Department of

# Civil Engineering

**LAB MANUAL**

**Environment Engineering-2 LAB**

## B.Tech– VI Semester



## KCT College OF ENGG AND TECH.

## VILLAGE FATEHGARH

## DISTT.SANGRUR

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# Experiment-1

# ACID-BASE TITRATION

**OBJECTIVE:** To determine the normality of Hydrochloric acid present in given solution using 0.05N Standard solution of sodium carbonate.

**APPARATUS**: 1. Burette 2. Burette Stand 3. Pipette 4. Conical Flask
5 Beakers 6. Glazed tile 7.Wash bottle

**CHEMICALS** : 1. Hydrochloric acid solution 2. Distilled water
3. Standard Sodium carbonate solution 4. Methylorange indicator
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**PRINCIPLE** : Hydrochloric acid reacts with Sodium carbonate solution according to the following equation . This is known as neutralization reaction

$$Na_2CO_3 + 2HCl \ 2NaCl + H_2O + CO_2$$

Methyl orange may be used as an indicator, since it is not affected by the liberated carbon dioxide gas.

**PROCEDURE:**

1. The burette is rinsed with tap water, then with distilled water finally with the given Hydrochloric acid solution. The burette is filled with Hydrochloric acid and the nozzle portion is also completely filled with the solution with out any air bubbles in it. The initial reading of the burette is adjusted to ' 0'. The burette is clamped vertically to a burette stand.

2. A 20ml pipette is taken. It is rinsed with tap water, then with distilled water and finally with the given sodium carbonate solution. 20ml of Sodium Carbonate is transferred into a clean Conical flask by means of a Pipette.

3. 1 or 2 drops of methyl orange are added to the solution. The solution turns yellow in colour.

4. The flask is placed under the burette on a glazed tile. The HCl is added slowly while shaking the flask. The addition is continued till the colour changes from yellow to pink. It is the 'End point'. Just before the end point, any drops of the solution adhering to inner walls of the flask are washed down into the flask with a few drops of distilled water.

5. The final reading of the burette is noted. The difference between the two readings gives the volumes of Hydrochloric acid rundown. The contents of flask are thrown away.

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6. Again 20 ml sodium carbonate solution is transferred to the flask and is titrated in a similar way. The titration are repeated till two consecutive readings coincide. The readings are entered in a Tabular form.
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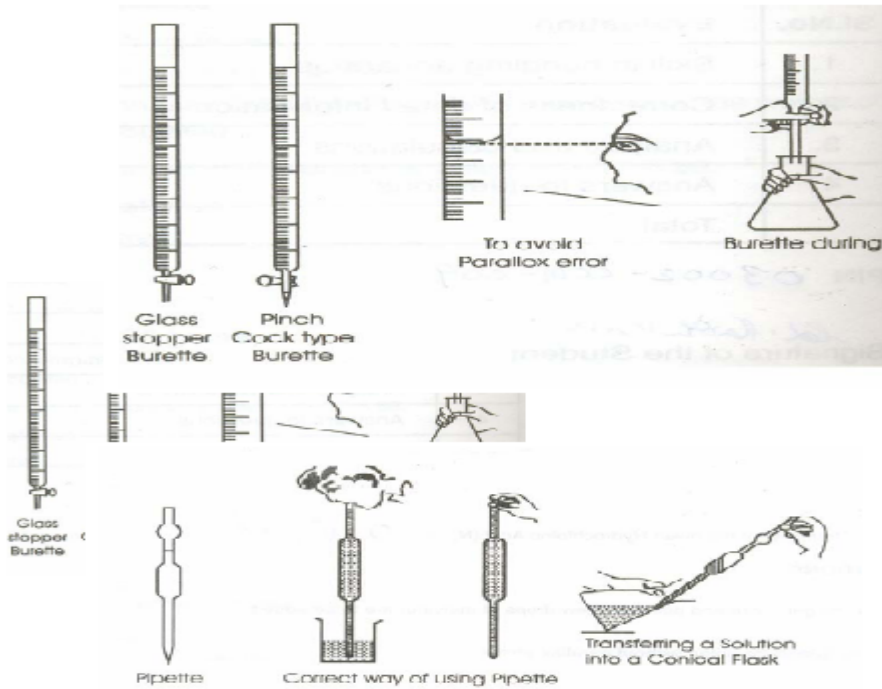
## OBSERVATIONS:

**Titration of HCI Using Standard $Na_2CO_3$ Solution**
Burette : HCI Solution
Conical Flask : 20 ml of Std $Na_2CO_3$+ Methyle orange 1 or 2 drops
End Point : Yellow to pink

| SI. No | Volume of $Na_2CO_3$ Solution In ml '$V_2$' | Burette Readings | | Volumes of HCI rundown. |
|---|---|---|---|---|
| | | Initial | Final | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

## CALCULATIONS :

Where: $V_1$ = Volume of Hydrochloric acid
$N_1$ = Normality of Hydrochloric acid
$V_2$ = Volume of sodium Carbonate solution
$N_2$ = Normality of sodium Carbonate Solution
$$N_1 = \frac{N_2 \times V_2}{V_1}$$

## RESULT

The Normality of the given Hydrochlolric Acid ( $N_1$ ) =

## PRECAUTIONS:

1. In order to get sharp end point only few drops of Indicator are to be added.
2. Reading should be noted without parallax errors.
3. The lower meniscus should be taken for colorless solution.
4. Burette should be vertically clamped to the burette stand and there should not be any air bubbles and nozzle part of the burette should also be filled with the above solution.
5. Phenophthalein can not be used an indicator in this titration, since it reacts with $CO_2$ liberated.

# Experiment-2

# TOTAL HARDNESS

**AIM**: To determine the hardness of given sample of water.

**GENERAL**: Hardness is the capacity of water to react with soap, hard water requiring more amount of soap to produce lather. Scaling of hot water pipes, boilers and other household appliances is due to hard water. It is caused by dissolved ions of calcium and magnesium. The degree of hardness of drinking water has been classified in terms of the equivalent $CaCO_3$ concentration as follows:

Soft 0 - 60 mg/L.
Medium 60 - 120 mg/L.
Hard 120 -180 mg/L
Very hard > 180 mg/L.

**PRINCIPLE**:

In alkaline conduction, EDT A reacts with Ca and Mg to form a soluble chelated complex. Ca and Mg ions develop wine red colour with eriochrome black *T* under alkaline condition. When EDT A is added as a titrant, Ca and Mg divalent ions get complexed resulting in a sharp change from wine red to blue which indicates end-point of the titration.

**REAGENTS:**

1. Standard EDTA solution O.OIM : Dissolve 3.723 g EDT A sodium salt and dilute to 1000 ml
2. Eriochrome black *T* indicator : Mix O.5g dye with 100g NaCl to prepare dry powder.
3. Buffer solution : Dissolve 16.9g $NH_4Cl$ in 143 ml Of $NH_4OH$.

**PROCEDURE:**

1.Take 50 ml of well mixed sample in a conical flask.
2.Add 1-2 ml of buffer solution.
3. Add a pinch of Eriochrome black T and titrate with standard EDTA (0.0IM) till wine red colour changes to blue colour. Note down the Vol. of EDTA required. (A).
4. Run a reagent blank, Note the Vol.of EDTA (B).
5. Calculate the Vol. of EDTA required by sample, C == (A - B).

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**CALCULATIONS**: Calculate the hardness of the sample using the relation

Total Hardness = $\dfrac{C \times D \times 1000}{\text{Volume of sample(ml)}}$ as $CaCO_3$ mg/L

C = Volume of EDTA required by sample (ml)
D = mg of $CaCO_3$ equivalent to 1.0 ml of EDTA titrant = 1mg of $CaCO_3$

**OBSERVATIONS**:

| S.No. | Burette reading EDT A | | | Hardness |
|---|---|---|---|---|
| | Initial | Final | Difference | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

**RESULT:** Total Hardness of the water sample =---------mg/l as CaCO3

# Experiment-3

# PH-VALUE

**AIM**: To determine the pH of the given sample.

**GENERAL:** pH of aqueous solutions can be defined as negative logarithm of hydrogen ion concentration. pH values ranging from 0 to 7 are acidic, and from 7 to 14 are alkaline. The effect of pH on the chemical and biological properties of liquids makes its determination very important. It is used in several calculations in analytical work. The pH determination is usually done by electrometric method which is the most accurate method and free of interference.

