubing. If tubing is dedicated to the well, the equipment blank needs only to include the pump in subsequent sampling rounds. If the pump and tubing are dedicated to the well, the equipment blank is collected prior to its placement in the well. If the pump and tubing will be used to sample multiple wells, the equipment blank is normally collected after sampling from contaminated wells and not after background wells.

<u>Field duplicates</u>: Field duplicates are collected to determine precision of the sampling procedure. For this procedure, collect duplicate for each analyte group in consecutive order (VOC original, VOC duplicate, SVOC original, SVOC duplicate, etc.).

<u>Indicator field parameters</u>: This SOP uses field measurements of turbidity, dissolved oxygen, specific conductance, temperature, pH, and oxidation/reduction potential (ORP) as indicators of when purging operations are sufficient and sample collection may begin.

Matrix Spike/Matrix Spike Duplicates: Used by the laboratory in its quality assurance program. Consult the laboratory for the sample volume to be collected.

<u>Poteniometric Surface</u>: The level to which water rises in a tightly cased well constructed in a confined aquifer. In an unconfined aquifer, the potentiometric surface is the water table.

QAPP: Quality Assurance Project Plan

SAP: Sampling and Analysis Plan

SOP: Standard operating procedure

<u>Stabilization</u>: A condition that is achieved when all indicator field parameter measurements are sufficiently stable (as described in the "Monitoring Indicator Field Parameters" section) to allow sample collection to begin.

<u>Temperature blank</u>: A temperature blank is added to each sample cooler. The blank is measured upon receipt at the laboratory to assess whether the samples were properly cooled during transit.

<u>Trip blank (VOCs)</u>: Trip blank is a sample of analyte-free water taken to the sampling site and returned to the laboratory. The trip blanks (one pair) are added to each sample cooler that contains VOC samples.

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## **SCOPE & APPLICATION**

The goal of this groundwater sampling procedure is to collect water samples that reflect the total mobile organic and inorganic loads (dissolved and colloidal sized fractions) transported through the subsurface under ambient flow conditions, with minimal physical and chemical alterations from sampling operations. This standard operating procedure (SOP) for collecting groundwater samples will help ensure that the project's data quality objectives (DQOs) are met under certain low-flow conditions.

The SOP emphasizes the need to minimize hydraulic stress at the well-aquifer interface by maintaining low water-level drawdowns, and by using low pumping rates during purging and sampling operations. Indicator field parameters (e.g., dissolved oxygen, pH, etc.) are monitored during purging in order to determine when sample collection may begin. Samples properly collected using this SOP are suitable for analysis of groundwater contaminants (volatile and semi-volatile organic analytes, dissolved gases, pesticides, PCBs, metals and other inorganics), or naturally occurring analytes. This SOP is based on Puls, and Barcelona (1996).

This procedure is designed for monitoring wells with an inside diameter (1.5-inches or greater) that can accommodate a positive lift pump with a screen length or open interval ten feet or less and with a water level above the top of the screen or open interval (Hereafter, the "screen or open interval" will be referred to only as "screen interval"). This SOP is not applicable to other well-sampling conditions.

While the use of dedicated sampling equipment is not mandatory, dedicated pumps and tubing can reduce sampling costs significantly by streamlining sampling activities and thereby reducing the overall field costs.

The goal of this procedure is to emphasize the need for consistency in deploying and operating equipment while purging and sampling monitoring wells during each sampling event. This will help to minimize sampling variability.

This procedure describes a general framework for groundwater sampling. Other site specific information (hydrogeological context, conceptual site model (CSM), DQOs, etc.) coupled with systematic planning must be added to the procedure in order to develop an appropriate site specific SAP/QAPP. In addition, the site specific SAP/QAPP must identify the specific equipment that will be used to collect the groundwater samples.

This procedure does not address the collection of water or free product samples from wells containing free phase LNAPLs and/or DNAPLs (light or dense non-aqueous phase

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liquids). For this type of situation, the reader may wish to check: Cohen, and Mercer (1993) or other pertinent documents.

This SOP is to be used when collecting groundwater samples from monitoring wells at all Superfund, Federal Facility and RCRA sites in Region 1 under the conditions described herein. Request for modification of this SOP, in order to better address specific situations at individual wells, must include adequate technical justification for proposed changes. <u>All changes and modifications must be approved and included in a revised SAP/QAPP before implementation in field.</u>

## **BACKGROUND FOR IMPLEMENTATION**

It is expected that the monitoring well screen has been properly located (both laterally and vertically) to intercept existing contaminant plume(s) or along flow paths of potential contaminant migration. Problems with inappropriate monitoring well placement or faulty/improper well installation cannot be overcome by even the best water sampling procedures. This SOP presumes that the analytes of interest are moving (or will potentially move) primarily through the more permeable zones intercepted by the screen interval.

Proper well construction, development, and operation and maintenance cannot be overemphasized. The use of installation techniques that are appropriate to the hydrogeologic setting of the site often prevent "problem well" situations from occurring. During well development, or redevelopment, tests should be conducted to determine the hydraulic characteristics of the monitoring well. The data can then be used to set the purging/sampling rate, and provide a baseline for evaluating changes in well performance and the potential need for well rehabilitation. Note: if this installation data or well history (construction and sampling) is not available or discoverable, for all wells to be sampled, efforts to build a sampling history should commence with the next sampling event.

The pump intake should be located within the screen interval and at a depth that will remain under water at all times. It is recommended that the intake depth and pumping rate remain the same for all sampling events. The mid-point or the lowest historical midpoint of the saturated screen length is often used as the location of the pump intake. For new wells, or for wells without pump intake depth information, the site's SAP/QAPP must provide clear reasons and instructions on how the pump intake depth(s) will be selected, and reason(s) for the depth(s) selected. If the depths to top and bottom of the well screen are not known, the SAP/QAPP will need to describe how the sampling depth will be determined and how the data can be used.

Stabilization of indicator field parameters is used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU, and stable drawdowns of less than 0.3 feet, while desirable, are not mandatory. Sample collection

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may still take place provided the indicator field parameter criteria in this procedure are met. If after 2 hours of purging indicator field parameters have not stabilized, one of three optional courses of action may be taken: a) continue purging until stabilization is achieved, b) discontinue purging, do not collect any samples, and record in log book that stabilization could not be achieved (documentation must describe attempts to achieve stabilization), c) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analytes, may reflect a sampling bias and therefore, the data may not meet the data quality objectives of the sampling event).

It is recommended that low-flow sampling be conducted when the air temperature is above 32°F (0°C). If the procedure is used below 32°F, special precautions will need to be taken to prevent the groundwater from freezing in the equipment. Because sampling during freezing temperatures may adversely impact the data quality objectives, the need for water sample collection during months when these conditions are likely to occur should be evaluated during site planning and special sampling measures may need to be developed. Ice formation in the flow-through-cell will cause the monitoring probes to act erratically. A transparent flow-through-cell needs to be used to observe if ice is forming in the cell. If ice starts to form on the other pieces of the sampling equipment, additional problems may occur.

## **HEALTH & SAFETY**

When working on-site, comply with all applicable OSHA requirements and the site's health/safety procedures. All proper personal protection clothing and equipment are to be worn. Some samples may contain biological and chemical hazards. These samples should be handled with suitable protection to skin, eyes, etc.

## **CAUTIONS**

The following cautions need to be considered when planning to collect groundwater samples when the below conditions occur.

If the groundwater degasses during purging of the monitoring well, dissolved gases and VOCs will be lost. When this happens, the groundwater data for dissolved gases (e.g., methane, ethane, ethane, dissolved oxygen, etc.) and VOCs will need to be qualified. Some conditions that can promote degassing are the use of a vacuum pump (e.g., peristaltic pumps), changes in aperture along the sampling tubing, and squeezing/pinching the pump's tubing which results in a pressure change.

When collecting the samples for dissolved gases and VOCs analyses, avoid aerating the groundwater in the pump's tubing. This can cause loss of the dissolved gases and VOCs in

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the groundwater. Having the pump's tubing completely filled prior to sampling will avoid this problem when using a centrifugal pump or peristaltic pump.

Direct sun light and hot ambient air temperatures may cause the groundwater in the tubing and flow-through-cell to heat up. This may cause the groundwater to degas which will result in loss of VOCs and dissolved gases. When sampling under these conditions, the sampler will need to shade the equipment from the sunlight (e.g., umbrella, tent, etc.). If possible, sampling on hot days, or during the hottest time of the day, should be avoided. The tubing exiting the monitoring well should be kept as short as possible to avoid the sun light or ambient air from heating up the groundwater.

Thermal currents in the monitoring well may cause vertical mixing of water in the well bore. When the air temperature is colder than the groundwater temperature, it can cool the top of the water column. Colder water which is denser than warm water sinks to the bottom of the well and the warmer water at the bottom of the well rises, setting up a convention cell. "During low-flow sampling, the pumped water may be a mixture of convecting water from within the well casing and aquifer water moving inward through the screen. This mixing of water during low-flow sampling can substantially increase equilibration times, can cause false stabilization of indicator parameters, can give false indication of redox state, and can provide biological data that are not representative of the aquifer conditions" (Vroblesky 2007).

Failure to calibrate or perform proper maintenance on the sampling equipment and measurement instruments (e.g., dissolved oxygen meter, etc.) can result in faulty data being collected.

Interferences may result from using contaminated equipment, cleaning materials, sample containers, or uncontrolled ambient/surrounding air conditions (e.g., truck/vehicle exhaust nearby).

Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and/or proper planning to avoid ambient air interferences. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

Clean and decontaminate all sampling equipment prior to use. All sampling equipment needs to be routinely checked to be free from contaminants and equipment blanks collected to ensure that the equipment is free of contaminants. Check the previous equipment blank data for the site (if they exist) to determine if the previous cleaning procedure removed the contaminants. If contaminants were detected and they are a concern, then a more vigorous cleaning procedure will be needed.

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## PERSONNEL QUALIFICATIONS

All field samplers working at sites containing hazardous waste must meet the requirements of the OSHA regulations. OSHA regulations may require the sampler to take the 40 hour OSHA health and safety training course and a refresher course prior to engaging in any field activities, depending upon the site and field conditions.

The field samplers must be trained prior to the use of the sampling equipment, field instruments, and procedures. Training is to be conducted by an experienced sampler before initiating any sampling procedure.

The entire sampling team needs to read, and be familiar with, the site Health and Safety Plan, all relevant SOPs, and SAP/QAPP (and the most recent amendments) before going onsite for the sampling event. It is recommended that the field sampling leader attest to the understanding of these site documents and that it is recorded.

## EQUIPMENT AND SUPPLIES

## A. Informational materials for sampling event

A copy of the current Health and Safety Plan, SAP/QAPP, monitoring well construction data, location map(s), field data from last sampling event, manuals for sampling, and the monitoring instruments' operation, maintenance, and calibration manuals should be brought to the site.

### **B.** Well keys.

## **C.** Extraction device

Adjustable rate, submersible pumps (e.g., centrifugal, bladder, etc.) which are constructed of stainless steel or Teflon are preferred. Note: if extraction devices constructed of other materials are to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

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If bladder pumps are selected for the collection of VOCs and dissolved gases, the pump setting should be set so that one pulse will deliver a water volume that is sufficient to fill a 40 mL VOC vial. This is not mandatory, but is considered a "best practice". For the proper operation, the bladder pump will need a minimum amount of water above the pump; consult the manufacturer for the recommended submergence. The pump's recommended submergence value should be determined during the planning stage, since it may influence well construction and placement of dedicated pumps where water-level fluctuations are significant.

Adjustable rate, peristaltic pumps (suction) are to be used with caution when collecting samples for VOCs and dissolved gases (e.g., methane, carbon dioxide, etc.) analyses. Additional information on the use of peristaltic pumps can be found in Appendix A. If peristaltic pumps are used, the inside diameter of the rotor head tubing needs to match the inside diameter of the tubing installed in the monitoring well.

Inertial pumping devices (motor driven or manual) are not recommended. These devices frequently cause greater disturbance during purging and sampling, and are less easily controlled than submersible pumps (potentially increasing turbidity and sampling variability, etc.). This can lead to sampling results that are adversely affected by purging and sampling operations, and a higher degree of data variability.

## **D.** Tubing

Teflon or Teflon-lined polyethylene tubing are preferred when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics. Note: if tubing constructed of other materials is to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

PVC, polypropylene or polyethylene tubing may be used when collecting samples for metal and other inorganics analyses.

The use of 1/4 inch or 3/8 inch (inside diameter) tubing is recommended. This will help ensure that the tubing remains liquid filled when operating at very low pumping rates when using centrifugal and peristaltic pumps.

Silastic tubing should be used for the section around the rotor head of a peristaltic pump. It should be less than a foot in length. The inside diameter of the tubing used at the pump rotor head must be the same as the inside diameter of tubing placed in the well. A tubing connector is used to connect the pump rotor head tubing to the well tubing. Alternatively, the two pieces of tubing can be connected to each other by placing the one end of the tubing inside the end of the other tubing. The tubing must not be reused.

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## E. The water level measuring device

Electronic "tape", pressure transducer, water level sounder/level indicator, etc. should be capable of measuring to 0.01 foot accuracy. Recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include check measurements with a water level "tape" at the start and end of each sampling event.

## F. Flow measurement supplies

Graduated cylinder (size according to flow rate) and stopwatch usually will suffice.

Large graduated bucket used to record total water purged from the well.

## G. Interface probe

To be used to check on the presence of free phase liquids (LNAPL, or DNAPL) before purging begins (as needed).

## H. Power source (generator, nitrogen tank, battery, etc.)

When a gasoline generator is used, locate it downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate samples.

## I. Indicator field parameter monitoring instruments

Use of a multi-parameter instrument capable of measuring pH, oxidation/reduction potential (ORP), dissolved oxygen (DO), specific conductance, temperature, and coupled with a flow-through-cell is required when measuring all indicator field parameters, except turbidity. Turbidity is collected using a separate instrument. Record equipment/instrument identification (manufacturer, and model number).

Transparent, small volume flow-through-cells (e.g., 250 mLs or less) are preferred. This allows observation of air bubbles and sediment buildup in the cell, which can interfere with the operation of the monitoring instrument probes, to be easily detected. A small volume cell facilitates rapid turnover of water in the cell between measurements of the indicator field parameters.

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It is recommended to use a flow-through-cell and monitoring probes from the same manufacturer and model to avoid <u>incompatibility</u> between the probes and flow-through-cell.

Turbidity samples are collected before the flow-through-cell. A "T" connector coupled with a valve is connected between the pump's tubing and flow-through-cell. When a turbidity measurement is required, the valve is opened to allow the groundwater to flow into a container. The valve is closed and the container sample is then placed in the turbidimeter.

Standards are necessary to perform field calibration of instruments. A minimum of two standards are needed to bracket the instrument measurement range for all parameters except ORP which use a Zobell solution as a standard. For dissolved oxygen, a wet sponge used for the 100% saturation and a zero dissolved oxygen solution are used for the calibration.

Barometer (used in the calibration of the Dissolved Oxygen probe) and the conversion formula to convert the barometric pressure into the units of measure used by the Dissolved Oxygen meter are needed.

## J. Decontamination supplies

Includes (for example) non-phosphate detergent, distilled/deionized water, isopropyl alcohol, etc.

## K. Record keeping supplies

Logbook(s), well purging forms, chain-of-custody forms, field instrument calibration forms, etc.

## L. Sample bottles

M. Sample preservation supplies (as required by the analytical methods)

#### N. Sample tags or labels

## **O.** PID or FID instrument

If appropriate, to detect VOCs for health and safety purposes, and provide qualitative field evaluations.

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## P. Miscellaneous Equipment

Equipment to keep the sampling apparatus shaded in the summer (e.g., umbrella) and from freezing in the winter. If the pump's tubing is allowed to heat up in the warm weather, the cold groundwater may degas as it is warmed in the tubing.

## EQUIPMENT/INSTRUMENT CALIBRATION

Prior to the sampling event, perform maintenance checks on the equipment and instruments according to the manufacturer's manual and/or applicable SOP. This will ensure that the equipment/instruments are working properly before they are used in the field.

Prior to sampling, the monitoring instruments must be calibrated and the calibration documented. The instruments are calibrated using U.S Environmental Protection Agency Region 1 *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction [ORP], and turbidity)*, January 19, 2010, or latest version or from one of the methods listed in 40CFR136, 40CFR141 and SW-846.

The instruments shall be calibrated at the beginning of each day. If the field measurement falls outside the calibration range, the instrument must be re-calibrated so that all measurements fall within the calibration range. At the end of each day, a calibration check is performed to verify that instruments remained in calibration throughout the day. This check is performed while the instrument is in measurement mode, not calibration mode. If the field instruments are being used to monitor the natural attenuation parameters, then a calibration check at mid-day is highly recommended to ensure that the instruments did not drift out of calibration. Note: during the day if the instrument reads zero or a negative number for dissolved oxygen, pH, specific conductance, or turbidity (negative value only), this indicates that the instrument drifted out of calibration or the instrument is malfunctioning. If this situation occurs the data from this instrument will need to be qualified or rejected.

## PRELIMINARY SITE ACTIVITIES (as applicable)

Check the well for security (damage, evidence of tampering, missing lock, etc.) and record pertinent observations (include photograph as warranted).

If needed lay out sheet of clean polyethylene for monitoring and sampling equipment, unless equipment is elevated above the ground (e.g., on a table, etc.).
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Remove well cap and if appropriate measure VOCs at the rim of the well with a PID or FID instrument and record reading in field logbook or on the well purge form.

If the well casing does not have an established reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook (consider a photographic record as well). All water level measurements must be recorded relative to this reference point (and the altitude of this point should be determined using techniques that are appropriate to site's DQOs.

If water-table or potentiometric surface map(s) are to be constructed for the sampling event, perform synoptic water level measurement round (in the shortest possible time) before any purging and sampling activities begin. If possible, measure water level depth (to 0.01 ft.) and total well depth (to 0.1 ft.) the day before sampling begins, in order to allow for re-settlement of any particulates in the water column. This is especially important for those wells that have not been recently sampled because sediment buildup in the well may require the well to be redeveloped. If measurement of total well depth is not made the day before, it should be measured after sampling of the well is complete. All measurements must be taken from the established referenced point. Care should be taken to minimize water column disturbance.

Check newly constructed wells for the presence of LNAPLs or DNAPLs before the initial sampling round. If none are encountered, subsequent check measurements with an interface probe may not be necessary unless analytical data or field analysis signal a worsening situation. This SOP cannot be used in the presence of LNAPLs or DNAPLs. If NAPLs are present, the project team must decide upon an alternate sampling method. All project modifications must be approved and documented prior to implementation.

If available check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practicable, the intake depth and extraction rate (use final pump dial setting information) from previous event(s). If changes are made in the intake depth or extraction rate(s) used during previous sampling event(s), for either portable or dedicated extraction devices, record new values, and explain reasons for the changes in the field logbook.

#### PURGING AND SAMPLING PROCEDURE

Purging and sampling wells in order of increasing chemical concentrations (known or anticipated) are preferred.

The use of dedicated pumps is recommended to minimize artificial mobilization and entrainment of particulates each time the well is sampled. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each

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sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

#### A. Initial Water Level

Measure the water level in the well before installing the pump if a non-dedicated pump is being used. The initial water level is recorded on the purge form or in the field logbook.

#### **B.** Install Pump

Lower pump, safety cable, tubing and electrical lines slowly (to minimize disturbance) into the well to the appropriate depth (may not be the mid-point of the screen/open interval). The Sampling and Analysis Plan/Quality Assurance Project Plan should specify the sampling depth (used previously), or provide criteria for selection of intake depth for each new well. If possible keep the pump intake at least two feet above the bottom of the well, to minimize mobilization of particulates present in the bottom of the well.

Pump tubing lengths, above the top of well casing should be kept as short as possible to minimize heating the groundwater in the tubing by exposure to sun light and ambient air temperatures. Heating may cause the groundwater to degas, which is unacceptable for the collection of samples for VOC and dissolved gases analyses.

#### C. Measure Water Level

Before starting pump, measure water level. Install recording pressure transducer, if used to track drawdowns, to initialize starting condition.

#### **D.** Purge Well

From the time the pump starts purging and until the time the samples are collected, the purged water is discharged into a graduated bucket to determine the total volume of groundwater purged. This information is recorded on the purge form or in the field logbook.

Start the pump at low speed and slowly increase the speed until discharge occurs. Check water level. Check equipment for water leaks and if present fix or replace the affected equipment. Try to match pumping rate used during previous sampling event(s). Otherwise, adjust pump speed until there is little or no water level drawdown. If the minimal drawdown that can be achieved exceeds 0.3 feet, but remains stable, continue purging.

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Monitor and record the water level and pumping rate every five minutes (or as appropriate) during purging. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump to ensure stabilization of the water level. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover" somewhat as pump flow adjustments are made. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. If the initial water level is above the top of the screen do not allow the water level to fall into the well screen. The final purge volume must be greater than the stabilized drawdown volume plus the pump's tubing volume. If the drawdown has exceeded 0.3 feet and stabilizes, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are collected.

Avoid the use of constriction devices on the tubing to decrease the flow rate because the constrictor will cause a pressure difference in the water column. This will cause the groundwater to degas and result in a loss of VOCs and dissolved gasses in the groundwater samples.

Note: the flow rate used to achieve a stable pumping level should remain constant while monitoring the indicator parameters for stabilization and while collecting the samples.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (e.g., bladder, peristaltic), and/or the use of dedicated equipment. For new monitoring wells, or wells where the following situation has not occurred before, if the recovery rate to the well is less than 50 mL/min., or the well is being essentially dewatered during purging, the well should be sampled as soon as the water level has recovered sufficiently to collect the volume needed for all anticipated samples. The project manager or field team leader will need to make the decision when samples should be collected, how the sample is to be collected, and the reasons recorded on the purge form or in the field logbook. A water level measurement needs to be performed and recorded before samples are collected. If the project manager decides to collect the samples using the pump, it is best during this recovery period that the pump intake tubing not be removed, since this will aggravate any turbidity problems. Samples in this specific situation may be collected without stabilization of indicator field parameters. Note that field conditions and efforts to overcome problematic situations must be recorded in order to support field decisions to deviate from normal procedures described in this SOP. If this type of problematic situation persists in a well, then water sample collection should be changed to a passive or no-purge method, if consistent with the site's DQOs, or have a new well installed.

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#### **E. Monitor Indicator Field Parameters**

After the water level has stabilized, connect the "T" connector with a valve and the flowthrough-cell to monitor the indicator field parameters. If excessive turbidity is anticipated or encountered with the pump startup, the well may be purged for a while without connecting up the flow-through-cell, in order to minimize particulate buildup in the cell (This is a judgment call made by the sampler). Water level drawdown measurements should be made as usual. If possible, the pump may be installed the day before purging to allow particulates that were disturbed during pump insertion to settle.

During well purging, monitor indicator field parameters (turbidity, temperature, specific conductance, pH, ORP, DO) at a frequency of five minute intervals or greater. The pump's flow rate must be able to "turn over" at least one flow-through-cell volume between measurements (for a 250 mL flow-through-cell with a flow rate of 50 mLs/min., the monitoring frequency would be every five minutes; for a 500 mL flow-through-cell it would be every ten minutes). If the cell volume cannot be replaced in the five minute interval, then the time between measurements must be increased accordingly. Note: during the early phase of purging emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments followed by stabilization of indicator parameters. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings are within the following limits:

**Turbidity** (10% for values greater than 5 NTU; if three Turbidity values are less than 5 NTU, consider the values as stabilized),

**Dissolved Oxygen** (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),

Specific Conductance (3%), Temperature (3%), pH (± 0.1 unit), Oxidation/Reduction Potential (±10 millivolts).

All measurements, except turbidity, must be obtained using a flow-through-cell. Samples for turbidity measurements are obtained before water enters the flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values measured within the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and continue monitoring activities. Record start and stop times and give a brief description of cleaning activities.

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The flow-through-cell must be designed in a way that prevents gas bubble entrapment in the cell. Placing the flow-through-cell at a 45 degree angle with the port facing upward can help remove bubbles from the flow-through-cell (see Appendix B Low-Flow Setup Diagram). All during the measurement process, the flow-through-cell must remain free of any gas bubbles. Otherwise, the monitoring probes may act erratically. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must remain submerged in water at all times.

#### **F.** Collect Water Samples

When samples are collected for laboratory analyses, the pump's tubing is disconnected from the "T" connector with a valve and the flow-through-cell. The samples are collected directly from the pump's tubing. Samples must not be collected from the flow-through-cell or from the "T" connector with a valve.

VOC samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

If the pump's flow rate is too high to collect the VOC/dissolved gases samples, collect the other samples first. Lower the pump's flow rate to a reasonable rate and collect the VOC/dissolved gases samples and record the new flow rate.

During purging and sampling, the centrifugal/peristaltic pump tubing must remain filled with water to avoid aeration of the groundwater. It is recommended that 1/4 inch or 3/8 inch (inside diameter) tubing be used to help insure that the sample tubing remains water filled. If the pump tubing is not completely filled to the sampling point, use the following procedure to collect samples: collect non-VOC/dissolved gases samples first, then increase flow rate slightly until the water completely fills the tubing, collect the VOC/dissolved gases samples, and record new drawdown depth and flow rate.

For bladder pumps that will be used to collect VOC or dissolved gas samples, it is recommended that the pump be set to deliver long pulses of water so that one pulse will fill a 40 mL VOC vial.

Use pre-preserved sample containers or add preservative, as required by analytical methods, to the samples immediately after they are collected. Check the analytical methods (e.g. EPA SW-846, 40 CFR 136, water supply, etc.) for additional information on preservation.

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If determination of filtered metal concentrations is a sampling objective, collect filtered water samples using the same low flow procedures. The use of an in-line filter (transparent housing preferred) is required, and the filter size ( $0.45 \ \mu m$  is commonly used) should be based on the sampling objective. Pre-rinse the filter with groundwater prior to sample collection. Make sure the filter is free of air bubbles before samples are collected. Preserve the filtered water sample immediately. Note: filtered water samples are not an acceptable substitute for unfiltered samples when the monitoring objective is to obtain chemical concentrations of total mobile contaminants in groundwater for human health or ecological risk calculations.

Label each sample as collected. Samples requiring cooling will be placed into a cooler with ice or refrigerant for delivery to the laboratory. Metal samples after acidification to a pH less than 2 do not need to be cooled.

#### G. Post Sampling Activities

If a recording pressure transducer is used to track drawdown, re-measure water level with tape.

After collection of samples, the pump tubing may be dedicated to the well for re-sampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth (to 0.1 ft.), if not measured the day before purging began. Note: measurement of total well depth annually is usually sufficient after the initial low stress sampling event. However, a greater frequency may be needed if the well has a "silting" problem or if confirmation of well identity is needed.

Secure the well.

#### DECONTAMINATION

Decontaminate sampling equipment prior to use in the first well and then following sampling of each well. Pumps should not be removed between purging and sampling operations. The pump, tubing, support cable and electrical wires which were in contact with the well should be decontaminated by one of the procedures listed below.

The use of dedicated pumps and tubing will reduce the amount of time spent on decontamination of the equipment. If dedicated pumps and tubing are used, only the initial sampling event will require decontamination of the pump and tubing.

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Note if the previous equipment blank data showed that contaminant(s) were present after using the below procedure or the one described in the SAP/QAPP, a more vigorous procedure may be needed.

#### Procedure 1

Decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump and tubing. The pump may be disassembled and flushed with the decontaminating solutions. It is recommended that detergent and alcohol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is removed. The pump exterior and electrical wires must be rinsed with the decontaminating solutions, as well. The procedure is as follows:

Flush the equipment/pump with potable water.

Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.

Flush with potable or distilled/deionized water to remove all of the detergent solution. If the water is recycled, the water must be changed periodically.

Optional - flush with isopropyl alcohol (pesticide grade; must be free of ketones {e.g., acetone}) or with methanol. This step may be required if the well is highly contaminated or if the equipment blank data from the previous sampling event show that the level of contaminants is significant.

Flush with distilled/deionized water. This step must remove all traces of alcohol (if used) from the equipment. The final water rinse must not be recycled.

#### Procedure 2

Steam clean the outside of the submersible pump.

Pump hot potable water from the steam cleaner through the inside of the pump. This can be accomplished by placing the pump inside a three or four inch diameter PVC pipe with end cap. Hot water from the steam cleaner jet will be directed inside the PVC pipe and the pump exterior will be cleaned. The hot water from the steam cleaner will then be pumped from the PVC pipe through the pump and collected into another container. Note: additives or solutions should not be added to the steam cleaner.

Pump non-phosphate detergent solution through the inside of the pump. If the solution is recycled, the solution must be changed periodically.

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Pump potable water through the inside of the pump to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.

Pump distilled/deionized water through the pump. The final water rinse must not be recycled.

#### FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the groundwater samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation. Quality control samples include field duplicates, equipment blanks, matrix spike/matrix spike duplicates, trip blanks (VOCs), and temperature blanks.

#### **FIELD LOGBOOK**

A field log shall be kept to document all groundwater field monitoring activities (see Appendix C, example table), and record the following for each well:

Site name, municipality, state.

Well identifier, latitude-longitude or state grid coordinates.

Measuring point description (e.g., north side of PVC pipe).

Well depth, and measurement technique.

Well screen length.

Pump depth.

Static water level depth, date, time and measurement technique.

Presence and thickness of immiscible liquid (NAPL) layers and detection method.

Pumping rate, drawdown, indicator parameters values, calculated or measured total volume pumped, and clock time of each set of measurements.

Type of tubing used and its length.

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Type of pump used.

Clock time of start and end of purging and sampling activity.

Types of sample bottles used and sample identification numbers.

Preservatives used.

Parameters requested for analyses.

Field observations during sampling event.

Name of sample collector(s).

Weather conditions, including approximate ambient air temperature.

QA/QC data for field instruments.

Any problems encountered should be highlighted.

Description of all sampling/monitoring equipment used, including trade names, model number, instrument identification number, diameters, material composition, etc.

#### **DATA REPORT**

Data reports are to include laboratory analytical results, QA/QC information, field indicator parameters measured during purging, field instrument calibration information, and whatever other field logbook information is needed to allow for a full evaluation of data usability.

Note: the use of trade, product, or firm names in this sampling procedure is for descriptive purposes only and does not constitute endorsement by the U.S. EPA.

#### REFERENCES

Cohen, R.M. and J.W. Mercer, 1993, *DNAPL Site Evaluation*; C.K. Smoley (CRC Press), Boca Raton, Florida.

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U.S. Environmental Protection Agency, 1987, A Compendium of Superfund Field Operations Methods; Washington, DC (EPA/540/P-87/001).

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Vroblesky, Don A., Clifton C. Casey, and Mark A. Lowery, Summer 2007, Influence of Dissolved Oxygen Convection on Well Sampling, *Ground Water Monitoring & Remediation* 27, no. 3: 49-58.

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#### APPENDIX A PERISTALTIC PUMPS

Before selecting a peristaltic pump to collect groundwater samples for VOCs and/or dissolved gases (e.g., methane, carbon dioxide, etc.) consideration should be given to the following:

- The decision of whether or not to use a peristaltic pump is dependent on the intended use of the data.
- If the additional sampling error that may be introduced by this device is NOT of concern for the VOC/dissolved gases data's intended use, then this device may be acceptable.
- If minor differences in the groundwater concentrations could effect the decision, such as to continue or terminate groundwater cleanup or whether the cleanup goals have been reached, then this device should NOT be used for VOC/dissolved gases sampling. In these cases, centrifugal or bladder pumps are a better choice for more accurate results.

EPA and USGS have documented their concerns with the use of the peristaltic pumps to collect water sample in the below documents.

- "Suction Pumps are not recommended because they may cause degassing, pH modification, and loss of volatile compounds" *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001, December 1987.
- "The agency does not recommend the use of peristaltic pumps to sample ground water particularly for volatile organic analytes" *RCRA Ground-Water Monitoring Draft Technical Guidance*, EPA Office of Solid Waste, November 1992.
- "The peristaltic pump is limited to shallow applications and can cause degassing resulting in alteration of pH, alkalinity, and volatiles loss", *Low-flow (Minimal drawdown) Ground-Water Sampling Procedures*, by Robert Puls & Michael Barcelona, April 1996, EPA/540/S-95/504.
- "Suction-lift pumps, such as peristaltic pumps, can operate at a very low pumping rate; however, using negative pressure to lift the sample can result in the loss of volatile analytes", USGS Book 9 Techniques of Water-Resources Investigation, Chapter A4. (Version 2.0, 9/2006).

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#### APPENDIX B

#### SUMMARY OF SAMPLING INSTRUCTIONS

These instructions are for using an adjustable rate, submersible pump or a peristaltic pump with the pump's intake placed at the midpoint of a 10 foot or less well screen or an open interval. The water level in the monitoring well is above the top of the well screen or open interval, the ambient temperature is above 32°F, and the equipment is not dedicated. Field instruments are already calibrated. The equipment is setup according to the diagram at the end of these instructions.

