

Biochemical Oxygen Demand (BOD), SM 5210 B, 22nd edition (2001) – 5-Day BOD Test

Initial Demonstration of Capability (DOC)

- 1020 B. 1 – As a minimum, include a reagent blank and at least 4 LFBs
- 4020 B.1.a. – Before analysts run any samples, verify their capability with the method. Run a laboratory fortified blank at least four times and compare to the limits listed in the method.
 - If no limit is specified, use the following procedure to establish limits.
 - Calculate the standard deviation of the four samples. Then calculate the LFB's recovery limits (see formula at end)
- **Real people language – each operator running this test needs to analyze 4 samples of GGA at a concentration of 198 ± 30.5 mg/L**
 - **Keep a folder for each analyst, keep a copy here**
 - **Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.**
 - **Recommend backup analyst do this once a year.**

Method Detection Limit (MDL)

- NONE

Initial Calibration Verification (ICV)

- 1020 B.11.b. – Perform initial calibration
- Hach's Method: 7.1.1 – Add approximately 1 inch (2.54 cm) of reagent water to a clean BOD bottle and stopper).
- 7.1.2 – Shake vigorously for ~ 10 seconds.
- 7.1.3 – Allow for the BOD bottle and its contents to equilibrate to room temperature. Room temperature should be approximately $20 \pm 3^{\circ}\text{C}$.
- 7.1.4 – The stopper may now be removed from the BOD bottle and the LBOD probe inserted for calibration purposes.
- **Real people language – calibrate DO probe **daily** (day of) by following manufacturer's instructions.**

Method Blank

- 1020 B.5.– A reagent blank (method blank) consists of reagent water and all reagents that normally are in contact with a sample during the entire analytical procedure.
- 5020 B.2.d. – Include at least one method blank daily or with each batch of 20 or fewer samples, whichever is more frequent.
- 5210 B.6.c. – With each batch of samples, incubate one or more bottles of dilution water that contains nutrient, mineral and buffer solutions but no seed or nitrification inhibitor.
 - The DO uptake in 5 days must not be more than 0.20 mg/L and preferably not more than 0.10 mg/L, before making seed corrections.
- **Real people language – analyze dilution water blanks **daily** (day of), preferably one at beginning and one at end**
 - **Target value is less than 0.20 mg/L (preferably less than 0.10 mg/L)**

Laboratory Fortified Blank (LFB)

- 1020 B.6.– A laboratory-fortified blank is a reagent water sample to which a known concentration of the analyte of interest has been added.
 - Sample batch = 5% basis
- 5020 B.2.e. – Calculate percent recovery, plot control charts and determine control limits (see Control Charts below)
 - When appropriate, include at least one LFB daily or per each batch of 20 or fewer samples.
- **Real people language – analyze GGA sample at a concentration of 198 ±30.5 mg/L**
 - **Run on a 5% basis (see batch size for more information).**
 - **If permit requires cBOD, add nitrification inhibitor (NI) to one GGA bottle once/quarter (or more often if the Lot # of NI changes), which should be equal to 164 ±30.7 mg/L**

Duplicate

- 1020 B.12.f. – Calculate RPD (relative percent difference)
- 5020 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.
- **Real people language – analyze 2 samples for BOD or cBOD, run an extra sample dilution bottle**
 - **Example: if you run 100mL, 200mL and 300mL for your effluent, run a second 300mL sample. You will have 4 bottles total for your effluent dilutions.**
 - **Target value should be close to the first value (same dilution) and have a small RPD (less than 20%)**
- **For reporting purposes, average results that meet method criteria.**

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

- NONE

Continuing Calibration Verification (CCV)

- Hach Method 10360 7.2 and 9.4 – Calibration Verification for membranes and LDO probes
 - 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 ± 3°C.
 - 7.2.3 – With a steady gentle stream of filtered air (≈ 10-40 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 ± 3°C) for 30 minutes and note the barometric pressure of the laboratory during preparation. The barometric temperature 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...
- 9.4.3 – Initially and at the end of each analytical batch of samples, analyze a dilution water sample that is air-saturated
- **Real people language – prepare dilution water that is air-saturated and analyze bottles and compare to the theoretical dissolved oxygen concentration (± 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.**

Control Charts – 1020 B.13.

- **Real people language**
 - **Create and maintain control charts if you have 20-30 data points within 90 days.**
 - **If you do not meet the above criteria, follow QC Acceptance Criteria below.**

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

- 5210 B.7.b. – Identify results in the test reports when any of the following quality control parameters is not met:
 - Dilution water exceeds 0.20 mg/L (5210B.6c)
 - Glucose-glutamic acid check falls outside of acceptable limits (5210B.6b)
 - Test replicates show more than 30% difference between high and low values
 - Seed control samples do not meet the above criteria in all dilutions (5210B.6d) or
 - Minimum DO is less than 1.0 mg/L (5210B.7a3)

QC Acceptance Criteria

- Blanks < 0.20 mg/L
- GGA = 198 ± 30.5 mg/L (if running cBOD, add NI to one bottle once/quarter or more often if NI Lot# changes, and it should = 164 ± 30.7 mg/L)
- RPD < 20%
- Minimum of three dilutions for each sample, at least one sample must have valid data with at least 2.0 mg/L depletion and a residual of 1.0 mg/L

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:
- Influent and Effluent are 2 different samples
 - If a permit stated that 3 analyses per week, that would be 6 samples per week, we would allow for a duplicate to be analyzed at least twice a month.
 - Pick a date and be consistent, the 1st and 15th of every month or the 1st and 3rd Thursday of every month. Mark your calendar!!
 - If a permit stated 5 analyses per week, that would be 10 samples per week, we would allow once a week.
 - Pick a date and be consistent, every Monday. Mark your calendar!!

- If sampling only once a month, need to run QC once a month.

Calculations

- % Recovery for LFB
 - = $\frac{\text{LFB concentration}}{\text{Expected concentration}} \times 100\%$
- RPD – relative percent differences for duplicates
 - = $\frac{\text{Difference between sample and duplicate}}{\text{Average of the sample and duplicate}} \times 100\%$
- Unseeded - BOD₅, mg/L = $\frac{D_1 - D_2}{P}$
- Seeded - BOD₅, mg/L = $\frac{(D_1 - D_2) - (B_1 - B_2)f}{P}$
 - Where:
 - D₁ = Initial Dissolved Oxygen Concentration in Sample, mg/L
 - D₂ = Final Dissolved Oxygen Concentration in Sample, mg/L
 - B₁ = Initial Dissolved Oxygen Concentration in Seed Control, mg/L
 - B₂ = Final Dissolved Oxygen Concentration in Seed Control, mg/L
 - P = Sample Concentration, % (expressed as a decimal)
 - f = $\frac{\text{Seed in Sample, \%}}{\text{Seed in Seed Control, \%}}$