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#### U.S. ENVIRONMENTAL PROTECTION AGENCY REGION 1

DRAFT CALIBRATION OF FIELD INSTRUMENTS (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential [ORP], and turbidity)

#### I. SCOPE & APPLICATION

The purpose of this standard operating procedure (SOP) is to provide a framework for calibrating field instruments used to measure water quality parameters for ground water and surface water. Water quality parameters include temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential [ORP], and turbidity. This SOP supplements, but does not replace, EPA analytical methods listed in 40 CFR 136 and 40 CFR 141 for temperature, dissolved oxygen, conductivity/specific conductance, pH and turbidity.

This SOP is written for instruments that utilize multiple probes (temperature, pH, dissolved oxygen, conductivity/specific conductance, and/or oxidation/reduction potential [ORP]) and the probe readings for pH, dissolved oxygen, and specific conductance are automatically corrected for temperature. Communications to the instrument (programming and displaying the measurement values) are performed using a display/logger or a computer. Information sent to the instrument is entered through the keypad on the display/logger or computer. It is desirable that the display/logger or computer have data storage capabilities. If the instrument does not have a keypad, follow the manufacturer's instructions for entering information into the instrument.

For ground water monitoring, the instrument must be equipped with a flow-through-cell, and the display/logger or computer display screen needs to be large enough to simultaneously contain the readouts of each probe in the instrument. Turbidity is measured using a separate instrument because turbidity cannot be measured in a flow-through-cell. This procedure is applicable for use with the EPA Region 1 Low Stress (low flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells.

#### II. GENERAL

All monitoring instruments must be calibrated before they are used to measure environmental samples. Part of the calibration is performed prior to the field event. For instrument probes that rely on the temperature sensor (pH, dissolved oxygen, conductivity/specific conductance, and

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oxidation/reduction potential [ORP]), each temperature sensor needs to be checked for accuracy against a thermometer that is traceable to the National Institute of Standards and Technology (NIST). Before any instrument is calibrated or used to perform environmental measurements, the instrument must stabilize (warm-up) according to manufacturer's instructions.

Most instruments will require at least two standards to bracket the expected measurement range, that is, one standard less than the expected value and one higher. Calibration must be performed at the beginning of each sampling day prior to sample collection. To determine if the instruments have remained in calibration during transport to each sampling location, use one of the previously used standards as a check standard at the sampling site. If the check measurement does not agree with the initial calibration or to within the specifications of the instrument, then the instrument must be re-calibrated. When an environmental sample measurement falls outside the calibration range, the instrument must be re-calibrated to bracket the new range before continuing measurements.

This SOP requires that the manufacturer's instruction manual (including the instrument specifications) accompany the instrument into the field.

# **III. CALIBRATION PROCEDURES**

Prior to calibration, all instrument probes must be cleaned according to the manufacturer's instructions. Failure to perform this step (proper maintenance) can lead to erratic measurements.

Program the multi-probe instrument so that the following parameters to be measured will be displayed: temperature, pH, percent dissolved oxygen, mg/l dissolved oxygen, conductivity, specific conductance, and ORP.

The volume of the calibration solutions must be sufficient to cover both the probe and temperature sensor (see manufacturer's instructions for additional information).

While calibrating or measuring, make sure there are no air bubbles lodged between the probe and the probe guard.

## TEMPERATURE

Most instrument manuals state there is no calibration of the temperature sensor, but the temperature sensor must be checked to determine its accuracy. This accuracy check is performed at least once per year and the accuracy check date/information is kept with the

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instrument. If the accuracy check date/information is not included with the instrument or the last check was over a year, the temperature sensor accuracy needs to be checked at the beginning of the sampling event. If the instrument contains multiple temperature sensors, each sensor must be checked.

#### Verification Procedure

1. Allow a container filled with water to come to room temperature.

2. Place a thermometer that is traceable to the National Institute of Standards and Technology (NIST), and the instrument's temperature sensor into the water and wait for both temperature readings to stabilize.

