USE OF MULTIPARAMETER 6.8 INSTRUMENTS FOR ROUTINE FIELD MEASUREMENTS

By Jacob Gibs, Franceska D. Wilde, and Heather A. Heckathorn

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USE OF MULTIPARAMETER 6.8 INSTRUMENTS FOR ROUTINE FIELD MEASUREMENTS

By Jacob Gibs, Franceska D. Wilde, and Heather A. Heckathorn

The miniaturization of sensors and other technological advances in electronics have resulted in water-quality instruments that house multiple sensors capable of simultaneous readings for various field measurements in environmental waters. With the use of these multiparameter instruments, field measurements can be determined with considerable reduction in the field work that generally is required when using multiple single-parameter instruments (table 6.8–1). This section addresses the short-term or discrete-measurement use of portable multiparameter instruments. Refer to Wagner and others (2006) for long-term or continuous-monitor deployment in surface water.

MULTIPARAMETER INSTRUMENT: An electronic instrument that contains sensors (each specific to the measurement of a given physical, chemical, or biological property) that are bundled in a single housing (a sonde) and deployed in environmental waters.

¹The term "field measurement(s)" is synonymous in this report with the terms field properties and field parameters. USGS field measurements include, for example, water temperature, pH, specific electrical conductance, turbidity, oxidation-reduction potential, barometric pressure, and calculations such as salinity and percent of dissolved oxygen in milligrams per liter. The term "field parameter" commonly is used in the environmental literature.

Table 6.8-1. Advantages and limitations of multiparameter instruments for field use

Advantages	Limitations
Efficiency is increased. Instruments are easy to clean, calibrate, and deploy. The time required to collect discrete samples for determining field properties is minimized. The time needed to measure and record multiple field properties is reduced. In situ measurement is likely to be more accurate and precise than measurements made in samples removed from their source. Instruments can store data, either in a display device or to internal memory. Instruments may be capable of long-term deployment.	Repair of sensors while working onsite often is not possible. Sensor replacement in the field may be unwieldy or not possible. Sensors must be replaced in a clean, dry environment. Backup field instruments (single parameter or multiparameter) are recommended to prevent data loss and extended field time. Purchase, repair, and replacement costs for multiparameter instruments are higher than for single-parameter instruments.

Quality assurance. To ensure the quality of the data collected, this section of NFM 6 describes standard operating procedures and recommendations that have been developed for routine U.S. Geological Survey (USGS) field studies. The instrument manufacturer is, however, the primary source of information about the maintenance and use of a specific instrument. The protocols and recommendations described in this section are meant to complement and enhance the manufacturer's guidelines, providing the level of quality assurance for which USGS data are held accountable.²

 $^{^2}$ USGS personnel should discuss any discrepancies between the protocols and recommendations described in this manual and the instructions provided by the instrument manufacturer with their water-quality specialists or with the USGS Office of Water Quality.

EQUIPMENT AND SUPPLIES 6.8.1

Multiparameter instruments are available for long- or short-term deployment at a stream, lake, reservoir, ground-water well, or other environmental setting, and their sondes³ are suitable for water that is fresh, brackish, saline, or polluted. Sensor housings (the sonde) of multiparameter instruments generally are available in a range of diameters from about 4 inches (in.) (10 centimeters (cm)) to less than 2 in. (5.1 cm). Small-diameter sondes may be used for downhole measurements in wells and may have more limited sensor capability than the larger diameter sonde. Depending on the manufacturer, some instruments can store instantaneous or continuous measurements to internal or external memory in a format compatible with a hand-held display, personal digital assistant (PDA), or laptop computer.

Advances in technology and design are expanding the sensor³ capabilities of multiparameter instruments and are improving instrument utility. The configuration and sensors that are available for multiparameter instruments can vary considerably among manufacturers. The procedures required for the maintenance, calibration, and use of these instruments also can change over time as a result of the technological changes being implemented; such information generally is available from the manufacturer, either online or as a manual or other document. Users must stay current as to how their instrument operates and is maintained.

- ► Sensors for the determination of water temperature, specific electrical conductance (SC), pH, dissolved-oxygen concentration (DO)⁴, and turbidity commonly are bundled in sondes used for USGS water-quality studies, as these measurements are routine for much USGS work.
- ► Sensors that determine oxidation-reduction potential (ORP or redox) and barometric pressure, and that calculate salinity, also are commonly included in the sonde.

³The term sensor, which is used in this report, is synonymous with the term "probe" that is common in the environmental literature. For multiparameter measurements, the sensors are bundled in a submersible sonde.

⁴DO is calculated as the percent of dissolved-oxygen concentration at saturation.

- ► For some instruments, sensors are available to measure fluorescence,⁵ water depth, and velocity. In some cases, specific-ion electrodes (for example, nitrate, ammonia, ammonium, and chloride) can be incorporated in a sonde. Use of sensors to measure cholorphyll and concentrations of specific nutrient species are becoming more common in USGS work. Some instruments include global positioning systems.
- When making field measurements in surface water, the sondes commonly are immersed in situ (directly within the water body). As an alternative to in situ deployment, a flowthough cell containing the sonde can be set up above land surface, to which sample water is pumped. The flowthrough cell commonly is used to monitor field measurements for ground-water investigations and for calibration of the sensors.

The types and number of sensors that can be bundled in a given sonde depend on the instrument model and manufacturer. When selecting a sensor, consult the manufacturer's recommendations and specifications for the instrument, taking into consideration the environmental conditions to be encountered, the data-quality objectives of the study, and the specific benefit of a particular sensor technology that might be applicable to the anticipated field conditions. The manufacturer is the most knowledgeable source of information for a given instrument. Consult the manufacturer's maintenance instructions for each instrument model before using the instrument.

Table 6.8–2 lists the equipment specifications and calibration solutions required when determining field-measurement values using a multiparameter instrument. The ancillary supplies needed for measuring field properties using multiparameter instruments (table 6.8–3) are the same or similar to those required for the calibration and maintenance of single-parameter instruments, and are discussed in greater detail in the individual field-measurement sections (NFM 6.1 through 6.7) of Chapter 6.

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⁵Fluorescence sensors indicate different algal pigment concentrations; see NFM 7.4 for additional information.

Table 6.8–2. Specifications and calibration solutions for multiparameter instruments

[\pm , plus or minus; -, minus; +, plus; °C, degrees Celsius; mV, millivolt; >, greater than; SC, specific electrical conductance; μ S/cm, microsiemens per centimeter at 25°C; DO, dissolved oxygen; mg/L, milligrams per liter; ORP, oxidation-reduction (redox) potential; NIST, National Institute of Standards and Technology; L, liter; \le , less than or equal to; μ m, micrometer; mL, milliliter; MSDS, Material Safety Data Sheet; SDVB, styrene-divinylbenzene beads; TDS, total dissolved solids; NFM, *National Field Manual for the Collection of Water-Quality Data*; USGS, U.S. Geological Survey.]

Item ¹	Description ²
Instrument (sensor) specifications:	Visual display - digital readout.
pH and millivolt	pH sensor - range of at least 2 to 12, preferably 0 to 14, pH units. Accuracy, ±0.2 pH units. Millivolt readout - accuracy, ±1.0 mV.
Temperature	Temperature sensor - thermistor range, at least -5 to +45°C. Accuracy, ±0.2°C.
SC	SC sensor - temperature compensating. Accuracy, the greater of 0.5±0.5 percent of reading or ±2 μS/cm.
DO	 DO polarographic sensor (amperometric method) - range from 0.05 to 20 mg/L. Accuracy, the greater of ±2 percent of reading or ±0.2 mg/L. DO optical sensor (luminescent-sensor method) - range from 0.05 to 20 mg/L. Accuracy, the greater of ±1 percent of reading or ±0.1 mg/L.
Turbidity	Turbidity sensor ³ - range and accuracy depend on the instrument type, manufacturer, and field conditions (see NFM 6.7). Choice of instrument will depend on application. Most multiparameter-instrument turbidity sensors use a monochrome light source with a spectral output typically near infrared (780 to 900 nanometers), usually a light-emitting diode. Note: Instrument should include a calibration cup specifically designed by the manufacturer, if available.
ORP ³	ORP sensors - accuracy, ±20 mV. For guidance on Eh measurements using the platinum electrode, refer to NFM 6, section 6.5 and the manufacturer's instructions.
Air pressure	Select instruments that incorporate an altimeter/barometer (measures to the nearest 1 millimeter).
Other sensors ³	Check the text for this section and the manufacturer's instructions for the availability of other sensors.

