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## مانيوال

# لاب بيئة

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الجامعة الهاشمية

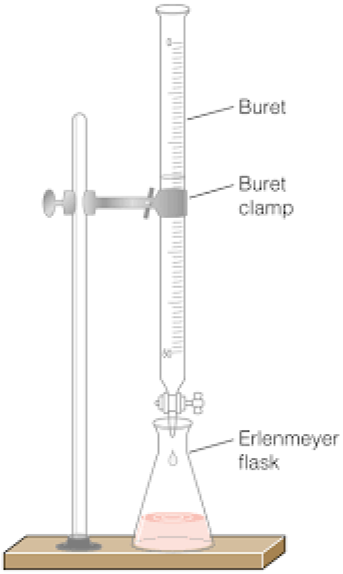
The Hashemite University

The Department of Civil Engineering

Faculty of Engineering

Laboratory Manual For

# Environmental Engineering



Prof. Ahmed . N. A. Bdour

Eng. Nisrein M. A. Al-Qutaish

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# LABORATORY SAFETY GUIDLINES

Never attempt to carry out an experiment without knowing the safety rules and procedures. Determine the potential hazards of all chemicals and any other appropriate information on chemicals, equipment and procedures. Remember laboratory safety is a full time job, all day and every day.

## GENERAL SAFETY

1. No smoking in the laboratory.
2. No food or drinks in the laboratory.
3. No using laboratory glassware in your food.
4. Don’t play in the laboratory
5. Lab coats and gloves must be worn at all times. Avoid chemical contact on skin.
6. No children in the laboratory.
7. Never work alone.
8. Only enclosed shoes are to be worn in the laboratory. No sandals.
9. Keep work area clean and uncluttered.

## CHEMICAL HAZARDS

1. Know the hazards of chemicals you are using.
2. Don’t touch, drink, eat and smell any of chemicals.
3. Don’t drink from distilled water.
4. Hands should be washed after contact with hazardous materials and before leaving.
5. Use fume hood if noxious gases are involved.
6. Dilute acids in the fume hood. Always add acid to water. DO NOT add water to acid.
7. Do not pipette by mouth.

## EQUIPMENT SAFETY

1. Calibrate all equipment prior to use.
2. Laboratory equipment is expensive and care should be taken when using it.
3. Don’t operate equipment if you are not familiar with.
4. Return all chemicals and supplies to the proper location after use.
5. Clean glassware with water and laboratory detergent, and let it to dry.
6. No experiment is complete until the laboratory is cleaned.

# EXPERIMENT #1: DETERMINATION OF CHLORIDE

## 1.1 AIM

To determine the chlorides concentrations present in given water sample.

## 1.2 INTRODUCTION

Chlorides are widely distributed as salts of calcium, sodium and potassium in water and wastewater. In potable water, the salty taste produced by chloride concentrations is variable and dependent on the chemical composition of water. The major taste producing salts in water are sodium chloride and calcium chloride. The salty taste is due to chloride anions and associated cations in water. Chlorides associated with sodium (Sodium Chloride) exert salty taste when its concentration is more than 250 mg/L and this is the limit of chloride concentration in public water supplies. On the other hand, a typical salty taste may be absent even if the water is having very high chloride concentration for example 1000 mg/L. This is because the predominant cation present in the water is not sodium but either calcium or magnesium may be present.

In many areas of the world where water supplies are scarce, sources containing as much as 2000 mg/L are used for domestic purposes without the development of adverse effect, once the human system becomes adapted to the water.

The measured chloride ions can be used to know salinity of different water sources. For brackish water (or sea water or industrial brine solution), it is used to determine the type of desalting of apparatus required and to control pumping of ground water from locations where intrusion of seawater is a problem. It also interferes with COD determination.

## 1.3 PRINCIPLE

The amount of chloride present in water can be easily determined by titrating the given water sample with silver nitrate solution. After all the chloride has been precipitated as white silver chloride, the first excess of titrant results in the formation of a silver chromate precipitate, which signals the end point (1). The reactions are:

$$Ag^+ + Cl^- \leftrightarrow AgCl(s)$$

$$2Ag^+ + CrO_4^{2-} \leftrightarrow Ag_2CrO_4(s)$$

The end of titration is indicated by formation of red silver chromate from excess silver nitrate.

## 1.4 Materials Required

### 1.4.1 Apparatus required

1. Burette
2. Pipette
3. Conical flask
4. Measuring cylinder
5. Funnel

### 1.4.2 REAGENTS

1. Silver nitrate
2. Potassium chromate indicator

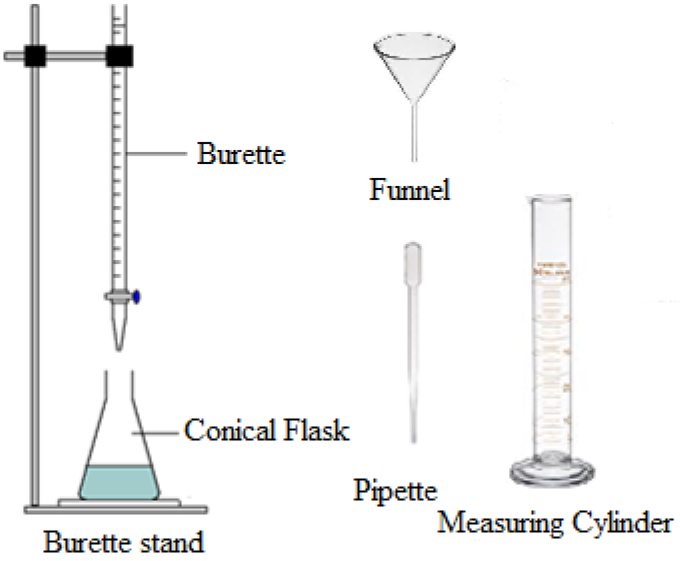


**Figure 1.15: Apparatus required for Chloride experiment**

## 2.5 PROCEDURE

1. Take 20 mL of the sample in a clean 250mL conical flask
2. Adjust pH to be between (7.0 and 8.0) either with sulpheric acid or sodium hudroxide solution.
3. Add 1 mL of Potassium Chromate indicator to get light yellow color
4. Titrate the sample against silver nitrate solution until the color changes from yellow to brick red. i.e., the end point.
5. Note the volume of Silver nitrate added ($V_S$).
6. Repeat the procedure for distilled water and note the volume of silver nitrate ($V_B$).