3. Compare the two measurements. The instrument's temperature sensor must agree with the reference thermometer measurement within the accuracy of the sensor (usually  $\pm 0.15^{\circ}$ C). If the measurements do not agree, the instrument may not be working properly and the manufacturer needs to be consulted.

## pH (electrometric)

The pH of a sample is determined electrometrically using a glass electrode.

Choose the appropriate buffered standards that will bracket the expected values at the sampling locations. For ground water, the pH will usually be close to seven. Three standards are needed for the calibration: one close to seven, one at least two pH units below seven and the other at least two pH units above seven. For those instruments that will not accept three standards, the instrument will need to be re-calibrated if the water sample's pH is outside the initial calibration range described by the two standards.

#### Calibration Procedure

1. Allow the buffered standards to equilibrate to the ambient temperature.

2. Fill calibration containers with the buffered standards so each standard will cover the pH probe and temperature sensor.

3. Remove probe from its storage container, rinse with distilled water, blot dry with soft tissue.

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4. Select monitoring/run mode. Immerse probe into the initial standard (e.g., pH 7).

5. Stir the standard until the readings stabilize. If the reading does not change within 30 seconds, select calibration mode and then select "pH". Enter the buffered standard value into instrument. Select monitoring/run mode. The readings should remain within manufacturer's specifications; if they change, re-calibrate. If readings continue to change after re-calibration, consult manufacturer.

6. Remove probe from the initial standard, rinse with distilled water, and blot dry.

7. Immerse probe into the second standard (e.g., pH 4). Repeat step 5.

8. Remove probe from the second standard, rinse with distilled water, and blot dry. If instrument only accepts two standards, the calibration is complete. Go to step 11. Otherwise continue.

9. Immerse probe in third buffered standard (e.g., pH 9) and repeat step 5.

10. Remove probe from the third standard, rinse with distilled water, and blot dry.

11. Select monitoring/run mode, if not already selected. To ensure that the initial calibration standard (e.g., pH 7) has not changed, immerse the probe into the initial standard. Wait for the readings to stabilize. The reading should read the initial standard value within the manufacturer's specifications. If not, re-calibrate the instrument. If re-calibration does not help, the calibration range may be too great. Reduce calibration range by using standards that are closer together.

12. The calibration is complete. Place pH probe in its storage container.

# DISSOLVED OXYGEN

Dissolved oxygen (DO) content in water is measured using a membrane electrode. The DO probe's membrane and electrolyte solution should be replaced prior to the sampling period. Failure to perform this step may lead to erratic measurements.

#### Calibration Procedure

1. Gently dry the temperature sensor according to manufacturer's instructions.

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2. Place a wet sponge or a wet paper towel on the bottom of the DO calibration container.

3. Place the DO probe into the container without the probe coming in contact with the wet sponge or paper towel. The probe must fit tightly into the container to prevent the escape of moisture evaporating from the sponge or towel.

4. Allow the confined air to become saturated with water vapor (saturation occurs in approximately 10 to 15 minutes). During this time, turn-on the instrument to allow the DO probe to warm-up. Select monitoring/run mode. Check temperature readings. Readings must stabilize before continuing to the next step.

5. Select calibration mode; then select "DO %".

6. Enter the local barometric pressure (usually in mm of mercury) for the sampling location into the instrument. This measurement must be determined from an on-site barometer. Do not use barometric pressure obtained from the local weather services unless the pressure is corrected for the elevation of the sampling location. [Note: inches of mercury times 25.4 mm/inch equals mm of mercury or consult Oxygen Solubility at Indicated Pressure chart attached to the SOP for conversion at selected pressures].

7. The instrument should indicate that the calibration is in progress. The instrument will take approximately one minute to calibrate. After calibration, the instrument should display percent saturated DO.