Table 6.8–2. Specifications and calibration solutions for multiparameter instruments — *continued*

ltem ¹	Description ²
Sensor-calibration	(Keep the respective MSDS guidance on hand in the laboratory and
solutions:	in the field. Dispose of hazardous waste according to regulations,
	using a licensed disposal company.)
pH buffers	Standard buffers are pH 4, 7, and 10. Temperature-correction chart(s)
	supplied by the buffer manufacturer or distributor are required.
SC standards	Use the SC standard(s) recommended by the manufacturer for
	calibration. NOTE: The manufacturer might require a proprietary
	calibration solution. For field verification of the calibration, select
	additional standard(s) that bracket the expected or known sample
	SC. Do not dilute a concentrated standard to prepare a standard of
	lower conductivity.
DO standard	Zero DO calibration solution. Dissolve 1 gram of sodium sulfite and
	a few crystals of cobalt chloride ⁴ in 1 liter of deionized water
	(prepared during the week of use). Cobalt chloride is toxic; check
	the MSDS for safe handling.
ORP standard	ZoBell's solution ⁵
	- This solution contains cyanide and may be harmful if absorbed
	through skin, inhaled, or swallowed. Check the MSDS for safe
	handling.
	- Use a dedicated hazardous waste disposal container for ZoBell's
	solution. Do not pour ZoBell's solution down the sink drain of
	onto the ground. Do not mix with acids or combustible
T 1 ' 1'	materials.
Turbidity	Turbidity standard solutions with various ranges are available
standard	commercially. Most sensor manufacturers recommend either
	formazin-based or SDVB-polymer standards for calibrating their
	turbidity sensors. Do not use gels or solids for calibrating instruments (see sections 6.8.2 and NFM 6.7).
	- Turbidity-free water (deionized water filtered through a ≤0.2-μn
	- furbidity-free water (deformed water intered through a ≤0.2-μm membrane filter).
	- Formazin stock suspension can be obtained commercially or
	prepared in-house from hydrazine sulfate and
	hexamethylenetetramine (safety precautions for handling these
	chemicals are described in NFM 6.7, section 6.7.2)

¹Modify this list to meet the specific needs of the field effort and the specific requirements for the multiparameter instrument to be used.

²The accuracy specification provided in this table has been generalized, based on a survey of three or more manufacturers with instruments in common use among USGS field studies. Consult the manufacturer's operators' manual for the level of accuracy for a specific instrument.

³The turbidity sensor commonly is required or recommended for use (section 6.7). ORP sensors are less commonly used for USGS studies; see the description in section 6.5. Follow the manufacturer's guidance for use of the salinity or TDS option, and for other ion-selective sensors (for example, for nitrogen species and chlorophyll).

⁴Prepare fresh zero DO solution for each use. CAUTION: Use of cobalt chloride is recommended in Standard Methods (American Public Health Association, 2005); however, this is a toxic substance that must be handled with care and disposed of in accordance with prevailing regulations. If possible, prepare a zero-DO solution without using cobalt chloride.

⁵Alternatives to Zobell's are being investigated (January 2008).

EQUIPMENT TRANSPORT 6.8.1.A

Transport the multiparameter instrument in a case that is designed to protect this equipment.

- ► To avoid damaging the sensitive and expensive fieldmeasurement sensors, keep either the sensor guard or transportation/calibration cup installed. Some manufacturers specify adding a small amount of water to the transportation/calibration cup for transport between field sites; follow the manufacturer's recommendations.
- ▶ When packing the instrument for transport, use a case provided by the manufacturer; alternatively, obtain a suitable case, such as a PelicanTM case, Otter[®] box⁶, or a tool box, and modify it as needed.
 - Cases must be padded to absorb shock, using material that does not absorb water.
 - Pelican and Otter boxes are airtight; the case needs to be vented if using sensors that have a flexible or semipermeable membrane.
 - A white or light-colored case should be used to help deflect solar heating of the sonde.

INSTRUMENT MAINTENANCE 6.8.1.B AND STORAGE

Each instrument requires its own (dedicated) log book that accompanies the instrument, in which permanent records of instrument calibrations, bench checks, sensor replacements, general maintenance, and repairs are logged. The following recommendations pertain to maintenance of the multiparameter instrument that is deployed over discrete or short (attended) time intervals. For maintenance of instruments intended for long-term or unattended instrument deployment, refer to Wagner and others (2006) and the instructions provided by the manufacturer.

⁶Examples of transport cases can be found at www.otterbox.com or www.pelican.com (accessed 5/22/2007).

▶ Sensor and sonde care and maintenance:

- Rinse the sensors immediately after each use with deionized water (DIW).
- If the multiparameter instrument (handheld display and sensors-containing sonde) is particularly dirty or will be stored for longer than one day, clean it with a mild, nonphosphate detergent solution using a small, nonabrasive brush or cotton swab or cloth, followed by a thorough water rinse.
- Avoid using organic solvents or other corrosive solutions to clean the sensors.
- O-rings used for some types of dissolved-oxygen sensors are not very robust; inspect such O-rings before each DO measurement and replace if damaged.
- Do not coat the sonde or sensors with protective or anti-fouling paint, except as specifically instructed by the manufacturer.
- Manufacturers may have instructions specific to their sensors—check the manufacturer's operating manual for each instrument that will be in use.

▶ Wiper and wiper-brush maintenance:

- Inspect the wiper pad and (or) wiper brush for dirt, deterioration, and damage after each use of the sonde. (Not all instruments have a wiper or wiper-brush mechanism.)
- Check wiper pads for wear, excessive discoloration, and particle accumulation, and change the pads as needed.
 Check that the wiper arm is parking properly. Follow the manufacturer's guidance for conditions requiring changing the pads and for wiper maintenance.
- A soft toothbrush can be used to clean wiper-brush bristles. Rinse with fresh tap water or DIW.
- Wiper-brush bristles should be kept moist at the start of the operation to prevent them from drying. If the bristles have dried, soak them in DIW and manually loosen them before deploying the sonde.

▶ General care of multiparameter instruments:

- Do not leave instruments in vehicles for long periods of time during extremes in temperature.
- At least once a year inspect cables for damage, and electronic connectors and sensor ports for corrosion.
- Inspect and clean the bulkhead O-rings and grease them with silicone lubricant annually, at a minimum. Replace any damaged O-rings.
- Store cables in a plastic container only after they are clean, dry, and neatly coiled (no tighter than 6-inchdiameter coils). Use protective plugs when cable connectors are not in use. When in use, protect cables from abrasion or unnecessary tension.
- Make sure that the instrument is running on software and firmware that is up-to-date. Check for updates from the manufacturer every 6 months or more frequently and follow the download or other installation instructions.

► General storage recommendations for multiparameter instruments and instrument cases:

- For short-term storage, some sensors need a small amount of the storage solution added to the protective (transport) cap or calibration cup; check the manufacturer's instructions.
- For long-term storage (longer than several weeks), remove the internal batteries; however, be sure to check the instrument manual for guidance before removing all of the batteries.
- Store multiparameter instruments in a carrying case or plastic container with foam cushioning (for shock protection). Keep the instrument and case out of direct sunlight and protected against extremely hot or cold temperatures.
- Insert a sensor-port plug into any vacant sensor port to prevent damage to the vacant port during maintenance, operation, or storage.

Table 6.8–3. General supplies related to field-measurement activities

[DO, dissolved oxygen; mL, milliliter; L, liter; μ S/cm, microsiemens per centimeter at 25 degrees Celsius; ASTM, ASTM International Company; NFM, National Field Manual for the Collection of Water-Quality Data; USGS, U.S. Geological Survey]

ltem ¹	Information
Flowthrough cell	Standard flowthrough cell, obtained from the manufacturer of the instrument. (Commonly used for ground water or other water pumped from the water source to the airtight cell for measurement of field properties.)
Extra sensors and meters	Single-parameter meters and sensors or a multiparameter sonde (as a field backup). Refer to equipment lists and descriptions and instructions provided in NFM 6, sections 6.1 through 6.7.
Membrane- replacement kit for amperometric DO	Membrane-replacement kit (includes membranes or screw-on membrane caps, O-rings, filling solution).
Calibration (laboratory) thermometer	Liquid-in-glass or electronic-thermistor thermometer, either NIST-certified or manufacturer-certified as NIST-traceable. (See NFM 6, section 6.1 for USGS standard specifications.)
Field thermometer	Non-mercury liquid-in-glass or thermistor thermometer that has been office-laboratory certified against a properly certified calibration thermometer. (See NFM 6, section 6.1 for USGS standard specifications.)
Turbidity container and flasks	Bottle for turbidity-free water, cleaned and rinsed three times with filtered water before starting each field trip. Volumetric flask, Class A, 100 mL or 500 mL, if dilution of stock solutions is necessary (see section 6.8.2).
Carrying case	Protective case, vented, white or other reflective color, to hold the multiparameter instrument during transport and storage.
Holding stand ²	A stand to support the multiparameter sonde during calibration.
Log book(s) ³	One log book per instrument (multiparameter and single- parameter), for recording instrument calibrations, maintenance, and repairs. Log book travels with the instrument.
Flasks, beakers, and other measurement vessels	Insulated flask or beaker and additional polyethylene or Teflon® preferable beakers for temperature check or other field needs. Assorted sizes, 50 to 150 mL. Beakers must be clean but not acid rinsed.
Deionized water (DIW)	1 L of DIW with a maximum conductivity of 1 μS/cm (ASTM Type 1) for rinsing sensors.
Paper tissues	Laboratory grades (for example, lint free and (for turbidity) extra lint free Kimwipes [®]), soft, disposable.

Table 6.8–3. General supplies related to field-measurement activities — *continued*

Item ¹	Information
Dispenser (squeeze) bottles	Polyethylene to contain DIW; for rinsing instruments and instrument sensors.
Disposable gloves	Laboratory gloves, disposable, non-powdered and of a material suitable to contact anticipated chemical solutions and environmental waters or wastewater. Keep a supply on hand in the field vehicle.
Brushes for equipment cleaning	Brushes of various sizes, but generally small and soft to prevent scratching the sensor(s) or other surfaces.
Minnow bucket with tether, mesh bag, or equivalent	Used to contain fresh sample water into which tightly capped calibration solutions are immersed for thermal equilibration with the temperature of the sample water before being used for sensor calibration.
Antistatic spray or polish	Used on the digital display screen of a multiparameter instrument.
Cleaning solution	1 L of nonphosphate laboratory detergent (see NFM 3 for solution-concentration guidelines).
Batteries and/or battery pack(s)	Check that batteries are fully charged; bring spares.
Safety equipment	Select safety equipment appropriate for the field effort conditions, such as gloves, eye protection, face mask, apron, chemical spill kit, first-aid kit.
Waste-disposal containers	Appropriate for safe containment of regulated (hazardous or toxic) substances and dedicated to use for the respective waste material (examples: ZoBell's solution, methanol, and acid and turbidity calibration solutions).