1. Review well installation information. Record well depth, length of screen or open interval, and depth to top of the well screen. Determine the pump's intake depth (e.g., mid-point of screen/open interval).

2. On the day of sampling, check security of the well casing, perform any safety checks needed for the site, lay out a sheet of polyethylene around the well (if necessary), and setup the equipment. If necessary a canopy or an equivalent item can be setup to shade the pump's tubing and flow-through-cell from the sun light to prevent the sun light from heating the groundwater.

3. Check well casing for a reference mark. If missing, make a reference mark. Measure the water level (initial) to 0.01 ft. and record this information.

4. Install the pump's intake to the appropriate depth (e.g., midpoint) of the well screen or open interval. Do not turn-on the pump at this time.

5. Measure water level and record this information.

6. Turn-on the pump and discharge the groundwater into a graduated waste bucket. Slowly increase the flow rate until the water level starts to drop. Reduce the flow rate slightly so the water level stabilizes. Record the pump's settings. Calculate the flow rate using a graduated container and a stop watch. Record the flow rate. Do not let the water level drop below the top of the well screen.

If the groundwater is highly turbid or colored, continue to discharge the water into the bucket until the water clears (visual observation); this usually takes a few minutes. The turbid or colored water is usually from the well being disturbed during the pump installation. If the water does not clear, then you need to make a choice whether to continue purging the well (hoping that it will clear after a reasonable time) or continue to

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the next step. Note, it is sometimes helpful to install the pump the day before the sampling event so that the disturbed materials in the well can settle out.

If the water level drops to the top of the well screen during the purging of the well, stop purging the well, and do the following:

Wait for the well to recharge to a sufficient volume so samples can be collected. This may take awhile (pump maybe removed from well, if turbidity is not a problem). The project manager will need to make the decision when samples should be collected and the reasons recorded in the site's log book. A water level measurement needs to be performed and recorded before samples are collected. When samples are being collected, the water level must not drop below the top of the screen or open interval. Collect the samples from the pump's tubing. Always collect the VOCs and dissolved gases samples first. Normally, the samples requiring a small volume are collected before the large volume samples are collected just in case there is not sufficient water in the well to fill all the sample containers. All samples must be collected, preserved, and stored according to the analytical method. Remove the pump from the well and decontaminate the sampling equipment.

If the water level has dropped 0.3 feet or less from the initial water level (water level measure before the pump was installed); proceed to Step 7. If the water level has dropped more than 0.3 feet, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are be collected.

7. Attach the pump's tubing to the "T" connector with a valve (or a three-way stop cock). The pump's tubing from the well casing to the "T" connector must be as short as possible to prevent the groundwater in the tubing from heating up from the sun light or from the ambient air. Attach a short piece of tubing to the other end of the end of the "T" connector to serve as a sampling port for the turbidity samples. Attach the remaining end of the "T" connector to a short piece of tubing and connect the tubing to the flow-through-cell bottom port. To the top port, attach a small piece of tubing to direct the water into a calibrated waste bucket. Fill the cell with the groundwater and remove all gas bubbles from the cell. Position the flow-through-cell in such a way that if gas bubbles enter the cell they can easily exit the cell. If the ports are on the same side of the cell and the cell is cylindrical shape, the cell can be placed at a 45-degree angle with the ports facing upwards; this position should keep any gas bubbles entering the cell away from the monitoring probes and allow the gas bubbles to exit the cell easily (see Low-Flow Setup Diagram). Note,

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make sure there are no gas bubbles caught in the probes' protective guard; you may need to shake the cell to remove these bubbles.

8. Turn-on the monitoring probes and turbidity meter.

9. Record the temperature, pH, dissolved oxygen, specific conductance, and oxidation/reduction potential measurements. Open the valve on the "T" connector to collect a sample for the turbidity measurement, close the valve, do the measurement, and record this measurement. Calculate the pump's flow rate from the water exiting the flow-through-cell using a graduated container and a stop watch, and record the measurement. Measure and record the water level. Check flow-through-cell for gas bubbles and sediment; if present, remove them.

10. Repeat Step 9 every 5 minutes or as appropriate until monitoring parameters stabilized. Note at least one flow-through-cell volume must be exchanged between readings. If not, the time interval between readings will need to be increased. Stabilization is achieved when three consecutive measurements are within the following limits:

**Turbidity** (10% for values greater than 5 NTUs; if three Turbidity values are less than 5 NTUs, consider the values as stabilized),

**Dissolved Oxygen** (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),

Specific Conductance (3%), Temperature (3%), pH (± 0.1 unit), Oxidation/Reduction Potential (±10 millivolts).

If these stabilization requirements do not stabilize in a reasonable time, the probes may have been coated from the materials in the groundwater, from a buildup of sediment in the flow-through-cell, or a gas bubble is lodged in the probe. The cell and the probes will need to be cleaned. Turn-off the probes (not the pump), disconnect the cell from the "T" connector and continue to purge the well. Disassemble the cell, remove the sediment, and clean the probes according to the manufacturer's instructions. Reassemble the cell and connect the cell to the "T" connector. Remove all gas bubbles from the cell, turn-on the probes, and continue the measurements. Record that the time the cell was cleaned.

11. When it is time to collect the groundwater samples, turn-off the monitoring probes, and disconnect the pump's tubing from the "T" connector. If you are using a centrifugal or peristaltic pump check the pump's tubing to determine if the tubing is completely filled with water (no air space).

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All samples must be collected and preserved according to the analytical method. VOCs and dissolved gases samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

If the pump's tubing is not completely filled with water and the samples are being collected for VOCs and/or dissolved gases analyses using a centrifugal or peristaltic pump, do the following:

All samples must be collected and preserved according to the analytical method. The VOCs and the dissolved gases (e.g., methane, ethane, ethene, and carbon dioxide) samples are collected last. When it becomes time to collect these samples increase the pump's flow rate until the tubing is completely filled. Collect the samples and record the new flow rate.

12. Store the samples according to the analytical method.

13. Record the total purged volume (graduated waste bucket). Remove the pump from the well and decontaminate the sampling equipment.



E-101

Comments of screen WELL PURGING-FIELD WATER QUALITY MEASUREMENTS FORM 10%Turb-idity NTU Pump Intake at (ft. below MP) bottom Purging Device; (pump type) Total Volume Purged 10%mg/L DO top **EXAMPLE** (Minimum Requirements)  $\pm 0.1 \pm 10 \, \text{mv}$ ORP<sup>3</sup> шv (below MP) Depth to Hd 3% Spec. Cond.<sup>2</sup> μS/cm 3% °C Volume Purged Cum. liters Date ml/min Purge Rate Location (Site/Facility Name) Pump Dial<sup>1</sup> Sampling Organization Stabilization Criteria Depth below MP ft Water Field Personnel Well Number Identify MP 24 HR Clock Time

APPENDIX C

1. Pump dial setting (for example: hertz, cycles/min, etc).

μSiemens per cm(same as μmhos/cm)at 25°C.
 Oxidation reduction potential (ORP)

## CDFG Standard Operating Procedures for Conducting Field Measurements

SOP for Field Measurements of Surface Water / Field Collection Procedures for [Surface] Water Samples

## **Field Measurements**

## **Field Data Sheets**

Field data sheets are used to record field observations, probe measurements, and water and sediment chemistry sampling. Field data sheets are provided through the Marine Pollution Studies Laboratory website at:

http://mpsl.mlml.calstate.edu/swdwnlds.htm

Click on the *Field Data Sheets* for the most recent versions. There are guidelines provided below to standardize what is recorded on all data sheets and that should be helpful in completing each form. The Beaufort Scale (see at the end of this document) is also used for specifications and equivalent wind speeds for water conditions. The entries discussed below and on the field data sheets are recorded at each sampling site.

## Notes to Standardize SWAMP Field Data Sheets (For in the field use)

Upon arrival at a sampling site, record visual observations on the appearance of the water and other information related to water quality and water use.

Key Reminders to identify samples:

- 1. **Sample Time** is the SAME for all samples (Water, Sediment, & Probe) taken at the sampling event. Use time of FIRST sample as it is important for the chain of custody (COC).
- 2. Left Bank/Right Bank

*Left bank* is defined as the bank to the left of the observer when facing downstream, and the *right bank* is to the right of the observer when facing downstream

**FIELD OBSERVATIONS:** (each one of these observations has a *Comment* field in the database so use comment space on data sheet to add information about an observation if necessary)

- 1. **DOMINANT SUBSTRATE**: if possible; describe DOMINANT substrate type; use UNK if you cannot see the dominant substrate type
- 2. **WADEABILITY**: in general, is the water body being sampled wadeable to the average person AT the POINT of SAMPLE
- 3. **BEAUFORT SCALE**: use scale 0-12; refer to scales listed at the end of this document.
- 4. WIND DIRECTION: records the direction from which the wind is blowing
- 5. **PICTURES:** Digital photos are taken to help document the actual sampling site. The convention is to take photos facing DOWNSTREAM, overlooking the site. Right bank and left bank are thus defined in this downstream-facing direction. Document any discrepancies from this convention. Only one photo is necessary, if both, left and right

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bank, fit into one frame. Record all photos in the field data sheet space to record picture numbers given by camera; be sure to rename accordingly back in the office. All photos should be renamed and saved with the StationCode\_yyyy\_mm\_dd\_uniquecode (e.g. 123ABC123\_2007\_07\_01\_BBDS).

- 6. **SITE ODOR**: Note if hydrogen sulfide odor, musty odor, sewage odor, etc. is in the sampling reach
- 7. **SKY CODE**: Note recent meteorological events that may have impacted water quality
- 8. **OTHER PRESENCE**: VASCULAR refers to terrestrial plants or submerged aquatic vegetation (SAV) and NONVASCULAR refers to plankton, periphyton etc.
- 9. **PRECIPITATION**: Note if any precipitation is occurring during sampling
- 10. **PRECIPITATION LAST 24 HOURS**: Note how much precipitation has occurred within the last 24-h of sampling
- 11. WATER ODOR: Note if the sample water being collected has odor
- 12. **WATER CLARITY**: this describes the clarity of the water while standing creek side; clear represents water that is clear to the bottom, cloudy may not be clear to bottom but greater than 4" can be seen through the water column.
- 13. WATER COLOR: This is the color of the water from standing creek side
- 14. **OBSERVED FLOW**: Visual estimates in cubic ft/s.

## SAMPLE DETAILS:

- 1. EVENT TYPE: Note the event type based which type of media is being collected
- SAMPLE TYPE: GRAB samples are when bottles are filled from a single depth; INTEGRATED sample are taken from MULTIPLE depths and combined.
   a. GRAB: use 0.1 for subsurface samples; if too shallow to submerge bottle; depth =0
   b. INTEGRATED: -88 in depth sampled, record depths combined in sample comments
- 3. SAMPLING CREW: J. Smith, S. Ride (first person listed is crew leader)
- 4. **STARTING BANK**: Which side of the stream was accessed first. Bearings are always recorded looking downstream
- 5. OCCUPATION METHOD: What media was used to access the site
- 6. **TARGET LAT/LONG**: Refers to the existing station location that the sampling crew is trying to achieve; can be filled out prior to sampling
- 7. ACTUAL LAT/ LONG: is the location of the current sample event.
- 8. **SAMPLE LOCATION**: describes from where IN water body sample was taken: Can be combined; ex: bank/thalweg or midchannel /thalweg
- 9. **HYDROMODIFICATION:** Describe existing hydromodifications such as a grade control, drainage pipes, bridge, culvert
- 10. **HYDROMOD LOC**: if there was an IMMEDIATE (with in range potentially effecting sample) hydromodification; was sample taken upstream or downstream of modification; if there is no hydromodification, NA is appropriate
- 11. **STREAM DEPTH, WIDTH & DISTANCE FROM BANK**: describe in meters at point of sample. Distance from bank should be recorded from the starting bank

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### Field Data Logbook

A Field Data Logbook or a Field Folder is taken into the field on each sampling trip. The use of bound or loose-leaf notebooks is left up to the entity conducting the monitoring. A good safety precaution against the loss of a bound field data logbook is to photocopy the current pages upon returning from the field. These pages are kept on file at the specific sample collection entity's office. If a loose-leaf notebook is used, take care to remove original field data log sheets from the notebook and file in the office. Copies of the field data log sheets may be left in the notebook for future reference.

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**Field Data Logbooks (bound or loose leaf sheets) are maintained on file indefinitely in each regional office or contract laboratory office.** They are never discarded, since the logbook may be the only written record of field measurements. Field Data Logbooks are reviewed periodically during SWAMP QA site visits. At this point, these field notes are not inclusive of the information that would be collected for biological assessment work, and several other data measurement types.

### Flow

Sampling crews should be notified on reconnaissance forms if it is known that there is an operational United States Geological Survey (USGS) gage is located at or nearby a sampling site. If there is a USGS gage nearby, a gage height in feet is recorded and later converted to an instantaneous flow value and recorded in the logbook. The gage height is always to be reported to the USGS for conversion to flow. If a USGS gage is not available, a flow measurement should be taken, if requested. See Instantaneous Flow Measurement information starting on page 13 in this document. In addition, it is recommended that a flow severity value is recorded at each stream or river station that is not tidally influenced. See the Flow Severity section starting on page 13 of this document. Centroid velocity measurements may also be taken as a minimum acceptable rough characterization of the stream flow as requested, although this measurement is not to be recorded as a flow, since it is only a velocity measurement.

### **Record of Samples Collected for Purposes of Chemical Analysis**

The general types of chemical samples to be collected are listed for each site, since this may vary from site-to-site (e.g., metals-in-water, pesticides-in-sediments, routine water quality). Analyses authorization forms are recommended since different authorized laboratories perform different chemical analyses. The method of preservation for each chemical sample is recorded, as appropriate.

### **Record of Data Submission**

The *Logbook* field must indicate in some manner whether data recorded in the logbook has been transcribed onto data forms and submitted to the SWAMP data management staff.

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Other Observations	
Water Appearance	Note general appearance (e.g., color, unusual amount of suspended matter, debris or foam)
Sediment Appearance	Color, Odor and sediment composition should be noted.
Weather	Note recent meteorological events that may have impacted water quality; (e.g., heavy rains, cold front, very dry, very wet)
<b>Biological Activity</b>	Note excessive macrophyte, phytoplankton or periphyton growth. The observation of water color and excessive algal growth is very important in explaining high chlorophyll a values. Other observations such as presence of fish, birds and spawning fish are noted.
Watershed or Instream Activities	Note instream or drainage basin activities or events that are impacting water quality (e.g., bridge construction, shoreline mowing, livestock watering upstream).
Record of Pertinent Observations Related to Water Quality and Stream Uses	If the water quality conditions are exceptionally poor, note that standards are not met in the observations, (e.g., dissolved oxygen is below minimum criteria). Note uses (e.g., swimming, wading, boating, fishing, irrigation pumps, navigation). Eventually, for setting water quality standards, the level of use will be based on comments related to the level of fishing and swimming activities observed at a station.
Specific Sample Information	Note specific comments about the sample itself that may be useful in interpreting the results of the analysis (e.g., number of sediment grabs, or type and number of fish in a tissue sample). If the sample was collected for a complaint or fish kill, make a note of this in the observation section.
Missing Parameters	If a scheduled parameter or group of parameters is not collected, make some note of this in the comments.

## **Field Data Measurements**

While collecting water samples (see Field Collection Procedures for Water Samples section), record appropriate field measurements. When field measurements are made with a multiparameter instrument, it is preferable to place the sonde in the body of water to be sampled and allow it to equilibrate in the dissolved oxygen (D.O.) mode while water samples are collected. Field measurements are made at the centroid of flow, if the stream visually appears to be completely mixed from shore to shore. *Centroid* is defined as the midpoint of that portion of the stream width which contains 50% of the total flow. For routine field measurements, the date, time and depth are reported as a grab. Measure Quality Objectives (MQO's) for field measurements are listed in appendix C of the SWAMP QAMP.

Water Depth Less than 5 ft (<1.5 m)	If the water depth is less than 5 ft (1.5 m), grab samples for water are taken at approximately 0.1 m (4 in.), and multi-probe measurements are taken at approximately 0.2 m (8 in.). This is because all sensors have to be submerged, so 0.1 m would not be deep enough. But taking a grab sample at 0.2 m is not always feasible, as it is difficult to submerge bottles to that depth, and in many cases the bottle will hit the stream bottom.
Water Depth Greater than 5 ft (>1.5 m)	If the water depth at the sampling point exceeds 5 ft (1.5 m) in depth, a vertical profile of dissolved oxygen, temperature, pH and specific conductance are made using the multiparameter probe equipment. The depth of the sonde at the time of measurement is most accurately determined from the depth sensor on the multiparameter sonde rather than depth labels on the cable.
Vertical Depth Profiles and Depth-Integrated Sample Collection	If depth integration sampling is being conducted, or if vertical profile measurements are requested, multi-probe measurements are made starting at a depth of 0.2 m, and are then conducted at 1.0, 2.0, 3.0, 4.0, and 5.0 m depths after that until 5.0 m depth is reached. Beginning at 5.0 m, measurements are made every 5.0 m through depth profile.

Field data for multiparameter vertical depth profiles are recorded in final form on the SWAMP Field Data Sheets and submitted to the SWAMP data management staff. Go to <u>http://mpsl.mlml.calstate.edu/swdwnlds.htm</u> for detailed information on data reporting.

## Water Temperature (<sup>0</sup>C)

Water temperature data are recorded for each SWAMP visit in final form in a Field Data Logbook and submitted to the SWAMP data management staff. See <u>http://mpsl.mlml.calstate.edu/swdwnlds.htm</u> for detailed information on data reporting.

## **Temperature Sampling Procedures**

Temperature is measured in-stream at the depth(s) specified above. Measuring temperature directly from the stream by immersing a multiprobe instrument or thermometer is preferred.

## Hand Held Centigrade Thermometer

If an electronic meter is not available, the temperature is measured with a hand-held, centigrade thermometer (Rawson, 1982).

< In wadeable streams, stand so that a shadow is cast upon the site for temperature measurement.

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- < Hold the thermometer by its top and immerse it in the water. Position the thermometer so that the scale can be read.
- < Allow the thermometer to stabilize for at least one minute, then without removing the thermometer from the water, read the temperature to the nearest  $0.1^{\circ}$  C and record.
- < Do not read temperature with the thermometer out of the water. Temperature readings made with modern digital instruments are accurate to within  $\pm 0.1^{\circ}$  C.

### **Temperature Measurement from a Bucket**

When temperature cannot be measured in-stream, it can be measured in a bucket-Nalgene or plastic. Care must be taken to insure a measurement representative of in-stream conditions.

The following conditions must be met when measuring temperature from a bucket:

- < The bucket must be large enough to allow full immersion of the probe or thermometer.
- < The bucket must be brought to the same temperature as the water before it is filled.
- < The probe must be placed in the bucket immediately, before the temperature changes.
- < The bucket must be shaded from direct sunlight and strong breezes prior to and during temperature measurement.
- < The probe is allowed to equilibrate for at least one minute before temperature is recorded.
- < After these measurements are made, this water is discarded and another sample is drawn for water samples which are sent to the laboratory.

## pH (standard units)

pH data is recorded for each SWAMP visit in final form on the Field Data Sheets and submitted to the SWAMP data management staff. See <u>http://mpsl.mlml.calstate.edu/swdwnlds.htm</u> for detailed information on data reporting.

## pH Sampling Equipment

The pH meter should be calibrated according to the recommended procedures for calibration and maintenance of SWAMP field equipment. Calibration directions are listed in the manufactures field equipment operations manual. The pH function is pre and post calibrated every 24 h of use for multiparameter instruments.

## pH Sampling Procedures

### In-stream Method

Preferably, pH is measured directly in-stream at the depth(s) specified earlier in this document. Allow the pH probe to equilibrate for at least one minute before pH is recorded to the nearest 0.1 pH unit.

### pH Measurement from a Bucket

When pH cannot be measured in-stream, it can be measured in a bucket-Nalgene or plastic. The following precautions are outlined above; "Temperature Measurement from a Bucket".

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### **Potential Problems**

- < If the pH meter value does not stabilize in several minutes, out gassing of carbon dioxide or hydrogen sulfide, or the settling of charged clay particles may be occurring (Rawson, 1982).
- < If out gassing is suspected as the cause of meter drift, collect a fresh sample, immerse the pH probe and read pH at one minute.
- < If suspended clay particles are the suspected cause of meter drift, allow the sample to settle for 10 min, then read the pH in the upper layer of sample without agitating the sample.
- < With care, pH measurements can be accurately measured to the nearest 0.1 pH unit.

## **Dissolved Oxygen (mg/L)**

Dissolved oxygen (D.O.) data is recorded for each SWAMP visit in final form on a Field Data Sheet and submitted to the SWAMP data management staff. See http://mpsl.mlml.calstate.edu/swdwnlds.htm for detailed information on data reporting.

#### **Dissolved Oxygen Sampling Equipment**

The dissolved oxygen meter should be calibrated according to the recommended procedures for calibration and maintenance of SWAMP field equipment. Calibration directions are listed in the manufactures field equipment operations manual.

### **Multiprobe Instrument**

Pre and post calibrate the D.O. sensor every 24 h and for elevations greater than 500 ft on the multiprobe instrument. Preferably, D.O. is measured directly in-stream at the depth(s) specified in the Field Measurements section above. The D.O. probe must equilibrate for at least 90 s before D.O. is recorded to the nearest 0.1 % saturation or mg/L. Care must be taken at profile stations to insure that the reading is stable for each depth. Since dissolved oxygen takes the longest to stabilize, record this parameter after temperature, conductivity and pH. If the D.O. probe has an operable, automatic stirrer attached, the D.O. probe does not have to be manually stirred. However, if the probe is not equipped with an automatic stirrer, manual stirring must be provided by raising and lowering the probe at a rate of 1 ft/s (0.3m/s) without agitating the water surface. If the stream velocity at the sampling point exceeds 1 ft/s, the probe membrane can be pointed upstream into the flow and manual stirring can be avoided (Rawson, 1982).

### **D.O.** Measurement from a Bucket

When D.O. cannot be measured in-stream, it can be measured in a bucket-Nalgene or plastic, following precautions outlined in the Temperature Measurement from a Bucket listed above. During equilibration and reading, water should be moved past the membrane surface at a velocity of 1 ft/s (0.3 m/sec), either by automatic stirrer or manual stirring. If stirred manually in a bucket, the water surface is not agitated (Rawson, 1982).

## 24-Hour Average D.O. (if requested in special study)

## **Unattended 24-Hour D.O. Data Collection**

## Why Collect 24-Hour Data

Dissolved oxygen sampling for standards compliance is targeted to water bodies where low instantaneous D.O. levels indicate partial or nonsupport of designated aquatic life uses. Intensive monitoring is conducted with automated equipment that is preset to record and store field measurements hourly over one 24-h period. Four or more dissolved oxygen measurements may also be made manually at 4-6-h intervals over one 24-h period, as long as one is made near sunrise (0500-0900 h) to approximate the daily minimum. However, data collected with automated equipment is preferred.

### When to Take Measurements

All 24-h D.O. monitoring events must be spaced over an index period representing warmweather seasons of the year (approx March 15-October 15), with between one-half to two-thirds of the measurements occurring during the critical period (July 1-September 30). The *critical period* of the year is when minimum stream flows, maximum temperatures, and minimum dissolved oxygen concentrations typically occur in area streams. A flow measurement must be taken at the time of deployment. In a perennial stream, a 24-h data for standards compliance can not be used if the flow is less than the 7Q2. In perennial streams, the D.O. criterion to do not apply for flows under the 7Q2. A period of about one month must separate each 24-h sampling event. Additional samples may be collected outside the index period to further characterize a water body, but that information is generally not used for assessing standards compliance.

### **Frequency of Measurements**

The measurement interval should be no more than once per 15 min and no less than once per hour.

### Where to Take Measurements

For purposes of determining standards compliance with the 24-h average criteria, samples collected near the surface will be considered representative of the mixed surface layer. In deep streams, reservoirs, and tidally influenced water bodies, automated equipment is positioned between 1 foot (from the surface) to one-half the depth of the mixed surface layer. At least 10 24-h monitoring events (using the 24-h criteria and/or absolute minimum criteria) at each site within a 5-year period are recommended to provide adequate data for assessment.

### When to Collect Other Routine Samples, if doing 24-hour D.O. measurements

Other routine field measurements and water samples should be collect at either the time of deployment, at the reference check, or when the multiprobe recording 24-h data is retrieved. When ever possible, flow must be measured at the 24-h site.

## **Priority for Scheduling 24-Hour Sampling Events**

- < 303d listed waterbodies
- < Waterbodies with Concerns for DO problems (too few samples available for full use assessment).

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- < Occurrence of low D.O. concentrations observed during the day
- < Waterbodies with trends indicating declining D.O. concentrations
- < Waterbodies which would contribute to an Ecoregion data set

#### Data Reporting for 24-hour D.O. measurements

Dissolved oxygen values recorded over the 24-h period are summed and divided by the number of measurements to determine the average concentration, which is compared to the 24-h criterion. The lowest D.O. value from each 24-h set is compared to the minimum criterion. There will be occasions when a complete 24-h data set won't be possible. For example, if there are 18 measurements instead of 24, a time weighted diurnal average needs to be calculated. This can be easily done using GW Basic.

Support of assigned aquatic life use is based on 24-h D.O. average and minimum criteria for each monitoring event. Report the 24-h average D.O. value, number of measurements over a 24-h period, and the minimum, and maximum values. Report data as a time composite sample with a beginning and ending date and time, covering the 24-h period measured.

## Specific Conductance (µS/cm)

Specific conductance should be recorded for each SWAMP visit in final form on a Field Data Sheet and submitted to the SWAMP data management staff. See <u>http://mpsl.mlml.calstate.edu/swdwnlds.htm</u> for detailed information on data reporting.

#### **Specific Conductance Sampling Equipment**

The conductivity meter should be calibrated according to the recommended procedures for calibration and maintenance of SWAMP field equipment. Calibration directions are listed in the manufactures field equipment operations manual.

#### **Specific Conductance Sampling Procedure**

Preferably, conductivity is measured directly in-stream at the depth(s) specified earlier in this document. Allow the conductivity probe to equilibrate for at least one minute before specific conductance is recorded to three significant figures (if the value exceeds 100). The primary physical problem in using a specific conductance meter is entrapment of air in the conductivity probe chambers. The presence of air in the probe is indicated by unstable specific conductance values fluctuating up to  $\pm 100 \,\mu$ S/cm. The entrainment of air can be minimized by slowly, carefully placing the probe into the water; and when the probe is completely submerged, quickly move it through the water to release any air bubbles.

If specific conductance cannot be measured in-stream, it should be measured in the container it can be measured in a bucket-Nalgene or plastic. The following precautions are outlined above; "Temperature Measurement from a Bucket".

#### Salinity (parts per thousand--ppt, or ‰)

The value for salinity is computed from chloride concentration or specific conductance. The calculation assumes a nearly constant ratio for major ions in an estuary when seawater is diluted E-112

## **Field Collection Procedures for Water Samples**

### **Scope and Application**

This protocol describes the techniques used to collect water samples in the field in a way that neither contaminates, loses, or changes the chemical form of the analytes of interest. The samples are collected in the field into previously cleaned and tested (if necessary) sample bottles of a material appropriate to the analysis to be conducted. Pre-cleaned sampling equipment is used for each site, whenever possible and/or when necessary. Appropriate sampling technique and measuring equipment may vary depending on the location, sample type, sampling objective, and weather. Trade names used in connection with equipment or supplies do not constitute an endorsement of the product.

## **Summary of Method**

Appropriate sample containers and field measurement gear as well as sampling gear are transported to the site where samples are collected according to each sample's protocol. Water velocity, turbidity, temperature, pH, conductivity, dissolved oxygen as well as other field data are measured and recorded using the appropriate equipment. These field data measurement protocols are provided in the SWAMP Field Measurement SOP. Samples are put on ice and appropriately shipped to the processing laboratories. This procedure has been modified from the Texas Natural Resources Conservation Commission's Procedure Manual for Surface Water Quality Monitoring, with major input from the United State's Geological Survey's (USGS's) National Water Quality Assessment (NAWQA) Protocol for Collection of Stream Water Samples, for which due credit is herewith given.

## WATER SAMPLE COLLECTION

Water chemistry and bacteriological samples, as requested, are collected at the same location. *Water samples are best collected before any other work is done at the site*. If other work (e.g., sediment sample collection, flow measurement or biological/habitat sample collection or assessment) is done after or downstream of the collection of water samples, it might be difficult to collect representative samples for water chemistry and bacteriology from the disturbed stream. Care must be taken, though, to not disturb sediment collection sites when taking water samples.

The following general information applies to all types of water samples, unless noted otherwise:

Sample CollectionSub-Surface Grab SampleSamples are collected at 0.1 mDepthbelow the water surface. Containers should be opened and re-<br/>capped under water in most cases.

**Depth-integrated Sample** If a depth-integrated sample is

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	taken, the sample is pumped from discrete intervals within the entire water column.
	<b>Surface Grab Sample</b> Samples are collected at the surface when water depth is <0.1 m. Since there is a difference in water chemistry on the surface, compared to subsurface, surface water should be noted on the field data sheet as 0 m.
Where to Collect Samples	Water samples are collected from a location in the stream where the stream visually appears to be completely mixed. Ideally this would be at the centroid of the flow ( <i>Centroid</i> is defined as the midpoint of that portion of the stream width, which contains 50% of the total flow), but depth and flow do not always allow centroid collection. For stream samples, the sampling spot must be accessible for sampling physicochemical parameters, either by bridge, boat or wading. Sampling from the shoreline of any water body (meaning standing on shore and sampling from there) is the least acceptable method, but in some cases is necessary.
	In reservoirs, lakes, rivers, and coastal bays, samples are collected from boats at designated locations provided by Regional Water Quality Control Boards (Regional Boards).
Sampling Order if Multiple Media are Requested to be Collected	The order of events at every site has to be carefully planned. For example, if sediment is to be collected, the substrate can not be disturbed by stepping over or on it; water samples can not be taken where disturbed sediment would lead to a higher content of suspended matter in the sample. <i>For the most part,</i> <i>water samples are best collected before any other work is done</i> <i>at the site</i> . This information pertains to walk-in sampling.
Sample Container Labels	Label each container with the station ID, sample code, matrix type, analysis type, project ID, and date and time of collection (in most cases, containers will be pre-labeled). After sampling, secure the label by taping around the bottle with clear packaging tape.
Procedural Notes	For inorganic and organic water samples, bottles do not have to be rinsed if they are I-Chem 200 series or higher or ESS PC grade or higher. This means that the sample bottles are analyzed for contamination, and a certification of analysis is included with the bottles. Other sample containers are usually rinsed at least three times if the bottles do not meet these

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requirements. See filling instruction for each type of analyses if there is uncertainty. If applicable to the sample and analysis type, the sample container should be opened and re-capped under water.

Sample Short-term Storage and Preservation	Properly store and preserve samples as soon as possible. Usually this is done immediately after returning from the collection by placing the containers on bagged, crushed or cube ice in an ice chest. Sufficient ice will be needed to lower the sample temperature to at least 4 °C within 45 min after time of collection. Sample temperature will be maintained at 4 °C until delivered to the laboratory. Care is taken at all times during sample collection, handling and transport to prevent exposure of the sample to direct sunlight. Samples are preserved in the laboratory, if necessary, according to protocol for specific analysis (acidification in most cases).
Field Safety Issues	Proper gloves must be worn to prevent contamination of the sample and to protect the sampler from environmental hazards (disposable polyethylene, nitrile, or non-talc latex gloves are recommended, <u>however, metals and mercury sample</u> containers can only be sampled and handled using polyethylene gloves as the outer layer). Wear at least one layer of gloves, but two layers help protect against leaks. One layer of shoulder high gloves worn as a first (inside) layer is recommended to have the best protection for the sampler. Safety precautions are needed when collecting samples, especially samples that are suspected to contain hazardous substances, bacteria, or viruses.
Sample Handling and Shipping	Due to increased shipping restrictions, samples being sent via a freight carrier require additional packing. Although care is taken in sealing the ice chest, leaks can and do occur. Samples and ice should be bagged placed inside a large trash bag inside the ice chest for shipping. Ice should be double bagged to prevent melted ice water from leaking into the sample. The large trash bag can be sealed by simply twisting the bag closed (while removing excess air) and taping the tail down. Prior to shipping the drain plug of the ice chests have to be taped shut. Leaking ice chests can cause samples to be returned or arrive at the lab beyond the holding time.

collection, bubble wrap must be used when shipping glass.

