

Guidance for NPDES and WPCF Permit Monitoring

Version 2.0

March 5, 2024



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Executive Summary

This guidance document is intended to aid in self-monitoring performed by NPDES (National Pollutant Discharge Elimination System) and WPCF (Water Pollution Control Facilities) permit applicants and holders. NPDES/WPCF permit holders must collect samples according to requirements in 40 CFR part 122.21 and 40 CFR part 403 Appendix E. For biosolids, samples must be collected according to 40 CFR part 503. This document does not supersede DEQ wastewater permit conditions. The information is provided for informational purposes only.

This guidance is not intended to be used as guidance for compliance under national safe drinking water regulations.

DEQ used the analytical methods and federal regulations as reference while preparing this document. In case of a discrepancy, the actual content of regulations and analytical referenced methods will supersede this document.



State of Oregon
Department of
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Quality



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1 pH – Grab Sampling

This section explains methodology for sampling pH in municipal wastewater, partially or fully treated wastewater, stormwater, and surface water with a handheld or benchtop pH meter. It describes the maintenance, calibration, and use of a single parameter (pH) meter or a multi-parameter water quality meter (multiprobe) for obtaining real-time in-situ measurements of pH. Continuous measurement of pH is described in the next section.

Procedures in this section describe the use of a single monitoring device to take real-time measurements of pH. The intent of this document is to provide operators with adequate information in order to follow procedures outlined in 40 CFR and issued NPDES or WPCF permits.

What is pH?

pH is the measure of how acidic or basic/alkaline water is. It is measured on a logarithmic scale from 0 to 14, where 7 is “neutral”. If the pH is measured between 0 and 6.9, the substance being measured is an acid. If the pH is measured between 7.1 and 14, the substance being measured is a base. The unit for pH is Standard Units or SU. Examples of acids that might be found in a wastewater laboratory include hydrochloric acid, sulfuric acid, or nitric acid. Examples of bases/alkaline substances that might be found in a wastewater laboratory include ammonia, sodium hydroxide, or sodium bicarbonate (baking soda). An acid can also be described as a substance that produced H⁺ ions (hydrogen ions) in aqueous solution, and a base as a substance that produces OH⁻ ions (hydroxide ions) in aqueous solution.

Approved Methods

To measure pH for a NPDES permit, one of the following methods must be used, according to CFR Title 40, Chapter I, Subchapter D, Part 136, found here: <https://www.ecfr.gov/current/title-40/chapter-I/subchapter-D/part-136/section-136.3>

PDF Version of CFR: <https://www.govinfo.gov/content/pkg/CFR-2021-title40-vol25/pdf/CFR-2021-title40-vol25-sec136-3.pdf>

- 1) Standard Methods 4500-H⁺ B-2011 (or newer):
<https://www.standardmethods.org/doi/full/10.2105/SMWW.2882.082>
- 2) ASTM D1293-99 (A or B): <https://www.astm.org/d1293-99.html>
- 3) USGS
 - a. USGS Method I-1586-85 “pH, Electrometric, Glass-Electrode” as in Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resource Investigations of the U.S. Geological Survey, Book 5, Chapter A1., unless otherwise stated. 1989. USGS. <https://pubs.usgs.gov/twri/05a01/report.pdf>
 - b. 2021 USGS pH Method, supersedes previous methods:
<https://pubs.usgs.gov/tm/09/a6.4/tm9a6.4.pdf>

- c. USGS webpage for up to date method publication:
<https://pubs.er.usgs.gov/publication/tm9A6.4>
- 4) 973.41: AOAC Official Methods of Analysis of the Association of Official Analytical Chemists, Methods Manual, Sixteenth Edition, 4th Revision, 1998. AOAC International.
<https://www.aoac.org/scientific-solutions/standards-and-official-methods/>

Prior to taking samples, the facility should select and acquire one of the above methods and keep it on file for frequent reference. The references to Standard Methods in 40 CFR 136.3 reference the year the method was adopted (and therefore the oldest method version that can be used), rather than referencing the multiple editions that contain a given method. The change was made so EPA would not have to continue adding each Standard Method edition in the CFR (Oregon DEQ Laboratory and Environmental Assessment Division, 2019). Note that 40 CFR 136 is periodically updated through a Method Update Rule. The permittee should check CFR 136 prior to selecting one of the above methods to confirm that they are still CFR approved.

Alternative Methods

Where a facility wants to utilize a new method or technology that is not listed in 40 CFR part 136.3, they may apply for an Alternate Testing Procedure (ATP) approval. *40 CFR Part 136.5 Approval of alternate test procedures for limited use* gives direction on this procedure. The Approval must be submitted to the permit inspector once obtained. For more information see DEQ09-LAB-0071-QAG, QA Guidance for Self-Monitoring Laboratories, most recent version. Also see *40 CFR § 136.6 Method modifications and analytical requirements* for additional information about method modifications.

OAR 340-041-0061(10): *“(10) Testing methods. The analytical testing methods for determining compliance with the water quality standards in this rule must comply with 40 CFR Part 136 or, if Part 136 does not prescribe a method, with the most recent edition of Standard Methods for the Examination of Water and Waste Water published jointly by the American Public Health Association, American Water Works Association, and Water Pollution Control Federation; if the department has published an applicable superseding method, testing must comply with the superseding method. Testing in accordance with an alternative method must comply with this rule if the department has published the method or has approved the method in writing.”*

Types of pH Measurements

pH can be measured in three ways:

- 1) Color changing test strips like litmus paper or pH test strips
- 2) Colorimetric or spectrophotometric methods where an indicator pigment is added to the water and then measured by shining a light through it and measuring the absorbance
- 3) Electrometric/potentiometric methods (a.k.a. a digital pH meter) where pH is determined by using the electrical potential of a pH-sensitive electrode

Approved methods in 40 CFR use electrometric methods only. pH test strips and colorimetric methods are not approved methods for measuring pH for an NPDES permit.

pH meters

A digital pH meter has a pH electrode (aka a probe). pH electrodes come in two general types:

1. Glass/glass bulb electrodes
2. Non-glass electrodes (metal/enamel)

pH measurements of surface water and wastewater are most commonly made with glass bulb electrodes. A thorough explanation of how glass bulb electrodes work can be found in the USGS National Field Manual for the Collection of Water-Quality Data, Book 9, Handbooks for Water-Resources Investigations, Chapter 6.4, section 2.2 – pH Electrodes (<https://pubs.usgs.gov/tm/09/a6.4/tm9a6.4.pdf>) (subsequently referred to as the USGS NFM).

Electrodes should be selected based on their intended use. For a NPDES permittee treating domestic wastewater, this might be the measurement of raw or partially treated wastewater, or the measurement of surface or groundwater. Special care should be taken when selecting an electrode in the following situations sometimes applicable to domestic NPDES wastewater treatment facilities: 1) Measurement of water with conductivity less than 100 $\mu\text{S}/\text{cm}$. 2) Measurement of water with a temperature $> 30^\circ\text{C}$ or with a pH outside of the range of 4-11.

The pH value of a solution is dependent on the temperature of that solution. pH values measured for a NPDES permit must be from a pH meter equipped with automatic temperature compensation. Although some pH meters have the option of manually entering the temperature of the solution and subsequently reporting the temperature compensated value, the easier solution is to select a pH meter and electrode with a built in thermistor and Automatic Temperature Compensation. A non-temperature compensated pH value is not an acceptable data point to submit as part of a NPDES permit DMR in the State of Oregon.

In summary, a pH meter should be:

- Accurate and reproducible to 0.1 pH unit
- With a range of 0 to 14
- Equipped with Automatic Temperature Compensation (ATC) or Manual Temperature Compensation
- Equipped with a pH electrode appropriate for the solution being measured

“Temperature sensors for ATC automatically adjust pH measurements for the change in electrode calibration that happens when the temperature changes. Therefore, the ATC corrects for the effects of changing temperature on sensor response. No pH temperature compensation corrects a pH reading back to a value expected at 25°C. That is why the verification is done by comparing the measured buffer reading to the reading expected at the measurement temperature – not to the 25°C value printed on the bottle or pouch. This is why recording the temperature for every pH measurement is important, as it has an impact on the pH reading.”

From ThermoFisher Scientific’s Measuring pH in Surface Water Fact Sheet, accessed 12/4/2023

- Equipped with a thermistor built into the pH electrode or as a second probe on the same pH meter
- Able to be calibrated
- Able to calculate and display the calibration slope in percent

Calibration

pH meters must be calibrated according to the specifications of the manufacturer and the 40 CFR 136 approved methodology being used. The process of calibration compares the pH value the pH meter is reading to the value of a *known standard*. A known standard is a solution with a known value, and therefore can be used to align the initial measurements of the pH meter. Known standards for pH are commonly referred to as buffer solutions, or buffers. Buffers are commonly sold for pHs of 4, 7, and 10 and are often dyed different colors so that they may be easily identified during use.

Buffers selected for a 2 point calibration (4 and 7, or 7 and 10, for example), should bracket the expected value of the pH sample. This makes the measurement more precise. If a facility expects that the pH value of their wastewater will be 7.5, then the 7 and 10 buffers should be selected for use in the calibration process. If the expected value of the sample is 5.7, then the 4 and 7 buffers should be used for calibration. If the pH meters returns a measurement outside of the range of calibration, the pH meters should be recalibrated using the appropriate buffers. For situations where the pH value of the wastewater or other sample is unknown or variable, a 3 point calibration (4, 7, and 10) should be performed. An alternative option when measuring a solution of unknown or variable pH is to use two pH meters. One meter can be calibrated from 4-7, and the second from 7-10. Place both probes into the solution and if the value is below 7, record the value from the meter with the calibration range between 4-7. If the solution's pH value is above 7, record the value from the meter with the calibration range between 7-10.

The method selected outlines details regarding the Recommended Calibration Slope Values. Typically, however, a slope during calibration between 92% and 102% is acceptable.

pH and temperature

The pH value of the pH buffers change as the temperature of the buffer solution changes. When "7 buffer" is referred to, it means that the solution is a pH 7.00 when the solution is at 25°C. However, the solution's pH value changes when the solution changes temperature. The manufacturer should provide or have available a chart of the value of their specific pH buffer at various temperatures. Below is an example of a chart for one brand of pH buffer.

Table 1: Value of pH Buffers at Temperature. EXAMPLE ONLY, use chart provided by buffer manufacturer

| Temperature (deg. C) | pH 7 | pH 10 | pH 4 |
|----------------------|------|-------|------|
| 10 | 7.06 | 10.15 | 4.00 |
| 11 | 7.06 | 10.14 | 4.00 |
| 12 | 7.05 | 10.12 | 4.00 |
| 13 | 7.05 | 10.12 | 4.00 |
| 14 | 7.04 | 10.11 | 4.00 |
| 15 | 7.04 | 10.10 | 4.00 |
| 16 | 7.04 | 10.09 | 4.00 |
| 17 | 7.03 | 10.08 | 4.00 |
| 18 | 7.03 | 10.07 | 4.00 |
| 19 | 7.02 | 10.06 | 4.00 |
| 20 | 7.02 | 10.05 | 4.00 |
| 21 | 7.02 | 10.04 | 4.00 |
| 22 | 7.01 | 10.03 | 4.00 |
| 23 | 7.01 | 10.02 | 4.00 |
| 24 | 7.00 | 10.01 | 4.00 |
| 25 | 7.00 | 10.00 | 4.00 |
| 26 | 7.00 | 9.99 | 4.00 |
| 27 | 7.00 | 9.98 | 4.00 |
| 28 | 6.99 | 9.98 | 4.01 |
| 29 | 6.99 | 9.97 | 4.01 |
| 30 | 6.99 | 9.96 | 4.01 |

Some pH meters require the user to manually enter the value and/or temperature of the buffer being used. This means the user has to measure the temperature of the buffer, then reference the manufacturer’s chart to determine the value of the pH buffer. The user then enters that value into the pH meter to tell it what it should be reading. Some newer pH meters will automatically detect the temperature of the buffer and select the appropriate pH value. The user should still verify that the value of the pH buffer entered during the calibration process is correct. The pH meter’s manual should be used in conjunction with the method selected to determine how to calibrate each brand and model of meter.

pH meter calibration tips

- Store pH buffers in the container provided by the supplier, and keep them closed at all times when not pouring buffer out for use. pH buffer, especially pH 10 buffer, can be altered over time by exposure to air.

