

**DWR–NPDES–SOP–G–13–Oxygen, Dissolved 4500-O G/H-01012024  
Oxygen (Dissolved), SM 4500-O G (2016) Membrane-Electrode Method &  
SM 4500-O H (2016) Optical-Probe Method**

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**1) EFFECTIVE DATE: 01/01/2024**

**2) SIGNATURES:**

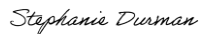


April Grippo (May 30, 2024 16:25 CDT)

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Drafter / Preparer



Stephanie Durman, Deputy General Counsel  
Reviewer

***Initial Demonstration of Capability (DOC)***

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- Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.
- Calibrate the probe daily according to manufacturer’s instructions.
- Follow Hach Method 10360 9.2.1 – Prepare and measure four samples of air-saturated water according to section 7.2.
  - 7.2.1 – Add approximately 1500 mL of organic-free water or BOD dilution water to a 2-L beaker or PET bottle.
  - 7.2.2 – Allow the water to equilibrate to room temperature. Room temperature should be approximately  $20 \pm 3^{\circ}\text{C}$ .
  - 7.2.3 – With a steady gentle stream of filtered air ( $\approx 10\text{-}40$  mL per minute), aerate the water for a minimum of 30 minutes. Alternatively, vigorously shake the reagent water or BOD dilution water for several minutes.
  - 7.2.4 – At the completion of aeration, let water re-equilibrate to room temperature ( $20 \pm 3^{\circ}\text{C}$ ) for 30 minutes and note the barometric pressure of the laboratory during preparation. The barometric pressure reading is used in the calculation and determination of the theoretical DO concentration for the preparation of air-saturated water.
  - 7.2.5 – Transfer the aerated water to a BOD bottle until overflowing and stopper.
  - 7.2.6 – Calculate the theoretical dissolved oxygen concentration using a dissolved oxygen table such as Hitchman.
- **Summary: each analyst should calibrate the probe, prepare dilution water that is air-saturated, and analyze four bottles and compare to the theoretical dissolved oxygen concentration ( $\pm 0.2$  mg/L).**
  - **Theoretical dissolved oxygen can be found at USGS’s website at <http://water.usgs.gov/software/DOTABLES/> or by using a DO Saturation Table.**

**Method Detection Limit (MDL)**

- None

**Initial Calibration Verification (ICV)**

- 1020 B.11.b. – Perform initial calibration using at least three concentrations of standards for linear curves.
- **Summary: calibrate daily by following manufacturer’s instructions.**

**Method Blank**

- NONE

**Laboratory Fortified Blank (LFB)**

- NONE

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**Duplicate**

- 1020 B.12.f. – Calculate RPD (relative percent difference).
- 4020 B.2.f. – Randomly select routine samples to be analyzed twice.
  - Process duplicate sample independently through the entire sample preparation and analysis.
  - Include at least one duplicate for each matrix type daily or with each batch of 20 or fewer samples.
- **Summary: on a 5% basis (one for every 20 samples or once per month, whichever is more frequent) analyze two samples for DO.**
  - **First sample is result, second sample is duplicate.**
  - **Target value is to get close to the first value and have a small RPD (less than 20%).**

**Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD)**

- NONE

**Continuing Calibration Verification (CCV)**

- Follow Hach Method 7.2.1 – Add approximately 1500 mL of organic-free water or BOD dilution water to a 2-L beaker or PET bottle.
- 7.2.2 – Allow the water to equilibrate to room temperature. Room temperature should be approximately  $20 \pm 3^{\circ}\text{C}$ .
- 7.2.3 – With a steady gentle stream of filtered air ( $\approx 10\text{-}40$  mL per minute), aerate the water for a minimum of 30 minutes. Alternatively, vigorously shake the reagent water or BOD dilution water for several minutes.
- 7.2.4 – At the completion of aeration, let water re-equilibrate to room temperature ( $20 \pm 3^{\circ}\text{C}$ ) for 30 minutes and note the barometric pressure of the laboratory during preparation. The barometric pressure reading is used in the calculation and determination of the theoretical DO concentration for the preparation of air-saturated water.
- 7.2.5 – Transfer the aerated water to a BOD bottle until overflowing and stopper.
- 7.2.6 – Calculate the theoretical dissolved oxygen concentration using a dissolved oxygen table such as Hitchman.
- 9.3.1 – Upon air calibration, prepare a calibration verification standard with each analytical batch of 20 samples or less in an 8-hour period.
- 9.4.3 – Initially and at the end of each analytical batch of samples, analyze a dilution water sample that is air-saturated.
- **Summary: prepare dilution water that is air-saturated and analyze bottles and compare to the theoretical dissolved oxygen concentration ( $\pm 0.2$  mg/L).**
  - **Theoretical dissolved oxygen can be found at USGS’s website at <http://water.usgs.gov/software/DOTABLES/> or by using a DO Saturation Table.**

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**Control Charts**

- 1020 B.13.b. – The precision chart is also constructed from the average and standard deviation of a specified number of measurements [e.g., %RSD or relative percent difference (RPD)] for replicate or duplicate analyses of the analyte of interest. Perfect agreement between replicates or duplicates results in a difference of zero when the values are subtracted, so the baseline of the chart is zero. Therefore, for precision charts, only upper WLs and upper CLs are meaningful.
- **Summary: Create and maintain control charts once you have 20-30 data points.**

**Corrective Action** - 1020 B.5., B.8., & B.15.

**Batch Size**

- For samples that need to be analyzed on a 5% basis (1 for every 20 samples or once per month, whichever is more frequent) follow these criteria:
  - If a permit stated that 3 analyses per week, we would allow for a duplicate to be analyzed at least once per month.
    - Pick a date and be consistent, the 1<sup>st</sup> of every month or the 1<sup>st</sup> Thursday of every month. Mark your calendar!!
  - If a permit stated 5 analyses per week, we would suggest twice a month.
    - Pick a date and be consistent, the 1<sup>st</sup> and 15<sup>th</sup> of every month or the 1<sup>st</sup> and 3<sup>rd</sup> Thursday of every month. Mark your calendar!!

Revision Number	Date	Brief Summary of Change
0	November 2013	Initial issuance of the Guidance
1	July 5, 2018	Method editorial revision date updated from 2001 to 2011
2	December 2021	Method editorial revision date updated from 2011 to 2016
3	December 11, 2023	Revised Summary, added SM 4500-O H (2016) information