***End point: Changing the color from yellow to brick red**

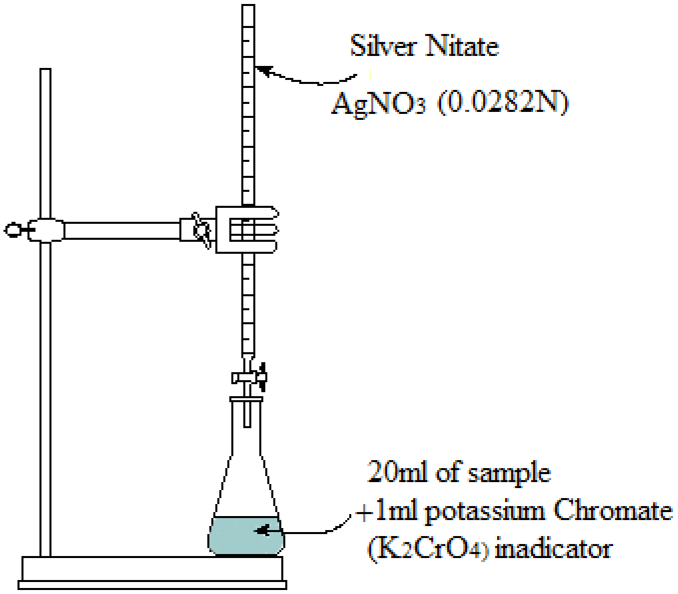


**Figure 1.16: Procedure of Chloride experiment**

## 2.6 DATA SHEET

DETERMINATION OF CHLORIDE DATA SHEET

Date Tested: August 30, 2010

Tested By: CEM Class, Group A

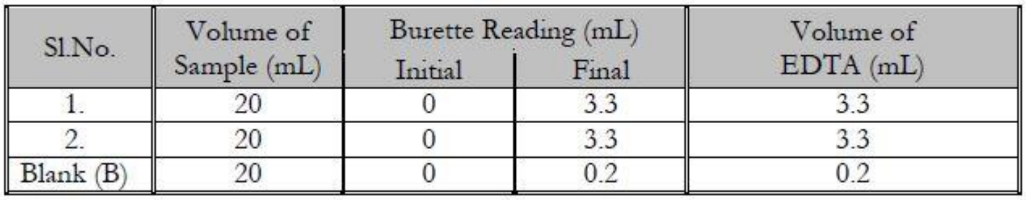
Project Name: CEM, NITTTR Lab

Sample Number: BH1

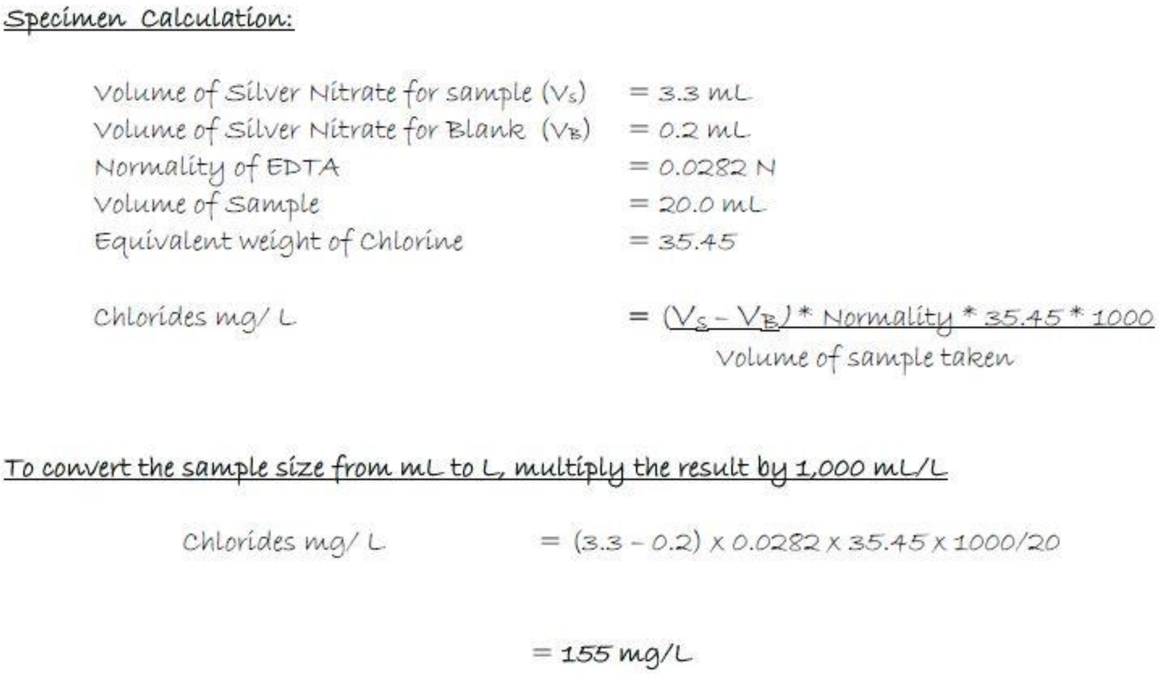
Sample Location: Perungudi (Lat 12’ 57’’ 31.74 & Long 80’14’’ 8.82)

