STANDARD OPERATING PROCEDURE NO. 8.0

FIELD PARAMETER MEASUREMENTS (INCLUDING INSTRUMENT CALIBRATION)

Powertech

R Squared

Attachment A

Field Parameter Measurements

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1.0 PURPOSE AND SCOPE

This standard operating procedure (SOP) describes procedures that will be used to obtain field parameter measurements for surface water and groundwater parameter samples. These parameters are pH, temperature, specific conductivity (S.C.), dissolved oxygen (D.O.), oxidation reduction potential (O.R.P), and turbidity. This SOP describes field measurement procedures, personnel responsibilities and qualifications, and quality assurance/quality control (QA/QC) procedures.

2.0 RESPONSIBILITIES AND QUALIFICATIONS

The Project Manager or Field Manager has the overall responsibility for implementing this SOP. They will be responsible for assigning appropriate environmental staff to implement this SOP and for ensuring that the procedures are followed by all personnel.

All personnel performing these procedures are required to have the appropriate health and safety training. In addition, all personnel are required to have a complete understanding of the procedures described within this SOP and receive specific training regarding these procedures, if necessary.

All environmental staff and assay laboratory staff are responsible for reporting deviations from this SOP to the Project Manager or Field Manager.

3.0 RELATED STANDARD OPERATING PROCEDURES

The procedures for field measurements set forth in this SOP are intended for use with the following SOPs:

SOP No. 1.0 Surface Water and Seep Sampling

SOP No. 26.0 Groundwater Sampling

SOP No. 10.0 Decontamination of Sampling Equipment

4.0 EQUIPMENT LIST

In addition to the equipment cited in associated SOPs for the collection of surface and groundwater samples (SOP Nos. 1.0 and 26.0, respectively), the following meters which are the current equipment available, or their equivalent, are required for the implementation of this SOP.

pH meter with a temperature scale (Geotech Multiline P3 pH/LF with integrated SenTix41 pH/temperature probe)

Specific conductivity meter (pH/LF standard conductivity cell)

Dissolved oxygen meter (pH/OXI Dissolved oxygen probe with air calibration beaker Oxical - SL)

Oxidation Reduction Potential meter (pH/OXI with UAg/AgCl redox electrode probe)

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Turbidity meter (portable turbidity meter).

Combination meters or flow-through cell meters may also be used.

5.0 FIELD PARAMETER MEASUREMENTS

Several of the parameters required to be measured are physically or chemically unstable and must be tested either in situ or immediately after sample collection using a field test kit or instrument. Examples of unstable parameters include pH, temperature, and dissolved oxygen. Although the specific conductivity of a substance is relatively stable, it is recommended that this characteristic be measured in the field. Most instruments measuring specific conductivity require temperature compensation; therefore, the temperature of the samples should be measured at the time the specific conductivity is measured.

Sampling personnel shall wear chemical-resistant gloves, which will be disposed of between locations when performing field measurements.

5.1 PROCEDURES FOR COLLECTING SAMPLE FOR FIELD PARAMETER MEASUREMENTS

Collect water samples for chemical analysis as described in SOP Nos. 1.0 and 26.0. Additional sample (volume) will be collected and placed in a separate container (if not using an insitu probe) for measuring field parameters. Field parameter measurements may be taken immediately before or after sample collection depending on the type of sample collected. If determining parameter stabilization during well development, samples for field parameter measurements will be collected periodically. The sample container can be used to measure all field parameters of the sample. After the measurements have been recorded, the water should be discarded. Do not use this sample for chemical analysis.

5.2 METER USE AND CALIBRATION

Field instruments will be calibrated daily prior to commencement of field measurement activities. Solutions used for standardizing and calibrating will be checked prior to field mobilization to determine if the expiration dates have been exceeded. Any expired solution will be discarded appropriately and replaced with a new solution.

5.2.1 pH Meter

The pH meter must be calibrated each day prior to well evacuation and water sample collection. Calibration and operation of the pH meter should follow the manufacturer's specific instructions. In general, calibration is done by adjusting the meter with standard buffers that bracket the expected pH of the aqueous samples. For specific instructions refer to the manufacturer's instruction manual. General calibration procedures for Powertech's on-site equipment are provided in Attachment 1 (Calibration of Powertech Field Instruments).