- Buffers expire, and expired buffers cannot be used for pH meter calibration. Purchase an appropriate amount of buffer for use in the period before it expires. Discard the buffer when it expires and replace it with fresh buffer.
- Record the lot number and expiration date of the pH buffers used in the lab, as well as the date the container was first opened and the date the container was discarded or emptied. This ensures that if there is a quality problem with the buffer lot used, the samples associated with the bad buffer lot can be identified.
- Each time the pH meter is calibrated, pour unused buffer directly from the original buffer container into a clean container. A dirty container may contaminate the buffer.
- Once sampling is completed, use fresh pH buffer to perform a drift check (see below) on the meter before returning the meter to storage.
- Rinse pH probes with tap or DI water before placing the probe into the storage solution, before placing the probe into a sample, or before placing the probe into buffer solution to calibrate or drift check (be sure to rinse the probe between different buffers).

Calibration Records

Pre-Measurement Calibration

When calibrating prior to measurement, record the following information:

- 1) The date
- 2) The name of the personnel who performed the calibration
- 3) If freshly poured buffer was used (yes/no)
- 4) The time the calibration was performed
- 5) The temperature of each pH buffer
- 6) The pH that the meter was reading for each buffer prior to calibration
- 7) The slope (in %)
- 8) If the calibration was completed successfully

An example of a calibration (pre-measurement) record is shown below:

| Calibration Record | | | | | | | | |
|--------------------|-----------------|--------------------|----------------|-----------------------------------|-----------------------------|------------------------------------|------------------------------|-------|
| Date M/D/YYYY | Initials xxx | Pre-Measurement | | | | | | |
| | | Fresh cal. buffer? | Check-out time | pre-calibration meter low reading | Low Buffer pH & temperature | pre-calibration meter high reading | High Buffer pH & temperature | Slope |
| | | Y/N | 24 Hr | std. units | SU/°C | std. units | SU/°C | % |
| 9/1/2016 | JXD | Y | 0935 | 4.02 | 4.00/18 | 7.05 | 7.03/18 | 98.5 |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |

Figure 1: Pre-Measurement Calibration Record

Measurement

After calibration, follow the procedure outlined in the selected method for measurement.

- pH must be sampled within 15 minutes of the sample being collected.
 - If collecting an in-situ sample (placing the probe in the sample media without collecting it into a secondary container), then the hold time does not apply.
 - If collecting a sample of the sample media into a secondary container, the pH probe must be placed in the secondary container to begin the equilibration process within 15 minutes of the sample being collected.
- The pH probe can be placed directly into the wastewater/water to take the measurement (not advisable if the flow is fast-paced, contains significant debris, or poses a safety hazard) or;
- A decontaminated secondary container (see below – Decontamination) can be used to collect at least 100mL of wastewater/water and the probe can then be placed into the solution for sampling. Rinse the secondary container with the solution to be measured, dump the rinsate out, and then collect a second fresh sample for measurement.
- Record the location, time the sample was collected, staff who collected the sample, meter that was used, and the pH reading to the nearest 0.1 S.U.
- Always record the temperature at the same time in the same media that pH was measured. Even if temperature is not a reporting requirement on your permit, the method requires that you record temperature at the same time (for example see SM 4500-H)

Decontamination

Equipment needed:

- Cellulose scrub pads and/or plastic scrub brushes
- Decontamination solutions:
 - Laboratory grade phosphate-free detergent (1% solution)
 - Tap water
 - Deionized water (DI water)

Secondary buffer containers

The container used to hold buffer solution for calibration/drift-checks can be glass, stainless steel, or plastic. Wash the container daily, prior to calibration, with 1% detergent/tap water solution and a non-scouring scrub brush or pad. Then rinse the container with tap water. Follow this step with a rinse with DI water. The container can then be filled with buffer as outlined in the calibration step above.

Sample containers

The container used to collect a wastewater or water sample or measurement with a pH probe should be washed according to the following procedure: Wash the container prior to sample collection with 1% detergent/tap water solution and a non-scouring scrub brush or pad. Rinse the container with tap water. Collect the sample as described in the approved method and as outlined in the measurement step above.

Post-Measurement Drift Check

After pH measurements have been completed on any given day the meter needs to be checked against the same known standards used to calibrate it to ensure the meter is still working as expected and did not drift over the course of the time which measurements were taken. The user will check the pH probe by measuring the buffers used to calibrate the meter prior to taking a measurement (e.g., if buffers 7 and 10 were used to calibrate, then buffers 7 and 10 should be measured at the end of the sampling day). Note that the user should perform this measurement with a freshly poured aliquot of buffer. The user records the value the meter reads and writes it down. The meter should read within +/- 0.2 SU of the known standard (or another assigned acceptance range as described by the standard method used). Performing this part of the procedures ensures the data quality.

If the buffers used to calibrate the pH meter end up not bracketing the sample value, then check in the meter using both the buffers that were used to calibrate AND buffers that bracket the value of the sample.

When performing a drift check after measurement, record the following information:

- 1) The date
- 2) The name of the personnel who performed the calibration
- 3) The time the check was performed
- 4) The temperature of each pH buffer
- 5) The value of the calibration standard/pH buffer
- 6) The pH that the meter reads in the buffer

- 7) The acceptance range and if the post-measurement reading was within the acceptance range
- 8) Any maintenance comments. If the pH meter does not check in, the value should be flagged as an estimate.

Below is an example of the check-in portion of a Calibration record:

| Post-Measurement | | | | | Comments |
|------------------|----------------------|-----------------------------|-----------------------|------------------------------|----------|
| Check-in time | Low pH meter reading | Low Buffer pH & temperature | High pH meter reading | High Buffer pH & temperature | |
| 24 Hr | std. units | SU/°C | std. units | SU/°C | |
| 1645 | 4.00 | 4.00/21 | 7.02 | 7.02/21 | |
| | | | | | |
| | | | | | |

Figure 2: Post-Measurement Calibration Record

These records should be maintained on site or submitted as described in the facility’s NPDES or WPCF permit.

Quality Checks for pH Meters

The manufacturer of a pH meter can often provide guidance and technical support on how to address quality issues for a specific product. Some commonalities exist in addressing technical issues, so the following list is provided to help troubleshoot when pH meters are not performing as expected.

- Probes
 - How old is the probe and when was it placed into service? If it is over a year old, it might need to be replaced.
 - Is the probe being used appropriate for the type of sample? For example, is the probe being used in a solution with low ionic strength, raw wastewater, or a high acid solution and is the probe designed for that type of solution?
 - pH probes should be stored in either a 100% water saturated air environment or in KCl solution, or as directed by the manufacturer. If the probe was left dry or stored tap or DI water the probe may require the electrolyte solution to be refilled, or replaced entirely.
 - Does the probe being used have refillable electrolyte solution? If it does, the solution may need to be refilled or refreshed.
- Buffers
 - Are the buffer solutions expired or very near their expiration date? If so, replace immediately. pH buffers may not be used if past their manufacturer provided expiration date.
 - Was the pH meter calibrated using buffer solutions that bracketed the final value of the sample?
 - It is a good practice to record the lot codes of the pH buffers used. In the case that a faulty batch of standard is provided by the manufacturer, you can use the lot code, the date the buffer was opened, and the day the buffer was emptied/disposed of to determine which samples may have been affected.
 - Has the buffer solution been kept capped/closed? If left uncapped, the buffer solution should be replaced.
- Meter
 - Most pH meters provide the ability to read the millivolts (mV) read by the meter. The mV reading can be a helpful diagnostic tool.
 - The mV difference read by the pH meter when placed in 7 buffer and 4 buffer should be between 165-180mV. If not, the probe needs to be cleaned, refilled, or changed.
 - The mV reading for 7 buffer should be between -50 and 50 mV.
 - The mV reading for 4 buffer should be 165-180 mV away from the 7 buffer reading in the positive direction.
 - The mV reading for 10 buffer should be 165 – 180 mV away from the 10 buffer reading in the negative direction.

Quality Assurance

It is important that when referencing an approved method for NPDES regulatory permit monitoring, that the associated QA/QC section of the method is referenced and including into sampling and analysis practices. For Standard Methods, Section 4020 Quality Assurance/Quality Control is referenced in methods for pH: <https://www.standardmethods.org/doi/abs/10.2105/SMWW.2882.069>

- "Quality assurance (QA) is a laboratory operations program that specifies the measures requires to produce defensible data with known precision and accuracy. This program is defined in a QA manual, written procedures, work instructions, and records. The manual should include a policy that defines the statistical level of confidence used to express data precision and bias, as well as method detection levels (MDLs) and reporting limits." - SM 4020 A. (2017)

For example, if using SM 4500-H then the following QA/QC is required as shown in SM 4020B:

- Run duplicates of the sample
- Run an additional pH standard with a value that is bracketed by the calibration standards
- Verify slope according to manufacturer's instructions

These practices are captured in the guidance outlined for pH in this document.

Low-Ionic Strength

Waters that have a conductivity less than 100 $\mu\text{S}/\text{cm}$ are considered low-specific-conductance and also low-ionic-strength (USGS, 2021). Waters that have a conductivity between 100 $\mu\text{S}/\text{cm}$ and 200 $\mu\text{S}/\text{cm}$ can exhibit some of the same characteristics as waters less than 100 $\mu\text{S}/\text{cm}$, but to a lesser degree. Measuring these low-ionic strength waters is challenging because (1) the water is not sufficiently conductive to complete the electrical circuit in the pH probe and allow for a stable pH reading using a combination pH electrode, (2) differences in ionic strength between the sample and buffers are large, and (3) the potential of the liquid junction is larger than normal (USGS, 2021). Modern pH probes and their reference junctions are designed to decrease the effects of low specific conductance, but still are not always able to produce the desired accuracy or equilibration time (USGS, 2021). The method approved for NPDES Permitting in 40 CFR 136 that addresses low ionic strength is the 2021 USGS Method for Measurement of pH which can be accessed at the following link (and see 'Approved Methods' section above): <https://pubs.usgs.gov/publication/tm9A6.4>

The method describes collecting a grab sample for pH, then placing a measured amount of potassium chloride (KCl) into the sample to raise the specific conductance of the sample by approximately 150 $\mu\text{S}/\text{cm}$. The addition of KCl in particular is not expected to change the pH of the sample by more than 0.1 S.U. and is not expected to harm the pH probe. The USGS method describes how field staff can prepare the KCl ionic strength adjuster. Several field meter manufacturers offer pH probes specific to sampling low ionic strength, as well as KCl ionic strength adjusters (Hach Company, 2009; Thermo Fisher Scientific, 2014).

Other methods that are not approved in 40 CFR 136 may describe calibration with low ionic strength buffers or checks with a low ionic strength standard, however, the use of those methods alone is not federally approved for the measurement of pH in effluent for NPDES permits. These methods may be used for sampling surface waters only if specified in the permittee's NPDES permit and are part of a permittee's written Quality Assurance Plan.

It is recommended that the permittee determine the range of specific conductance of the surface water when establishing a new surface water monitoring location. If the specific conductance at that location is below 200 $\mu\text{S}/\text{cm}$, then the permittee should evaluate their probe and associated SOP to ensure that issues associated with measuring pH in low ionic strength waters are appropriately addressed.

2 pH – Continuous

This section explains methodology for sampling pH in municipal wastewater, partially or fully treated wastewater, stormwater, and surface water utilizing an in-situ stand-alone pH meter with an integrated data logger (pH loggers). This section only provides guidance for pH loggers designed to be temporarily deployed and operated with a temporary power supply.

Procedures in this section describe the use of a single monitoring device to take continuous measurements of pH. The intent of this document is to provide operators with adequate information in order to follow procedures outlined in 40 CFR and issued NPDES or WPCF permits.

Approved Methods

To measure pH for a NPDES permit, one of the following methods must be used, according to CFR Title 40, Chapter I, Subchapter D, Part 136, found here: <https://www.ecfr.gov/current/title-40/chapter-I/subchapter-D/part-136/section-136.3>

PDF Version of CFR: <https://www.govinfo.gov/content/pkg/CFR-2021-title40-vol25/pdf/CFR-2021-title40-vol25-sec136-3.pdf>

When performing continuous monitoring, the methods approved in the CFR are as listed next to “automated electrode”:

- 1) EPA Method 150.2 (published Dec 1982) https://www.epa.gov/sites/default/files/2015-08/documents/method_150-2_1982.pdf
- 2) USGS
 - a. Method I-2587-85 “pH, Electrometric, Glass-Electrode” as in Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resource Investigations of the U.S. Geological Survey, Book 5, Chapter A1., unless otherwise stated. 1989. USGS. <https://pubs.usgs.gov/twri/05a01/report.pdf>
 - b. 2021 USGS pH Method, supersedes previous methods: <https://pubs.usgs.gov/tm/09/a6.4/tm9a6.4.pdf>
 - c. USGS webpage for up to date method publication: <https://pubs.er.usgs.gov/publication/tm9A6.4>

Prior to taking samples, the facility should select and acquire one of the above methods and keep it on file for frequent reference. The most recent version of the method is the most appropriate method to

use. Note that 40 CFR 136 is periodically updated through a Method Update Rule. The permittee should check CFR 136 prior to selecting one of the above methods to confirm that they are still CFR approved.