8. Select monitoring/run mode. Compare the DO mg/l reading to the Oxygen Solubility at Indicated Pressure chart attached to the SOP. The numbers should agree. If they do not agree to the accuracy of the instrument (usually  $\pm 0.2$  mg/L), repeat calibration. If this does not work, change the membrane and electrolyte solution.

9. Remove the probe from the container and place it into a 0.0 mg/L DO standard (see note). The standard must be filled to the top of its container and the DO probe must fit tightly into the standard's container (no head space). Check temperature readings. They must stabilize before continuing.

10. Wait until the "mg/l DO" readings have stabilized. The instrument should read 0.0 mg/L or to the accuracy of the instrument (usually  $\pm$  0.2 mg/L). If the instrument cannot reach these values, it will be necessary to clean the probe, and change the membrane and electrolyte solution. If this does not work, prepare a new 0.0 mg/L DO standard. If these measures do not work, contact manufacturer.

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Note: To prepare a zero mg/L DO standard follow the procedure stated in Standard Methods (Method 4500-O G). The method basically states to add excess sodium sulfite (until no more dissolves) and a trace amount of cobalt chloride to water. The standard container must be completely filled (no head space). This solution is prepared prior to the sampling event. If some of the solution is lost during instrument calibration, add more water to the container so that the standard is stored with no head space.

## SPECIFIC CONDUCTANCE

Conductivity is used to measure the ability of an aqueous solution to carry an electrical current. Specific conductance is the conductivity value corrected to 25°C.

Most instruments are calibrated against a single standard which is near, but below the specific conductance of the environmental samples. A second standard which is above the environmental sample specific conductance is used to check the linearity of the instrument in the range of measurements.

## Calibration Procedure

1. Allow the calibration standard to equilibrate to the ambient temperature.

2. Remove probe from its storage container, rinse the probe with a small amount of the conductivity/specific conductance standard (discard the rinsate), and place the probe into the conductivity/specific conductance standard.

3. Select monitoring/run mode. Wait until the probe temperature has stabilized.

4. Look up the conductivity value at this temperature from the conductivity versus temperature correction table usually found on the standard bottle or on the standard instruction sheet. You may need to interpolate the conductivity value between temperatures. Select calibration mode, then conductivity. Enter the temperature corrected conductivity value into the instrument.

5. Select monitoring/run mode. The reading should remain within manufacturer's specifications. If it does not, re-calibrate. If readings continue to change after re-calibration, consult manufacturer.

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6. Read the specific conductance on the instrument and compare the value to the specific conductance value on the standard. The instrument value should agree with the standard within the manufacturer's specifications. If not, re-calibrate. If the re-calibration does not correct the problem, the probe may need to be cleaned or serviced by the instrument manufacturer.

7. Remove probe from the standard, rinse the probe with a small amount of the second conductivity/specific conductance standard (discard the rinsate), and place the probe into the second conductivity/specific conductance standard. The second standard will serve to verify the linearity of the instrument. Read the specific conductance value from the instrument and compare the value to the specific conductance on the standard. The two values should agree within the specifications of the instrument. If they do not agree, recalibrate. If readings do not compare, then the second standard may be outside the linear range of the instrument. Use a standard that is closer, but above the first standard and repeat the verification. If values still do not compare, try cleaning the probe or consult the manufacturer.

8. When monitoring ground water or surface water, use the specific conductance readings.

# **OXIDATION/REDUCTION POTENTIAL (ORP)**

The oxidation/reduction potential is the electrometric difference measured in a solution between an inert indicator electrode and a suitable reference electrode. The electrometric difference is measured in millivolts and is temperature dependent.

## Calibration or Verification Procedure

1. Allow the calibration standard (a Zobell solution) to equilibrate to ambient temperature.

2. Remove the probe from its storage container, and place it into the standard.

3. Select monitoring/run mode.

4. While stirring the standard, wait for the probe temperature to stabilize, then read the temperature.

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5. Look up the millivolt (mv) value at this temperature from the millivolt versus temperature correction table usually found on the standard bottle or on the standard instruction sheet. You may need to interpolate millivolt value between temperatures. Select "calibration mode", then "ORP". Enter the temperature-corrected ORP value into the instrument.