 $^{^{1}\}mathrm{Modify}$ this list to meet the specific needs of the field effort.

²USGS personnel may check for the availability of instrument stands (HIF # 6103032 or #6103035) at the USGS Hydrologic Instrumentation Facility.

³Bound log books with water-resistant pages are available to USGS personnel through the USGS One Stop Shopping store.

6.8.2 CALIBRATION

Multiparameter instruments must be tested and the sensors calibrated before each field use. With some exceptions (for example, turbidity calibration), calibrations are performed in the field in preparation for making measurements.

When visiting more than one site for field measurements, the sensors and sonde housing must be cleaned and then the sensor calibration verified for each site. Field calibration should be completed in an area sheltered from wind, dust, and temperature fluctuations. Consult the manufacturer's guidelines before beginning the calibration process and contact the manufacturer's technical support if problems or questions arise.

Ensure that the sensors are properly installed in the sonde. Before beginning the calibration process, check the power source; only use batteries that indicate a full charge.

- ▶ Most multiparameter instruments perform best if allowed to warm up for at least 10 minutes after being turned on, or according to the manufacturer's recommendation.
- ► The following order is recommended for performing calibration or accuracy checks in the field:
 - 1. **Temperature** (using a thermometer that has been calibrated and office-certified, as described in NFM 6.1)
 - 2. **Specific electrical conductance (SC)** (note that the value of the SC standard solution changes by more than 3 percent when the temperature is less than (<) 6°C or greater than (>) 40°C; do not calibrate with standards <6°C or >40°C.
 - 3. **Dissolved oxygen (DO)** (amperometric or luminescent-sensor methods using polarographic or optical sensors, respectively)
 - 4. **pH** (be sure to check and adjust for the buffer pH value at the buffer temperature)
 - 5. Oxidation-reduction potential (ORP)
 - 6. **Turbidity** (most manufacturers recommend that the turbidity calibration be performed in a laboratory or other stable environment)
 - 7. Ion-selective electrodes, followed by chlorophyll-fluorescence and other sensors.

- ► Complete the calibration field form during calibration (Appendix 6.8–A). Accurate calibration records must be maintained and entered into the appropriate instrument log book at the time of calibration.⁷
 - Keep a hard copy of the field form in the field or site folder. These records contain vital information that can be referenced if technical or legal questions arise.
 Interpretation of data analyses or data quality may depend on the documentation regarding instrument performance and the calibration solutions and the methods used, in addition to the results recorded. This record should be checked and verified by a second or third party.
 - The field form documents that a sensor has met the dataquality objectives of the study and that the calibration was performed according to the required standard operating procedures. Lot numbers and expiration dates of calibration solutions are recorded on the electronic or paper field form (Appendix 6.8–A).
 - The instrument log book is the archival document for recording details chronologically, including calibrations, maintenance specific to the sensors, and general repairs. Log book entries should be recorded using black or blue ballpoint ink, preferably on water-resistant paper with the pages consecutively numbered and bound to deter page removal. To ensure the legal viability of the log-book record, a page never should be removed and a single line should be drawn through any erroneous information or data and initialed. (USGS personnel can obtain log books through One Stop Shopping).
- ► Clean the instrument onsite after each use to reduce the potential for site and sample cross contamination and loss of calibration.

Reagents used for calibration may be hazardous to health and require special handling. Review the MSDS for the reagent of concern. Keep the safety sheets handy.

⁷For USGS studies, the worksheet is included in the electronic (PCFF) and paper national surface-water and ground-water water-quality field-notes forms. Meter-calibration log books are available to USGS personnel through One Stop Shopping.

6.8.2.A STANDARD USGS CALIBRATION PROCEDURES FOR MULTIPARAMETER INSTRUMENTS

The results of sensor calibrations are recorded on a field form at the time of calibration (Appendix 6.8–A provides an example of a field form for recording calibrations and field measurements). In addition, a historical record of calibrations for each sensor used in a given multiparameter instrument must be kept in an instrument log book that accompanies the instrument to the field. This log book also is used to document maintenance, repairs, and sensor replacements for the instrument

When calibrating multiparameter-instrument sensors:

- 1. **Follow the manufacturer's instructions** for the instrument model and sensors being used.
 - Become familiar with the operation and setup of the handheld or other display hardware and software. Make sure that the batteries are fully charged, or install fresh batteries.
 - Ensure that the instrument has been set for the appropriate measurement unit, if this option is available.
 - Ensure that the instrument has been warmed up for the amount of time recommended by the manufacturer.
- 2. Bring calibration solutions (calibrants) to the temperature of the sample source, to the degree possible. Note there are exceptions to this protocol for SC and turbidity, as described below. To allow equilibration of the calibration solutions with ambient sample-water temperature, calibrant containers can be partially immersed in the stream being sampled, or in a bucket to which the ground water being sampled is pumped. Great care must be taken to prevent water from getting close to the top of the calibrant container and contaminating the calibrant.

⁸For calibration of sensors for turbidity and specific electrical conductance, check with the manufacturer for guidance.

- Calibrate the instrument in a temperature-stable environment, out of the wind and direct sunlight.
- Use the calibration cup that comes with the instrument for calibration, unless otherwise instructed by the manufacturer. If a calibration cup is not available, follow the manufacturer's alternative recommendations.
- Use the recommended volume of calibrant when filling the calibration cup. The calibrant must cover the temperature sensor, as most sensors require temperature compensation.
- Be careful not to overtighten the calibration cup. This is especially important for DO calibration. Many calibration cups have vents that allow their equilibration with ambient pressure.
- For SC, do not equilibrate the temperature of the standards to that of the sample source if source temperature is less than 6°C or greater than 40°C, because the value of the SC calibration standard changes significantly (by more than 3 percent) as a function of temperature at these temperature extremes. In such situations, perform the SC calibration inside a room or vehicle in which the ambient temperature of the standards is maintained at a temperature >6°C and < 40°C.
- For turbidity, calibrations should be performed in an environment that is protected from wind and thermal fluctuations.
- 3. Rinse sensors thoroughly three times with deionized water after use of each calibrant, followed by three rinses with the next calibrant to be used.
 - To avoid dilution of calibration solutions, gently shake excess rinse water from sensors.
 - Use a lint-free laboratory tissue (for example, Kimwipes[®]) to absorb water droplets without touching or wiping the sensor surface; never touch or wipe the transparent surfaces associated with luminescent DO, pH, and turbidity sensors.
- 4. Calibrate the SC and DO sensors before calibrating the pH sensor. This helps prevent contamination of the SC sensor from pH buffer solutions (pH buffers have much higher conductivities than most environmental waters).
- 5. **Periodic removal and cleaning of sensors** may be needed for any multiparameter sonde that is deployed for long-term monitoring. The time interval between cleanings will depend on site conditions and study requirements.

Bring calibration solutions (standards and buffers) to the ambient temperature of the environmental sample to the degree possible.

6.8.2.B SENSOR-SPECIFIC CALIBRATION TIPS

The following guidelines comprise standard USGS procedures.

- ► Check sensor ports to be sure that either the ports have a properly installed sensor or that the empty ports are sealed. Sensors from which data are not being collected routinely can be removed from the sonde for safe storage, provided that the sensor is not necessary for the measurements of interest and provided that the empty port is sealed according to the manufacturer's instructions. The temperature sensor should not be removed. All electrical connections must be clean, dry, waterproof, and protected from dust.
- ► Clean sensors after each use and keep them maintained and stored according to the manufacturer's instructions.
 - Before calibrating and using an instrument in the field, inspect the sensors to be sure that they are clean and are not damaged.
 - Periodic cleaning may be needed for any instrument that is deployed for continuous monitoring (see Wagner and others, 2006).

Temperature (see also NFM 6.1, section 6.1.2)

Check to ensure the accuracy of the temperature sensor at least every 3 months if the multiparameter instrument is in frequent use. The accuracy of pH and other field measurements depends on the accuracy of the temperature measurement.

- 1. Verify the accuracy of the temperature sensor against a certified NIST-traceable digital or liquid-in-glass thermometer, following the guidelines provided in NFM 6.1 (annual laboratory verification and biannual field checks are mandatory for USGS studies). For the calibration check, the NIST thermometer and sonde thermistor should be as close together as possible without touching. For field verification, use a non-mercury thermometer that has been certified as accurate within the past 6 months and is tagged as such by the verifier. When making the field check, record the temperature readings of both the multiparameter instrument and the NIST-traceable thermometer in the instrument log book.
 - If the difference between the readings does not fall within the manufacturer-specified accuracy, return the instrument to the manufacturer for repair or replacement.
 - See NFM 6.1 for a description of the annual and biannual calibration protocol for liquid-in-glass and digital thermometers, which also require calibration checks.
- 2. Make sure that the temperature sensor is completely submerged.
- 3. Allow at least 1 minute for temperature equilibration and stabilization before recording the temperature value and proceeding with the other measurements.