Sample Description: Surface water

**TABULATION**



| Sl.No. | Volume of Sample (mL) | Burette Reading (mL) | | Volume of EDTA (mL) |
|---|---|---|---|---|
| | | Initial | Final | |
| 1. | 20 | 0 | 3.3 | 3.3 |
| 2. | 20 | 0 | 3.3 | 3.3 |
| Blank (B) | 20 | 0 | 0.2 | 0.2 |



Specimen Calculation:

Volume of Silver Nitrate for sample ($V_S$) = 3.3 mL
Volume of Silver Nitrate for Blank ($V_B$) = 0.2 mL
Normality of EDTA = 0.0282 N
Volume of Sample = 20.0 mL
Equivalent weight of Chlorine = 35.45

$$\text{Chlorides mg/ L} = \frac{(V_S - V_B) * \text{Normality} * 35.45 * 1000}{\text{Volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L

Chlorides mg/ L = (3.3 – 0.2) x 0.0282 x 35.45 x 1000/20

= 155 mg/L

# EXPERIMENT #2: DETERMINATION OF RESIDUAL CHLORINE

## 2.1 AIM

To determine the amount of total residual chlorine present in the given sample of chlorinated water by starch Iodide method

## 2.2 INTRODUCTION

Water chlorination is the process of adding chlorine as a gas ($Cl_2$) or as sodium hypochlorite solution (NaOCl) or solid calcium hypochlorite ($Ca(OCl)_2$ to water. This method is used to kill certain bacteria and other microbes in tap water as chlorine is highly toxic. In particular, chlorination is used to destroy or deactivate disease-producing microorganism in the public water supplies and polluted water.

When chlorine is added to water, some of the chlorine reacts first with organic materials and metals in the water and is not available for disinfection (this is called the chlorine demand of the water). The remaining chlorine concentration after the chlorine demand is accounted for is called total chlorine. Total chlorine is further divided into: 1) the amount of chlorine that has reacted with nitrates and is unavailable for disinfection which is called combined chlorine and, 2) the free chlorine, which is the chlorine available to inactivate disease-causing organisms, and thus a measure to determine the potability of water.

Residual chlorine is the amount of chlorine that remains in the water after a certain period or contact time.

## 2.3 PRINCIPLE

Chlorine will liberate free Iodine from Potassium Iodide solution at pH 8.0 or less. The liberated Iodine is titrated against standard sodium thiosulphate ($Na_2S_2O_3$) with starch as indicator. This Iodometric Method used for total chlorine testing at levels above 1 mg/l $Cl_2$.

## 2.4 Materials Required

### 2.4.1 Apparatus required

1. Burette
2. Pipette
3. Conical flask
4. Spatula
5. Balance
6. Funnel

### 2.4.2 REAGENTS

1. Concentrated Acetic acid
2. Potassium Iodide, KI, crystal
3. Sodium Thiosulphate (0.025N)
4. Starch solution

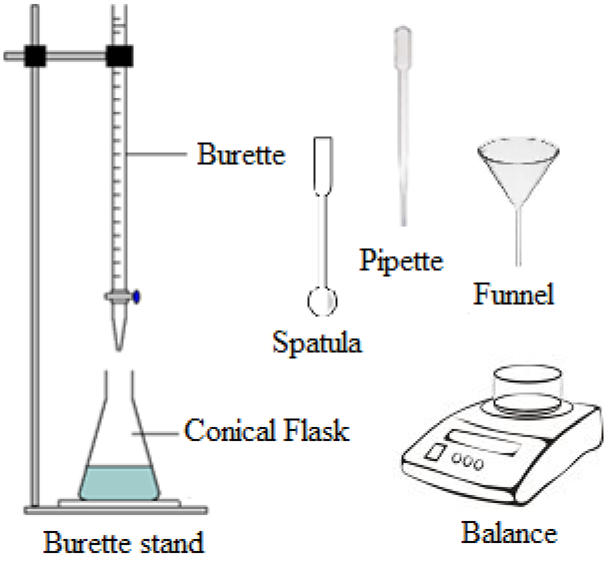


**Figure 2.17:Apparatus required for Residual chlorine experiment**

## 2.5 PROCEDURE

1. Take 200ml of sample in an Erlenmeyer flask
2. Add 5ml of Acetic acid to bring pH (3.0 to 4.0)
3. Add 1g of potassium iodide and mix thoroughly. Yellow color is obtained
4. Titrate against standard sodium thiosulphate solution in the burette until a pale yellow color is obtained
5. Add 1ml of starch indicator and continue the titration till the blue color disappears.
6. Note down the volume (V1)

***End point: Disappearance of blue color**

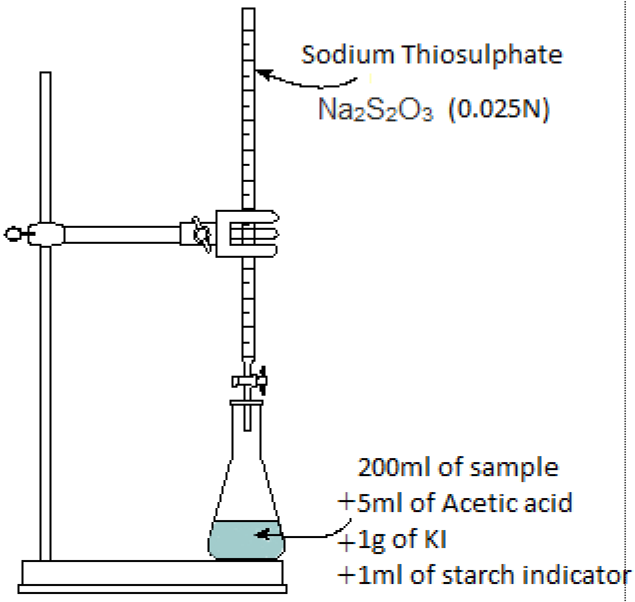


**Figure 2.18: Procedure for Residual chlorine experiment**

## 2.6 DATA SHEET

DETERMINATION OF RESIDUAL CHLORINE DATA SHEET

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Date Tested: August 30, 2010