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Chain of Custody (COC) Forms	Every shipment must contain a complete Chain of Custody (COC) Form that lists all samples collected and the analyses to be performed on these samples.
	Make sure a COC is included for every laboratory, every time you send a shipment of samples. Electronic COCs can also be emailed to the various laboratories but must be sent before the samples arrive at their destinations. Include region and trip information as well as any special instructions to the laboratory on the COC.
	The original COC sheet (not the copies) is included with the shipment (insert into ziplock bag) One copy goes to the sampling coordinator, and the sampling crew keeps one copy.
	Samples collected should have the salinity (in ppt), depth of collection, and date/time collected for each station on every COC.
	Write a comment on this form, if you want to warn the laboratory personnel about possibly hazardous samples that contain high bacteria, chlorine or organic levels.
Field QC Samples for Water Analyses	Field duplicates are currently submitted at an annual rate of 5%. Field travel blanks are required for volatile organic compounds at a rate of one per cooler shipped. Field blanks are required for trace metals (including mercury and methyl mercury), DOC, and volatile organic compounds in water at a rate of 5%. See Appendix C of the SWAMP QAMP for detailed Field QC requirements.
Field Site Data Sheets	Each visited field site requires a field observation completed SWAMP Field Data Sheet, even if no samples are collected (i.e. at a site which is found to be dry). If water and/or sediment samples are collected, all elements of the SWAMP Field Data Sheet must be completely filled out.
General Pre- Sampling Procedures	<b>Instruments</b> . All instruments must be in proper working condition. Make sure all calibrations are current. Multi-probe sondes should be pre-calibrated every morning prior to sampling and post-calibrated within 24 h of the original calibration. Conductivity should also be calibrated between stations if there is a significant change in salinity. Dissolved oxygen sensors should be re-calibrated if there is a 500 ft

change in elevation.

**Calibration Standards**. Pack all needed calibration standards.

**Sample Storage Preparations**. A sufficient amount of cube ice, blue ice and dry ice as well as enough coolers of the appropriate type/size must be brought into the field, or sources for purchasing these supplies identified in advance.

**Sample Container Preparation**. After arriving at the sample station, pack all needed sample containers for carriage to the actual collection site, and label them with a pre-printed label containing Station ID, Sample Code, Matrix info, Analysis Type info, Project ID and blank fields for date and time (if not already pre-labeled).

**Safety Gear.** Pack all necessary safety gear like waders, protective gloves and safety vests.

**Walk to the site**. For longer hikes to reach a sample collection site, large hiking backpacks are recommended for transport of gear, instruments and containers. Tote bins can be used, if the sampling site can be accessed reasonably close to the vehicle.

**GPS**. At the sampling site, compare/record reconnaissance GPS reading with current site reading and note differences. GPS coordinates should be in Decimal Degrees (e.g. 38.12345 -117.12345).

# COLLECTION OF WATER SAMPLES FOR ANALYSIS OF CONVENTIONAL CONSTITUENTS

In most streams, sub-surface (0.1 m below surface) water is representative of the water mass. A water sample for analysis of conventional constituents is collected by the grab method in most cases, immersing the container beneath the water surface to a depth of 0.1 m. Sites accessed by bridge can be sampled with a sample container-suspending device. Extreme care must be taken to avoid contaminating the sample with debris from the rope and bridge. Care must also be taken to rinse the device between stations. If the centroid of the stream cannot be sampled by wading, sampling devices can be attached to an extendable sampling pole.

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In some cases, depth-integrated sampling is required, as requested by Regional Boards. This is useful when lakes or rivers are stratified and a sample is wanted that represents the entire water column. Depth-integrated sample collection is explained later in this document.

Conventional Water Constituents, Routinely Requested in SWAMP	Chloride, sulfate, nitrite, nitrate (or nitrate+nitrate), ortho- phosphate, fluoride, total phosphorus, ammonia, TKN, alkalinity, chlorophyll a.
Conventional Water Constituents, Occasionally Requested in SWAMP	Total Suspended Solids (TSS) or Suspended Sediment Concentration (SSC), Total Dissolved Solids (TDSespecially if total metals requested), Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), hardness (if trace metals analysis is requested).
Conventional Water Constituents Sample Volume	Due to the potential for vastly different arrays of requested analyses for conventional constituents, please refer to table at the end of this document, as well as the Sample Handling Requirements Tables in Appendix C of the QAMP, for information on the proper volume to collect for the various types of analyses.
Conventional Water Constituents Sample Container Type	Due to the potential for vastly different arrays of requested analyses for conventional constituents, please refer to table at the end of this document, as well as the Sample Handling Requirements Tables in Appendix C of the QAMP, for information on the proper type of sample containers.
Chlorophyll a Syringe Sample Method	<b>Chlorophyll a syringe method:</b> Chlorophyll a is sampled by forcing water with a 60-mL syringe through a filter holder containing a 25-mm glass microfiber filter. The 60-mL syringe and an in-line filter holder are rinsed three times with the ambient water before filtration. The syringe is then filled with 60 mL of ambient water. The filter holder is then removed and a 25-mm glass microfiber filter is placed inside. The filter holder is then screwed onto the syringe and the ambient water is then flushed through the filter. The filter holder is removed every time more water needs to be drawn into the syringe. The process is then repeated until the desired amount of Chlorophyll a is present (usually 60 to 360 mL depending on the water clarity). When filtering is complete the filter holder is opened and the filter is removed with tweezers without

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touching the Chlorophyll a. The filter is then folded in half, then again, in half with the Chlorophyll a inside the folds. The folded filter is then wrapped in aluminum foil and placed in an envelope labeled with the site information and the volume filtered. The envelope is then immediately placed on dry ice until transferred to the lab.

## **Collection of Water Samples for Analysis of Trace Metals (Including Mercury)**

When deciding to measure total and dissolved metals in water the purpose of the sampling must be considered. Water quality standards for the protection of aquatic life are determined for the dissolved form of heavy metals in most cases, although this, too, can vary within different Basin Plans for different regions. The exception to routinely conducting dissolved metals analyses is usually mercury (and often selenium). Water quality standards usually apply to the total form of mercury (and often selenium), and not the dissolved form of these elements. Several regions are interested in conducting total metals analyses, in order to address specific issues. In order to budget inputs, transport, and accumulation of metals, it is necessary to know the concentration of total metals in the water column, sediments, effluent, etc. Sample collection for trace metals and mercury in water requires "Clean Hands/Dirty Hands" methodology.

Metals-in-water: General Information	Unless otherwise requested to collect for total metals analysis, dissolved metals are collected for all elements with the exception of mercury. Metals-in-water samples should <u>not</u> be collected during periods of abnormally high turbidity if at all possible. Samples with high turbidity are unstable in terms of soluble metals, and it is difficult to collect a representative grab sample. Special study sampling, however, may be an exception. For example, wet weather sampling is likely to include some samples with high turbidity.
Metals-in-water:	Collect a metals sample from a depth of 0.1 m using a sub - surface grab method, or at discrete depths using a depth-
Sample Collection Depth	integrated sampling method with a peristaltic pump (described further down). In most streams, sub-surface water is representative of the water mass. For the purpose of determining compliance with numerical toxic substance standards, a sample taken at the surface is adequate.
Metals-in-water:	Refer to table at end of this document, as well as Sample Handling Requirements Tables in Appendix C of the QAMP,
Sample Volume	for specific information on the proper volume to collect for trace metals analyses. Generally, for procedures most commonly used for analysis of metals in water (total or dissolved metals); one 60-mL polyethylene container is filled with the salinity recorded on the field data sheet and COC. Generally, for the procedures most commonly used for analysis of mercury in water (whether total or dissolved), one 250-mL glass or teflon container is filled, regardless of the salinity. All containers are pre-cleaned in the lab using HNO <sub>3</sub> .

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Metals-in-water: Sampling Equipment	The method of choice for the collection of water samples for trace metals analysis in small, wadeable streams is the grab method, where the sampler submerges the sample bottle or syringe beneath the surface of the water until filled. The procedure for filtration of water samples for trace metals (including mercury) analysis must be performed within the 48-h maximum holding time (as well as acid preservation), and with extreme care to avoid contamination of the water sample. Considering these factors, it is best to use a <b>field</b> filtration system, such as a set-up with peristaltic pump with in-line filter, or a set-up with a syringe filter, if filtered water is required. Samples are pumped and/or filtered directly into the sample container. This minimizes contamination by using no intermediate sampling device. Samples can also be filtered in lab if need be Un-powdered (no-talc) polyethylene gloves are always worn during sampling for metals-in-water.
	Depth-integrated sampling is useful when lakes or rivers are stratified and a representative sample is wanted which represents the entire water column. The method involves a peristaltic pump system with enough Teflon tubing to pump at the desired depth with an inline filter. Alternatively, mercury and metal samples can be filtered in the laboratory as long as they are filtered within the 48-hr maximum holding time and filter equipment blanks are analyzed for five percent of all cleaned equipment.
Equipment Preparation	<ul> <li>It is best if the metals-in-water sampling materials are prepared by a laboratory that can guarantee contamination-free sampling supplies. If a laboratory assembles a Metals-in-Water Sample Collection Kit, it should contain the following items packaged together <u>for each sample</u>:</li> <li>Tubing with an in-line filter (disposable, 0.45 μm) attached for dissolved metals-in-water sampling. This same tubing is used for total metals-in-water samples without filter. If an in-line pumping system is not used, an acid cleaned syringe and filter are packed.</li> <li>Sample containers- polyethylene for total and dissolved samples and blanks; Glass or Teflon for total and dissolved mercury.</li> <li>Acid preservation is performed in the laboratory.</li> <li>Metals-free DI water (for blanks).</li> </ul>
• Powder-free polyethylene gloves

If a laboratory is not assembling collection kits, individuals should take care to keep containers in the original packaging. When removed from the box, sample containers are placed in plastic bags (ziplock bags). Although filters come individually wrapped, they should also be stored in new ziplock bags to avoid possible contamination.

The filtering equipment is pre-cleaned according to laboratory protocol. Clean tubing is put into clean containers, such as large ziplock bags. Metals-free filter cartridges with the capacity to filter several liters are commercially available. Equipment blanks are run at the laboratory on batches of metals-in-water sampling equipment prior to their distribution to field staff. One to two liter containers with metals-free deionized water are taken into the field for travel blanks. Metals-free deionized water is supplied by the laboratory performing metals analysis. The deionized water containers are kept clean and dust-free on the outside by wrapping in two plastic bags.

# **Dissolved and Total Metals-in-Water: Detailed Collection Techniques**

- ✤ Sub-Surface Grab Method
- Syringe Filtration Method (for sub- surface collection)
- Peristaltic Pumping Method (Using Tubing/In-line Cartridge Filters) for sub- surface collection or for depth-integrated collection

Metals-in-water	Unfiltered Samples (for total metals analysis, if requested,	
Sample Collection:	and for mercury almost always, unless otherwise	
	requested): Some samples can be sampled directly from the	
Sub-Surface Grab	ambient water either by wading into the stream and dipping	
Method	bottles under the surface of the water until filled, or by	
	sampling from a boat and dipping the bottle under the surface	
	of the water until it is filled. The bottles are cleaned according	
	to laboratory protocol. It is very critical that all the acid is	
	rinsed out of the bottles before the samples are taken.	
	Personnel involved in field sample collection/processing wear	
	polyethylene gloves. The laboratory pre-cleaned glass or	
	Teflon <sup>™</sup> 250 mL (for mercury) or polyethylene 60 mL (for	

metals) sample bottles are taken from the double-wrapped plastic bags using "Clean Hands/Dirty Hands" techniques. The dirty hands person opens the first bag, and the clean hands person opens the inner bag around the bottle. The clean hands person then removes the bottle from the inner bag. The clean hands person dips the bottle into the ambient water, with the cap on, to approximately 0.1 m (avoiding disturbing surface scums), placing the cap back on the bottle before being removed from the water, rinses the bottle five times with ambient water, making sure the threads of the bottle get rinsed as well, and fills the bottle to the top. The lid is secured and the bottle is put back into the inner clean bag and sealed by the clean collector. The dirty hands collector then seals the outer bag. Metals-in-water Filtered Samples (for dissolved metals analyses): Subsurface water samples are filtered for dissolved trace metals Sample Collection: analysis (not for mercury, however, in almost all cases) using Syringe Filtration the following syringe filtration method. Method (for subsurface collection) The syringe (60 cc size, pre-cleaned in the laboratory) and inline filter are pre-packed in two ziplock bags. The syringe and filter are taken out of the bags using "Clean Hands/Dirty Hands" technique, as previously described. The sub-surface water sample is collected by 1) wading out into the centroid portion of the stream, or by leaning over the edge of the boat, and aspirating water into the syringe, filling and rinsing the syringe five times with ambient water; 2) attaching the filter onto the syringe and filling the syringe body; 3) rinsing the filter with a few milliliters of the sample; 4) rinsing the sample bottle five times with the filtered ambient water; and 5) extruding the sample through the syringe filter and completely filling each bottle. The bottles are taken out of and put back into their bags using "Clean Hands/Dirty Hands". Metals-in-water The basic "Clean Hands/Dirty Hands" technique is also Sample Collection-applied in the use of a peristaltic pump with an in-line filter cartridge for metals-in-water sample collection. Dirty Hands **Peristaltic Pump** removes the plastic cover from the end of the pump tubing and inserts the tubing into the sampling container. Dirty Hands holds the tubing in place. The in-line cartridge filter is attached to the outlet end of the tubing.

Clean Hands takes the plastic cover off the other end of the tubing. Dirty Hands turns on the pump and flushes l L of ambient water through the tubing to purge it for dissolved

metals.

Clean Hands removes the cap from the sample bottle and uses the pump to fill it with ambient water. Clean Hands puts the cap back on the bottle and places it in the plastic bag.

#### Metals-in-water Sample Collection:

Depth-Integrated Sampling, using Inline Cartridge Filter and Peristaltic Pump **Preparation for Depth-integrated sample collection:** Depth-integrated sampling is useful when lakes or rivers are stratified, and a representative sample is wanted that represents the entire water column to the extent possible. The method utilized to date for SWAMP involves a peristaltic pump system with enough Teflon tubing to pump from the desired depth. Regional Boards must request depth-integrated sampling.

The tubing set consists of a small length of CFLEX tubing that fits in the peristaltic pump, with an appropriate length of Teflon tubing on the suction side of the pump and a 3-ft section of Teflon tubing on the discharge side of the pump.

The tubing set is pre-cleaned in 10% reagent grade HCL at the laboratory, and to date in SWAMP, a new pre-cleaned tubing set is used for each site. However, the same peristaltic tubing set <u>can</u> be used at multiple sites, as long as it has been cleaned in the field between stations, according to protocol as outlined below. If this is to be done, however, and Dissolved or Total Organic Carbon samples are collected, equipment blanks should be collected at each site until it is determined that the blanks are acceptably low.

The field cleaning procedure for tubing that is to be re-used is:

- Pump phosphate free detergent through tubing.
- Pump 10% HCL through tubing.
- Pump methanol through tubing.
- Pump 1 l of blank water (Milli-Q) through.

All reagents must be collected in appropriate hazardous waste containers (separated by chemical), and transport, as well as disposal, must follow appropriate local, state, and federal regulations.

If a field blank is needed, collect it after the 1 L of blank water is pumped through. Pump the amount of ambient water

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equivalent to 3 times the volume of the tubing before sampling the next site.

#### Filtered and Unfiltered Samples, Depth-integrated:

It is recommended to attach the tubing to a line with depth measurement markers (preferably in meters). At the end of this line should be a trace metal-safe weight, which hangs about one meter below the tubing end, avoiding any sediment intake from the bottom of the water column with the pump tubing.

At the site, Dirty Hands sets up the pump, while Clean Hands takes a bottle from the plastic bag and places it in a container holder or on a clean surface. A container holder can be anything trace metal clean that supports the bottle, freeing up the collector's hands. Clean Hands takes the outlet-end of the tubing (with the in-line filter cartridge attached) out of the bag. and places it in the peristaltic pump head. The outlet end is long enough to allow easy bottle filling; the other end is long enough to easily reach beneath the water surface and to the desired depth. Dirty Hands closes the pump head, locking the tubing in place.

Make sure that all bottles are filled with a depth-integrated water sample. This can be accomplished by dividing the total vertical length of the water column into 2 to 10 equal intervals, and sampling each interval equally, filling the bottles at each depth proportional to the number of intervals sampled. For example, if 10 intervals are sampled, every bottle is filled 1/10<sup>th</sup> full at each depth sampled. A very common method of dividing the water column is by first determining the depth of the thermocline. Samples are taken at the midpoint between the surface and the thermocline, at the midpoint between the top of the thermocline and the bottom of thermocline, and at the midpoint between the bottom of the thermocline and just above the bottom of the water column. For these methods, all containers have to be filled at the same time. Note the number of intervals sampled on the data sheet.

When filling bottles, Clean Hands immerses the intake tube directly into the water at the appropriate depth, and Dirty Hands operates the pump to flush the tubing with a minimum of 1L of ambient water through the tubing and filter.

Clean Hands removes the cap from the sample bottle, holds

the tubing outlet with the in-line filter cartridge over the container opening (without touching the container), and allows the container to fill. The container is filled and rinsed five times with ambient water, and is then filled to the top for the actual sample. Clean Hands puts the cap back on the bottle, and places the bottle back it in the plastic bag. Whenever Clean Hands touches the boat or equipment, which may be contaminated, gloves should be changed immediately. (Note for Unfiltered samples: If an unfiltered sample is required for total metals, total mercury, conventional constituents, toxicity, or synthetic organics, the same procedure is used as described above, except the filter is detached from the end of the tubing before filling the bottles.) When sampling is finished, the tubing is brought to the surface, clean water (Milli-Q or deionized) is pumped through system, and the tubing is stored in a polyethylene bag. The tubing set can be used at multiple sites, as long as it has been cleaned in the field between stations (see field cleaning procedure above). However, if Dissolved or Total Organic Carbon samples (in water) are collected, equipment blanks should be collected at enough sites until it is determined the blanks are appropriate. **Collecting the Sample:** Metals-in-water **Sample Collection:** The sample collection methodologies are identical to those described above except the sample is collected first into a *Composite Bottle* composite bottle(s). The sample is collected in an amber glass 4-L bottle for mercury and methyl mercury, and a 4-L polyethylene bottle for other trace metals. The compositing bottle is cleaned according to SWAMP SOP.SC.G.1. It is very critical that all the acid is rinsed out of the bottle and that the bottle is rinsed with sample water (five times) before the sample is taken. The sample is collected by the grab or pumping method after being rinsed five times with ambient water and is brought inside the water quality vehicle or sampling box for processing. Personnel involved in sample processing don polyethylene gloves. During sampling the dirty hands person opens the bag holding the composite bottle and opens the outer plastic bag. The clean hands person opens the inner plastic bag, removes the bottle and holds the bottle while the Dirty Hands sampler controls the flow of water

through the pump into the bottle.

#### Preparing sample aliquots from a composite bottle into smaller sample bottles using an inline pump and filter:

The dirty hands person opens the first bag, and the clean hands person opens the inner bag around the composite bottle. The clean hands person then removes the bottle from the inner bag and places the bags and the bottle in a designated clean place.

This process is repeated until all sample bottles are lined up on the clean bench with their tops still on.

The top of the bottles are loosened so that they fit very loosely on top of the bottles so the clean hands person can remove the caps and pour or pump water into the bottles easier.

The clean hands person shakes the 4-L sample in a steady and slow up and down motion for two full minutes.

Samples that are not to be filtered (including TSS/SSC) are subsampled out of the bottle by pouring out of the large compositing bottle into the sample bottles. The compositing bottle is shaken for 15 s between these subsamples.

Each sample bottle is rinsed five times with ambient water before filling.

For the clean pumping system setup procedure, see above.

(The equipment or field blank is processed exactly like a sample following the same steps.)

The clean end of the tubing used for suction is placed into 1 L bottle. Approximately 750 mL of Milli-Q are then pumped through the system to purge any residual contamination.

The 250-mL sample bottles are then filled to the neck and capped as soon as possible.

Note: if volatile organics are to be collected they should be pumped directly into the sample containers before the compositing procedure.

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Metals-in-water:	After collecting the sample, the double-bagged container is placed in another plastic bag for shipping, and placed on ice in
Short-term Sample Preservation	the ice chest, cooled to 4 °C. This is to prevent possible contamination from other samples in the ice chest. Metals-in- water samples are acid-preserved in the lab.
Metals-in-water:	Label each outer sample-bag with the station ID, sample code, matrix type, analysis type, project ID, and date and time of
Sample Container Label	collection.
Metals-in-water: Field Equipment Blank	<b>Pumping Method.</b> If required, field blanks are collected at the last site of a sampling trip, with the same tube and filter used to collect the last dissolved metals-in-water sample of the day (before the ambient sample is collected); and with the tube used for the last total metals-in-water sample of the day. If each sample is taken using a new set of tubing, a separate tubing-set should be used for the blank.
	The same Clean Hands/Dirty Hands collection techniques are followed for the field blank as the samples, pumping trace metal-free water from a clean container supplied by the laboratory.
	<b>Syringe Method.</b> If required, field blanks are collected in much the same way as in the pumping method. "Clean Hands/ Dirty Hands" techniques are used. The syringe is taken out of the double bags, deionized water is aspirated into the syringe, syringe is rinsed five times with ambient water, the filter is attached, and the blank water is extruded into a sample bottle. A minimum of one blank per trip is taken, if required.
	<b>Grab Method.</b> Bottles full of deionized water or Milli-Q are opened at the site for the same length of time the sample bottles are open.

#### **COMPANION SAMPLES FOR METALS-IN-WATER**

A hardness analysis should be requested by the Regional Water Control Board whenever metalsin-water are to be analyzed from an inland (freshwater) site. Estuarine/marine sites do not require hardness analysis.

If a total metals sample is collected, it is recommended to submit a sample for total suspended solids/suspended sediment concentration (TSS/SSC) in a companion sample for "conventionals in water".

# **Hexavalent Chromium**

Very rarely, a request may be made for conducting hexavalent chromium analysis in water samples. Acidification alters the hexavalent form of chromium. A separate (un-acidified) sample must be submitted if hexavalent chromium is to be analyzed. Filter and submit a minimum of 500 mL water. The sample is collected in a DI-water-rinsed plastic or glass container, placed on ice, and shipped to the lab in time for analysis to begin within 24 h of collection. The lab must be notified when a hexavalent chromium sample will arrive. Hexavalent chromium is not usually analyzed on unfiltered samples.

#### FIELD QC SAMPLE COLLECTION REQUIREMENTS FOR METALS-IN-WATER

In order to assess contamination, "blanks" are submitted for analysis. Special projects may have other requirements for blanks. The same group of metals requested for the ambient samples are requested for the blank(s). Run a blank for each type of metal sample collected. Blanks results are evaluated (as soon as available) along with the ambient sample results to determine if there was contamination or not. See Appendix C of the QAMP for MQO's regarding frequency and types of field QC samples.

Field Equipment S Blank (Ambient 1 Blank) 1 5 5 6 7 7 7 7	Submit an equal volume (equal to the ambient sample) of metals-free deionized water that has been treated exactly as the sample at the same location and during the same time period. Use the same methods as described above (Grab sample, pumping method, syringe method). At least one ambient blank per field trip is required each for trace metal and Mercury samples in water. <i>If contamination is detected in</i> <i>field equipment blanks, blanks are required for every metals-</i> <i>in-water sample until the problem is resolved.</i>
Laboratory I Equipment 6 Blank 2 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	Laboratory Equipment Blanks for pumping and sampling equipment (Metals-in-Water Sample Collection Kits and Syringe Filtration Kits) are run by the laboratory that cleans and distributes the collection materials. It documents that the materials provided by the laboratory are free of contamination. When each batch of tubes, filters, bottles, acid and deionized water are prepared for a sampling trip, about five percent of the Mercury sampling materials are chosen for QC checks. Trace metal equipment needs to be subjected to an initial plank testing series. If these blanks are acceptable only poccasional re-testing is required for TM equipment. The QC checks are accomplished by analyzing metals-free water

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in a sample container; and preserved.

Field DuplicatesFive percent Field Duplicates are submitted every year. (If<br/>less than 20 samples are collected during an event, submit one<br/>set of duplicates per event.)

# **Collection of Water Samples for Analysis of Synthetic Organic Compounds**

Collect organic samples at a depth of 0.1 m by submerging the sample container by hand. If depth-integrated sampling is required, use the in-line peristaltic pump methodology described previously. Since organic compounds tend to concentrate on the surface of the sampling device or container, the sampling device and sample container are <u>not</u> to be rinsed with ambient water before being filled.

#### Sample Containers and Collection

Also refer to Appendix C of the SWAMP QAMP for a list of sample volumes and containers.

Pesticides/	The sample container for pesticides and herbicides is a new,
Herbicides	clean, unused amber glass jar with a Teflon-liner inside the
	cap. Collect one liter of water for each of the three sample
	types (Organophosphorus Pesticides, Organochlorine
	Pesticides and Chlorinated Herbicides). EACH ANALYSIS
	TYPE REQUIRES A SEPARATE JAR. Minimize the air
	space in the top of the jar. Preserve immediately after
	collection by placing on ice out of the sunlight.
Semi-volatile	The sample container for semi-volatile organics must also be
Organics	new, clean, unused amber glass bottles with a Teflon-liner
	inside the cap, and pre-rinsed with pesticide-grade hexane,
	acetone, or methylene chloride. Fill jars to the top and place
	on ice in the dark. In addition to other sample information,
	label the jar Semi-volatiles.
Volatile Organics:	The sample containers for volatiles are VOA vials. Fill the 40-
	mL VOA vials to the top and cap without trapping any air
Volatile Organic	bubbles. If possible, collect directly from the water, keeping
Carbon (VOC),	the vial under water during the entire collection process. To
Methyl-Tert Butyl	keep the vial full while reducing the chance for air bubbles,
Ether (MTBE) and	cap the vials under the water surface. Fill one vial at a time
(BTEX)	and preserve on ice. The vials are submitted as a set.
	If the vial has been pre-acidified for preservation, fill the vial quickly, without shaking using a separate clean glass jar. Fill the vial till the surface tension builds a meniscus, which extends over the top end of the vial, then cap tightly and check for bubbles by turning the vial on its head.
	Ensure that the pH is less than 2. If the water may be alkaline or have a significant buffering capacity, or if there is concern that pre-acidified samples may have the acid wash out, take a

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	few practice vials to test with pH paper. It may take more than two drops, and it will then be known how to preserve the other samples that are being submitted to the lab. If an alternative method has proven successful, continue with that method.
	<u>Note</u> : If vigorous foaming is observed following acidification, discard that sample and collect another set. <u>Do not</u> acidify the second set. Mark the sample clearly "not acidified" and the lab will run them immediately. Holding time is 14 days with acid, 24 h without acid.
	Collect three VOA vials, if VOC, MTBE and BTEX are required, two vials, if only VOC is required and two vials, if only MTBE and BTEX are require. The vials may be taped together to keep them together.
Perchlorate	Surface water samples for perchlorate should be collected in a new unused polyethylene or glass container. Perchlorate samples should be placed immediately on ice to maintain temperature at 4 °C. The sample holding time is 28 days, under refrigeration.
Sample Treatment in Presence of Chlorine	(NOTE: This treatment has not been performed in SWAMP, but may be in the future, or if a known or suspected chlorine residual is suspected and this information is made known by a Regional Board SWAMP contact beforehand.)
	If in stream chlorine residual is suspected, measure the chlorine residual using a separate water subsample. Free chlorine will oxidize organic compounds in the water sample even after it is collected. If chlorine residual is above a detectable level, (i.e., the pink color is observed upon adding the reagents) immediately add 100 mg of sodium thiosulfate to the pesticides, herbicides, semivolatiles and VOA samples; invert until sodium thiosulfate is dissolved. Record the chlorine residual concentration in field logbook. If chlorine residual is below detectable levels, no further sample treatment necessary
VOA Trip Blank	Submit one Trip Blank for VOA samples (2- 40 mL VOA vials) for each sampling event. Trip Blanks are prepared in advance just before the sampling trip and transported to the field. Ask the laboratory for DI water and specify that it is for a VOA trip blank. VOA blanks require special purged water. Trip blanks demonstrate that the containers and sample handling did not introduce contamination. The trip blank vials

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Field QC Samplesare never opened during the trip.Field QC SamplesIf required, field Duplicates and field blanks are submitted at a<br/>rate subject to the discretion of the project manager. Refer to<br/>Appendix C of the SWAMP QAMP for details on required<br/>blanks and duplicates.

# **BACTERIA AND PATHOGENS IN WATER SAMPLES**

#### Summary of Collection Procedure (Based on EPA water quality monitoring procedures)

Make sure the containers are sterilized; either factory-sealed or labeled.

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Whirl-pak® bags	<ul> <li>Label the bottle as previously described for SWAMP.</li> <li>Tear off the top of the bag along the perforation above the wire tab just prior to sampling. Avoid touching the inside of the bag. If you accidentally touch the inside of the bag, use another one.</li> <li>If wading into the stream, try to disturb as little bottom sediment as possible. Be careful not to collect water</li> </ul>
	that has sediment from bottom disturbance. Stand facing upstream. Collect the water sample on your upstream side, in front of you. You may also attach your bottle to an extension pole to sample from deeper water.
	• If taking sample from a boat, carefully reach over the side and collect the water sample on the upstream side of the boat.
	• Hold the two white pull-tabs in each hand and lower the bag into the water on your upstream side with the opening facing upstream. Open the bag midway between the surface and the bottom by pulling the white pull-tabs. The bag should begin to fill with water. You may need to "scoop" water into the bag by drawing it through the water upstream and away from you. Fill the bag no more than 3/4 full.
	<ul> <li>Lift the bag out of the water. Pour out excess water. Pull on the wire tabs to close the bag. Continue holding the wire tabs and flip the bag over at least 4-5 times quickly to seal the bag. Don't try to squeeze the air out of the top of the bag. Fold the ends of the wire tabs together at the top of the bag, being careful not to puncture the bag. Twist them together, forming a loop.</li> <li>If the samples are to be analyzed in the lab, place them in a cooler with ice or cold packs for transport to the</li> </ul>
Screw cap containers	<ul> <li>lab.</li> <li>Label the bottle as previously described for SWAMP.</li> <li>Remove the cap from the bottle just before sampling. Avoid touching the inside of the bottle or cap. If you accidentally touch the inside, use another bottle.</li> </ul>
	• If wading into the stream, try to disturb as little bottom sediment as possible. Be careful not to collect water that has sediment from bottom disturbance. Stand facing upstream. Collect the water sample on your upstream side, in front of you. You may also attach your bottle to an extension pole to sample from deeper

water.

- If taking sample from a boat, carefully reach over the side and collect the water sample on the upstream side of the boat.
- Hold the bottle near its base and plunge it (opening downward) below the water surface. If you are using an extension pole, remove the cap, turn the bottle upside down, and plunge it into the water, facing upstream. Collect a water sample 2" beneath the surface. You can only use this method if the sample bottles do not contain sodium thiosulfate.
- Turn the bottle underwater into the current and away from you. In slow moving stream reaches, push the bottle underneath the surface and away from you in an upstream direction.
- Alternative sampling method: In case the sample bottle contains preservatives/chlorine removers (i.e. Sodium-Thiosulfate), it cannot be plunged opening down. In this case hold the bottle upright under the surface while it is still capped. Open the lid carefully just a little to let water run in. Fill the bottle to the fill mark and screw the lid tight while the bottle is still underneath the surface.
- Leave a 1-in. air space so that the sample can be shaken just before analysis. Recap the bottle carefully, remembering not to touch the inside.
- If the samples are to be analyzed in the lab, place them in a cooler with ice or cold packs for transport to the lab.

#### Pouring from another clean bottle

• Due to different sampling conditions (high turbidity, rough water etc.) it is sometimes easy to pour water from another clean bottle into the bacteria bottle. This helps to make sure that the sample water is only being filled to the desired line and no overfilling occurs.

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#### TOXICITY IN WATER

**Sample Collection** Using the standard grab sample collection method described previously for water samples, fill (for typical suite of water toxicity tests conducted) the required amount of 2.25-L amber glass bottles with water, put on ice, and cool to 4 °C. Label the containers as described above and notify the laboratory of the impending sample delivery, since there is a 48-hr maximum sample hold time. Sample collection must be coordinated with the laboratory to guarantee appropriate scheduling.

Caltrans Guidance Manual: Storm Water Monitoring Protocols

SOP for Equipment Installation and Maintenance / [Storm Drain Outfall] Sample Collection

# JULY 2000

# GUIDANCE MANUAL: Stormwater Monitoring Protocols

# (Second Edition)

California Department of Transportation 1120 N Street Sacramento, CA 95814



## CTSW-RT-00-005

This section provides guidance for the installation and maintenance of stormwater monitoring stations that are equipped with automated monitoring equipment. Automated monitoring stations typically include the following basic elements:

- > Protective Equipment Enclosures
- Power Source
- ► Flow Meter
- ► Automated Sampler
- ► Rain Gauge
- ► Confined Space Entry
- ► Bottle and Equipment Cleaning and Installation

# ► PROTECTIVE EQUIPMENT ENCLOSURES

Stormwater monitoring stations containing automated monitoring equipment require a proper protective enclosure that is lockable, resistant to vandalism and tampering, and provides protection from the elements. In areas where equipment tampering may be a concern, the protective enclosures should be surrounded by chain link fencing with a locked gate and razor wire along the top. Figure 7-1 illustrates security for a sampling station equipped with steel enclosure, chain-link fencing, and razor wire.