Specific Electrical Conductance (SC) (see NFM 6.3, section 6.3.2)

- 1. Most multiparameter instruments use a one-point calibration to calibrate the SC sensor. Use a standard having the conductivity recommended by the instrument manufacturer; otherwise, select a standard that is close in conductivity to that of the environmental water.
 - Rinse the calibration cup and sonde using a small amount of standard. **Repeat this two more times** and then fill the cup with the recommended volume of standard.
 - The sensor should be completely submerged in the standard (if a hole exists in the side of the sensor, it must be covered by the standard). Low fluid level can cause an erroneous calibration or may result in an error message on the instrument display.

- The presence of air bubbles in SC electrodes will cause erroneous readings and incorrect calibration.
- Although most SC sensors are shielded from effects caused by proximity to transmission lines and to alternating-current (AC) electrical outlets and radio-frequency noise sources, be aware of the possibility of this interference and check with the instrument manufacturer.
- 2. Wait for readings to stabilize (approximately 30 seconds under normal conditions) before adjusting and saving the calibration point.
 - The USGS reports SC in units of microsiemens per centimeter (μS/cm). The default SC setting on many multiparameter instruments often is in units of millisiemens per centimeter (mS/cm). Either change the setting to μS/cm (if this option is available) or measure in mS/cm and then convert to μS/cm (multiply mS/cm by 1,000). To fulfill USGS data protocols, record the SC value in μS/cm on paper or electronic (PCFF) field forms.
 - Do not override a calibration error message without troubleshooting and correcting the cause of the error. For example, check the fluid level and check for air bubbles in the sensor.
- 3. To verify that the accuracy of the SC sensor is within the range of the conductivities to be measured:
 - Ensure the linearity of response of the SC sensor at low-conductivity values and check the zero response of the dry sensor in air (Wagner and others, 2006).
 - Select two standards ("check standards") that bracket the expected SC range of your water as closely as possible; a third standard that is at or close to the actual ambient conductivity helps to pinpoint the accuracy of the sensor. Equilibrate the temperature of the standard to that of the water body, unless the water temperature is < 6°C or > 40°C (use of this protocol can depend also on instrument software consult the manufacturer's guidance). Follow the same procedure as for an actual calibration, but do not lock in or adjust these readings—this is an accuracy check, not a calibration point.

Handle conductivity standards in a manner so as to prevent their dilution or contamination.

- Do not use expired standards.
- Do not reuse standard or pour used standard back into the bottle.

Dissolved Oxygen (DO) (see also NFM 6.2, section 6.2.1.B)

Two sensor options are available for the DO measurement when using multiparameter instruments: the polarographic (or Clark cell) sensor or the luminescent (optical) sensor. Referring to NFM 6.2 on DO measurement methods, the polarographic-sensor option corresponds to the amperometric method, and the optical-sensor option corresponds to the luminescent-sensor method.

General comments:

- Follow the manufacturer's guidelines for care, proper setup, and calibration of the DO sensor for the instrument in use. For either sensor type, most manufacturers recommend that the sensor be allowed to equilibrate to the temperature of the air-saturated water or water-vapor-saturated air for at least 15 minutes before calibration.
- Before calibrating for 100-percent saturation of DO, loosen the calibration cup. (It should contain less than 1/8 in. (~0.32 cm) of water, or as recommended by the manufacturer.)
- Remove any water droplets from the thermistor or the DO membrane without wiping the membrane. Water droplets on these surfaces can cause a temperature compensation error in the DO calibration.
- Store and transport the sonde in a padded, vented, white (or light-colored) case to make DO calibration checks quicker and reduce
 the chance of DO sensor drift (since the instrument is in a more
 temperature-stable environment and can be calibrated within the
 cooler).
- Calibrate the DO sensor on the morning of the field day and check the calibration at each measurement station. Enter the barometric pressure (see NFM 6.2 for an explanation of corrected and uncorrected values).

TECHNICAL NOTE: Check the manufacturer's instructions regarding the need to recalibrate amperometric-instrument sensors with changes in altitude. For some instruments, the DO sensor should be recalibrated at each site at which there is a change of approximately 900 ft (~ 300 m) in altitude. Luminescent sensors tend to keep calibration over extended time periods; however, verification of sensor performance with appreciable altitude change is recommended to quality assure and document sensor performance. To convert inches (in.) of mercury (Hg) to millimeters (mm), multiply inches by 25.4.

- The calibration procedure depends on the type of DO sensor being used. Note the type of sensor being used—amperometric or optical (luminescent)—and follow the appropriate instructions provided by the manufacturer and as described below. Allow the sensor to equilibrate to the temperature of the solution for at least 15 minutes or as recommended by the manufacturer.
- Always perform a 100-percent saturation calibration before beginning the zero DO calibration.

Amperometric method for DO measurement (polarographic or Clark-cell sensor):

Instrument makes and models can vary considerably; always refer to the manufacturer's instructions for the instrument that is in use. To prevent water damage to the sonde's internal parts, maintain the O-rings and sealing surfaces on the sonde as directed by the manufacturer. Be aware that extreme temperatures and instrument vibrations may cause the DO sensor to drift out of calibration on a day when a series of measurements is made.

- 1. Inspect the DO sensor anodes and cathodes—if they are not bright and shiny, recondition them as instructed by the manufacturer.
- Install a new membrane or membrane cap of the desired membrane thickness. If not using the membrane cap, the membrane should be tightly stretched, and have no bubbles, wrinkles, or tears. Replace any worn or stretched (loose) O-rings.
 - Membrane replacement should take place 24 hours before use (USGS standard procedure). Manufacturer guidance generally specifies membrane replacement at least 3 to 4 hours before use (M. Lizotte, YSI and Bruce Wilcox, Hach Environmental, written communs., May 2007).

- A tight-fitting O-ring is critical to good sensor performance.
- Run or power up the newly membraned sensor for 15 minutes.
- Do not allow electrolyte solution to wet the sensor or sonde connector or other O-ring sealed areas. Electrolyte solution is highly conductive and will short out electrical connections.
- 3. A wet towel can facilitate the water-saturated air calibration of the DO sensor as follows: wrap the sensor guard with a white towel wetted in field temperature water, forming an enclosed moist environment around the instrument sensor guard and sonde body. Allow time for the air inside the sensor guard and wet towel to become saturated with water vapor (10 to 15 minutes).
- 4. Rinse the DO sensor thoroughly, at least three times, with DIW or tap water after being calibrated in the zero-percent solution, to avoid cross contamination and faulty readings. Inadequate rinsing will cause negatively biased readings.

Luminescent-sensor method for DO measurement (optical sensor):

Great care is required when calibrating optical DO sensors in the field. Optical DO sensors (like polarographic sensors) can be calibrated in either water vapor-saturated air or in air-saturated water (see NFM 6.2). The air-saturated water method is recommended for calibrating optical sensors. Temperature equilibration of the sensor with the calibration solution must be achieved before proceeding with the calibration; follow the manufacturer's instructions.

- 1. To create an air-saturated water bath, one method is to fill a 5-gallon pail with tap water and aerate the water using a mid-sized aquarium air pump with air stone. Check the manufacturer's recommendations. Some manufacturers have developed their own bath aeration system to help avoid effects from variance of temperature and hydrostatic pressure on the calibration (R. Mooney, In-Situ Inc., written commun., May 2007).
 - The air-saturated water method is faster and guarantees temperature equilibration of the optical DO sensor and calibration medium.
 - If the water bath is kept air-saturated and ready to use, calibration time can be reduced, as there is no need to wait for a calibration cup or wet towel to saturate the air.

- 2. Aerate the water for at least 1 hour prior to use.
- 3. When measuring in low DO environments or after replacing a luminescent-sensor membrane, a two-point DO calibration and (or) a zero DO check is needed or required.
 - If the sensor is equipped with a wiper, remove the wiper before starting the calibration (see the warning in step 5 below).
 - Calibrate the saturated and zero DO levels following each manufacturer's specific instructions.
 - To prepare a zero DO calibration solution, dissolve 1 gram of sodium sulfite and a few crystals of cobalt chloride in 1 liter of DIW (prepare this solution during the week of use). Check the Material Safety Data Sheet (MSDS) for handling of cobalt chloride, which is a toxic substance.
- 4. Observe the readings for DO; when there is no appreciable change for approximately 30 seconds, lock in or adjust the reading.
- 5. After calibrating the sensor with the zero-percent solution, take extra care in rinsing the sensor thoroughly to remove any residue of the solution. Inadequate rinsing will cause negatively biased DO readings and can result in cross contamination, possibly causing faulty SC or pH readings. The three-time tapwater or DIW rinse recommended for the amperometric-instrument sensor may not be sufficient. One manufacturer recommends rinsing the sensor under running tap water for at least 10 minutes.

WARNING: On optical sensors equipped with wipers, remove the wiper before beginning the zero-DO calibration to prevent the wiper from soaking up sodium sulfite and thus contaminating the membrane when the wiper is activated.

pH (see also NFM 6.4, section 6.4.2)

- 1. **The pH measurement requires a two-point calibration.** Select the pH 7 buffer plus a second pH buffer (for example, pH 4 or pH 10) that brackets the expected range of sample pH.
 - Use historical pH data for the sampling site, if available, to select the correct buffers.