Tested By: CEM Class, Group A

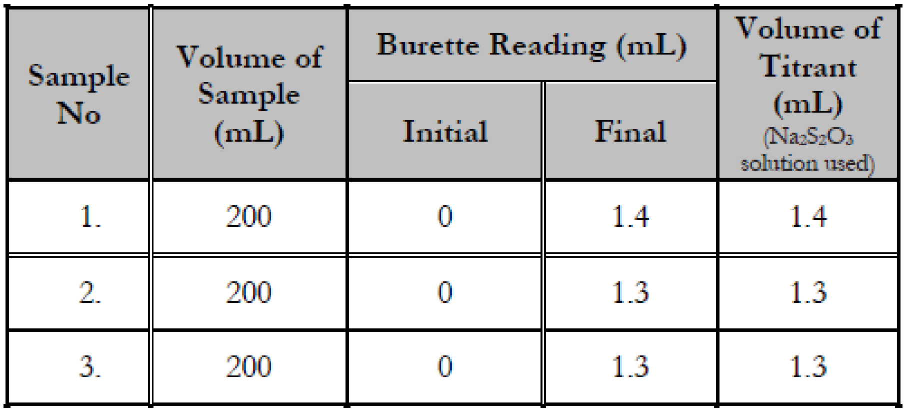
Project Name: CEM, NITTTR Lab

Sample Number: BH1

Sample Location: Perungudi (Lat 12’ 57’’ 31.74 & Long 80’14’’ 8.82)

Sample Description: Surface water

**TABULATION**



| Sample No | Volume of Sample (mL) | Burette Reading (mL) | | Volume of Titrant (mL) ($Na_2S_2O_3$ solution used) |
|---|---|---|---|---|
| | | Initial | Final | |
| 1. | 200 | 0 | 1.4 | 1.4 |
| 2. | 200 | 0 | 1.3 | 1.3 |
| 3. | 200 | 0 | 1.3 | 1.3 |

Specimen Calculation:

Volume of Sodium thiosulphate $V_1$ = 1.3 mL

Normality of Sodium thiosulphate $N_1$ = 0.01 N

Volume of Sample $V_2$ = 200.0 mL

Equivalent weight of Chlorine = 35.45

$$\text{Residual Chlorine} = \frac{\text{Volume of Sodium thiosulphate} * N * 35.45 * 1000}{\text{Volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L

Residual Chlorine (mg/L) = 1.3 x 0.01 x 35.45 x 1000/200 = 2.3 mg/L

# EXPERIMENT #3: DETERMINATION OF TOTAL HARDNESS

## 3.1 AIM

To determine the hardness of the given sample by EDTA Titrimetric method

## 3.2 INTRODUCTION

Hard water is water that has high mineral content (in contrast with "soft water"). Scaling of hot water pipes, boilers and other household appliances is due to hard water and it cause's excessive consumption of soap used for cleaning. Hard water is not seriously harmful to human health its useful to growth of children due to the presence of calcium and magnesium is an essential nutrient for plants.

Total hardness is determined by the multivalent cations' concentrations present in water. The most common cations present in hard water are $Mg^{2+}$ and $Ca^{2+}$.

Total hardness is defined as the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate in mg/L.

when hard water is heated, $Ca^{2+}$ ions react with bicarbonate ($HCO_3^-$) ions to form insoluble calcium carbonate (CaCO3) (Equation 1). This precipitate, known as **scale**, coats the vessels in which the water is heated, producing the mineral deposits on your cooking dishes. (Equation 2) presents magnesium hardness.

$$Ca^{2+}_{(aq)} + 2HCO_3^-{}_{(aq)} \leftrightharpoons CaCO_{3(s)} + CO_{2\,(aq)} + H_2O_{(l)} \qquad \text{Eq. 1}$$

$$Mg^{2+}_{(aq)} + 2OH^-_{(aq)} \leftrightharpoons Mg(OH)_{2(s)} \qquad \text{Eq. 2}$$

## 3.3 PRINCIPLE

In alkaline conduction, Ethylenediaminetetraacetic acid (abbreviated EDTA) reacts with Ca and Mg to form a soluble chelated complex. If a small amount of dye such as Eriochrome black T (abbreviated EBT) is added to an aqueous solution containing small calcium and magnesium ions at a pH of a 10±0.50 the solution become wine red. When EDTA is added as a titrant, $Ca^{2+}$ and $Mg^{2+}$ divalent ions get complexes resulting in a sharp change from wine red to blue which indicates end-point of the titration.

