

APPENDIX U: Actual Water Resource Sampling and Analysis Plan

**WATER RESOURCES MONITORING
FIELD SAMPLING AND ANALYSIS PLAN
BLACK BUTTE COPPER PROJECT**

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**WATER RESOURCES MONITORING
FIELD SAMPLING AND ANALYSIS PLAN
BLACK BUTTE COPPER PROJECT**

1.0 INTRODUCTION

Baseline water resource monitoring has been conducted at the Black Butte Copper Project (Project) since 2011. The monitoring to date has been conducted in accordance with the 2013 Water Resource Monitoring Field Sampling and Analysis Plan (Hydrometrics, 2013). This Field Sampling and Analysis Plan (FSAP) provides an updated summary of the groundwater, spring/seep, and surface water monitoring activities to be conducted as part of the continued baseline water resource monitoring program for the BBC Project. The FSAP will also provide a basis for monitoring that will be conducted during future activities such as construction, operations, and closure for the proposed BBC Project. These future monitoring programs are will be finalized through the Mine Operating Permit application and development of the Environmental Impact Statement; once the future monitoring programs are finalized the FSAP will be updated in conjunction with the Record of Decision.

The FSAP is intended to provide guidance regarding sampling locations, sample collection methodologies, sample handling, documentation, and custody, and analytical requirements for groundwater, spring, and surface water samples collected as part of routine water resources monitoring. Any additional groundwater and/or surface water monitoring performed at the site will be conducted in accordance with the procedures set forth in this FSAP.

The water resources FSAP is structured as follows:

- Section 1.0 – Introduction;
- Section 2.0 – Sampling Locations and Frequency;

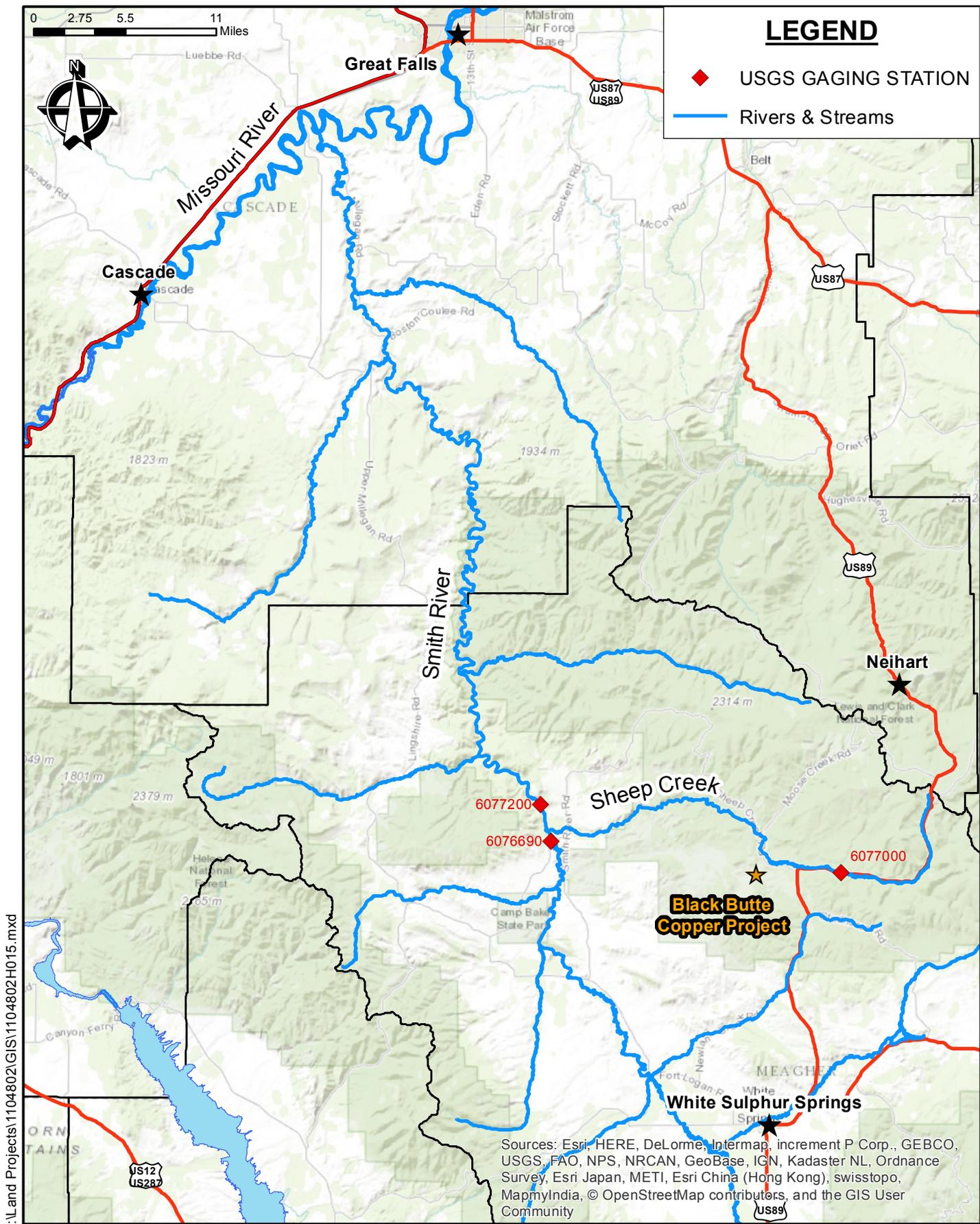
- Section 3.0 – Sampling Methodology;
- Section 4.0 – Sample Handling and Documentation;
- Section 5.0 – Laboratory Analytical Procedures and Reporting; and
- Section 6.0 – References.

1.1 PROJECT BACKGROUND

The Project is located approximately 16 miles north of White Sulphur Springs, Montana in Meagher County (Figure 1). The project is in the early stages of permitting an underground copper deposit and is collecting baseline data for use in project development. The ore body consists of a massive sulfide deposit within the Newland Formation of the Precambrian Belt Supergroup. The Newland Formation can be divided into a lower member that consists of primarily dolomitic shale and an upper member of interstratified shales and carbonates (Nelson, 1963). The project site lies within the Sheep Creek drainage.

Water resource monitoring conducted at the site will be used to establish baseline data including surface water flows, groundwater level elevations, and water quality in the vicinity of the project area to be used in project development and future permitting. Monitoring events will be conducted during each calendar quarter (i.e., January-March, April-June, July-September, and October-December time periods).

Groundwater, including springs and seeps, and surface water monitoring will be conducted in accordance with Hydrometrics' Standard Operating Procedures (SOPs). Water quality samples will be submitted to Energy Laboratories in Helena, MT for analyses of physical parameters, common constituents, nutrients, and a comprehensive suite of trace constituents. With the exception of aluminum, trace constituents will be analyzed for the total recoverable fraction for surface water samples; aluminum will be analyzed for the dissolved fraction. All trace constituents for groundwater samples will be analyzed for the dissolved fraction.



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Figure 1
Project Location Map
Black Butte Copper Project
Meagher County, Montana

2.0 SAMPLING LOCATIONS AND FREQUENCY

This section of the FSAP describes groundwater, spring/seep, and surface water sampling locations. Details on sampling methodologies, sample handling, and analytical requirements are presented in Sections 3.0, 4.0, and 5.0, respectively.

2.1 GROUNDWATER MONITORING

The groundwater quality monitoring will be conducted to establish pre-mining conditions in the vicinity of the proposed mine with an emphasis on the area surrounding the underground workings. The proposed underground mine will be accessed through a single decline which will penetrate dolomitic and silicic shales of the Newland Formation. There are upper (UCZ) and lower (LCZ) ore zones within the Johnny Lee copper-cobalt deposit hosted within the Upper and Lower Sulfide Zones (USZ, LSZ) of the lower Newland Formation. The upper ore zone lies at a depth of approximately 250 to 350 feet below ground surface and is overlain by shale and dolostone (Ynl A), and dolomite (Ynl 0) interbeds. The upper ore zone is underlain by the lower Newland shale and conglomerate (Ynl B). Quaternary alluvial deposits are present beneath the stream channels and along the axis of the drainages.

Quarterly groundwater monitoring is currently conducted at a total of 17 monitoring wells (Figure 2). A series of paired monitoring wells (MW-1A, -1B, MW-2A, -2B, MW-4A, -4B, and MW-6A, -6B) were installed between 2011 and 2013 to document baseline conditions within the unconsolidated Quaternary/Overburden and in the underlying shallow bedrock groundwater system. Monitoring well MW-3 was completed in November 2011 near the proposed terminus of the exploration decline within the UCZ. Paired wells MW-6A and MW-6B and two single wells (MW-7 and MW-8) were completed as groundwater quality and water level monitoring wells to document baseline water quality in the vicinity of the proposed underground LAD/infiltration system where treated mine water will be discharged. MW-6A is completed in shallow alluvial gravels, and MW-6B, MW-7, and MW-8 are completed in shallow YNL dolostone bedrock. In 2014, an additional monitoring well, MW-9, was installed in the YNL-A zone above the sulfide and ore zones as a monitoring

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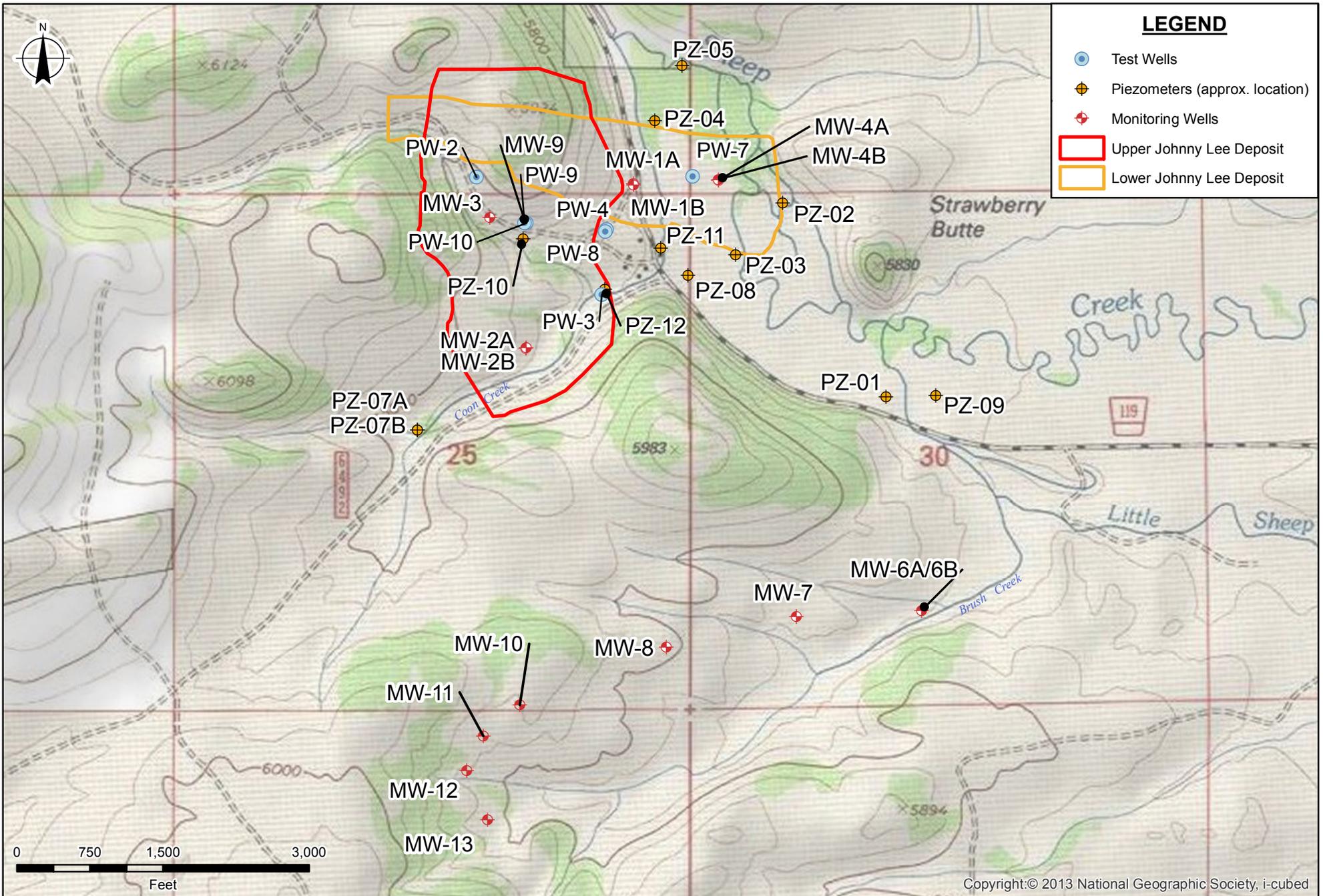


Figure 2
Groundwater Monitoring Locations
Black Butte Copper Project
Meagher County, Montana

point to assess the effects of ore zone dewatering on overlying units during mine development. MW-10, MW-11, MW-12, and MW-13 were installed in 2016 to assess water quality and quantity in the area of the proposed cemented tailings facility. MW-10, MW-11, and MW-12 are completed in granodiorite and MW-13 is completed in dolomitic shale. Well completion details for the monitoring wells are included in Table 1.

Seven additional test wells (PW-2, PW-3, PW-4, PW-7*, PW-8, PW-9, and PW-10) will be added to the quarterly monitoring during the third quarter of 2016 and thereafter to provide further water quality data for the groundwater resources in the vicinity of the proposed underground facilities. Well PW-7 has been observed to be contaminated with drill mud; therefore, it will not be included in the quarterly monitoring until it has cleaned and purged and shows no signs of drill mud in the water quality results. Wells PW-3 and PW-8 will provide additional data for the Ynl-A. Wells PW-2, PW-4, and PW-9 are completed in the USZ and UCZ. Additional data from the Ynl-B and LCZ will be provided from wells PW-10 and PW-7, respectively. The test wells are deep, four to six-inch wells and will produce significant volumes of water during purging; therefore, the wells will not be sampled during freezing conditions as water disposal is not possible during freezing conditions.

2.2 SPRING AND SEEP MONITORING

As a part of the initial water resource evaluation, nine seeps and 13 springs in the Project area have been identified, mapped, and some monitored for water quality and flow as a part of an inventory completed in 2011 (Hydrometrics, 2012). Seeps and springs are listed in Table 2 and their location is shown on Figure 3.

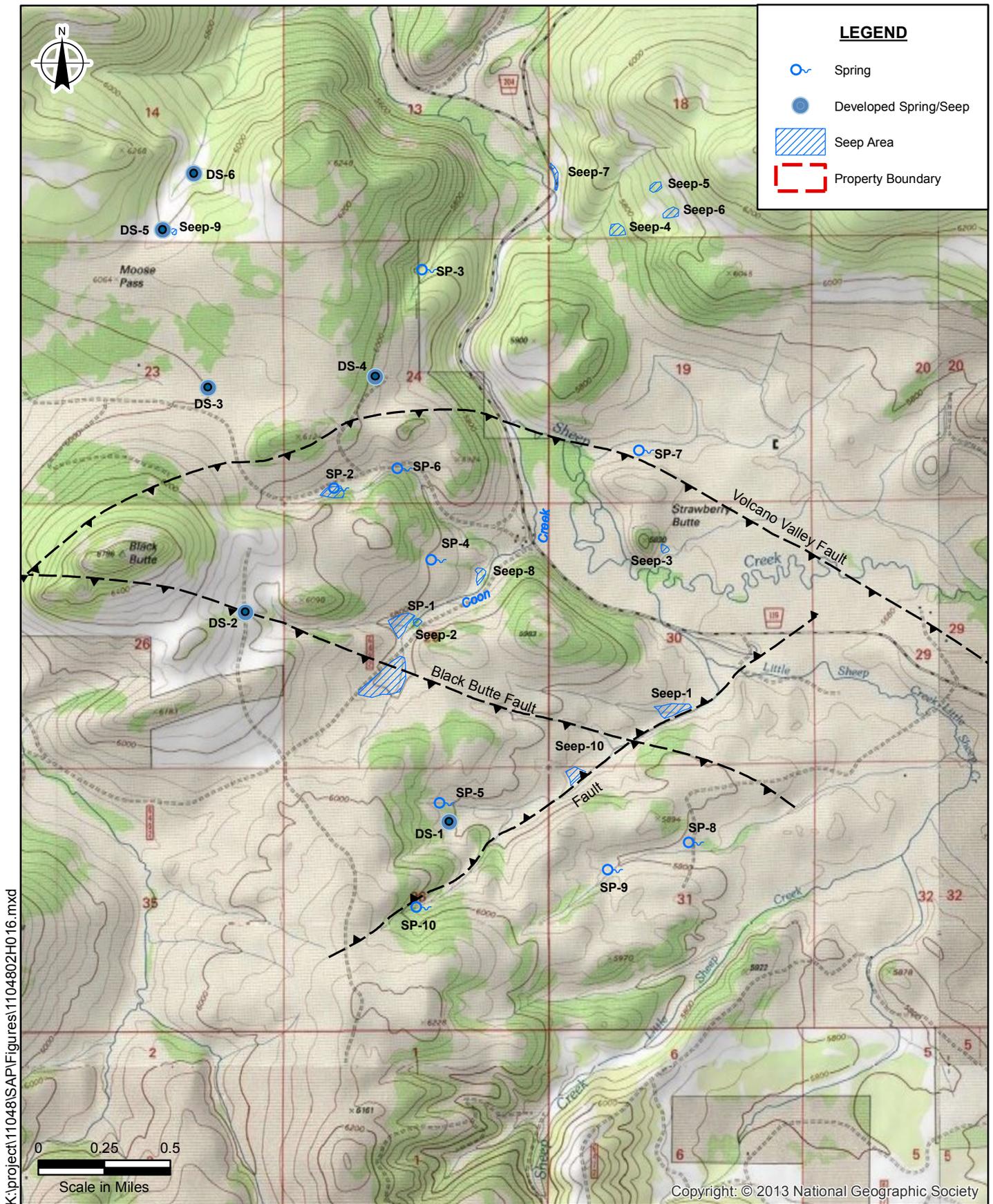
The majority of the identified sites consist of small springs or seeps located in ephemeral channels in the headwaters of small unnamed tributaries. These springs form small boggy areas with limited flow and generally re-infiltrate within a few hundred feet downstream. A number of these springs have been developed for stock watering (indicated by a DS designator in Figure 3 and Table 2) and feed small livestock watering tanks. The seeps are similar to springs, but are typically more disperse and have little to no measurable flow.