- After performing the calibration, a calibration check with a third buffer can be useful if the pH range is unknown or if sites with differing range in pH value will be measured.
- Do not use expired buffers. Discard decanted buffer after one use—do not reuse buffers or pour decanted buffer back into the original container.
- 2. Bring the buffers as close as possible to the ambient temperature of the water being sampled.
- 3. Normally the sensor is calibrated first against the pH 7 buffer; however, this may differ among manufacturers.
- 4. Rinse the sensors and calibration cup, first with DIW and then with the buffer.
 - a. Before using the first buffer, rinse the pH and temperature sensors and the calibration cup three times with the first buffer.
 - b. Fill the calibration cup with enough buffer to completely cover the temperature and pH sensors.
- 5. Allow time for the pH and temperature sensors to equilibrate to the temperature of the buffer.
- 6. Record the temperature reading after it has stabilized. The pH value is temperature dependent. Use the chart provided by the buffer manufacturer to determine the true pH value for the buffer at that temperature. You will need to adjust the calibration reading to that value. NOTE: Buffers from different manufacturers can yield somewhat different pH values for a given temperature.
- 7. Follow the manufacturer's instructions for calibration with the first buffer.
 - a. Record the temperature, pH, and millivolt (if available) readings before and after calibration with the first buffer.
 - b. If your instrument does not display the percent slope, then calculate and record the slope of the pH sensor.

EXAMPLE: The acceptable tolerance for the pH 4 buffer is 180±50 mV; for the pH 7 buffer, 0±50 mV; and for the pH 10 buffer, -180±50 mV. If a value of +3 mV were recorded for the pH 7 buffer and -177 mV were recorded for the pH 10 buffer, the slope would be 180 mV. The acceptable range for the slope is from 165 to 180 mV.

- 8. Repeat steps 4, 5, 6, and 7 using the second buffer.
- 9. If a third buffer will be used to check the calibration range of the sensor, follow the same general procedures described above for the first and second buffers, but do not lock in a calibration. The instrument reading should be within ±0.2 pH units of the theoretical pH value at the buffer temperature.

Oxidation-Reduction Potential (ORP or Eh) (see also NFM 6.5, section 6.5.2)

- 1. The pH sensor must be calibrated and working properly before calibrating the ORP sensor, if the instrument uses a combination pH-ORP electrode.
 - For most multiparameter instruments, the ORP electrode usually is combined with pH electrodes in one sensor body in order to utilize a common reference electrode (usually the silver/silver-chloride electrode).
 - Recommended calibration procedures differ among instrument manufacturers. Follow the manufacturer's recommendations for calibration of the specific instrument and electrodes being used.
- 2. A one-point calibration at a known temperature generally is used to calibrate the ORP sensor. The ORP measurement should stabilize within 1 to 3 minutes.
 - Table 6.8–4 shows the true readings in millivolts for ZoBell's solution as a function of temperature for the platinum/silver-silver chloride paired electrodes. These values must be converted to a standard hydrogen reference electrode when the field measurements are reported in the USGS National Water Information System (NWIS) QWDATA database. See NFM 6.5 for more detailed information about ORP sensors, data conversion to the standard hydrogen reference electrode, and use of ZoBell's solution.
 - The calibration values should be within a tolerance of ±5 millivolts of the values listed in table 6.8–4.
 - ZoBell's solution is toxic; handle with care.⁹
- 3. Calibration can be affected by static electricity. Avoid touching the sensors during calibration and measurement.

⁹Alternatives to ZoBell's solution are being investigated (January 2008).

- 4. The ORP sensors of some manufacturers must be oriented near the vertical ±45 degrees for proper operation. Be thoroughly familiar with the manufacturer's instructions before using the instrument.
- 5. Follow proper procedures for handling and disposal of ZoBell's solution and keep an MSDS for ZoBell's solution with the ORP equipment. Minimize the volume of ZoBell's solution being used and store the spent solution in a separate, dedicated container.

Table 6.8–4. Voltage of ZoBell's solution as a function of temperature for the platinum/silver-silver chloride paired electrodes

[°C, degrees Celsius; mV, millivolts]

Temperature, in °C	ZoBell's solution, ¹ in mV
-5	270.0
0	263.5
5	257.0
10	250.5
15	244.0
20	237.5
25	231.0
30	224.5
35	218.0
40	211.5
45	205.0
50	198.5

¹This table is provided as a courtesy by YSI (M. Lizotte, written commun., February 2006). See table 6.5–3 in NFM 6.5 for a chart showing the Eh of ZoBell's solution as a function of temperature.

ZoBell's solution is a toxic solution and considered a hazardous waste. Check with a chemical-substances safety officer and the MSDS for safe handling information and proper and legal disposal of spent ZoBell's solution.

Turbidity (see also NFM 6.7, section 6.7.2)

The methods and standards used for turbidity sensor calibration should be those that are recommended by the instrument manufacturer for the specific instrument type and model, using NFM 6.7 as a guide for USGS work.

Calibration of the turbidity sensor is highly sensitive to environmental fluctuations and should be performed away from wind, sunlight, and temperature fluctuations. (Most manufacturers recommend that the turbidity calibration be performed in a laboratory or other stable environment before departing for the field site. To some extent this is dependent upon the calibrant being used; for example, formazin use is confined to a laboratory environment. USGS protocol stipulates that calibration of the turbidity sensor be verified at each field site. Refer to NFM 6.7 for a detailed explanation.)

- ▶ Calibrants are not necessarily interchangeable. Serious calibration errors can result from using the wrong standards. Three types, or levels, of standard turbidity solutions (calibrants) are used to calibrate and (or) verify the accuracy of turbidity sensors (section 6.7.2). Use only those calibrants that are prescribed for the sensor by the instrument manufacturer. Refer to NFM 6.7 for detailed information on turbidity calibrants and for turbidity units of measurement as operationally assigned according to instrument type by the USGS. ¹⁰ The following terminology, taken from ASTM Method D6855, is used by the USGS to distinguish among classes of turbidity standards (C.W. Anderson, U.S. Geological Survey, written commun., December 2006; ASTM International, 2003):
 - Reference standard: 4000 NTU formazin solution, obtained commercially or prepared in-house ("from scratch").
 - Calibration standard: Diluted scratch formazin, StablCal[®] or styrene-divinylbenzene (SDVB) polymer.
 - Verification standard: Gels, solids, or diluted SDVB or StablCal.

¹⁰The guidelines for reporting turbidity units described in NFM 6.7 were developed jointly by the USGS, ASTM International, and participating instrument manufacturers.

- ▶ Diluting a reference standard for turbidity calibration can result in erroneous data and, in general, is not recommended.
 - Precise laboratory technique is essential for dilutions and should be performed only by experienced personnel. If not handled carefully, the dilutions can become unstable and particle suspension may be lost.
 - Discard a diluted scratch formazin calibration standard within 24 hours of preparation.
- ► The quality of the turbidity measurement is dependent on the type of standard (that is, on the particulate matter contained in the suspension) that is used to prepare instrument calibration curves.
 - Turbidity-free water, used as a zero-turbidity standard and for the preparation of standard solutions, dilutions, and equipment rinsing, is prepared as described in NFM 6.7.
 - Formazin-based calibration standards are freshly prepared by diluting a 4,000 NTU reference standard, using the dilution formula provided in NFM 6.7. Because the dilution process is subject to preparation errors, document that a calibration standard was used and report it as "calibration standard, prepared by dilution of a 4,000 NTU standard." A calibration standard must be prepared on the day of use and be discarded on the same day.
 - Record the source of the 4,000 NTU reference standard.
 The 4,000 NTU standard has a shelf life not to exceed 1 year.
 - o The diluted scratch formazin (calibration standard) has a shelf life of less than 24 hours.
 - o Do not use expired standards (American Public Health Association, 2005, Method 2130B, p. 2–9 to 2–11).
 - Do not dilute SDVB polymer or StablCal standard for use as a calibration standard. Although a diluted polymersuspension (less than 10:1) sometimes is used as a verification or calibration check (verification standard), this is not recommended by the USGS and should not be used for USGS studies.
 - Store the verification standards out of sunlight and in PVC bottles.
 - Handle verification standards carefully to maintain the stability of the suspension.

► Check the turbidity standards for expiration before performing a dilution, calibration, or calibration verification. Note that higher range formazin standards tend to settle and thus are less stable than lower range formazin standards.

The following summary of turbidity sensor calibration does not replace the more detailed information to be found throughout NFM 6.7, and specifically in section 6.7.2.

- 1. If the sonde includes a wiper brush and (or) pad for cleaning the DO, pH, and SC sensors, this brush must be removed before calibrating the turbidity sensor. If the wiper occupies a sensor port, be sure to plug the open port before starting the calibration.
- Perform the turbidity-sensor calibration in a protected environment, away from wind and thermal fluctuations.
 Standard USGS procedure is to calibrate sensors onsite, but in a location in which stable environmental conditions can be maintained.
 - Prevent disturbance to the standard solutions that might result in forming bubbles, and prevent exposure of these standards to direct sunlight.
 - Verify calibration of the turbidity sensor in an environment in which stable readings can be obtained.
 - If the calibration is performed in a laboratory just before departing for the field site, use a verification standard onsite to check the sensor calibration.
- 3. Use only the recommended calibration standards for actual calibration of the sensors. A verification standard may be used to check the calibration in the field.
- 4. Use the manufacturer-supplied calibration (or storage) cup with a non-reflective endcap.
 - Do not use plastic beakers or containers when working with sensors that use infrared light; clear plastics can reflect the infrared light beam and cause errors.
 - Clear glassware may be used with the sensor guard installed on the sonde.
 - Do not use small-diameter or small-volume containers (for example, 35-mm film-storage containers) for this purpose.