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## 3.4 Materials Required

### 3.4.1 Apparatus required

1. Burette
2. Pipette
3. Conical flask
4. Measuring cylinder
5. Funnel

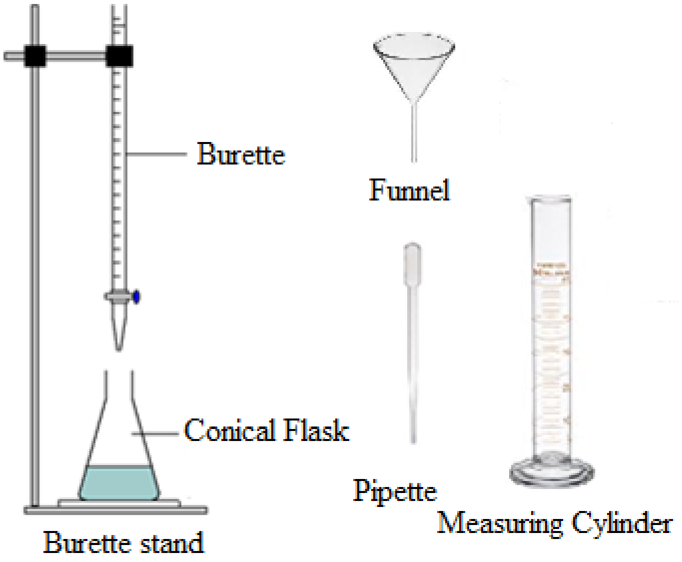

**Figure 3.19: Apparatus required for Total hardness experiment**

### 2.4.2 REAGENTS

1. EBT (Erichrome Black T) indicator
2. Ammonia buffer solution
3. EDTA(Ethylenediaminetetraacetic acid)

## 3.5 PROCEDURE

1. Pipette 20mL of water sample and transfer it to a clean 250mL conical flask.
2. Add 2mL of Ammonia buffer solution to the water sample so that the pH will be maintained between (9 and 10).
3. Add few drops of EBT indicator to the conical flask and the sample turns to wine red in color.
4. Before starting the titration rinse the burette with few mL of EDTA. Fill the burette with 0.02M EDTA solution and adjust to zero then fix it in burette stand.
5. Titrate the sample against the EDTA solution in the burette till all calcium and magnesium ions present in the sample reacts with the EDTA. The appearance of blue color indicates that all Ca & Mg ions are complexes with EDTA and forms a metal EDTA complex i.e., the end point of the titration.
6. Note down the burette reading

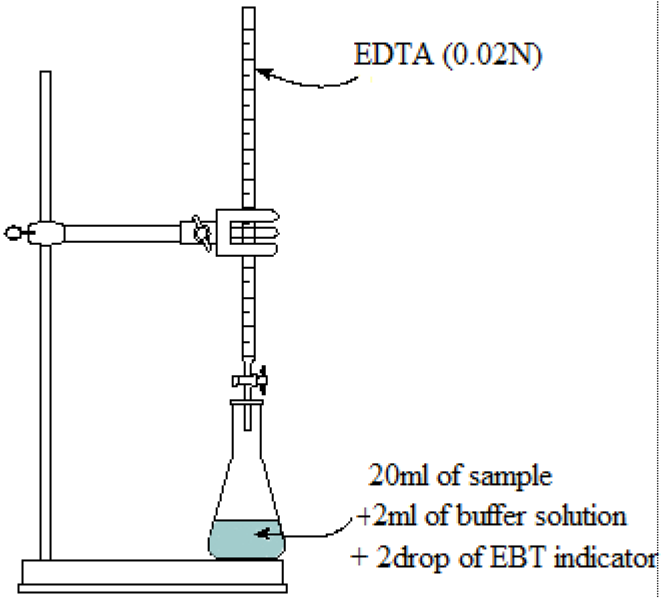

**Figure 3.20: Procedure of Total hardness experiment**

***End point: Changing color from wine red to blue**

## 2.6 DATA SHEET

DETERMINATION OF Total Hardness DATA SHEET

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Date Tested: August 30, 2010

Tested By: CEM Class, Group A

Project Name: CEM, NITTTR Lab

Sample Number: BH1

Sample Location: Perungudi (Lat 12’ 57’’ 31.74 & Long 80’14’’ 8.82)

Sample Description: Surface water

| Sl.No. | Volume of Sample (mL) | Burette Reading (mL) | | Volume of EDTA (mL) |
|---|---|---|---|---|
| | | Initial | Final | |
| 1. | 20 | 0 | 10.1 | 10.1 |
| 2. | 20 | 0 | 10 | 10 |
| 3. | 20 | 0 | 10 | 10 |

**Specimen Calculation:**

Volume of EDTA = 10 mL

Normality of EDTA = 0.02 N

Volume of Sample = 20.0 mL

Equivalent weight of $CaCO_3$ = 50

Total Hardness = $\frac{\text{Volume of EDTA} * N * 50 * 1000}{\text{Volume of sample taken}}$

**To convert the sample size from mL to L, multiply the result by 1,000 mL/L**

Calcium Hardness as $CaCO_3$ equivalent (mg/L) = 10 x 0.02 x 50 x 1000/20

= 500 mg/L as $CaCO_3$ equivalent

# EXPERIMENT #4: DETERMINATION OF CALCIUM HARDNESS

## 4.1 AIM

To determine the calcium hardness of the given sample by EDTA Titrimetric method

## 4.2 INTRODUCTION

Water hardness is an expression for the sum of the calcium and magnesium cations concentration in a water sample. Calcium hardness is the estimation of hardness due to calcium in water.

The presence of calcium in water results from deposits of lime stone, gypsum etc.

Calcium is one of the principal cations involved in water hardness. These cations form insoluble salts with soap and decrease the cleaning effectiveness of soap.

## 4.3 PRINCIPLE

The quantity of calcium in water will be determined by titrating the water sample with a standard Ethylenediaminetetraacetic acid (abbreviated EDTA) of known volume and conc.

An indicator, ammonium purpurate which combines only with calcium is used. The indicator imparts a pink color to the solution while there are calcium and magnesium ions that have not complexes with EDTA. Once the endpoint has been reached and there is no more uncomplexed Ca or Mg, the solution will turn to purple color.