There are two basic types of enclosures typically used to house monitoring equipment: 1) a heavy gauge steel box with hinged lid, and 2) a walk-in shed type enclosure. The advantages of a walk-in enclosure include shelter for field personnel from rain, more room for field crew to work, and storage for extra sample bottles and equipment. However, walk-in enclosures tend to be more costly than box type enclosures.

The protective enclosure should be secured to a concrete pad, with all wiring and tubing entering/exiting the enclosure routed through appropriately sized conduit. Examples of typical steel box and walk-in monitoring station enclosures are presented in Figures 7-2 and 7-3.

# ► POWER SOURCE

Commercially available automated stormwater monitoring equipment typically has the capability of running on either AC or DC power. At monitoring stations where AC power is available, the preferred setup is to operate the equipment using AC power with

DC battery backup. This will allow monitoring to continue in the event of a power outage. However, refrigerated samplers cannot be powered by DC power. Making AC power available to a monitoring station typically involves having an electrician run power from nearby power lines to a metered fuse box, and finally into the enclosure.

Sampling locations that do not have AC power lines nearby typically make providing AC power cost prohibitive. At installations where AC power is not readily available, automated monitoring equipment must be powered using DC batteries. Solar panels may be installed to provide continuous DC battery charging. Immediately prior to each stormwater monitoring event, all monitoring equipment batteries should be checked and replaced with freshly charged batteries, as necessary.



Figure 7-1. Example of Equipment Security





Figure 7-2. Steel Box Monitoring Station Enclosure



Figure 7-3. Walk-In Monitoring Station Enclosure (Source: American Sigma, Model 6989)

#### ► FLOW METER

There are two basic types of flow measuring devices typically used for flow weighted stormwater sampling: 1) depth sensors, which convert level measurements to flow rates based on the known pipe or channel geometry and an assumed relationship of depth to flow rate (usually using Manning's equation – see Figure 5-5), and 2) area velocity measuring devices, which measure both the depth and velocity of flow to produce a more accurate estimation of flow rate. The applicability of flow measuring devices and Manning's equation are described in detail in *Section 5*.

Flow monitoring equipment should be installed and maintained according to manufacturer specifications. Flow monitoring equipment should be calibrated, at a minimum, according to manufacturer recommended frequencies. For some applications, more extensive calibration procedures may be required to insure accurate flow measurement.

#### Installation

The flow meter should be securely fastened to the inside of the protective enclosure in such a way that all controls, display windows, and cable connections are easily accessed. All cables entering/exiting the flow meter should be secured, in a well organized fashion, to the inside of the protective enclosure. This will reduce the potential for accidental disconnection or damage.

The flow monitoring sensor(s) must be installed in the channel, pipe, or flume according to manufacturer specifications. Typically, stainless steel expanding bands are used to mount sensors inside pipes. Sensors may also be mounted at the base of the channel, pipe, or flume using a stainless steel base plate and hardware. The cable(s) that connect the sensor(s) to the flow meter should be housed in conduit from the point at which the cable(s) exits the protective enclosure. Because turbulence can significantly influence flow reading accuracy, placement of the sensor is extremely important. Conveyance pipe segment connecting joints are typical generators of turbulence. Additionally, sensor cables should be secured in such a way as not to create turbulence. Once installed, flow accuracy can be determined by releasing a known quantity of water at varying rates through the conveyance and comparing actual volume released with the volume measured. An example of flow sensor installation in a pipe is shown in Figure 7-4.



Figure 7-4. Flow Sensor Installation

#### Maintenance

The flow monitoring equipment should be calibrated according to manufacturer specifications. Flow meters typically contain desiccant packets and moisture indicators to keep the internal components of the equipment dry. The moisture indicators should be checked during each site visit, or at least once between each monitoring event. Often, system malfunctions can be attributed to high moisture levels inside the equipment. Any time a moisture indicator reads above the acceptable level, the desiccant should be replaced with new packets. At a minimum, the sensor(s) should be inspected and calibration checked prior to each monitoring event. The sensor(s) should be calibrated on an as-needed basis. The sensor cable(s) should be inspected at least prior to each stormwater monitoring season. All connections into the flow meter should be visually inspected prior to each monitoring event.

# ► AUTOMATED SAMPLER

The automated sample collection equipment should be installed and maintained according to manufacturer specifications. See *Section 5* regarding selection of automated equipment.

#### Installation

The automated sampler should be installed inside the protective enclosure in such a way that all controls, display windows and cable connections are easily accessed. All wiring should be secured, in a well organized fashion to the inside of the enclosure to prevent accidental disconnection or damage. The sampler must be oriented in a way that will allow the sample intake tubing to enter the sampler without sharp bends or kinking, and to allow easy access for tubing replacement (see Figure 7-5).

At the sampler peristaltic pump, where the sample intake tubing is connected to the pump tubing, no metallic fittings or clamps should be used. Using "clean techniques" (*Appendix F*), the Teflon intake tubing should be inserted (at least a half inch) into the flexible pump tubing and fastened using a non-metallic clamp or cable tie. At no time during this procedure should the ends of the tubing be allowed to touch any object that is not known to be clean (see page 7-10). The flexible pump tubing should then be fed through the peristaltic pump and into the area of the sampler where the sample bottle(s) are housed (see Figure 7-6).

Adequate space must be available in the equipment enclosure to easily remove and replace sample bottles from the automatic sampler.

Proper placement of the sampler intake assures the collection of representative samples. The intake strainer should be placed in the main flow. The vertical position of the intake strainer in the flow is important. Placement at the bottom may result in excess heavy solids and no floating material, while placement at the top may result in excess floating material and no heavy solids. The constituents of interest must be considered when positioning the intake strainer. Placement of the intake strainer is usually at the channel invert, but may be mounted slightly above the invert on one side of the channel wall if high solids loadings are expected. This will reduce the amount of solids that may enter the intake strainer, and help prevent blockages. However, with the intake strainer offset above the channel invert, low flows may not adequately submerge the strainer, thus preventing sample collection.

#### Maintenance

Using laboratory provided blank water, the automated sampler should be calibrated according to manufacturer specifications to collect the desired sample aliquot. At a minimum, the calibration should be checked prior to each stormwater monitoring season. After each stormwater monitoring event, the sample bottle(s) should be checked to verify that the programmed sample volume was delivered to the sample bottle(s). If the programmed sample volume was not delivered accurately to the sample bottle(s), the automatic sampler should be recalibrated prior to the next monitoring event. Detailed information on the programming of automated equipment is presented in *Section 9*.



Figure 7-5. Automated Sampler Installation



Figure 7-6. Pump Tubing Installation

# ► CONFINED SPACE ENTRY

The installation and maintenance of stormwater monitoring devices often requires entry into designated confined spaces. At no time during storm conditions, or at any other time when significant flows are present, should any person enter a confined space. Any below-ground-level space that requires entry for equipment installation must be evaluated by personnel trained and certified in confined space entry. Only confined space certified personnel, with proper equipment and training, may enter a confined space. This holds true in the event of an accident. If an accident occurs, do not enter the confined space, but immediately request assistance from confined space certified personnel.

# ► BOTTLE AND EQUIPMENT CLEANING AND INSTALLATION

Prior to each stormwater monitoring event, sample bottles and sampling equipment should be cleaned and installed as specified in the following subsections.

### Sample Bottle Cleaning

Prior to each stormwater monitoring event, clean sample bottles must be ordered from the analytical laboratory. Sample bottles must be prepared by the laboratory as specified by analytical method protocols. This includes the addition of sample preservatives where applicable. See Table 12-1 for specific bottle and preservative requirements. Sample preservation is discussed in more detail in *Section 10*. Specific bottle cleaning procedures are presented in *Appendix E*. Sample bottles should be stored in a clean environment with lids securely on until the time of use.

# **Composite Sample Bottle Installation**

Clean composite sample bottles should be installed into automated sampling stations using "clean techniques" as described in *Section 10* and *Appendix F*.

# Sampling Equipment Cleaning

Any sampling equipment that comes in contact with the sample must be cleaned according to protocols presented in *Appendix E*. Sampling equipment that most frequently comes in contact with the sample includes pump and sample intake tubing, intake strainers, composite bottle lids, and any grab sampling device, such as a bailer. After cleaning, each clean item should be individually double bagged (sealed in a plastic bag, then sealed in a second plastic bag) and stored until the time of installation or use. The plastic bags used for this purpose must be new and not previously used for any other purpose. Ziplock bags are ideal for this application and are available in a wide variety of sizes.

#### Sample Tubing and Intake Strainer Installation

Clean intake and pump tubing should be installed using "clean techniques", as described in *Section 5, 10* and *Appendix F*, so as not to contaminate the tubing. The tubing should remain double bagged until the time of installation. The clean tubing should have both ends covered with clean, non-metallic, non-contaminating material (i.e. polyethylene caps or clean latex gloves) until the intake strainer is installed at one end and a clean sample bottle is installed at the other end. The tubing ends should be covered with clean latex material to keep the tubing clean during installation, which typically involves feeding the tubing through protective conduit and pipelines. During installation, the intake and pump tubing should only be handled wearing clean, powder-free, nitrile gloves. During installation, the tubing ends should not touch any item not known to be clean. It is important to avoid kinking of the intake tubing during installation, as this will hinder sample collection.

Once the tubing has been installed, the intake strainer should be installed using "clean techniques". During installation, the strainer should only be handled while wearing clean, powder-free nitrile, gloves. The strainer should be attached to the end of the intake tubing and secured at the designated sampling location. All hardware in the immediate area of the intake strainer (hardware used to secure the suction tubing and intake strainer) must be stainless steel, polyethylene, or Teflon to minimize the possibility of contamination.

#### Sample Tubing Inspection

The sample intake and pump tubing should be inspected prior to each monitoring event. Intake tubing should be checked for kinks or cracks, and for adequate connection to pump tubing. Tubing clamps or cable ties, which secure the intake tubing to the pump tubing, should also be inspected prior to each monitoring event. Pump tubing should be checked for wear after each monitoring event. Pump tubing will show wear from the peristaltic sample pump. The frequency of pump tubing replacement will vary from site to site, depending primarily on head height, intake tubing length (may range from 3 to 99 feet), and temperature. If pump tubing wear is detected, the tubing should be replaced with new clean tubing prior to the next monitoring event.

Project:	Stormwater Quality Monitoring Program		
Station:	Neal Road	Event:	Event #1
Date:	12/15/98	Time:	0300
Sample Type:	X Grab Composite	Bottle:	<u>1</u> of <u>2</u>
Preservative:	Sodium thiosulfate	Collected By:	John Smith
Analysis:	Fecal Coliform		

Figure 9-3. Typical Sample Bottle Label Example

Computer labeling programs can save a great deal of time in generating bottle labels. The sites and analytical constituent information can be entered in the computer program for each monitoring program in advance, and printed as needed prior to each monitoring event.

# ► FIELD EQUIPMENT PREPARATION

Prior to the first targeted storm event of each monitoring season, and immediately after each monitored event, the field crews will inventory, restock, replace, clean, calibrate, maintain, and test field equipment as needed. Field equipment is inventoried using a comprehensive checklist of all required field equipment (tools, sample bottles, flashlights, extra batteries, safety equipment, first-aid kit, cellular telephone, etc.). Field equipment should be kept in one location which is used as a staging area to simplify field crew mobilization. An example field equipment checklist is provided as Figure 9-4.

The following equipment preparation procedures should be conducted prior to each targeted storm:

- ✓ Inspect the pump tubing and replace if necessary,
- ✓ Inspect intake tubing condition and connections,
- ✓ Inspect desiccant cartridges in sampler and flow meter,
- ✓ Inspect rain gauge for blockage,
- ✓ Check all electrical connections,
- ✓ Ensure that batteries are adequately charged and positioned,
- ✓ Insert sample bottles into sampler (see *Section 10* and *Appendix F* for detailed bottle changing procedures),
- ✓ Place ice around sample bottle in non-refrigerated samplers,
- $\checkmark\,$  Reset automatic sampler, and

✓ Calibrate any portable analytical meters that will be used to make field measurements.

At a minimum, the frequency and nature of equipment maintenance for all field equipment should be consistent with the manufacturer's recommendations.

# ► MOBILIZATION OF FIELD CREWS

When a potentially acceptable storm is approaching (i.e., the storm meets the storm selection criteria, as discussed previously in this section), the field crew and analytical laboratory will be alerted by the monitoring task manager. Field crews will be given notice to mobilize when precipitation is imminent or has begun.

Establishing deployment criteria is recommended for the purpose of standardizing field crew mobilization. A flow chart that combines project specific storm selection criteria (i.e., antecedent conditions, storm size, storm duration) with storm action levels (Figure 9-1) can be useful for standardizing field crew mobilization for a given project. An example of a typical deployment flow chart is presented as Figure 9-5.

When first alerted, field crew members should consult their sampling plan and check monitoring equipment and supplies to ensure they are ready to conduct monitoring. Once given the go-ahead by the monitoring task manager, the field crew members will travel to their assigned locations and conduct final preparations for monitoring. Upon arrival at the monitoring site, the field crew should:

- ✓ Check battery levels,
- ✓ Check tubing and all connections,
- ✓ Install clean composite bottle(s) and remove lid(s) as necessary (see Section 10 and Appendix F for detailed bottle changing procedures),
- $\checkmark$  Add ice to sampler if necessary, and
- ✓ Program automatic flow meter and sampler.

Storm Kit Equipment List		Storm Mobilization Equipment List	
	First aid kit		Storm kit
	Keys (to gates and to enclosures)		Waterproof log books/log sheets
	Flashlights (2) - hand held and head mounted		Paper towels
	Maps		D.I. water squirt bottles
	Large flat screwdriver		Ice scoop
	Small flat screwdriver		Chain of custody forms
	Umbrella - large size		Appropriate number of composite bottles with mesh carriers and buckets
	Alkaline batteries for flashlights		Appropriate number of grab sample bottles
	Write in the rain" pens (2), waterproof markers-fine point (2)		Bottle labels
	Spare sample bottle labels		Coolers and ice
	Desiccant (for samplers and flow meters)		Grab pole, rope and duct tape
	Diagonal cutters		Laboratory-provided blank water
	Electrical tape		Cellular phone
	Cable ties (assorted sizes)		Personal extra change of clothes
	Utility knife		Lighting
	Zip-lock baggies (assorted sizes)		Personal rain gear
	Gloves - powder free nitrile		Hard hats and orange safety vests
	Duct tape		Traffic cones/signs
	Rubber bands		Sampling Plan/Health and Safety Plan

# Figure 9-4. Field Equipment Checklist



Note: Specific precipitation levels, etc. presented in this example are project specific.

# Figure 9-5. Example Deployment Flow Chart

If sample collection is conducted at a station without a refrigerated sampler, or if grab samples are required, the field crew will need to obtain ice (for sample preservation) on the way to the sampling station. Composite sample bottles are required to be kept in a refrigerated sampler, or surrounded with ice during sample collection. Ice for grab samples should be kept in ice chests where full grab sample bottles will be placed. Keeping ice in double zip-lock bags facilitates clean easy ice handling. Refreezable ice packets are generally not recommended because they are susceptible to leakage.

# > PROGRAMMING OF AUTOMATED EQUIPMENT

The steps involved will vary depending upon the software programs selected; however, the following steps illustrate what is generally involved when a stormwater monitoring event is anticipated:

- ✓ System counters (precipitation, runoff volume, sample count, etc.) are reset to zero,
- ✓ The mode is switched from non-monitoring mode to monitoring mode. This typically will increase data collection frequency and allow the system to begin sample collection once thresholds are met,
- ✓ Thresholds are set for all sampling locations to allow stations to enter sample collection mode. Thresholds can include the minimum precipitation amount, flow depth, or flow volume required to initiate the sample collection routine,
- ✓ For flow paced sampling, the flow volume per sample (i.e., the flow volume that passes between each composite aliquot collected) is set based on the expected amount of rainfall and runoff,
- ✓ The system should have "start sampling" and "stop sampling" options that can be selected when appropriate (e.g., at the beginning of a storm, during bottle changes, at the end of a storm),
- ✓ When all samples have been collected, the sampling event is terminated and software is switched back to the non-monitoring mode, and
- ✓ Data are downloaded from the data logger to a personal computer (PC) immediately following the storm event.

Most automated monitoring stations typically contain continuous flow measurement devices and data logging software. To collect flow-proportioned composite samples, the flow measurement device must be programmed to send a pulse to the sampler each time a specified flow volume has passed the flow sensor. The sampler, in turn, is programmed to collect a sample each time it receives a pulse. Therefore, each time the programmed flow volume per sample has passed the sampling location, a composite sample aliquot is collected.

To insure the collection of representative samples, automatic samplers should be programmed to perform a full back purge cycle between each sample aliquot collected. When multiple sample containers are used, samplers should be programmed to perform a full back purge cycle prior to the filling of each individual container. Purging the sample intake tube prior to the collection of each aliquot or individual container sample helps to keep the line clear. Debris at sample tubing intake may cause flow restriction, which reduces velocities within the intake tube. When intake tube velocities are reduced heavy particulates may not adequately represented in the sample. Additionally, reduced velocities may result in sampler aliquot volume calibration problems, or increased pump tubing wear. Automatic samplers may also be programmed to perform rinse cycles after the back purge cycle and prior to the collection of sample aliquots. However, for stations that have a high sampling head height or a long intake tubing length, rinse cycles are not advised because of additional wear on the pump tubing. Worn or split pump tubing will result in missed sample aliquots.

The flow volume per sample (the amount of flow that passes the sampling point between each aliquot collected) must be programmed into the flow meter in proportion to the predicted rainfall amount for each storm event, to set the sample pacing so as to fill the composite bottle(s) at an appropriate rate.

Calculation of the flow volume per sample is performed using the predicted rainfall amount (quantity of precipitation forecast, or QPF), the known or estimated drainage area, and the composite runoff coefficient for the area monitored to calculate the expected runoff flow volume for the storm event. Flow volume per sample can be determined using the formula presented below:

$$V_r (acre - feet) = QPF (inches) * \frac{1(ft)}{12(inches)} * A(acres) * C$$
$$V_r (cf) = V_r (acre - feet) * \frac{43,560(cf)}{1(acre - foot)}$$
$$V_s (cf) = \frac{V_r (cf)}{CSA}$$

QPF = quantity of precipitation forecast

A = drainage area

C = runoff coefficient

 $V_r$  = total runoff volume for forecast storm (calculated)

CSA = number of composite sample aliquots required for complete composite

 $V_s$  = flow volume per sample

## Example:

0.33 inches = QPF (quantity of precipitation forecast)
150 acres = A (drainage area)
0.6 = C (runoff coefficient)
20 = CSA (number of composite sample aliquots required for complete composite)

$$V_r (acre - feet) = 0.33 (inches) * \frac{1(ft.)}{12(inches)} * 150 (acres) * 0.6 = 2.475 (acre - feet)$$
$$V_r (cf) = 2.475 (acre - feet) * \frac{43,560(cf)}{1(acre - foot)} = 107,811(cf)$$
$$V_s (cf) = \frac{107,811(cf)}{20(aliquots)} = 5,391(cf / aliquot)$$

The flow volume per sample is calculated so that if the predicted precipitation is delivered by the targeted storm, the automatic sampler will collect enough samples to conduct the set of analytical measurements specified in the sampling plan, plus any required QA/QC analyses. If the required analytical volume is less than the capacity of the composite bottle(s), then a margin of safety can be provided by setting the sample pacing to collect the needed composite sample volume at some fraction (typically one half to three quarters) of the predicted rainfall amount. This is done by using an appropriate fraction of the QPF in calculating the flow volume per sample. If less rainfall is received than predicted by the QPF, this may allow for collection of an adequate composite volume during the storm event.

The automatic sampler is programmed to collect a specific number of composite sample aliquots of specific volume before halting the sampling program, so as to fill the composite bottle(s) to the desired level, without overfilling (see *Section 10*, Table 10-1 for minimum acceptable number of aliquots required to meet monitoring event representativeness requirements).

The number of composite sample aliquots ("CSA") used in the above equation may be determined based on total composite sample volume required and the desired sample aliquot volume. An adequate number of sample aliquots should be collected to produce a composite sample that is representative of the runoff for the entire sampling event. The total sample volume required for the laboratory to conduct all planned analyses, including QA/QC analyses, may be divided by the selected sample aliquot volume to produce the required sample aliquot number.

Because automated samplers tend to exhibit slight variations in aliquot volume delivered, it is recommended that the sample aliquot volume be a minimum of 200 milliliters. A typical sample aliquot volume is 500 milliliters. If 10 liters of composite sample volume is required to perform the specified laboratory analyses, then 20 composite sample aliquot volume of 500 milliliters. The input value used for the CSA variable would therefore be 20.

The runoff coefficient for a specific drainage area is defined as the fraction of total precipitation volume delivered to the area that ends up as stormwater runoff at the point of discharge. Runoff coefficients may be available from Caltrans District personnel or from local flood control or public works agencies. If not, the coefficient may initially be estimated to correspond to the fraction of impervious area within the drainage area. Or, if the pervious and impervious areas are known in a drainage area, it may be useful to modify the above equation to include separate runoff coefficients for pervious and impervious areas as follows:

$$V_r \left( acre - ft \right) = QPF \left( in. \right) * \frac{1(ft)}{12(in.)} * \left[ \left( A_{pervious} * C_{pervious} \right) + \left( A_{impervious} * C_{impervious} \right) \right]$$

The number of sample aliquots per composite and the sample aliquot volume, once determined, are typically programmed into the automatic sampler. The CSA is used in the above equation throughout the monitoring season.

The flow volume per sample calculation normally requires input of only one variable for each storm event (the QPF), once the equations are set up for each monitoring station.

If a storm delivers more precipitation than expected, composite bottle replacement may be required to capture runoff from the entire storm event. *Section 10* describes composite bottle replacement procedures.

If less precipitation is received than predicted, the resulting composite sample volume may be insufficient to conduct all planned analyses. It may be possible to salvage a successful monitoring event in such cases by reducing the planned QA/QC analyses, or by eliminating some analytes while retaining others. Metals, for example, are key stormwater constituents which require relatively small sample volumes for analysis. To make well-informed decisions, the monitoring task manager should be familiar with the minimum sample volumes necessary to conduct each type of analysis (see *Section 12*), as well as the overall goals and priorities of the monitoring program.

After one or two storms have been monitored, the flow volume per sample formula for each monitoring site should be checked for accuracy. To check the formula, the actual measured storm event precipitation should be plugged into the formula as the quantity of precipitation forecast (QPF). Then the resulting calculated total runoff volume ( $V_r$ ) should be compared to the actual measured storm event total runoff volume. The formula may then be modified, if necessary, by modifying the runoff coefficient (C). Keep in mind that there will naturally be some variability from storm to storm at any given site because of non-uniform rainfall intensity throughout the drainage area and soil saturation (for sites with a significant pervious surfaces).

# **SECTION 10** SAMPLE COLLECTION

Equipment and bottles used in the collection of samples to be analyzed for trace metals, trace organics, nutrients, and bacteriological constituents must be handled with great care to minimize the possibility of contamination. The ease with which stormwater samples can be unintentionally contaminated cannot be overemphasized. The following procedures include sample handling techniques that maximize the ability of sampling personnel to collect samples reliably and with minimal sample contamination.

The following are basic sample collection and handling elements required during stormwater monitoring:

- ► Personnel Safety
- ► Sampling Equipment and Bottles
- ► Clean Sampling Techniques
- ► Grab Sample Collection
- ► Composite Sample Collection
- ► Flow Monitoring
- ► Composite Bottle Changing
- ► Sample Representativeness Evaluation
- Multi-Bottle Compositing, and Composite Sample Splitting
- ► Sample Preservation
- ► Sample Filtration
- ► Sample Delivery/Chain of Custody

These elements are described below to provide sample collection and handling guidance for field personnel engaged in stormwater monitoring.

# ► PERSONNEL SAFETY

Before stormwater samples are collected, personnel must ensure the safety of such activities at each sampling location. As mentioned in *Section 3*, personnel safety should be considered when selecting monitoring sites. Adherence to the following recommendations will minimize risks to sampling personnel:

✓ At no time during storm conditions or when significant flows are present should sampling personnel enter a manhole or standpipe.
- ✓ Two-person field crews should be available for all field work to be conducted under adverse weather conditions, or whenever there are risks to personal safety.
- ✓ Use of automated samplers can eliminate many of the hazards associated with manual sample collection, as personnel are not required to be at the site for composite sampling.
- ✓ Personnel must be trained regarding appropriate traffic control measures. If appropriate, a traffic control plan should be developed for each site and included in the sampling plan and analysis plan prior to conducting sampling events.
- ✓ Only personnel properly trained and equipped for confined space entry may enter a space designated as "confined".
- ✓ When appropriate, an encroachment permit must be filed with the district.

#### ► SAMPLING EQUIPMENT AND BOTTLES

Generally, field personnel are responsible for collecting composite samples and/or grab samples. It is important to use the appropriate sample bottles and equipment for each parameter to be measured (see *Section 5*). Improper bottles and equipment can introduce contaminants and cause other errors which can invalidate the data. For example, chemicals may leach from the bottle into the sample, or waterborne constituents may cling to sampling equipment or to the sides of the bottle.

As general guidelines, all sampling equipment and sample bottles used for trace metals determination must be nonmetallic and free from any material that may contain metals. Only high density plastic or Teflon containers should be used for metals analytical sample storage bottles. All sampling equipment and sample bottles used for trace organics determination must be glass or Teflon. Borosilicate glass is acceptable for composite sample containers because it is considered an acceptable compromise for collection of stormwater samples that will be analyzed for both metals and organic compounds. Nutrients and most "conventional" parameters may be sampled using plastic or glass bottles.

The size and type of sample composite sample bottle(s) will depend on the analyses selected. Composite sample bottle(s) must hold sufficient volume to provide the analytical laboratory with enough sample volume to conduct all of the selected analyses, plus any QA/QC requirements. The bottle type also must be appropriate for all planned analytical constituents. When composite samples are to be analyzed for both metals and organics, for example, the composite bottle(s) must be borosilicate glass or Teflon.

Certain constituents cannot be analyzed from composite bottles, and must be collected as "grab" samples. For example, all sampling equipment and sample bottles used for bacteriological determinations must be sterile. This normally requires field collection of

the sample directly into the sterile "bacti" bottle, or use of a sterile Teflon bailer as an intermediate device. Samples for oil & grease or petroleum hydrocarbons analysis must be collected directly into the glass bottle that the laboratory will use for analyses, because the use of any intermediate container or tubing may result in some loss of the material being analyzed. Other analytes, such as ammonia and volatile organic compounds, must be collected as grab samples because of the risk of losing the constituent(s) to volatilization in a composite bottle. *Section 12* provides information on the appropriate bottles for specific constituents and analytical methods.

Before samples are collected, all sampling equipment and bottles are cleaned in a laboratory using appropriate detergent, mineral acids, and deionized water as described in *Appendix E*. The laboratory is responsible for generating acceptable equipment blanks and sample bottle blanks to demonstrate that the sampling equipment and bottles are free from trace metals and organics contamination before they are delivered to field sampling personnel. An acceptable blank is one that is free from contamination below the minimum level specified in the referenced analytical method. *Section 11* provides additional information on collection of equipment blanks.

After cleaning, sample bottles and laboratory-cleaned sampling equipment are handled only while wearing clean, powder-free nitrile gloves. All laboratory-cleaned sampling equipment and metals analysis storage bottles are double bagged in clean zip-lock plastic bags for storage or shipment. Clean bottles are stored in a clean area with lids properly secured.

Immediately prior to the filling of grab sample bottles, the bottle labels should be checked, and date and time added using a waterproof pen (see *Section 9* for sample bottle labeling). Attempting to label grab sample bottles after sample collection may be difficult because of wet labels.

#### ► CLEAN SAMPLING TECHNIQUES

Caltrans stormwater monitoring projects employ "clean" sampling techniques to minimize potential sources of sample contamination, particularly from trace pollutants. Experience has shown that when clean sampling techniques are used, detected concentrations of constituents tend to be lower. Clean sample collection techniques that should be followed during the collection of stormwater samples are described below. More extensive clean sampling techniques may be required under certain conditions, such as monitoring to assess receiving water impacts. See *Appendix F* for a detailed description of more extensive clean sampling techniques.

Extreme care must be taken during all sampling operations to minimize exposure of the samples to human, atmospheric, and other potential sources of contamination. Care must be taken to avoid contamination whenever handling composite bottles, lids, sample

tubing, and strainers. Whenever possible, grab samples should be collected by opening, filling and capping the sample bottle while submerged, to minimize exposure to airborne particulate matter. Additionally, whenever possible, samples should be collected upstream and upwind of sampling personnel to minimize introduction of contaminants.

To reduce potential contamination, sample collection personnel must adhere to the following rules while collecting storm water samples:

- ✓ No smoking
- ✓ Never sample near a running vehicle. Do not park vehicles in immediate sample collection area (even non-running vehicles)
- ✓ Always wear clean, powder-free nitrile gloves when handling composite bottles, lids, sterile grab sample bottles, tubing or strainers.
- ✓ Never touch the inside surface of a sample bottle or lid, even with gloved hands.
- ✓ Never touch the exposed end of a sampling tube.
- ✓ Never allow the inner surface of a sample bottle, lid, or sampling tube to be contacted by any material other than the sample water.
- ✓ Never allow any object or material to fall into or contact the collected sample water.
- ✓ Avoid allowing rain water to drip from rain gear or other surfaces into sample bottles.
- ✓ Do not eat or drink during sample collection.
- $\checkmark$  Do not breathe, sneeze or cough in the direction of an open sample bottle.

#### ► GRAB SAMPLE COLLECTION

Grab sampling is required for monitoring parameters that transform rapidly, require special preservation, or adhere to bottles. For example, samples to be analyzed for oil and grease, petroleum hydrocarbons, ammonia, volatile organics, and bacteria are required to be collected as grab samples only.

When grab samples are only collected once during a storm event, it is important to collect those samples under flow conditions that will provide the most representative sample possible. In an attempt to provide grab samples representative of an entire storm event, to the greatest extent possible, grab samples should be collected during event peak flow. However, peak flow conditions are typically difficult to determine during a monitoring event. Therefore, grab samples should be collected using best professional judgement, during the estimated midpoint of a monitoring event, under moderate (not low) runoff flow conditions. Grab samples are typically collected by direct submersion of each individual sample container. It is acceptable for some grab samples to be collected using intermediate containers and for some (e.g., oil and grease, petroleum hydrocarbons) it is not. When use of an intermediate container is appropriate (e.g., for ammonia, volatile organics), grab samples should be collected by holding an appropriate container (bucket, bailer, sample bottle, etc.) under the outfall of a discharge pipe, at the lip of an inlet grate, or by dipping a container downstream of a discharge with the container opening facing upstream, depending on monitoring site configuration. The sample is then poured immediately into the appropriate grab sample bottle. Samples for bacteriological analysis must be collected in sterile containers. Sterile Teflon bailers are available for this purpose; otherwise, the sample must be collected directly into the sterile "bacti" bottle. Clean techniques must be used when collecting bacteriological samples.

When collecting samples for oil and grease or petroleum hydrocarbon analysis, the sample must be collected directly into the bottle that will be used in the laboratory, because petroleum-derived compounds may adhere to the sample container (the laboratory analyzes samples for these constituents by extracting the entire contents of the sample bottle). Because oil and grease and other petroleum hydrocarbons tend to float, these grab samples should be collected so as to include sampling of the air/water interface.

#### ► COMPOSITE SAMPLE COLLECTION

A composite sample is made up of multiple sub-samples (aliquots) collected over some spatial or temporal range. Stormwater runoff composite samples are typically collected from a single location during a period of runoff. Such temporal composites can be collected on a time-proportioned basis (equal sample aliquot volumes collected at equal time intervals) or flow-proportioned basis (samples are collected either on an even-time-interval basis, with sample aliquot size proportional to instantaneous measured flow rate, or on an even-flow-interval basis, with a set aliquot volume collected at passage of each equal, pre-set flow volume). Because stormwater runoff flow typically varies throughout a storm event, flow-proportioned composite sampling is the standard composite sample collection method (see *Section 5* for guidance on the selection of sample collection methods and equipment). Composite samples are typically collected using automated sampling equipment, but can also be collected manually.

Flow-proportional sampling requires determination of several key parameters:

- ✓ Storm event quantity of precipitation forecast ("QPF"; from forecast information),
- ✓ Expected runoff volume (determined from the QPF and watershed characteristics),
- ✓ Expected storm duration (for even-time-interval methods),

- ✓ Minimum required composite sample volume for all planned analyses (see *Section 12* obtained from contract laboratories),
- ✓ Minimum acceptable number of sample aliquots (see Table 10-1 and discussion below), and
- ✓ Sample aliquot size (varies proportional to measured flow throughout the event for even-time-interval methods, and set to a single volume per event for even-flow-volume methods)

See *Section 9* for a detailed discussion of how to program flow-proportioning parameters.