TABLE 1. WELL COMPLETION SUMMARY

Well Name	Northing (meters)	Easting (meters)	Ground Surface Elev.	Measuring Point Elev.	Borehole Total Depth	Well Total Depth	Screen Interval	Hydro- stratigraphic Unit	Year Drilled	Purpose
			(feet, amsl)							
UTM Zone 12 North					(feet, bgs)					
Monitoring Wells										
MW1A	5180841.55	506935.22	5635.81	5637.73	38	34	25 - 34	Overburden	2011	Baseline
MW1B	5180845.46	506934.19	5636.14	5637.9	98	98	88 - 98	YNL-A		East of USZ
MW2A	5180331.93	506598.18	5743.72	5745.31	62	62	52 - 62	Shallow Bedrock	2011	Baseline East of Coon Creek
MW2B	5180328.73	506596.96	5743.44	5745.53	80	80	70 - 80	YNL-A		
MW3	5180740.22	506484.07	5760.06	5762.17	305	305	285 - 305	USZ	2011	Baseline USZ
MW4A	5180855.43	507201.47	5610.12	5612.12	23	23	14-23	Sheep Creek Alluvium	2012	Baseline Sheep Cr. Alluvium
MW4B	5180858.49	507200.12	5610.07	5612.07	59	59	39-59	YNL-B	2012	Baseline YNL-B below Sheep Cr. Alluvium
MW-5	Not Drilled									
MW-6A	5179492.85	507809.18	5680.08	5681.87	20	15	5-15	Quaternary	2013	Proposed UG Infiltration Gallery
MW-6B	5179490.71	507792.76	5683.41	5685.31	50	50	40-50	Dolostone	2013	
MW-7	5179500.71	507451.7	5747.48	5749.46	50	50	40-50	Dolostone	2013	
MW-8	5179398.31	507036	5809.1	5810.93	80	80	70-80	Dolostone	2013	
MW-9	5180725.46	506592.96	5744.35	5745.8	143.7	128	108-128	YNL-A	2014	Baseline YNL-A Characterization
MW-10	5179215.05	506578.57	5882.78	5886.11	90	90	70-90	Granodiorite	2016	Baseline CTF
MW-11	5179117.47	506464.72	5854.74	5857.86	70	70	50-70	Granodiorite	2016	
MW-12	5179010.38	506412.82	5841.51	5844.75	60	60	40-60	Granodiorite	2016	
MW-13	5178855.81	506477.79	5819.07	5822.48	40	40	20-40	Dolostone	2016	
Test Wells										
PW-2	5180865.03	506443.15	5793.08	5794.88	215	212	132 - 212	USZ	2011	Previous Decline
PW-3	5180479.42	506846.43	5655.21	5657.42	131	127	90-127	YNL-A	2012	Baseline near Decline
PW-4	5180701.75	506849.44	5678.13	5680.01	242	239	200-239	USZ	2012	
PW-7	5180867.59	507122.89	5609.11	5611.15	1350	1346	1306-1346	LCZ	2013	Baseline LCZ Characterization
PW-8	5180695.53	506846.19	5679.12	5680.6	184	178.5	138.5-178.5	YNL-A	2014	Baseline YNL-A Characterization
PW-9	5180721.88	506598.38	5743.59	5745.05	255.5	255.5	215.5-255.5	UCZ	2014	Baseline UCZ Characterization
PW-10	5180721.88	506593.55	5743.57	5744.84	369.5	358.5	318.5-358.5	YNL-B	2014	Baseline YNL-B Characterization

TABLE 2. SPRING AND SEEP SITES

Monitoring Site	Easting (meters)	Northing (meters)	Monitoring Frequency	Flow or Water Level	Field Parameters	Water Quality
	UTM-WGS 1984 Zone 12 North					
Developed Springs						
DS-1	506507.08	5178870.81	Annual	X	X	X
DS-2	505263.49	5180150.61	Annual	X	X	--
DS-3	505037.62	5181520.61	Annual	X	X	X
DS-4	506056.53	5181588.64	Annual	X	X	X
DS-5	504761.45	5182484.96	Annual	X	X	--
DS-6	504949.66	5182827.88	Annual	X	X	--
Seeps						
Seep-1	507876.19	5179570.54	Annual	--	X	--
Seep-2	506310.60	5180089.20	Annual	--	X	--
Seep-3	507821.16	5180537.25	Annual	--	X	--
Seep-4	507530.57	5182486.29	Annual	--	X	--
Seep-5	507768.38	5182748.77	Annual	--	X	--
Seep-6	507853.49	5182587.27	Annual	--	X	--
Seep-7	507155.40	5182821.06	Annual	--	--	--
Seep-8	506701.44	5180381.64	Annual	--	X	--
Seep-9	504825.48	5182475.68	Annual	--	X	--
Seep-10	507270.05	5179164.80	Annual	--	X	--
Springs						
SP-1	506273.00	5180099.00	Annual	X	X	--
SP-2	505833.97	5180907.34	Annual	X	X	--
SP-3	506370.58	5182241.55	Annual	X	X	X
SP-4	506425.17	5180468.94	Annual	X	X	X
SP-5	506478.82	5178985.42	Annual	X	X	X
SP-6	506219.58	5181027.89	Annual	X	X	X
SP-7	507693.69	5181137.92	Annual	X	X	X
SP-8	507995.89	5178745.24	Annual	X	X	--
SP-9	507502.03	5178577.92	Annual	X	X	--
SP-10	506335.42	5178351.00	Annual	X	X	--



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Figure 3
Spring & Seep Monitoring Locations
Black Butte Copper Project
Meagher County, Montana

Slightly larger spring and seep areas were identified along the lower reaches of Coon Creek and on Little Sheep Creek and support perennial flow on these lower stream reaches.

Springs and seeps are monitored annually during the third quarter water resource monitoring event. Field parameters are collected at all spring and seep sites and flow is measured at all springs. In addition, water quality samples are collected at select springs (Table 2).

2.3 SURFACE WATER MONITORING

The project site lies within the Sheep Creek drainage. Sheep Creek originates in the Little Belt Mountains at an elevation of about 7,600 feet and discharges to the Smith River approximately 34 river miles to the west at an elevation of 4,380 feet. The project area is approximately 17 miles above the confluence with the Smith River. Sheep Creek flows in a meandering channel through a broad alluvial valley upstream of the project site but enters a constricted bedrock canyon just downstream.

Primary tributaries to Sheep Creek in the immediate project area are Little Sheep Creek, and Coon Creek (Figure 4). There are also two un-named tributaries that collect flow from the far side of the valley (to the northeast) and discharge to Sheep Creek immediately upstream and downstream of Strawberry Butte. Black Butte Creek lies just to the southwest of the project area and flows to the west away from the site until it discharges to Sheep Creek further downstream.

Baseline surface water monitoring is conducted on thirteen surface water stations within the project vicinity on a quarterly basis. Quarterly monitoring is intended to document surface water conditions at a range of flows. All thirteen sites will be monitored for field parameters and flow (as described in Section 3.2); eight sites will be samples for water quality monitoring. The location and monitoring conducted at each surface water monitoring site is listed in Table 3. Figure 4 shows the location of each site.

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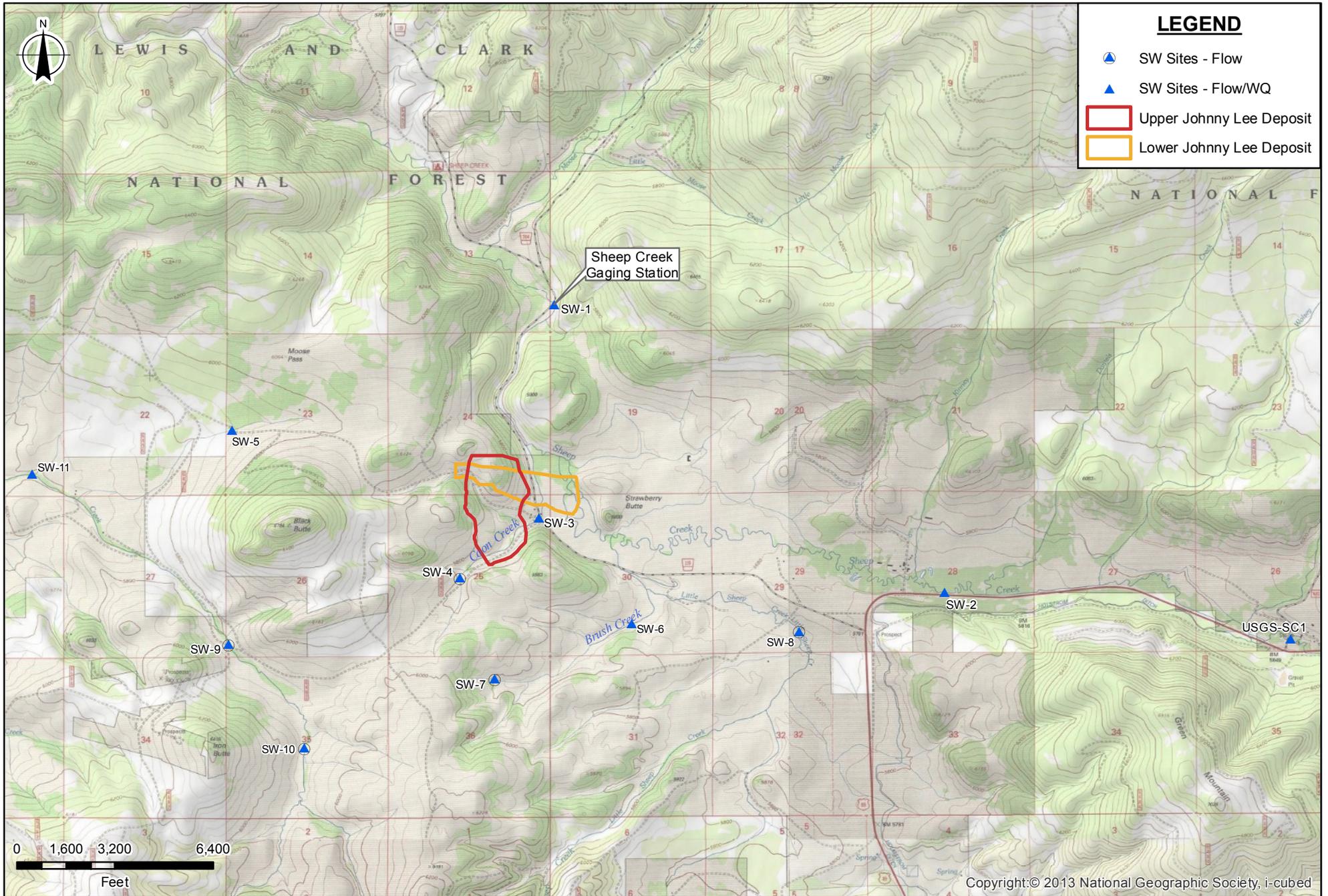


Figure 4
Surface Water Resource Monitoring Locations
 Black Butte Copper Project
 Meagher County, Montana

TABLE 3. SURFACE WATER MONITORING SITES

Site	Location	Northing	Easting	Field Parameters	Laboratory Analyses
		UTM - WRS 1984 (meters)			
SW-1	Sheep Creek - Downgradient site; at bridge on county road 119	5182710	507148	X	X
SW-2	Sheep Creek - Upgradient site; Highway 89 right away approximately 0.6 miles east of county road intersection	5179844	511040	X	X
SW-3	Unnamed Trib. to Sheep Creek - at intersection of county road 119 and forest service road.	5180581	506996	X	X
SW-4	Unnamed Trib. to Sheep Creek - approximately 0.6 miles southwest of Co. Rd/USFS Rd intersection	5180114	506308	X	
SW-5	Unnamed Trib. To Butte Creek - West of Moose Pass, where jeep trail crosses drainage.	5181465	503914	X	X
SW-6	Unnamed Trib to Little Sheep Creek - approximately 0.25 miles south of county road.	5179536	507919	X	X
SW-7	Unnamed Trib to Little Sheep Creek - Upgradient site, approximately 1-mile upgradient of SW-6.	5179000	506420	X	
SW-8	Little Sheep Creek - Approximately 0.5 miles from Highway 89.	5179476	509575	X	
SW-9	Butte Creek - at USFS road crossing.	5179271	503944	X	
SW-10	Butte Creek - approximately 0.7 miles upstream of SW-9.	5178322	504665	X	
SW-11	Butte Creek - Downgradient of confluence with Unnamed Trib to Butte Creek (west of Moose Pass).	5181021	501951	X	X
SW-14	Little Sheep Creek- Approximately 0.25 miles upstream of confluence with Sheep Creek	5180050	507876	X	X
USGS-SC1	Historical USGS station 06077000 ; approximately 4 miles upstream of the project site on Sheep Creek	514462	5179373	X	X

2.4 ADDITIONAL/FUTURE MONITORING

Additional water resource monitoring has consisted of spring and seep, surface water monitoring during spring runoff, and additional groundwater monitoring associated with hydrological investigations. As noted in Section 1.0, future monitoring programs are being developed in conjunction with the Mine Operating Permit application and the Environmental Impact Statement for different phases of the project (construction, operations, and closure). The monitoring conducted under these programs and any future programs will be conducted in accordance with this FSAP. A new FSAP will be developed for the different phases of the project once the future monitoring programs are completely developed and a record of decision has been finalized.

3.0 SAMPLING METHODOLOGY

The sampling methodologies to be utilized for groundwater, spring/seep, and surface water monitoring conducted as part of the quarterly monitoring program are detailed below: Groundwater Monitoring Section 3.1, Spring and Seep Monitoring 3.2, and Surface Water Monitoring Section 3.3. Spring and seep monitoring will be conducted in accordance with surface water monitoring for field parameters and sample collection methods; however, sample containers and preservation will be conducted in accordance with groundwater procedures. SOPs for performing field activities are located in Appendix A. Collection of field quality control (QC) samples for all sample media is discussed in Section 3.3.

3.1 GROUNDWATER MONITORING

Groundwater monitoring will include collection of field parameters and water quality samples from seven monitoring wells subsequent to well completion. The collection of groundwater samples from site monitoring wells will generally consist of three steps:

1. Measurement of static water level;
2. Well purging and monitoring for field parameter stabilization; and
3. Water quality sample collection.

3.1.1 Static Water Level Measurement

Prior to collection of samples or removal/introduction of any equipment into the well, the static water level will be measured at each well using an electric water level probe to determine the depth of groundwater below a specified measuring point (typically top of PVC). Water level measurements will be combined with surveyed measuring point elevations to compute groundwater elevations at each monitoring point.

3.1.2 Field Parameters and Water Quality Sample Collection

Dedicated HDPE tubing will be installed in monitoring wells, and a submersible pump will be used to purge and sample the monitoring well. Purging consisted of removing three well volumes while routinely monitoring field parameters (pH, dissolved oxygen (DO),

temperature, specific conductance (SC)) during removal of each well volume. Field meters will be calibrated daily according to factory instructions, with calibration results recorded on calibration forms. Purge water will be discharged in such a manner that it will not discharge to surface water.

Samples for laboratory analysis will be collected after one of the following purge conditions are met:

- A minimum of three well volumes have been removed and successive field parameter measurements agree to within the stability criteria given below; or
- At least five well volumes have been removed although field parameter stabilization criteria are not yet met; or
- The well has been pumped dry and allowed to recover sufficiently such that adequate sample volumes for rinsing equipment and collecting samples can be removed.

Criteria for field parameter stabilization are as follows:

Parameter (Units)	Stability Criteria
pH (standard units)	± 0.1 s.u.
Water temperature (°C)	± 0.2 °C
Specific conductance (µmhos/cm)	± 5% (SC ≤ 100 µmhos/cm) ± 3% (SC > 100 µmhos/cm)
Dissolved oxygen (mg/L)	± 0.3 mg/L

NOTE: Stability criteria obtained from USGS *National Field Manual for the Collection of Water Quality Data: Chapter A4, Collection of Water Samples* (September 1999).

Following well purging, final field parameter measurements will be collected and recorded, and groundwater quality samples obtained. Samples for trace constituents will be filtered through a 0.45 µm filter prior to preservation, to allow analysis for the dissolved fraction. Sample containers will be rinsed three times with sample water prior to sample collection, then preserved as appropriate for the intended analysis (e.g., nitric acid preservation to pH <2 for metals analysis), and stored on ice in coolers at approximately 4±2°C during transport.

Groundwater sampling equipment reused between monitoring locations (e.g., 12-volt sampling pump and short piece of discharge line) will be thoroughly decontaminated between uses. Equipment decontamination will consist of the following steps:

- Rinse with about two gallons of soapy water (Alconox or other non-phosphate detergent); and
- Rinse with about two gallons of distilled water.

3.2 SPRING AND SEEP MONITORING

Spring and seep monitoring will include generally consist of three steps:

- Collecting field parameters
- Water quality sample collection
- Flow measurement (excluding seeps)

3.2.1 Field Parameters

Spring and seep monitoring includes the collection of field parameters that consist of pH, SC, DO, and water temperature. Field parameters will be collected before spring flow measurements, or upstream of the location that spring flows will be measured to ensure the measurements are not affected by streambed disturbance.

Field meters will be calibrated daily according to factory instructions, with calibration results recorded in the field notebook and/or on calibration forms. Field parameter measurements will be obtained directly in the spring; however, in developed springs, field parameters will be taken in a clean container filled with sample water. Results will be recorded in the field notebook.

3.2.2 Water Quality Sampling

Water quality grab samples will be collected from spring and seep monitoring sites by passing an uncapped sample container across the area of flow. Water quality samples will be collected in containers and preserved. Samples for trace constituents will be filtered through a 0.45 µm filter prior to preservation, to allow analysis for the dissolved fraction. Sample

containers will be rinsed three times with sample water prior to sample collection, then preserved as appropriate for the intended analysis (e.g., nitric acid preservation to pH <2 for metals analysis), and stored on ice in coolers at approximately $4\pm 2^{\circ}\text{C}$ during transport.

3.2.3 Flow Measurement

Spring flow measurements will be collected using an appropriate flume (e.g., 90° v-notch cutthroat flume) or visually estimated when the flow is too low to be able to use a flume. To measure spring flow, the flume will be placed and leveled in the channel of spring flow in a location where the full spring flow can be directed through the flume throat. Water depth or head measurements will then be collected at specified locations in the converging and (if applicable) diverging sections of the flume. The head measurements will be used to verify proper functioning of the flume and to calculate stream flow based on the water depth. When it is impracticable to use a flume, a visual flow estimate will be made. Visual flow estimates are typically less than two gallons per minute.

3.3 SURFACE WATER MONITORING

Surface water monitoring will include the collection of flows and field parameters at all 11 sites; water quality samples will be collected at six of the 11 monitoring sites. Below is a summary of the methodologies to be used for the surface water monitoring, which consists of the following steps:

1. Measurement of stream flow and stage (at sites instrumented with staff gages);
2. Collection of field parameters; and
3. Water quality sample collection (if required).

3.3.1 Flow Measurement

Surface water flow measurements will be collected using a Marsh-McBirney current meter and wading rod (area-velocity method), appropriate flume, or estimated using the float method (when it is unsafe to wade in the river).

The Marsh-McBirney current meter is used to measure stream flow at larger, wadeable stream sites. Measurement of stream flow will be performed in accordance with the area-velocity method developed by the USGS (USGS, 1977). In general, the entire stream width is divided into subsections and the stream velocity is measured at the midpoint of each subsection at a depth equivalent to six-tenths of the total subsection depth. The velocity in each subsection is then multiplied by the cross-sectional area to obtain the flow volume through each subsection. The subsection flows are then summed to obtain the total stream flow rate. Stream flow measurements are typically collected in a stream reach as straight and free of obstructions as possible, to minimize potential measurement error introduced by converging or turbulent flow paths.