5. Inspect the instrument carefully.

- a. Check the instrument—ensure that all submerged parts of the multiparameter instrument are clean before beginning turbidity calibration. Sediment or other particulates from the sonde, wiper, or other parts can contaminate the standard, leading to an incorrect calibration and measurement.
- b. Check the optical ports—the optical surface of the turbidity sensor must be clean and free of bubbles, fingerprints, scratches, or other interferences.
- c. Check the wiper—if your turbidity sensor has a wiper with a pad or brush, inspect the condition of the pad/brush and replace it if necessary. Check that the wiper is parking properly and is operational.
- d. If the sensor is without a mechanical wiper (for example, during discrete sampling), take extra care to maintain a clean, bubble- and solid-material-free optical face. To remove bubbles from the optical face during calibration or field measurement, agitate the sonde by moving it in a vertical or circular motion.
- 6. Check the manufacturer's instructions for the minimum distance between the sensor face and the bottom of the calibration chamber, before and during the calibration process. Take care to avoid interference from the bottom of the calibration vessel.
- 7. Note that if the sensor is equipped with a wiper (or brush), the wiper (or brush) needs to be activated immediately before the calibration data are acquired.
- 8. When verifying the turbidity-sensor calibration, a three-point check is recommended before deciding to adjust the calibration.
 - If the sensor readings exceed the established calibration criteria for project data-quality objectives (for example, the greater of ±5 percent of the measured value or 0.5 turbidity units) during the inspection process, the sensor requires calibration.
 - If instrument calibration allows only a two-step process, use two
 calibration standards that cover the expected turbidity range and
 check for linearity using a third midpoint standard. If the
 instrument calibration requires only turbidity-free water and one
 calibration standard, select a midpoint standard to check for
 linearity.

TECHNICAL NOTE: The range of standards recommended for verification of turbidity-sensor calibration varies, depending on the manufacturer and the linearity of the instrument being used.

- 9. Perform multipoint calibrations in the order of increasing turbidity.
 - a. First rinse the calibration cup, turbidity sensor (and sensor guard) three times, each time using a small amount of zero-turbidity solution.
 - b. Using the zero-turbidity solution, carefully fill the calibration cup along the inside surface, so as to avoid aerating the solution. Set the multiparameter instrument on top of the calibration cup (do not engage the threads). Verify that there are no air bubbles on the sensor face; then run the wiper (if present) at least once before accepting the first calibration point. Record the first calibration point. Use 2 Formazin Nephelometric Units (FNU) as the low-end calibration point.

TECHNICAL NOTE: Consult the instrument manufacturer if the accuracy and precision of measurements below 2 FNU are important for the study, as calibration procedures within the 0 to 2 FNU range can differ depending on the instrument. Some manufacturers advise that instruments can be better calibrated to 2 or to 10 FNU than to 0 FNU

- c. Before using the next standard, re-rinse the calibration cup, sensor guard (if present), and sensor three times with the zero-turbidity solution. Repeat this rinse between each new standard.
- d. To assess the actual performance of the instrument near the detection limit, periodically check using standards in the 1 to 5 turbidity-unit (low-level) range.
- e. Calibrate at the second point, again removing air bubbles and wiping the sonde or sensor at least once before accepting the value.

- f. Monitor each output carefully to ensure that turbidity readings are stable before confirming the calibration value. Report the measurements in the proper units, as specified in NFM 6.7, table 6.7–4.
- g. Never override a calibration-error message without fully troubleshooting the cause of the problem. Calibration-error messages usually indicate that a problem exists that will result in incorrect field readings.
- 10. While in the field, check instrument performance periodically using either a calibration standard (StablCal, SDVB polymer, or diluted scratch formazin) or a verification standard (gels, solids, or diluted SDVB or StablCal) and turbidity-free water.

TECHNICAL NOTE: Field experience is the best guide as to how often the turbidity sensor will benefit from recalibration. The need for recalibration depends on the condition of the optical windows, which in turn depends on the environment in which the instrument is deployed. Instruments deployed in biologically active environments, for example, require frequent cleaning and calibration checks. Periodic checks of the sensor against calibrants can be beneficial for indicating how well the sensor is holding its calibration.

WARNING: Contamination of the zero turbidity standard (from inadequately cleaned equipment) often is the cause of negative turbidity readings in clear environmental waters. Contact the instrument manufacturer for recommendations if negative turbidity readings cannot be eliminated.

6.8.3 **MEASUREMENT**

The field-measurement procedures implemented depend on the type of water body for which the chemical and physical properties are being determined, onsite characteristics and conditions at the time of measurement, and on the study objectives and data-quality requirements of the project. Refer to the respective sections of this chapter for detailed information regarding field measurement of temperature, specific electrical conductance, dissolved-oxygen concentration, pH, oxidation-reduction potential, and turbidity.

- ► Record a description of site conditions and any anomalies at the time of sampling.
- ► Allow time for the readings on the display to stabilize within the criteria shown on table 6.8–5.
- ▶ Record all required and targeted field measurements on the appropriate paper or electronic field forms, laboratory analytical request forms, project log books, chain-of-custody logs, and other documentation that might be required for the study (Appendix 6.8–A). Note on the appropriate forms any onsite conditions that could have affected the quality of the data.

Table 6.8–5. Standard criteria for stabilization of common multiparameter-instrument sensors

[\pm , plus or minus; °C, degrees Celsius; %, percent; \leq , less than or equal to; μ S/cm, microsiemens per centimeter; >, greater than; mg/L, milligrams per liter; FNU, formazin nephelometric units]

Sensor	Standard sensor stabilization criteria (Note that the actual accuracy of the sensor varies, depending on sensor model and manufacturer)
Temperature (thermistor)	± 0.2°C
Specific electrical conductance (SC)	± 5% for SC ≤100 μS/cm, or ± 3% for SC >100 μS/cm
Dissolved oxygen (polarographic or optical)	± 0.2 mg/L
рН	± 0.1 pH unit; if drifting persists or if measuring low- conductivity waters (≤75 μS/cm), allow ± 0.2 to ± 0.3 pH units
Turbidity	± 0.5 FNU or 5% of the measured value, whichever is greater, for turbidity 100 FNU, or 10% of the measured value, for turbidity >100 FNU

SURFACE WATER 6.8.3.A

Field measurements commonly are monitored within a cross section of the surface-water body to (a) help determine how well mixed the stream is, and consequently the sampling method to be used (NFM 4.1), and (b) determine the field-property values of the water body at the selected site. In situ use of a multiparameter instrument is the most efficient means of obtaining such data.

- Many instruments include a pressure transducer that produces a value for water depth or level. For instruments without pressure transducers, the approximate depth of the sonde as it is lowered through a transect can be noted by placing incremental marks along the instrument cable or be connected to a pressure transducer. Depending on site conditions, the sonde might need to be weighted (consult the manufacturer).
- ▶ Wait a minimum of 60 seconds for the sensors to reach thermal equilibrium with the water temperature at each new location. Some instruments require a longer equilibration time; check the manufacturer's recommendations.
- At each measuring point, allow the field-measurement values on the instrument display to stabilize within an established criterion before recording final field measurements (table 6.8–5).
 - Field-measurement values generally are considered stable if the variability among three or more consecutive readings, spaced some number of minutes apart, conforms to the designated criteria. See NFM 6.0 for a discussion on sensor-stabilization criteria.
 - After making multiple measurements across a stream transect, return to the original measurement location within the transect and make a second measurement at that location, to check for measurement stability. Repeat the transect measurements if the original measurement is not replicated within the stabilization criterion shown on table 6.8–5.
 - When aggregating the data from a cross section, document the median of the cross-sectional data for each field measurement.
- ▶ Biological growth or debris in the water can foul sensors, which will adversely affect sensor readings. If field conditions and quality-assurance protocols allow, adjust the spacing of the measurement intervals along the cross section or transect in order to avoid areas that will result in having to stop and clean algae, sediment, or debris from the sensors.

6.8.3.B GROUND WATER

The stability of field-measurement values is monitored toward the end of well purging to help indicate when the water being withdrawn represents fresh formation water and when sample collection for other analytes should begin (NFM 4.2). The final field measurement typically is recorded after three or more well volumes have been purged and stability criteria have been met.

If the purpose of sampling is to obtain field measurements only, these data can be obtained in situ by deploying the multiparameter sensor or sonde downhole, followed by a submersible pump to draw water upward. If water-quality samples will be collected, pumping the water from the well to and through a flowthrough cell that contains the sonde or sensors is a preferable and efficient method for collecting field-measurement data without having to remove and redeploy sampling equipment. Flowthrough cells are supplied by the manufacturers of the multiparameter instruments.

- ➤ Connect all sampling-pump discharge-tubing fittings securely so that atmospheric oxygen does not enter the flowthrough cell of the multiparameter instrument, as this can affect the accuracy and quality of the measurements.
- ▶ Shield the flowthrough cell from direct sunlight to minimize changes in the temperature of the ground-water sample as it is withdrawn; changes in temperature also can affect the accuracy of the pH, ORP, and DO measurements, with respect to their ambient ground-water values, and incident light can affect turbidity readings.
- ▶ Wait a minimum of 60 seconds for the sensors to equilibrate to ambient ground-water conditions before monitoring field-measurement values. Some instruments require a longer equilibration time; check the manufacturer's recommendations.
 - Allow the value(s) on the instrument display to stabilize before recording a final field-measurement value (table 6.8–5).