#### Automated Composite Sample Collection

Automated flow-proportional composite sampling is typically done on an even flowvolume-per-sample basis; that is, a sample aliquot of equal size is collected every time a pre-selected flow volume passes by the flow sensor. The flow volume per sample is determined based on the quantity of precipitation forecast (QPF) and the required composite sample volume (see *Section 9*), with consideration of the minimum required number of sample aliquots for the storm event (see Table 10-1 and discussion below). At automated monitoring stations, sample collection will begin automatically once the programmed thresholds (triggers) have been met (see *Section 9* for automated station programming and preparation).

Automatic sampling stations should be checked periodically throughout a monitored storm event to make sure the station is function properly. If the composite sample bottle (or bottles, in the case of multi-bottle composite sample collection) fills more rapidly than expected, field personnel should be mobilized to conduct a bottle change (bottle changing is described in detail later in this section). If the composite sample collection period exceeds 24 hours, the composite sample bottle(s) should be replaced with (a) clean bottle(s) at or prior to the end of each 24-hour period. For constituents with short holding times, such as 48 hours or less, composite sample volume should be removed from each 24-hour composite for analysis as necessary to comply with holding time requirements.

After the storm event has ended, field personnel are mobilized to retrieve the full composite sample bottle(s) and interrogate sampling equipment. Sample splitting and delivery to the laboratory are described later in this section.

#### Manual Composite Sample Collection

Manual composite sample collection may be conducted at monitoring sites that are not equipped with automatic equipment. Manual composite sample collection can be done on a time-proportional compositing basis, but it is generally possible to perform flowproportional manual composite sampling using one of the techniques described below. Manual flow-proportional composite sample collection is conducted using the same basic principals as automatic composite sample collection. Typical manual composite sample collection methods are described below.

#### <u>Flow-Proportioning – Even Time Interval Basis</u>

This method involves the collection of sample aliquots at a specified time interval, with the aliquot volume set proportional to the measured flow rate. The sampling time interval is set to ensure collection of the minimum number of aliquots required for adequate storm representativeness (see discussion later in this section), based on the expected (forecast) storm duration. Sample aliquot volumes are set to ensure collection of the required composite volume over the course of the expected storm event, based on the storm QPF and the expected runoff volume (this requires some advance knowledge of rainfall/runoff relationships in the monitored watersheds; see Section 9 for a discussion of the relevant calculations). At each sampling interval the flow rate is measured (using one of the methods described in *Section 5*), and a sample aliquot volume is collected in proportion to the measured flow prior to collection. Sample aliquots are composited to generate a single event composite sample. This method requires advance preparation of a table showing the aliquot volumes to collect for a range of expected flow rates, over a range of possible storm event QPFs. In this table, the aliquot volumes are set so as to ensure collection of the full composite sample volume required to perform all planned analyses for a given QPF.

#### <u>Flow-Proportioning – Even Flow Volume Basis</u>

This method is typically more difficult than the even-time-interval method described above, because it requires keeping a cumulative running tally of flow volume, which is not normally practical without automated flow monitoring equipment. As with automated composite sampling, flow volume per sample is calculated using the target storm QPF, with consideration for the required composite sample volume and the minimum acceptable number of aliquots, as discussed below (also see Section 9 for guidance on calculating flow volume per sample). Once runoff begins, flow rate is measured periodically (using one of the methods described in Section 5), and sample aliquots are collected each time the pre-determined flow volume per sample has passed the sampling location. Instantaneous and cumulative flow volume can be calculated by inputting the flow data into a portable computer and applying Manning's equation or other appropriate flow equation in the field (see Section 5). Sample aliquots are collected either as manual grabs or by using a peristaltic pump equipped with appropriate tubing and strainer (see Section 5). Each sample aliquot collected is of equal volume (typically 250-1000 mL) and combined into a single composite bottle immediately after collection.

#### Flow-Proportioning Using Precipitation Measurement

For sites where flow measurement is extremely difficult (such as sites where sheet flow is prevalent) flow-proportional samples may be collected using precipitation measurements

as an analog for runoff flow. The assumption is made that runoff volume is directly proportional to event precipitation. So, instead of calculating flow volume per sample, rainfall depth per sample is determined. A sample aliquot is then collected each time the selected precipitation increment has fallen. Therefore, an on-site rain gauge is required for precipitation measurement (see *Section 5*). To determine appropriate rainfall amount per sample for a target storm event, simply divide the event QPF by the number of sample aliquots required (the number of sample aliquots required is determined by the total composite volume required and the desired sample aliquot volume, subject to the minimum numbers of sample aliquots per event, as discussed later in this section).

#### Multi-Bottle Flow-Proportioning

This method of flow-proportional sample collection involves collecting sample aliquots of equal volume at a predetermined time interval throughout the storm event, and recording the flow rate at the time of sample collection. The sample aliquot volume is set in advance, based on the expected duration of the storm and the required composite sample volume, and considering the minimum number of aliquots required (see discussion below). After the storm has ended, a portion of each aliquot is composited to generate a single flow-proportional composite sample. The volume used from each aliquot is directly proportional to the flow rate that was recorded during the aliquot collection. Using this method, the highest flow rate measured during the event is used to determine the scale for the sample volumes added to the composite from each aliquot. For example, for each aliquot, the flow reading taken during sample collection can be divided by the highest flow reading during the event, and that percentage is the percentage of the aliquot that is added to the composite. This method requires more bottles and bottle handling that the other methods described above.

#### ► FLOW MONITORING

Flow measurements are necessary to produce flow-weighted composite samples. Flow measurements should be performed utilizing one of the methods discussed in *Section 5*. If using manual methods, field crews should begin taking flow measurements as soon as possible after stormwater runoff begins (concurrently with sample collection). If automated sampling equipment is utilized, the equipment must be programmed to obtain the desired composite sample volume every time a specified flow volume is recorded, based on the predicted rainfall amount. Methods for programming automatic samplers and flow meters are described in *Section 9*.

If multiple bottles are used for composite sample collection, or if more flow volume is delivered than expected during a storm event, requiring one or more composite bottle changes per monitoring station, it is necessary to combine the multiple composite bottles to produce a single flow-weighted composite sample. To do this, it is normally necessary to use the collected flow data to determine the amount of sample from each composite bottle to be used to form the final composite. When using automated equipment, the field crew will typically download data from the flow meter or datalogger, to determine the flow volume represented by each composite bottle. The sample volumes to be used out of each composite bottle can be calculated by the monitoring crew, and the completed calculations faxed or otherwise delivered to the lab. The methods for calculating appropriate sample volumes from each bottle are described below under "Multi-bottle Compositing".

#### ► COMPOSITE BOTTLE CHANGING

If an automated monitoring station is used for the collection of composite stormwater samples and a composite bottle change is required, composite bottle changing is conducted using the steps listed below. When conducting monitoring to determine compliance with water quality objectives or to determine receiving water impacts, the more extensive composite-changing protocols presented in *Appendix F* should be followed.

- 1. The automated sampling equipment is placed in pause mode prior to the initiation of a composite bottle change. This action is accomplished in the field or by remote monitoring personnel if the monitoring station is equipped with telemetry.
- 2. Field personnel should wear clean, powder-free nitrile gloves and practice clean sampling techniques (see above).
- 3. To change a sample bottle, the end of the pump tubing is removed from the full sample bottle, the full bottle is removed from the sampler and capped with a clean lid, a clean bottle is placed in the sampler, and the tubing end is placed into the clean bottle. Do not allow the exposed tubing end to contact hands or any other surface.
- 4. After the sample bottle has been changed, the sampler is closed and the sampler keypad is used to place the sampler in sampling mode. The field supervisor or remote operation personnel are notified as soon as the bottle change is complete.
- 5. The sampling team fills out the appropriate information on the label of the collected composite sample bottle(s).
- 6. The collected composite bottle(s) are surrounded with ice, and secured inside the vehicle for transport.
- 7. Verify that the automatic sampler has been placed in sampling mode, if sampling is to continue. Visually inspect the components for possible damage or clogging, to be sure the system will be ready to continue sampling, or is ready to sample the next storm.

#### ► SAMPLE REPRESENTATIVENESS EVALUATION

Immediately following sample collection, composite sample representativeness must be evaluated to determine whether samples meet the project minimum acceptable storm capture parameters (number of aliquots and percent storm capture). Samples not meeting these criteria are generally not analyzed. However, the Caltrans Project Coordinator should be consulted to make the decision whether or not to analyze the samples.

Percent storm capture is the percentage of the total event flow that passes the sampling station during which sample collection occurred (i.e., the portion of the runoff represented by the composite sample). This is calculated simply by dividing the flow volume that passed the sampling station during sample collection by the total flow that passed the sampling station during the entire monitoring event.

The minimum acceptable number of sample aliquots and minimum acceptable storm percent capture depend on the total event precipitation, as shown in Table 10-1. The specified minimum number of sample aliquots is intended to ensure adequate representativeness of the composite sample throughout the monitoring event. Higher numbers of sample aliquots are desirable whenever possible, subject to the practical limitations of sample collection.

Total Event Precipitation	Minimum Acceptable Number of Aliquots	Percent Capture Requirement
0-0.25"	6	85
0.25-0.5"	8	80
0.5-1"	10	80
>1"	12	75

 Table 10-1. Monitoring Event Representativeness Requirements

#### ► MULTI-BOTTLE COMPOSITING AND COMPOSITE SAMPLE SPLITTING

Procedures for combining multiple composite samples to produce a single sample, and the procedures for splitting samples into multiple sample bottles are described below.

#### Multi-bottle Compositing

When multiple composite sample bottles are filled at a single site during a single storm monitoring event, the sample bottles are typically composited together to produce a single composite sample representing the entire monitoring event.

In order to combine multiple sample bottles to generate a single representative composite sample, the following two items must be determined: 1) the percent of the sampling event flow represented by each individual sample bottle, and 2) which of the sample bottle(s), if

any, will limit the compositing of samples. Because individual sample bottles will likely contain different volumes, one bottle will likely dictate the total available sample volume. Individual sample bottles may contain different sample volumes for several reasons. For example, the number of aliquots may differ in each bottle if runoff ceased before triggering all programmed sample aliquots. Composite bottle volumes may also differ slightly from unequal aliquot volumes, sometimes caused by pump tubing blockages or wear.

Each individual composite sample corresponds to the volume of stormwater runoff that passed the sampling point during the collection of that composite sample. Composite samples are mixed in relative proportion, according to the percentage of total volume that passed the sampling point during the storm event. Therefore, to properly combine multiple composite samples, the following must be known:

- ✓ Individual volumes in each sample bottle
- ✓ Total runoff flow volume that passed during the collection of each individual sample bottle, and the total runoff volume for the monitoring event

Multiple composite samples should be combined using the following formulas:

 $V_n/V_t = P_n$ , and

 $S_t * P_n = S_n$ 

Where;

 $V_n$  = the volume of flow that passed during the collection of bottle n

- $V_t$  = the total volume of flow that passed during the sample collection event
- $P_n$  = the percent of the total sampled flow represented by bottle n
- $S_t$  = the total volume of sample collected in all bottles combined
- $S_n$  = the volume of sample contributed from bottle n toward the combined composite sample

The following is an example of how multiple composite samples are combined:

Bottle #1 = 8 L of sample 10,000 CF passed during the collection of bottle #1 (V<sub>1</sub>) Bottle #2 = 10 L of sample 15,000 CF passed during the collection of bottle #2 (V<sub>2</sub>) Total volume passed during sample collection = 25,000 CF (V<sub>t</sub>) Total composite sample collected = 18 liters (S<sub>t</sub>)

Bottle #1, % of total flow = % of composite = 10,000/25,000 = 40% (P<sub>1</sub>)

Bottle #2, % of total flow = % of composite = 15,000/25,000 = 60% (P<sub>2</sub>)

Therefore, the single composite is made up of 40% from bottle #1 and 60% from bottle #2. Since 40% of the total sample volume collected (18 liters) equals 7.2 (S<sub>1</sub>) liters and 60% equals 10.8 liters (S<sub>2</sub>), it is apparent that bottle #2 has limited sample volume for compositing. Therefore, the entire 10 liters of sample from bottle #2 is mixed with the following volume (X) from bottle #1.

10 liters/60% = X liters/40%

X (the volume required from bottle #1) = 6.7 liters

The two composite samples are combined by adding 6.7 liters from sample bottle #1 to 10 liters from sample bottle #2.

Each sample bottle must be well-mixed prior to pouring off into another composite bottle. The sample is mixed thoroughly by shaking or otherwise agitating the composite bottle to prevent sediment from remaining on the bottom of the bottle. Throughout the sample compositing procedures, clean, powder-free nitrile gloves are used for bottle and lid handling. This process can be done by analytical laboratory personnel, or by field sampling personnel in a clean, dry setting.

#### **Composite Sample Splitting**

Composite samples collected in a single composite sample bottle are poured, by the analytical laboratory or the sampling team, into individual sample bottles for analysis (to limit contamination it is recommended that splitting be conducted by the laboratory). When a composite sample duplicate is required, the sampling team will be required to split the composite sample into two composite bottles to generate a subsampling duplicate. As with field duplicate samples (replicate samples collected simultaneously in the field), subsampling duplicates (replicate samples generated from a single composite sample bottle) should be submitted to the analytical laboratory "blind" (labeled using a pseudonym site name). Below are three examples of composite sample splitting procedures:

#### Sample Splitting Example #1

- 1. Sample storage bottles are labeled for specific analyses.
- 2. Clean powder-free nitrile gloves are worn for handling of bottles and lids.
- 3. Any item that will contact the sample is cleaned using protocols presented in *Appendix E*.
- 4. Clean, specially blown borosilicate glass vessels are used to composite and mix samples (vessels have glass spigots with Teflon stopcocks).

5. Clean, Teflon coated magnetic mixing bars are used to stir the sample continuously before and during sample pour-off into individual sample containers (check with analytical laboratories for recommended sample volumes for individual constituents).

#### Sample Splitting Example #2

- 1. Sample storage bottles are labeled for specific analyses.
- 2. Clean powder-free nitrile gloves are worn for handling of bottles and lids.
- 3. Any item that will contact the sample is cleaned using protocols presented in *Appendix E*.
- 4. During continuous manual composite sample agitation, sample is drawn from the bottle into individual sample containers using a portable peristaltic pump and clean tubing (check with analytical laboratories for recommended sample volumes for individual constituents).

#### Sample Splitting Example #3 (USGS method)

- 1. Sample storage bottles are labeled for specific analyses.
- 2. Clean powder-free nitrile gloves are worn for handling of bottles and lids.
- 3. The composite sample is mixed thoroughly by carefully shaking composite bottle, with lid in place, until the sample is well mixed.
- 4. Immediately after mixing, the composite sample is poured into a clean Teflon funnel/splitter with clean tubes leading to individual sample containers (check with analytical laboratories for recommended sample volumes for individual constituents).

#### Sample Splitting Example #4

- 1. Sample storage bottles are labeled for specific analyses.
- 2. Clean powder-free nitrile gloves are worn for handling of bottles and lids.
- 3. The composite sample is mixed thoroughly by carefully shaking composite bottle, with lid in place, until the sample is well mixed. For large composite bottles, the use of a swiveling mechanical bottle holding/mixing/pouring device is recommended.
- 4. Immediately after mixing, the composite sample is poured off into individual sample storage bottles and the bottles are capped (check with analytical laboratories for recommended sample volumes for individual constituents). Step 3 is repeated immediately prior to filling each sample storage bottle.

#### ► SAMPLE PRESERVATION

All samples are kept on ice or refrigerated to  $4^{\circ}$  Celsius from the time of sample collection until delivery to the analytical laboratory. Refrigerated automatic samplers are ideal for keeping composite samples cool during sample collection. Where refrigerated automatic samplers are not used, composite samples are kept on ice from the time sampling is initiated. Ice is checked regularly to insure that the sample is kept cool. Grab samples are placed in an ice chest with ice immediately following collection.

In addition to keeping stormwater samples cool it is also important to minimize the exposure of the samples to direct sunlight, as sunlight may cause biochemical transformation of the sample, resulting in unreliable analytical results. Therefore, all samples are covered or placed in an ice chest with a closed lid immediately following collection or removal from the automatic sampler enclosure.

Samples to be analyzed for certain constituents (depending on analytical laboratory method) require that special chemical preservatives be added to the sample, either in the bottle prior to collection, or after collection, at the analytical laboratory. The later is recommended, as this may reduce logistical problems in the field (e.g., loss of preservative during sample collection). *Section 12* discusses appropriate sample preservation in more detail.

#### ► SAMPLE FILTRATION

Sample filtration is required when collecting samples for dissolved metals determinations. Because of the added possibilities for field contamination during stormwater sampling, it is recommended that filtration for dissolved metals be performed in the laboratory. The laboratory provides a controlled environment for sample filtration. However, it is important that the samples be transported promptly to the laboratory, and filtered immediately upon receipt. The laboratory must be notified in advance that samples will be delivered and that immediate filtration for dissolved metals analysis will be required.

If filtration can be done in the field in such a way as to minimize the possibility of contamination, and if the analytical laboratory is a substantial distance from the monitoring location, field filtration may be preferable because filtration could be more immediate. Filtration should be performed using methods and filtration apparatus specified in EPA Method 1669 (USEPA, 1996). Field filtration is a viable alternative particularly when a mobile laboratory or a walk-in shelter is available for the filtering activity.

#### ► SAMPLE DELIVERY/CHAIN OF CUSTODY

All samples must be kept on ice, or refrigerated, from the time of onset of sample collection to the time of receipt by laboratory personnel. If samples are being shipped to the laboratory, place sample bottles inside coolers with ice, ensure that the sample bottles are well packaged (i.e., with bubble wrap, foam, etc.), and secure cooler lids with packaging tape.

It is imperative that all samples be delivered to the analytical laboratory and analysis begun within the maximum holding times specified by laboratory analytical methods (see *Section 12*). For example, if the fecal coliform test is required, analysis must be started within 8 hours of sample collection (the analytical method allows 6 hours for transportation to the laboratory and 2 hours to begin analysis). Similarly, soluble reactive phosphorus or nitrite analyses must be performed within 48 hours after sample collection. To minimize the risk of exceeding the holding times, samples must be transferred to the analytical laboratory as soon as possible after sampling. The field crew must in such cases coordinate activities with the analytical laboratory to ensure that holding times can be met.

Chain-of-custody (COC) forms must be filled out by the sampling team for all samples submitted to the analytical laboratory. The purpose of COC forms is to keep a record of the transfer of sample custody, and requested analyses. Sample date, sample location, and analysis requested are noted on each COC, including specification of lab quality control requirements (e.g., laboratory duplicate samples and matrix spike/matrix spike duplicate (MS/MSD) samples; see *Section 11*). Any special instructions for the laboratory should also be noted, for example, requesting that filtration for dissolved metals be conducted immediately. Customized project specific COCs, that include standard information (e.g. contact information, constituents and methods, and special notes) are recommended. Copies of COC forms are kept with field notes in a field log book. COC forms should be checked to be sure all analyses specified by the sampling plan are included. Review of the COC forms immediately following a storm event gives the data reviewer a chance to review the field crews' requests and then to notify the laboratory of additional analyses or necessary clarification. An example of a customized COC form is presented as Figure 10-1.

## SWAMP SOP for Collecting BMI Samples and Associated Physical and Chemical Data

SOP for Biological Assessment





SWAMP Bioassessment Procedures 2007

Standard Operating Procedures for Collecting Benthic Macroinvertebrate Samples and Associated Physical and Chemical Data for Ambient Bioassessments in California

February 2007





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The protocols described here represent the contributions of a wide range of researchers and field crews. Most of the physical habitat methods are close modifications of those used in the U.S. Environmental Protection Agency's (EPA's) Environmental Monitoring and Assessment Program (EMAP) and developed by EPA's Office of Research and Development (ORD, Peck et al. 2004). The benthic macroinvertebrate collection methods are based on EMAP methods (EPA's targeted riffle methods were derived in turn from methods developed at Utah State University; Hawkins et al. 2003).

The current version of these protocols was established by Peter Ode (Department of Fish and Game's (DFG's) Aquatic Bioassessment Laboratory (ABL)) and David Herbst (UC Santa Barbara's Sierra Nevada Aquatic Research Laboratory) with significant contributions from staff at the ABL (Jim Harrington, Shawn McBride, Doug Post, Andy Rehn, and Jennifer York), the Surface Water Ambient Monitoring Program (SWAMP) Quality Assurance (QA) Team, Thomas Suk and other members of the SWAMP bioassessment committee (Mary Adams, Lilian Busse, Matt Cover, Robert Holmes, Sean Mundell, and Jay Rowan) and three external reviewers: Chuck Hawkins, Dave Peck, and Phil Kaufmann.

Ode, P.R.. 2007. Standard operating procedures for collecting macroinvertebrate samples and associated physical and chemical data for ambient bioassessments in California. California State Water Resources Control Board Surface Water Ambient Monitoring Program (SWAMP) Bioassessment SOP 001.



# swamp **SG**

#### SWAMP GUIDANCE FOR MACROINVERTEBRATE FIELD PROTOCOLS FOR WADEABLE STREAMS

**Background:** The SWAMP Bioassessment Committee met in December, 2004, and agreed that the SWAMP Quality Assurance Management Plan (QAMP) should be amended to provide greater consistency in bioassessment sampling protocols for wadeable streams. The Committee's recommendations were reviewed and accepted by the full SWAMP Roundtable<sup>1</sup> in February, 2005 (some of the key considerations are contained in Appendix A).

The current guidance for macroinvertebrate sampling under the SWAMP program is as follows:

- 1. For ambient bioassessment monitoring of wadeable streams in California, two methods are to be used at sites with riffle habitats (i.e., one "multihabitat" sample, and one sample that targets the "richest" habitat):
  - For sites with sufficient riffle habitat, the two samples shall be: (1) the reachwide benthos (RWB) method (also known as "multihabitat" sampling.); and (2) the targeted-riffle composite (TRC) method.
  - For low-gradient sites that do not have sufficient riffle habitat, the RWB method is the standard method, but we also recommend the option of collecting a sample with (2) the "Margin-Center-Margin" (MCM) method until ongoing methods comparisons are completed (see Appendix A).
  - Notes: (1) The protocols for each method are provided in this document; (2) Other appropriate method(s) will be allowed if the specific monitoring objectives require use of alternative method(s). (See Item #2, below.); (3) The protocol recommendations specified above will be reevaluated as results become available from ongoing methods comparison studies. (See Appendix A for more information.)
- 2. The SWAMP QAMP allows flexibility in sampling methods so that the most appropriate method(s) may be used to address hypothesis tests and project-specific objectives that differ from program objectives. Such situations may include, but are not necessarily limited to, special studies (e.g., evaluation of point source discharges, above/below comparisons where statistical replication is needed), stressor identification investigations, and long-term monitoring projects where consistent data comparability is desired and an alternative method is needed to achieve that comparability. In addition, in some rare cases where funding limitations would make it cost-prohibitive to complete a project in compliance with the protocols listed in #1, above, the project proponent may request to complete laboratory analysis of only one sample, and "archive" one of the macroinvertebrate samples (i.e., the RWB sample in streams with riffles) to reduce lab costs. Deviations from the protocols specified in #1 above may be granted by the SWAMP Bioassessment Coordinator or the full SWAMP Roundtable.

The SWAMP Roundtable is the coordinating entity for the program. Participants include staff from the State and Regional Water Boards, USEPA, the Department of Fish and Game, the Marine Pollution Studies Laboratory, Moss Landing Marine Laboratories, contractors, and other interested entities.



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## SECTION INTRODUCTION

This document describes two standard procedures (TRC and RWB) for sampling benthic macroinvertebrate (BMI) assemblages for ambient bioassessments. This document also contains procedures for measuring instream and riparian habitats and ambient water chemistry associated with BMI samples. These sampling methods replace previous bioassessment protocols referred to as the California Stream Bioassessment Procedure (CSBP, Harrington 1995, 1999, 2002).

These procedures can produce quantitative and repeatable measures of a stream's physical/habitat condition and benthic invertebrate assemblages, but they require field training and implementation of QA measures throughout the field season.

The sampling layout described here provides a framework for systematically collecting a variety of physical, chemical, and biological data. The biological sampling methods are designed to nest within the overall framework for assessing the biotic, physical, and chemical condition of a reach. The layout used in these procedures and most of the physical habitat methods are close modifications of those used in EPA's EMAP and developed by EPA's ORD (Peck et al. 2004). Data collected using this methodology are generally directly comparable to equivalent EMAP data, except for the difference in reach length. Other exceptions are noted in the text.

The following steps are presented in an order suggested for efficient data collection. The specific order of collection for the physical parameters may be modified according to preferences of field crews, with the caveat that care must always be taken to not disturb the substrates within the streambed before BMI samples are collected.

#### **PHYSICAL HABITAT METHODS**

The physical habitat scoring methods described here can be used as a stand-alone evaluation or used in conjunction with a bioassessment sampling event. However, measurements of instream and riparian habitat and ambient water chemistry are essential to interpretation of bioassessment data and should always accompany bioassessment samples. This information can be used to classify stream reaches, associate physical and chemical condition with biotic condition, and explain patterns in the biological data.





Because bioassessment samples can be collected to answer a variety of questions, this document describes the component measures of instream and riparian habitat as independent modules. Although individual modules can be added or subtracted from the procedure to reflect specific project objectives, a standard set of modules will normally accompany bioassessment samples. This document describes two standard groupings of modules that represent two different levels of intensity for characterizing the chemical and physical habitat data (Table 1). The BASIC physical habitat characterization represents a minimum amount of physical and chemical data that should be taken along with any ambient BMI sample, the FULL physical habitat characterization represents the suite of data that should be collected with most professional level bioassessment samples (e.g., SWAMP regional monitoring programs). In addition to these data, we also briefly introduce additional data modules (e.g., excess sediment, periphyton) that can be collected as supplements to the full set (OPTIONAL). Table 1 lists the physical and chemical variables that should be measured under the different levels.

**Note:** SWAMP intends to develop guidance for selecting appropriate physical habitat modules to the intended uses of data. Until this guidance is available, users of these protocols should consult with representatives of the Regional Water Quality Control Boards (Regional Boards) or the SWAMP Bioassessment Coordinator when selecting modules.

#### FIELD CREW SIZE AND TIME ESTIMATES

These methods are designed to be completed by either two or three (or more) person field crews. A very experienced field crew can expect to complete the full suite of physical habitat measurements and the two BMI sampling protocols in approximately two hours. Less experienced crews will probably take closer to three or four hours to complete the work depending on the complexity of the reach. Note that this estimate includes only time at the site, not travel time between sites.

#### **Equipment and Supplies**

Recommended equipment and supplies are listed in Table 2.

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Survey Task	Parameter(s)	Basic	Full	Option	Comments
REACH DELINEATION and WATER QUALITY Conducted before entering	Layout reach and mark transects, record GPS coordinates	х	Х		Use 150-m reach length if wetted width ≤10 m; Use 250-m reach length if wetted width > 10 m
stream to sample Bivils or conduct any habitat surveys]	Temperature, pH, specific conductance, DO, alkalinity	х	Х		Multi-meter (e.g., YSI, Hydrolab, VWR Symphony)
	Turbidity, Silica			х	Use test kit or meter
	Notable field conditions	Х	Х		Recent rainfall, fire events, domin local landuse
CROSS-SECTIONAL	Wetted width	Х	Х		Stadia rod is useful here
TRANSECTS	Flow habitat delineation	Х	Х		Record proportion of habitat class each inter-transect zone
BASIC Measurements at main 11 transects only	Depth and Pebble Count + CPOM		Х		5 -point substrate size, depth and C records at all 21 transects
FULL Measurements at 11 main transects (A, B, C, D, E, F, G, H, I, J, K) or 21	Cobble embeddedness		Х		All cobble-sized particles in pebl count. Supplement with "random v if needed for 25
transects (11 main plus 10 inter-transects) for substrate size classes only	Slope (%)	See reach scale	х		Average slope calculated from 10 transect to transect slope measurements. Use autolevel for slopes ≤ 1%; clinometer is 0 for steeper gradients
	Sinuosity		Х		Record compass readings betwe transect centers
	Canopy cover	х	Х		Four densiometer readings at cer of channel (facing L bank R ban Upstream +Downstream)
	Riparian Vegetation		Х		Record % or categories
	Instream Habitat		Х		
	Human Influence		Х		
	Bank Stability	Х	Х		Eroding / Vulnerable / Stable
	Bankfull Dimensions		Х		
	Excess Sediment Transect Measures (optional)				
	Bankfull width and height, bank angles			Х	
	Large woody debris counts			х	Tallies of woody debris in several size classes



Survey Task	Parameter(s)	Basic	Full	Option	Comments
DISCHARGE TRANSECT	Discharge measurements		Х		Velocity-Area Method or Neutrally Buoyant Object Method
REACH SCALE MEASURE- MENTS:	EPA-RBP visual scoring of habitat features	*		х	*Used for citizen monitoring and comparison with legacy data
	Selected RBP visuals:		х		Channel alteration, sediment deposition, epifaunal substrate (redundant if doing EPA-RBP scoring)
	Slope (%, not degrees)	х	See transect scale		Single measurement for entire reach only for BASIC. Use autolevel for slopes ≤ 1%, clinometer is OK for higher gradients
	Photo documentation	Х	Х		Upstream (A, F, K) Downstream (F)
OTHER OPTIONAL COMPONENTS					
FOOD RESOURCE QUANTIFICATION	Periphyton (3 replicates)			х	Qualitative characterization of diatom growth and filamentous algal growth, quantification of biomass (AFDM, chl-a)
	CPOM & FPOM (3 replicates)			Х	CPOM field measure of wet mass >1 mm particles, FPOM as 0.25 – 1 mm fraction (AFDM in lab)

I I I I I I I I I I I I I I I I I I I	s	
Physical Habitat	BMI Collection	General/ Ambient Chemistry
<ul> <li>GPS receiver</li> <li>topographic maps</li> <li>measuring tape (150-m)</li> <li>small metric ruler or gravelometer for substrate measurements</li> <li>digital watch, random number table or ten-sided die</li> <li>stadia rod</li> <li>clinometer</li> <li>autolevel (for slopes &lt; 1%)</li> <li>handlevel (optional)</li> <li>current velocity meter</li> <li>stopwatch for velocity measurements</li> <li>convex spherical densitometer</li> <li>flags/ flagging tape</li> <li>rangefinder</li> </ul>	<ul> <li>D-frame kick net (fitted with 500-µ mesh bag)</li> <li>standard # 35 sieve (500-µ mesh)</li> <li>wide-mouth 500-mL or 1000 mL plastic jars</li> <li>white sorting pan (enamel or plastic)</li> <li>95% Et0H</li> <li>fine tipped forceps or soft forceps</li> <li>waterproof paper and tape for attaching labels</li> <li>10-20-L plastic bucket for sample elutriation</li> <li>preprinted waterproof labels (e.g., Rite-in-the-Rain<sup>™</sup>)</li> <li>disposable gloves/ elbow length insulated gloves</li> </ul>	<ul> <li>sampling SOP (this document)</li> <li>hip or chest waders, or wading boots/shoes</li> <li>field forms printed on waterproof paper (e.g., Rite-in-the-Rain™)</li> <li>clip board and pencils</li> <li>digital camera</li> <li>centigrade thermometer</li> <li>pH meter</li> <li>D0 meter</li> <li>conductivity meter</li> <li>field alkalinity meter</li> <li>water chemistry containers</li> <li>calibration standards</li> <li>spare batteries for meters</li> <li>first aid kit</li> </ul>

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# REACH DELINEATION AND WATER QUALITY 2

The systematic positioning of transects is essential to collecting representative samples and to the objective quantification of physical habitat measures. The standard sampling layout consists of a 150-m reach (length measured along the bank) divided into 11 equidistant transects that are arranged perpendicular to the direction of flow (Figure 1, Figure 2). Ten additional transects (designated "inter-transects") located between the main transects give a total of 21 transects per reach. Main transects are designated A through K while inter-transects are designated by their nearest upstream and downstream transects (e.g., AB, BC, etc.). In extreme circumstances, reach length can be shorter than 150 m (e.g., if upstream and downstream barriers preclude a 150-m reach), but this should be avoided whenever possible. If the actual reach length is other than 150 m or 250 m this should be noted and explained on the field forms.

**Note 1:** The standard reach length differs from that used in the EMAP design, in which reach length was defined as 40x stream width, with a minimum reach length of 150 m. The EMAP reach length approach is used to ensure that enough habitat is sampled to support accurate fish assemblage estimates and relatively precise characterization of channel characteristics (e.g., residual pool volumes and woody debris estimates, which that are critical for relative bed stability estimates). Programs wishing to sample fish assemblages or produce relative bed stability estimates should strongly consider adopting the EMAP guidance for setting reach length.

**Note 2:** Streams > 10 m wetted width should use a reach length of 250 m. Some very large streams (i.e., > 20-m wetted width) may not be adequately represented even by a 250-m reach. In these cases, field crews should define a reach length that is representative of the larger stream segment being studied (i.e., attempt to include two to three meander cycles, or four to six riffle-pool sequences when possible).

*Note 3:* When the exact reach location is not restricted by the sampling design, attempt to position reaches upstream of bridges to avoid this influence.