Field sampling personnel will measure pH as follows:

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- 1. If the pH is measured in a container, rinse the sample container with deionized water and then rinse it three times with the water to be sampled prior to measurement. If using an inflow multi-measurement system, measurements are continuously displayed.
- 2. Rinse the pH probe with deionized water. Be sure to protect the fragile glass bulb at the end of the probe from damage.
- 3. Immerse the electrode in the water, allow the pH to stabilize, and monitor the drift of the instrument. Do not immerse the electrode above the top of the pH probe.
- 4. When the pH reading stabilizes, record in the field logbook the temperature to the nearest 0.1°C and the pH reading to the nearest 0.01 unit.
- 5. Between measurements, store the electrode in pH electrode storage solution or equivalent solution, if possible, or put a cotton swab soaked in electrode solution in the protective cap of the electrode.
- 6. Decontaminate the pH meter/in-flow multi-measurement system and associated equipment in accordance with SOP No. 10.0, Decontamination of Sampling Equipment.

QC requirements for pH measurements are provided in Section 5.3.

5.2.2 Specific Conductivity Meter

Conductance is a measure of the ability of an aqueous solution to conduct electrical current and is expressed in reciprocal ohms (mhos). The International System of Units uses the siemen(s) to represent mhos. Calibration procedures are to be conducted according to the manufacturer's instructions. Record time, temperature, and instrument response in the field logbook. General calibration procedures for Powertech's on-site equipment are described in Attachment 1 (Calibration of Powertech Field Instruments).

The procedures for measuring conductivity are as follows:

- 1. If the conductivity is measured in a container, rinse the sample container with deionized water and then rinse it three times with the water to be sampled prior to measurement. Rinse the probe with deionized water. If using an in-flow multi-measurement system, measurements are continuously displayed.
- 2. Insert the probe into the sample.
- 3. Allow the reading to stabilize before recording measurements.
- 4. Record in the field logbook the conductivity, the units of measure, any scale multipliers and temperature of the sample.
- 5. During normal use, rinse the probe thoroughly with deionized water between measurements to minimize the buildup of interfering substances on the probe element.
- 6. Decontaminate conductivity meter/in-flow multi-measurement system and associated equipment in accordance with SOP No. 10.0, Decontamination of Sampling Equipment.

QC requirements for conductivity measurements are provided in Section 5.3.

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5.2.3 Dissolved Oxygen Meter

The concentrations of dissolved oxygen in groundwater samples provide an indication of the whether aerobic or anaerobic respiration is occurring in an aquifer system. Dissolved oxygen concentrations greater than 1 mg/L are favorable for aerobic respiration. Manufacturer's operating manuals, calibration; maintenance procedures will be followed and documented in the field logbook. General calibration procedures Powertech's on-site equipment is provided in Attachment 1 (Calibration of Powertech Field Instruments.

The procedures for using a DO meter are as follows:

- 1. Inspect the membrane before each field use for air bubbles, oily film, and/or holes. If the membrane is defective, it must be replaced and soaked before recalibration in accordance with manufacturer's instructions.
- 2. If the D.O. is measured in a container, rinse the sample container with deionized water and then rinse it three times with the water to be sampled prior to measurement.
- 3. Read the D.O. meter to the nearest 0.1 mg/l. Record in the field logbook the D.O. concentration and the range setting of the D.O. meter.
- 4. If the D.O. meter is equipped with an operational thermometer, read the water temperature at the time the D.O. is measured. Protect the D.O. probe when not in use to prevent the membrane from drying out or freezing.
- 5. Sampling tools, instruments, and equipment will be protected from sources of contamination before use and decontaminated after use as specified in SOP No. 10.0, Decontamination of Sampling Equipment.

QC requirements for D.O. measurements are provided in Section 5.3.

5.2.4 Oxidation Reduction Potential Meter

Oxidation-reduction potential is measure of the tendency of a solution to donate or accept electrons. ORP is measured in units of milliVolts (mV). The oxidation-reduction potential (ORP) may be measured at the time of sample collection. Manufacturer's operating manuals, calibration; maintenance procedures will be followed and documented in the field logbook. General calibration procedures for Powertech's on-site equipment are described in Attachment 1 (Calibration of Powertech Field Instruments).

Oxidation-reduction potential is measured as follows:

- 1. Rinse the ORP meter probe with deionized water. If the ORP is measured in a container, rinse the sample container with deionized water and then rinse it three times with the water to be sampled prior to measurement
- 2. Dip the electrode ½ to 1-inch into the sample and stir once. The electrode should stabilize within 2 to 5 minutes. When stable, take a reading and record it in the field logbook. Any instability will be noted and recorded.



3. Decontaminate the ORP meter probe and associated equipment in accordance with SOP No. 11.0, Decontamination of Sampling Equipment.

QC requirements for ORP measurements are provided in Section 5.3.

5.2.5 Turbidity

The turbidity may be measured at the time of sample collection. A portable turbidity meter will be used for field determination of turbidity in the parameter samples.