**ELECTROMETRIC METHOD**:

The pH is determined by measurement of the electromotive force of a cell comprising an indicator electrode (an electrode responsive to hydrogen ions such as glass electrode) immersed in the test solution and a reference electrode or a combined electrode. The contact between the test solution and the reference electrode is usually achieved by means of a liquid junction. The emf of this cell is measured with pH meter. This is a high impedance electrometer calibrated in terms of pH.

**REAGENTS:** .

Calibrates the electrode system against standard buffer solution of known pH. Buffer tablets having pH 4.0, 7.0 and 9.2 are available.

**pH 4, pH7, pH 9.2 BUFFER SOLUTIONS**:

Dissolve buffer tablet of pH 4 in 100 ml of distilled water to get pH 4 buffer solution. Similarly other buffer solutions can be prepared by dissolving corresponding buffer tablets.

**PROCEDURE:**

1. Rinse the electrode thoroughly with distilled water.
2. Dry electrode by gently blotting with a soft tissue paper and standardise instrument with electrode immersed in a buffer solution.
3. Remove the electrode from buffer, rinse thoroughly and blot. dry.
4. Immerse in a second buffer below pH 10, approximately 3 pH units different from the first, the reading should be within 0.1 unit for the pH of the second buffer.
5. Rinse the electrode thoroughly, blot and dry and determine the pH of unknown sample.

**Sample No. pH Remarks**

# Experiment- 4

# DISSOLVED OXYGEN

**AIM**: To determine the dissolved oxygen content in given sample.

**THEORY:** The solubility of atmospheric oxygen in fresh water ranges from 14.4 mg/l at 0º C to about 7.0 mg/L at 35ºC at one atmospheric pressure. Since it is poorly soluble gas, its solubility directly varies with the atmospheric pressure at any given temperature. Analysis of DO is important in sanitary engineering practice. It is necessary to know DO levels to keep a check on stream pollution, and also to assess raw water quality. DO is necessary for all aerobic biological treatment processes. DO is the basis for BOD test which is an important parameter to evaluate pollution potential of wastes.

**PRINCIPLE**: (WINKLER METHOD WITH AZIDE MODIFICATION)

Oxygen present in a sample rapidly oxidizes the dispersed divalent manganese hydroxide to its higher valency which precipitates as a brown hydrated oxide after addition of NaOH and KI. Upon acidification, manganese reverts to divalent state and liberates iodine from KI equivalent to the original DO content. The liberated iodine is titrated against $Na_2S_2O_3$ (N/80) using starch as an indicator.

**APPARATUS:**

1. BOD bottles of capacity 300 ml.
2. Sampling device for collection of samples.

**REAGENTS**:

1. **Manganous sulphate**: Dissolve 480g tetrahydrate manganous sulphate and dilute to 1000 ml. Filter if necessary. This solution should not give colour starch when added to an acidified solution of KI.

2. **Alkali iodide-azide reagent**: Dissolve 500g NaOH and 150 g KI or 135 g. NI dilute to 1000 ml. Add 10 g $NaNO_3$ dissolved in 40 ml distilled water. This solution should not give colour with starch solution when diluted and acidified.

3. **Concentrate sulphuric acid**:

4. **Starch indicator**: Prepare paste or solution of 2.0 g L.R. grade soluble starch powder and 0.2 g salicylic acid as preservative in distilled water. Pour this solution in 100 ml boiling water. Allow to boil for few minutes, cool and then use.

5. **Stock sodium thiosuphate** 0.1N: Dissolve 24.82 g $Na_2S_2O_3$, $5H_2O$ in boiled distilled water and dilute to 1000 ml. Preserve by adding 5 ml of chloroform per litre.

6. **Standard sodium thiosulphate** 0.025 N: Dilute 250 ml stock $Na_2S_2O_3$ solution to 1000 ml with freshly boiled and cooled distilled water. Preserve by adding 5 ml chloroform per titre.