**Step 1.** Upon arrival at the sampling site, fill out the reach documentation section of the field forms (site and project identification, stream and watershed name, crew members, and date/time). If known at the time of sampling, record the Site Code following SWAMP site code formats. Determine the geographic coordinates of the downstream end of the reach (preferably in decimal degrees to at least four decimal places) with a GPS receiver and record the datum setting of the unit (preferably NAD83/ WGS84).

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Figure 1. Reach layout geometry for physical habitat and biological sampling showing positions of 11 main transects (A – K) and the 10 supplemental inter-transects (AB- JK). The area highlighted in the figure is expanded in Figure 2. Note: reach length = 150 m for streams  $\leq 10\text{-m}$  average wetted width, and reach length = 250 m for streams > 10-m average wetted width.

**Step 2.** Once a site has been identified, make an initial survey of the reach from the stream banks (being sure to not disturb the instream habitat). If TRC samples will be collected, identify all riffle habitats suitable for sampling (see Section IIIa for suitable habitat types) and note their positions so that a subset can be identified for sampling.

**Step 3.** Determine if the average wetted width is greater or less than 10 m. If the average wetted width  $\leq$  10 m, use a 150-m reach length. If the average wetted width > 10 m, use a 250-m reach length.



**Step 4.** Starting at one end of the reach, establish the position of the 11 main transects (labeled A-K from downstream to upstream) by measuring 15 m (25 m for streams > 10 m wetted width) along the bank from the previous transect. The 10 inter-transects should be established equidistant from the adjacent main transects (i.e., 7.5 m from main transects for 150-m reaches, 12.5 m for 250-m reaches). Since the data collection will start at the downstream end, is often easiest to establish transects starting from the upstream end. For easy setup and breakdown, mark the main transects with easily removable markers (e.g., large washers tied with strips of flagging, surveyor's flags).

**Note 1:** While it is usually easiest to establish transect positions from the banks (this also reduces disturbance to the stream channel), this can result in uneven spacing of transects in complex stream reaches. To avoid this, estimate transect positions by projecting from the mid-channel to the banks.

#### Note 2: Flagging of a single bank is recommended to reduce mistakes caused by missed markers.

**Step 5.** Measure and record common ambient water chemistry measurements (pH, DO, specific conductance, alkalinity, water temperature) at the downstream end of the reach (near same location as the GPS coordinates were taken). These are typically taken with a handheld water quality meter (e.g., YSI, Hydrolab), but field test kits (e.g., Hach) can provide acceptable information if they are properly calibrated. For appropriate calibration methods and calibration frequency, consult the current SWAMP QAMP (Appendix F), or follow manufacturer's guidelines.

**Note 1:** If characteristics of the site prohibit downstream entry, measurements may be taken at other points in the reach. In all cases, ambient chemistry measurements should be taken at the beginning of the reach survey.

*Note 2:* Alkalinity test kits may not perform well in low ionic strength waters. Programs should consider collecting lab samples for these sites (see SWAMP QAMP for guidance on collecting water chemistry samples).

**Step 6.** Take a minimum of four (4) photographs of the reach at the following locations: a) Transect A facing upstream, b) Transect F facing upstream, c) Transect F facing downstream, and d) Transect K facing downstream. It may also be desirable to take a photograph at Transect A facing downstream and Transect K facing upstream to document conditions immediately adjacent to the reach. Digital photographs should be used when possible. Record the image numbers on the front page of the field form.

**Note 1:** When possible, photograph names should follow SWAMP coding conventions ("StationCode\_yyyy\_ mm\_dd\_uniquecode"). The unique code should include one of the following codes to indicate direction: RB (right bank), LB (left bank), BB (both banks), US (upstream), DS (downstream). SWAMP suggests using unique codes created by the camera to facilitate file organization. Example: 603WQLB02\_2004\_03\_20\_ RBDS1253.





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**Step 7.** Record the dominant land use and land cover in the area surrounding the reach (evaluate land cover within 50 m of either side of the stream reach).

**Step 8.** At the bottom of the form, record evidence of recent flooding, fire, or other disturbances that might influence bioassessment samples. Especially note if flow conditions have been affected by recent rainfall, which can cause significant under-sampling of BMI diversity (see note in the following section). If you are unaware of recent fire or rainfall events, select the "no" option on the forms.





### SECTION 3 COLLECT BENTHIC MACROINVERTEBRATES 3 MULTIPLE HABITAT AND TARGETED RIFFLE PROTOCOLS

**Note 1:** BMI samples intended for ambient bioassessments are generally collected when streams are at or near base flow (i.e., not influenced by surface runoff) as sudden flow increases can dramatically alter local community composition.

**Note 2:** Guidance for choosing among TRC sampling, RWB sampling or both will be provided in a separate document (see Appendix A for current guidance for sampling under SWAMP).

Once the reach transects have been laid out, the biological samples (BMIs and algae if included) should be collected before any other physical habitat measures so that substrates are not disturbed prior to sampling. Both TRC and RWB methods use 500-µ mesh D-frame nets (see list of BMI sampling equipment in Table 2). The two samples can be collected at the same time by carrying two D-nets and compositing the material from the two samples in their respective nets. If a two person field crew is responsible for both the physical habitat data and benthic invertebrate samples, it is generally best to collect the benthos at each transect, then immediately record the physical habitat data before moving to the next transect. Obviously, this requires especially careful handling of the D-nets during the course of sampling to avoid loss or contamination of the samples. It can be helpful to clearly label the two D nets as RWB and TRC. Larger field crews may choose to split the sampling between biological team and a physical habitat team and have the biological team go through the reach first. The positions of the TRC and RWB subsampling locations are illustrated in Figure 2.

#### SECTION III A. TARGETED RIFFLE COMPOSITE PROCEDURE

The TRC method is designed for sampling BMIs in wadeable streams that contain fast-water (riffle/run) habitats and is not appropriate for waterbodies without fastwater habitats. The RWB protocol should be used in these situations. Riffles are often used for collecting biological samples (e.g., the old CSBP methods) because they often have the highest BMI diversity in wadeable streams. This method expands the definition to include other fast water habitats, however care should be taken when attempting to apply this method in low gradient streams.

*Note:* Since all streams (even low gradient streams) have variation in flow habitats within the channel, this guidance should not be interpreted as including areas within low gradient streams that are only marginally faster than the surrounding habitats. The RWB protocol should be applied in these situations.





The TRC was developed by the Western Center for Monitoring and Assessment of Freshwater Ecosystems (www.cnr.usu.edu/wmc) in Logan, Utah (Hawkins et al. 2003) and slightly modified by the EPA program (Peck et al. 2004). The TRC has been widely used in California (US Forest Service (USFS), the EMAP Western Pilot, and the California Monitoring and Assessment Program (CMAP)), and in the interest of methodological consistency between state and federal water resource agencies, has been adopted as the standard riffle protocol for bioassessment in California. The version described here is the EMAP modification, which distributes the sampling effort throughout the reach.

#### Sampling Locations – Acceptable Habitat Types

Riffles are the preferred habitat for TRC sampling, but other fast water habitats are acceptable for sampling if riffles are sparse. Common flow-defined habitat types are listed in Table 3 in decreasing order of energy. Most streams contain some or all of the following fast water habitat types: 1) cascades/falls, 2) rapids, 3) riffles, 4) runs. All of these are acceptable for TRC sampling if riffles are not available.

*Note:* Because the common habitat types are arranged on a continuum between high to low energy environments, the categories grade into each other continuously and are not discrete. Thus, determination of habitat types requires somewhat subjective decision-making.

Table 3. Com	mon habitat types in stream channels, arranged in decreasing order of energy			
Flow Habitat Type	Description			
Cascades	Short, high gradient drop in stream bed elevation often accompanied by boulders and considerable turbulence			
Falls	High gradient drop in elevation of the stream bed associated with an abrupt change in the bedrock			
Rapids	Sections of stream with swiftly flowing water and considerable surface turbulence. Rapids tend to have larger substrate sizes than riffles			
Riffles	Shallow sections where the water flows over coarse stream bed particles that create mild to moderate surface turbulence; (< 0.5 m deep, > 0.3 m/s)			
Step-Runs	A series of runs that are separated by short riffles or flow obstructions that cause discontinuous breaks in slope			
Runs	Long, relatively straight, low-gradient sections without flow obstructions. The stream bed is typically even and the water flows faster than it does in a pool; (> 0.5 m deep, > 0.3 m/s)			
Glides	A section of stream with little or no turbulence, but faster velocity than pools; (< 0.5 m deep, < 0.3 m/s)			
Pools	A reach of stream that is characterized by deep, low-velocity water and a smooth surface ; (> 0.5 m deep, < 0.3 m/s)			





Figure 2. Section of the standard reach expanded from Figure 1 showing the appropriate positions for collecting benthic macroinvertebrate samples, instream and riparian habitat measurements and flow habitat proportion measurements.

#### **Sampling Locations – Selecting Habitat Units**

A TRC sample is a composite of eight individual kick samples of 1 ft<sup>2</sup> (0.09 m<sup>2</sup>) of substrate each. During your initial layout of the reach, take a mental note of the number and position of the main riffles in a reach (and other fast water habitats if needed). Randomly distribute the eight sub-samples among the fast water habitats in the reach, giving preference to riffles where possible. Unless you are sampling in small streams, try to avoid very small riffle units (i.e., <5 ft<sup>2</sup>). If fewer than eight riffles are present in a reach, more than one sample may be taken from a single riffle, especially if the riffles are large.

#### **Sampling Procedure**

Begin sampling at the downstream end of the reach at the first randomly selected riffle and work your way upstream.



**TRC-Step 1.** Determine net placement within each habitat unit by generating a pair of random numbers between 0 and 9. Examples of convenient random number generators include the hundredths place on the stopwatch feature of a digital watch, a 10 sided die and a random number chart. The first number in each pair (multiplied by 10) represents the percent upstream along the habitat unit's length. The second number in each pair represents the percent of the riffle width from right bank. For example, if the two generated random numbers are 4 and 7, you will walk upstream 40% of the distance of the riffle and then go 70% of the distance across the riffle (see Figure 3). This position is the center of the 1 ft<sup>2</sup> (0.09 m<sup>2</sup>) sampling quadrat for that riffle. If you are unable to sample this location because it is too deep or it is occupied by a large boulder, select a new pair of random numbers and pick a new spot.

**TRC-Step 2.** Position a 500-µ D-net (with the net opening perpendicular to the flow and facing upstream) quickly and securely on the stream bottom to eliminate gaps under the frame. Avoid, and if necessary remove, large rocks that prevent the sampler from seating properly on the stream bottom.

**TRC-Step 3.** Holding the net in position on the substrate, visually define a square quadrat that is one net width wide and one net width long upstream of the net opening. Since D-nets are 12 inches wide, the area within this quadrat is 1ft<sup>2</sup> (0.09 m<sup>2</sup>). Restrict your sampling to within that area. If desired, a wire frame of the correct dimensions can be placed in front of the net to help delineate the quadrat to be sampled, but it is often sufficient to use the net dimensions to keep the sampling area consistent.

**TRC-Step 4.** Working backward from the upstream edge of the sampling plot, check the quadrat for heavy organisms such as mussels, snails, and stone-cased caddisflies. Remove these organisms from the substrate by hand and place them into the net. Carefully pick up and rub stones directly in front of the net to remove attached animals. Remove and clean all of the rocks larger than a golf ball (~3 cm) within your sampling quadrat such that all the organisms attached to them are washed downstream into your net. Set these rocks outside your sampling quadrat after you have cleaned them. If the substrate is consolidated or comprised of large, heavy rocks, use your feet to kick and dislodge the substrate to displace BMIs into the net. If you cannot remove a rock from the stream bottom, rub it (concentrating on cracks or indentations) thereby loosening any attached insects. As you are disturbing the plot, let the water current carry all loosened material into the net.

*Note 1:* Brushes are sometimes used in other bioassessment protocols to help loosen organisms, but in the interest of standardizing collections, do not use a brush when following this protocol.

*Note 2:* In sandy-bottomed streams, kicking within run habitats can quickly fill the sampling net with sand. In these situations, follow the standard procedures but use care to disturb the substrate gently and avoid kicking.

**TRC-Step 5.** Once the coarser substrates have been removed from the quadrat, dig your fingers through the remaining underlying material to a depth of about 10 cm (this material is often comprised of gravels and finer particles). Thoroughly manipulate the substrates in the quadrat.





*Note:* The sampler may spend as much time as necessary to inspect and clean larger substrates, but should take a standard time of 30 seconds to perform Step 5.

**TRC-Step 6.** Let the water run clear of any insects or organic material before carefully lifting the net. Immerse the net in the stream several times to remove fine sediments and to concentrate organisms at the end of the net, but be careful to avoid having any water or foreign material enter the mouth of the net during this operation.

**TRC-Step 7.** Move upstream to the next randomly selected habitat unit and repeat steps one through six, taking care to keep the net wet but uncontaminated by foreign material when moving the net from riffle to riffle. Sometimes, the net will become so full of material from the streambed that it is no longer effective at capturing BMIs. In these cases, the net should be emptied into sample jars as frequently as necessary, following guidelines described below in the "Preparation of BMI Sample Jars" section. Continue until you have sampled eight 1ft<sup>2</sup> (0.09 m<sup>2</sup>) of benthos.

TRC-Step 8. PROCEED to Section IIIc. Filling and Labeling BMI Sample Jars.



Figure 3. Example showing the method for selecting a subsampling position within a selected riffle under the TRC method. In this example, the random numbers 4 and 7 were selected

#### SECTION III B. REACHWIDE BENTHOS (MULTIHABITAT) PROCEDURE

The RWB procedure employs an objective method for selecting subsampling locations that is built upon the 11 transects used for physical habitat measurements. The RWB procedure can be used to sample any wadeable stream reach since it does not target specific habitats. Because sampling locations are defined by the transect layout, the position of individual sub-samples may fall in a variety of erosional or depositional habitats.

**Note:** Sampling locations should be displaced one meter downstream of the transects to avoid disturbing substrates for subsequent physical habitat assessments.

**RWB** -Step 1. The sampling position within each transect is alternated between the left, center and right positions along a transect (25%, 50% and 75% of wetted width, respectively) as you move upstream from transect to transect. Starting with the downstream transect (Transect



A), identify a point that is 25% of the stream width from the right bank (note that the right bank will be on your left as you face upstream). If you cannot collect a sample at the designated point because of deep water obstacles or unsafe conditions, relocate the point as close as possible to the designated position.

**Note:** A modification to this procedure is currently being investigated by SWAMP. This "margin-center-margin" (MCM) modification replaces the samples at 25% and 75% of wetted width with samples of the marginal habitats (including emergent and submergent vegetation).

**RWB** -Step 2. Place a 500-µ D-net in the water so the mouth of the net is perpendicular to and facing into the flow of the water. If there is sufficient current in the area at the sampling point to fully extend the net, use the normal D-net collection technique to collect the sub-sample (TRC-Step 3 through TRC-Step 6 above). If flow volume and velocity is not sufficient to use the normal collection technique, use the sampling procedure for "slack water" habitats (RWB-Step 3 through RWB-Step 7 below).

**RWB** -Step 3. Visually define a 1 ft<sup>2</sup> (0.09 m<sup>2</sup>) quadrat that is one net-width wide and one net-width long at the sampling point.

**RWB** -Step 4. Working backward from the upstream edge of the sampling plot, check the quadrat for heavy organisms such as mussels and snails. Remove these organisms from the substrate by hand and place them into the net. Carefully pick up and rub stones directly in front of the net to remove attached animals. Remove and clean all of the rocks larger than a golf ball within your sampling quadrat such that all the organisms attached to them are washed downstream into your net. Set these rocks outside your sampling quadrat after you have cleaned them. Large rocks that are less than halfway into the sampling area should be pushed aside. If the substrate is consolidated or comprised of large, heavy rocks, use your feet to kick and dislodge the substrate to displace BMIs into the net. If you cannot remove a rock from the stream bottom, rub it (concentrating on cracks or indentations) thereby loosening any attached insects.

**RWB** -Step 5. Vigorously kick the remaining finer substrate within the quadrat with your feet while dragging the net repeatedly through the disturbed area just above the bottom. Keep moving the net all the time so that the organisms trapped in the net will not escape. Continue kicking the substrate and moving the net for 30 seconds. For vegetation-choked sampling points, sweep the net through the vegetation within a 1ft<sup>2</sup> (0.09 m<sup>2</sup>) quadrat for 30 seconds.

*Note:* If flow volume is insufficient to use a D- net, spend 30 seconds hand picking a sample from 1ft<sup>2</sup> of substrate at the sampling point, then stir up the substrate with your gloved hands and use a sieve with 500-µ mesh size to collect the organisms from the water in the same way the net is used in larger pools.

**RWB** -Step 6. After 30 seconds, remove the net from the water with a quick upstream motion to wash the organisms to the bottom of the net.





RWB -Step 7. PROCEED to Section IIIc: Filling and Labeling BMI Sample Jars

#### SECTION III C. FILLING AND LABELING BENTHIC MACROINVERTEBRATE SAMPLE JARS

**Step 1.** Once all sub-samples (eight for TRC, 11 for RWB) have been collected, transfer benthos to a 500-mL or 1000-mL wide-mouth plastic sample jar using one of the following methods.

*Note:* Field elutriation should only be used by well-trained field crews who are proficient at removing all benthic organisms from the discarded inorganic material. Training in the recognition of aquatic invertebrates is highly recommended.

**Step 1a. Complete Transfer of all Sampled Material** – Invert the contents of the kick net into the sample jar. Perform this operation over a white enameled tray to avoid loss of any sampled material and make recovery of spilled organisms easier. If possible, remove the larger twigs and rocks by hand after carefully inspecting for clinging organisms, but be sure not to lose any organisms. Use forceps to remove any organisms clinging to the net and place these in the sample jar.

**Step 1b. Field Elutriation of Samples** – Empty the contents of the net into a large plastic bucket (10-20 L is sufficient). Use forceps to remove any organisms clinging to the net and place these in the bucket. Add stream water to the bucket and gently swirl the contents of the bucket in order to suspend the organic material (being certain to not introduce entrained organisms from the source water). Pour the organic matter from the bucket through a 500- $\mu$  sieve (or use the 500- $\mu$  net). Repeat this process until no additional material can be elutriated (i.e., only inorganic material is left in the bucket). If possible, remove the larger twigs and rocks by hand after carefully inspecting for clinging organisms, but be sure not to lose any organisms. Transfer all of the material in the sieve (invertebrates and organic matter) into the sample jar. Carefully inspect the gravel and debris remaining in the bottom of the bucket for any cased caddisflies, clams, snails, or other dense animals that might remain. Remove any remaining animals by hand and place them in the sample jar.

Date: Collector:	Time:	circle one;
County:	Jar #:	of
Site Name/ Code:		
Stream Name:		
Longitude: N	W	NAD83
Latitude: N	W	NAD27

Figure 4. Example date - locality label for all BMI samples.

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**Step 2.** Place a completed date/locality label (see Figure 4) on the inside of the jar (use pencil only as most "permanent" inks dissolve in ethanol) and completely fill with 95% ethanol. Place a second label on the outside of the jar. Note that the target concentration of ethanol is 70%, but 95% ethanol is used in the field to account for dilution from water in the sample. If organic and inorganic material does not accumulate in the net quickly, it may be possible to transfer all the material in the net into one jar. Otherwise, divide the material evenly among several jars


(being careful to clearly label them as part of a set). To ensure proper preservation of benthic macroinvertebrates it is critical that the ethanol is in contact with the BMIs in the sample jar. Never fill a jar more than 2/3 full with sampled material, and gently rotate jars that contain mostly mud or sand to ensure that the ethanol is well distributed. If jars will be stored for longer than a month prior to processing, jars should not contain more than 50% sample material.





# SECTION 4 MAIN CROSS-SECTIONAL TRANSECT MEASURES SECTION IVA. PHYSICAL MEASURES

The majority of physical habitat measurements in this protocol are made relative to the main cross-sectional transects (Figure 5). All the measures taken relative to each transect are recorded on forms specific to that transect. Start with the downstream transect (Transect A) and repeat steps 6-15 for all 11 main transects.

# Module A. Transect Dimensions: Wetted Width and Bankfull Dimensions

**Wetted Width** – The wetted channel is the zone that is inundated with water and the wetted width is the distance between the sides of the channel at the point where substrates are no longer surrounded by surface water. Measure the wetted stream width and record this in the box at the top of the transect form.

**Bankfull Width and Depth** – The bankfull channel is the zone of maximum water inundation in a normal flow year (one to two year flood events). Since most channel formation processes are believed to act when flows are within this zone (Mount 1995), bankfull dimensions provide a valuable indication of relative size of the waterbody.

**Note:** Bankfull dimensions are notoriously difficult to assess, even by experienced field crews (see Heil and Johnson 1995). It is often useful to discuss the interpretation of bankfull locations among the field crew members to reach a consensus. The USFS Stream Team provides a good set of instructional videos for improving consistency in accurate bankfull measurements (http://www.stream.fs.fed.us/publications/videos.html).

**Step 1.** Scout along the stream margins to identify the location of the bankfull margins on either bank by looking for evidence of annual or semi-annual flood events. Examples of useful evidence includes topographic, vegetative, or geologic cues (changes in bank slope, changes from annual to perennial vegetation, changes in the size distribution of surface sediments). While the position of drift material caught in vegetation may be a helpful aid, this can lead to very misleading measurements.

**Note:** The exact nature of this evidence varies widely across a range of stream types and geomorphic characteristics. It is helpful to investigate the entire reach when attempting to interpret this evidence because the true bankfull margin may be obscured at various points along the reach. Often the bankfull position is easier to interpret from one bank than the other; in these cases, it is easiest to infer the opposite bank position by projecting across the channel. Additionally, height can be verified by measuring the height from both edges of the wetted channel to the bankfull height (these heights should be equal).







Figure 5. Cross sectional diagram of a typical stream channel showing locations of substrate measurements, wetted and bankfull width measurements, and bank stability visual estimates.

**Step 2.** Stretch a tape from bank to bank at the bankfull position. Measure the width of the bankfull channel from bank to bank at bankfull height and perpendicular to the direction of stream flow.

**Step 3.** Measure bankfull height (the vertical distance between the water height of the water and the height of the bank, Figure 5) and record.

#### Module B. Transect Substrate Measurements

Particle size frequency distributions often provide valuable information about instream habitat conditions that affect BMI distributions. The Wolman pebble count technique (Wolman 1954) is a widely used and cost-effective method for estimating the particle size distribution and produces data that correlates with costly, but more quantitative bulk sediment samples. The method described here follows the EMAP protocol, which records sizes of 105 particles in a reach (five particles from each of 11 main transects and 10 inter-transects).

**Note:** The size cutoff for the finest particle sizes in the EMAP protocol (< 0.06 mm) differs from that used by the Sierra Nevada Aquatic Research Laboratory (SNARL) program (0.25 mm), although the narrative description for this cutoff is the same (the point at which fine particles rubbed between one's fingers no longer feel gritty).

Coarse particulate organic matter (CPOM, particles of decaying organic material such as leaves that are greater than 1.0 mm in diameter) is a general indicator of the amount of allochthonous organic matter available at a site, and its measurement can provide valuable information about the basis of the food web in a stream reach. The presence of CPOM associated with each particle is quantified at the same time that particles are measured for the pebble counts.





**Step 1.** Transect substrate measurements are taken at five equidistant points along each transect (Figure 5). Divide the wetted stream width by four to get the distance between the five points (Left Bank, Left Center, Center, Right Center and Right Bank) and use a measuring device to locate the positions of these points (a stadia rod is especially helpful here). Once the positions are identified, lower a graduated rod (e.g., a marked ski pole) though the water column perpendicular to both the flow and the transect to objectively select the particle located at the tip of the rod.

**Step 2.** Measure the depth from the water surface to the top of the particle with the graduated rod and record to the nearest cm.

**Step 3.** Record the presence or absence of CPOM > 1mm within 1 cm of the particle.

**Step 4.** If the particle is cobble-sized (64-250 mm), record the percent of the cobble that is embedded by fine particles ( < 2 mm) to the nearest 5% (see cobble embeddedness text below).

**Step 5.** Remove the particle from the streambed, then measure and record the length of its intermediate axis to the nearest mm (see Figure 6). Alternatively, assign the particle to one of the size classes listed in the bottom of the transect form. Particle sizes classes can be estimated visually or with a quantitative measuring device (e.g., pass/ no-pass template, "gravelometer"). Regardless of the method, all particles less than 0.06 mm should be recorded as fines, all particles between 0.06mm and 2.0 mm recorded as sand. Field crews may want to carry vials containing sediment particles with these size ranges until they are familiar with these particles.



Figure 6. Diagram of three major perpendicular axes of substrate particles. The intermediate axis is recorded for pebble counts.

## Module C. Cobble Embeddedness

The quantification of substrate embeddedness has long been a challenge to stream geomorphologists and ecologists (Klamt 1976, Kelley and Dettman 1980). It is generally agreed that the degree to which fine particles fill interstitial spaces has a significant impact on the ecology of benthic organisms and fish, but techniques for measuring this impact vary greatly (this is summarized well by Sylte and Fischenich 2002, http://stream.fs.fed.us/news/ streamnt/pdf/StreamOCT4.pdf ). Here we define embeddedness as the volume of cobble-sized particles (64-250 mm) that is buried by fine particles ( < 2.0 mm diameter).

**Note:** This method differs from the EMAP method for measuring embeddedness, which measures embeddedness of all particles larger than 2 mm.



Size Class Code	Size Class Description	Common Size Reference	Size Class Rang
RS	bedrock, smooth	larger than a car	>4 m
RR	bedrock, rough	larger than a car	> 4 m
ХВ	boulder, large	meter stick to car	1 - 4 m
SB	boulder, small	basketball to meter stick	25 cm - 1.0 m
СВ	cobble	tennis ball to basketball	64 - 250 mm
GC	gravel, coarse	marble to tennis ball	16 - 64 mm
GF	gravel, fine	ladybug to marble	2 – 16 mm
SA	sand	gritty to ladybug	0.06 – 2 mm
FN	fines	not gritty	< 0.06 mm
HP	hardpan (consolidated fines)		< 0.06 mm
WD	wood		
RC	concrete/ asphalt		
ОТ	other		

**Step 1.** Every time a cobble-sized particle is encountered during the pebble count, remove the cobble from the stream bed and visually estimate the percentage of the cobble's volume that has been buried by fine particles. Since visual estimates of volume and surface area are subject to large amounts of observer error, field crews should routinely calibrate their estimates with each other and with other field crews.

**Step 2.** In the spaces to the right of the pebble count data, record the embeddedness of all cobble-sized particles encountered during the pebble count.

# **Note:** The cobble embeddedness scores do not correspond with the specific particles in the pebble count cells to the left, but are merely a convenient place to record the data.

**Step 3.** If 25 cobbles are not encountered during the pebble count, supplement the cobbles by conducting a "random walk" through the reach. Starting at a random point in the reach, follow a transect from one bank to the other at a randomly chosen angle. Once at the other bank reverse the process with a new randomly chosen angle. Record embeddedness of cobble-sized particles in the cobble embeddedness boxes on the transect forms until you reach 25 cobbles. If 25 cobble-sized particles are not present in the entire reach, then record the values for cobbles that are present.





## Module D. Canopy Cover

This method uses the Strickler (1959) modification of a convex spherical densiometer to correct for overestimation of canopy density that occurs with unmodified readings. Read the densiometer by counting the number of line intersections that are obscured by overhanging vegetation (see Figure 7). Taping off the lower left and right portions of the mirror emphasizes overhead vegetation over foreground vegetation (the main source of bias in canopy density measurements). All densiometer readings should be taken with the bubble leveled and 0.3 m (1 ft) above the water surface.

**Step 1.** Using a modified convex spherical densitometer, take and record four 17-point readings all taken from the center of each transect: a) facing upstream, b) facing downstream, c) facing the left bank, d) facing the right bank.

**Note:** This method deviates slightly from that of EMAP (in which two additional readings are taken at the left and right wetted edges to increase representation of bank vegetation).



Figure 7. Representation of the mirrored surface of a convex spherical densiometer showing the position for taping the mirror and the intersection points used for the densiometer reading. The score for the hypothetical condition in (b) is 10 covered intersection points out of 17 possible. Note the position of the bubble level in (b) when the densiometer is leveled.



## Module E. Gradient and Sinuosity

The gradient of a stream reach is one of the major stream classification variables, giving an indication of potential water velocities and stream power, which are in turn important controls on aquatic habitat and sediment transport within the reach. The gradient (slope) of a stream reach is often strongly correlated with many BMI metrics and other physical habitat measures and is therefore very useful when interpreting BMI data.

The "full" physical habitat method uses 10 transect to transect measurements to calculate the average slope through a reach. Although this is a little more time intensive than the reach-scale transect measures used in the "basic" protocol, it results in more precise slope determination and the ability to quantify slope variability within a reach. Sinuosity (calculated as the ratio of the length of the flow path between the ends of the reach and the straight line distance between the ends of the reach, Kaufmann et al. 1999) is measured at the same time as slope. These two measurements work best with two people, one taking the readings at the upstream transect ("backsighting") and the other holding a stadia rod at the downstream transect. If you cannot see the mid point of the next transect from the starting point, use the supplemental sections (indicating the proportion of the total length represented by each section). Otherwise, leave these blank.

**Note 1:** An auto level should be used for reaches with a percent slope of less than or equal to 1%. All methods (clinometer, hand level, or auto level) may be used for reaches with a percent slope of greater than 1%. The following description is for clinometer-based slope measurements, but the same principles apply to use of an auto or hand level.

*Note 2:* In reaches that are close to 1%, you will not know whether you are above or below the 1% slope cutoff before taking readings. In these cases, default to use of an autolevel.

**Step 1.** Beginning with the upper transect (Transect K), one person (the measurer) should stand at the water margin with a clinometer held at eye level. A second person should stand at the margin of the next downstream transect (Transect J) with a stadia rod flagged at the eye level of the person taking the clinometer readings. Be sure you mark your eye level while standing on level ground! Adjust for water depth by measuring from the same height above the water surface at both transects. This is most easily accomplished by holding the base of the pole at water level.

# *Note:* An alternative technique is to use two stadia rods pre-flagged at the eye-height of the person taking the readings.

**Step 2.** Use a clinometer to measure the percent slope of the water surface (not the streambed) between the upstream transect and the downstream transect by sighting to the flagged position on the stadia rod. The clinometer reads both percent slope and degree of the slope. Be careful to read and record percent slope rather than degrees slope (these measurements differ by a factor of ~ 2.2). Percent slope is the scale on the right hand side as you look through most clinometers (e.g., Suunto models).

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**Note:** If an auto level or hand level is used, record the elevation difference (rise) between transects and the segment length (run) instead of the percent slope.

**Step 3.** If the stream reach geometry makes it difficult to sight a line between transects, divide the distance into two or three sections and record the slope and the proportion of the total segment length between transects for each of these sections in the appropriate boxes on the slope form (supplemental segments).

#### *Note:* Never measure slope across dry land (e.g., across a meander bend).

**Step 4.** Take a compass reading from the center of each main transect to the center of the next main transect downstream and record this bearing to the nearest degree on the slope and bearing section of the form. Bearing measurements should always be taken from the upstream to downstream transect.

**Step 5.** Proceed downstream to the next transect pair (I-J) and continue to record slope and bearing between each pair of transects until measurements have been recorded for all transects.

# SECTION IVB. VISUAL ESTIMATES OF HUMAN INFLUENCE, INSTREAM HABITAT, AND RIPARIAN VEGETATION

The transect-based approach used here permits semi-quantitative calculations from visual estimates even though most are categorical data (i.e., either presence/ absence or size classes) because we can calculate the percentage of transects that fall into different categories. These modules are adapted directly from EMAP protocols with some modifications as noted.

## **Module F. Human Influence**

The influence of human activities on stream biota is of critical concern in bioassessment analyses. Quantification of human activities for these analyses is often performed with GIS techniques, which are very useful but are not capable of accounting for human activities occurring at the reach scale. Reach scale observations are often critical for explaining results that might seem anomalous on the basis of only remote mapping tools.

**Step 1.** For the left and right banks, estimate a 10 x 10 m riparian area centered on the edges of the transect (see Figure 2). Record the presence of 11 human influence categories in three spatial zones relative to this 10 x 10 m square (between the wetted edge and bankfull margin, between the bankfull margin and 10 m from the stream, and between 10 m and 50 m beyond the stream margins): 1) walls/rip-rap/dams, 2) buildings, 3) pavement/cleared lots, 4) roads/railroads, 5) pipes (inlets or outlets), 6) landfills or trash, 7) parks or lawns (e.g., golf courses), 8) row crops, 9) pasture/ rangelands, 10) logging/ timber harvest activities, 11) mining activities, 12) vegetative management (herbicides, brush removal, mowing), 13) bridges/ abutments, 14) orchards or vineyards. Circle all combinations of impacts and locations that apply, but be careful to not double-count any human influence observations.





**Step 2.** Record the presence of any of the 11 human influence categories in the stream channel within a zone 5 m upstream and 5 m downstream of the transect.