Alternative Methods

Where a facility wants to utilize a new method or technology that is not listed in 40 CFR part 136.3, they may apply for an Alternate Testing Procedure (ATP) approval. The Approval must be submitted to the permit inspector once obtained. For more information see DEQ09-LAB-0071-QAG, QA Guidance for Self-Monitoring Laboratories, most recent version.

Logging pH meters

A variety of continuously evolving technologies exist for automatic logging of pH measurements. Some examples of types of loggers:

- Multiparameter sondes equipped with logging and batteries that have the capacity to measure multiple parameters and can hang into a water body or be placed into a flow through cell.
- Single parameter pH loggers in a plastic or metal casing equipped with logging and replaceable or rechargeable batteries.
- Shore-powered logging controller units with permanently installed pH probes – these units are outside of the scope of this document and should be installed and maintained according to the approved method and manufacturer specifications.

It is necessary to supplement the direction in the approved method with direction from the manufacturer of the pH logger chosen for use. This following sections provide an overview of the calibration and measurement process for pH loggers in general, but is not a definitive guide to pH logger operation.

In summary, a pH logger should be:

- Accurate and reproducible to 0.1 pH unit
- With a range of 0 to 14
- Equipped with Automatic Temperature Compensation (ATC)
- Equipped with a pH electrode appropriate for the solution being measured
- Equipped with a built in thermistor
- Able to be calibrated
- Able to calculate and display the slope in percent
- Able to store enough data for the expected deployment period

Equipment

A suggested equipment list for the calibration, programming, deployment, removal, calibration check, and data retrieval for pH data loggers is listed below. Depending on the equipment selected, additional or fewer equipment could be necessary:

- Ph/Temperature logger
- pH 4,7, and 10 standard (buffer)
- Flask or beaker sufficiently large to accommodate the pH logger, one for each pH standard used
- pH electrode storage solution and electrode cap
- Water bath with a Reference Thermometer (PRT) (see section – Temperature)
- Deionized water (DI water)
- Device specific batteries or other appropriate power source
- Communication device (typically laptop, tablet, or smartphone), with pH logger software
- Logger mounting equipment (specific to monitoring location)
- Handheld pH meter for field audit

Calibration

pH meters must be calibrated according to the specifications of the manufacturer and the 40 CFR 136 approved methodology being used. The process of calibration compares the pH value the pH meter is reading to the value of a known standard. A known standard is a solution with a known value, and therefore can be used to align the initial measurements of the pH meter. Known standards for pH are commonly referred to as buffer solutions, or buffers. Buffers are commonly sold for pHs of 4, 7, and 10 and are often dyed different colors so that they may be easily identified during use.

Temperature sensors typically cannot be calibrated, they are instead verified for accuracy against a Reference Thermometer (see Temperature)

It is recommended that for logging pH meters deployed in situations where the pH measurements may fluctuate above and below 7 (typical in wastewater), that a 3 point calibration (4, 7, and 10) should be performed. A description of two and three-point calibrations are described in the section **pH-Grab Sampling**.

Calibration for logging pH meters can be broken down into the following steps:

1. Verify and/or adjust the timeclock on the logger, so that it matches the current time within 3 minutes.
2. Logger is placed into a water bath with a PRT, logging is initiated, and temperature allowed to equilibrate. Once the logging temperature has equilibrated, compare the temperature the logger is recording to the temperature measured by the PRT. The temperature read by the logger must be within +/- 0.5°C of the temperature read by the PRT.

3. Logger is calibrated with each buffer as specified by the manufacturer. Typically, the logger is placed into a container with the buffer and allowed to equilibrate. The value of the pH buffer at the temperature is then entered, and the meter adjusts the pre-calibration measurement to the value of the buffer. Rinse the probe with DI water between each buffer solution.

Programming

The logger will be programmed:

- To start at the desired date and time
- With the desired sampling interval (e.g., 5-minutes, 15-minutes, 1-hour etc.)
- Loggers can typically be programmed to stop logging on a prescribed date and time, programmed to “slate” (cease logging when memory fills), or “wrap-around” (oldest data is overwritten when memory fills). This setting will be determined by the monitoring objectives; however, “slate” is the most commonly used mode.
- If the pH logger is equipped with an immersion indicator, enable logging of that channel. This will assist in the interpretation of the data and will enable any measurements taken while out of water to be quality flagged as such.

Installation

Deployment configurations will vary based on the monitoring site selected, the type of water being monitoring, and the equipment selected.

Tips for installation in common wastewater applications:

- Consider the velocity, depth, and amount of debris present in the wastewater stream where the logger will be installed. The logger could be secured with polypropylene rope, nylon rope, or stainless steel cable depending on the application or configuration. The point where the logger is attached to land should be a secure feature such as a manhole rung, or an eyebolt installed in concrete.
- If water level fluctuates widely, allow enough cable so that the logger stays submerged even during low water level events.
- Rags, oil and grease, and trash pose significant problems when pH loggers are installed in raw wastewater, such as prior to a headworks barscreen. pH loggers will need frequent checks to ensure they have not been ripped of their mount or ensnared in rags. Often a permanently installed pH logger with a flow through cell or other protective mount will be more reliable.

Once the logger is installed, use a handheld pH meter (as described in pH – Grab) to take an audit of the logger. The purpose of the audit is to confirm the accuracy of the logged pH values. This will help determine the necessary calibration frequency for the logger and ensure that the pH values record in between audits are of a known quality. When the logger is offloaded, the audit values recorded can be

compared to the logged values. Both the logged values and the audit values should be submitted to netDMR if continuous pH values are requested as part of a NPDES or WPCF permit.

Interim Site Visits

Based on individual project objectives, interim site visits may be conducted during the course of the monitoring period. Reasons for interim site visits could include collection of additional audit/reference readings for data quality verification, data retrieval as logger capacity is approached, and data evaluation to inform project progress and determine needs for continued data collection. Any interim site visit should include an audit/reference measurement with a calibrated pH meter or multiprobe. Prior to removing the pH logger from the flow for any data retrieval activities, collect an audit/reference measurement as described above. After collecting the audit/reference measurement, the pH logger can be removed from the flow. Take this opportunity to clear any debris from the pH probe and inspect it for damage. If retrieving data use the logger software to establish communication with the logger and download the data file. Record the date/time, staff performing the activity, and data file name. The pH logger can then be returned to the flow.

Removal

Prior to removing the pH logger from the flow, collect an audit/reference measurement with a calibrated pH meter or multiprobe as described. Record the date and time of the measurement, the pH and temperature reading, the audit meter used at each audit. Remove the pH logger from the flow, along with any accompanying installation materials. Record the date and time that the logger was removed. The pH logger should be immediately placed into storage solution as specified by the manufacturer. This might be a Ziploc bag or storage cap containing water from the site, tap water, or storage solution. Never store a pH probe in DI water.

Drift Check

After removal, the logger needs to be checked against the same known standards used to calibrate it, to ensure the meter is still working as expected and did not drift over the course of the time which measurements were taken.

Drift Checks for logging pH meters can be broken down into the following steps:

1. Verify the timeclock on the logger, ensuring that it matches the current time within 3 minutes. If it does not match, the data may need to be adjusted or flagged.
2. Logger is placed into a water bath with a PRT, logging is initiated, and temperature allowed to equilibrate. Once the logging temperature has equilibrated, compare the temperature the logger is recording to the temperature measured by the PRT. The temperature read by the logger must be within +/- 0.5°C of the temperature read by the PRT.

3. Logger is placed each buffer and allowed to equilibrate. Compare the value displayed by the logger to the value of the pH buffer at its current temperature. Rinse the probe with DI water between each buffer solution.

The meter should read within +/- 0.2 SU of the known standard (or another assigned acceptance range as described by the standard method used, manufacturer specifications, and actual experience with the achievable quality of the technology). Performing this part of the procedures ensures the data quality.

If the buffers used to calibrate the pH meter end up not bracketing the sample value, then check in the meter using both the buffers that were used to calibrate AND buffers that bracket the value of the sample.

These records should be maintained on site or submitted as described in the facility's NPDES or WPCF permit.

An example of a pH logger calibration and audit log:

Logging pH Calibration, Audit, and Drift Check Log (Example)

Facility Name: _____

pH Logger ID: _____

Monitoring Location: _____

Data File Name: _____

Start Date: _____

Date of Battery Installation: _____

Interval: _____

Memory Available: _____

Audit Thermometer Name: WWTP pH Meter 1

Audit Thermometer Model: Name ##

Audit Thermometer Serial Number: #####

PRT Model: _____

Audit Temp Probe Serial #: #####

PRT Serial #: _____

Audit Temp Probe Model: Name #

PRT last date of calibration: _____

Audit pH meter Name: WWTP pH Meter 1

pH logger Model: _____

Audit pH meter Model: Name ##

pH logger Serial Number: _____

Audit pH meter Serial Number: #####

Audit pH Probe Serial #: #####

Audit pH Probe Model: Name #

Pre-Deployment Temperature Check
Temperature 1

| Date/Time | Reference Thermometer Temp | pH Logger Temp | Difference | Status |
|-----------|----------------------------|----------------|------------|--------|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

Pre-Deployment Temperature Check
Temperature 2

| Date/Time | Reference Thermometer Temp | pH Logger Temp | Difference | Status |
|-----------|----------------------------|----------------|------------|--------|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

**temperature from logger must be within +/- 0.5°C of reference thermometer*

Pre-Deployment pH Calibration

pH Buffer 1

| Date/Time | Buffer value @ temp | pH Logger pre-calibration pH |
|-----------|---------------------|------------------------------|
| | | |
| | | |

Pre-Deployment pH Calibration

pH Buffer 2

| Date/Time | Buffer value @ temp | pH Logger pre-calibration pH |
|-----------|---------------------|------------------------------|
| | | |
| | | |

**pH from logger must be within +/-0.2 S.U of the value of the buffer @ temp*

Figure 4: Logging pH calibration, audit, and drift check log (pg. 1)

3 Temperature – Grab Sampling

This section explains methodology for collecting grab (or instantaneous) field temperature data. Grab temperature measurements provide a snapshot of water temperature at the time of collection. This guidance is applicable to measurements of municipal wastewater, partially or fully treated wastewater, stormwater, surface water, and groundwater. It describes the maintenance, calibration, and use of a digital thermometer or field meter with integrated thermometer for obtaining real-time in-situ measurements of temperature. Continuous measurement of temperature is described in the next section.

The intent of this section is to provide operators with adequate information in order to follow procedures outlined in 40 CFR and issued NPDES or WPCF permits.

Thermometers

National Institute of Standards and Technology (NIST): U.S. Physical Science laboratory now part of the U.S. Department of Commerce. NIST provides the standard for calibrating or verifying reference thermometers. <https://www.nist.gov/about-nist>

Thermometer: From USGS National Field Manual, Chapter A6, Temperature, 2006, page T-3: “A *thermometer is any device used to measure temperature, consisting of a temperature sensor and some type of calibrated scale or readout device. Liquid-in-glass thermometers and thermistor thermometers are commonly used to measure air and water temperature.*” Thermometers may be interchangeably referred to as ‘Temperature Sensors’ and/or ‘Thermistors’ in this document.

Reference Thermometer (RT) Calibration: The reference thermometer (see below) is sent to a laboratory to be verified against a SI traceable reference standard platinum resistance thermometer. The range of temperatures that the reference thermometer will be used for is included in the calibration request. The result of the test are compiled into a report that gives the test temperatures and the test thermometer response with the uncertainties associated with the test.

Reference Thermometer: One thermometer is designated as the reference and is used as the standardized unit for checking all other thermometers and water quality meters. The reference thermometer needs to be calibrated/verified yearly by the manufacturer or an accredited laboratory. If the reference is a model that cannot be adjusted (the most common), it needs to be checked against a NIST-certified thermometer by the manufacturer or an accredited laboratory. If a difference greater than 0.1 °C exists, the reference thermometer requires replacement.

Audit thermometer: NIST traceable (calibrated and maintained) thermometer accurate to $\pm 0.2^{\circ}\text{C}$ or a thermometer accurate to $\pm 0.2^{\circ}\text{C}$ or better, that has been checked against an NIST traceable thermometer. (NIST temperatures are given in Celsius.)