Stream flow measurements on smaller streams will be obtained by using a portable 90° v-notch cutthroat flume. To measure stream flow, the flume will be placed and leveled in the streambed, and the full stream flow directed through the flume throat. Water depth or head measurements will then be collected at specified locations in the converging and (if applicable) diverging sections of the flume. The head measurements will be used to verify proper functioning of the flume and to calculate stream flow based on the water depth.

The float method can be used when larger streams are not safe to wade due to strong flow. This method tends to underestimate the flow due to slower velocity near the surface, but it is more accurate than a visual estimate.

This method requires a straight and uniform stretch within a stream reach for best results. Stakes or flagging will be placed at the high water line at a distance apart of approximately twice the length of the mean wetted width (>50 feet is preferred). The mean width (from the water's edge) and the mean depth are then estimated and recorded in the field notebook. The measured distance between stakes and a description and sketch of each stake's location is recorded in the field notebook. Photographs of both stakes are taken to record their location along the streambank and the water level.

Toss a small stick or other biodegradable floating object (i.e., an orange) heavy enough to stay in and move consistently with the main current into the middle of the stream above the upstream marker of the measured reach. Begin timing when the object passes the upstream marker. Count (with a watch or stopwatch) the seconds it takes the object to reach the downstream marker. The object must stay in the main current. If it does not, repeat the measurement. Complete three measurable floats.

Record the following information:

- Reach length (ft or m);
- Mean depth (ft or m);
- Mean width (ft or m); and
- Float times (sec).

Complete the following calculations on the Total Discharge Form for high flow:

- Cross-sectional area (m^2 or ft^2) = Mean width x Mean depth;
- Average float time (sec) = (Float time 1 + Float time 2 + Float time 3) / 3;
- Float velocity (ft/sec or m/s) = Reach Length / Average float time; and
- Discharge (ft^3/sec or m^3/sec) = Cross-sectional area x Float velocity.

3.3.2 Field Parameters

Surface water monitoring includes the collection of field parameters that consist of pH, SC, DO, and water temperature. Field parameters will be collected before stream flow measurements, or upstream of the location that stream flows will be measured to ensure the measurements are not affected by streambed disturbance.

Field meters will be calibrated daily according to factory instructions, with calibration results recorded in the field notebook and/or on calibration forms. Field parameter measurements will be obtained directly in the stream; however, in high velocity areas pH may be measured in a clean container filled with sample water to limit possible errors due to streaming potentials. Results will be recorded in the field notebook.

3.3.3 Water Quality Sampling

Water quality grab samples will be collected from surface water monitoring sites by passing an uncapped sample container across the area of flow. Sample containers will be rinsed three times with sample water prior to sample collection. Water quality samples will be collected in containers and preserved as summarized in Table 4.

TABLE 4. SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Parameters	Sample Containers	Preservative
Field Parameters	None	None
Common Constituents	500 mL HDPE	Cool to 4°C
Nutrients (Nitrate+Nitrite)	250 mL HDPE	H ₂ SO ₄ to pH <2 Cool to 4°C
Surface Water Trace Constituents (total recoverable, except dissolved for aluminum)	250 mL HDPE	Filter dissolved samples (0.45 µm) HNO ₃ to pH <2 Cool to 4°C

Following preservation, samples will be stored on ice in coolers at approximately 4±2°C for transport. Dissolved trace constituents will be filtered by passing unpreserved sample water through a 0.45 µm filter using a peristaltic pump. All raw sample containers, tubing and filters will be discarded after each use to eliminate any cross contamination.

All water quality sampling information, including sample sites, sample numbers, date and time of sample collection, field parameter measurements, flow measurements, and other notes and observations, will be documented in waterproof ink in a dedicated project field notebook. Photos will be taken at each site to document conditions at the time of sampling and to provide reference for future monitoring events.

3.4 FIELD QUALITY CONTROL

Field QC samples will be used to provide quality assurance for field sampling and subsequent laboratory analysis. Field QC samples will include collection of field duplicates, rinsate blanks, D.I. blanks.

Field Duplicates

Field duplicate samples are replicate samples from a single sampling location submitted to a laboratory for the same set of analyses. For the purposes of this project, field duplicates will be collected by filling two samples containers consecutively from the sampling location. Duplicates will be sent to the same laboratory, but identified with different sample numbers. One field duplicate for each sample type (groundwater, spring, surface water) will be collected during each monitoring event to evaluate the reproducibility of the field sampling protocols.

Field Blanks (Rinsate Blanks and D.I. Blanks)

Rinsate (equipment) blanks will be collected for groundwater samples as there is not any equipment that is reused to collect spring or surface water samples. For groundwater samples, rinsate blanks will be collected each day and consist of deionized water processed through decontaminated sampling equipment (including filtration equipment as appropriate), collected into sample bottles and preserved. D.I. blanks will be collected for each monitoring event, and will consist of deionized water placed into sample containers and preserved.

4.0 SAMPLE HANDLING AND DOCUMENTATION

All samples transferred to the laboratory for analysis will follow standard documentation, packing, and chain-of-custody procedures. Samples will be stored in iced coolers or refrigerated following collection, then hand-delivered to the laboratory in iced coolers to maintain sample temperatures of approximately $4\pm 2^{\circ}\text{C}$. The SOPs for sample labeling, documentation and chain-of-custody procedures are in Appendix A of this document.

Sample custody (responsibility for the integrity of samples and prevention of tampering) will be the responsibility of sampling personnel until samples are shipped or delivered to the laboratory. Any containers used to ship samples via independent courier will be sealed with custody seals prior to shipping and the receiving laboratory will record the condition of the seals upon arrival to ensure that the containers have not been opened during transport. Custody seals are not required for samples that are maintained under the direct custody of sampling personnel until being hand-delivered to the laboratory. Upon arrival at the laboratory, sample custody shifts to laboratory personnel, who are responsible for tracking individual samples through login, analysis, and reporting. At the time of sample login, the laboratory will assign a unique laboratory sample number, which can be cross-referenced to the field sample number and used to track analytical results.

Documents generated during sample collection will consist of:

1. Sample collection field notes and forms;
2. Chain-of-Custody forms; and
3. Shipping receipts in the event that samples are sent to a laboratory via independent courier.

Sampling activities will be recorded in a project-specific field notebook. Each sample will be identified with a unique sample number, along with the date and time of collection, on adhesive labels attached to sample bottles. All labels will be completed using waterproof ink.

Field notebooks used to record pertinent sampling information will include, at a minimum, the following:

- Project name;
- Date and time;
- Sample location;
- Sample number;
- Sample depth (if applicable);
- Media type;
- Field meter calibration information;
- Sampling personnel present;
- Analyses requested;
- Sample preservation;
- Field parameter measurements;
- Weather observations; and
- Other relevant project-specific site or sample information.

Entries will be made in permanent ink. Corrections to field notebooks will be made by crossing out erroneous information with a single line and initialing the correction. Field books will be signed and dated at the bottom of each page by personnel making entries on that page.

Individual samples (including QC samples) will be assigned unique sample numbers according to the following sample numbering scheme:

AAA[A]-YYMM-XXX

where AAA[A] is a three- or four-character code denoting the project, YYMM is a four-digit code denoting the year and month (e.g., 1109 for September 2011), and XXX is a three-digit code that is incremented sequentially for each successive sample.

5.0 LABORATORY ANALYTICAL PROCEDURES AND REPORTING

Laboratory analysis will be conducted by Energy Laboratories' Helena, Montana branch. Energy Laboratories is certified by EPA Region 8 and the State of Montana under the Safe Drinking Water Act. Field parameters will be analyzed by Hydrometrics' field personnel using the procedures outlined in Sections 3.1.2 and 3.2.2 above, and in the applicable SOPs collected in Appendix A of this document. All laboratory analysis will be fully documented and conducted in accordance with EPA-approved and/or industry standard analytical methods.

5.1 GROUNDWATER, SPRING, AND SEEP ANALYSES

Required parameters, analytical methods, and project-required detection limits for groundwater quality samples collected from wells and springs are shown in Table 5. Groundwater samples, including spring samples, will be analyzed for physical parameters, common constituents, Nitrite + Nitrate, and a comprehensive suite of trace constituents. The project required detection limits (PRDLs) for individual parameters have been set at concentrations normally achievable by routine analytical testing in the absence of unusual matrix interference (laboratory's practical quantitation limit). It must be recognized that the PRDL is a detection limit goal, which may not be achieved in all samples due to sample matrix interference or other problems. If a PRDL is not met by the laboratory, the data will be reviewed to determine if any actions (e.g., sample reanalysis or selection of an alternative analytical method) are required.

**TABLE 5. ANALYTICAL METHODS AND DETECTION
LIMITS FOR GROUNDWATER SAMPLES**

Parameter	Analytical Method⁽¹⁾	Project-Required Detection Limit (mg/L)
Physical Parameters		
TDS	SM 2540C	10
TSS	SM 2540C	10
Common Ions		
Alkalinity	SM 2320B	4
Sulfate	300.0	1
Chloride	300.0/SM 4500CL-B	1
Fluoride	A4500-F C	0.1
Calcium	215.1/200.7	1
Magnesium	242.1/200.7	1
Sodium	273.1/200.7	1
Potassium	258.1/200.7	1
Nutrients		
Nitrate+Nitrite as N	353.2	0.01
Trace Constituents (Dissolved)⁽²⁾		
Aluminum (Al)	200.7/200.8	0.009
Antimony (Sb)	200.7/200.8	0.0005
Arsenic (As)	200.8/SM 3114B	0.001
Barium (Ba)	200.7/200.8	0.003
Beryllium (Be)	200.7/200.8	0.0008
Cadmium (Cd)	200.7/200.8	0.00003
Chromium (Cr)	200.7/200.8	0.01
Cobalt (Co)	200.7/200.8	0.01
Copper (Cu)	200.7/200.8	0.002
Iron (Fe)	200.7/200.8	0.02
Lead (Pb)	200.7/200.8	0.0003
Manganese (Mn)	200.7/200.8	0.005
Mercury (Hg)	245.2/245.1/200.8/SM 3112B	0.000005
Molybdenum (Mo)	200.7/200.8	0.002
Nickel (Ni)	200.7/200.8	0.001
Selenium (Se)	200.7/200.8/SM 3114B	0.0002
Silver (Ag)	200.7/200.8	0.02
Strontium (Sr)	200.7/200.8	0.0002
Thallium (Tl)	200.7/200.8	0.0002
Uranium	200.7/200.8	0.008
Zinc (Zn)	200.7/200.8	0.002
Field Parameters		
Stream Flow	HF-SOP-37/-44/-46	NA
Water Temperature	HF-SOP-20	0.1 °C
Dissolved Oxygen (DO)	HF-SOP-22	0.1 mg/L
pH	HF-SOP-20	0.1 s.u.
Specific Conductance (SC)	HF-SOP-79	1 µmhos/cm

(1) Analytical methods are from *Standard Methods for the Examination of Water and Wastewater* (SM) or EPA's *Methods for Chemical Analysis of Water and Waste* (1983).

(2) Samples to be analyzed for dissolved constituents will be field-filtered through a 0.45 µm filter.

5.2 SURFACE WATER ANALYSES

Required parameters, analytical methods, and project-required detection limits for surface water quality samples collected at in the vicinity of the Project are shown in Table 6. Similar to groundwater, samples will be analyzed for physical parameters, common constituents, nutrients, and a comprehensive suite of trace constituents. As for groundwater, the PRDLs for individual parameters have been set at concentrations normally achievable by routine analytical testing in the absence of unusual matrix interference (laboratory's practical quantitation limit). If a PRDL is not met by the laboratory, the data will be reviewed to determine if any actions (e.g., sample reanalysis or selection of an alternative analytical method) are required.

**TABLE 6. PARAMETERS, METHODS, AND DETECTION LIMITS FOR
BASELINE SURFACE WATER MONITORING**

Parameter	Analytical Method ⁽¹⁾	Project-Required Detection Limit (mg/L)
Physical Parameters		
TDS	SM 2540C	4
TSS	SM 2540C	4
Common Ions		
Alkalinity	SM 2320B	4
Sulfate	300.0	1
Chloride	300.0/SM 4500CL-B	1
Fluoride	A4500-F C	0.1
Calcium	215.1/200.7	1
Magnesium	242.1/200.7	1
Sodium	273.1/200.7	1
Potassium	258.1/200.7	1
Nutrients		
Nitrate+Nitrite as N	353.2	0.003
Total Persulfate Nitrogen	A 4500-N-C	0.04
Total Phosphorus	E365.1	0.003
Trace Constituents (SW - Total Recoverable except Aluminum [Diss], GW - Diss)⁽²⁾		
Aluminum (Al)	200.7/200.8	0.009
Antimony (Sb)	200.7/200.8	0.0005
Arsenic (As)	200.8/SM 3114B	0.001
Barium (Ba)	200.7/200.8	0.003
Beryllium (Be)	200.7/200.8	0.0008
Cadmium (Cd)	200.7/200.8	0.00003
Chromium (Cr)	200.7/200.8	0.01
Cobalt (Co)	200.7/200.8	0.01
Copper (Cu)	200.7/200.8	0.002
Iron (Fe)	200.7/200.8	0.02
Lead (Pb)	200.7/200.8	0.0003
Manganese (Mn)	200.7/200.8	0.005
Mercury (Hg)	245.2/245.1/200.8/SM 3112B	0.000005
Molybdenum (Mo)	200.7/200.8	0.002
Nickel (Ni)	200.7/200.8	0.001
Selenium (Se)	200.7/200.8/SM 3114B	0.0002
Silver (Ag)	200.7/200.8	0.02
Strontium (Sr)	200.7/200.8	0.0002
Thallium (Tl)	200.7/200.8	0.0002
Uranium	200.7/200.8	0.008
Zinc (Zn)	200.7/200.8	0.002
Field Parameters		
Stream Flow	HF-SOP-37/-44/-46	NA
Water Temperature	HF-SOP-20	0.1 °C
Dissolved Oxygen (DO)	HF-SOP-22	0.1 mg/L
pH	HF-SOP-20	0.1 s.u.
Specific Conductance (SC)	HF-SOP-79	1 µmhos/cm

(1) Analytical methods are from *Standard Methods for the Examination of Water and Wastewater* (SM) or EPA's *Methods for Chemical Analysis of Water and Waste* (1983).

(2) Samples to be analyzed for dissolved constituents will be field-filtered through a 0.45 µm filter.

5.3 DATA REVIEW AND REPORTING

All data deliverables containing analytical data and QC information will be reviewed for overall completeness of the data package. Completeness checks will be administered on all data to determine whether deliverables identified in this FSAP are present. At a minimum, deliverables will include field notes and/or forms, transmittal information, sample chain-of-custody forms, analytical results, methods and PQLs, and laboratory QC summaries. The reviewer will determine whether all required items are present and request copies of missing deliverables. Procedures for data review, validation, and reporting are discussed in HSOP-58 located in Appendix A.

The number and type of samples collected will be compared with project specifications. Review of sample collection and handling procedures will include verification of the following:

- Completeness of submittal packages;
- Completeness of field documentation, including chain-of-custody documentation;
- Field equipment calibration and maintenance and/or quality of field measurements; and
- Adherence to proper sample collection procedures.

Data validation will include a detailed review of all analytical results, including:

- Reporting limits (RLs) and PQLs vs. PRDLs;
- Holding times;
- Analytical methods;
- Field QC sample results; and
- Laboratory QC sample results.

6.0 REFERENCES

- EPA, 1983. Methods for Chemical Analysis of Water and Wastes. EPA-600/14-79-020. Revised March 1983.
- Hydrometrics, Inc., 2012. 2011 Spring and Seep Inventory, Black Butte Copper Project. January 2012.
- Hydrometrics, Inc., 2013. Water Resources Monitoring Field Sampling and Analysis Plan, Black Butte Copper Project. Revised March 2013.
- Nelson, W.H. 1963. Geology of the Duck Creek Pass Quadrangle, U.S. Geological Survey Bulletin 1121J, 56 p.
- USGS, 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Chapter 1: Surface Water.
- USGS, 1999. National Field Manual for the Collection of Water Quality Data: Chapter A4, Collection of Water Samples. September.

APPENDIX A

STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURES

HSOP-4	Chain-of-Custody Procedures, Packing and Shipping Samples
HSOP-29	Labeling and Documentation of Samples
HSOP-31	Field Notebooks
HSOP-58	Guidelines for Quality Assurance of Environmental Data Collection Activities Data Quality Planning, Review, and Management
HF-SOP-3	Preservation and Storage of Inorganic Water Samples
HF-SOP-10	Water Level Measurement With An Electric Probe
HF-SOP-11	Sampling Monitoring Wells For Inorganic Parameters
HF-SOP-19	Obtaining Water Quality Samples from Streams
HF-SOP-20	Field Measurement of pH Using a pH Meter
HF-SOP-22	Field Measurement of Dissolved Oxygen
HF-SOP-37	Streamflow Measurement Using a Marsh-McBirney Water Current Meter
HF-SOP-49	Use of a Flow Cell For Collecting Field Parameters
HF-SOP-73	Filtration of Water Samples
HF-SOP-79	Field Measurement of Specific Conductivity
HF-SOP-84	Field Measurement of Temperature

REVISION HISTORY

Revised by: *W. T. Walker* Date: 10/2010

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1.0 SCOPE AND APPLICATION

HSOP-4 presents procedures to be followed when shipping samples of environmental media (e.g., air, water, soil, waste material) to a laboratory for analysis. All samples submitted should be accompanied by chain-of-custody documentation.

2.0 SUMMARY OF METHOD

Samples of environmental media submitted to laboratories for analysis are often shipped via commercial carrier. Samples are packed in shipping containers to minimize the potential for container breakage or leaking. Each shipment will be accompanied by sample documentation, including chain-of-custody forms and a list of required analytical parameters, methods, and detection limits. Samples are cooled with ice during transport, to maintain temperature at approximately 4°C ($\pm 2^\circ\text{C}$). Shipments of hazardous materials must conform to International Air Transport Association (IATA) Dangerous Goods regulations and/or Department of Transportation (DOT) regulations, as well as any carrier-specific requirements.

3.0 HEALTH AND SAFETY WARNINGS

Field personnel should be aware of the health and safety precautions to be followed during any field event, and should be familiar with any project-specific hazards. This may include review of project-specific health and safety plans, site-specific and/or organization-specific safety requirements and training.

- Care should be exercised when handling samples of hazardous or potentially hazardous waste. Personal protective equipment (PPE) should be utilized (gloves, safety glasses, coveralls) as appropriate.
- Glass sample containers should be handled with extreme care to avoid breakage, loss of sample, and possible injury.

4.0 INTERFERENCES

Not Applicable

5.0 PERSONNEL QUALIFICATIONS

Personnel should be familiar with the project work plan and objectives, and with the operation of equipment listed in Section 6.0 below. Personnel should also familiarize themselves with the schedule of the shipping location to be used for shipping samples. For projects involving hazardous materials, consult the project work plan, courier regulations,

and any state and federal air or ground shipping regulations for details on shipping hazardous material.