# Module G. Riparian Vegetation

Riparian vegetation (vegetation in the region beyond the bankfull margins) has a strong influence on the composition of stream communities through its direct and indirect roles in controlling the food base, moderating sediment inputs and acting as a buffer between the stream channel and the surrounding environment. These methods provide a cursory survey of the condition of the riparian corridor. Observations are made in the same 10 x 10 m riparian area used for assessing human influence (see Figure 2).

#### Note: Riparian vegetation measurements should only include living or recently dead vegetation.

The riparian vegetation categories used here were condensed from the EMAP version, which further breaks the canopy classes into different components. However, because we have consolidated EMAP categories into fewer categories rather than creating new categories, existing EMAP data can be easily converted to this format simply by combining the appropriate categories.

**Step 1.** Divide the riparian zone into three elevation zones: 1) ground cover (<0.5 m), 2) lower canopy (0.5 m - 5 m), and 3) upper canopy (>5 m). Record the density of the following riparian classes: 1) Upper Canopy–Trees and Saplings, 2) Lower Canopy–Woody Shrubs and Saplings, 3) Woody Ground Cover–Shrubs, Saplings, 4) Herbaceous Ground Cover–Herbs and Grasses, and 5) Ground Cover–Barren, Bare Soil and Duff. Artificial banks (e.g., rip-rap, concrete, asphalt) should be recorded as barren.

**Step 2.** Indicate the areal cover (i.e., shading) by each riparian vegetative class as either: 1) absent, 2) sparse (<10%), 3) moderate (10-40%), 4) heavy (40-75%), or 5) very heavy (>75%).

#### Module H. Instream Habitat Complexity

Instream habitat complexity was developed by the EMAP program to quantify fish concealment features in the stream channel, but it also provides good information about the general condition and complexity of the stream channel. Estimates should include features within the banks and outside the wetted margins of the stream.

**Step 1.** Record the amount of nine different channel features within a zone 5m upstream and 5m downstream of the transect (see Figure 2): 1) filamentous algae (long-stranded algal forms that are large enough to see with the naked eye), 2) aquatic macrophytes (include mosses and vascular plants), 3) boulders (>25 cm), 4 and 5) woody debris (break into two classes- larger and smaller than 30 cm diameter), 6) undercut banks, 7) overhanging vegetation, 8) live tree roots and 9) artificial structures (includes any anthropogenic objects including large trash objects like tires and shopping carts). Indicate the areal cover of each feature as either: 1) absent, 2) sparse (<10%), 3) moderate (10-40%), 4) heavy (40-75%), or 5) very heavy (>75%).





# SECTION SECTION SECTION

While most measures are taken at or relative to the main transects, a few measures are recorded at transects located at the midpoint between main transects. These are called "inter-transects".

# Module B (Part 2) Pebble Counts (same as for transects, but no cobble embeddedness measures)

**Step 1.** Divide the wetted stream width by four to get the distance between the five points (Left Bank, Left Center, Center, Right Center and Right Bank) and use a measuring device to locate the positions of these points (a stadia rod is especially helpful here, see Figure 5). Once the positions are identified, lower a graduated rod through the water column perpendicular to both the flow and the transect to objectively select the particle located at its tip.

**Step 2.** With the graduated rod, measure the depth from the water surface to the top of the particle and record to the nearest cm.

**Step 3.** Remove the particle from the streambed, then measure and record the length of its intermediate axis to the nearest mm (see Figure 6). Alternatively, assign the particle to one of the size classes listed in the bottom of the transect form (see Table 3 for a list of size classes). Particle size classes may be estimated visually or with a quantitative measuring device (e.g., pass/ no-pass template, gravelometer). Regardless of the method, all particles less than 0.06 mm should be recorded as fines, while all particles between 0.06 mm and 2.0 mm should be recorded as sand. Field crews may want to carry vials containing sediment particles with these size ranges until they are familiar with these particle size classes.

Step 4. Record the presence (P) or absence (A) of any CPOM within 1 cm of each particle.

# **Module J. Flow Habitats**

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Because many benthic macroinvertebrates prefer specific flow and substrate microhabitats, the proportional representation of these habitats in a reach is often of interest in bioassessments. There are many different ways to quantify the proportions of different flow habitats (for example, see text on EMAP's "thalweg profile" below). Like the riparian and instream measures listed above, this procedure produces a semi-quantitative measure consisting of 10 transect-based visual estimates.

*Note:* The categories used here are based on those used in the EMAP protocol, with pools combined into one class and cascades and falls combined into another class.



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Step 1. At each inter-transect, identify the proportion of six different habitat types in the region between the upstream transect and downstream transect: 1) cascades/falls, 2) rapids, 3) riffles, 4) runs, 5) glides, 6) pools, 7) dry areas. Record percentages to the nearest 5% — the total percentage of surface area for each section must total 100%.





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# SECTION **B**

Stream discharge is the volume of water that moves past a point in a given amount of time and is generally reported as either cubic meters per second (cms) or cubic feet per second (cfs). Because discharge is directly related to water volume, discharge affects the concentration of nutrients, fine sediments and pollutants; and discharge measurements are critical for understanding impacts of disturbances such as impoundments, water withdrawals and water augmentation. Discharge is also closely related to many habitat characteristics including temperature regimes, physical habitat diversity, and habitat connectivity. As a direct result of these relationships, stream discharge is often also a strong predictor of biotic community composition. Since stream volume can vary significantly on many different temporal scales (diurnal, seasonal, inter-annually), it can also be very useful for understanding variation in stream condition.

This procedure (modified from the EMAP protocol) provides for two different methods for calculating discharge. It is preferable to take discharge measurements in sections where flow velocities are greater than 0.15 m/s and most depths are greater than 15 cm, but slower velocities and shallower depths can be used. If flow volume is sufficient for a transect-based "velocity-area" discharge calculation, this is by far the preferred method. If flow volume is too low to permit this procedure or if your flow meter fails, use the "neutrally buoyant object/ timed flow" method.

**Note:** Programs that sample fixed sites repeatedly may want to consider installing permanent discharge estimation structures (e.g., stage gauges, wiers).

# Module K. Discharge: Velocity Area Method

The layout for discharge measurements under the velocity-area (VA) method is illustrated in Figure 8. Flow velocity should be measured with either a Swoffer Instruments propeller-type flow meter or a Marsh-McBirney inductive probe flow meter. Refer to the manufacturers' instrument manuals for calibration procedures.

**VA-Step 1.** Select the best location in the reach for measuring discharge. To maximize the repeatability of the discharge measurement, choose a transect with the most uniform flow (select hydraulically smooth flow whenever possible) and simplest cross-sectional geometry. It is acceptable to move substrates or other obstacles to create a more uniform cross-section before beginning the discharge measurements.

**VA-Step 2.** Measure the wetted width of the discharge transect and divide this into 10 to 20 equal segments. The use of more segments gives a better discharge calculation, but is impractical in small channels. A minimum of 10 intervals should be used when stream width permits, but interval width should not be less than 15 cm.





**VA-Step 3.** Record the distance from the bank to the end of the first interval. Using the top-setting rod that comes with the flow velocity meter, measure the median depth of the first interval.

**VA-Step 4**. Standing downstream of the transect to avoid interfering with the flow, use the top-setting rod to set the probe of the flow meter (either the propeller or the electromagnetic probe) at the midpoint of each interval, at 0.6 of the interval depth (this position generally approximates average velocity in the water column), and at right angles to the transect (facing upstream). See Figure 8 for positioning detail.

**VA-Step 5.** Allow the flow velocity meter to equilibrate for 10-20 seconds then record velocity to the nearest m/s. If the option is available, use the flow averaging setting on the flow meter.

**Note:** Under very low flow conditions, flow velocity meters may register readings of zero even when there is noticeable flow. In these situations, record a velocity of 0.5x the minimum flow detection capabilities of the instrument.



Figure 8. Diagram of layout for discharge measurements under the velocity-area method showing proper positions for velocity probe (black dots).



VA-Step 6. Complete Steps 3 through 5 on the remaining intervals.

## Note: The first and last intervals usually have depths and velocities of zero.

### Module L. Discharge: Neutrally Buoyant Object Method

If streams are too shallow to use a flow velocity meter, the neutrally buoyant object (NBO) method should be used to measure flow velocity. However, since this method is less precise than the flow velocity meter it should only be used if absolutely necessary. A neutrally buoyant object (one whose density allows it to just balance between sinking and floating) will act as if it were nearly weightless, thus it's movement will approximate that of the water it floats in better than a light object. To estimate the flow velocity through a reach, three transects are used to measure the cross-sectional areas within the test section sub-reach and three flow velocity estimates are used to measure average velocity through the test reach. To improve precision in velocity measurements, the reach segment should be long enough for the float time to last at least 10-15 seconds.

**NBO-Step 1.** The position of the discharge sub-reach is not as critical as it is for the velocity-area method, but the same criteria for selection of a discharge reach apply to the neutrally buoyant object method. Identify a section that has relatively uniform flow and a uniform cross sectional shape.

**NBO-Step 2.** The cross sectional area is estimated in a manner that is similar but less precise than that used in the velocity area method. Measure the cross sectional area in one to three places in the section designated for the discharge measurement (three evenly-spaced cross sections are preferred, but one may be used if the cross section through the reach is very uniform). Record the width once for each cross section and measure depth at five equally-spaced positions along each transect.

**NBO-Step 3.** Record the length of the discharge reach.

**NBO-Step 4.** Place a neutrally buoyant object (e.g., orange, rubber ball, heavy piece of wood, etc.) in the water upstream of the discharge reach and record the length of time in seconds that it takes for the object to pass between the upstream and downstream boundaries of the reach. Repeat this timed float three times.

# SECTION 7

# Module M. Rapid Bioassessment Procedures Visual Assessment Scores (for Basic Physical Habitat, or optional supplement)

EPA's Rapid Bioassessment Procedures (RBPs, Barbour et al. 1999) include a set of 10 visual criteria for assessing instream and riparian habitat. The RBP has been used in the CSBP since its first edition (1995) and thus, this information is often valuable for comparison to legacy datasets. The criteria also have a useful didactic role since they help force the user to quantify key features of the physical environment where bioassessment samples are collected.

# Module N. Additional Habitat Characterization (Full Physical Habitat only)

The RBP stream habitat visual estimates described in Step 1 are not included in the Full Physical Habitat version because they are generally replaced by more quantitative measurements of similar variables. However, we have found that three of the RBP measures are reasonably repeatable and include them in the reachwide assessment portion of the Full Physical Habitat version.

*Note:* This is the only case in which a measurement included in the basic procedure is not included in the full.

# Module O. Reach Slope (for Basic Physical Habitat only)

Reach slope should be recorded as percent slope as opposed to degrees slope to avoid confusion. Slope measurements work best with two people, one taking the readings at the upstream transect and the other holding a stadia rod at the downstream transect. If you cannot see the mid point of the next transect from the starting point, use the supplemental sections (indicating the proportion of the total length represented by each section).

An auto level (with a tripod) should be used for reaches with a percent slope of less than or equal to 1%. All methods (clinometer, hand level, or auto level) may be used for reaches with a percent slope of greater than 1%. In reaches that are close to 1%, you will not know whether you are above or below the 1% slope cutoff. In these cases, default to use of an autolevel.

**Step 1.** Divide the reach into multiple segments such that stadia rod markings can be easily read with the measuring device to be employed (this is especially a factor for clinometer and hand level readings).





**Step 2.** Use a clinometer, hand level, or auto level to measure the percent slope of the water surface (not the streambed) between the top and bottom of each segment. Be sure to adjust for water depth by measuring from the same height above the water surface at both transects. Also be sure to record percent slope, not degrees slope. Record the segment length for each of these sections in the appropriate boxes on the BASIC slope form.





# OPTIONAL EXCESS SEDIMENT MEASURES

Future editions of these protocols will include supplemental modules, including a full discussion of the measurements used for calculating the excess sediment index (sometimes referred to as log relative bed stability, LRBS). However, since several of the measurements in EMAP's physical habitat protocols are interwoven into the layout of this protocol, a brief overview of the additional measurements collected for the LRBS calculations is included here for information purposes only. For detailed explanations of these measurements, consult Peck et al. 2004.

# **Woody Debris Tallies**

Large woody debris (logs, snags, branches, etc.) that is capable of obstructing flow when the channel is at bankfull condition (just short of flood stage) contributes to the "roughness" of a channel. The effect of this variable is to reduce water velocity and thereby reduce the stream's competence to move substrate particles. The EMAP protocol tallies all woody debris with a diameter greater than 10 cm ( $\sim 4$ ") into one of 12 size classes based on the length and width of each object. Tallies are conducted in the zone between the main transects.

# **Thalweg Measurements**

A stream's thalweg is a longitudinal profile that connects the deepest points of successive cross-sections of the stream. The thalweg defines the primary path of water flow through the reach. Thalweg measurements perform many functions in the EMAP protocols, producing measurements for the excess sediment calculations (residual pool volume, stream size, channel complexity) and flow habitat variability.



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# SECTION OPTIONAL PERIPHYTON QUANTIFICATION

# **Periphyton Quantification**

Characterization of periphyton has a dual role in bioassessments, as periphyton is both a food and habitat resource for benthic macroinvertebrates and fish and an effective bioindicator on its own. Quantification of periphytic resources will be covered under a separate SWAMP bioassessment protocol, but will include procedures for qualitative characterization of diatom assemblages, documentation of filamentous algal growth, and biomass quantification (e.g., ash-free dry mass and chlorophyll a).





# SECTION 1 O

The SWAMP bioassessment group is currently developing guidelines for quality assurance and quality control for bioassessment procedures. Future revisions to this document will include guidance covering personnel qualifications, training and field audit procedures, procedures for field calibration, procedures for chain of custody documentation, requirements for measurement precision, health and safety warnings, cautions (actions that would result in instrument damage or compromised samples), and interferences (consequences of not following the standard operating procedure, SOP).

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# DEFINITIONS OF TERMS USED IN SOP

Terms & Definitions		
TERM	DEFINITION	
ABL	California Department of Fish and Game's Aquatic Bioassessment Laboratory	
Allocthonous	Derived from a source external to the stream channel (e.g., riparian vegetation) as opposed to autochthonous, which indicates a source inside the stream channel (e.g., periphyton).	
Ambient Bioassessment	Biological monitoring that is intended to describe general biotic condition as opposed to a diagnosis of sources of impairment	
Bankfull	The bankfull channel is the zone of maximum water inundation in a normal flow year (one to two year flood events)	
вмі	Benthic macroinvertebrates: bottom-dwelling invertebrates large enough to be seen with the unaided eye	
Cobble Embeddedness	The volume of cobble-sized particles (64-250 mm) that is buried by fine particles (<2.0 mm diameter)	
СРОМ	Coarse particulate organic matter (CPOM, particles of decaying organic material such as leaves that are greater than 1.0 mm in diameter)	
CSBP	California State Bioassessment Procedures	
DFG	California Department of Fish and Game	
EMAP	The U.S. Environmental Protection Agency's Environmental Monitoring and Assessment Program	
EPA	The U.S. Environmental Protection Agency	
Fines	Substrate particles less than 0.06 mm diameter (not gritty to touch)	
Inter-transects	Transects established at points equidistant between the main transects	
MCM	Margin-Center-Margin alternative procedure for sampling low gradient habitats	
ORD	EPA's Office of Research and Development	
QAMP	Quality assurance management plan	
RBP	EPA's Rapid Bioassessment Procedures	
Reach	A segment of the stream channel	
Riparian	An area of land and vegetation adjacent to a stream that has a direct effect on the stream.	
RWB	Reach-wide benthos composite sampling method for benthic macroinvertebrates, also referred to as multi-habitat method	
SCCWRP	Southern Coastal California Water Research Project	
SNARL	Sierra Nevada Aquatic Research Laboratory	
Substrate	The composition of a streambed, including both inorganic and organic particles	
SWAMP	The State Water Resources Control Board's Surface Water Ambient Monitoring Program	
Thalweg	A longitudinal profile that connects the deepest points at successive cross-sections of the stream. The thalweg defines the primary path of water flow through the reach	

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TERM	DEFINITION
Transects	Lines drawn perpendicular to the path of flow used for standardizing sampling locations
TRC	Targeted riffle composite sampling method for benthic macroinvertebrates
USFS	The United States Forest Service
Wadeable Streams	Streams that can be sampled by field crews wearing chest waders (generally less that 0.5 m - 1.0 meters deep)





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# APPENDIX A

# FACTORS TO CONSIDER WHEN RECOMMENDING/ CHANGING BIOASSESSMENT METHODS

Beyond the primary considerations of precision and accuracy, there are at least five other key issues that SWAMP has considered and should consider in the future, when recommending or changing its official methods for bioassessment. These issues include:

**1. Costs of Collecting Samples via Multiple Protocols** – Collecting, processing, and interpreting samples using more than one method for each indicator (e.g., algae, macroinvertebrates, fish) per site adds significant costs to bioassessment monitoring programs. SWAMP should strive to identify the minimum set of protocols necessary for each indicator. However, this should not come at the expense of sound monitoring. If more than one method is needed to interpret the biological response, then this decision should be based on a cost-benefit assessment.

**2.** Costs of Maintaining Multiple SWAMP Protocols – While multiple methods for monitoring a given indicator may provide additional accuracy in specific habitats, there are significant costs to maintaining multiple protocols:

- **a.** Need to maintain method-specific infrastructure (e.g., separate reference samples, separate indices of biotic integrity (IBIs), separate O/E models, etc.).
- **b.** May lose or impair ability to compare across sites if different methods are used (see Issue 5 below).
- **c.** Guidance on when to use methods becomes more complex. For example, we need to define very specifically which methods to use at each water body type; and thus, which tools can be used to interpret them.

**Recommendation:** SWAMP should maintain as few protocols as necessary. If we elect to add new or modified protocols it should be because we have determined that the added value is worth all of the costs listed above.

**3. Separating Physical Impairment from Water Quality Impairment** – One of the original reasons for adding a multihabitat component to SWAMP bioassessment programs was the potential for distinguishing physical and water quality impairment sources (see recommendations in Barbour and Hill 2002). In regards to macroinvertebrate indicators, the conventional wisdom has been that reachwide (RW, sometimes referred to as multihabitat or MH) samples should be relatively more responsive to physical habitat alteration (i.e., fine sediment inputs) than targeted-riffle (TR) samples because it is believed that erosional habitats take longer





to respond to sediment stresses, and because pockets of riffle habitat are thought to act as refugia from habitat loss. To the extent that this is true, RW and TR samples may offer complementary information that allows us to separate these sources of impairment.

While very few studies have addressed this conventional wisdom directly, recent studies suggest that this may not be as much a factor as previously believed. In a recent comparison of TR and RW samples at nearly 200 sites statewide, the ABL found at most weak evidence to support this notion (Rehn et al. 2007). Gerth and Herlihy (2006) came to the same conclusion in their analysis of ~ 500 sites in the eastern and western United States. However, this issue is far from resolved and SWAMP scientists currently are not in agreement regarding this issue. Since the majority of bioassessment programs in California have emphasized targeted riffle sampling, SWAMP will undoubtedly want to evaluate this question further before making any policy decision to discontinue TR sampling.

**Recommendation:** Until this issue can be evaluated further and resolved to SWAMP's satisfaction, ambient macroinvertebrate sampling should include collection of both RW samples and richest targeted habitat (TR or MCM) samples at every site. (The TR method should be used where sufficient riffles are present, and the MCM method should be used at low-gradient sites where sufficient riffle habitat is not available.)

**4. Compatibility with Previous Data** – To address this issue, at least three sets of macroinvertebrate sampling method comparisons have been conducted in California.

- a. Targeted Riffle Methods Comparisons are complete. Samples collected under the current TR protocols are considered interchangeable with both CSBP and SNARL samples (Ode et al. 2005, Herbst and Silldorff 2006).
- **b.** Low Gradient Sand-Dominated Streams Collaborative studies are currently underway between Water Board Regions 3 and 5, the Southern California Coastal Water Research Project (SCCWRP), and ABL to compare the performance of: (1) the "low-gradient" CSBP; (2) RW samples; and (3) a modification of the RW method designed to emphasize habitats along stream margins (MCM). The results of these low-gradient methods comparisons are not yet available.
- **c.** Targeted Riffle vs. Reachwide Methods A recent comparison of RW and TR samples collected from nearly 200 EMAP/ CMAP sites is in peer review press (Rehn et al. 2007). Results demonstrate remarkably similar performance of the methods across a wide range of habitats. Gerth and Herlihy (2006) recently published a similar analysis with the same conclusions. However, the bioassessment committee has yet to carefully review and discuss these analyses and their implications for SWAMP biomonitoring.

**5. Comparability Among Sites** – The ability to compare biological condition across sites is a common requirement of most ambient bioassessment programs. This type of analysis is confounded if different methods are used at these sites. One of the big advantages of reachwide (i.e., multihabitat) methods is that they can be applied anywhere because they don't require a specific habitat for sampling. Statewide



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bioassessments and most regional programs will require the ability to compare their bioassessment results among multiple sites (e.g., within a watershed, within a region, statewide).

# INTERIM RECOMMENDATIONS FOR MACROINVERTEBRATE SAMPLING (UPDATED DECEMBER 2006):

1. Until we can reach consensus on the outstanding issues (i.e., whether a single method for macroinvertebrate sampling will meet our needs, and the outcome of RW vs. MCM comparison studies for low-gradient wadeable streams/rivers), SWAMP recommends collecting both a reachwide (i.e., multihabitat) and a targeted habitat sample at each site. In high gradient streams, this means using both the RW and TR methods. In low-gradient streams, we recommend collecting both RW and MCM samples until the results are available from the low-gradient ("non-riffle") comparison. In rare cases where monitoring objectives cannot be met following these recommendations, the SWAMP Bioassessment Coordinator may authorize deviations. For example, where project-specific objectives differ from ambient monitoring, the SWAMP Bioassessment Coordinator may authorize alternate methods. In rare cases where funding is extremely limited and the cost of following the above recommendations would be prohibitive, the SWAMP Bioassessment Coordinator may authorize laternate methods. Such as collecting both samples, but archiving one of the samples for later lab analysis.

2. SWAMP should develop guidance specifying when and where different methods should be used. For example, at "low gradient" sites, what is the slope cut-off (or other channel feature criteria to use) when deciding whether to apply TR or MCM? In addition, while SWAMP may eventually choose to adopt a single method (such as RW) at most sites, some regions may determine that the value of targeted habitat sampling merits continued sampling with supplemental protocols. In the latter case, or if SWAMP determines that distinct methods are needed for different habitat types, the guidance should specify the types of waterbodies or classes of waterbodies that require different methods.

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# **Appendix F**

# Laboratory Quality Assurance Management Plan for Weck Laboratories

for the Newhall Ranch Specific Plan Water Quality Monitoring Plan Conditions of Approval and Clean Water Act Section 401 Water Quality Certification and Waste Discharge Requirements



engineers | scientists | innovators

924 Anacapa Street, Suite 4A Santa Barbara, CA 93101

Project Number LA0170

March 27, 2013



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	<b>Resumes of Key Personnel</b>
Appendix 2	Code of Ethics
Appendix 3	Organization Chart
Appendix 4	List of Major Equipment
Appendix 5	Chain of Custody Form
Appendix 6	Sample Collection and Holding Times
Appendix 7	List of SOPs
Appendix 8	Acceptance Limits for QC Determinations
Appendix 9	Initial Demonstration of Capability Procedure
Appendix 10	<b>Corrective Action Report Form</b>
Appendix 11	Laboratory Accreditations
Appendix 12	Flags Used for Data Qualifiers

# 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.

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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.

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# **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.

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# 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 laboratory
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.

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# 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.

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## 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	<b>Tests Performed</b>
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	General Chemistry	1	UV-Visible Spectrophotometer Hach model DR4000U	Various
ISE/pH	General Chemistry	1	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	<b>Tests Performed</b>
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

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	RELINQUISHED BY	DATE / TIME	RECEIVED BY	L.	reserved at Lab	SO = Soll 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	l 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	l 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	Va	arious	Wet Chem methods	various
General Minerals (metals only)	Water	Poly	250 ml	HNO3	HNO3	6 M	Nonths	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		l4 days	GC/ECD	SM 5710B/EPA 552.2
Herbicides-DW	Water	Amber Glass	250 ml (*)	None	Thiosulfate	1	l4 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	2	28 Days	Cold Vapor AAS	EPA 245.1/7470
Methanol	Water	Glass	1 x 40 ml	None	None	1	4 Days	Dir. Inj./FID	EPA 8015B
Mercury in soil/solid/sludge	Soil/Solid	Glass jar	4 oz.	None	None	2	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	4	8 Hours	IC or FIA	EPA 300.0/353.2
Nitrite	Water	Poly	250 ml	None	None	4	8 Hours	IC or FIA	EPA 300.0/353.2
Nitrite+Nitrate as N	Water	Poly	250 ml	H2SO4	H2SO4	2	28 Days	FIA-Colorimetric	EPA353.2
Nitrogen, Total Kjeldahl (TKN)	Water	Poly	250 ml	H2SO4	H2SO4	2	28 Days	FIA-Colorimetric	EPA 351.2
Nitrogen-Ammonia	Water	Poly	250 ml	H2SO4	H2SO4	2	28 Days	FIA-Colorimetric	EPA 350.1
Nitrogen-Ammonia in ww with distillation	Water	Poly	250 ml	H2SO4	H2SO4	2	28 Days	FIA-Colorimetric	EPA 350.1
Nitrosamines	Water	Amber Glass	2 x 1 L (*)	None	Ascorbic	1	14 days	GC/MS/CI SIM	EPA 521
Odor	Water	Glass	500 ml	None	None	2	4 Hours	Odor	SM 2150B
Oil and Grease	Water	Glass	1 L	HCL	HCL	2	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	2	4 Hours	O2 Probe	SM 4500-OG
PBDEs	Water	Amber Glass	2 x 1 L (*)	None	None	1	l4 days	GC/MS SIM	EPA 1614M
Perchlorate	Water	Poly	250 ml	None	None	2	28 Days	IC	EPA 314
Perchlorate - Low Level by LC/MS/MS	Water	Poly Sterile	125 ml	Sterile field filtration	Sterile field filtration	2	28 Days	LC/MS/MS	EPA 331/332
Perchlorate in soils	Soil	Glass jar	4 oz	None	None	2	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	1	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	Rev	Rev	Method	Title
Name	No	Date		
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
				Chemicals, Standards and consumable materials, Receipt, Storage and Preparation of
MIS004	5	Apr-08	General	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
				Sampling Instructions for Protected Groundwater Supplies and Water Supplies with
MIS010	2	Mar-08	Sampling	Treatment
MIS011	4	Mar-08	General	Preparation, Approval, Distribution, & Revision of standard Operating Procedures
MIS012	2	Mar-08	General	Significant Figures and Rounding
MIS013	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 Kaw 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
NIIS044	3	Mar 09	General	Control of Nonconforming Environmental Testing
IVII5045	4	Mar 09	General	Control of Records and Documents
WIS046	3	mar-09	General	I raining of Laboratory Personnel

MIS047	3	Mar-09	General	Estimating the Uncertainty of Measurements
MIS048	З	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

Fil		R		
е	Rev	ev	Method	Title
N				
a m		Dat		
е	No	e		
М		S		
Е		ер		
T0		-		
01	6	07	1311	Toxicity Characteristic Leaching Procedure (TCLP)
		3		
TO		р-		Acid Digestion of Agueous Samples and Extracts for Total Metals by ICP and ICP-MS. EPA
05	6	08	3010A	Method 3010A Modified
М		S		
E		е		
10	F	p-	2050P	Acid Direction of Sodimente, Sludges and Soile, EDA Method 2050P
M	5	S	3030D	
E		e		
Т0		p-	3050B	
09	3	08	Mod	Acid Digestion of Sediments, Sludges, Soils and Wipes, EPA Method 3050 Modified.
M		S		
E TO		e		
10	7	-4 08	74710	Analysis of Mercury in Solid Matrices by Cold Vanor Atomic Absorption, EPA 7471A
M	,	S	17111	
Е		е		
Т0		p-		
11	5	08	245.1	Analysis of Hg in water by manual cold vapor technique EPA method 245.1
		J		
		n-		
17	8	08	6010	Analysis of Trace Metal in Water and Solid Matrices by ICP-AES, EPA Method 6010
М		S		
E		е		
10	10	p-	200.9	Analysis of Trace Metals in Water by ICP MS, EPA Method 200.8
M	10	S	200.0	Analysis of Trace Metals III Water by ICF-INS, EFA Method 200.0
E		e		
Т0		p-		
19	7	08	6020	Analysis of Trace Metal in Water and Solid Matrices by ICP-MS, EPA Method 6020
M		S		
E TO		e n-		Sample Proparation Procedure for Spectrochemical Determination of Total Paceverable
20	5	08	200.2	Elements, EPA Method 200.2
M		S		
Е		е		
T0		p-	==	
21	3	80	WET	Waste Extraction Test Procedure, Title 22 Part 66261.126 Appendix II

M E T0			S e p-	As-				
23		3	08	ICPN	1S	Analysis	of Arsenic by Hydride Generation-ICPMS, EPA Method 200.8 Modified	
E T0			e p-	Se-				
24 M	:	3	08 D	ICPM	1S	Analysis of Selenium by Hydride Generation-ICPMS, EPA Method 200.8 Modified		
E			ec					
10 25		5	- 08	200.	7	Analysis	Analysis of Trace Metals in Water by ICP-AES, EPA Method 200.7	
M F			S e					
T0 31		3	р- 08	747	0	Analysis EPA 74	of Mercury in Aqueous Samples and Liquid Waste by Cold Vapor Atomic Absorption, 70A	
M E			M ar					
T0		1	-	162	1	Analysis	of Low Lovel Mercury by CVAES with Gold Amalgamation EDA Method 1621E	
<u>34</u> М		I	M	103	1	Analysis	S of Low Level Mercury by CVAPS with Gold Amagamation, EPA Method 1031E	
E TO			ay -					
35		1	07	245.	7	Analysis	s of Low Level Mercury by CVAFS, EPA Method 245.7	
M E			J u					
Т0 36		1	n- 08	164	Determine 0 ICP-MS		nation of Trace Elements in Saline Waters by Direct Injection and Preconcentration and - EPA Method 1640	
M F			J					
T0			n-	3500	Fe	<b>.</b>		
37 M		1	08 O	В		Determi	nation of Ferrous Iron by the Phenantrioline Colorimetric Method, SM3500-Fe B	
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38		1	- 08	163	8	Analysis	s of Trace Elements in Ambient Waters by ICP-MS - EPA Method 1638	
M F			Mav					
T0		•	-	SM2	33			
39		2	09	08		Determi	nation of Corrosivity (Langlier Index) in Water, SM 2330B	
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Nd	ille	INU	U	ale			Analysis of Total Coliform and F. Coli in Water by P/A Colilert <sup>TM</sup> and Enumeration by	
MIC	003	8	Fe	b-09	SM	19223	the Quanti-Tray® method, SM9223	
MIC	004	6	Fe	b-09	SM /Sir	l9215B mPlate	Analysis of Heterotrophic Plate Count by Pour Plate and SimPlate Methods, SM 9215B	
MIC	005	7	Ар	or-09	SM	19221	Analysis of Total and Fecal Coliform in Water by Multiple Tube Fermentation Technique, SM9221	
MIC	006	5	Ma	iy-09	QA		Quality Assurance for Microbiological Tests	
MIC	007	∠ 3	Ju	il-09	QA		Verification of Support Equipment Used for Microbiological Determinations	
		-	-				Bacteriological Analysis of Ambient Water Samples for Enterococci by Enterolert	
MIC	009	2	Ар	or-09	Ent	terolert	Presence/Absence and Quanti-Tray® Method	
MIC	010	1	Ар	or-09	DIS	posal	Disposal of Material Used for Microbiological Determinations	

