Neutralization of Total Residual Chlorine in BOD_5/$CBOD_5$ Samples Policy

(NC WW/GW LCB 08/06/2021)

It is acceptable to screen samples with DPD powder for the presence of Total Residual Chlorine (use pillows appropriate for the volume of sample tested). If pink color is observed, chlorine is present. Ultra-low type Total Residual Chlorine test strips capable of detecting ≤0.1 mg/L may also be used. If chlorine is present, the titration procedure outlined below must be used to determine the proper amount of Sodium Sulfite needed to neutralize chlorine in the sample. If chlorine is absent, document and proceed to set sample.

Chemicals:

1. Sodium Sulfite solution - Dissolve 0.1575 g Na_2SO_3 in 100 mL distilled water. Prepare fresh daily.
2. 2% H_2SO_4 - Add 2 mL concentrated H_2SO_4 to 100 mL distilled water. 10 mL of 1:1 acetic acid may be substituted for the H_2SO_4 solution.
3. Potassium Iodide (KI) solution - Add 10 g KI to 100 mL distilled water. Initially this solution will be clear, but in a few days it will turn greenish yellow. That's ok.
4. Starch - Commercially available or use either an aqueous solution or soluble starch powder mixture. To prepare an aqueous solution, dissolve 2 g laboratory-grade soluble starch and 0.2 g salicyclic acid (as a preservative) in 100 mL hot distilled water.

Procedure:

To 100 mL of sample, add approximately 1 mL of 2% H_2SO_4, 1 mL KI solution, and 1 mL starch.

If the solution remains clear, no chlorine is present. Document this on the bench sheet and proceed to set up the sample for BOD analysis.

If the solution turns blue, chlorine is present. Add the Sodium Sulfite solution, drop by drop, while stirring the sample, until the sample is clear again. Count the drops of Sodium Sulfite solution needed to neutralize the 100 mL sample. Add the relative volume of Sodium Sulfite solution to the volume of sample needed. For example, if it took 6 drops to neutralize the 100 mL sample volume and you need 300 mL of sample to set the dilutions you want, add 18 drops of Sodium Sulfite solution to 300 mL of sample. Document this on the bench sheet. Wait about 10 to 20 minutes and recheck the sample to verify the chlorine has been neutralized. Proceed to set up the sample.

Note: If the blue color returns after a few seconds, do not add more Sodium Sulfite solution. Recheck with DPD to verify that returning blue color is not caused by chlorine still in sample. If no chlorine is still present, add amount of Sodium Sulfite solution equivalent to when blue color first disappears. Adding an excessive amount of Sodium Sulfite solution creates an oxygen demand and will result in a false high BOD value.