6.0 EQUIPMENT AND SUPPLIES

- Shipping container (metal or plastic cooler);
- Packing material (bubble wrap, Styrofoam peanuts);
- Absorbent material (clay absorbents, rock wool);
- Shipping tape;
- Shipping strap;
- Custody seals;
- Chain-of-custody (COC) forms;
- Heavy-duty or contractor grade garbage bags or similar plastic bags;
- Ziploc bags; and
- Ice.

7.0 CHAIN-OF-CUSTODY PROCEDURE

1. Chain-of-custody involves ensuring that samples are traceable from the time of collection until received by the analytical laboratory. The laboratory is responsible for custody during processing and analysis. A sample is under custody if:
 - It is in your possession;
 - It is in your view, after being in your possession; or
 - It was in your possession and you then placed it in a designated secure or locked area to prevent tampering.
2. When ready to ship samples, set out samples in a clean, secure area to complete chain-of-custody forms. Chain-of-custody forms may be obtained from the project laboratory, or from Hydrometrics' Data Quality Department. An example COC form is shown in Attachment 1. Each sample should be identified on the form by its sample number, date and time of collection, and analysis requested. Check sample labels against information recorded in field notebook and on chain-of-custody to ensure consistency and guard against transcription errors (HSOP-29). It is usually best to use one chain-of-custody form per shipping container, covering the samples included in the container. When shipping multiple coolers to the laboratory, label chain-of-custody forms as "Cooler 1 of 3," "Cooler 2 of 3," etc. While chain-of-custody forms obtained from various sources may differ, certain information regarding sampling dates and times, sample identification, contact information, and requested parameters for analysis should be included on all acceptable forms. Complete all fields on the chain-of-custody form, as applicable to the

particular sampling event. Examples of typical COC information to be completed are as follows:

- a) **Company Name:** Enter “Hydrometrics, Inc.”
- b) **Project Name:** Enter the project name and Hydrometrics’ project number
- c) **Report Mail Address:** Enter the name, address, and e-mail address of the person who should receive the laboratory report.
- d) **Contact Name:** Enter the name of the project manager, sampling personnel, or other responsible contact.
- e) **Phone/Fax:** Enter the phone and fax number of the contact person for the project.
- f) **E-mail:** Enter the e-mail address for the contact person.
- g) **Sampler:** Print the name of the person who collected the samples.
- h) **Invoice Address:** Enter the address where the invoice should be sent.
- i) **Invoice Contact and Phone:** Enter the name and phone number of the person responsible for approving the invoice.
- j) **Purchase Order:** Enter the Hydrometrics’ Purchase Order number for the sample order.
- k) **Quote/Bottle Order:** Enter the laboratory quote number for the project or bottle order number provided with the sample bottle order.
- l) Note any special reporting requirements or formats.
- m) **Sample Identification:** Enter the unique sample number assigned to the sample.
- n) **Collection Date:** Enter the date each sample was collected. Do not use ditto (“) marks, arrows or lines to represent the same date.
- o) **Collection Time:** Enter the time each sample was collected. Do not use ditto (“) marks, arrows or lines to represent the same time.
- p) **Number of Containers and Matrix:** Enter the number of bottles the sample is contained in followed by a dash and then a letter representing the type of sample matrix (i.e. A=Air, W=Water, S=Soil/Solid, V=Vegetation, B=Bioassay, O=Other).

- q) **Analysis Requested:** Write the analysis to be performed on each sample and check the box for each sample you want to receive this analysis. Also include an analytical parameter list.
 - r) **Remarks:** Use this field to make notes or comments to the laboratory.
- (Note: If a laboratory-provided COC form is used, be sure to follow any additional instructions included from the laboratory.)
3. Record shipping information (tracking numbers, name of courier, other pertinent information) on chain-of-custody form. Sign and date chain-of-custody form, and retain one copy of form for project file.

8.0 PACKING AND SHIPPING PROCEDURE

1. Seal drain holes in bottom of shipping cooler (inside and out) to prevent leakage. Check sample container lids to ensure they are tightly sealed.
2. Line bottom of cooler with packing material (bubble wrap). Open and place two heavy-duty plastic bags in cooler (one inside the other).
3. Seal samples within individual plastic or bubble wrap bags, as necessary. All glass containers (VOAs, amber glass bottles, glass soil jars) should be placed in individual bubble wrap bags. Place sealed sample containers in shipping cooler, inside double plastic bags. In most instances, a labeled temperature blank should be included with the samples to allow the laboratory to check the sample temperature upon arrival. The temperature blank is generally a small vial or bottle filled with tap water and labeled "Temperature Blank." Ensure that temperature blank meets temperature requirements upon receipt by laboratory.
4. Cover samples with ice, inside double plastic bags.
5. Close and seal double plastic bags, by knotting or with shipping tape. Fill any empty space in cooler with additional packing material or absorbent material.
6. Record shipping information (tracking numbers, name of courier, other pertinent information) on chain-of-custody form. Sign and date chain-of-custody form, and retain one copy of form for project file.
7. Place original chain-of-custody, sample parameter list, cover letter, and any other documentation needed by the laboratory into a plastic Ziploc bag. Seal Ziploc bag and tape to the inside of the shipping container lid.

8. Label outside of shipping container with sampling organization name, address, and phone number, laboratory destination name, address, and phone number, and any required DOT shipping labels.
9. Place custody seals on front and back of cooler (see Attachment 2) and tape in place with shipping tape to avoid accidental breakage. Wrap cooler securely in at least two places with a minimum of three wraps of shipping tape. Shipping strap may also be used to provide additional insurance against the cooler opening during shipment.
10. Deliver sample containers to the shipping location. Since samples should reach the laboratory as soon as possible to protect sample integrity, **overnight shipping is required**, unless unavailable at the shipping location. Retain copies of shipping receipts for the project file. Shipping receipts and tracking numbers serve as chain-of-custody documentation during sample transport from the sampler to the laboratory.
11. Additional guidance may be found in the EPA's *Contract Laboratory Program Guidance for Field Samplers* (EPA, 2004). More stringent shipping requirements may apply to samples collected under CLP protocols. The project work plan should be consulted to determine any special requirements.

9.0 DATA AND RECORDS MANAGEMENT

The following documents generated during sample packing and shipping will be retained in the project file:

- Chain-of-custody form;
- Analytical parameter list;
- Cover letter; and
- Shipping receipts.

10.0 QUALITY CONTROL/QUALITY ASSURANCE

- Field personnel should cross-reference information on sample labels, in the field notebook, and on sample chain-of custody forms during the sample packing and shipping process.
- Data quality review will include checking of sample documentation to ensure consistency.
- Temperature blank measurements by the laboratory upon arrival of samples will document that samples were maintained at the appropriate temperature during shipping.

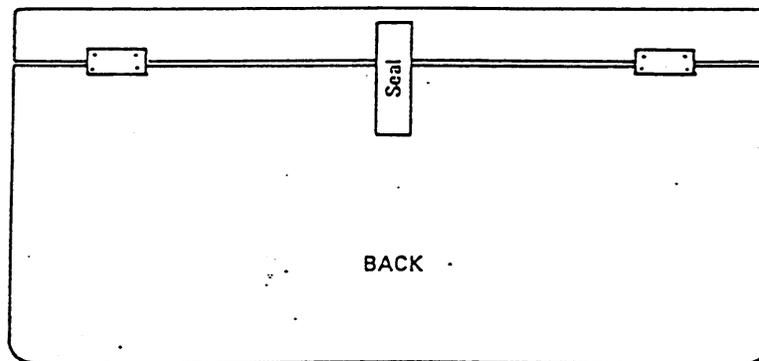
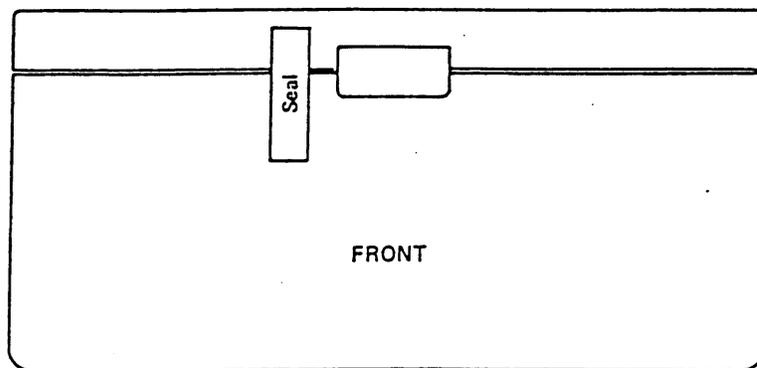
11.0 REFERENCES

EPA, 2004. Contract Laboratory Program Guidance for Field Samplers (Draft Final). EPA 540-R-00-003. January, 2004.

Hydrometrics HSOP-29: Labeling and Documentation of Samples

Attachment 2: Example of Custody Seals and Placement

 UNITED STATES ENVIRONMENTAL PROTECTION AGENCY OFFICIAL SAMPLE SEAL	SAMPLE NO.	DATE
	SIGNATURE	
	PRINT NAME AND TITLE (<i>Inspector, Analyst or Technician</i>)	
	SEAL BROKEN BY	DATE
		EPA FORM 7500-2 (R7-7B)



HSOP-29

LABELING AND DOCUMENTATION OF SAMPLES

Prepared by: *W. F. Walker* Date: 6/04

Reviewed by: *R. D. M.* Date: 6/04

Approved by: *M. L. M. Wright* Date: 6/04

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1.0 SCOPE AND APPLICATION

HSOP-29 describes typical procedures used to label sample containers, to ensure that information on the label is complete and correct, and to document the number and type of samples collected at a particular site. Samples must be thoroughly documented so that analytical data received from the laboratory can be correlated to the correct sampling site.

2.0 SUMMARY OF METHOD

Hydrometrics uses unique sample codes to identify individual samples. Sample codes are distinct from site identification codes, to ensure that the laboratory is unaware of the sample source, and whether the sample is a quality control (QC) or routine sample. Sample codes and other pertinent information is written on adhesive labels affixed to the sample container, or directly on the sample container in some cases. Sample documentation includes recording information in the field notebook (and on sampling forms if required), and completing chain-of-custody documentation for sample storage and shipping.

3.0 HEALTH AND SAFETY WARNINGS

Field personnel should be aware of the health and safety precautions to be followed during any field event, and should be familiar with any project-specific hazards. This may include review of project-specific health and safety plans, site-specific and/or organization-specific safety requirements and training.

4.0 INTERFERENCES

Some common problems with sample labeling and documentation might include the following:

- Use of incorrect sample numbers;
- Transcription errors during sample labeling or recording information in the field notebook; and
- Duplication of sample numbers.

These errors may be avoided by having an additional member of the sampling team check the labeling and documentation during the field event. If one person is conducting the sampling event, information entered on the sample label and in the field notebook should be double-checked for accuracy.

5.0 PERSONNEL QUALIFICATIONS

Labeling and documentation of samples should be conducted by personnel familiar with the project work plan and the proposed sample numbering scheme.

- performance evaluation sample, used to evaluate lab performance with a standard of known concentration) be identified as such on the sample label. QC samples are assigned sample numbers in the same manner as other samples.
4. When multiple sample containers are used at the same site due to differing preservation requirements or additional volume requirements, the same sample numbers should be used on each container.
 5. Due to requirements for cooling samples and field conditions, sample containers often become wet. If possible, it is advisable to place clear shipping tape over the label to ensure that it stays on the container. In addition, some sample information may be written on the sample lid, to aid in sample identification should the label become separated from the container.
 6. If required by the project, signed and dated seals may be placed over the container lid to prevent opening without breaking the seal.
 7. Sample information is recorded in the field notebook, including the same information recorded on the sample label (date and time, sample number, etc.), as well as identifying information for the sampling site, and QC sample information (see HSOP-31). If desired, sampling forms may also be used to record sampling information.
 8. On large projects, with multiple field sampling activities occurring at the same time, multiple field notebooks may be used to document sampling activities. Each notebook should clearly state in the initial entry what tasks will be recorded in the particular book.
 9. After collection and documentation, samples should be handled in accordance with standard chain-of-custody procedures (see HSOP-4).
 10. Any corrections made to sample labels, field notebooks, or chain-of-custody documentation should be made by crossing out the incorrect information with a single line, entering the correct information, and signing and dating the correction.

8.0 DATA AND RECORDS MANAGEMENT

Copies of all sample documentation, including field notebooks, sampling forms, and chain-of-custody forms will be maintained in the project file. Sampling crews are responsible for submitting this information to the Data Quality Department for filing at the completion of each sampling event.

9.0 QUALITY CONTROL/QUALITY ASSURANCE

- At the conclusion of the sampling event, field personnel should collate and review all sampling documentation materials for accuracy, prior to submitting the information to the Data Quality Department.
- Sample codes and associated sampling sites will be cross-referenced during data review and validation procedures stipulated by the project work plan and QAPP.
- Field samplers should ensure that complete documentation of samples has occurred prior to the close of sampling activities each day, by counting the number of samples collected and checking the field notebook for entries related to each sample.

10.0 REFERENCES

Hydrometrics HSOP-4: Chain-of-Custody Procedures, Packing, and Shipping Samples

Hydrometrics HSOP-31: Field Notebooks

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1.0 SCOPE AND APPLICATION

HSOP-31 presents general guidance on recording field activities in a dedicated project notebook. Field books are intended to provide sufficient data and observations to enable participants to reconstruct events that occurred during the implementation of the project. In legal proceedings, field notes are typically admissible as evidence and subject to cross-examination.

2.0 SUMMARY OF METHOD

Bound notebooks with sequentially numbered pages are used to record observations, sampling information, weather conditions, and other pertinent information during field activities. Entries are made in permanent ink, and signed and dated at the bottom of each page. Both original notebooks and copies of field notes are retained as part of the project file.

3.0 HEALTH AND SAFETY WARNINGS

Field personnel should be aware of the health and safety precautions to be followed during any field event, and should be familiar with any project-specific hazards. This may include review of project-specific health and safety plans, site-specific and/or organization-specific safety requirements and training.

4.0 INTERFERENCES

The primary potential problem with recording information in field notebooks is dealing with incorrect entries. In no case should erasures be made or information be obliterated or made illegible. Errors should simply be crossed out with a single line, dated, and initialed by the person making the original entry.

5.0 PERSONNEL QUALIFICATIONS

No specific qualifications are necessary for recording information in field notebooks. Personnel should be familiar with the scope and objectives of the project in order to record more meaningful field observations.

6.0 EQUIPMENT AND SUPPLIES

- Bound notebook with water resistant, sequentially numbered pages
- Pen (indelible ink)

7.0 PROCEDURE

1. New field notebooks should be labeled with the project title and number on the cover. Inside the front cover, write Hydrometrics' address and phone number as contact

- information, in case the notebook is lost. Multiple field notebooks may be required for large or ongoing projects; these should be assigned sequential numbers or labeled on the cover with the inclusive dates of observations recorded in the notebook (e.g., Project X, May 2002 through May 2004).
2. Notebook entries should begin on a fresh page for each day during a field event. While specific entry formats may vary with personal preference, the intent of the field notebook is to provide a daily record of significant events, observations, and measurements, as well as sampling information. All entries should be accompanied by date and time. Examples of information to be recorded in the field notebook includes:
 - Weather conditions;
 - Personnel on-site, including arrival and departure times and identities of visitors and observers;
 - Purpose of daily activities;
 - Site sketch maps;
 - Health and safety briefing information;
 - Field meter calibration information;
 - Identification and description of sampling sites (see HSOP-2); and
 - Descriptions of photos taken;
 - Communication logs;
 - Documentation of deviation from methods;
 - Sampling instrument decontamination records.

Sampling-specific information should include (see also HSOP-29):

- Sample number, date, and time;
- Site identifier;
- Description of sample containers, preservation, and sample collection method;
- Sample tag number (if applicable);
- Field parameter measurements and water calibration (static water level, total well depth, pH, specific conductance, water temperature, turbidity, color, odor, etc.); and
- Soil depth intervals and descriptions.

This list is not meant to be exhaustive, and other pertinent information should also be recorded in the field notebook as determined by field personnel.

3. The field notebook will be used to record communication with individuals on-site and on the phone that could result in a deviation from the SAP or that could impact the quality of the data being collected as part of the investigations.
4. Observations and measurements should be recorded in indelible ink, at the time they are made.

5. If erroneous entries are recorded, corrections should be made by deleting incorrect information with a single line, and dating and initialing the deletion in the notebook. Do not erase or obliterate incorrect entries, or remove pages from the notebook.
6. Blank and unused portions of notebook pages should be crossed out with a single line.
7. At the conclusion of the field event, review notebook entries, sign and date each page (if not already done), and photocopy notebook pages for inclusion in the project file. Original notebooks may be maintained in the project file, or in the files of individual field personnel at the discretion of the project manager.

8.0 DATA AND RECORDS MANAGEMENT

Copies of field notes are retained in the project file. Original field notebooks are maintained in the project file, or in the files of individual field personnel at the discretion of the project manager. Completed (filled) notebooks should be placed in the project files or the Data Quality Department notebook library, at the discretion of the project manager. Copies of field notebooks should be updated in project files at the end of each field event.

9.0 QUALITY CONTROL/QUALITY ASSURANCE

Standard procedure requires review of field notes by a person other than the person who recorded the field notes, prior to entering the information into the project files, to check for inaccurate, incomplete, or unclear entries, blank pages, or other problems with documentation. Peer review of notebook entries should also be conducted at least once per day during field activities.

10.0 REFERENCES

Hydrometrics HSOP-2: Determination, Identification, and Description of Field Sampling Sites

Hydrometrics HSOP-29: Labeling and Documentation of Samples

HSOP-58

**GUIDELINES FOR QUALITY ASSURANCE OF
ENVIRONMENTAL DATA COLLECTION ACTIVITIES
DATA QUALITY PLANNING, REVIEW, AND MANAGEMENT**

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1.0 PURPOSE AND APPLICABILITY

This Standard Operating Procedure (SOP) outlines Hydrometrics' standard data review and data management policies and procedures. These policies and procedures provide a general framework to guide the collection, analysis, technical review, and management of data obtained during an environmental investigation. Although the required level of rigor will vary based on individual project goals and objectives, some provisions for assessment of data quality and data usability should be incorporated into all projects involving collection and analysis of environmental samples. This SOP describes aspects of data review, validation, and management that are applicable throughout the full duration of a typical environmental investigation, from initial project planning through preparation and submittal of any final reports. Note that project-specific requirements for data review, data validation, and data management are frequently detailed in project planning documents such as Work Plans, Sampling and Analysis Plans (SAPs), and/or Quality Assurance Project Plans (QAPPs). The procedures outlined in this SOP are intended to function as a basis for development of project-specific requirements, and also to provide a fundamental set of review, validation, and management practices applicable to all environmental investigations.