6. Select monitoring/run mode. The readings should remain unchanged within manufacturer's specifications. If they change, re-calibrate. If readings continue to change after re-calibration, consult manufacturer.

7. If the instrument instruction manual states that the instrument is factory calibrated, then verify the factory calibration against the standard. If they do not agree within the specifications of the instrument, the instrument will need to be re-calibrated by the manufacturer.

# TURBIDITY

The turbidity method is based upon a comparison of intensity of light scattered by a sample under defined conditions with the intensity of light scattered by a standard reference suspension. A turbidimeter is a nephelometer with a visible light source for illuminating the sample and one or more photo-electric detectors placed ninety degrees to the path of the light source.

Some instruments will only accept one standard. For these instruments, the standards will serve as check points.

Calibration Procedures

1. Allow the calibration standards to equilibrate at the ambient temperature. The use of commercially available polymer primary standards (AMCO-AEPA-1) is preferred, however, the standards can be prepared using Formazin according to the EPA analytical Method 180.1.

2. If the standard cuvette is not sealed, rinse a cuvette with deionized water. Shake the cuvette to remove as much water as possible. Do not wipe dry the inside of the cuvette because lint from the wipe may remain in the cuvette. Add the standard to the cuvette.

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3. Before performing the calibration procedure, make sure the cuvettes are not scratched and the outside surfaces are dry, free from fingerprints and dust. If the cuvette is scratched or dirty, discard or clean the cuvette respectively.

4. Zero the instrument by using either a zero or 0.02 NTU standard. A zero standard (approximately 0 NTU) can be prepared by passing distilled water through a 0.45 micron pore size membrane filter.

5. Using a standard in the range of 5 - 20 NTUs, calibrate according to manufacturer's instructions or verify calibration if instrument will not accept a second standard. If verifying, the instrument should read standard value to within the specifications of the instrument. If the instrument has range of scales, check each range that will be used during the sampling event with a standard that falls within that range.

7. Using a standard between 20 and 100 NTUs, calibrate according to manufacturer's instructions or verify calibration if instrument does not accept a third standard. If verifying, the instrument should read standard value to within the specifications of the instrument. If the instrument has range of scales, check each range that will be used with the proper standard for that scale.

# IV. DATA MANAGEMENT AND RECORDS MANAGEMENT

All calibration records must be documented in the project's log book. At a minimum, include the instrument manufacturer, model number, instrument identification number, standards used to calibrate the instruments (including source), calibration date, and the instrument readings.

# References

Standard Methods for the Examination of Water and Wastewater, 19th edition, 1995.

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983.

Turbidity - Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993.