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**PROCEDURE:**

1. Collect sample in a BOD bottle using DO sampter.
2. Add 2 ml $MgSO_4$ followed by 2 ml of alkali-iodide-azide reagent. The tip of the pipette should be below the liquid level while adding these reagents. Stopper immediately.
3. Mix well by inverting the bottle 2-3 times and allow the precipitate to settle leaving 150 ml clear supernatant.
4. At this stage, add 2 ml conc. $H_2SO_4$. Mix well till precipitate goes into solution.
5. Allow the solution to stand at least 5 minutes.
6. Withdraw 100 ml. of the solution into an conical flask and immediately add 0.025 N sodium thiosulfate drop by drop from a burette until the yellow color almost disappears.
7. Add about 1 ml. of starch solution and continue the addition of the thiosulfate until the blue color just disappears. Record the ml. of thiosulfate used.
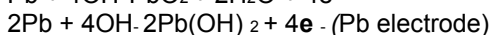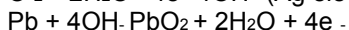
**CALCULATION**: Dissolved oxygen mg/L = ML of 0.025 N sodium thiosulphats used x 2

**RESULT:**

Dissolved oxygen = ---------------- mg/l.

**MEMBRANE ELECTRODE METHOD (MACKERETH OXYGEN CELL)**

Two metal electrodes, one of Ag and the other of Pb, are immersed in a saturated $KHCO_2$ solution separated from the test solution by a poly ethylene membrane, around 0.06 mm thick.. Thus, a galvonic cell can be plugged to a pH meter to give a direct reading of D.O. in mg $L^{-1}$ (the scale of 0 to 14 pH becomes 0 to 14 mg $L^{-1}$ D.O.). The current is measured for sample, for a standard (sample after air saturation) and for a blank (sample after treatment with a little $Na_2SO_3$ to expel $O_2$)

$O_2 + 2H_2O + 4e^- \, 4OH^-$ (Ag electrode)

$Pb + 4OH^- \, PbO_2 + 2H_2O + 4e^-$

$2Pb + 4OH^- \, 2Pb(OH)_2 + 4e^-$ (Pb electrode)

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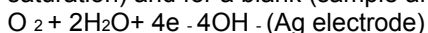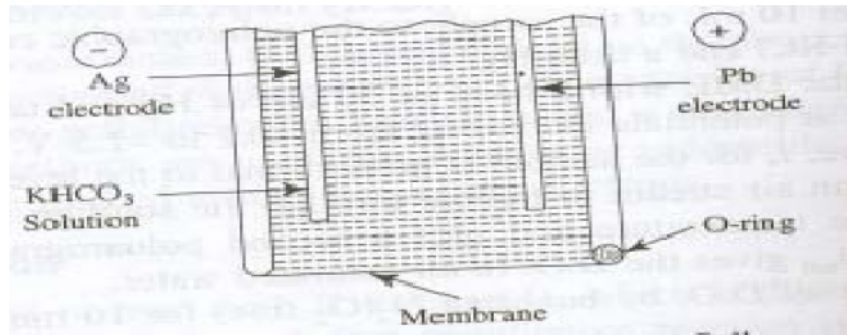
**The Mackereth Oxygen cell**

# Experiment-5

# TOTAL SOLIDS

**AIM:** To determine the total solids present in water sample.

**GENERAL:** The term solid refers to the matter either filterable or infilterable that remains as residue after evaporation and subsequent drying at a defined temperature. Different forms of solids are defined on the basis of method applied for their determination.

Residue after the evaporation and subsequent drying in oven at specific temperature, 103-105ºC of a known volume of a sample are total solids. The loss in weight on ignition of the same sample at 550°C (in which organic matter is converted to $CO_2$ and $H_2O$) gives organic solids present in the sample.

## PROCEDURE:

1. Take empty weight of beaker. ($W_1$)
2. Take a known volume of a well mixed sample in the above beaker.
3. Evaporate the sample to dryness at 103°C for 24 hours.
4. Cool and weigh and record the reading ($W_2$)
5. Keep the beaker for 15-20 min. in a muffle furnace maintained at 550 + 50°C.
6. Cool the beaker and record the final weight ($W_3$).