## 4.4 Materials Required

### 4.4.1 Apparatus required

1. Burette
2. Pipette
3. Conical flask
4. Measuring cylinder
5. Funnel

### 4.4.2 REAGENTS

1. Ammonium Purpurate indicator
2. Sodium hydroxide
3. EDTA (Ethylenediaminetetraacetic acid)

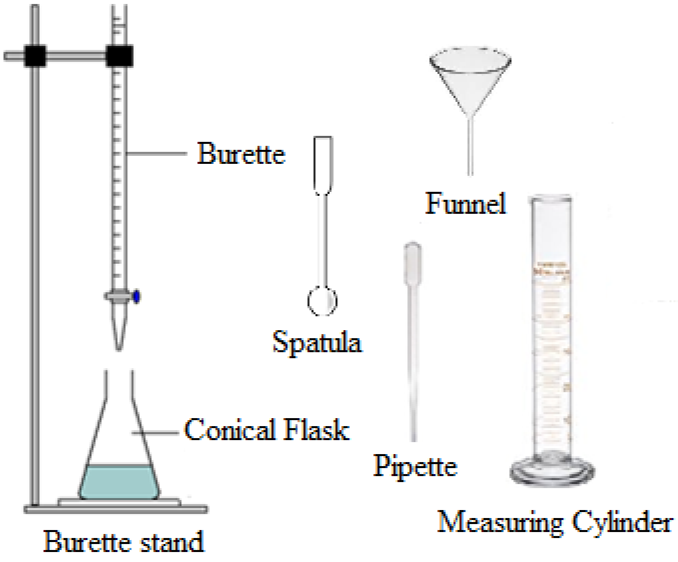

**Figure 4.21: Apparatus required for calcium hardness experiment**

## 4.5 PROCEDURE

1. Pipette 20mL of water sample and transfer it to a clean 250mL conical flask.
2. Add 2mL of Sodium hydroxide solution to the water sample so that the pH will be maintained between 12 and 13.
3. Add few drops of Ammonium Purpurate indicator to the conical flask and the sample turns into pink color. This color change is due to the calcium and magnesium contents present in water.
4. Before starting the titration rinse the burette with few mL of EDTA. Fill the burette with 0.02M EDTA solution and adjust to zero then fix it in burette stand.
5. Titrate the water sample against the EDTA solution in the burette till all calcium and magnesium ions present in the sample reacts with the EDTA to form a metal EDTA complex by changing the color of the sample to purple. i.e., the end point.
6. Note down the burette reading

***End point: Changing color from pink red to purple**

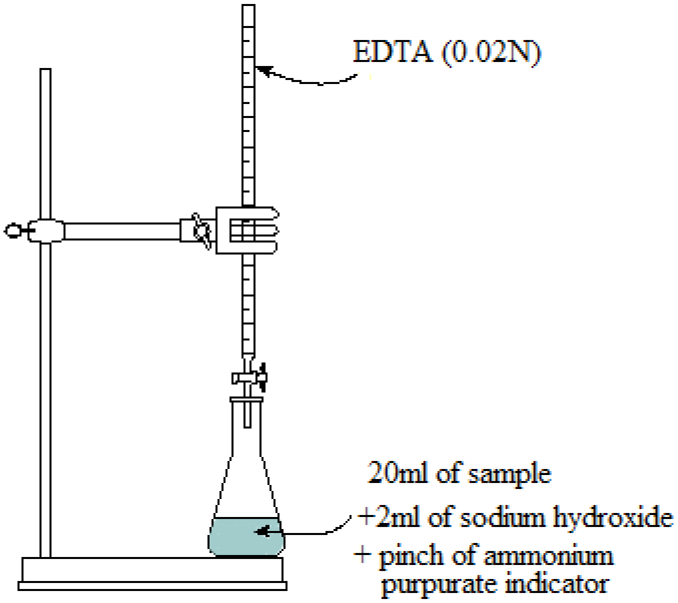

**Figure 4.22: Procedure of calcium hardness experiment**

## 4.6 DATA SHEET

DETERMINATION OF Calcium Hardness DATA SHEET

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Date Tested: August 30, 2010

Tested By: CEM Class, Group A

Project Name: CEM, NITTTR Lab

Sample Number: BH1

Sample Location: Perungudi (Lat 12' 57'' 31.74 & Long 80'14'' 8.82)

Sample Description: Surface water

| Sl.No. | Volume of Sample (mL) | Burette Reading (mL) | | Volume of EDTA (mL) |
|---|---|---|---|---|
| | | Initial | Final | |
| 1. | 20 | 0 | 5.6 | 5.6 |
| 2. | 20 | 0 | 5.7 | 5.7 |
| 3. | 20 | 0 | 5.7 | 5.7 |

**Specimen Calculation:**

| | |
|---|---|
| Volume of EDTA | = 5.7 mL |
| Normality of EDTA | = 0.02 N |
| Volume of Sample | = 20.0 mL |
| Equivalent weight of $CaCO_3$ | = 50 |

$$\text{Calcium Hardness} = \frac{\text{Volume of EDTA} * N * 50 * 1000}{\text{Volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L

$$\text{Calcium Hardness as } CaCO_3 \text{ equivalent (mg/L)} = 5.7 \times 0.02 \times 50 \times 1000/20$$

$$= 285 \text{ mg/L as } CaCO_3 \text{ equivalent}$$

$$\text{Calcium Present in the sample} = \frac{\text{Ca hardness in mg/L as CaCO3* molecular weight of Ca}}{\text{molecular weight of Calcium carbonate}}$$

$$= \frac{285 \times 40.078}{100.09} = 114 \text{ mg/L as } Ca^{+2}$$

Total Hardness = 500 mg/L as $CaCO_3$ equivalent

Magnesium Hardness = Total Hardness – Calcim Hardness

= 500 – 285 = 215 mg/l as $CaCO_3$ equivalent

$$\text{Magnesium Present in the sample} = \frac{\text{Mg hardness in mg/L as CaCO3* molecular weight of Ca}}{\text{molecular weight of Calcium carbonate}}$$

$$= \frac{215 \times 24}{100.09} = 51.6 \text{ mg/l as } Mg^{+2}$$

# EXPERIMENTS #5: DETERMINATION OF ALKALINITY

## 5.1 AIM:

To determine phenolphthalein alkalinity and total alkalinity of given water sample.