2.0 ORGANIZATION AND RESPONSIBILITY

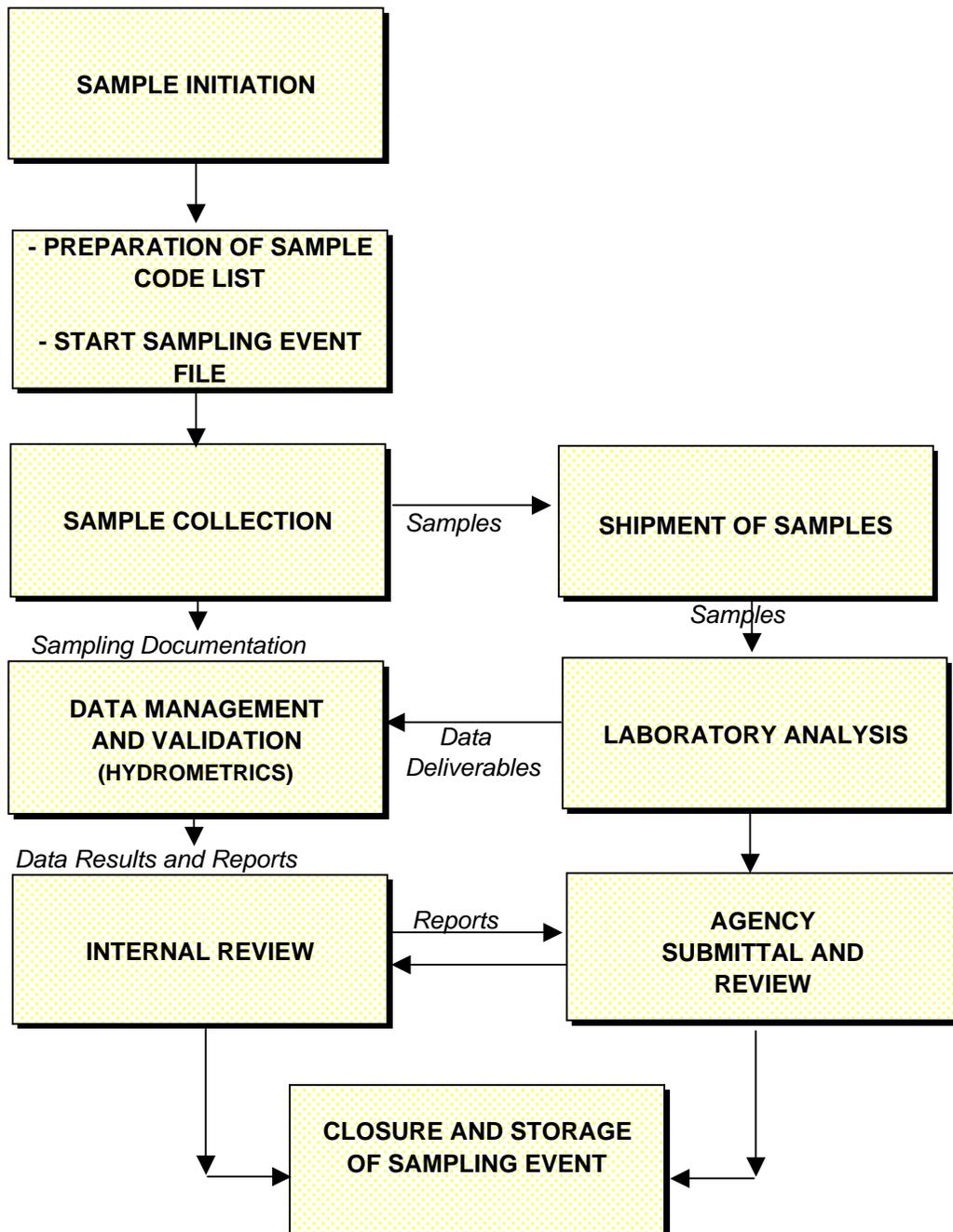
A QA manager is assigned to each individual project. The QA manager has the primary responsibility of overseeing implementation of field activities and laboratory analysis, to ensure that requirements in the project planning documents (Work Plan, SAP, QAPP) are met. These requirements may include specified field and laboratory methodologies, sample types and locations, data quality objectives, quality control sample types and frequencies, and data review, validation, and management procedures. At the direction of the client or QA manager, periodic audits may be performed to evaluate project-specific QA/QC and data management procedures and to provide an avenue for corrective actions.

The QA manager and project manager are responsible for assigning personnel to additional roles, including field team leaders and data quality review and management coordinators. Maintenance of complete and accurate field and laboratory documentation should be a focus of the QA team throughout the life of the project. The integrity of the data is maintained throughout all transfers and manipulations between principal data handlers/users. The flow of information is shown in Figure 1.

3.0 DATA QUALITY OBJECTIVES

Project-specific Data Quality Objectives (DQOs) should be developed during the project planning phase. The DQO process is designed to ensure that the type, quantity and quality of data collected during the investigation are appropriate for the intended application (EPA, 2006). The DQO process sets the stage for development and implementation of the project work plan.

FIGURE 1. SAMPLE INFORMATION AND DOCUMENTATION FLOW CHART



4.0 DATA REVIEW AND VALIDATION

Data review and validation involve the evaluation of the completeness, correctness and conformance of a specific data set against requirements set forth in the project planning documents (EPA, 2002). The level of review used for a particular data set will therefore depend on a comprehensive consideration of not only the intended end use and project objectives but also of project documentation requirements, QA/QC procedures, and inherent limitations in various sampling techniques and analytical methods. These levels are fairly fluid and can be customized to meet project requests/requirements. Table 1 lists Hydrometrics' established validation levels and their applications. Additionally, for any Montana Department of Environmental Quality CECRA site, the MDEQ's Data Validation Guidelines will be performed and will take precedence over any inconsistencies with this SOP. The MDEQ guidance document is located at <http://deq.mt.gov/StateSuperfund/PDFs/DataValidationReport.pdf>.

- **Level I - Visual Validation** - At this level the verification of completeness and accuracy of all sampling information takes place. This includes the following: confirming all results (both field and lab); all parameters, units and measurement basis, as being correct; cross checking of field notes and forms; and the verification of flow calculations. The results of this validation, at this level, are documented in a data review report memo. This level of validation generally corresponds to "data verification" as discussed in EPA (2002).
- **Level II - Standard Validation** - This level of validation encompasses the visual validation plus a more comprehensive review of all of the sampling information. The additional review includes the following: an examination of both field and laboratory QC (any laboratory QC that is included within the analytical package) using validation criteria limits as specified in the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic/Organic Data Review (EPA, 2010; 2008); a survey of the achievement of the project data quality objectives; qualification of the data per project requirements; data evaluation; historic trend comparison and/or graphs; ion balance; and statistical comparisons. The results of this validation, at this level, are documented in a comprehensive data review report.
- **Level III - (Contract Laboratory Program) CLP Validation** - At this level of review, both the visual and standard validation tasks are performed. Analytical data is characterized by rigorous QA/QC protocols and documentation. Validation procedures utilize such documentation as necessary to support project needs. Additional review requirements are: verification of the laboratory's raw data and quality control for frequency; accuracy; completeness; and procedures as required by the criteria limits specified in the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic/Organic Data Review.

Performance criteria for the following sampling and analytical specific data quality indicators (DQIs) for the precision, accuracy, representativeness, completeness and comparability

TABLE 1. VALIDATION LEVELS AND APPLICATIONS

VALIDATION LEVELS	APPLICATION
Level I - Visual Validation	<ul style="list-style-type: none"> • Verify Completeness and Accuracy of Input Data: <ul style="list-style-type: none"> - Results - Sampling Information - Parameters - Units - Measurement Basis • Cross Check Field Notes and Forms • Verify Flow Calculations • Report via Validation Memo
Level II - Standard Validation	<ul style="list-style-type: none"> • Visual Validation • Quality Control Review <ul style="list-style-type: none"> - Field Quality Control - Laboratory Batch Quality Control • Data Quality Objectives(DQO) Summary for Precision, Accuracy, Representativeness Comparability, Completeness (PARCC) • Qualify Data as per Project Requirements • Data Evaluation <ul style="list-style-type: none"> - Statistical Comparison - Ion Balance - Trend Comparison and Graphs • Report via Standard Comprehensive Data Review Report
Level III - CLP Validation (EPA, 2010; 2008)	<ul style="list-style-type: none"> • Visual • Standard • Quality Control Validation <ul style="list-style-type: none"> - Laboratory Quality Control - Field Quality Control • Verification with Raw Data <ul style="list-style-type: none"> - Frequency - Accuracy - Completeness - Procedure • Quality Data as per Project Requirements • Report via Standard Comprehensive Data Review Report

(PARCC) parameters are typically specified in the project SAP or QAPP. Assessment of these non-direct measurements provides the basis for the evaluation of overall data quality.

Precision Objective

Precision is defined as a measure of reproducibility of replicate measurements, and is inversely related to the variability among the results obtained (e.g., highly variable results have low precision). Precision of field duplicates is a measure of both field sampling variability and the laboratory analytical variability. Precision will be assessed using field and laboratory duplicates, and laboratory matrix spike duplicates.

Accuracy Objective

Accuracy is the agreement between a measured value and a 'true' value. Accuracy will be assessed using field trip blanks, field equipment/rinsate blanks, laboratory matrix spikes, laboratory control standards (LCS), laboratory method blanks, laboratory fortified blanks, and laboratory surrogate standard checks.

Representativeness Objective

Representativeness is the extent to which discrete measurements and testing accurately describe the environmental system. Representative data are achieved through careful selection of sampling sites, and proper sampling and analytical procedures.

Completeness Objective

Completeness is achieved when the number of valid measurements is sufficient to satisfactorily address all-important issues about the site. Completeness is assessed as the number of "valid" measurements. A "valid" measurement is one in which the sample was properly collected and considered representative of the material sampled, and which was not rejected during the data quality review process. Results qualified during the data quality review process as estimated will be considered valid measurements, unless extenuating circumstances or professional judgment indicate otherwise.

Comparability Objective

Comparability is the degree to which two or more data sets from the same site are generated using consistent procedures. Inherent compositional differences aside, discrete data sets may differ as a result of non-random (biased) sampling, variability in sampling technique, and variations in methods of analysis. To ensure comparability of data collected under the plan, the following actions will be implemented:

1. Standard Operating Procedures (SOPs) will be employed for sampling and analytical activities, as appropriate;
2. Field personnel will be thoroughly trained in sampling techniques;
3. Data results will be reported in standard units;
4. Data qualifiers will be consistent for all project data;

5. All sampling sites will be accurately delineated and recorded (HSOP-2); and
6. Analyses will be performed using EPA-accepted methods, as available and appropriate.

5.0 DATA MANAGEMENT ACTIVITIES

The process of collecting, analyzing, managing, tracking, evaluating, and reporting data involves many steps. The data management system for a project should address documentation requirements, document control and storage, and reporting formats. Figure 2 gives an overview of typical data management activities.

5.1 DOCUMENTATION

All sampling and analytical related project documents, field notes, laboratory analyses and/or testing results, as well as supporting documentation, should be maintained as part of the data management records organized by sampling events in the project file. Figure 3 outlines the flow of data documentation. The types of documentation that may be part of the data management records are as follows.

5.1.1 Field Sampling Documents

Field sampling documents contain all pertinent information recorded in the field and/or associated with samples collected in the field they include:

Calculated Flow Sheets	Field Sampling Forms	Transmittal Letter(s)
Calibration Logs	Shipping Records	Pump Tests
Parameter Lists	Well Logs	Sample Code List
Site Maps	Field Notebooks	

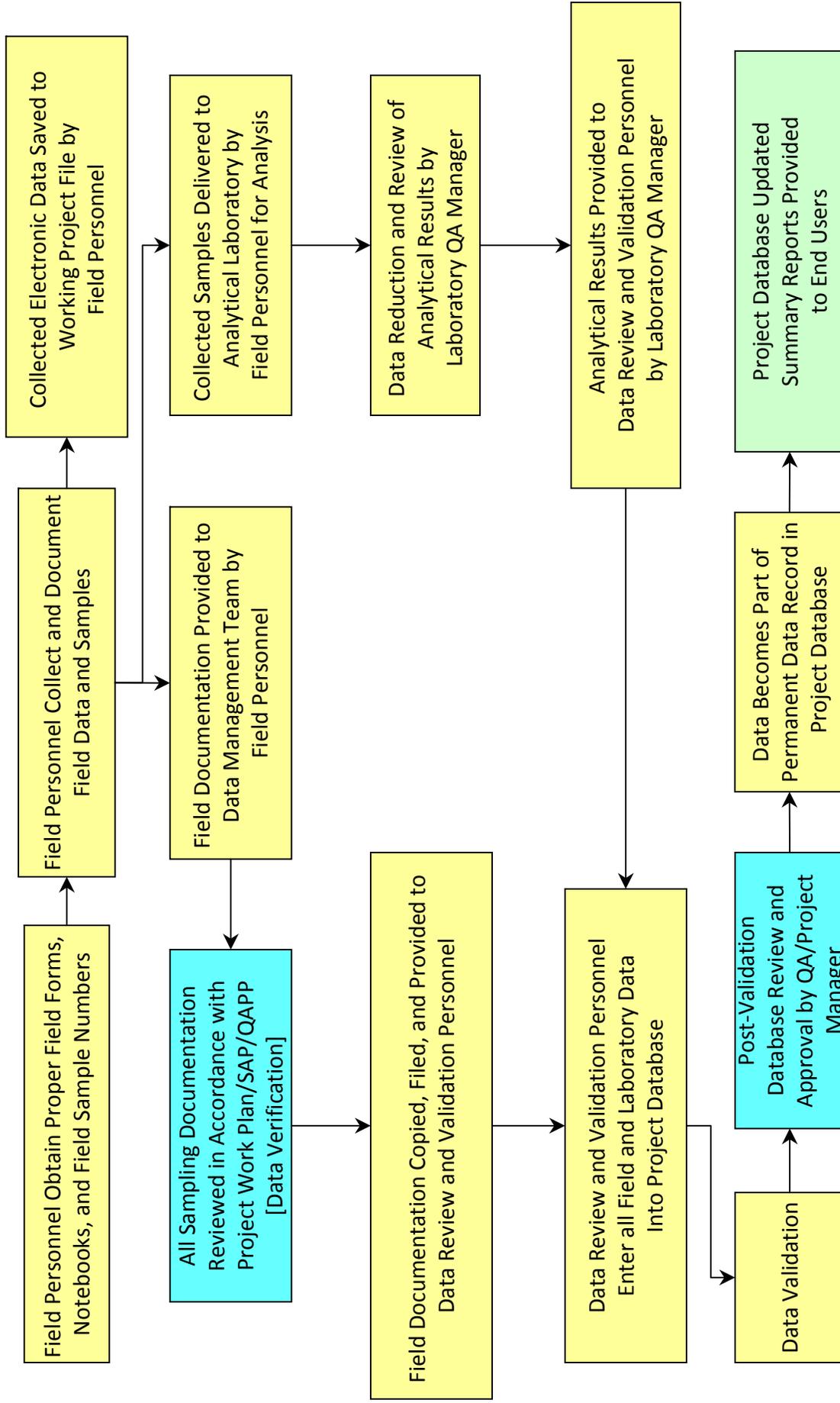
5.1.2 Laboratory Documents

Laboratory documents contain all pertinent information relating to the handling, processing, and subsequent analysis of the samples. Laboratory documents fall within the following categories:

- **Transmittal Records** - allow for tracking of the samples, and aid in communication between the laboratory and the Hydrometrics QA/QC personnel.

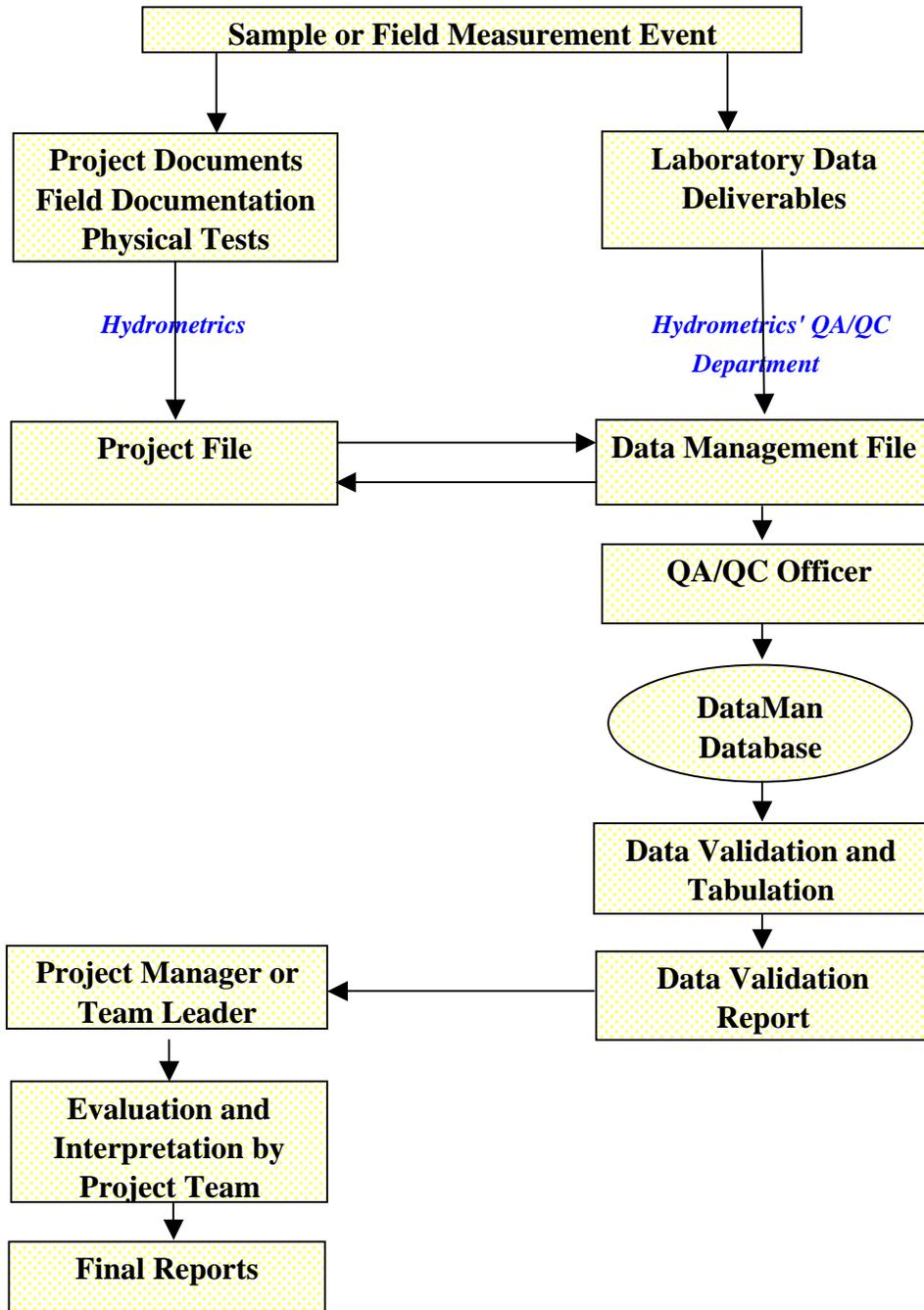
Cover Letter	Parameter List
Case Narrative	Sample Login Records
Chain of Custody Documents	Sample Preservation Check

- **Hard Copy Data Deliverables** - all deliverables received as part of the analytical package. The amount and type are dependent on the level of analysis and may range from a summarization of results to complete CLP deliverables (e.g., raw instrument output, lab bench logs, etc.).