#### Oxygen Solubility at Indicated Pressure

Temp.					re (Hg			
. ~	760	755	750	745	740	735	730	mm
°C	29.92	29.72	29.53	29.33	29.13	28.94	28.74	in
0	14.57	14.47	14.38	14.28	14.18	14.09	13.99	mg/l
1	14.17	14.08	13.98	13.89	13.79	13.70	13.61	
2	13.79	13.70	13.61	13.52	13.42	13.33	13.24	
3	13.43	13.34	13.25	13.16	13.07	12-98	12.90	
4	13.08	12.99	12.91	12.82	12.73	12.65	12.56	
5	12.74	12.66	12.57	12.49	12.40	12.32	12.23	
6	12.42	12.34	12.26	12.17	12.09	12.01	11.93	
7	12.11	12.03	11.95	11.87	11.79	11.71	11.63	
8	11.81	11.73	11.65	11.57	11.50	11.42	11.34	
9	11.53	11.45	11.38	11.30	11.22	11.15	11.07	
10	11.28	11.19	11.11	11.04	10.96	10.89	10.81	
11	10.99	10.92	10.84	10.77	10.70	10.62	10.55	
12	10.74	10.67	10.60	10.53	10.45	10.38	10.31	
13	10.50	10.43	10.36	10.29	10.22	10.15	10.08	
14	10.27	10.20	10.13	10.06	10.00	9.93	9.86	
15	10.05	9.98	9.92	9.85	9.78	9.71	9.65	
16	9.83	9.76	9.70	9.63	9.57	9.50	9.43	
17	9.63	9.57	9.50	9.44	9.37	9.31	9.24	
18	9.43	9.37	9.30	9.24	9.18	9.11	9.05	
19 20	9.24	9.18	9.12	9.05	8.99	8.93	8.87	
20	9.06	9.00	8.94	8.88	8.82	8.75	8.69	
21	8.88	8.82	8.76	8.70	8.64	8.58	8.52	
22	8.71	8.65	8.59	8.53	8.47	8.42	8.36	
23	8.55	8.49	8.43	8.38	8.32	8.26	8.20	
24 25	8.39	8.33	8.28	8.22	8.16	8.11	8.05	
25 26	8.24	8.18	8.13	8.07	8.02	7.96	7.90	
26	8.09	8.03	7.98	7.92	7.87	7.81	7.76	
27 28	7.95 7.81	7.90	7.84 7.70	7.79	7.73	7.68 7.54	7.62 7.49	
		7.76 7.63	7.70	7.65 7.52	7.60 7.47	7.34	7.49	
29 30	7.68 7.55	7.50	7.45	7.32	7.47	7.42	7.24	
30 31		7.30	7.43	7.27	7.22	7.16	7.24	
31 32	7.42 7.30	7.25	7.20	7.15	7.10	7.05	7.00	
32 33	7.08	7.13	7.08	7.03	6.98	6.93	6.88	
33 34	7.08	7.02	6.97	6.92	6.87	6.82	0.88 6.78	
35	6.95	6.90	6.85	6.80	6.76	6.71	0.78 6.66	
36	6.84	6.79	6.76	6.70	6.65	6.60	6.55	
30	6.73	6.68	6.64	6.59	6.54	6.49	6.45	
38	6.63	6.58	6.54	6.49	6.44	6.49 6.40	6.35	
39	6.52	6.47	6.43	6.38	6.35	6.29	6.24	
40	6.42	6.37	6.33	6.28	6.24	6.19	6.15	
41	6.32	6.27	6.23	6.18	6.14	6.09	6.05	
42	6.22	6.18	6.13	6.09	6.04	6.00	5.95	
43	6.13	6.09	6.04	6.00	5.95	5.91	5.87	
44	6.03	5.99	5.94	5.90	5.86	5.81	5.77	
45	5.94	5.90	5.85	5.81	5.77	5.72	5.68	
<i>ч</i> ,	J.J+	5.70	5.05	5.01	5.11	5.14	5.00	

(Continued)

Source: Draft EPA Handbook of Methods for Acid Deposition Studies, Field Operations for Surface Water Chemistry, EPA/600/4-89/020, August 1989.