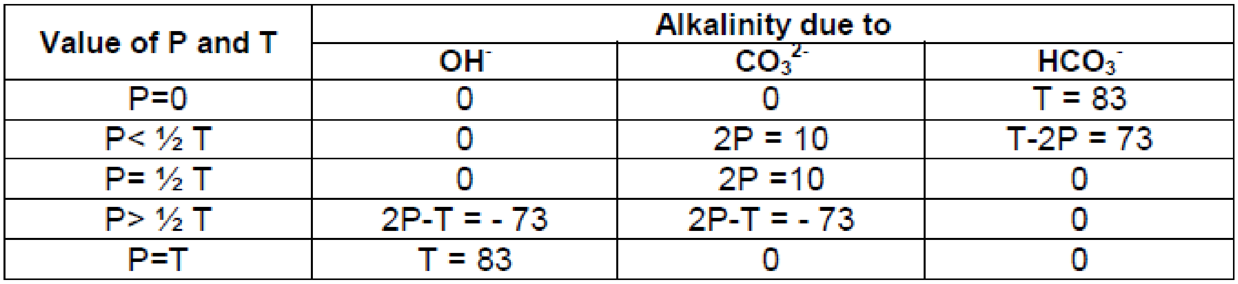
## 5.2 INTRODUCTION:

Alkalinity is a measure of the capability of water to absorb $H^+$ ions or to neutralized acid (ANC) without significant change of pH. In other words, alkalinity is a measure of the acid buffering capacity of water.

The ability of natural water to act as a buffer is controlled in part by the amount of calcium and carbonate ions in solution. water that comes in contact with limestone will contain high levels of both $Ca^{+2}$ and $CO_3^{2-}$ ions and have elevated hardness and alkalinity, so they are expressed as mg/l as $CaCO_3$.

Alkalinity of a sample of water is due to the presence of $OH^-$ (hydroxide ion), $HCO_3^-$ (bicarbonate ion) and $CO_3^{2-}$ (carbonate ion) or the mixture of two ions present in water. The possibility of $OH^-$ and $HCO_3^-$ ions together is not possible since they combine together to form $CO_3^{2-}$ ions. They are behaving as a basis so determined by titration with strong acid. Need two steps to find Total alkalinity (T alkalinity), the first end point determine (T alkalinity) and (T alkalinity) can be founded after the second end point Table 5.1 represent the 5-Combination of P and T alkalinity.

**Table 5.1: Combination of P and T alkalinity**



| Value of P and T | Alkalinity due to | | |
|---|---|---|---|
| | $OH^-$ | $CO_3^{2-}$ | $HCO_3^-$ |
| P=0 | 0 | 0 | T = 83 |
| P< ½ T | 0 | 2P = 10 | T-2P = 73 |
| P= ½ T | 0 | 2P =10 | 0 |
| P> ½ T | 2P-T = - 73 | 2P-T = - 73 | 0 |
| P=T | T = 83 | 0 | 0 |

The determination of alkalinity of water is necessary for controlling the corrosion and it's important for fish and aquatic life because it protects or buffers against rapid pH changes. Higher alkalinity levels in surface waters will buffer acid rain and other acid wastes and prevent pH changes that are harmful to aquatic life.

## 5.3 PRINCIPLE

The alkalinity of water can be determined by titrating the water sample with Sulphuric acid of known values of pH, volume and concentrations. Based on stoichiometry of the reaction and number of moles of Sulphuric acid needed to reach the end point, the concentration of alkalinity in water is calculated.

For the pH more than 8.3 need two step of titration firstly lowering pH to 8.3 using phenolphthalein indicator the color changes to **pink** color, This pink color is due to presence of hydroxyl ions. Titrate with sulphuric acid until pink color **disappears** (i.e. $OH^-$ ions are neutralized). This is the **1st end point (P alkalinity).** Then Lowering pH to 4.5 using mixed indicator, the presence of $CO_3^{2-}$ and $HCO_3^-$ ions in the solution changes the color to **blue**. While adding sulphuric acid, the color changes to **red**, this color change indicates that all the of $CO_3^{2-}$ and $HCO_3^-$ ions has been neutralized. This is the **2ndend point.**

The following reactions are occurring during titration;

***pH range above 8.3:***

$OH^- + H^+ \rightarrow H_2O$

$CO_3^{2-} + H^+ \rightarrow HCO_3^-$

***pH range between 8.3 and 4.5:***

$HCO_3^- + H^+ \rightarrow H_2CO_3$

## 5.4 Materials Required

### 5.4.1 Apparatus required

1. Burette
2. Pipette
3. Conical flask
4. Measuring cylinder

### 5.4.2 REAGENTS

1. Standard sulphuric acid
2. Phenolphthalein
3. Mixed Indicator

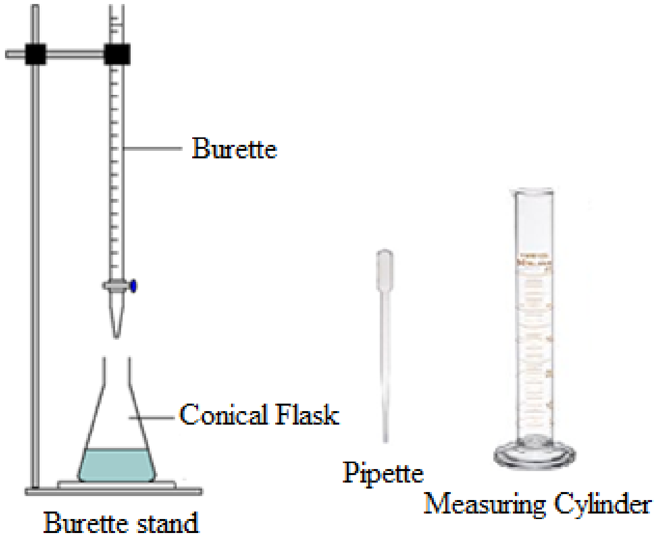


**Figure 5.23: Apparatus required for Alkalinity experiment**

## 5.5 PROCEDURE

1. Rinse the burette with 0.02N Sulphuric acid $H_2SO_4$ and discard the solution.
2. Fill the burette with 0.02N sulphuric acid and adjust it to zero.
3. Using a measuring cylinder exactly measure 100 mL of sample and pour it into a 250 mL of conical flask. Pipette 100mL of water sample and transfer it to a clean 250mL conical flask.
4. Add few drops of phenolphthalein indicator to the contents of conical flask. The color of the solution will turn to pink. This color change is due to alkalinity of hydroxyl ions in the water sample.
5. Titrate it against 0.02N sulphuric acid till the pink color disappears. This indicates that all the hydroxyl ions are removed from the water sample. Note down the titter value (V1). The value of titration is 0.5mL .This value is used in calculating the phenolphthalein alkalinity.
6. To the same solution in the conical flask add few drops of mixed indicator. The color of the solution turns to blue. This color change is due to $CO_3^{2-}$ & $HCO^{3-}$ ions in water sample.
7. Continue the titration from the point where stopped for the phenolphthalein alkalinity. Titrate till the solution becomes red. The entire volume (V2) of sulphuric acid is noted down and it is accountable in calculating the total alkalinity.

