# ESTIMATION OF DISSOLVED OXYGEN

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Dissolved oxygen (DO) levels in environmental water depend on the physiochemical and biochemical activities in water body and it is an important useful in pollution and waste treatment process control.
Two methods are commonly used to determine DO concentration:

(1) The iodometric method which is a titration-based method and depends on oxidizing property of DO

(2) The membrane electrode procedure, which works based on the rate of diffusion of molecular oxygen across a membrane. In the Iodometric method, divalent manganese solution is added to the solution, followed by addition of strong alkali in a glass-stopper bottle. DO rapidly oxidize an equivalent amount of the dispersed divalent manganese hydroxide precipitates to hydroxides of higher valence states. In the presence of iodide ions in an acidic solution, the oxidized manganese reverts to the divalent state, with the liberation of iodine equivalent of the original DO content. The iodine is then titrated with a stranded solution of thiosulfate. The titration end point can be detected visually with a starch indicator. Some oxidizing and reducing agents present in solution can interfere with the iodometric method. Oxidizing agents liberate iodine from iodides (positive interference) and some reducing agents reduce iodine to iodide (negative interference).

Collection of Samples for DO Determination Samplers are designed to ensure that air cannot enter into the sample. Most samplers are designed

to retain 3-4 times the volume of samples which is required for analysis purposes. As oxygen values change with time due to any biological activity, it is important to fix it in field immediately after collection. This is done using reagents using in DO test and then the titration is done in laboratory. This method gives low results for samples with high iodine demand and in this case it is better to preserve sample using 0.7 mL concentrated sulfuric acid and 0.02 g sodium azide. When this is doe it is necessary to add 3 mL of alkali-iodide reagent rather than the usual 2 mL because of the extra acid the sample contains. Better results are also obtained if the sample is fixed and stored in the dark and on the ice until the analyses are conducted. The final titration should not be delayed more than 6 hours.

Reagents:

1. Manganese sulfate solution: Dissolve 480 g $MnSO_4.4H_2O$, 400 g $MnSO_4.2H_2O$ or 364 g $MnSO_4.H_2O$ in distilled water, filter, and dilute to 1L. The $MnSO_4$ solution should not give a color with starch when added to an acidified potassium iodide (KI) solution.

2. Alkali-iodide-azide reagent

3. Sulfuric acid: One mL is equivalent to ~ 3mL alkali-iodide-azide reagent.

4. Starch solution: Dissolve 2 g laboratory-grade soluble starch and 0.2 g salicyclic acid as preservative in 100 mL hot distilled water.

5. Standard sodium thiosulfate titrant: Dissolve 6.205 g $Na_2S_2O_3 .5H_2O$ in distiller water and add 1.5 mL 6N NaOH or 0.4 g solid NaOH and dilute to 1000 mL. Standardize with biiodate solution.

6. Standard potassium bi-iodate solution (0.0021M): Dissolve 812.4 mg KH(IO3) in distilled water and dilute to 1000 mL.

7. Standardization: Dissolve e ~ 2 g KI, free from iodate in an Erlenmeyer flask with 100 to 150 mL distilled water; add 1 mL 6N H2SO4 or a few drops of conc. H2SO4 and 20.00 mL standard bi-iodate solution. Dilute to 200 mL and titrate librated iodine with thiosulfate titrant, adding starch toward end of titration, when a pale straw color is reached. When the solution is of equal, 20.00 mL 0.025M Na2S2O3 should be required. If not, adjust the Na2S2O3 solution to 0.025M.

Apparatus: Incubation bottle 300mL volume; Air compressor

Steps:

1. Make dilution water by adding 2mL/L of following reagents in distilled water:
a. Phosphate buffer solution
b. Magnesium sulfate solution
c. Calcium chloride solution
d. Ferric chloride solution
e. Sodium Sulfite solution

2. Take 300 mL sample in BOD bottle. Prepare two sets of this sample. Keep one set for DO analysis for day 0 (i.e., Sample0Day) and another sample in BOD incubator for 5 days at 20°C (Sample5Day) (this is how 5-day BOD experiment is done). Here you will prepare duplicate samples and analyze at Day 0 (i.e., Sample 0 Day_A and Sample 0 Day_B).

3. For a given sample bottle, add 1 mL of alkali azide and then 1 mL manganous sulfate solution. Shake well the bottle and keep it open for 5 minutes to settle the precipitate. Add 2 mL concentrated $H_2SO_4$ and place the cap on the bottle. Shake well the bottle till all the precipitate is dissolved.

4. Take 203 mL of sample in conical flask and titrate with standard sodium thiosulfate solution (0.025N) till the colour changes from dark yellow to light yellow. Then add few drops of starch indicator and continue to titrate till the color of the solution becomes either colorless or changes to its original sample colour. Note down volume of 0.025N sodium thiosulfate consumed.

5. Calculate DO value of the sample. Remember that in 200 mL sample, 1 mL of sodium thiosulfate of 0.025N equals to 1 mg/L dissolved oxygen: =>Dissolved oxygen (DO) (in mg/L) = mL of sodium thiosulfate (0.025N) consumed.

Dilution of Sample

1. 0.1, 0.5, and 1% for strong waste water
2. 1.0, 2.5, and 5% for raw and settled sewage
3. 5.0, 12.5 and 25% for oxidized effluent
4. 25, 50 and 100% for polluted river water