Approved Methods

To measure temperature for a NPDES or WPCF permit, one of the following methods must be used, according to CFR Title 40, Chapter I, Subchapter D, Part 136, found here:

<https://www.ecfr.gov/current/title-40/chapter-I/subchapter-D/part-136/section-136.3>

PDF Version of CFR: <https://www.govinfo.gov/content/pkg/CFR-2021-title40-vol25/pdf/CFR-2021-title40-vol25-sec136-3.pdf>

- 1) Standard Methods 2550 B-2010 (or newer):
<https://www.standardmethods.org/doi/full/10.2105/SMWW.2882.031?role=tab>
- 2) USGS
 - a. USGS Method “Water Temperature-Influential Factors, Field Measurement and Data Presentation,” Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1. 1975. USGS.
 - b. 2006 USGS temperature Method, supersedes previous methods:
<https://doi.org/10.3133/twri09A6.1>
 - c. USGS webpage for up to date method publication:
<https://pubs.er.usgs.gov/publication/twri09A6.1>

Prior to taking samples, the facility should select and acquire one of the above methods and keep it on file for frequent reference. The most recent version of the method is the most appropriate method to use. Note that 40 CFR 136 is periodically updated through a Method Update Rule. The permittee should check CFR 136 prior to selecting one of the above methods to confirm that they are still CFR approved.

Calibration/Verification

Facilities have two options for thermometer calibration. The first option is to acquire a reference thermometer that has been calibrated against a NIST SI traceable reference standard platinum resistance thermometer, and then use that thermometer for temperature measurements. This option is acceptable, but be aware that if the reference thermometer is brought into the field, it may be more at risk of breaking or losing calibration. The second option is to acquire both a reference thermometer and a field thermometer. The reference thermometer is typically more expensive and of higher quality, while the field thermometer can be an inexpensive model. The field thermometer is verified against the reference thermometer prior to and after taking temperature measurements. While both options are acceptable, the second option may be preferable because it is easier to acquire a new field thermometer than a calibrated reference thermometer in case the thermometer used in the field is lost or broken.

Steps for verification of a field thermometer:

- 1) Acquire a reference thermometer that has been calibrated against a NIST SI traceable reference standard platinum resistance thermometer.
- 2) Fill a container big enough for both temperature sensors with water and allow to equilibrate to room temperature. Ensure the container is well mixed to ensure equal water temperature throughout the container. The water bath could be mixed by hand, aerated with an air stone, or stirred with a mixer, for example.
- 3) Place the reference thermometer into the water bath and allow to equilibrate
- 4) Place the temperature sensor in the water bath and allow to equilibrate
- 5) Compare the reading on the reference thermometer to the field thermometer. The thermometers must be within 0.5°C (or another specified quality control range)
- 6) Record the value of the reference thermometer, the field thermometer, date, time, and personnel. Keep or submit these records as described in the relevant NPDES or WPCF permit.

Reference Thermometers

The reference thermometer should also be used for the verification of the thermistor on pH probes with Automatic Temperature Compensation (ATC), or for verifying thermometers used to measure the temperature of pH buffer solutions/pH samples and then entered for manual temperature compensation.

Measurement

After calibration, follow the procedure outlined in the selected method for measurement.

- Temperature should be collected *in-situ* or immediately following collection of the sample into a secondary container
- *In-situ* measurements: The temperature probe can be placed directly into the wastewater/water to take the measurement (not advisable if the flow is fast-paced, contains significant debris, or poses a safety hazard) or;
- Secondary container: A secondary container can be used to collect at least 100mL of wastewater/water (or enough to fully submerge the thermometer) and the thermometer can then be placed into the solution for sampling.
- Record the location, time the sample was collected, staff who collected the sample, meter that was used, and the pH reading to the nearest 0.1 S.U.

Drift Check

After removal, the thermometer needs to be re-checked against the reference thermometer used to verify it prior to measurement, to ensure the meter is still working as expected and did not drift over the course of the time which measurements were taken.

Steps for drift check of a field thermometer:

- 1) Place the reference thermometer into the well mixed water bath and allow to equilibrate. The reference thermometer can be kept in the water bath all the time.
- 2) Place the temperature sensor in the water bath and allow to equilibrate
- 3) Compare the reading on the reference thermometer to the field thermometer. The thermometers must be within 0.5°C (or another specified quality control range)
- 4) Record the value of the reference thermometer, the field thermometer, date, time, and personnel. Keep or submit these records as described in the relevant NPDES or WPCF permit. The meter should read within +/- 0.5°C of the known standard (or another assigned acceptance range as described by the standard method used, manufacturer specifications, and actual experience with the achievable quality of the technology). Performing this part of the procedures ensures the data quality.

These records should be maintained on site or submitted as described in the facility's NPDES or WPCF permit.

4 Temperature – Continuous

This section explains methodology for sampling temperature in municipal wastewater, partially or fully treated wastewater, stormwater, and surface water utilizing an in-situ stand-alone temperature meter with an integrated data logger (temperature loggers). This section only provides guidance for temperature loggers designed to be temporarily deployed and operate with a temporary power supply.

Procedures in this section describe the use of a single monitoring device to take continuous measurements of temperature. The intent of this document is to provide operators with adequate information in order to follow procedures outlined in 40 CFR and issued NPDES or WPCF permits.

Approved Methods

To measure temperature for a NPDES or WPCF permit, one of the following methods must be used, according to CFR Title 40, Chapter I, Subchapter D, Part 136, found here:

<https://www.ecfr.gov/current/title-40/chapter-I/subchapter-D/part-136/section-136.3>

PDF Version of CFR: <https://www.govinfo.gov/content/pkg/CFR-2021-title40-vol25/pdf/CFR-2021-title40-vol25-sec136-3.pdf>

- 1) Standard Methods 2550 B-2010 (or newer):
<https://www.standardmethods.org/doi/full/10.2105/SMWW.2882.031?role=tab>
- 2) USGS
 - a. USGS Method “Water Temperature-Influential Factors, Field Measurement and Data Presentation,” Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1. 1975. USGS.
 - b. 2006 USGS temperature Method, supersedes previous methods:
<https://doi.org/10.3133/twri09A6.1>
 - c. USGS webpage for up to date method publication:
<https://pubs.er.usgs.gov/publication/twri09A6.1>

Prior to taking samples, the facility should select and acquire one of the above methods and keep it on file for frequent reference. The most recent version of the method is the most appropriate method to use. Note that 40 CFR 136 is periodically updated through a Method Update Rule. The permittee should check CFR 136 prior to selecting one of the above methods to confirm that they are still CFR approved.

Continuous temperature measurement protocols are not outlined in either Standard Methods 2550-B or the USGS National Field Manual, Chapter 6 – Temperature. The calibration procedures outlined in those standards should be maintained for temperature audit meters, however.

In lieu of a national standard, Oregon NPDES and/or WPCF Permittees may use the guidance as outlined in the list below.

- 1) USGS Techniques and Methods 3-A25, Monitoring Stream Temperatures –A Guide for Non-Specialists: <https://doi.org/10.3133/tm3A25>
- 2) Best Practices for Continuous Monitoring of Temperature and Flow in Wadeable Streams (EPA/600/R-13/170F), Published 7/37/2014, last revised 5/13/2016:
https://cfpub.epa.gov/si/si_public_record_Report.cfm?Lab=NCEA&dirEntryId=280013
 - a. Direct Report Link:
https://cfpub.epa.gov/si/si_public_file_download.cfm?p_download_id=520122&Lab=NCEA

The guidance outlined above are specific to continuous temperature monitoring in streams. These methods are appropriate to use in their entirety if monitoring receiving water as part of a NPDES or WPCF permit. These methods do not describe the installation of temperature loggers in oceans, bays, estuaries, lakes, or wetlands, but the principles for data collection described in the methods apply.

Consider the following additional guidance when deploying a continuous temperature logger in raw or partially treated wastewater, biosolids lagoons, recycled water storage lagoons, or other wastewater treatment plant applications. This guidance only applies to the deployment of temperature loggers designed to be temporarily deployed and operate with a temporary power supply. In the case that a temperature logger is installed that is designed to be a permanent installation and with shore power, the manufacturer’s specifications should be followed, as well as the guidance in the subsequent section – Permanently Installed Temperature Loggers.

Permanently Installed Temperature Loggers

Permanently Installed Temperature Loggers must demonstrate quality as described below:

- Calibrated accuracy within 0.2°C
- Monthly calibration with a NIST certified or manufacturer-certified as NIST traceable thermometer (less frequent calibration frequency can be approved if data and documentation prove data quality), acceptance range is +/- 0.5°C.
- Monthly audits with a reference thermometer or a verified field thermometer (less frequent audit frequency can be approved if data and documentation prove data quality), acceptance range is +/- 0.5°C.

Temperature Loggers

A variety of continuously evolving technologies exist for automatic logging of temperature measurements. Some examples of types of loggers:

- Multiparameter sondes equipped with logging and batteries that have the capacity to measure multiple parameters and can hang into a water body or be placed into a flow through cell
- Single parameter temperature loggers in a plastic or metal casing equipped with logging and replaceable or rechargeable batteries.

- Shore-powered logging controller units with permanently installed temperature probes – these units are outside of the scope of this document and should be installed and maintained according to the approved method and manufacturer specifications as well as the specifications under “Permanently Installed Temperature Loggers” above.

It is necessary to supplement the direction in the approved method with direction from the manufacturer of the temperature logger chosen for use. The following sections provide an overview of the calibration and measurement process for temperature loggers in general, but is not a definitive guide to temperature logger operation.

In summary, a temperature logger should:

- Have a calibrated accuracy within $\pm 0.2^{\circ}\text{C}$
- Digital readout to at least 0.1°C
- Be verified against a calibration thermometer
- Have memory sufficient enough for the necessary monitoring period
- Have battery sufficient enough for the deployment period
- Be sturdy enough for the monitoring application or be able to be mounted in an appropriate protective structure

Equipment

National Institute of Standards and Technology (NIST): U.S. Physical Science laboratory now part of the U.S. Department of Commerce. NIST provides the standard for calibrating or verifying reference thermometers. <https://www.nist.gov/about-nist>

Thermometer: From USGS National Field Manual, Chapter A6, Temperature, 2006, page T-3: “A *thermometer is any device used to measure temperature, consisting of a temperature sensor and some type of calibrated scale or readout device. Liquid-in-glass thermometers and thermistor thermometers are commonly used to measure air and water temperature.*” Thermometers may be interchangeably referred to as ‘Temperature Sensors’ and/or ‘Thermistors’ in this document.

Reference Thermometer Calibration: The reference thermometer (see below) is sent to a laboratory to be verified against a SI traceable reference standard platinum resistance thermometer. The range of temperatures that the reference thermometer will be used for is included in the calibration request. The result of the test are compiled into a report that gives the test temperatures and the test thermometer response with the uncertainties associated with the test.

Reference Thermometer: One thermometer is designated as the reference and is used as the standardized unit for checking all other thermometers and water quality meters. The reference thermometer needs to be calibrated/verified yearly by the manufacturer or an accredited laboratory. If the reference is a model that cannot be adjusted (the most common), it needs to be checked against a

NIST-certified thermometer by the manufacturer or an accredited laboratory. If a difference greater than 0.1 °C exists, the PRT requires replacement.

Audit thermometer: NIST traceable (calibrated and maintained) thermometer accurate to $\pm 0.2^{\circ}\text{C}$ or a thermometer accurate to $\pm 0.2^{\circ}\text{C}$ or better, that has been checked against an NIST traceable thermometer. (NIST temperatures are given in Celsius.)

A suggested equipment list for the calibration, programming, deployment, removal, calibration check, and data retrieval for temperature loggers is listed below. Depending on the equipment selected, additional or fewer equipment could be necessary:

- Temperature logger(s)
- Water bath with a Reference Thermometer
- Device specific batteries or other appropriate power source
- Communication device (typically laptop, tablet, or smartphone), with temperature logger software
- Logger mounting equipment (specific to monitoring location)
- Field thermometer for audit

Calibration

Calibration for temperature loggers can be broken down into the following steps:

- 1) Verify and/or adjust the timeclock on the logger, so that it matches the current time within 3 minutes.
- 2) Logger is placed into a water bath with a PRT, logging is initiated, and temperature allowed to equilibrate. Once the logging temperature has equilibrated, compare the temperature the logger is recording to the temperature measured by the PRT. The temperature read by the logger must be within $\pm 0.5^{\circ}\text{C}$ of the temperature read by the PRT.

Programming

The logger will be programmed:

- To start at the desired date and time
- With the desired sampling interval (e.g., 5-minutes, 15-minutes, 1-hour etc.)
- Loggers can typically be programmed to stop logging on a prescribed date and time, programmed to “slate” (cease logging when memory fills), or “wrap-around” (oldest data is overwritten when memory fills). This setting will be determined by the monitoring objectives; however, “slate” is the most commonly used mode.
- If the temperature logger is equipped with an immersion or light indicator, enable logging of that channel. This will assist in the interpretation of the data and will enable any measurements taken while out of water to be quality flagged as such.

Installation

Deployment configurations will vary based on the monitoring site selected, the type of water being monitored, and the equipment selected.