The turbidity meter is factory calibrated and requires no field calibration. The factory calibration shall be checked before the turbidity meter is sent to the field. Additionally, the calibration of the turbidity meter will be checked daily before collection of samples against a known formazin standard. The instrument will be recalibrated as necessary based on the daily check. Refer to the manufacturer's instruction manual for proper use.

The turbidity test measures an optical property of the water sample that results from the scattering and absorbing of light by the particulate matter present. The amount of turbidity registered is dependent on such variables as the size, shape, and refractive properties of the particles. This procedure is commonly calibrated using formazin turbidity standards, and the readings are in terms of nephelometric turbidity units (NTUs).

Turbidity is measured as follows:

- 1. Rinse the turbidity meter probe with deionized water. If the turbidity is measured in a container, rinse the sample container with deionized water and then rinse it three times with the water to be sampled prior to measurement
- 2. Insert the probe into the parameter sample and allow the turbidity reading to stabilize. When stable, take a turbidity reading and record it in the field logbook.
- 3. Decontaminate the turbidity meter probe and associated equipment in accordance with SOP No. 11.0, Decontamination of Sampling Equipment.

QC requirements for turbidity measurements are provided in Section 5.3.

5.2.6 In-flow Multi-Parameter Monitoring System

The in-flow multi-parameter monitoring system or similar multi-parameter instruments may be used for measuring pH, temperature, conductivity, D.O., ORP, and turbidity. In-flow meters allow for the analysis of purge water, in-line, as it flows so that sampling can begin as soon as water stabilizes. The manufacturer's operation and maintenance manual shall be followed when operating or calibrating the instrument.

5.3 QUALITY ASSURANCE/QUALITY CONTROL

QA/QC activities will be conducted in accordance with the governing applicable documents as well as quality requirements presented in this SOP. As noted in Section 5.2, all meters will be calibrated daily prior to use. In addition, each meter will be inspected daily prior to calibration.

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Furthermore, as described below a duplicate set of field parameter measurements will be collected for every 10 sampling stations.

5.3.1 QC Checks and Acceptance Criteria

One duplicate field parameter measurement shall be collected for every ten sampling locations (10% frequency). Field parameter measurements are considered satisfactory if the duplicate measurements fall within the acceptable range in the table below.

Field Parameter	Acceptable Range
рН	<u>+</u> 0.1 pH unit
Temperature	<u>+</u> 10%
Oxidation-Reduction Potential	<u>+</u> 5 mV
Conductivity	<u>+</u> 10%
Turbidity	<u>+</u> 10%
Dissolved Oxygen	<u>+ 0.1 mg/L</u>

If duplicate acceptance criteria are not met, the instrument must be re-calibrated and new parameter measurements obtained at the sample location where the original duplicate sample did not meet acceptance criteria.

6.0 DOCUMENTATION

Documentation of observations and data acquired in the field will provide information on the activities conducted and also provide a permanent record of field activities. The observations and data will be recorded with waterproof ink in a permanently bound weatherproof field logbook with consecutively numbered pages, and/or on field data sheets as appropriate.

6.1 FIELD LOGBOOK

All information regarding calibration of field instruments will be recorded in the field logbook. The following information will be recorded in the field logbook:

- Type of meter calibrated and instrument manufacture (the instrument serial number should be recorded at the start of work and if instruments are changed)
- Date of calibration
- Person(s) performing the calibration
- Calibration solutions used
- Results of calibration measurements
- Any deviations from the SOPs

6.2 FIELD NOTES

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All information regarding field parameter measurements taken at a sample location will be recorded on the appropriate field sample collection datasheet. At a minimum, this documentation will include:

- Sample location and sample identification number
- Date and time of measurement
- Weather conditions
- Measurement results, including units and scaling factors
- Person(s) conducting measurements
- Sample characteristics (color, odor, etc.)
- Results for any QC measurements (i.e. results for duplicate set of measurements)
- Any instrument maintenance or corrective actions necessary (including rinsing of probes between sampling locations)
- Any deviations from the SOPs

The field sample collection datasheet will cross-reference the logbook in which the applicable calibration information is recorded. If the sample collection datasheet does not include spaces to record the information noted above, this information will be recorded in the field logbook.