GUIDELINES FOR QUALITY ASSURANCE OF ENVIRONMENTAL DATA COLLECTION ACTIVITIES DATA QUALITY PLANNING, REVIEW, AND MANAGEMENT	TYPICAL DATA MANAGEMENT ACTIVITIES	FIGURE 2
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FIGURE 3. DATA DOCUMENTATION FLOW



- **Electronic Data Deliverables** - allows for rapid transfer of laboratory data results into the specific client project database. Electronic deliverables contain analytical results and associated quality control data. Analytical results can be converted either into the Microsoft® FoxPro database program DataMan, developed by Hydrometrics, or data can be converted to other spreadsheet or database software.

5.1.3 Data Management Records

Data management records integrate client and project information with the field and laboratory data documentation for specific sampling events. The data management files may contain the following information.

A. Project specific client project information

- Work Plan
- Sampling and Analysis Plan (SAP)
- Quality Assurance Project Plan (QAPP)
- Site List
- Map
- Well Inventory
- Project Detection Limits
- Communications
- Any Other Relevant Project Information

B. Event specific files

- Field Documentation
- Laboratory Hard Copy Deliverables
- Electronic Deliverables
- Supporting Documentation
- Retest Requests
- Validation Reports
- Correspondence
- Communications

5.2 DATA STORAGE

Formats for handling data storage involve both electronic formats via the database system or spreadsheets, and physical hard copy files. The finalized data records and documents are always unique. A complete set of all project documents and data analyses will be stored in accordance with Hydrometrics' records management procedures, and/or as stipulated in the project QAPP or Data Management Plan. A set of project documents related to data or data analyses will also be stored at the originating Hydrometrics' office along with associated electronic files. All data documentation will be received by the Hydrometrics' QA/QC data management department to be entered into the data management files as appropriate, to allow efficient retrieval of information.

5.3 DATA/DOCUMENT RETRIEVAL

Retrieval of documents will be accomplished through the use of the data management filing system. Project data are maintained in project information files, and sampling event files, as well as the client database. Retrieval is quick and efficient with the use of these tools and can readily be provided in hard copy format and/or electronic format depending on client needs.

5.4 EXTERNAL DOCUMENT SOURCES

In order to maintain project information flow, it will be necessary to include any relevant project analytical/physical testing information generated by contractors or subcontractors. Analyses and documentation generated by external sources can be maintained in the data management system.

5.5 REPORTING

A schedule for reports will be established by the client and the project manager. The reporting schedule and specific report formats and content are normally outlined in the project work plan or contract.

Reports may include any of the following formats:

- General Information Summary - summarizes overall activity of the project.
- Status Report - updates the recipient as to the progress of specific activities.
- Data Evaluation/Interpretive Reports - includes and elaborates on topics covered in the General Information Summary; additionally, these reports highlight and may attempt to explain any data anomalies or trends that have been noted.
- Data Validation Reports - summarizes data quality in a formal report that is distributed both in-house and to external agencies.

5.6 SYSTEMS AUDIT/CONTROL

Database and electronic file security is controlled via network access limitations. Only authorized personnel have access to create or revise data files based on assigned user rights. A change log form documents all changes to the DataMan database files. Electronic data and document files are backed up daily.

Periodic system audits, if required by the client or oversight agencies, may be performed on field collection activities, laboratories and the data management activities. System audits are qualitative evaluations conducted for the purpose of determining compliance with the organizational and work element requirements for the specific client project activities. Performance will be assessed and non-compliance will be addressed and/or corrected. The schedule and content of the audits will be dictated by the client and QA or project manager.

6.0 REFERENCES

- EPA, 2002. Guidance on Environmental Data Verification and Data Validation (EPA QA/G-8). EPA/240/R-02/004. Office of Environmental Information. November 2002.
- EPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA QA/G-4). EPA/240/B-06/001. Office of Environmental Information. February 2006.
- EPA, 2008. USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review. USEPA-540-R-08-01. Office of Superfund Remediation and Technology Innovation. June 2008.
- EPA, 2010. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review. USEPA-540-R-10-011. Office of Superfund Remediation and Technology Innovation. January 2010.

STANDARD OPERATING PROCEDURE

PRESERVATION AND STORAGE OF INORGANIC WATER SAMPLES[®] HF-SOP-3

1.0 PURPOSE

An important factor in obtaining representative water quality data is the preservation and storage of samples. Preservation is designed to:

1. Retard biological activity;
2. Retard chemical reactions; and
3. Reduce volatility of constituents.

Preservation generally includes chemical additives, pH control, refrigeration, proper container materials, and immediate field filtration for dissolved constituents.

2.0 EQUIPMENT

Table 1 (attached) lists recommended preservatives, containers and holding times for various parameters. Be sure to assemble all the required containers, preservatives, and filters, as required, before leaving for the field.

3.0 PROCEDURE

In all cases where dissolved constituents are to be measured, the sample will be field-filtered through a 0.45 micron filter prior to addition of a preservative. Samples will be preserved according to guidelines presented in **Table 1**, and will remain refrigerated or in coolers with ice until analysis.

Complete sampling form for groundwater or surface water (**HF-FORM-430**).

4.0 REFERENCES

U.S. EPA, 1983. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, 3rd Edition.

TABLE 1. REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES

<u>Parameters</u>	<u>Container</u> ¹	<u>Preservative</u>	<u>Maximum Holding Time</u>
Specific Electrical Conductivity	T, P, G	Field determined	None
Total Dissolved Solids (TDS)	P, G	Cool, 4°C	7 Days
Total Suspended Solids (TSS)	P, G	Cool, 4°C	7 Days
pH	T, P, G	Field determined	None
Dissolved Oxygen (DO)	G bottle and top	None required	Analyze immediately
Temperature	P, G	None required	Analyze immediately
Eh	P, G	None required	Analyze immediately
Alkalinity	P, G	Cool, 4°C	14 days
Calcium (Ca)	P, G	HNO ₃ to pH <2	6 months
Magnesium (Mg)	P, G	HNO ₃ to pH <2	6 months
Sodium (Na)	P, G	HNO ₃ to pH <2	6 months
Potassium (K)	P, G	HNO ₃ to pH <2	6 months
Bicarbonate (HCO ₃)	P, G	Cool, 4°C	14 days
Carbonate (CO ₃)	P, G	Cool, 4°C	14 days

¹ T = Teflon; P = Polyethylene; G = Glass

TABLE 1 (Continued). REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES

<u>Parameters</u>	<u>Container</u> ¹	<u>Preservative</u>	<u>Maximum Holding Time</u>
Sulfate (SO ₄)	T, P, G	Cool, 4°C	28 days
Chloride (Cl)	T, P, G	Cool, 4°C	28 days
Silica (Si)	P	Cool, 4°C	28 days
Fluoride (F)	T, P	HNO ₃ to pH <2	28 days
<u>METALS*</u>			
Aluminum (Al)	T, P	HNO ₃ to pH <2	6 months
Antimony (Sb)	T, P	HNO ₃ to pH <2	6 months
Arsenic (As)	T, P	HNO ₃ to pH <2	6 months
Barium (Ba)	T, P	HNO ₃ to pH <2	6 months
Beryllium (Be)	T, P	HNO ₃ to pH <2	6 months
Cadmium (Cd)	T, P	HNO ₃ to pH <2	6 months
Chromium (Cr)	T, P	HNO ₃ to pH <2	6 months
Cobalt (Co)	T, P	HNO ₃ to pH <2	6 months
Copper (Cu)	T, P	HNO ₃ to pH <2	6 months
Iron (Fe)	T, P	HNO ₃ to pH <2	6 months
Lead (Pb)	T, P	HNO ₃ to pH <2	6 months
Manganese (Mn)	T, P	HNO ₃ to pH <2	6 months

¹ T = Teflon; P = Polyethylene; G = Glass

* Dissolved metals are filtered on site with 0.45 micron filter. Total metals are not filtered.

TABLE 1 (Continued). REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES

<u>Parameters</u>	<u>Container</u> ¹	<u>Preservative</u>	<u>Maximum Holding Time</u>
Mercury (Hg)	T, P	HNO ₃ to pH <2	28 days
Nickel (Ni)	T, P	HNO ₃ to pH <2	6 months
Selenium (Se)	T, P	HNO ₃ to pH <2	6 months
Silver (Ag)	T, P	HNO ₃ to pH <2	6 months (in dark place)
Tin (Sn)	T, P	HNO ₃ to pH <2	6 months
Thallium (Th)	T, P	HNO ₃ to pH <2	6 months
Vanadium (V)	T, P	HNO ₃ to pH <2	6 months
Zinc (Zn)	T, P	HNO ₃ to pH <2	6 months

PHOSPHORUS (P)

Orthophosphate (PO ₄), Dissolved	P, G	Filter on site, Cool, 4°C	48 hours
Orthophosphate, Total	P, G	Cool, 4°C	48 hours
Hydrolyzable	P, G	Cool, 4°C H ₂ SO ₄ to pH <2	28 days
Total	P, G	Cool, 4°C H ₂ SO ₄ to pH <2	28 days
Total, Dissolved	P, G	Filter on site Cool, 4°C H ₂ SO ₄ to pH <2	24 hours

¹ T = Teflon; P = Polyethylene; G = Glass

TABLE 1 (Continued). REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES

<u>Parameters</u>	<u>Container</u> ¹	<u>Preservative</u>	<u>Maximum Holding Time</u>
<u>NUTRIENTS</u>			
Ammonia	P, G	Cool, 4°C H ₂ SO ₄ to pH <2	28 days
Kjeldahl, Total	P, G	Cool, 4°C H ₂ SO ₄ to pH <2	28 days
Nitrate plus Nitrite	P, G	Cool, 4°C H ₂ SO ₄ to pH <2	28 days
Nitrate (NO ₃)	T, P, G	Cool, 4°C or Cool, 4°C H ₂ SO ₄ to pH <2	48 hours 14 days
Nitrite (NO ₂)	P, G	Cool, 4°C	48 hours

¹ T = Teflon; P = Polyethylene; G = Glass

Project Name: _____
 Project Code: _____
 Sample Team Member(s): _____
 Laboratory Used: _____

Site Designation: _____
 Sample Code Number: _____
 Sample Date: _____
 Sample Time: _____ (military)

**If Duplicate Sample Collected,
Please Record Below**

Duplicate Sample Code #: _____
 Duplicate Sample Time: _____

Site Conditions

New Site: Yes No Photo taken: Yes No
 Site Type: DRY surface water process water
 monitoring well domestic well adit seep
 spring- other: _____
 Weather Conditions: calm breeze windy
 no precip. rain snow
 clear p. cloudy overcast
 Air Temperature: _____ °C _____ °F

For Groundwater Samples

well volume formula:	$V = (TD-SWL) \times (Dia.^2) / 25$	Comments
TD (ft):	_____	
SWL (ft):	_____	no access/pumping
Casing Diameter (I.D.):	_____	
Water Volume (V) (gal):	_____	
x 3=(gal.)	_____	
Actual Vol. Removed (gal.):	_____	
Water Level Recovery:	slow moderate rapid	

For Surface Water Samples

Flow Method: Marsh McBirney Volumetric Flume Weir Estimate
 Other Flow or Description: _____
 Flow: gpm cfs Staff Gage: _____

Field Parameter Stabilization

Time (military)	Oxidation Reduction Potential (mV)	Dissolved Oxygen (mg/l)	pH	S.C. (µmhos/cm)	Turbidity (n.t.u.)	Temperature (°C)	Additional Parameters or Notes

Turbidity: (circle) clear slight moderate very Sample Method: (describe) grab composite large peristaltic pump bailer other

Field Parameters

Bottles Collected

	Sample	Duplicate	Quantity	Size	Filter or Unfilt.	Preservative	Parameter	Additional Notes
ORP (mV)				ml	F or UF			
DO (mg/l)				ml	F or UF			
pH				ml	F or UF			
SC (µmhos/cm)				ml	F or UF			
Turbidity (ntu)				ml	F or UF			
H ₂ O Tmp. (°C)				ml	F or UF			
Color				ml	F or UF			
Other: _____				ml	F or UF			
				ml	F or UF			

Comments: _____

Sample Team Member Signature: _____ Page _____ of _____

STANDARD OPERATING PROCEDURE

WATER LEVEL MEASUREMENT WITH AN ELECTRIC PROBE

HF-SOP-10

1.0 PURPOSE

This procedure applies to all water level measurements obtained using an electric probe. Normally, this procedure is used for measurement of water levels in wells. All electrical probes used, such as an Olympic Well Probe or Solinst, must have permanent depth markers placed at a minimum of every five feet on the probe wire or must have a direct reading tape.

2.0 EQUIPMENT

- Electronic probe;
- Water level measurement form (**HF-FORM-430**, Water Sampling Form);
- Field notebook; and
- Probe calibration data.

3.0 PROCEDURE

The water level is obtained by lowering the probe until contact is made between the probe tip and the water surface. The contact point is carefully checked by a slight lowering and raising of the probe and simultaneously observing the needle deflection, buzzer or light on the meter. For accurate measurements, the wire line must be straight as the probe is lowered. This is particularly important for the first few feet of line. Water depth is determined by direct reading of the probe wire or by measurement of the wire to the center of the nearest large marker and addition or subtraction from the marker value.

Water level measurements are referenced to the measuring point (MP). Normally, the MP is the top of a well casing but may be some other point. The MP used must be described. The north edge of the casing is usually marked or notched and all water level measurements are referred to this marked point.

3.1 CALIBRATION

All electric probes must be periodically calibrated. Normally, calibration is once or twice per year but, if the probe has been rebuilt, stretched, or replaced, it also must be recalibrated. For

recalibration, the electrical line is laid out on a flat surface and stretched to approximate its normal hanging weight. A steel tape graduated in 0.01 foot increments is used to determine probe accuracy. Additionally, the probe must be placed in wells with differing water levels and water depth measured and compared with a steel tape. A calibration record with correction factor is developed and placed in the equipment calibration file. This calibration record is used in the field to correct probe readings.

3.2 MEASUREMENT ACCURACY

All water levels and calibrations are normally measured to the nearest 0.01 foot. Probe data are considered accurate to 0.05 feet under good measurement and calibration conditions and to 0.10 feet under normal conditions. For deep or difficult conditions, accuracy may be less than 0.10 feet.

3.3 PROBE DECONTAMINATION

For projects where cross-contamination of wells may be a problem, the well probe and line must be decontaminated between measurement sites. This is particularly important when measuring wells containing substances such as PAH (polyaromatic hydrocarbons), pesticides, petroleum products and some metals.

Decontamination must include cleaning the probe and wire line. Most organics can be removed by wiping the line, then using detergent in water followed by acetone or methanol, followed by rinsing with DI (deionized) water.

Many inorganics can be removed by wiping the wire line and rinsing the probe in DI water. Specific attention must be paid to any sediment, rust or dirt on the wire line.

Project Name: _____
Project Code: _____
Sample Team Member(s): _____
Laboratory Used: _____

Site Designation: _____
Sample Code Number: _____
Sample Date: _____
Sample Time: _____ (military)

**If Duplicate Sample Collected,
Please Record Below**

Duplicate Sample Code #: _____
 Duplicate Sample Time: _____

Site Conditions

New Site: Yes No Photo taken: Yes No
 Site Type: DRY surface water process water
 monitoring well domestic well adit seep
 spring- other: _____
 Weather Conditions: calm breeze windy
 no precip. rain snow
 clear p. cloudy overcast
 Air Temperature: _____ °C _____ °F

For Groundwater Samples

well volume formula:	$V = (TD-SWL) \times (Dia.^2) / 25$	Comments
TD (ft):	_____	
SWL (ft):	_____	no access/pumping
Casing Diameter (I.D.):	_____	
Water Volume (V) (gal):	_____	
x 3=(gal.)	_____	
Actual Vol. Removed (gal.):	_____	
Water Level Recovery:	slow moderate rapid	

For Surface Water Samples

Flow Method: Marsh McBirney Volumetric Flume Weir Estimate
 Other Flow or Description: _____
 Flow: gpm cfs Staff Gage: _____

Field Parameter Stabilization

Time (military)	Oxidation Reduction Potential (mV)	Dissolved Oxygen (mg/l)	pH	S.C. (µmhos/cm)	Turbidity (n.t.u.)	Temperature (°C)	Additional Parameters or Notes

Turbidity: (circle) clear slight moderate very Sample Method: (describe) grab composite large peristaltic pump bailer other

Field Parameters

Bottles Collected

	Sample	Duplicate	Quantity	Size	Filter or Unfilt.	Preservative	Parameter	Additional Notes
ORP (mV)				ml	F or UF			
DO (mg/l)				ml	F or UF			
pH				ml	F or UF			
SC (µmhos/cm)				ml	F or UF			
Turbidity (ntu)				ml	F or UF			
H ₂ O Tmp. (°C)				ml	F or UF			
Color				ml	F or UF			
Other:				ml	F or UF			
				ml	F or UF			

Comments: _____

Sample Team Member Signature: _____ Page _____ of _____

STANDARD OPERATING PROCEDURE

SAMPLING MONITORING WELLS FOR INORGANIC PARAMETERS

HF-SOP-11

1.0 PURPOSE

This procedure describes the methods to be used in collection of groundwater samples from wells. The procedure is designed for wells where inorganic constituents are of primary concern. Methods presented in this SOP are based on recent USGS guidance (USGS, 1999).

2.0 EQUIPMENT

Bailers, submersible pumps, sample containers and water level electric probe. Other sampling equipment may be required for specific tasks. Other general equipment may include:

- Distilled or deionized water;
- Sampling sheets;
- Samplers notebook;
- Coolers;
- Preservatives;
- 0.45 µm filter apparatus with inert filters;
- Chemical-free paper towels;
- Properly cleaned sample containers of an appropriate volume; and
- Stopwatch or watch with second hand.

3.0 PROCEDURE

- A. Unlock and open well.
- B. Obtain water level measurement (see water level **HF-SOP-10**). If total well depth is unknown, measure total depth by sounding well. NOTE: electric water level probes are typically not recommended for sounding wells; instead, use a weighted measuring tape or other equipment.
- C. Calculate well volume (see calculation on HF-FORM-430) as $[(H) \times (D)^2] / 25$, where H = height of water column (feet), and D = well diameter (inches).

D. Purge well using an appropriate device (bailer, pump, etc.). Standard procedure involves removal of a minimum of three well volumes of water while monitoring field measurements and water level over time. In addition, purge volume should be adequate to remove water from the well annulus (filter pack). Record all pertinent purging information in field notebook and/or on field sampling forms, including:

- Purge method, rate, and total volume;
- Field parameter measurements;
- Water level changes (drawdown/recovery);
- Location of pump intake; and
- Other information.