Tips for installation in common wastewater applications:

- Consider the velocity, depth, and amount of debris present in the wastewater stream where the logger will be installed. The logger could be secured with polypropylene rope, nylon rope, or stainless steel cable depending on the application or configuration. The point where the logger is attached to land should be a secure feature such as a manhole rung, or an eyebolt installed in concrete.
- If water level fluctuates widely, allow enough cable so that the logger stays submerged even during low water level events.
- Rags, oil and grease, and trash pose significant problems when temperature loggers are installed in raw wastewater, such as prior to a headworks barscreen. Temperature loggers will need frequent checks to ensure they have not been ripped of their mount or ensnared in rags. Often a permanently installed logger with a flow through cell or other protective mount will be more reliable.

Once the logger is installed, use a field thermometer to take an audit of the logger. The purpose of the audit is to confirm the accuracy of the logged temperature values. This will help determine the necessary calibration frequency for the logger and ensure that the temperature values record in between audits are of a known quality. When the logger is offloaded, the audit values recorded can be compared to the logged values. Both the logged values and the audit values should be submitted to netDMR if continuous temperature values are requested as part of a NPDES or WPCF permit.

Interim Site Visits

Based on individual project objectives, interim site visits may be conducted during the course of the monitoring period. Reasons for interim site visits could include collection of additional audit/reference readings for data quality verification, data retrieval as logger capacity is approached, and data evaluation to inform project progress and determine needs for continued data collection. Any interim site visit should include an audit/reference measurement with a calibrated thermometer or multiprobe. Prior to removing the temperature logger from the flow for any data retrieval activities, collect an audit/reference measurement as described above. After collecting the audit/reference measurement, the temperature logger can be removed from the flow. Take this opportunity to clear any debris from the temperature logger and inspect it for damage. If retrieving data use the logger software to establish communication with the logger and download the data file. Record the date/time, staff performing the activity, and data file name. The temperature logger can then be returned to the flow.

Removal

Prior to removing the temperature logger from the flow, collect an audit/reference measurement with a calibrated thermometer or multiprobe as described. Record the date and time of the measurement, the temperature reading, the audit meter used at each audit. Remove the temperature logger from the flow, along with any accompanying installation materials. Record the date and time that the logger was removed.

Drift Check

After removal, the logger needs to be checked against the same known standards used to calibrate it, to ensure the meter is still working as expected and did not drift over the course of the time which measurements were taken.

Drift Checks for temperature loggers can be broken down into the following steps:

4. Verify the timeclock on the logger, ensuring that it matches the current time within 3 minutes. If it does not match, the data may need to be adjusted or flagged.
5. Logger is placed into a water bath with a reference thermometer, logging is initiated, and temperature allowed to equilibrate. Once the logging temperature has equilibrated, compare the temperature the logger is recording to the temperature measured by the reference thermometer. The temperature read by the logger must be within +/- 0.5°C of the temperature read by the reference thermometer.

The meter should read within +/- 0.5°C of the known standard (or another assigned acceptance range as described by the standard method used, manufacturer specifications, and actual experience with the achievable quality of the technology). Performing this part of the procedures ensures the data quality.

These records should be maintained on site or submitted as described in the facility's NPDES or WPCF permit.

Continuous Temperature – Calibration Record

An example of a continuous temperature calibration and audit record:

5 Dissolved Oxygen – Grab as “Composite”

This section explains methodology for sampling Dissolved Oxygen (DO) in municipal wastewater, partially or fully treated wastewater, stormwater, and surface water with a handheld Dissolved Oxygen meter. It describes the maintenance, calibration, and use of a single parameter Dissolved Oxygen meter or a multi-parameter water quality meter (multiprobe) installed with a DO probe for obtaining real-time in-situ measurements of DO. The distinction between a “Grab” and “Composite” for Dissolved Oxygen is described below.

Procedures in this section describe the use of a single monitoring device to take real-time measurements of Dissolved Oxygen. The intent of this document is to provide operators with adequate information in order to follow procedures outlined in 40 CFR and issued NPDES or WPCF permits.

What is Dissolved Oxygen?

Dissolved Oxygen (DO) is a measure of how much oxygen is dissolved in water. Aquatic animals require dissolved oxygen to breathe. The amount of dissolved oxygen in a stream or lake is an important indicator of water quality and aquatic health. In the context of wastewater treatment, Biological Oxygen Demand (BOD) is used more often to determine how much dissolved oxygen a waste stream would consume. This section, however, describes direct measurement of the amount of dissolved oxygen in water or wastewater.

Approved Methods

To measure DO for a NPDES permit, one of the following methods must be used, according to CFR Title 40, Chapter I, Subchapter D, Part 136, found here: <https://www.ecfr.gov/current/title-40/chapter-1/subchapter-D/part-136/section-136.3>

PDF Version of CFR: <https://www.govinfo.gov/content/pkg/CFR-2021-title40-vol25/pdf/CFR-2021-title40-vol25-sec136-3.pdf>

Winkler (Azide modification)

- 1) Standard Methods 4500-O (B-F)-2016: <https://www.standardmethods.org/doi/10.2105/SMWW.2882.091>
- 2) ASTM D888-12(A): <https://www.astm.org/d0888-12.html>
- 3) 973.45B (Official Methods of Analysis of the Association of Official Analytical Chemists, Methods Manual, Sixteenth Edition, 4th Revision, 1998. AOAC International) : <https://www.aoac.org/>
- 4) USGS

- a. USGS Method I-1575-78 as in Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1. 1979. USGS.
<https://pubs.usgs.gov/twri/05a01/report.pdf>
- b. 2020 USGS DO Method, supersedes previous methods:
<https://pubs.usgs.gov/tm/09/a6.2/tm9a6.2.pdf>
- c. USGS webpage for up to date method publication:
<https://pubs.usgs.gov/publication/tm9A6.2>

Electrode

- 1) Standard Methods 4500-O G-2016:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.091>
- 2) ASTM D888-12 (A): <https://www.astm.org/d0888-12.html>
- 3) USGS
 - a. USGS Method I-1575-78 as in Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1. 1979. USGS.
<https://pubs.usgs.gov/twri/05a01/report.pdf>
 - b. 2020 USGS DO Method, supersedes previous methods:
<https://pubs.usgs.gov/tm/09/a6.2/tm9a6.2.pdf>
 - c. USGS webpage for up to date method publication:
<https://pubs.er.usgs.gov/publication/tm9A6.2>

Luminescence-Based Sensor

- 1) Standard Methods 4500-O H-2016:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.091>
- 2) ASTM D888-12 (C): <https://www.astm.org/d0888-12.html>
- 3) Hach Method 10360, Luminescence Measurement of Dissolved Oxygen in Water and Wastewater and for Use in the Determination of BOD5 and CBOD5. Revision 1.2, October 2011. Hach Company. This method may be used to measure dissolved oxygen when performing the methods approved in Table IB for measurement of biochemical oxygen demand (BOD) and carbonaceous biochemical oxygen demand (CBOD).
<https://www.hach.com/epa?origin=footer&c1=customer-service&c2=epa-methods&clickedon=epa-methods>
- 4) In-Situ Method 1002-8-2009, Dissolved Oxygen (DO) Measurement by Optical Probe. 2009. In-Situ Incorporated.: <https://in-situ.com/pub/media/support/documents/Alternate-Test-Procedure-N05-0014-In-Situ-Method-1002-8-2009-Dissolved-Oxygen-DO-Measurement-by-Optical-Probe.pdf>
 - a. In-Situ Documentation Page: <https://in-situ.com/us/support/categories/documentation?srch=1002-8>

Prior to taking samples, the facility should select and acquire one of the above methods and keep it on file for frequent reference. The most recent version of the method is the most appropriate method to

use. Note that 40 CFR 136 is periodically updated through a Method Update Rule. The permittee should check CFR 136 prior to selecting one of the above methods to confirm that they are still CFR approved.

Alternative Methods

Where a facility wants to utilize a new method or technology that is not listed in 40 CFR part 136.3, they may apply for an Alternate Testing Procedure (ATP) approval. *40 CFR Part 136.5 Approval of alternate test procedures for limited use* gives direction on this procedure. The Approval must be submitted to the permit inspector once obtained. For more information see DEQ09-LAB-0071-QAG, QA Guidance for Self-Monitoring Laboratories, most recent version. Also see *40 CFR § 136.6 Method modifications and analytical requirements* for additional information about method modifications.

OAR 340-041-0061(10): *“(10) Testing methods. The analytical testing methods for determining compliance with the water quality standards in this rule must comply with 40 CFR Part 136 or, if Part 136 does not prescribe a method, with the most recent edition of Standard Methods for the Examination of Water and Waste Water published jointly by the American Public Health Association, American Water Works Association, and Water Pollution Control Federation; if the department has published an applicable superseding method, testing must comply with the superseding method. Testing in accordance with an alternative method must comply with this rule if the department has published the method or has approved the method in writing.”*

Types of DO Measurements

DO can be measured in three ways:

- 4) Winkler (Azide modification): a titrimetric procedure performed in a laboratory
 - a. The collection of these samples requires multiple considerations to avoid adding or depleting the dissolved oxygen content of the sample. The sample must then be analyzed or preserved as soon as possible. This method is often impracticable if a laboratory with the capability for this method is not at the site where the sample is to be collected.
- 5) Electrode: a handheld meter and probe with a small membrane stretched over the end
 - a. These probes have two solid metal electrodes submersed in electrolyte solution, and a membrane stretched over the top. They are easy to use for field measurements and variety of manufacturers produce these types of probes. The membrane can be easily torn or displaced by passing debris, such as in streams or raw wastewater.
- 6) Luminescence-Based Sensor: also called “optical DO probes”, these are a handheld meter and probe with a luminescence-based sensor
 - a. These probes have two LEDs and luminescent material that is able to determine the concentration of oxygen in the sample. These probes can be impacted by biofouling (bacterial or algal growth), oils, or damaged by alcohols and organic solvents. Generally, these probes can be more reliable than membrane in fast moving streams with a variety of debris, or in wastewater streams with debris. Refer to the

manufacturer and method when determining what probe might be best for your application.

There are colorimetric methods for measuring dissolved oxygen, but none are approved for NPDES monitoring.

Dissolved Oxygen meters

Environmental monitoring companies offer a wide variety of products that use the methods listed under Electrode and Luminescence Based Sensor above. Companies will typically publish a method specific to their instruments. That method may be based on one of the methods approved in 40 CFR Part 136 listed above. If the method is based on the approved methods, then the manufacturer may have approval from the Environmental Protection Agency (EPA) for that instrument to be used for NPDES permit monitoring. Contact the manufacturer to ask if the company method is approved for NPDES permit monitoring.

A manufacturer may also have Alternate Test Procedure (ATP) approval, where the EPA approved a method that does not align with a Standard Methods, ASTM, AOAC, or USGS method, but the method is still acceptable for use for NPDES permit monitoring. Those methods with ATP approval may be listed in 40 CFR 136, such as Hach Method 10360 and In-Situ Method 1002-8-2009 (listed above). Contact the manufacturer if you are unsure if the method has ATP approval.

Calibration

DO meters must be calibrated according to the specifications of the manufacturer and the 40 CFR 136 approved methodology being used. Since many different models of probe can be used with the approved methods, this document will not outline calibration procedures for each individual model of probe. Contact the manufacturer if unsure of calibration procedures.

DO meter calibration tips

The following is a list of common areas to troubleshoot for polarographic or optical sensors. This list does not apply to every manufacturer's equipment or style of probe that can be used with the approved methods. Contact the manufacturer if unsure of calibration procedures, or if the calibration issue is not addressed below.

Polarographic sensors

- A polarographic sensor often utilizes a cathode and anode in an electrolyte solution, sealed by a semi-permeable membrane. Visually check the membrane for wrinkles, air bubbles, rips and tears, or evidence of having been dried-out. The DO sensor will need to be maintained if any of these conditions exist. Maintenance could include membrane replacement, cleaning, or replacement. Check the specifications on the sensor for maintenance information.

- If the unit has a polarographic sensor and DO will not calibrate because the adjustment is beyond the acceptable tolerance, or if the readings do not seem accurate, examine the dissolved oxygen sensor membrane for wrinkles or bubbles under the membrane, and replace if necessary (calibration cannot occur for several hours after the membrane is changed).
- If there are chronic problems with calibration or stabilization of readings, the gold cathode or silver anode may need maintenance. Consult the manufacturer for information on how to resurface or repair a fouled anode or cathode.
- Consult the manufacturer for instructions on how to replace the membrane on your probe, and the time for which the membrane must rest after replacement prior to use.