725 720 715 710 705 700 695 690 mm $^{\circ}$ C 28.34 28.35 28.15 27.95 27.76 27.36 27.17 in 3.22 mg/l   1 13.51 13.42 13.33 13.23 13.14 13.04 12.95 12.86   2 13.15 13.06 12.07 12.88 12.79 12.60 12.21 12.13 12.04 11.95 11.87   3 12.81 12.72 12.23 12.21 12.13 12.04 11.95 11.87   5 12.15 12.06 11.98 11.81 11.43 11.35 11.27   11.55 11.47 11.39 11.31 11.22 11.14 11.06 10.98   8 11.26 11.18 11.01 11.02 10.95 10.87 10.79 10.71   9 10.99 10.92 10.84 10.76 10.69 10.61 10.53 10.44 </th <th>Temp.</th> <th colspan="9">Pressure (Hg)</th>	Temp.	Pressure (Hg)								
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	-	725	720	715	710	705	700	695	690	mm
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	°C	28.54	28.35	28.15	27.95	27.76	27.56	27.36	27.17	in
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	0	13.89	13.80	13.70	13.61	13.51	13.41	13.32	13.22	mg/l
312.8112.7212.6312.5412.4512.3612.2712.18412.4712.3912.3012.2112.1312.0411.9511.87512.1512.0611.9811.8911.8111.7311.6411.56611.8411.7311.6811.0011.5111.4311.3511.27711.5511.4711.3911.3111.2211.1411.0610.98811.2611.1811.1011.0210.9510.8710.7910.71910.9910.9210.8410.7610.6910.6110.5310.461010.7410.6610.5910.5110.4410.3610.2910.211110.4810.1710.1010.029.959.889.819.461310.199.449.589.519.459.389.31159.589.519.449.589.319.249.179.189.119.058.988.928.858.798.73188.998.928.868.808.738.678.618.54198.818.748.688.628.568.498.438.37208.638.578.518.458.398.338.278.21218.468.408.348.288.228.168.108.04228.30	1	13.51	13.42	13.33	13.23	13.14	13.04	12.95	12.86	-
312.8112.7212.6312.5412.4512.3612.2712.18412.4712.3912.3012.2112.1312.0411.9511.87512.1512.0611.9811.8911.8111.7311.6411.56611.8411.7311.6811.0011.5111.4311.3511.27711.5511.4711.3911.3111.2211.1411.0610.98811.2611.1811.1011.0210.9510.8710.7910.71910.9910.9210.8410.7610.6910.6110.5310.461010.7410.6610.5910.5110.4410.3610.2910.211110.4810.1710.1010.029.959.889.819.461310.199.449.589.519.459.389.31159.589.519.449.589.319.249.179.189.119.058.988.928.858.798.73188.998.928.868.808.738.678.618.54198.818.748.688.628.568.498.438.37208.638.578.518.458.398.338.278.21218.468.408.348.288.228.168.108.04228.30	2	13.15	13.06	12.07	12.88	12.79	12.69	12.60	12.51	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	3	12.81	12.72	12.63	12.54	12.45	12.36	12.27	12.18	
		12.47	12.39	12.30	12.21	12.13	12.04	11.95	11.87	
		12.15	12.06	11.98	11.89	11.81	11.73	11.64		
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$\begin{array}{cccccccccccccccccccccccccccccccccccc$	34	6.73	6.68	6.63	6.58	6.53	6.48	6.43	6.38	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	35	6.61	6.56	6.51	6.47	6.42	6.37	6.36	6.27	
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396.266.156.116.066.015.975.925.87406.106.066.015.965.925.865.835.78416.005.965.915.875.825.785.735.69425.915.865.825.775.735.695.645.60435.825.785.735.695.655.605.565.51445.725.685.645.595.555.515.465.42	37	6.40	6.35	6.31	6.26	6.21	6.16	6.12	6.07	
406.106.066.015.965.925.865.835.78416.005.965.915.875.825.785.735.69425.915.865.825.775.735.695.645.60435.825.785.735.695.655.605.565.51445.725.685.645.595.555.515.465.42	38	6.30	6.26	6.21	6.16	6.12	6.07	6.02	5.98	
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425.915.865.825.775.735.695.645.60435.825.785.735.695.655.605.565.51445.725.685.645.595.555.515.465.42	40	6.10	6.06	6.01	5.96	5.92	5.86	5.83	5.78	
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	44							5.46		
	45	5.64	5.59	5.55	5.51	5.47	5.42	5.38	5.34	

Source: Draft EPA Handbook of Methods for Acid Deposition Studies, Field Operations for Surface Water Chemistry, EPA/600/4-89/020, August 1989.