ENVIRONMENTAL CHEMISTRY LABORATORY MANUAL

1. Standard Solutions, pH, conductivity, ionic strength

Group 1	Group 2	Group 3	Group 4
Prepare: 1 N NaOH, 100 mL 1 N H_2SO_4, 100 mL 10% (mass/mass) salt solution, 100 mL ***Dilute and prepare***: 0.02 N NaOH, 250 mL 0.02 N H_2SO_4, 250 mL ***Measure***: pH, conductivity, ionic strength of each solutions	***Prepare***: 1 N NaOH, 50 mL 1 N H_2SO_4, 50 mL 25% (mass/mass) salt solution, 100 mL ***Dilute and prepare***: 0.02 N NaOH, 100 mL 0.02 N H_2SO_4, 100 mL ***Measure***: pH, conductivity, ionic strength of each solutions	***Prepare***: 2 N NaOH, 100 mL 2 N H_2SO_4, 100 mL 50% (mass/mass) salt solution, 100 mL ***Dilute and prepare***: 0.02 N NaOH, 100 mL 0.02 N H_2SO_4, 100 mL ***Measure***: pH, conductivity, ionic strength of each solutions	***Prepare***: 2 N NaOH, 50 mL 2 N H_2SO_4, 50 mL 35% (mass/mass) salt solution, 100 mL ***Dilute and prepare***: 0.02 N NaOH, 50 mL 0.02 N H_2SO_4, 50 mL ***Measure***: pH, conductivity, ionic strength of each solutions

2. Acidity Test

- Measure 50 mL of sample using graduated cylinder and pour into an erlen
- Add 2-3 drops of methyl-orange indicator reagent
- Measure the pH value using a pH-meter
- Titrate the solution using 0.02 N NaOH and finish the titration when methyl orange turning point is observed.
- Repeat the steps using phenolftalein indicator reagent.
- Calculate the acidity types using the formula below.

$$Acidity\ (mg\ CaCO_3 / L) = \frac{V_{NaOH}\ x\ N_{NaOH}\ x\ 50000}{V_{sample}}$$

3. Alkalinity Test

- Take 50 mL of sample
- Add 2 – 3 drops of phenol ftalein indicator reagent
- Titrate the sample with 0.02 N H_2SO_4 solution till the turning point.
- Add 2 – 3 drops of methyl ftalein indicator reagent.
- Titrate the sample with 0.02 N H_2SO_4 solution till the turning point.
- Calculate the alkalinity types using alkalinity dominant species table and the formula below.

Volume	Dominant alkalinity type	Concentration (mg $CaCO_3$ /L)
$V_{PP} = 0$	HCO_3^-	$[HCO_3^-] = V_{mo} * N * 50000 / V_{sample}$
$V_{MO} = 0$	OH^-	$[OH^-] = V_p * N * 50000 / V_{sample}$
$V_{PP} = V_{MO}$	CO_3^{-2}	$[CO_3^{-2}] = V_p * N * 50000 / V_{sample}$
$V_{MO} > V_{PP}$	CO_3^{-2} & HCO_3^-	$[CO_3^{-2}] = V_p * N * 50000 / V_{sample}$ $[HCO_3^-] = (V_{mo} - V_p) * N * 50000 / V_{sample}$
$V_{MO} < V_{PP}$	OH^- & CO_3^{-2}	$[CO_3^{-2}] = V_{mo} * N * 50000 / V_{sample}$ $[OH^-] = (V_p - V_{mo}) * N * 50000 / V_{sample}$

$$Alkalinity\ (mg\ CaCO_3 / L) = \frac{V_{H2SO4}\ x\ N_{H2SO4}\ x\ 50000}{V_{sample}}$$

$$\mathbf{Alkalinity = [HCO_3^-] + 2[CO_3^{2-}] + [OH^-] - [H^+]}$$

4. Turbidity and Color Test

Turbidity Test:

- Homogenize (mix well) the sample, and add sample into the turbidimeter tube (25±1 ml).
- Wipe out the tube and place it into the turbidimeter, and close the lid.
- Read the value from the digital screen.