Optical Sensor

- DO sensors require the input of the barometric pressure from a built in barometer. The solubility of a gas in water is directly related to the partial pressure of that gas in the surrounding atmosphere, as described in Henry's Law. Partial Pressure is the pressure exerted by an individual gas in a mixture. The total pressure in an atmosphere is calculated by adding the partial pressures of all the component gases that make up the atmosphere mixture. Because of those relationships, as the local atmospheric pressure increases, the partial pressure of oxygen in the atmosphere also increases, and therefore the solubility of oxygen in water will also increase. Ensure that the barometer is functioning prior to calibration. The barometer in the meter should be reading the current air pressure, so the calibration is determined using the correct saturation value. Given the lack of a barometer in a meter, input the atmospheric pressure as 760 mmHg (or the typical atmospheric pressure at the sampling location).
- Visually inspect the sensing element to ensure that the dye layer is intact and has not been scraped or scratched off.
- Check the replacement schedule for the sensing element – some require replacement every 12 to 18 months, and an expired probe should not be used for permit monitoring. Some manufacturers program a hard-stop date into the probe, after which it cannot be used.

A calibrated dissolved oxygen sensor should measure within 10% of the known standard. For example, if comparing the sensor against an environment with 100% dissolved oxygen saturation, the acceptance range is 90% to 110% saturation.

Calibration Records

Pre-Measurement Calibration

When calibrating prior to measurement, record the following information:

- 9) The date
- 10) The name of the personnel who performed the calibration
- 11) The time the calibration was performed
- 12) The barometric pressure at the time of calibration
- 13) The % saturation of the standard
- 14) The value that the meter was reading prior to calibration (pre-equilibrated value)

An example of a calibration (pre-measurement) record is shown below:

| CALIBRATION & DRIFT CHECK | | | | | |
|--|----------------|--------------------|------------------------|---------------|-------------------------------------|
| Parameter | Units | PRE | Calibration | | POST |
| | | Equilibrated Value | Standard | | Equilibrated Value |
| Dissolved Oxygen | (% saturation) | 98% | Pressure (mmHg) 760 | % Sat. 100 | plus/minus 10% of value of standard |
| If any reported parameter falls outside of the acceptable range add the following phrase in the comments section of the Chain of Custody/Field Data Sheet: The result for (DO, pH, Sp. Cond., Temp., Turb., ORP) should be considered an estimate due to the post-measurement check of the field meter being outside the acceptance range. In addition, if space permits, precede each reading on the COC or FDS with EST. | | | | | |
| Notes: | | | | | |
| | | | | | |

Figure 8: Pre-Measurement Calibration Record

Measurement

After calibration, follow the procedure outlined in the selected method for measurement. To ensure accurate measurement, the following is advised:

- The DO probe can be placed directly into the wastewater/water to take the measurement (not advisable if the flow is very fast, contains significant debris, or poses a safety hazard) or;
- A decontaminated secondary container (see below – Decontamination) can be used to collect at least 100mL of wastewater/water and the probe can then be placed into the solution for sampling. Rinse the secondary container with the solution to be measured, dump the rinsate out, and then collect a second fresh sample for measurement.
- Record the location, time the sample was collected, staff who collected the sample, meter that was used, and the DO in mg/L to the nearest 0.1 mg/L
- Note that calibration is typically performed using percent saturation, while measurements are recorded in mg/L.
- Oregon’s NPDES permits may have the following note in Schedule B: ‘For Dissolved Oxygen, the permittee must collect and analyze at least four discrete grab samples over the operating day with samples collected no less than one hour apart. The analytical results for all samples in a day must be averaged for reporting purposes.’
 - This note directs that the permittee should use their dissolved oxygen probe to collect four measurements (for example, one at 1000, 1100, 1200, and 1300). Each of the four measurements should be recorded, and then averaged. For example, if the four measurements are 12.5 mg/L at 1000, 10.1 mg/L at 1100, 9.9 mg/L at 1200, and 11.3

mg/L at 1300, then the average of those four results is 10.95 mg/L , which rounds to 11.0 mg/L using three significant figures. The value of 11.0 mg/L will be the single value reported in netDMR, and the 11.0 mg/L value is considered a “composite”.

- If a single measurement is taken, that single measurement is considered a “grab” for the purposes of NPDES reporting.

Post-Measurement Drift Check

After pH measurements have been completed on any given day the meter needs to be checked against the same known standards used to calibrate it to ensure the meter is still working as expected and did not drift over the course of the time which measurements were taken. The user will check the DO probe by measuring the buffers used to calibrate the meter prior to taking a measurement. The user records the value the meter reads and writes it down. The meter should read within 10% of the known standard (or another assigned acceptance range as described by the standard method used). Performing this part of the procedures ensures the data quality.

Below is an example of the check-in portion of a Calibration record:

| CALIBRATION & DRIFT CHECK | | | | | | |
|---|----------------|--------------------|-------------|-----------------|--------|-------------------------------------|
| Parameter | Units | PRE | Calibration | | POST | |
| | | Equilibrated Value | Standard | Pressure (mmHg) | % Sat. | Equilibrated Value |
| Dissolved Oxygen | (% saturation) | 98% | 760 | 100 | 101 | plus/minus 10% of value of standard |
| If any reported parameter falls outside of the acceptable range add the following phrase in the comments section of the Chain of Custody/Field Data Sheet: The result for (DO, pH, Sp. Cond., Temp., Turb.,ORP) should be considered an estimate due to the post-measurement check of the field meter being outside the acceptance range. In addition, if space permits, precede each reading on the COC or FDS with EST. | | | | | | |
| Notes: | | | | | | |
| | | | | | | |

Figure 9: Post-Measurement Calibration Record

These records should be maintained on site or submitted as described in the facility’s NPDES or WPCF permit.

Decontamination

Equipment needed:

- Cellulose scrub pads and/or plastic scrub brushes
- Decontamination solutions:

- Laboratory grade phosphate-free detergent (1% solution)
- Tap water
- Deionized water (DI water)

Sample containers

The container used to collect a wastewater or water sample or measurement with a DO probe should be washed according to the following procedure: Wash the container prior to sample collection with 1% detergent/tap water solution and a non-scouring scrub brush or pad. Rinse the container with tap water. Collect the sample as described in the approved method and as outlined in the measurement step above.

6 E. coli, Fecal Coliform, Total Coliform, and Enterococci

This section explains methodology for sampling E. coli, Fecal Coliform, Total Coliform, and/or Enterococci (referred to generally as “bacteria”) in municipal wastewater, partially or fully treated wastewater, stormwater, surface water, or groundwater. Guidance for the analysis of bacteria in the laboratory can be found in the DEQ guidance document QA Guidance for Self-Monitoring Laboratories (DEQ 09-LAB-0071-QAG). The intent of this section is to provide operators with adequate information in order to follow procedures outlined in 40 CFR and issued NPDES or WPCF permits.

Approved Methods

To measure for a NPDES permit, one of the following methods must be used, according to CFR Title 40, Chapter I, Subchapter D, Part 136, found here: <https://www.ecfr.gov/current/title-40/chapter-I/subchapter-D/part-136/section-136.3>

PDF Version of CFR: <https://www.govinfo.gov/content/pkg/CFR-2021-title40-vol25/pdf/CFR-2021-title40-vol25-sec136-3.pdf>

E. coli

- 1) MPN Multiple Tube
 - a. Standard Methods 9221 B2014/9221 F-2014:
<https://www.standardmethods.org/doi/abs/10.2105/SMWW.2882.192>
- 2) Multiple tube/Multiple well
 - a. Standard Methods 9223 B-2016:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.194>
 - b. Official Methods of Analysis of AOAC International. 16th Edition, 4th Revision, 1998. AOAC International - Method 991.15: <https://www.aoac.org/>
 - c. IDEXX Laboratories, Inc. - Colilert (this method is now included in Standard Methods for the Examination of Water and Wastewater, 23rd edition, Method 9223 B, Enzyme Substrate Coliform Test)
 - i. <https://www.idexx.com/files/5L-v4water-regulatory-article.pdf>
 - ii. <https://www.standardmethods.org/doi/10.2105/SMWW.2882.194>
 - d. IDEXX Laboratories, Inc. - Colilert-18[®] (this method is now included in Standard Methods for the Examination of Water and Wastewater, 23rd edition, Method 9223 B, Enzyme Substrate Coliform Test)
 - i. <https://www.idexx.com/files/5L-v4water-regulatory-article.pdf>
 - ii. <https://www.standardmethods.org/doi/10.2105/SMWW.2882.194>

- 3) MF, two step
 - a. Standard Methods 9223 B-2015/9222 I-2015 Membrane Filter Technique for Members of the Coliform Group:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.193>
- 4) Single step
 - a. EPA Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified Membrane-Thermotolerant *Escherichia coli* Agar (modified mTEC), EPA-821-R-14-010. September 2014: https://www.epa.gov/sites/default/files/2015-08/documents/method_1603_2009.pdf
 - b. Hach Company - m-ColiBlue24®: USEPA Simultaneous Detection and Enumeration of Total Coliforms and *Escherichia coli* using m-ColiBlue24 Membrane Filtration Medium
 - i. <https://www.hach.com/m-coliblu24-broth-plastic-ampules-50-pk/product-downloads?id=7640249626>

Fecal Coliform

- 1) MPN, 5 tube, 3 dilution
 - a. EPA Microbiological Methods for Monitoring the Environment, Water and Wastes, EPA/600/8-78/017. 1978. p. 132:
<https://nepis.epa.gov/Exe/ZyPURL.cgi?Dockey=300014TD.txt>
 - b. Standard Methods 9221 E-2014; 9221 F-2014:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.194>
- 2) Multiple tube/Multiple well
 - a. Colilert-18®(this method is now included in Standard Methods for the Examination of Water and Wastewater, 23rd edition, Method 9223 B, Enzyme Substrate Coliform Test)
 - i. <https://www.idexx.com/files/5L-v4water-regulatory-article.pdf>
 - ii. <https://www.standardmethods.org/doi/10.2105/SMWW.2882.194>
- 3) MF, single step
 - a. EPA Microbiological Methods for Monitoring the Environment, Water and Wastes, EPA/600/8-78/017. 1978. p. 124:
<https://nepis.epa.gov/Exe/ZyPURL.cgi?Dockey=300014TD.txt>
 - b. Standard Methods 9222 D-2015:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.193>

Total Coliform

- 1) MPN, 5 tube, 3 dilution
 - a. EPA Microbiological Methods for Monitoring the Environment, Water and Wastes, EPA/600/8-78/017. 1978. pg. 114:
<https://nepis.epa.gov/Exe/ZyPURL.cgi?Dockey=300014TD.txt>
 - b. Standard Methods 9221 B-2014:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.192>
- 2) MF, single step or two step

- a. EPA Microbiological Methods for Monitoring the Environment, Water and Wastes, EPA/600/8-78/017. 1978. pg. 108:
<https://nepis.epa.gov/Exe/ZyPURL.cgi?Dockey=300014TD.txt>
 - b. Standard Methods 9222 B-2015:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.193>
 - c. U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples. 1989. Method B-0025-85:
https://pubs.usgs.gov/twri/twri5a4/pdf/TWRI_5-A4.pdf
- 3) MF, with enrichment
- a. EPA Microbiological Methods for Monitoring the Environment, Water and Wastes, EPA/600/8-78/017. 1978. pg. 111:
<https://nepis.epa.gov/Exe/ZyPURL.cgi?Dockey=300014TD.txt>
 - b. Standard Methods 9222 B-2015:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.193>

Enterococci

- 1) MPN, 5 tube, 3 dilution
 - a. EPA Microbiological Methods for Monitoring the Environment, Water and Wastes, EPA/600/8-78/017. 1978. pg. 139:
<https://nepis.epa.gov/Exe/ZyPURL.cgi?Dockey=300014TD.txt>
 - b. Standard Methods 9230 B-2013:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.193>
- 2) MPN, multiple tube/multiple well
 - a. Standard Methods 9230 D-2013:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.197>
 - b. Annual Book of ASTM Standards-Water and Environmental Technology, Section 11.02. 2000, 1999, 1996. ASTM International. Method D6503-99:
<https://www.astm.org/d6503-99.html>
 - c. Enterolert® (this method is now included in the Standard Methods for the Examination of Water and Wastewater, 23rd edition, Method 9230)
 - i. <https://www.idexx.com/files/5GCwater-regulatory-art.pdf>
 - ii. <https://www.standardmethods.org/doi/10.2105/SMWW.2882.197>
- 3) MF, single step
 - a. EPA Method 1600: Enterococci in Water by Membrane Filtration Using Membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI), EPA-821-R-14-011. September 2014. Method 1600: https://www.epa.gov/sites/default/files/2018-06/documents/method_1600_sept-2014.pdf
 - b. Standard Methods 9230 C-2013:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.197>
- 4) Plate count

- a. EPA Microbiological Methods for Monitoring the Environment, Water and Wastes, EPA/600/8-78/017. 1978. p. 143:
<https://nepis.epa.gov/Exe/ZyPURL.cgi?Dockey=300014TD.txt>

Sample Bottles for E. coli, Fecal Coliform, Total Coliform, and Enterococci

If using an outside laboratory, the lab will provide the appropriate container.