- Field-measurement values generally are considered stable if, while purging the last of three well volumes of water, the variability among three or more consecutive readings spaced at least 3 to 5 minutes apart conforms to the designated criteria. See NFM 6.0, section 6.0.1 for a discussion on sensor-stabilization criteria and problems. See NFM 4.2.3 for detailed information about well purging.
- Good field judgment and experience are required to make a final determination when readings keep drifting or if what the values represent is in question. Such problems should be documented and advice (if needed) should be sought from a senior technician.

Field-measurement sensors must first be allowed to equilibrate to the ambient temperature of the water body being sampled or monitored. This can take from 60 seconds to more than 30 minutes, depending on the instrument and the start and final temperature range. Ensure that all field-measurement readings have stabilized before recording the final field measurement values.

6.8.3.C MEASUREMENT TIPS

Measurement accuracy depends on the adequacy of the calibration procedures used, and many of the precautions described in section 6.8.2 on calibration also apply when measuring the field properties of environmental waters. The following tips can enhance the quality of the field measurement and address some common onsite practices or issues.

- ► Equipment use: Each instrument must be tested and the sensors calibrated before use.
 - Apply the same precautions for measurement as were recommended for calibration.
 - Avoid faulty readings by cleaning calibration residues and dirt from sensors before use.
 - Instruments may be sensitive to static electricity. Keep the instrument at least 3 ft (about 1 m) away from objects that are not electrically grounded.
- Sensor-sample equilibration: Allow a minimum of 60 seconds for an instrument to warm up and the sensors to reach thermal equilibrium with the water temperature before recording field measurements. Some instruments require a longer equilibration time (up to 30 minutes); check the manufacturer's recommendations.
- ▶ Measurement accuracy: If the water matrix or other condition triggers a concern regarding the accuracy or replication of the measurement, check the sensor calibration and document any changes in the sensor response after sampling or completing a set of field measurements. This record will help to determine deterioration or malfunction of one or more of the sensors. A calibration check of the DO sensor is recommended as a routine practice, especially if the measurement was made in a suboxic environmental water.
- ▶ pH and ORP (see NFM 6.4, section 6.4.3, and NFM 6.5, section 6.5.3, respectively):
 - Check the slope of the pH electrode before use to verify that the electrode is working properly (the slope is determined as part of the calibration process; see section 6.8.2.B and NFM 6.4 for pH calibration tips).

- Record changes in ambient air or water temperature while onsite, as temperature affects pH and ORP readings.
- Depending on the sensor type and manufacture, pH or ORP sensors may or may not be designed for horizontal or near horizontal placement during measurement; check manufacturer's instructions (Hach pH sensors, for example, do allow for horizontal placement).
- ORP field values that are determined with a silver/silver chloride reference electrode must be converted to standard hydrogen electrode (SHE) values. See NFM 6.5 for calculation instructions.

► **Turbidity** (see NFM 6.7, section 6.7.3):

- Cover the flowthrough cell with aluminum foil to avoid potential bias to the readings from ambient light.
- Inspect the sensor body to ensure that no bubbles are on the optical surface before beginning measurement.
- If using a flowthrough cell, ensure that no bubbles are entrained in the sample water. The presence of bubbles will result in a high bias to readings.
- For sensors with wipers, follow the manufacturer's instructions for how to verify that the wiper arm is operating correctly.
- Instrument precision often decreases at turbidities less than 2 turbidity units—consult the manufacturer's specification for the expected accuracy of the measurement. Some instruments have the capability of processing low-turbidity data to improve reproducibility. Check whether the instrument has a user-adjustable turbidity data-filter option. If working in low-turbidity water, review the guidance in NFM 6.7 for selection of the appropriate multiparameter (or single-parameter) instrument type.

▶ Dissolved oxygen (see NFM 6.2, section 6.2.1):

— Table 6.8–6 provides general guidelines for use of the amperometric (polarographic or Clark cell) and luminescent (optical) sensors. Use of the luminescent-sensor method may be more practical for dissolved-oxygen measurement in the field, depending on site conditions.

- For surface-water measurements, selection of the DO amperometric or luminescent sensor should be based on flow regime and stratification of the water body.
- For an amperometric (polarographic sensor or Clark cell) measurement, some manufacturers recommend transporting the sonde with the sensor guard (instead of the storage/calibration cup) installed, keeping the sonde wrapped in the wet light-colored towel used for calibration. To reduce evaporation in hot weather, place the entire sonde and wet towel into a perforated plastic bag (that is kept unsealed). The wrapped sonde can be transported in a bucket or cooler.
 - Allow the amperometric instrument to warm up after turning on the display. The DO output should read saturation for the barometric pressure determined for the site.
 - Allow the polarographic sensor to equilibrate to the temperature of the stream, lake, or ground water.
 - For low-velocity water, follow the manufacturer's instructions when using an amperometric instrument.
 - Use the stirrer for the DO sensor that is provided or recommended by the manufacturer. Alternatively, use the luminescent-sensor method, which is not flow dependent.
 - If the instrument has no stirrer, move the sonde up and down (or side to side in shallow water) at the rate recommended by the manufacturer. (A stirrer is preferable to manually induced flow, especially under stratified conditions at the thermocline of a surfacewater body).
 - Flow dependence is diminished when using a "rapidpulse sensor;" however, some flow over the membrane is needed. Check the manufacturer's instructions.
- To verify the accuracy of the amperometric measurement, rinse the sensors and check the DO calibration by rewrapping it in the wet white towel. The instrument display should return to its saturation set point (±2 percent) within a few minutes. Record any post-measurement calibration check in the field notes.

Table 6.8–6. General guidelines for use of amperometric and luminescent dissolved-oxygen sensors on multiparameter instruments

A mnonometrie conce	
Amperometric sensor (polarographic or Clark cell) ¹	Luminescent sensor (optical) ¹
Inspect the sonde and sensor for damage, improper installation, or excessive buildup of biofouling matter. Follow the manufacturer's recommendations for cleaning and calibration.	Inspect the sonde and sensor for damage, improper installation, or excessive buildup of biofouling matter. Follow the manufacturer's recommendations for cleaning and calibration. The maintenance and use of optical dissolved-oxygen sensors are highly dependent on the
Inspect the membrane for damage or improper installation (the average replacement interval is 2 to 4	technology used by the specific manufacturer. Follow the instructions specified by the manufacturer.
weeks). Inspect the membrane for biofouling. Replace the membrane if biofouling is evident. Avoid contact of the membrane and	Example A – YSI "ROX" optical sensor. This sensor should not be left exposed to air for 2 hours or more or otherwise allowed to dry out. Store the sensor wet to avoid drift or having to rehydrate the sensor.
sensor with acids, bases, and organic solvents. Replace the potassium chloride (KCl) solution once a month or sooner if performance degrades, and when replacing the sensor. Inspect O-rings periodically and replace as needed or per the manufacturer's recommendation.	Example B – Hydrolab "LDO" optical sensor: This sensor should not be left exposed to air and allowed to dry out. The sensor needs to be stored in liquid with its cap on. If the sensor is in a dry environment for several hours it may need to be soaked for up to 5 days before use. The sensor drifts slightly during hydration and must be fully hydrated before being calibrated. Example C – In-Situ "RDO" optical sensor. This sensor can be exposed to ambient air for extended periods, can be stored dry, and does not require a hydration period before deployment.
For short-term storage, keep the DO sensor immersed in a calibration cup with enough water to keep electrolyte from evaporating.	Check the manufacturer's instructions for short-term and long-term sensor storage, as requirements can differ substantially among manufacturers.
Anode and cathode maintenance: The silver anode can be contaminated and might be the cause of poor sensor performance: clean according to the manufacturer's recommendation. The gold cathode must be bright. Follow the manufacturer's recommendations for cleaning.	Sensors with wipers require manufacturer-specific maintenance procedures: • Use only the wiper recommended by the manufacturer for the sensor in use. • Inspect the wiper pad periodically for wear and tear, and biofouling. • Change the wiper before each long-term deployment, or as recommended by the manufacturer.

¹Refer to Section 6.2.1 for detailed information on amperometric and luminescent-sensor methods for measuring dissolved-oxygen concentrations.

6.8.4 TROUBLESHOOTING

Multiparameter instruments that perform poorly can be tested and the cause can be identified. The complexity of the series of tests increases with the number of sensors in the sonde. The troubleshooting tests should be performed in a prescribed order that depends on the type of sensors in use and potential for sensor contamination. General troubleshooting tips are provided below in table 6.8–7. More detailed guidance is available from the manufacturer. **Consult the manufacturer's user manual for the specific instrument being used.**

- ► If the display shows a warning message, do not use the sensor until the error has been identified and corrected.
- ➤ Sensor ports on the instrument body should be dry before replacing sensors. Use compressed air, methanol, or isopropyl alcohol to dry the ports. When using methanol or isopropyl alcohol, gently shake off the excess liquid from the port and allow sufficient time for the liquid to evaporate.

WARNING: Alcohol or other solvents can damage certain types of plastics and can destroy the sensing surface of the optical DO sensor.

CAUTION: Avoid skin contact with, and fume inhalation of, potentially hazardous equipment-cleaning solutions such as methanol and isopropyl alcohol. If such substances will be used, wear a face mask and protective clothing. If possible, replace sensors under a fume hood.