## CALCULATIONS:

Total solids mg/L. = $(W_2 - W_1)$x 1000

ml of sample

Organic solids mg/L. = $(W_2 - W_3)$x 1000

ml of sample

## OBSERVATIONS:

Weight of empty beaker $W_1$= ---------------- mg

Weight of beaker after evaporation at 103°C $W_2$ =---------------- mg

Weight of beaker after evaporation in $W_3$ = -------------- mg

muffle furnace

## RESULT:

Total Solids = mg/l

# Experiment-6
# CHEMICAL OXYGEN DEMAND

**AIM:** To determine the chemical oxygen demand of given sample.

**THEORY:** Chemical oxygen demand (COD) test determines the oxygen required for chemical oxidation of organic matter with the help of strong chemical oxidant. The limitation of COD test is that it can not differentiate between the biologically oxidizable and biologically inert material. COD determination has an advantage over BOD determination in that the result can be obtained in about 3-4 hours as compared to 5 days required for BOD test.

**PRINCIPLE:** The organic matter gets oxidized completely by $K_2Cr_2O_7$ in the presence of $H_2SO_4$ to produce $CO_2 + H_2O$. The excess $K_2Cr_2O_7$ remaining after the reaction is titrated with $Fe(NH_4)_2(SO_4)_2$. The dichromate consumed gives the $O_2$ required for oxidation of organic matter.

## APPARATUS:
1. Reflux apparatus consisting of a flat bottom 250 to 500 m1 capacity flask
2. Burner or hot plate with temperature regulator .

## REAGENTS:
1. **Standard potassium dichromate 0.250 N**: Dissolve 12.259g by $K_2Cr_2O_7$ dried at 103°C for 24 hours in distilled water and dilute to 1000 ml. Add about 120 mg sulphamic acid to take care of 6 mg/L $NO_2$-N.
2. **Sulphuric Acid reagent:** Add 10 g of $Ag_2SO_4$ to 1000 ml cone. $H_2SO_4$ and keep over night for dissolution.
3. **Standard ferrous ammonium sulphate 0.1 N**: Dissolve 39 g $Fe(NH_4.)_2(SO_4)_2 6H_2O$ in about 400 ml distilled water. Add 20 ml cone. H2SO4 and dilute to 1000 ml.
4. **Ferroin indicator**: Dissolve 1.485 g of 1, 10 phenanthroline monohyderate and 695 mg $FeSO_4$, $7H_2O$ and dilute to 100 ml with distilled water.
5. **Mercuric Sulphate**: $HgS0_4$ crystals, analytical grade.

## PROCEDURE:
1. Place 0.4 g $HgSO_4$ in a reflux flask.
2. Add 20 ml sample and mix well.
3. Add pumice stone or glass beads followed by 10 ml of standard $K_2Cr_2O_7$.
4. Add slowly 30 ml $H_2SO_4$ containing $Ag_2SO_4$ mixing thoroughly. This slow addition along with swirling prevents fatty acids to escape out due to high temperature.
5. Connect the flask to condenser: Mix the contents before heating.
6. Reflux for a minimum of 2 hours cool and then wash down the condenser with distilled water.24
7. Dilute for minimum of 150 ml, cool and titrate excess $K_2Cr_2O_7$. with 0.1 N Ferr. Amm. Sulphate using ferroin indicator. Sharp colour change from blue green to wine red indicates end-point.
8. Reflux blank: in the same manner using distilled water instead of sample.
9. Calculate COD from the following formula:

**COD** mg/L = (a-b)xNx8000
ml of sample
where a = ml of Ferr. Amm. Sulphate for blank.
b = ml of Ferr. Amm. Sulphate for sample.
N = normality of Ferr. Amm. Sulphate.

## OBSERVATIONS:

| S.No. | Burette Reading of Ferr. Amm. Sulphate | | | Remarks |
|-------|---------|-------|------------|---------|
|       | Initial | Final | Difference |         |
|       |         |       |            |         |
|       |         |       |            | .       |
|       |         |       |            |         |
|       |         |       |            |         |
|       |         |       |            |         |
|       |         |       |            |         |
|       |         |       |            |         |

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## RESULT:
COD of the given sample = ------------------mg/l.