Glass sample bottles should be wide-mouth, reusable, nonreactive, autoclavable, borosilicate glass with screw caps or ground glass stoppers. Plastic bottles should have wide-mouth, reusable, nonreactive, autoclavable (ability to be heat sterilized without producing toxic materials), have screw caps (that do not produce bacteriostatic or nutritive compounds upon sterilization), and can be either linerless or have insert liners. Commercially prepared pre-sterilized plastic bottles or bags can also be used, as long as the container is large enough to collect the needed sample and still have 1 inch of headspace to shake the sample in the container. The sample size is determined by both the minimum volume in the approved method and the laboratory's needs. Typically, sample bottles are at least 250mL, and up to 1000mL in volume. Do not use bottles that have chips, cracks, or etched surfaces, Bottle closures must be water-tight. If preparing reusable bottles, follow the instructions in the approved method.

E. coli, Fecal Coliform, Total Coliform, and Enterococci Sample Collection

Bacteria samples must be collected without the use of a secondary container. It is not acceptable to fill another container and then fill the sample bottle from that container. The sterile sample bottle must be directly filled without the use of an intermediate vessel. The bottle can be dipped or lowered into the flow multiple times if necessary to achieve the necessary sample volume. If collecting the sample from the chlorinated location, use sterile bottles with sodium thiosulfate as outlined in the approved method. Bacteria samples cannot be collected through a pump/tubing.

Frequently bacteria samples need to be collected from a vault, manhole, from a bridge, or other inaccessible location. In this case the bacteria bottle should be directly placed into a sample apparatus to be lowered into the flow. Commercially available sampling devices may be available to fit the sample bottle. A sample apparatus can be constructed from two zip ties and string as shown below:

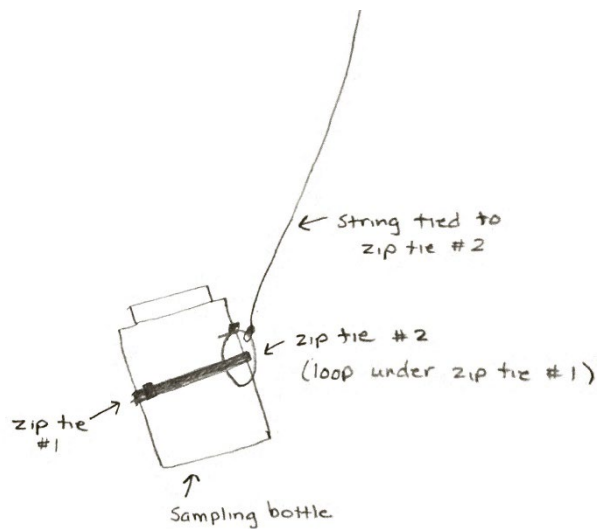


Figure 10: Simple sample apparatus using two zip ties and string to collect a sample directly into a sample bottle from a deep or unreachable location (Prichard 2022)

If a duplicate bacteria sample is needed, either hold two bottles side by side in the flow simultaneously to fill, or lower two bottles in two sampling apparatus' side by side simultaneously into the flow. This is also the appropriate way to collect two bacteria samples if split sampling is being conducted. Once collected, samples need to be analyzed within the EPA allowed 8 hour holding time (40 CFR 136).

7 BOD5

This section explains methodology for sampling for Biological Oxygen Demand analysis in municipal wastewater, partially or fully treated wastewater, stormwater, surface water, or groundwater. Guidance for the analysis of BOD in the laboratory can be found in the DEQ guidance document QA Guidance for Self-Monitoring Laboratories (DEQ 09-LAB-0071-QAG). The intent of this section is to provide operators with adequate information in order to follow procedures outlined in 40 CFR and issued NPDES or WPCF permits.

Approved Methods

To measure DO for a NPDES permit, one of the following methods must be used, according to CFR Title 40, Chapter I, Subchapter D, Part 136, found here: <https://www.ecfr.gov/current/title-40/chapter-I/subchapter-D/part-136/section-136.3>

PDF Version of CFR: <https://www.govinfo.gov/content/pkg/CFR-2021-title40-vol25/pdf/CFR-2021-title40-vol25-sec136-3.pdf>

- 1) Standard Methods 5210 B-2016:
<https://www.standardmethods.org/doi/10.2105/SMWW.2882.102>
- 2) Official Methods of Analysis of the Association of Official Analytical Chemists, Methods Manual, Sixteenth Edition, 4th Revision, 1998. AOAC International. Method 973.44:
<https://www.aoac.org/official-methods-of-analysis-21st-edition-2019/>
- 3) In-Situ Method 1003-8-2009, Biochemical Oxygen Demand (BOD) Measurement by Optical Probe. 2009. In-Situ Incorporated.: <https://in-situ.com/pub/media/support/documents/Alternate-Test-Procedure-N09-0020-In-Situ-Method-1003-8-2009-Biochemical-Oxygen-Demand-BOD-Measurement-by-Optical-Probe.pdf>
- 4) Hach Method 10360, Luminescence Measurement of Dissolved Oxygen in Water and Wastewater and for Use in the Determination of BOD₅ and CBOD₅. Revision 1.2, October 2011. Hach Company. This method may be used to measure dissolved oxygen when performing the methods approved in Table IB for measurement of biochemical oxygen demand (BOD) and carbonaceous biochemical oxygen demand (CBOD): https://www.hach.com/cms-portals/hach_com/cms/documents/pdf/Methods-Guidelines/Method-10360.pdf

Sampling for BOD₅ Analysis - Grab

This section applies to Standard Methods 5210-B and AOAC Method 973.44. In-Situ Method 1003-8-2009 and Hach Method 10360 use water quality meters for the measurement of BOD. Note that BOD is commonly collected as a 24-hour composite sample. This guidance is included in the event a permittee is allowed to collect BOD as a grab sample according to their specific permit or in case a permittee is collecting discrete grabs that will be composited together.

- If using an outside laboratory, the lab will provide the appropriate container.
- Typically, a BOD sample is collected in a secondary bottle, then transferred to the incubation containers in the laboratory. If another container is not provided by the laboratory, a 1L amber glass bottle can be used. Fill the bottle to the shoulder.
- The sampling container can be directly filled, filled from a secondary container, or from a peristaltic pump.
 - If using a secondary container, decontaminate the container or tubing prior to sampling with 1% detergent/tap water solution and a non-scouring scrub brush or pad. Then rinse the container with tap water. Let the container air dry, upside down, on a clean surface such as on a new, absorbent paper towel, in an area with minimal dust.
 - If using a peristaltic pump, use new silicone, Teflon, or polyethylene tubing; or decontaminate previously used tubing using the following method:
 - Calculate the internal volume of the tubing by dividing the internal diameter (ID) by 2 to get the radius. The internal volume is equal to $(\pi)(r^2)(\text{length of tubing})$.
 - Prepare 5x the internal volume of 1% detergent/tap water solution and tap water.
 - Pump the 1% detergent/tap water solution through the tubing
 - Pump the tap water through the tubing.
 - The tubing can now be used for BOD analysis.
 - Tubing connected to the autosampler should be replaced once a month or at a schedule justified by QA/QC samples including field blanks.
 - Protect tubing from sunlight exposure, replace tubing if sedimentation or biofilm growth occurs in tubing interior.
- If analysis begins within 2h of collection, cold storage is unnecessary. Otherwise, keep sample at less than or equal to 6°C between collection and analysis. Grab samples that will be composited together should be kept at less than or equal to 6°C before compositing. Ideally, begin analysis within 6 hours of sample collection. If impossible due to distance between sampling site and laboratory, then begin analysis within 24 hours of collection. The recommended holding time is 24 hours; however, the EPA allows for a 48-hour holding time (40 CFR 136). The EPA allowed holding time of 48 hours begins at the time of end of collection for the composite sample. For a grab sample that will not be part of a composite sample, the holding time begins at the time of collection (40 CFR 136)

Sampling for BOD₅ Analysis – 24-hour composite

This section applies to Standard Methods 5210-B and AOAC Method 973.44. In-Situ Method 1003-8-2009 and Hach Method 10360 use water quality meters for the measurement of BOD.

If the permit requires a 24-hour composite for the BOD sample, follow the subsequent guidance when collecting samples:

- Samples may be collected via an autosampler or as a composite of discrete grabs (see section above for grab sample collection). For discrete grabs, the permittee must collect a minimum of 4 samples over a 24 hour period (40 CFR § 122.21)
- Use new tubing prior to sampling, or decontaminate used silicone, Teflon, or polyethylene tubing using the following method:
 - Calculate the internal volume of the tubing by dividing the internal diameter (ID) by 2 to get the radius. The internal volume is equal to $(\pi)(r^2)(\text{length of tubing})$.
 - Prepare 5x the internal volume of 1% detergent/tap water solution and tap water.
 - Pump the 1% detergent/tap water solution through the tubing
 - Pump the tap water through the tubing.
 - The tubing can now be used for BOD analysis.
- If the sampler and tubing are permanently fixed in one location, the tubing may be reused between samples. However, if the tubing shows significant accumulation of biofilm, debris, FOG, or other material (interior or exterior); cracking, thinning, or other contamination or damage the tubing should be replaced. Recommended replacement or decontamination is once every 4 weeks in wastewater applications. If decontaminating tubing, it is recommended to replace all tubing annually.
- Composite samples should be kept at less than or equal to 6°C during collection. The EPA allowed holding time of 48 hours begins at the time of end of collection for the composite sample (40 CFR 136).

8 Total Suspended Solids

This section explains methodology for sampling for Total Suspended Solids (TSS) analysis in municipal wastewater, partially or fully treated wastewater, stormwater, surface water, or groundwater. Guidance for the analysis of TSS in the laboratory can be found in the DEQ guidance document QA Guidance for Self-Monitoring Laboratories (DEQ 09-LAB-0071-QAG). The intent of this section is to provide operators with adequate information in order to follow procedures outlined in 40 CFR and issued NPDES or WPCF permits.

Approved Methods

To measure DO for a NPDES permit, one of the following methods must be used, according to CFR Title 40, Chapter I, Subchapter D, Part 136, found here: <https://www.ecfr.gov/current/title-40/chapter-I/subchapter-D/part-136/section-136.3>

PDF Version of CFR: <https://www.govinfo.gov/content/pkg/CFR-2021-title40-vol25/pdf/CFR-2021-title40-vol25-sec136-3.pdf>

- 1) Standard Methods 2540 D-2015 – Total Suspended Solids Dried at 103°C – 105°C: <https://www.standardmethods.org/doi/10.2105/SMWW.2882.030>
- 2) ASTM D5907-13, Standard Test Methods for Filterable Matter (Total Dissolved Solids) and Nonfilterable Matter (Total Suspended Solids) in Water: <https://www.astm.org/d5907-13.html>
- 3) USGS Techniques of Water-Resources Investigations of the United States Geological Survey, Chapter A1, Methods for Determination of Inorganic Substances in Water and Fluvial Sediment, Book 5 – Laboratory Analysis; Method I-1750-85: Solids, residue on evaporation at 180°C, dissolved, gravimetric: https://pubs.usgs.gov/twri/twri5-a1/pdf/twri_5-A1_n.pdf

Sampling for TSS Analysis - Grab

- Use borosilicate glass, plastic, or Teflon bottles, so long as suspended solids in sample do not adhere to container walls.
- Rinse the container with sample water prior to filling.
- Fill the bottle to the shoulder.
- The container can be directly filled, filled from a secondary container, or from a peristaltic pump.
- The laboratory will specify the volume, but a typical volume for TSS sampling is 1 liter.
- Begin analysis as soon as possible, the maximum hold time is 7 days (40 CFR 136).
- Between collection and analysis, refrigerate (but do not freeze) sample at less than 6°C to minimize microbiological decomposition of solids.

Sampling for TSS Analysis – 24-hour composite

If the permit requires a 24-hour composite for the TSS sample, follow the subsequent guidance when collecting samples:

- Samples may be collected via autosampler or as a composite of discrete grabs (see section above for grab sample collection). For discrete grabs, the permittee must collect a minimum of 4 samples over a 24 hour period (40 CFR § 122.21(7)(i)).
- Use new tubing prior to sampling, or decontaminate used silicone, Teflon, or polyethylene tubing using the following method:
 - Calculate the internal volume of the tubing by dividing the internal diameter (ID) by 2 to get the radius. The internal volume is equal to $(\pi)(r^2)(\text{length of tubing})$.
 - Prepare 5x the internal volume of 1% detergent/tap water solution and tap water.
 - Pump the 1% detergent/tap water solution through the tubing
 - Pump the tap water through the tubing.
 - The tubing can now be used for TSS analysis.
- If the sampler and tubing are permanently fixed in one location, the tubing may be reused between samples. However, if the tubing shows significant accumulation of biofilm, debris, FOG, or other material (interior or exterior); cracking, thinning, or other contamination or damage the tubing should be replaced. Recommended replacement or decontamination is once every 4 weeks in wastewater applications. If decontaminating tubing, it is recommended to replace all tubing annually.