If turbidity value is > 1000 NTU:

- Homogenize the sample, and dilute the sample (For ex. 50%, 75% dilution).
- Add sample into the turbidimeter tube, place it into the turbidimeter and read the value.
- Multiply the value with dilution factor.

Color Test:

- Read the absorbance values of the colored standard solutions using a UV spectrophotometer.
- Draw a calibration graph using the measured points on the graph and generate the equation.
- Read the absorbance values of the unknown samples with spectrophotometer.
- Show the sample results on the graph and calculate the concentrations of the samples using the equation

5. Solid Matters Test

Total Solids (TS) Method

- Dry crucible at 103–105°C for 1 h and put it in a desiccator
- Weigh the crucible (A).
- Put a 50 mL of homogenized sample.
- Dry the sample at 103–105°C (usually it takes >3 h) and then put it in a desiccator.
- Weigh the sample + crucible (B).

TS (mg/L) = (B - A) / Vsample

B = Weight of crucible + sample (mg, g, kg, …)
A = Crucible (mg, g, kg, …) Vsample = mL, L

Total Suspended Solids (TSS) Method

- Dry the crucible + filter paper at 103–105°C for 1 h and put it in a desiccator
- Weigh the crucible + FP (A).
- Filter a 100 mL of homogenized sample.
- Dry the crucible+FP at 103–105°C for 1 h and then put it in a desiccator.
- Weigh the sample + crucible + FP (B).

TSS (mg/L) = (B - A) / Vsample

B = Weight of crucible + filter paper + sample (mg, g, kg, …)
A = Crucible + filter paper (mg, g, kg, …) Vsample = mL, L, m^3