The USGS (1999) recommends pumping or otherwise purging at a rate that does not significantly lower the water level. Toward the end of purging, a minimum of five sets of field parameters should be collected at regular intervals while pumping at the rate to be used for sampling. Use of a flow cell for field parameter monitoring is recommended. Field parameters are considered “stable” when the variability between five sequential measurements is as follows:

<u>Parameter</u>	<u>Stability Criteria</u>
pH	± 0.1
Temperature ($^{\circ}\text{C}$)	± 0.2
SC ($\mu\text{mhos/cm}$)	$\pm 5\%$ ($\text{SC} \leq 100$) or $\pm 3\%$ ($\text{SC} > 100$)
Dissolved oxygen (mg/L)	± 0.3
Turbidity (NTU)	$\pm 10\%$ ($\text{NTU} < 100$)

Modifications of the standard purge procedure are allowable if site conditions, the project work plan, or study objectives dictate such modifications. At a minimum, sufficient water must be removed to rinse equipment and sample bottles, and field measurements must be monitored prior to sampling. Low-flow (micropurge) techniques are discussed in a separate procedure (HF-SOP-105).

- E. Samples are collected after a sufficient purge volume is withdrawn and/or field parameters have stabilized and final field measurements have been collected. Bottles are filled directly from discharge from the well or from another clean container. Considerable care should be taken to minimize entrainment of air, particularly if bailers are used for sampling.
- F. Preserve and store samples as appropriate for the intended laboratory analysis. Collect final water level measurements if desired to determine water level recovery following purging.

4.0 DECONTAMINATION

If cross contamination of sampled wells is a potential problem, the following procedure should be followed:

- A. Design sampling to proceed from the best quality water to the poorest quality water; and
- B. Rinse the pumping apparatus or bailer between holes if well yields are too low to supply sufficient water to purge the pump, water hose or bailer.

If contamination is a problem, dedicated pumps or bailers should be used to ensure the samples are representative of site conditions (see Decontamination of Sampling Equipment **HF-SOP-7**).

5.0 ASSOCIATED DOCUMENTS

- A. Decontamination of Sampling Equipment (**HF-SOP-7**)
- B. Water Level Measurement with an Electric Probe (**HF-SOP-10**)

The following forms will be completed and retained in the project file:

- A. Water Sampling Form (**HF-FORM-430**);
- B. Chain-of-Custody Form (**HF-FORM-1**); and
- C. Shipping receipts.

6.0 REFERENCES

USGS, 1999. National Field Manual for the Collection of Water-Quality Data: Chapter A4, Collection of Water Samples. USGS TWRI Book 9, September 1999.

Project Name: _____
Project Code: _____
Sample Team Member(s): _____
Laboratory Used: _____

Site Designation: _____
Sample Code Number: _____
Sample Date: _____
Sample Time: _____ (military)

**If Duplicate Sample Collected,
Please Record Below**

Duplicate Sample Code #: _____
 Duplicate Sample Time: _____

Site Conditions

New Site: Yes No Photo taken: Yes No
 Site Type: DRY surface water process water
 monitoring well domestic well adit seep
 spring- other: _____
 Weather Conditions: calm breeze windy
 no precip. rain snow
 clear p. cloudy overcast
 Air Temperature: _____ °C _____ °F

For Groundwater Samples

well volume formula:	$V = (TD-SWL) \times (Dia.^2) / 25$	Comments
TD (ft):	_____	
SWL (ft):	_____	no access/pumping
Casing Diameter (I.D.):	_____	
Water Volume (V) (gal):	_____	
x 3=(gal.)	_____	
Actual Vol. Removed (gal.):	_____	
Water Level Recovery:	slow moderate rapid	

For Surface Water Samples

Flow Method: Marsh McBirney Volumetric Flume Weir Estimate
 Other Flow or Description: _____
 Flow: _____ gpm _____ cfs Staff Gage: _____

Field Parameter Stabilization

Time (military)	Oxidation Reduction Potential (mV)	Dissolved Oxygen (mg/l)	pH	S.C. (µmhos/cm)	Turbidity (n.t.u.)	Temperature (°C)	Additional Parameters or Notes

Turbidity: (circle) clear slight moderate very Sample Method: (describe) grab composite large peristaltic pump bailer other

Field Parameters

Bottles Collected

	Sample	Duplicate	Quantity	Size	Filter or Unfilt.	Preservative	Parameter	Additional Notes
ORP (mV)				ml	F or UF			
DO (mg/l)				ml	F or UF			
pH				ml	F or UF			
SC (µmhos/cm)				ml	F or UF			
Turbidity (ntu)				ml	F or UF			
H ₂ O Tmp. (°C)				ml	F or UF			
Color				ml	F or UF			
Other:				ml	F or UF			
				ml	F or UF			

Comments: _____

Sample Team Member Signature: _____ Page _____ of _____

STANDARD OPERATING PROCEDURE

OBTAINING WATER QUALITY SAMPLES FROM STREAMS[®] HF-SOP-19

1.0 PURPOSE

The type of samples described in the following are "grab samples". They represent the water quality at one point for one time period. This is a commonly employed method of water quality sampling and the purpose of this procedure is to standardize sampling.

2.0 EQUIPMENT

- Sampler's field notebook;
- Water Sampling Form (**HF-FORM-430**);
- Clean sample bottles and labels;
- Preservatives;
- Coolers, ice;
- 0.45 micron filter apparatus with inert filters;
- Distilled, deionized water; and
- Custody seals if required by project.

3.0 PROCEDURE

- 3.1 Select a station where the water quality sample would best represent the hydrochemistry of the stream segment. This could be a rapids or fast moving section of a stream. Avoid stagnant areas. Do not sample downstream from a tributary unless complete mixing has occurred. If possible, choose an accessible site for streams to be monitored regularly. Avoid sampling downstream of road crossings, sample upstream if at all possible.
- 3.2 Measure and record stage and/or flow (see appropriate stage and streamflow measurement Standard Operating Procedure).
- 3.3 Label each sample bottle with the appropriate information in accordance with the field procedure. Complete the Water Sampling Form (**HF-FORM-430**) (a copy of which is attached).
- 3.4 If the sample bottle does not contain preservatives, bottle and cap should be rinsed three times with sample water before the actual sample is collected. A distilled, deionized water rinse can be used as an alternative in some situations.

- 3.5** Conditions at the surface of stream environments may differ significantly from conditions within the water column due to the presence of buoyant contaminants (dust, pollen, leaves, etc.). In most cases, inclusion of the surface layer in the integrated sample is desirable. However, if conditions indicate that surface layer contamination would seriously compromise the representativeness of the sample, the sample bottle may be uncapped, filled, and recapped while submerged.
- 3.6** Obtain a stream width and depth integrated sample by collecting water while moving the open sample bottle up and down and across the width of the stream. Raise and lower the bottle through the entire depth while proceeding across the stream to assure a representative sample where needed or use an isokinetic depth-integrating sampler such as the USGS US DH-81, DH-48, or D-77 sampler (USGS, 1998). Be sure to leave sufficient volume in sample bottles such that required preservatives may be added without overfilling bottles. Total suspended sediment samples should always be collected using the depth and cross-section integration method. **When wading, be sure to collect the sample upstream of wading personnel to avoid sampling resuspended bed sediments caused by bed disturbances.**
- 3.7** Filter and preserve samples as required.
- 3.8** Immediately place filled sample bottles in cooler chest that is kept at the appropriate temperature.

4.0 ASSOCIATED REFERENCES

HF-FORM-430 Water Sampling Form

USGS, 1998. Techniques of Water-Resources Investigations, Book 9, Chapter AZ: Selection of Equipment for Water Sampling. August 1998.

Project Name: _____
Project Code: _____
Sample Team Member(s): _____
Laboratory Used: _____

Site Designation: _____
Sample Code Number: _____
Sample Date: _____
Sample Time: _____ (military)

**If Duplicate Sample Collected,
Please Record Below**

Duplicate Sample Code #: _____
 Duplicate Sample Time: _____

Site Conditions

New Site: Yes No Photo taken: Yes No
 Site Type: DRY surface water process water
 monitoring well domestic well adit seep
 spring- other: _____
 Weather Conditions: calm breeze windy
 no precip. rain snow
 clear p. cloudy overcast
 Air Temperature: _____ °C _____ °F

For Groundwater Samples

well volume formula:	$V = (TD-SWL) \times (Dia.^2) / 25$	Comments
TD (ft):	_____	
SWL (ft):	_____	no access/pumping
Casing Diameter (I.D.):	_____	
Water Volume (V) (gal):	_____	
x 3=(gal.)	_____	
Actual Vol. Removed (gal.):	_____	
Water Level Recovery:	slow moderate rapid	

For Surface Water Samples

Flow Method: Marsh McBirney Volumetric Flume Weir Estimate
 Other Flow or Description: _____
 Flow: gpm cfs Staff Gage: _____

Field Parameter Stabilization

Time (military)	Oxidation Reduction Potential (mV)	Dissolved Oxygen (mg/l)	pH	S.C. (µmhos/cm)	Turbidity (n.t.u.)	Temperature (°C)	Additional Parameters or Notes

Turbidity: (circle) clear slight moderate very Sample Method: (describe) grab composite large peristaltic pump bailer other

Field Parameters

Bottles Collected

	Sample	Duplicate	Quantity	Size	Filter or Unfilt.	Preservative	Parameter	Additional Notes
ORP (mV)				ml	F or UF			
DO (mg/l)				ml	F or UF			
pH				ml	F or UF			
SC (µmhos/cm)				ml	F or UF			
Turbidity (ntu)				ml	F or UF			
H ₂ O Tmp. (°C)				ml	F or UF			
Color				ml	F or UF			
Other:				ml	F or UF			
				ml	F or UF			

Comments: _____

Sample Team Member Signature: _____ Page _____ of _____

STANDARD OPERATING PROCEDURE

FIELD MEASUREMENT OF pH USING A pH METER HF-SOP-20

1.0 PURPOSE

The purpose of this procedure is to obtain accurate field measurements of the pH of water samples.

2.0 EQUIPMENT

This procedure written for Beckman pH meters is applicable to a variety of pH meters. Specific operating instructions accompanying each pH meter should be followed where in variance with the following.

2.1 INSTRUMENTS

- Beckman I-10 or I-21 pH meter or similar instrument;
- Beckman pH electrode/probe, Model 39841 or equivalent;
- Beckman temperature probe, Model 598115 or equivalent; and
- Field notebook.

2.2 REAGENTS

- Buffers pH 4.0, 7.0 and 10.0 (other buffers may be used in unusual waters);
- Deionized water; and
- Beckman filling and storage solution - 4 Molar KCl (potassium chloride).

3.0 PROCEDURE

Calibration of the instrument should be performed at least once per day, before sampling activities commence. Field calibration forms must be completed at this time, and calibration verification should be documented in field notebooks.

While field instruments are manufactured to be rugged and dependable, a reasonable amount of care is still required to ensure that instruments function properly and give accurate readings.

Field instruments must be cleaned and stored in accordance with established guidelines (see operating instructions) in order to maintain instrument integrity.

3.1 EQUIPMENT SET UP

3.1.1 Instrument Check

- Turn instrument on by pressing pH button, check display and confirm the low battery light is not illuminated; and
- Visually inspect probe for damage and fluid level. If damage is evident, replace probe. If low on fluid, refill using 4 Molar KCl potassium chloride. Be sure to leave vent hole uncovered while taking measurement so that liquid junction flows freely.

3.1.2 Connecting Electrodes

- Insert the pH electrode connector into the large input jack on the top of the instrument and twist to the locked position.
- Insert temperature electrode connector into the small input jack on the instrument top. Instrument is now ready to use.

3.2 pH MEASUREMENT

3.2.1 Select two buffers, one with a pH of 7.0. Select a second buffer (pH 4.0 or 10.0) so that the two buffers bracket the anticipated sample pH (use fresh buffers for calibration).

3.2.2 Uncap pH electrode, remove stopper from vent hole, rinse both pH probe and temp probe with deionized water and place in pH 7.0 buffer.

3.2.3 Depress the CLR button, then depress the ↓ button. The meter will automatically temperature adjust the reading and compensate to read the buffer in which it is reading. This reading will lock in memory and display on the bottom of the screen.

3.2.4 Remove electrodes from the solution. Rinse with distilled water and place in the second buffer.

3.2.5 Repeat step 3.2.3 with the second buffer.

3.2.6 Remove electrodes from the second buffer, rinse with distilled water then a portion of sample and place in sample. The instrument is calibrated daily or anytime a pH is measured, which is not in the buffer range for which the instrument is calibrated.

3.2.7 Record the pH of the sample in sample field notebook.

3.2.8 When measurements are complete, rinse probe with distilled water. Add a few drops of 4 Molar KCl solution to the protective cap and store probe in the protective cap. Replace cover over vent hole.

4.0 ASSOCIATED REFERENCES

Beckman Instruments, 1992. Instruction manuals for specific ion meter, models I-10, I-11, I-12; and I-21 pH meters.

STANDARD OPERATING PROCEDURE
FIELD MEASUREMENT OF DISSOLVED OXYGEN
HF-SOP-22

1.0 PURPOSE

The purpose of this procedure is to obtain accurate field measurements of dissolved oxygen (DO) in water.

2.0 EQUIPMENT

2.1 INSTRUMENTS

- YSI Model 55 Dissolved Oxygen Meter

2.2 REAGENTS

- Deionized water (DI H₂O); and
- Oxygen probe solution.

2.3 OTHER

- Flow Cell (strongly recommended)
- Field Notebook

3.0 PROCEDURE

When collecting measurements in surface water, the probe can be placed directly into the water body. **Similarly, the best method for measuring DO in groundwater is by using a downhole probe.** However, if this is not feasible, alternate acceptable methods are available. When measuring ground water, care should be taken to avoid adding oxygen to the water during sample collection. To avoid this condition, bailers should be moved slowly across the water surface and pumping rates should be reduced to avoid splashing or otherwise aerating the sample upon collection in the sample cup. Pumps which cause air to contact the water should not be used. **Use of a flow-through cell is strongly encouraged over collection in a sample cup.** A flow-through cell reduces potential sample aeration and allows for selection of a standard flow rate to proceed across the probe.

3.1 EQUIPMENT SET-UP AND CALIBRATION

- 3.1.1.** Switch probe on and allow to warm up for at least 15 minutes. Check probe storage chamber to ensure that sponge in chamber is moist.
- 3.1.2.** Press up and down arrow keys simultaneously to enter calibration mode. Input approximate elevation in feet above mean sea level and press Enter.
- 3.1.3.** Allow meter reading to stabilize. Record “Cal #” shown in lower area of display, as well as meter readout following stabilization. These numbers should be similar (i.e., for “Cal #” equal to 82, stabilized meter reading should be 80-84). Press Enter.
- 3.1.4.** Input salinity correction value (leave at 0.0 for fresh water, or input approximate salinity for brines, seawater, etc.) Press Enter. Meter is ready for use. If “Cal #” and stabilized meter reading are not similar, recalibrate.

3.2 DISSOLVED OXYGEN MEASUREMENT

- 3.2.1** Lower probe into sample. NOTE: Some motion of water past probe membrane is required, so if water sample is quiescent, manual movement of probe is required (do not aerate sample during movement).
- 3.2.2.** Allow reading to stabilize. MODE key selects unit readout (% saturation or mg/L). Record reading and temperature.
- 3.2.3.** Replace probe in storage chamber after decontamination. If meter is shut off, recalibration is required each time meter is turned on. Recalibration will also be required if elevation changes significantly (>200 ft) between sample locations.

4.0 ASSOCIATED REFERENCES

Yellow Springs Instrument Company. Instrument manual for YSI Model 55 dissolved oxygen meter.

HF-SOP-49 - Use of a Flow Cell for Collecting Field Parameters.

TABLE 1. SOLUBILITY OF OXYGEN IN FRESH WATER

Temperature °C	mg/L Dissolved Oxygen	Temperature °C	mg/L Dissolved Oxygen
0	14.60	23	8.56
1	14.19	24	8.40
2	13.81	25	8.24
3	13.44	26	8.09
4	13.09	27	7.95
5	12.75	28	7.81
6	12.43	29	7.67
7	12.12	30	7.54
8	11.83	31	7.41
9	11.55	32	7.28
10	11.27	33	7.16
11	11.01	34	7.05
12	10.76	35	6.93
13	10.52	36	6.82
14	10.29	37	6.71
15	10.07	38	6.61
16	9.85	39	6.51
17	9.65	40	6.41
18	9.45	41	6.31
19	9.26	42	6.22
20	9.07	43	6.13
21	8.90	44	6.04
22	8.72	45	5.95

Source: Derived from 15th Edition "Standard Methods for the Examination of Water and Wastewater".

TABLE 2. ALTITUDE CORRECTION FACTOR

<u>Atmospheric Pressure</u> <u>mmHg</u>	<u>or</u>	<u>Equivalent Altitude</u> <u>Ft.</u>	<u>=</u>	<u>Correction</u> <u>Factor</u>
775		-540		1.02
760		0		1.00
745		542		.98
730		1094		.96
714		1688		.94
699		2274		.92
684		2864		.90
669		3466		.88
654		4082		.86
638		4756		.84
623		5403		.82
608		6065		.80
593		6744		.78
578		7440		.76
562		8204		.74
547		8939		.72
532		9694		.70
517		10472		.68
502		11273		.66

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater".

STANDARD OPERATING PROCEDURE
STREAMFLOW MEASUREMENT USING A
MARSH-McBIRNEY WATER CURRENT METER[®]
HF-SOP-37

1.0 PURPOSE

The purpose of this procedure is to obtain an accurate streamflow measurement. The method described is the "midsection method" with a Marsh-McBirney current meter.

2.0 PROCEDURE

2.1 SITE SELECTION

2.1.1 Choose a stream section with the following conditions:

- A. A straight reach with stable streambed free of large rocks, weeds and protruding obstructions such as boulders which would create non-parallel flow.
- B. A uniformly sloped streambed profile to eliminate vertical components of velocity.

It is usually not possible to satisfy all these conditions, but select the best possible site using these criteria.

2.1.2 Modify the stream channel above the measuring cross-section to best approximate these conditions.

2.1.3 If the site is to be revisited, permanently mark cross-section location.

2.2 CALIBRATION PROCEDURE

2.2.1 Set scale knob to "CAL" and time constant switch to 2.

2.2.2 After approximately ten seconds, the digital readout should be on or between 9.8 and 10.2.

- A. If readout is not within limits, change batteries and repeat calibration. If the unit fails to calibrate (readout between 9.8 and 10.2) after the battery change, the unit is malfunctioning and should be returned to the manufacturer for repair.

B. If readout is within limits, the instrument is correctly calibrated.

3.0 MEASUREMENT PROCEDURE

3.1 Place a measuring tape or tag line across the selected section at right angles (if possible) to direction of flow. If it is not possible to establish a line perpendicular to flow, record the angle between the perpendicular and the actual flow measurement line. Record the total channel width. Estimate the number of sections needed to allow no more than 5 percent of the total flow in each section. For small streams, 10 percent of flow is permitted. Twenty-five to thirty sections are needed for a good measurement to get less than 5 percent of flow in each section. For less stringent accuracy, a lesser number of stations can be used.