References

- AOAC International. (1998a). AOAC 973.41. In AOAC Official Methods of Analysis of the Association of Official Analytical Chemists, Methods Manual, Sixteenth Edition, 4th Revision. <https://www.aoac.org/scientific-solutions/standards-and-official-methods/>
- AOAC International. (1998b). AOAC 973.45B . In Official Methods of Analysis of the Association of Official Analytical Chemists, Methods Manual, Sixteenth Edition, 4th Revision,. <https://www.aoac.org/>
- AOAC International. (1998c). AOAC 991.15. <https://www.aoac.org/>
- ASTM International. (1999). ASTM D1293-99 Standard Test Methods for pH of Water. 11.01, 1–9. <https://doi.org/10.1520/D1293-99>
- ASTM International. (2012). ASTM D888-12 Standard Test Methods for Dissolved Oxygen in Water.
- ASTM International. (2013). ASTM D5907-13. <https://doi.org/10.1520/D5907-13>
- Bordner, R., & Winter, J. (1978). EPA-600/8-78-017 Microbiological Methods for Monitoring the Environment. <https://nepis.epa.gov/Exe/ZyPURL.cgi?Dockey=300014TD.txt>
- Field Operations - City of Portland. (2000). Design for E. coli Sampling Apparatus ('TUSS').
- Hach Company. (1999). Simultaneous Detection and Enumeration of Total Coliforms and Escherichia coli using m-ColiBlue24 Membrane Filtration Medium. <https://www.hach.com/m-colibblue24-broth-plastic-ampules-50-pk/product-downloads?id=7640249626>
- Hach Company. (2021). Method 10360 Dissolved Oxygen DOC316.53.01243. <https://www.hach.com/assetsref/56933>
- Hach Company, Bier, A. W., & Hach-Lange. (2009). pH Measurement in Low-ionic Strength (Pure) Samples (Lit. No. 2073).
- Heck, M. P., Schultz, L. D., Hockman-Wert, D., Dinger, E. C., & Dunham, J. B. (2018). Monitoring stream temperatures—A guide for non-specialists. In U.S. Geological Survey Techniques and Methods, book 3, chap. A25. <https://pubs.usgs.gov/publication/tm3A25>
- Heck, M. P., Schultz, L. D., Hockman-Wet, D., Dinger, E. C., Dunham, J. B., U.S. Geological Survey, & National Park Service. (2018). Monitoring Stream Temperatures - A Guide for Non-Specialists. In U.S. Geological Survey Techniques and Methods, book 3, chap. A25 (Vol. 3). <https://doi.org/https://doi.org/10.3133/tm3A25>
- In-Situ Inc. (2009). Method 1003-8-2009 Biochemical Oxygen Demand (BOD) Measurement by Optical Probe. <https://in-situ.com/pub/media/support/documents/Alternate-Test-Procedure-N09-0020-In-Situ-Method-1003-8-2009-Biochemical-Oxygen-Demand-BOD-Measurement-by-Optical-Probe.pdf>

Michaud, J. P. (1991). A Citizen's Guide to Understanding and Monitoring Lakes and Streams. <https://apps.ecology.wa.gov/publications/documents/94149.pdf>

NIST. (2024). National Institute of Standards and Technology (NIST). <https://www.nist.gov/about-nist>

Oregon Department of Environmental Quality Laboratory and Environmental Assessment Division. (2021). Standard Operating Procedure Electrometric Measurement of Hydrogen Ion Activity (pH) - Revision 4.7 - DEQ97-LAB-0023-SOP.

Oregon DEQ Laboratory and Environmental Assessment Division. (2019). Guidance for Self Monitoring Laboratories (NPDES and WPCF).

Oregon Plan for Salmon and Watersheds. (2000). Water Quality Monitoring Guidebook Version 2.0. <https://www.oregon.gov/oweb/Documents/Water%20Quality%20Monitoring%20Guidebook.aspx>

Rice, E. W., Baird, R. B., & A.D., E. (2017). 9223 B. Enzyme Substrate Test. <https://www.idexx.com/files/5GCwater-regulatory-art.pdf>

Standard Methods. (2015). 2540 SOLIDS. In Standard Methods For the Examination of Water and Wastewater. <https://doi.org/10.2105/SMWW.2882.030>

Standard Methods. (2018). 4020 QUALITY ASSURANCE/QUALITY CONTROL. In Standard Methods For the Examination of Water and Wastewater.

Standard Methods Committee of the American Public Health Association, American Water Works Association, & Water Environment Federation. (2010). 2550 TEMPERATURE. In Standard Methods For the Examination of Water and Wastewater. <https://doi.org/10.2105/SMWW.2882.031>

Standard Methods Committee of the American Public Health Association, American Water Works Association, & Water Environment Federation. (2011). 4500-H+ pH. In W. Lipps, T. Baxter, & Braun-Howland E (Eds.), Standard Methods For the Examination of Water and Wastewater. APHA Press. <https://doi.org/10.2105/SMWW.2882.082>

Standard Methods Committee of the American Public Health Association, American Water Works Association, & Water Environment Federation. (2013). 9230 Fecal Enterococci. In Standard Methods For the Examination of Water and Wastewater. <https://doi.org/10.2105/SMWW.2882.197>

Standard Methods Committee of the American Public Health Association, American Water Works Association, & Water Environment Federation. (2014). 9221 MULTIPLE-TUBE FERMENTATION TECHNIQUE FOR MEMBERS OF THE COLIFORM GROUP. In W. Lipps, T. Baxter, & E. Braun-Howland (Eds.), Standard Methods For the Examination of Water and Wastewater. APHA Press. <https://doi.org/10.2105/SMWW.2882.192>

Standard Methods Committee of the American Public Health Association, American Water Works Association, & Water Environment Federation. (2015). 9222 Membrane Filter Technique for Members of

the Coliform Group. In Standard Methods For the Examination of Water and Wastewater.

<https://doi.org/10.2105/SMWW.2882.193>

Standard Methods Committee of the American Public Health Association, American Water Works Association, & Water Environment Federation. (2016a). 4500-O OXYGEN (DISSOLVED). In Standard Methods For the Examination of Water and Wastewater. <https://doi.org/10.2105/SMWW.2882.091>

Standard Methods Committee of the American Public Health Association, American Water Works Association, & Water Environment Federation. (2016b). 5210 BIOCHEMICAL OXYGEN DEMAND (BOD). In Standard Methods For the Examination of Water and Wastewater.

<https://doi.org/10.2105/SMWW.2882.102>

Standard Methods Committee of the American Public Health Association, American Water Works Association, & Water Environment Federation. (2016c). 9223 ENZYME SUBSTRATE COLIFORM TEST. In W. Lipps, T. Baxter, & E. Braun-Howland (Eds.), Standard Methods For the Examination of Water and Wastewater. APHA Press. <https://doi.org/10.2105/SMWW.2882.194>

Standard Methods Committee of the American Public Health Association, American Water Works Association, & Water Environment Federation. (2018a). 2550 TEMPERATURE. In Lipps WC (Ed.), Standard Methods For the Examination of Water and Wastewater (23rd Ed.).

<https://doi.org/10.2105/SMWW.2882.031>

Standard Methods Committee of the American Public Health Association, American Water Works Association, & Water Environment Federation. (2018b). 4500-H+ pH. In W. Lipps, T. Baxter, & E. Braun-Howland (Eds.), Standard Methods For the Examination of Water and Wastewater. APHA Press.

<https://doi.org/10.2105/SMWW.2882.082>

Standard Methods Committee of the American Public Health Association, American Water Works Association, & Water Environment Federation. (2018c). 4500-O OXYGEN (DISSOLVED). In Standard Methods For the Examination of Water and Wastewater. <https://doi.org/10.2105/SMWW.2882.091>

Standard Methods Committee of the American Public Health Association, American Water Works Association, & Water Environment Federation. (2018d). 9222 Membrane Filter Technique for Members of the Coliform Group. In Standard Methods For the Examination of Water and Wastewater.

<https://doi.org/10.2105/SMWW.2882.193>

ThermoFisher Scientific. (2014). Measuring pH in Low Ionic Strength Solutions (AN-PHLOWION-E 0914 REVA; Application Note 006).

ThermoFisher Scientific. (2022). Measuring pH in surface water Application Note AN008.

<https://www.thermofisher.com/document-connect/document-connect.html?url=https://assets.thermofisher.com/TFS-Assets%2FSLSG%2FApplication-Notes%2FAN-PHSFCWTR-E%201014-RevA-WEB.pdf>

U.S. Environmental Protection Agency. (1978). Microbiological Methods for Monitoring the Environment EPA-600/8-78-017. <https://nepis.epa.gov/Exe/ZyPURL.cgi?Dockey=300014TD.txt>

U.S. Environmental Protection Agency. (1982a). Method 150.2: pH, Continuous Monitoring (Electrometric) by pH Meter. https://www.epa.gov/sites/default/files/2015-08/documents/method_150-2_1982.pdf

U.S. Environmental Protection Agency. (1982b). Method 150.2: pH, Continuous Monitoring (Electrometric) by pH meter. https://www.epa.gov/sites/default/files/2015-08/documents/method_150-2_1982.pdf

U.S. Environmental Protection Agency. (2003). IDEXX Summary 5AP-v2: U.S. EPA Approval of Colilert*, Colilert-18*, Enterolert*, Quanti-Tray*, QuantiTray*/2000 for Ambient Water Testing. <https://www.idexx.com/files/water-reg-article5AP-v2.pdf>

U.S. Environmental Protection Agency. (2007). Guidelines Establishing Test Procedures for the Analysis of Pollutants; Analytical Methods for the Biological Pollutants in Wastewater and Sewage Sludge; Final Rule. <https://www.idexx.com/files/water-reg-article5BA.pdf>

U.S. Environmental Protection Agency. (2014a). Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl- β -D-Glucoside Agar (mEI) [EPA 821-R-14-011]. https://www.epa.gov/sites/default/files/2018-06/documents/method_1600_sept-2014.pdf

U.S. Environmental Protection Agency. (2014b). Method 1603: Escherichia coli (E. coli) in Water by Membrane Filtration Using Modified membrane-Thermotolerant Escherichia coli Agar (Modified mTEC).

U.S. Environmental Protection Agency. (2016). Best Practices for Continuous Monitoring of Temperature and Flow in Wadeable Streams (EPA/600/R-13/170F). https://cfpub.epa.gov/si/si_public_record_Report.cfm?Lab=NCEA&dirEntryId=280013

40 CFR 136.3 (July 19, 2021), (2021).

U.S. Environmental Protection Agency. (2021, July 19). 40 CFR 136.3 (Accessed via eCFR). <https://www.ecfr.gov/current/title-40/chapter-I/subchapter-D/part-136/section-136.3>

USGS. (1987). Chapter A4 METHODS FOR COLLECTION AND ANALYSIS OF AQUATIC BIOLOGICAL AND MICROBIOLOGICAL SAMPLES. https://pubs.usgs.gov/twri/twri5a4/pdf/TWRI_5-A4.pdf

USGS. (1989a). Chapter A1 Methods for Determination of Inorganic Substances in Water and Fluvial Sediments. In Techniques of Water-Resources Investigations of the United States Geological Survey (pp. 363–364). U.S. Geological Survey. <https://pubs.water.usgs.gov/TWRI5A1/>

USGS. (1989b). Chapter A1 Methods for Determination of Inorganic Substances in Water and Fluvial Sediments. In 5 LABORATORY ANALYSIS. <https://doi.org/https://doi.org/10.3133/tm9A6.2>

USGS. (2020). Dissolved Oxygen Chapter 6.2 of Section A, National Field Manual for the Collection of Water-Quality Data. <https://doi.org/https://doi.org/10.3133/tm9A6.2>

USGS. (2021). Measurement of pH. In Techniques and Methods (Vol. 9). <https://doi.org/https://doi.org/10.3133/tm9A6.4>

Wilde, F. D. (2006). Chapter A6. Section 6.1. Temperature. In Techniques of Water-Resources Investigations (Vol. 9). USGS. <https://doi.org/https://doi.org/10.3133/twri09A6.1>

YSI. (2015). The pH Handbook a practical guide to pH measurement.

Version History

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|-------------------------------------|--|
| Version 1.0, Published July 1, 2022 | First Version |
| Version 2.0, Published Feb 12, 2024 | <ul style="list-style-type: none">• Added Quality Assurance and Low Ionic Strength sections to pH – Grab Sampling• Added Dissolved Oxygen section• Updated references, links• Added version history |