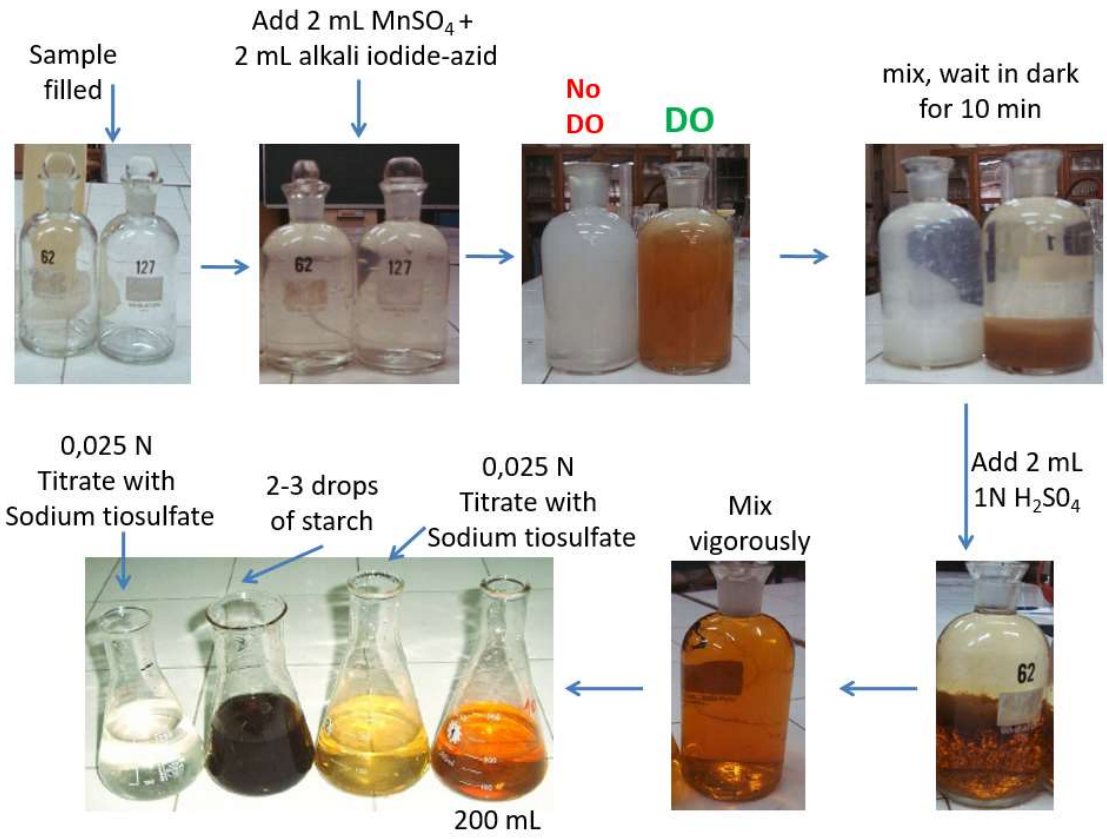
Total Dissolved Solids (TDS) = TS – TSS

6. Hardness Test

- Take 50 mL of water sample (or take a sample which would consume <15 mL of EDTA and dilute to 50 mL).
- Add 1 mL of buffer solution to increase pH to 10,0 ± 0,1 (and finalize the titration within 5 minutes).
- Add a very small amount of eriochrome black T (EBT) indicator reagent (Color of the sample should turn pink).
- Titrate the sample with EDTA till the color turns from pink to blue.
- Calculate the hardness using the formula given below

$$Hardness(mgCaCO3/mL = \frac{V_{EDTA} \times N_{EDTA} \times 50000}{Vs(mL)}$$

7. Dissolved Oxygen and Biochemical Oxygen Demand Test



- Apply dilution or add dilution solution to the sample and note the dilution factor.
- Fill two BOD bottles with sample (or diluted sample) and store one of them in the incubator immediately.
- Add 2 mL $MnSO_4$ and 2 mL alkali iodide azid solutions into the BOD bottle, put BOD bottle for 10 min in dark closet.
- Add 2 mL, 1 N H_2SO_4 into the bottle and mix it vigorously.
- Take 200 mL of sample into an erlene and start titration with 0.025 N sodium thiosulfate till light yellow color.
- Add 2-3 drops of starch and finalize titration when you reach the color of the sample.
- Repeat the procedure on the 5th day and calculate the BOD_5 and ultimate BOD of the sample using the formula below.

- Unseeded: $\mathrm{BOD}_5 = \dfrac{(D_0 - D_5)}{P}$
- Seeded: $\mathrm{BOD}_5 = \dfrac{(D_0 - D_5) - (B_0 - B_5)f}{P}$

$$\mathbf{BOD_t = L_U \cdot (1 - e^{-k.t})}$$

D_0 is the dissolved oxygen (DO) of the diluted solution after preparation (mg/l)

D_5 is the DO of the diluted solution after 5 day incubation (mg/l)

P is the decimal dilution factor

B_0 is the DO of diluted seed sample after preparation (mg/l)

B_5 is the DO of diluted seed sample after 5 day incubation (mg/l)

f is the ratio of seed volume in dilution solution to seed volume in BOD test on seed

8. Chemical Oxygen Demand Test

- 20 mL of sample (and 20 mL of blank) is filled into COD bottles (volumetric flasks).
    - If sample is highly polluted, then the sample should be diluted with distilled water.
- A few number of boiling stones and 0.4 g of HgSO4 (mercury sulfate) are added.
    - $HgSO_4$ is added to prevent the interference of Cl.
- 5 mL of $Ag_2SO_4.H_2SO_4$ (silver-sulphuric acid) is added and $HgSO_4$ is dissolved.
- 10 mL of standard $K_2Cr_2O_7$ is added and mixed.
- COD bottles are placed into reflux system, and cooling water is started to run in the system.
- 25 mL of $Ag_2SO_4.H_2SO_4$ is added from the top of the reflux system.
- Samples and blank are boiled for 2 hours in reflux system
- After boiling for 2 hrs, add 60 mL of distilled water into COD bottles and cool it till room temperature.
- After cooling, add 2–3 drops of ferroin indicator reagent.
- Samples and blank are titrated with standard iron ammonium sulfate (IAS) till the color changed from blue-green to dark red.
- Calculate the COD of the sample using the formula below.

$$\text{COD (mg/L)} = \frac{(B - S) \times N \times 8000}{V_{sample}\ (mL)}$$

B: Volume of IAS spent for blank (mL)

S: Volume of IAS spent for sample (mL)

N: Normality of IAS