3.2 Fill out the required information on stream gaging on the **Stream Gaging - Current Meter Form (HF-FORM-438)**. Much of the form is self-explanatory; however, the following explanation will assist in completing some parts of the form.

- A. Site: List the site number and its name.
- B. Distance from Initial Point: This is the measured distance from the initial point. For example: A measuring tape may be used and the edge of water may be several feet from the tape zero point.
- C. Width: Width of the cross section in feet.
- D. Depth: Depth of water in feet measured by wading rod or other measuring device.
- E. Area: Product of WIDTH x DEPTH in square feet.
- F. Point Velocity: Velocity as read from meter.
- G. Discharge: Product of area times the point velocity. This is the computed flow in cfs with attention paid to significant numbers and the error limits.
- H. Measurement Conditions and Rating: Estimate conditions.
- Good (5%): Bottom slightly rough, flow not completely straight and smooth.
- Fair (8%): Moderately rough bottom flow velocity varies across channel.

<u>Poor (over 12%):</u>	Rough bottoms; significant velocity variation across channel.
<u>Very Poor (20%):</u>	Very rough bottom; channel divided by boulders or weed-filled or other problems.
<u>Other (EXPLAIN):</u>	Some channels are rocky or weedy and are otherwise difficult to measure. Estimate error. Error can range from 20% to over 100%.

- I. Gage Height: Record reading of staff gage or other measuring device placed in the stream. This is a measurement of stream stage.
- 3.3** Identify stream bank by either LEOW or REOW (left or right edge of water, respectively, when facing downstream) and record starting time.
- 3.4** Note any changes in stage height during measurement.
- 3.5** To begin measurement note distance from end of tape to beginning edge of water. Try to start at an even increment.
- 3.6** Measure and record water depth at the edge of the water.
- 3.7** Move out to center of the first section.
- 3.8** Record the distance from the initial point.
- 3.9** Using the top-setting wading rod, measure and record the depth at that point.
- 3.10** Mean velocity of flow at the point is determined by measuring velocity at 0.6 depth from the surface, for depths less than 2.5 feet. To set the sensor at 0.6 depth using the top-setting wading rod, line up the foot scale on the sliding rod with the tenth scale at the top of the depth gauge rod so that the combined scales match the depth of water at the measuring point. For depths greater than 2.5 feet, measurements are collected at 0.2 and 0.8 depth below the surface and the average of these values is used as the average velocity for the cross-section.
- 3.11** Set wading rod so the sensor is facing directly into flow (record any angles). Be sure you are not disturbing flow around the meter, stand to the side and downstream while taking the measurement.
- 3.12** Allow meter readout to stabilize. Start with the smallest time constant setting. If, after a period of time (in seconds) equal to 5 times the time constant setting (e.g. 10, 30 and 100

seconds for settings of 2, 6 and 20), the readout has not stabilized move to the next highest time constant settling.

3.13 Record velocity.

3.14 Move to the center of the next section.

3.15 Continue through the sections using steps 8 through 15.

3.16 For streams with a fairly uniform flow regime, the section can remain of equal width.

3.17 In areas where velocity varies or flow is concentrated in a narrow area, divide the high flow sections up into smaller widths to account for higher velocities (discharge).

3.18 Record the distance at the edge of water and ending time and note which edge this is - either LEOW or REOW.

3.19 Compute flow using the mid-section technique (USGS, 1977).

4.0 MAINTENANCE PROCEDURES

- Keep sensor free of dirt and coatings such as grease. Clean sensor with mild soap and water.
- Routinely check batteries by calibrating the meter.

5.0 REFERENCES

USGS, 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Chapter 1: Surface Water.

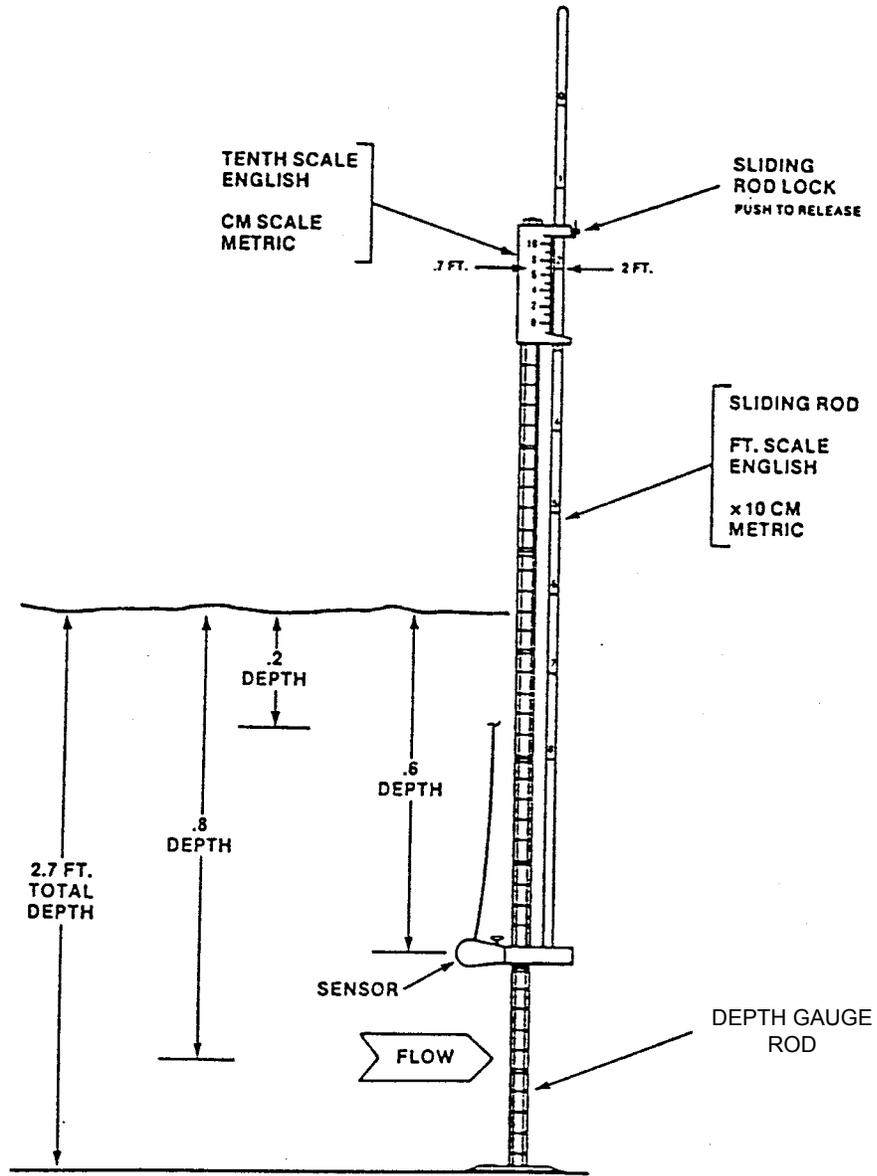


FIGURE 1. TOP SETTING WADING ROD

STANDARD OPERATING PROCEDURE

USE OF A FLOW CELL FOR COLLECTING FIELD PARAMETERS[®]

HF-SOP-49

1.0 PURPOSE

The purpose of using a flow cell is to increase the accuracy of field parameter values while sampling groundwater. The flow cell is designed to allow field personnel the ability to obtain field parameters from groundwater that are, with the exception of the pumping equipment, undisturbed. Specifically, use of a flow cell isolates the water sample from contact with the atmosphere at ground surface, providing a better representation of *in situ* groundwater chemistry for field parameter measurement.

2.0 EQUIPMENT

- A) Flow cell
Necessary fittings to connect pumping system to the flow cell.

3.0 PROCEDURE

- A) Connect flow cell to discharge tubing of pump system.
- B) Connect or place meter (YSI 556 or similar) in the flow cell
- C) Take readings as necessary from the field meter, according to the requirements outlined in the project work plan or sampling and analysis plan
- D) If performing low flow sampling, reference HSOP-105 for instruction on use of a flow cell during low flow sampling.

STANDARD OPERATING PROCEDURE

FILTRATION OF WATER SAMPLES[®]

HF-SOP-73

1.0 PURPOSE

Water is filtered to obtain a sample for analysis of dissolved constituents. Dissolved constituents are operational, defined as those which pass a 0.45 micron filter. This SOP describes three methods in which filtered water samples can be prepared in the field. Other types of filtering equipment can be employed. The essential points are use of the proper filter and adequate decontamination of reusable equipment.

2.0 EQUIPMENT

<u>Filter Barrel</u>	or	<u>Plate Filter</u>	or	<u>Disposable Filter Cartridges</u>
Tire pump		Peristaltic Pump		0.45 µm filter
Filter barrel		Plate filter		cartridges
Clean sample bottles		0.45 µm membrane		Peristaltic Pump
Prefilters (where needed)		filters		Plastic tubing
0.45 µm filter membranes		Prefilters (where		Clean sample bottles
Distilled or deionized water		needed)		Distilled or
Plastic tweezers		Plastic tubing		deionized water
		Clean sample bottles		
		Distilled or deionized water		
		Plastic tweezers		

3.0 PROCEDURE

A) General

1. Have at hand clean sample bottle pre-labeled with appropriate information.
2. Use a new filter membrane or disposable cartridge for each sampling site.
3. If water is very turbid, it must be first run through a larger pore size pre-filter.
4. Be sure you know the volume of sample required for analysis, check with laboratory if in doubt.
5. If collecting samples for low level analysis, rinse filter with an appropriate amount (usually 100 to 200 ml) of DI water prior to filtering any sample. This step should

remove contaminants (particularly zinc) which may be entrained within the filter matrix. Record the amount of DI water used to rinse the filter.

6. Rinse sample bottle with filtered water three times, before collecting actual sample. However, if water is hard to filter or of limited quantity, distilled or deionized water rinses are acceptable.
7. Avoid dusty locations and vehicle motor exhaust while filtering.
8. When a peristaltic pump is used, the pump and tubing should be cleaned immediately after obtaining a sample by pumping 500 ml of deionized water. After pumping 500 ml deionized water, remove inlet tubing from DI source and continue pumping until tubing is drained.

B) Filter Cartridge These are single-use, self-contained membrane filtration devices with inlet and outlet hose barbs designed for use when samples are pumped.

1. Examine a new filter cartridge and note direction of flow arrow imprinted on it.
2. Slip hose from pump over inlet nipple of cartridge. Sample may be collected directly from filter outlet (optional, place another short piece of tubing over outlet, if this is more convenient). Keep tubing length as short as possible.
3. It is important that water flow through filter in direction of imprinted arrow, as filter failure will likely result if flow direction is reversed. Also, inlet pressure should not exceed 25 PSI (pounds per square inch) for most filters of this type.
4. Turn pump on, discard initial 30 ml of filtrate (filter purge), then begin collecting sample.

C) Filter Barrel Filter barrels are reusable plastic cylinders with removable endcaps and fitted with a replaceable filter at one end (the outlet) and an air inlet at the opposite end by which the barrel is pressurized. Filter barrels are used where samples cannot be pumped.

1. Filter barrels must be decontaminated prior to going to the field. Remove both end caps, O-rings, and filter support. Wash components thoroughly with a non-phosphate detergent and water, thoroughly rinse with distilled or deionized water, re-assemble and store in plastic bag.
2. Ideally, the filter barrel should be rinsed with the water to be sampled. If an inadequate volume of sample water is available, a distilled or deionized water rinse is acceptable.

3. After rinsing, fill filter barrel 2/3 full with sample water.
4. Place clean 0.45 μm filter on filter support (do not touch filter with hands, use plastic tweezers or blue divider papers to move or adjust filter). Wet filter support will hold filter in place.
5. Assemble filter barrel carefully so as not to twist or put folds in filter paper.
6. Turn filter barrel over so sample water comes in contact with filter paper.
7. Connect tire pump to Shrader valve and pump several times. Do not allow static pressure on tire pump to go over 20 PSI.
8. Purge filter by draining approximately 100 ml of water from lower side of filter support. Discard this initial filtrate.
9. Once sample bottle is full, preserve sample as needed and place in cooler with ice. (see **HF-SOP-3, Preservation and Storage of Inorganic Water Samples**).
10. Before leaving the sampling site, disassemble filter barrel, remove and dispose of filter paper, and immediately rinse with distilled or deionized water. Partial decontamination, consisting of three successive distilled or deionized water rinses between sites is acceptable.

D) Plate Filter Plate filter is a reusable membrane filter holder, generally fitted with three removable legs. The filter holder is disassembled to replace the large diameter (typically 14.2 cm) membrane filter. Water is pumped through the filter, entering at the top and exiting through a port at the bottom.

1. Plate filters must be decontaminated prior to use. Disassemble plate filter, wash components thoroughly with a non-phosphate detergent and water, thoroughly rinse with distilled or deionized water, re-assemble and store in plastic bag.
2. Ideally, the plate filter should be rinsed with the water to be sampled. If an inadequate volume of sample water is available, a distilled or deionized water rinse is acceptable.
3. Place clean 0.45 μm membrane filter on filter support (do not touch filter with hands, use plastic tweezers or blue divider papers to move or adjust filter). Wet filter support will hold filter in place.
4. Assemble plate filter carefully so as not to twist or put folds in filter paper.

5. Connect plastic tubing from pump to top hose barb on filter. Sample may be collected directly from outlet, or keep tubing lengths as short as possible. A short piece of tubing may be connected to outlet barb at bottom.
6. Purge filter by pumping approximately 100 ml of water through the filter. Discard this initial filtrate.
7. Once sample bottle is full, preserve sample as needed and place in cooler with ice. (see **HF-SOP-3, Preservation and Storage of Inorganic Water Samples**).
8. Before leaving the sampling site, disassemble plate filter, remove and dispose of filter paper, and rinse with distilled or deionized water.

NOTES

- Use a new filter membrane for each sample.
- Run very turbid or muddy water through prefilter first and then a 0.45 micron filter.
- Check with lab performing analysis for adequate quantity and holding time for sample. Complete all appropriate documentation.
- Completely decontaminate filtering equipment after each day of use and whenever partial decontamination doesn't visually clean all filter parts.
- Do not attempt filtration in dusty locations or while your vehicle motor is running (due to exhaust).

STANDARD OPERATING PROCEDURE

FIELD MEASUREMENT OF SPECIFIC CONDUCTIVITY HF-SOP-79

1.0 PURPOSE

The purpose of this procedure is to obtain accurate field measurements of specific electrical conductance of water samples. This procedure is written for the Hydac Digital type meter; other meters may be used if they are calibrated and used according to manufacturer's recommendations.

2.0 EQUIPMENT

2.1 Instrument

- Hydac Digital Conductance Meter or equivalent meter.

2.2 Reagents

- Potassium Chloride (KCl) standard solutions with known conductivities: (e.g., 50, 74, 147, 400, 718, 1413, 6668, 12990 $\mu\text{mhos/cm}$ at 25°C).

2.3 Other Materials

- Distilled or deionized water for rinsing
- Field Sampling Notebook

3.0 PROCEDURE

3.1 Calibration

3.1.1 Rinse sample cup with distilled water before and after each conductivity standard used.

3.1.2 Select a standard with a conductivity value in the approximate range of the samples to be measured. After rinsing the sample cup with distilled water, rinse with the selected standard. Fill the cup with the standard, set function selector to TEMPERATURE and depress READ button. Set the temperature compensation knob on the conductivity side of the meter to the displayed temperature.

- 3.1.3 Switch function selector to CONDUCTIVITY and depress the READ button (READ button must be held down for display). Move the range selector to the lowest setting which will give a reading.
- 3.1.4 If the reading is not that of the standard, with a small screwdriver, adjust the calibration screw at the bottom of the meter (only small turns are required for fine-tuning).
- 3.1.5 Record reading, temperature, and time of calibration.

3.2 Sample Specific Conductivity Measurement

- 3.2.1 Rinse the sample cup with distilled water prior to filling with the sample. Rinse and fill with sample water.
- 3.2.2 Switch function selector to temperature scale and measure temperature of sample.
- 3.2.3 Adjust temperature compensator knob on the conductivity side of the meter to the displayed temperature.
- 3.2.4 Switch function selector to conductivity and depress READ button. Move the range selector to the lowest setting which will give a reading. Read conductivity and multiply by range. Record in field sampling notebook.
- 3.2.5 When measurements are complete, rinse probe with distilled water.

3.3 Calibration Check

- 3.3.1 At least once per day (or about once per every ten samples collected, whichever is more frequent), or when measuring conductivities of samples significantly different from the initial calibration solution, the meter should be checked against a standard of known conductivity. Record the check standard conductivity, temperature, and meter reading on appropriate documentation.
- 3.3.2 If the check standard reading differs from the true value by more than 10%, the meter should be recalibrated according to Section 3.1 of this SOP.

4.0 ASSOCIATED REFERENCES

Hydac Instruments -Instruction Manual for Digital Conductance, Temperature, and pH Tester.
Hydrometrics' Video Training Library -- Measurement of Conductivity.

STANDARD OPERATING PROCEDURE

FIELD MEASUREMENT OF TEMPERATURE[®] HF-SOP-84

1.0 PURPOSE

This procedure outlines the protocol for measurement of water temperature in the field. The procedure is applicable to lotic systems (rivers and streams), lentic systems (lakes, ponds, reservoirs, and impoundments), and groundwater systems. Special considerations for the various types of water environments are included in this procedure.

2.0 EQUIPMENT

- Liquid-filled thermometer, with scale divisions marked at a minimum of 1.0°C;
- Standard field meter equipped with a thermometer (for example, pH meters and conductivity meters often include temperature readout option);
- Temperature readout device with a remote probe (necessary for measuring temperature at depth in lakes or groundwater wells); and
- Field notebook.

3.0 PROCEDURE

Calibrate temperature measurement devices prior to field use with NIST-certified thermometers. When two methods of temperature measurement are available in the field (glass thermometer and pH water thermometer, for example) they may be used to cross-check one another.

It is preferable to measure temperature directly in the source to be sampled by immersing the thermometer into the stream, pond, etc., and allowing the reading to stabilize, when practical. Procedures for each of the main types of water samples are given below. If temperature must be measured on a sample that has been removed from the source, it is critical to measure and record the temperature immediately after collection, since equilibration with ambient air and container temperature will immediately begin to affect sample temperature.

A. Rivers and Streams

Wade stream and measure temperature directly, or measure from bank if unwadable. Temperature should be measured at multiple points across the stream transect, especially in large, slow-moving river systems or immediately downgradient of tributaries. The average of all measurements is taken as the water temperature and recorded in the field notebook.

B. Lakes and Ponds

Measure temperature from bank and record. Recall that static water bodies often stratify. If samples are collected at various depths, temperature should be recorded at each depth. Depth profiling of temperature should occur at 1 foot or smaller intervals, in most cases.

C. Groundwater

Measure temperature of pumped or bailed water while purging well to monitor stabilization of temperature. Record temperature immediately after obtaining sample. If a remote, "down-the-hole" temperature probe is available, its use is preferred.

4.0 ASSOCIATED REFERENCES

"Standard Methods for the Examination of Water and Wastewater," 18th edition (1992), page 2-59.