Table 6.8–7. Troubleshooting tips for use of multiparameter instruments

[DO, dissolved oxygen; NIST, National Institute of Standards and Technology; SC, specific electrical conductance; ORP, oxidation-reduction (redox) potential; Cl, chloride; NH₄, ammonium; NO₃, nitrate; NTU, nephelometric turbidity unit]

Symptom	Possible cause(s), corrective actions, and tips				
Erratic or jumpy readings	• May be caused by loose connections or sensitivity to the electrical capacitance of your body and to static electricity: avoid touching the sonde housing and try to keep a distance of about 1 meter from the sonde.				
Display does not turn on	 Check that the batteries are installed properly and are fully charged. Battery performance decreases with deceasing temperature. Batteries that charge at room temperature may not perform well when the temperature approaches freezing. Carry spare batteries. 				
The display does not show readings; the readings seem to be wrong	 Check that the readings are displayed in the appropriate units. Inspect all connectors for moisture, dirt, damage, or a loose connection. Clean as recommended by the manufacturer. Disconnect and reconnect and recalibrate the sensors. When replacing sensors, the waterproof and dustproof properties of the instrument must be maintained or instrument performance will degrade. 				
Data on the display appear scrambled	 Check for computer speed and software and hardware compatibility. Check for a damaged cable. Check that the correct units are displayed. If data remain scrambled, consult the manufacturer or authorized service center. 				
Initial drifting of the readings	 Increase the time for sensors to equilibrate to the water temperature. Check that the sensors are appropriately submerged and (if necessary for the instrument) that they are at the appropriate inclination from the horizontal. 				
Dissolved-oxygen reading is unstable or inaccurate	 Check that the sensor has been calibrated to the true onsite barometric pressure or altitude; recalibrate the sensor at the proper barometric pressure and, to the extent possible, with calibrants brought to sample temperature. Amperometric DO method: Inspect the membrane for a puncture, bubbles, or improper installation. Verify the integrity of the membrane, electrolyte solution, and O-ring by checking the reading against a zero-DO solution. Rinse the sonde thoroughly. 				
Temperature reading is unstable or inaccurate	 Check for water in the connector; dry the connector and reinstall the sensor. Check the accuracy of the reading with an NIST-traceable thermometer and have it replaced if necessary. Usually, only the manufacturer can replace a faulty thermistor. 				
Reading is unstable or inaccurate for SC, pH, ORP, turbidity, Cl, NH ₄ , or NO ₃	 Examine the sensor for dirt or damage. Clean dirty sensors according to the manufacturer's instructions. Replace damaged sensors and recalibrate. Ensure that the temperature reading is accurate by allowing sufficient time for the temperature sensor to equilibrate to the water temperature. Check that the calibration solutions used for SC, pH, and ORP were not expired or subject to contamination. Recalibrate the sensor(s), first bringing the calibration solutions as close to the ambient temperature of the sample as is practical, given ambient field conditions. Check pH reference junction: if dry, follow manufacturer's instructions for soaking the sensor in tap water or buffer solution until readings stabilize. Alternatively, replace the junction. Check the sensor connector for water; dry the connector and reinstall the sensor. If the ZoBell check fails, was temperature dependence of the ZoBell solution accounted for? The SC sensor must be fully immersed for proper calibration and sample measurement. There must be no bubbles in the cell. The turbidity sensor wiper must be clean, activated, and rotating properly. Check that expired turbidity calibrants were not used, including any diluted 4000-NTU formazin standard (which must be used within 24 hours of preparation). 				

6.8.5 REPORTING

USGS personnel are instructed to record all field-measurement values on electronic or paper field forms, and to complete the field-measurement fields on Analytical Services Request forms of the USGS National Water Quality Laboratory or other laboratory at which samples will be analyzed. Field-measurement entries should be checked by a second party and compared for accuracy and consistency with those entered into NWIS.

Table 6.8–8. USGS guidelines for reporting field-measurement values

[\pm , plus or minus; o C, degrees Celsius; μ S/cm, microsiemens per centimeter; >, greater than; mg/L, milligrams per liter; mV, millivolt; SHE, standard hydrogen electrode; FNU, formazin nephelometric units; ppt, parts per trillion; psu, practical salinity units calculated from specific electrical conductance at 25 degrees Celsius]

Field measurement ¹	USGS reporting convention for the National Water Information System (NWIS) ²	Unit	
Temperature	±0.1°C, depending on instrument accuracy and precision	°C	
Specific conductance	Three significant figures to the nearest whole number	μS/cm at 25°C	
Dissolved oxygen (DO)	Nearest 0.1 mg/L (for the amperometric or luminescent-sensor method) Nearest 0.01 mg/L (for the spectrophotometric/Rhodazine-D TM method) Report ">20 mg/L" for a DO measurement that exceeds 20 mg/L	mg/L	
pН	Nearest 0.1 unit for most applications. Can be reported at 0.05 pH unit, depending on accuracy and precision of the calibrated sensor		
Oxidation- reduction potential	reduction report raw data) and the temperature of the sample		
Turbidity Range: 0 to 10 to the nearest 0.1 FNU 10 to 100 to the nearest 1 FNU >100 to the nearest 10 FNU		FNU ³	
Salinity <1 to 10, to the nearest 0.1 ppt or psu 10 to 100, to the nearest 1 ppt or psu		ppt or psu	

¹Information is based on manufacturers' specifications for the following multiparameter systems: Hydrolab Quanta and DataSonde 5 and 5X, DS5; YSI 6600; In-Situ Troll 9500; and Eureka Manta.

² It is USGS practice to enter values into NWIS that have more significant figures than are the standard for data publication. The NWIS databases produce the values that are rounded correctly, which are then reported in publications. This practice eliminates investigator mistakes when reporting rounded values. NWIS data must be input with the correct parameter and method codes, which can be found by accessing QWDATA.

³ Most multiparameter instruments used for USGS turbidity measurement contain single-beam infrared wavelength turbidity sensors and are reported in FNU. Check the Excel spreadsheet at http://water.usgs.gov/owq/turbidity_codes.xls to determine the appropriate turbidity unit of measure and NFM 6.7 for detailed information on turbidity measurement and instrumentation.

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APPENDIX 6.8-A

Example of a USGS field form for recording sensor calibrations and field measurements

NOTE: USGS personnel are advised to use the latest available version of this and other field forms.

November 2006 METER CALIBRATIONS/FIELD MEASUREMENTS STN NO								
Calibrate Date:	ed by:							
TEMPER	ATURE Met	er MAKE/MODEL _		S/N		Thermis	ster S/N Thermometer ID	
Calibration criteria: ±1 percent or ±0.5 °C for liquid-filled thermometers ±0.2 °C for thermisters								
		hermometer/The					± °C	
						ROM LEFT RIGHT		
Field Re	adings #1	#2	#3	#4	#5	MEDIAN	: °C Method code Remark Qualifier	
pH Mete	r MAKE/MODEL _		S	6/N		Electrode No	Type: GEL LIQUID OTHER	
Sample:	FILTERED UN	FLTERED (HURN SPLITTE	R SINGLE	POINT AT	FT DEEP	VERTICAL AVG. OF POINTS CONE SPLITTER	
pH BUFFER	BUFFER TEMP	THEO- RETICAL pH FROM TABLE	pH BEFORE ADJ.	pH AFTER ADJ.	SLOPE	MILLI- VOLTS	TEMPERATURE CORRECTION FACTORS FOR BUFFERS APPLIED? Y N BUFFER LOT NUMBERS:	
pH 7							рн 7:	
pH 7							рн:	
pH 7							CHECK PH:	
pH							BUFFER EXPIRATION DATES:	
pH							рн 7:	
pH							рн:	
CHECK							СНЕСК РН:	
pH							Calibration Criteria: ± 0.1 pH units	
Field Re	eadings #1	#2	#3#	#4#5	MEDIAI	N:Unit	ts MethodcodeRemark Qualifier	
CDECLE	C CONDUCT	ANCE Material				CAL	Course Times and State of the	
							Sensor Type: DP FLOW-THRU OTHER	
Sample: C	HURN SPLITTER	SINGLE POINT AI	π DE	EP VERTICAL /	AVG. OF	POINTS CONE	AUTO TEMP COMPENSATED METER	
Std Valu μS/cm		SC Before	SC After		itd t No.	Std typ	INFANOAL I LIVIF CONFENSATED WETER	
,		Adj.	Adj.			(KCI; Na	CORRECTION FACTOR APPLIED? Y N	
							CORRECTION FACTOR=	
							Calibration Criteria: ± 5 % for SC ≤100 μS/	
							cm or 3% for SC >100 µS/cm	
Field rea	dings #1	#2#	3#4	#5	MEDIAN:	mS/cm	Method code Remark Qualifier	
DISSOLV	(ED OVVCEN	Meter MAKE/M	ODE!			S/N		
		ic Luminescent				3N		
	٠.	Saturated Water			Water Air 0	Calibration Chamb	per in Air Winkler Titration Other	
Sample: SINGLE POINT AT								
WATER	BAROMETRI		_		DO		mg/IL Adj. tomg/L Date:	
TEMP °C	PRESSURE mm Hg			BEFORE ADJ.	AFTER ADJ.		on Date Thermister Check? Y N Date	
						Membrane Cha	nged? N Y Date: Time:	
Barometer Calibrated? N Y Date:Tima								
Calibration Criteria: ± 0.2 mg/L Battery Check: REDLINE RANGE								
Field readings #1 #2 #3 #4 #5 MEDIAN: mg/L Method code Remark Qualifier								
Calibration form ver. 4.0								

Appendix 6.8–A. Example of a USGS field form for recording sensor calibrations and field measurements. (USGS personnel should use the latest available version of this and other field forms.)