Attachment 1 Calibration of Powertech Field Instruments

1.1 pH Meter

Calibration will consist of the following general procedures:

- 1. The meter will be calibrated using the conventional to point calibration method "ConCal." An admissible standard solution will be used: Buffer solution pH 7.0 +/- 0.5 and a buffer solution pH 4.0 +/- 0.5.
- 2. Press the CAL button until you see pH, ASY and the temperature on the screen of the meter. Also at the bottom should appear an icon labeled Cal in small print.
- 3. Immerse pH electrode into a <u>neutral</u> buffer solution. pH value of the solution: 7.0 +/- 0.5. Then press the button **RUN/ENTER** on the meter.
- 4. Set pH value of the buffer solution using the **up** and **down arrows** on the meter. CHECK THE TEMPERATURE OF THE BUFFER SOLUTION!! The specific pH value required based on the temperature is on the side of the bottle of the buffer solution.
- 5. Next press the **RUN/ENTER**. You will get an indication of asymmetry. The admissible range is +/- 30mV.
- 6. Press the **RUN/ENTER** button again. You should see pH, **SLO**, and the temperature on the screen of the meter. Rinse the pH electrode and immerse into the second buffer solution (pH 4.0).
- 7. Next press RUN/ENTER again.
- 8. Set pH value of the buffer solution using the **up** and **down arrows** on the meter. CHECK THE TEMPERATURE OF THE BUFFER SOLUTION!! The specific pH value required based on the temperature is on the side of the bottle of the buffer solution.
- 9. Next press **RUN/ENTER** again. You will get a slope range on the screen. The admissible slope range is -50mV/pH to -62.0mV/pH.
- 10. Next press **RUN/ENTER** again. You will get the indication offset voltage asymmetry. The admissible range is +/- 30mV.
- 11. Now check the readings (± 0.1 pH units) for all the buffers used for calibration.
- 12. Your meter is now ready for calibration of conductivity.

Note: Always use the same electrode for measurements that was used in the calibration. Recalibrate the meter if the electrode is replaced. Also, the temperature setting on the pH meter often does not match the sample temperature after calibration. The pH readings will be accurate in these cases provided that the response to the buffers is correct.

Record the time and temperature in the field logbook whenever the pH meter is calibrated.

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1.2 Specific Conductivity Meter

Calibration is done by determining the cell constant. The cell constant of the conductivity measuring cell can be determined by calibrating in the control standard in the range of 0.45 to 0.5 cm^{-1} .

- 1. Connect the conductivity cell TetraCon 325.
- 2. Set measuring mode to conductivity by pressing the button on the upper left hand corner labeled as **pH/mV X**. Using the **up and down arrows** select conductivity.
- 3. Press and hold down the CAL button until you see CAL and temperature on the screen of the meter.
- 4. Immerse measuring cell into control standard solution 0.01 mol/l KCl.
- 5. Next press the **RUN/ENTER** button. You should get a value between 0.45 and 0.5 cm⁻¹.
- 6. The MultiLine P3 automatically considers the temperature dependence of the control standard solution and stores the determined cell constant. You are now ready to do conductivity measurements.
- 7. Note that the probe must be placed in the center of the solution container to prevent disturbance of the electrical field.

1.3 Dissolved Oxygen Meter:

General procedures for using a D.O. meter will be as follows:

To calibrate the D.O. meter first connect the D.O. probe CellOx 325.

Press the button $pH/mV O_2$ until you see O_2 on the screen. Set measuring mode to dissolved oxygen concentration using the **up and down arrows** on the meter.

Press the CAL button until you see CAL on the screen of the meter.

Place probe in the air calibration beaker.

The sponge in the beaker must be moist (not wet!). See also the section of the operation manual entitled Oxical – SL in the appendix of the operating manual.

Press **RUN/ENTER** button. You will see the AR flashing on the screen of the meter wait until it stops and you will see the relative slope of the probe on the screen. The admissible range is 0.6 to 1.25. Your meter is now ready for use.

1.4 Oxidation Reduction Potential Meter

Calibration of the redox electrode is as follows:

Connect the redox electrode.

Set measuring mode to mV by pressing the pH/mV button. Using the up and down arrows on the meter select mV.

Immerse electrode into Mettler redox buffer solution 220mV (pH 7) UAg/Agcl

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Compare voltage and temperature value to the data on the bottle of the redox buffer solution or on the table below if the difference is higher than +/-30mV clean or change the electrode.

Temperature degrees Celsius	mV Range	рН
20	220	7.02
25	220	7.00
30	212	6.90

Record calibration verification on the log sheet. Admissible range is +/- 5mV.

1.5 Turbidity

The turbidity meter is factory calibrated and requires no field calibration. The factory calibration shall be checked before the turbidity meter is sent to the field. Additionally, the calibration of the turbidity meter will be checked daily before collection of samples again at a known formazin standard. The instrument will be recalibrated as necessary based on the daily check. Refer to the manufacturer's instruction manual for proper use.

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