# SOP's LIST AND INDEX Radio Chemistry Department - RadChem SOPs

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Name	No	Date		
				Determination of Gross Alpha and Gross Beta Radioactivity in Drinking Water, EPA
RAD001	2	Nov-07	900.0	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
				The Procedure for Monitoring Radiation Measurement Instrumentation for Radioactive
RAD005	1	Apr-06	All	Contamination
				The Procedure for Handling, Storing and Establishing Expiration Dates for Reference
RAD006	1	Apr-06	All	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
				Spectrometric Determination of Uranium in Water Samples for Radiological
RAD009	1	Sep-07	200.8	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	
				Analysis of Anions in Solid and Liquid Matrices by Ion Chromatography, EPA Method	
WET002	1	Sep-02	9056	9056	
			SM4500CN		
WET003	11	Oct-08	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	Analysis of Thiogyapate in Wastewater by SM 4500-CN M	
	2	Oct-00	140.4	Analysis of Oder in Drinking Water, EDA Method 140 4/SM 2450	
WET013	3		140.1	Analysis of Odol in Dinking Water, EPA Method 140. //SW 2150	
WE1015	3	Oct-08	E203	Analysis of Water Content by Karl Fisher Titration ASTM Method E203	
			SM4500CN	Analysis of Cyanide Amenable to Chlorination in Water - Manual Colorimetric, SM	
WET018	4	Oct-08	G	4500CN-G	
				Analysis of Low Level Total Recoverable Phenolics in Water by chloroform Extraction	
WET019	5	Mar-08	420.1	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	B Analysis of Acidity as CaCO3, SM 2310B		
				Alkaline Digestion for Analysis of Hexavalent Chromium in Solid Matrices, EPA Method		
WET027	3	Dec-08	3060	3060		
WET028	5	Jan-08	SM4500 H B	pH (Electrometric), SM 4500-H+ B		
WET029	4	Dec-08		Analysis of Hexavalent Chromium in Water - Manual Colorimetric, SM 3500-Cr D		
WET032	4	Dec-08	SM4500 S2 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	SM4500CI G	Analysis of Total Residual Chorine by Colorimetry with DPD, SM 4500CI G		
WET039	6	Jan-08	SM2510B	Determination of Specific Conductance, SM 2510B		
WET041	7	May-08	SM2540C	Filterable Residue (TDS) by Gravimetric analysis, SM 2540C		
WET042	7	Dec-08	SM2540D	Determination of Non-filterable Residue (TSS) by Gravimetry, SM 2540D		
				Determination of Methylene Blue Active Substances (MBAS) by Spectrophotometry,		
WET043	4	Jan-09	SM5540C	SM 5540C		
WE1044	2	Dec-08	253B	Analysis of Thiosulfate and Sulfite by Iodometric Titration, LACSD Procedure 253B		
WE1046	3	Dec-U8	SM2540B	Determination of Total Residue (TS) by Gravimetry, SM 2540B		
WE1047	4	Jun-ux	160.4	Determination of Volatile Residue (VS) by Gravimetry, EPA Method 160.4		
VVE1048	4	Dec-08	SM2540F	Determination of Settleable Residue (SS) by volumetric imnon Cone, Sivi 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		
MET070		D 00	SM4500CIO2			
WE1070	3	Dec-08	D	Analysis of Chlorine Dioxide by Colorimetric Method with DPD, SM 4500-CIO2 D		
	2	Dec 09	SM4500 O	Determination of Dissolved Overson by Membrane Electrode Method, SM 4500, O.C.		
VVEI072	3	Dec-08	G SM4500SO3	Determination of Dissolved Oxygen by Memorane Electrode Method, SM 4500-O G		
WET073	3	Dec-08	В	Analysis of Sulfite by Iodometric Method, SM4500SO3= B		
				Distillation and Analysis of Total and Amenable Cyanide in Waste and Solid Matrices		
WE1074	3	Dec-08	9010/9014	,EPA Method 9010B/9014		
WE1075	2	Dec-08	CCR ch10	Determination of Ignitability in Waste, CCR Chapter 10, Article 3		
WE1077	2	Dec-08	CCR ch10	Determination of Corrosivity in Waste, CCR Chapter 10, Article 3		
VVE1078	2	Dec-08	SM5910	Determination of UV Absorbing Constituents (UV-254), SNI 5910		
WET070	2	Dec 09	74.00	Analysis of Havavalant Chromium by Manual Spectrophotometria EDA Method 7106A		
VVE1079	2	Dec-00	7196	Analysis of Hexavalent Chromium by Manual Spectrophotometric, EPA Method 7196A		
	1	Dec-08	365.3	Analysis of Total Phosphorus and Ortho Phosphate in Water by Manual Colorimetric Method, EPA Method, 365.3		
WET080	2	Dec-08	ΔSTM2382	Determination of Heat of combustion ASTM Method 2382		
WEIGOI		200 00	A01102302			
WET084	2	Jan-09	353.2	Analysis of Nitrate and Nitrite in Water by Automated Colorimetry, EPA Method 353.2		
WFT086	2	Jan-09	350.1	Analysis of Ammonia in Water by Automated Colorimetry, EPA Method 350.1		
1121000			000.1	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	Mav-09	365.1	Analysis of Orthophosphate in Water by Automated Colorimetry, EPA Method 365.1		

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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
				Analysis of Total Cyanide in Water by Midi-Distillation and Automated Colorimetry, EPA
WET091	2	Jan-09	335.4	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
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WE1101	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

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Name	No	Date			
ORG003	7	Apr-05	SM5310C	Total Organic Carbon (TOC) and Dissolved Organic Carbon (DOC), SM 5310C	
				Determination of Total Organic Halides (TOX) in Water by Adsorption-Pyrolysis-	
ORG004	9	Mar-02	SM5320B	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	
ORG022	4	Mar-01	508	Analysis of Organochlorine Pesticides and PCBs in Drinking Water by LL Extraction 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	
00000	-	0 01	504.4	Analysis of EDB, DBCP and 123TCP in Water by Microextraction and GC/ECD, EPA	
ORG030	5	Sep-01	504.1	504.1	
ORG032	5	Mar-94	622	Analysis of Halogenated Hydrocarbons in Charcoar Tubes, NIOSH Method 1003	
ORG033	1	Lun-94	032 0SH457	Analysis of Didion by HELC-OV, EFA Method 652 Analysis of 4 4-Methylenedianiline (MDA) in Air Filters, OSHA Method 57	
01(0004	1	Jun-94	0011/07	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	Mav-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	
ORG046	3	Feb-02	3500	Sample Preparation and Extraction for Hazardous Waste Samples, EPA Method 3500B	
ORG047	3	Feb-02	3510	Separatory Funnel Liquid-Liquid Extraction, EPA Method 3510B	
ORG048	3	Feb-02	3550	Ultrasonic Extraction, EPA Method 3550B	
ORG049	2	Feb-02	3580	Waste Dilution Procedure, EPA Method 3580A	
ORG050	3	Mar-02	5030	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	
ORG058	5	Mar-02	8082	Analysis of Polychlorinated Biphenyl's (PCBs) in Liquid and Solid Matrices by GC-ECD, EPA Method 8082	
ORG059	1	Jul-99	1666	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 Determination of Total Organic Halides in Water by Adsorption-Pyrolysis-Titrimetric	
ORG062	6	Nov-03	9020B	Method, EPA Method 9020B	

ORG064         3         Mar-02         Construction of Utra Low Levels of N-Ntrosodimethytamic (DNA) by Continuous           ORG065         10         Dec-03         1625M         1-L-Extraction and Isolopic Dilution OC/MS. EPA Method 1525C Mod           ORG066         2         Feb-03         8270sim         Determination of Volatile Organic Compounds in Soil by Closed-System Purge and           ORG067         3         Mar-02         5005         Trap and GC/MS. EPA Method 5270 Modified           ORG071         2         Mar-02         5015         Trap and GC/MS. EPA Method 505           ORG071         2         Mar-02         5015         Trap and GC/MS. EPA Method 505           ORG072         2         Mar-02         5015         Achitysis of Alcohols by GC-FID, EPA Method 505           ORG073         3         Sep-01         505         C-ECD, EPA Method 505           ORG074         2         Jul-02         General         Establishing Retention Times Windows for Organic Analyses by GC-BID, CAN Method 505           ORG075         2         Mar-02         152.2         Analysis of Hotohaute Alexit by Unconstruction and GC-ECD, EPA Method 505           ORG076         2         Mar-02         ES2.4         Analysis of Hotohaute Alexit by Ion Chromatography, EPA 218.6           ORG077         4         Mar-0	ORG063	3	Jul-02	9020M	Determination of Total Halogens and Total Extractable Organic Halides in Solid and Oil Matrices, EPA Method 9020B Modified	
ORG065         Dec-03         TE2H retrinstion of Ultra Low Levels of N-Niroscilmethylamice (TDMA) by Continuous           ORG066         2         Feb-03         6270sim         Determination of Low Levels of Polynuclear Aromatic Compound in Water and Solid           Marces by GO/MS SIM Mode, EPA Method 5270 Modified         Determination of Volatile Organic Compounds in Soli by Closed-System Purge and           ORG067         Mar-02         5035         Trap and GC/MS, EPA Method 5270 Modified           ORG071         Mar-02         B016B         Analysis of Ichonby by GC-FID, EPA Method 5015B           ORG072         Mar-02         5015         Analysis of Ichonby by GC-FID, EPA Method 5015B           ORG074         Jul-02         Sep-01         505         Analysis of Ichonby by GC-FID, EPA Method 5015B           ORG074         Jul-02         General         Establishing Retention Times Windows for Organic Analyses by GC and GC/MS           ORG075         Mar-01         552.2         Analysis of Tehonby alcohon (Tahu alcohon (Tah	ORG064	3	Mar-02	608	Analysis of Organochlorine Pesticides and PCBs in Wastewater by GC-ECD, EPA Method 608.	
Determination of Low Levels of Polynuclear Anomatic Compound in Water and Solid           ORG066         2         Feb-03         8270sim         Matrices by GC/MS SM Mode, EPX Method 8270 Modified           ORG067         3         Mar-02         5035         Determination of Volatile Organic Compounds in Soli by Closed-System Purge and Trap and GC/MS, EPA 5035/8260           ORG071         2         Mar-02         80156         Analysis of Alcohols by GC-FID, EPA Method 80156           ORG072         2         Mar-02         551.3         Method 515.3           ORG075         2         Mar-02         General         Establishing Retention Times Windows for Organic Analyses by GC and GC/MS           ORG075         2         Mar-02         CEO. EPA Method 505         OC-FCD, EPA Method 505           ORG076         2         Mar-02         CAL         Analysis of Choinnated Pesticides and PCBs in Drinking Water by Microextraction and OC-FCD, EPA Method 505           ORG076         2         Mar-01         S52.2         Analysis of Hexavalent Chromium in Water by Ion Chromatography, EPA 218.6           ORG077         4         May-06         218.6         Analysis of Themane Torinking Water by SPE and GC/MS, EPA Method 528           ORG078         1         Jan-02         526         Analysis of Second SU/S 12.3-Trichioropropane by L-L extraction and GC/MS SIM <tr< td=""><td>ORG065</td><td>10</td><td>Dec-03</td><td>1625M</td><td>Determination of Ultra Low Levels of N-Nitrosodimethylamine (NDMA) by Continuous L-L Extraction and Isotopic Dilution GC/MS. EPA Method 1625C Mod</td></tr<>	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	
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 71199           ORG071         2         Mar-02         8015B         Analysis of Chlorinated Acid Herbicides in Water by Microextraction and GC-ECD, EPA Method 515.3           ORG073         3         Sep-01         505         GC-ECD, EPA Method 505           ORG074         2         Mur-02         Establishing Retention Times Windows for Organic Analysis of CACRDS           ORG075         Mar-01         552         Analysis of Haloacetic Acids by Microextraction and GC-ECD, EPA 52.2           ORG076         Mar-01         552.2         Analysis of Hexavalent Chromium in Water by Ion Chromatography, EPA 218.6           ORG077         Mar-02         524.2M         Analysis of TPH and BTEX by GC/MS LUFT Method           ORG076         Map-03         252.4         Analysis of Selected SVOA in Drinking Water by SPE and GC/MS, EPA Method 528           ORG080         1         Jan-02         526         Analysis of Selected SVOA in Drinking Water by SPE and GC/MS, EPA Method 526           ORG082         1         Apr-02         TCP-Ft         mode, SRL Method         MarOS	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	
ORG000         S         Image and Octimes, Enr A Social Control           ORG066         May-08         7199         Analysis of Hexavalent Chromium by Ion Chromatography, EPA Method 71199           ORG071         2         Mar-02         8015B         Analysis of Chlorinated Acid Herbicides in Water by Microextraction and GC-ECD, EPA           ORG072         2         Mar-02         505         Analysis of Chlorinated Acid Herbicides and PCBs in Drinking Water by Microextraction and GC-ECD, EPA           ORG073         Sep-01         505         GC-ECD, EPA Method 505         Organic Analysis           ORG074         Jul-02         General         Establishing Retention Times Windows for Organic Analysis           ORG077         Mar-01         522.2         Analysis of Hexavalent Chromium in Water by Ion Chromatography, EPA 218.6           ORG077         Mary-08         218.6         Analysis of Thana BTEX by GC/MS LUT Method           ORG076         Mary-01         524.2M         Analysis of Phenols in Drinking Water by SPE and GC/MS, EPA Method 528           ORG080         1         Jan-02         526         Analysis of Selected SVOA in Drinking Water by SPE and GC/MS, EPA Method 526           ORG081         1         Jan-02         TCP-F         Maryois of Low Levels of 1.2.3 Trichloropropane by L-L extraction and GC/MS SIM           ORG082         1 <td< td=""><td></td><td>3</td><td>Mar-02</td><td>5035</td><td>Determination of Volatile Organic Compounds in Soil by Closed-System Purge and Trap and GC/MS_EPA 5035/8260</td></td<>		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         80159         Analysis of Chiorinated Acid Herbicides in Water by Microextraction and GC-ECD, EPA           ORG072         2         Mar-02         515.3         Method 515.3           ORG073         3         Sep-01         505         GC-ECD, EPA Method Sto           ORG074         2         Jul-02         General         Establishing Retention Times Windows for Organic Analyses by GC and GC/MS           ORG076         2         Mar-02         Instrument Maintenance for Organic Analysis         Mayos by Microextraction and GC-ECD, EPA 552.2           ORG076         1         Apr-01         524.2M         Analysis of Hexavalent Chromium in Water by Ion Chromatography, EPA 218.6           ORG079         1         Iuft         Analysis of Orbit Sin Drinking Water by SPE and GC/MS, EPA Method 528           ORG080         1         Jan-02         526         Analysis of Iow Levels of 1.2.3-Trichloropropane by L-L extraction and GC/MS SIM           ORG082         1         Apr-02         TCP-E         Towalysis of Low Levels of 1.2.3-Trichloropropane by Purge and Trap and GC/MS SIM           ORG083         1         May-08         300.1         Analysis of Low Levels of 1.2.3-Trichloropr	0100007	5	11101-02	5055	Trap and 60/1003, ET A 3033/0200	
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           ORG073         3         Sep-01         505         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         Mar-01         552.2         Analysis of Haloacetic Acids by Microextraction and GC-ECD, EPA 552.2           ORG077         Mar-01         524.2M         Analysis of Haloacetic Acids by Ocganic Analysis           ORG076         Mar-01         524.2M         Analysis of therbutyl alcohol (TBA) in drinking water by EPA 524.2M           ORG078         1         Jan-02         528         Analysis of Selected SVOA in Drinking Water by SPE and GC/MS, EPA Method 528           ORG081         1         Jan-02         526         Analysis of Low Levels of 1.2,3-Trichloropropane by L-L extraction and GC/MS SIM           ORG083         1         May-02         TCP-PT         mode, SRL Method           ORG086         1	ORG069	6	Mav-08	7199	Analysis of Hexavalent Chromium by Ion Chromatography, EPA Method 7199	
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         GC-ECD, EPA Method 505           ORG074         2         Jul-02         General         Establishing Retention Times Windows for Organic Analyses by GC and GC/MS           ORG076         2         Mar-02         Instrument Maintenance for Organic Analysis         Orce CD, EPA Analysis of Havavalent Chronium in Water by Ion Chromatography, EPA 218.6           ORG076         1         Apr-01         552.2         Analysis of the-butyl alcohol (TEA) in drinking water by PA 524.2M           ORG078         1         Apr-01         524.2M         Analysis of The-butyl alcohol (TEA) in drinking water by PA 524.2M           ORG080         1         Jan-02         526         Analysis of Selected SVOA in Drinking Water by SPE and GC/MS, EPA Method 528           ORG081         1         Jan-02         526         Analysis of Low Levels of 1.2,3-Trichloropropane by L-L extraction and GC/MS SIM           ORG082         1         Apr-02         TCP-PT         mode, SRL Method         Analysis of Low Levels of 1.2,3-Trichloropropane by Purge and Trap and GC/MS SIM           ORG086         1         Jul-02         3535         Solid Phase Extraction Procedures - Manual and Automated, EPA Method 300.1 </td <td>ORG071</td> <td>2</td> <td>Mar-02</td> <td>8015B</td> <td>Analysis of Alcohols by GC-FID. EPA Method 8015B</td>	ORG071	2	Mar-02	8015B	Analysis of Alcohols by GC-FID. EPA Method 8015B	
ORG073         Sep-01         Analysis of Chlorinated Pesticides and PCBs in Drinking Water by Microextraction and ORG074         Analysis of Chlorinated Pesticides and PCBs in Drinking Water by Microextraction and GC-ECD, EPA Method 505           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         ORG076           ORG077         4         May-03         218.6         Analysis of Hexavalent Chromium in Water by Ion Chromatography, EPA 218.6           ORG078         1         Apr-01         524.2M         Analysis of PH and BTEX by GC/MS LUFT Method           ORG080         1         Jan-02         528         Analysis of PH and BTEX by GC/MS LUFT Method           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         mode, SRL Method           ORG083         1         May-02         TCP-PT         mode, SRL Method           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	ORG072	2	Mar-02	515.3	Analysis of Chlorinated Acid Herbicides in Water by Microextraction and GC-ECD, EPA Method 515.3	
ORG074         2         Jul-02         General         Establishing Retention Times Windows for Organic Analyses by GC and GC/MS           ORG076         2         Mar-01         552.2         Analysis of Haloacetic Acids by Microextraction and GC-ECD, EPA 552.2           ORG076         4         May-08         218.6         Analysis of Hexavalent Chromium in Water by Ion Chromatography, EPA 218.6           ORG077         4         May-08         218.6         Analysis of tert-butyl alcohol (TBA) in drinking water by EPA 524.2M           ORG079         1         Jun-02         528         Analysis of TPH and BTEX by GC/MS LUFT Method           ORG080         1         Jan-02         526         Analysis of Selected SVOA in Drinking Water by SPE and GC/MS, EPA Method 526           ORG081         1         Jan-02         526         Analysis of Low Levels of 1,2,3-Trichloropropane by L-L extraction and GC/MS SIM mode, SRL Method           ORG082         1         Apr-02         TCP-E         mode, SRL Method           ORG083         1         May-02         TCP-PT         mode, SRL Method           ORG086         1         Ju-02         3535         Solid Phase Extraction Procedures - Manual and Automated, EPA Method 3532           ORG087         2         May-08         300.1         Analysis of Low Levels of Oxyhalides by lon chromatography, EP	ORG073	3	Sep-01	505	Analysis of Chlorinated Pesticides and PCBs in Drinking Water by Microextraction and GC-ECD. EPA Method 505	
ORG075         2         Mar-01         552.2         Analysis of Haloacetic Acids by Microextraction and GC-ECD, EPA 552.2           ORG077         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 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 Low Levels of 1,2,3-Trichloropropane by SPE and GC/MS, EPA Method 526           ORG082         1         Apr-02         TCP-E         mode, SRL Method           ORG085         2         Aug-07         556         Analysis of Low Levels of 1,2,3-Trichloropropane by Purge and Trap and GC/MS SIM           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 lon chromatography, EPA Method 352           ORG089         1         Feb-04         1624         Analysis of Low Levels Construction and Cor/MS In SIM Mode, EPA	ORG074	2	Jul-02	General	Establishing Retention Times Windows for Organic Analyses by GC and GC/MS	
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 tern-burgl alcohol (TBA) in drinking water by EPA 524.2M           ORG079         1         Jan-02         528         Analysis of TPH and BTEX by GC/MS LUFT Method           ORG080         1         Jan-02         526         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-F         mode, SRL Method           ORG083         1         May-02         TCP-PT         mode, SRL Method           ORG086         1         Jul-02         3535         Solid Phase Extraction Procedures - Manual and Automated, EPA Method 3635           ORG087         2         May-08         300.1         Analysis of Low Levels of Oxyhalides by Ion chromatography, EPA Method 532           ORG089         1         Feb-04         1624         Analysis of Low Level Phenols in Water and Solid by GC/MS in SIM Mode, EPA           ORG098         2 </td <td>ORG075</td> <td>2</td> <td>Mar-01</td> <td>552.2</td> <td>Analysis of Haloacetic Acids by Microextraction and GC-ECD, EPA 552.2</td>	ORG075	2	Mar-01	552.2	Analysis of Haloacetic Acids by Microextraction and GC-ECD, EPA 552.2	
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         Iuft         Analysis of TPH and BTEX by GC/NS LUFT Method           ORG080         1         Jan-02         528         Analysis of TPH and BTEX by GC/NS LUFT Method           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           ORG083         1         May-02         TCP-PT         mode, SRL Method           ORG086         1         Jul-02         3535         Solid Phase Extraction Procedures - Manual and Automated, EPA Method 300.1           ORG086         1         Jul-02         3535         Solid Phase Extraction Procedures - Manual and Automated, EPA Method 300.1           ORG088         2         May-08         532         Analysis of Low Level of Oxyhalides by Ion chromatography, EPA Method 332           ORG089         1         Feb-04         1624         Analysis of Low Level Phenols in Water by EPA 1624           ORG	ORG076	2	Mar-02		Instrument Maintenance for Organic Analysis	
ORG078       1       Apr-01       524.2M       Analysis of trPh and BTEX by GC/MS LUFT Method         ORG080       1       Jan-02       528       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         ORG083       1       May-02       TCP-FE       mode, SRL Method         ORG086       1       Jul-02       3535       Solid Phase Extraction Procedures - Manual and Automated, EPA Method 3535         ORG086       1       Jul-02       3535       Solid Phase Extraction Procedures - Manual and Automated, EPA Method 300.1         ORG088       2       May-08       300.1       Analysis of Low Levels of Oxyhalides by Ion chromatography, EPA Method 532         ORG089       1       Feb-04       1624       Analysis of Low Level Phenols in Water by SPE and HPLC-UV, EPA Method 532         ORG089       1       Mar-04       8270SIM       Method 8270 Modified         ORG091       3       Jun-08       322       Analys	ORG077	4	May-08	218.6	Analysis of Hexavalent Chromium in Water by Ion Chromatography, EPA 218.6	
ORG079         Iuft         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           ORG083         1         May-02         TCP-FT         mode, SRL Method           ORG086         2         Aug-07         556         Analysis of Low Levels of 1,2,3-Trichloropropane by Purge and Trap and GC/MS SIM           ORG086         1         Jul-02         3535         Solid Phase Extraction Procedures - Manual and Automated, EPA Method 3535           ORG086         1         Jul-02         3535         Solid Phase Extraction Procedures - Manual and Automated, EPA Method 300.1           ORG088         2         May-08         300.1         Analysis of Low Levels of Oxyhalides by lon chromatography, EPA Method 302.1           ORG089         1         Feb-04         1624         Analysis of Low Level Phenols in Water by SPE and HPLC-UV, EPA Method 532           ORG090         1         Mar-04         8270SIM         Method 8270 Modified         Analysis of	ORG078	1	Apr-01	524.2M	Analysis of tert-butyl alcohol (TBA) in drinking water by EPA 524.2M	
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       Malysis of Low Levels of 1,2,3-Trichloropropane by L-L extraction 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 300.1         ORG086       2       May-08       300.1       Analysis of Low Levels of Oxyhalides by Ion chromatography, EPA Method 300.1         ORG087       2       May-08       532       Analysis of Low Level Phenols in Water by SPE and HPLC-UV, EPA Method 532         ORG089       1       Feb-04       1624       Analysis of Low Level Phenols in Water and Solid by GC/MS in SIM Mode, EPA         Mar09       3       Jun-08       326       Post-column derivatization, EPA Method 326         ORG091       3       Jun-08       326       Post-column derivatization, EPA Method 326         ORG092       2       Jan-08	ORG079			luft	Analysis of TPH and BTEX by GC/MS LUFT Method	
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       mode, SRL Method         ORG083       1       May-02       TCP-PT       Malysis of Low Levels of 1,2,3-Trichloropropane by Purge and Trap and GC/MS SIM         ORG083       1       May-02       TCP-PT       Analysis of Low Levels of 1,2,3-Trichloropropane by Purge and Trap and GC/MS SIM         ORG086       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 300.1         ORG087       2       May-08       300.1       Analysis of Low Levels of Oxyhalides by Ion chromatography, EPA Method 532         ORG089       1       Feb-04       1624       Analysis of Low Level Phenols in Water by SPE and HPLC-UV, EPA Method 532         ORG090       1       Mar-04       8270SIM       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       Jan-08       OSHA 20M       Analysis of Hydrazine by HPLC, OSHA Method 200M (Modified)         ORG093 </td <td>ORG080</td> <td>1</td> <td>Jan-02</td> <td>528</td> <td>Analysis of Phenols in Drinking Water by SPE and GC/MS, EPA Method 528</td>	ORG080	1	Jan-02	528	Analysis of Phenols in Drinking Water by SPE and GC/MS, EPA Method 528	
Analysis of Low Levels of 1,2,3-Inchloropropane by L-L extraction and GC/MS SIM         ORG082       1       Apr-02       TCP-E       mode, SRL Method         ORG083       1       May-02       TCP-PT       mode, SRL Method         ORG086       2       Aug-07       556       Analysis of Low Levels of 1,2,3-Trichloropropane by Purge and Trap and GC/MS SIM         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       300.1       Analysis of Low Level of Oxyhalides by Ion chromatography, EPA Method 532         ORG089       1       Feb-04       1624       Analysis of Low Level Phenols in Water by SPE and HPLC-UV, EPA Method 532         ORG090       1       Mar-04       8270SIM       Method 8270 Modified         ORG092       2       Jan-08       326       Post-column derivatization, EPA Method 326         ORG094       2       May-09       8316       Analysis of PBDEs by isotopic dilution GC/MS-EI, EPA Method 1614 Modified         ORG095       1       Sep-05       16144M       Analysis of Low Level Perchlorate by IC-MS/MS, EPA Method 331.0         ORG096	ORG081	1	Jan-02	526	Analysis of Selected SVOA in Drinking Water by SPE and GC/MS, EPA Method 526	
ORG002       1       Apj-02       TO-L       Index, State Inductors         ORG083       1       May-02       TCP-PT       Analysis of Low Levels of 1,2,3-Trichloropropane by Purge and Trap and GC/MS SIM mode, SRL Method         ORG085       2       Aug-07       556       Analysis of Low Levels of 1,2,3-Trichloropropane by Purge and Trap and GC/MS SIM mode, SRL Method         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 Low Level Phenols in Water by SPE and HPLC-UV, EPA Method 532         ORG089       1       Feb-04       1624       Analysis of Low Level Phenols in Water and Solid by GC/MS in SIM Mode, EPA         ORG090       1       Mar-04       8270SIM       Method 8270 Modified         Analysis of Low Level Chlorite, Chlorate and Bromate by Ion Chromatography and       Post-column derivatization, EPA Method 326         ORG092       2       Jan-08       326       Post-column derivatization, EPA Method 326         ORG094       1       May-09       8316       Analysis of Low Level Pherols or granotins by GC-MS.         ORG095       1       Sep-05	ORG082	1	Apr-02	TCP-F	Analysis of Low Levels of 1,2,3-Trichloropropane by L-L extraction and GC/MS SIM	
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ORG102         2         Infailuo         527         Analysis of Pesticides and Plane Relabants by SPE-GC/MS EPA Method 527           ORG103         2         Nov-08         529         Analysis of Explosives by SPE-GC/MS EPA Method 529           ORG104         1         May-06         300M         Analysis of lodide by Ion Chromatography, EPA Method 300 Modified           ORG105         1         Apr-06         LCMS         Tuning the Varian 1200 LC/MS	ORG101	1 0	Mar 09	527	Analysis of Nitrosamines by SPE-GC/MS/MS EPA Method 527	
ORG105         2         Norvoit         529         Analysis of Explosives by SPE-GC/NIS EPA Method 529           ORG104         1         May-06         300M         Analysis of Iodide by Ion Chromatography, EPA Method 300 Modified           ORG105         1         Apr-06         LCMS         Tuning the Varian 1200LLC/MS	ORG102	2	Nov-09	520	Analysis of resticides and riame relations by SPE-GU/MS EPA Method 52/	
ORG105 1 Apr-06 LCMS Tuning the Varian 1200 LC/MS	ORG103	∠1	May-06	300M	Analysis of Explosives by SEE-GU/NS EFA Method 300 Modified	
	ORG104	1	Apr-06		Tuning the Varian 1200LLC/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	
				Analysis of Pyrethroid Pesticides in Water and Soil/Sediment by Extraction and GC/MS in NCI	
ORG117	1	Nov-08	GCMS CI	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.

#### 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) 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 latest version of the test meth	od and SOP.
Analyst's Signature	Date	
□ Training courses or workshops o	on equipments, analytical techniques and la	b procedures:
Standard and sample preparation, of familiarization and operation of bo Ricci Tipon. GC and GC/MS semi-	lilution, and spiking using syringes and volum th software and hardware of GC/MS#1, 8(Ag nars provided by Full Spectrum and Tekmar.	netric flasks. On-site training for gilent 5890,6890)provided by
Analyst's Signature	Date	
Technical Director's Name and S	Signature Date	
$\square$ IDOC Certification Statement:	Julie Due	
Proficiency Demonstrated by: Accortable performance of a his	(See attachment)	
<b>b.</b> Another demonstration of capa	hility	
c. Acceptable at least 4 consecuti	ve LCS.	
<b>d.</b> Analysis of authentic sample a	nalyzed by another trained analyst with statistically indist	inguishable results
the undersigned, CERTIFY that:		
1 The Analyst identified above, using the cite	d test method(s), which is in use at this facility for the ana	lyses of samples under the
National Environmental Laboratory ac	creditation Program, have met the Demonstration of	of Capability
2 The test method(s) was performed by the an	alyst(s) identified on this certification.	
3 A copy of the test method(s) and the laborat	ory-specific SOPs are available for all personnel on-site	
4 The data associated with the demonstration	capability are true, accurate, complete and self-explanator	ry (*)
5 All raw data (including a copy of this certifi	ication form) necessary to reconstruct and validate these a	nalyses have been retained at
the facility, and that the associated info	ormation is well organized and available for review	by authorized assessors
Technical Director's Name and Signatu	ıre	Date
QA Officer's Name and Signature		Date

 \*: 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.

C	QUALITY ASSUR	ANCE N REPORT
Date:	Name of Analyst:	
Sample ID Number(s) Invo	olved:	
Corrective action to be in	nplemented (1):	
Were samples reanalyzed Were samples reported <b>:</b>	d and acceptable QC obtained: with qualifiers:	YES - NO YES - NO
Were samples reanalyzed Were samples reported Approval of corrective a	d and acceptable QC obtained: with qualifiers: ction by Technical Director:	YES - NO YES - NO
Were samples reanalyzed Were samples reported Approval of corrective ac Signed:	d and acceptable QC obtained: with qualifiers: ction by Technical Director:	YES - NO YES - NO Date:
Were samples reanalyzed Were samples reported Approval of corrective ac Signed: Technical I	d and acceptable QC obtained: with qualifiers: ction by Technical Director:	YES - NO YES - NO Date:
Were samples reanalyzed Were samples reported Approval of corrective ac Signed: Technical I Comments by TD:	d and acceptable QC obtained: with qualifiers: ction by Technical Director:	YES - NO YES - NO Date:
Were samples reanalyzed Were samples reported Approval of corrective ac Signed: Technical I Comments by TD:	d and acceptable QC obtained: with qualifiers: ction by Technical Director:	YES - NO YES - NO Date:
Were samples reanalyzed Were samples reported Approval of corrective ac Signed: Technical I Comments by TD: Verification of Implemen	d and acceptable QC obtained: with qualifiers: ction by Technical Director:  Director	YES - NO YES - NO Date:
Were samples reanalyzed Were samples reported Approval of corrective ac Signed: Technical I Comments by TD: Verification of Implemen	d and acceptable QC obtained: with qualifiers: ction by Technical Director:  Director	YES - NO YES - NO Date:
Were samples reanalyzed Were samples reported T Approval of corrective ad Signed: Technical I Comments by TD: Verification of Implemen Signed: QA Officer	d and acceptable QC obtained: with qualifiers: ction by Technical Director:  Director	YES - NO         YES - NO         Date:
Were samples reanalyzed Were samples reported Approval of corrective ac Signed: Technical I Comments by TD: Verification of Implemen Signed: QA Officer Comments by QA Officer:	d and acceptable QC obtained: with qualifiers: ction by Technical Director: Director	YES - NO         Date:

problem, probable cause of problem and how to prevent from happening again.

#### 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< th=""><th>&lt; 0.588</th></fis<>	< 0.588
_ <fl< th=""><th>No free liquids</th></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.
Е	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
	Due to the high concentration of analyte in the sample, sample extract was diluted prior to analysis. The
M-06	reporting limit was raised due to this dilution.
	Due to high concentration of solid particles in the sample, a smaller volume was used for analysis. The
M-07	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.
0-11	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.
	This sample was received unpreserved and with the recommended holding time for preservation of 48
O-25	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.
D 5	Due to the nature of the sample matrix a 1:10 dilution was necessary to perform a corrosivity
P-5	One or more quality control griteria foiled
Q	The recovery of this englyte in OC sample was outside control limits. Sample was justified as ND based on
Q-01	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) = $4 \text{ pCi/L}$ , and MCL (Maximum contaminant Level) = $50 \text{ pCi/L}$ .
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
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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.
T- AgBaHRP	The sample was treated with Silver, Barium, H+, and Organics cartridges to minimize chloride, sulfates, 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.