***First End point: Disappear of pink color**

***Second End point: Changing color from Blue to Red**

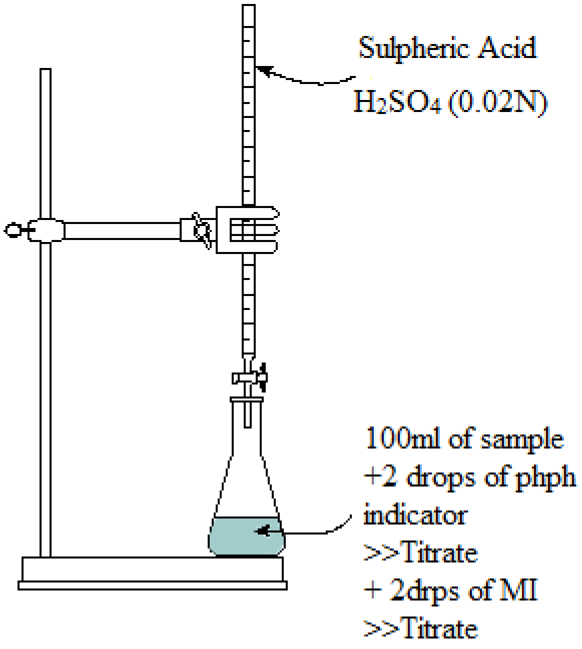


**Figure 5.24: Procedure of Alkalinity experiment**

### 4.6 DATA SHEET

DETERMINATION OF Calcium Hardness DATA SHEET

Date Tested: August 30, 2010

Tested By: CEM Class, Group A

Project Name: CEM, NITTTR Lab

Sample Number: BH1

Sample Location: Perungudi (Lat 12’ 57’’ 31.74 & Long 80’14’’ 8.82)

Sample Description: Surface water

| Sl.No. | Volume of Sample (mL) | Burette Reading (mL) | | Volume of Sulphuric acid (mL) |
|---|---|---|---|---|
| | | Initial | Final | |
| 4. | 100 | 0 | 0.6 | 0.6 |
| 5. | 100 | 0 | 0.5 | 0.5 |
| 6. | 100 | 0 | 0.5 | 0.5 |

Specimen Calculation:

Volume of Sulphuric Acid = 0.5 mL
Normality of Sulphuric Acid = 0.02 N
Volume of Sample = 50 mL
Equivalent weight of $CaCO_3$ = 1000

$$\text{Phenolphthalein Alkalinity} = \frac{(\text{Volume of } H_2SO_4 (V1) * \text{Normality} * 50 * 1000)}{\text{Volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L to convert the sample size from mL to L

Alkalinity as $CaCO_3$ equivalent (mg/L) = 0.5 x 0.02 x 50 x 1000/100

= 5 mg/L as $CaCO_3$ equivalent

## Total Alkalinity:

| Sl.No. | Volume of Sample (mL) | Burette Reading (mL) | | Volume of Sulphuric acid (mL) |
|---|---|---|---|---|
| | | Initial | Final | |
| 1 | 100 | 0 | 8.5 | 8.5 |
| 2 | 100 | 0 | 8.3 | 8.3 |
| 3 | 100 | 0 | 8.3 | 8.3 |

Specimen Calculation:

Volume of Sulphuric Acid = 8.3 mL
Normality of Sulphuric Acid = 0.02 N
Volume of Sample = 50 mL
Equivalent weight of $CaCO_3$ = 1000

$$\text{Total Alkalinity} = \frac{(\text{Volume of } H_2SO_4 (V1) * \text{Normality} * 50 * 1000)}{\text{Volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L to convert the sample size from mL to L

Alkalinity as $CaCO_3$ equivalent (mg/L) = 8.3 x 0.02 x 50 x 1000/100

T Alkalinity = 83 mg/L as $CaCO_3$ equivalent

P Alkalinity = 5 mg/l as CaCo3 equivalent

$P < 1/2\,T$ Then,

Alkalinity due $OH^-$ = 0
Alkalinity due $CO_3^{-2}$ = 2P = 10 mg/l
Alkalinity due $HCO_3^-$ = T-2P = 73 mg/l

# EXPERIMENT #6: DETERMINATION OF ACIDITY

## 6.1 AIM

To determine mineral and total acidity of given water sample.

## 6.2 INTRODUCTION

Acidity is a measure of the capacity of water to neutralize bases. Acidity of water is its quantitative capacity to react with a strong base to a designated pH. Acidity may be caused by mineral acids such as sulfuric acid or hydrochloric acid or by dissolved carbon dioxide

Usually dissolved carbon dioxide (CO2) is the major acidic component present in the unpolluted surface waters, so the water sample should be collected within a few hours of the time of analysis.

**Mineral acidity**: It is measured by titration to a pH of about 3.5, the methyl orange end point (also known as methyl orange acidity).

**Total acidity**: Titration of a sample to the phenolphthalein end point of pH 8.3 measures mineral acidity plus acidity due to weak acids, thus this is called as total acidity (or phenolphthalein acidity).

Aquatic life is affected by high water acidity. The organisms present are prone to death with low pH of water. High acidity water is not used for construction purposes, especially in reinforced concrete construction due to the corrosive nature of high acidity water. Water containing mineral acidity is not fit for drinking purposes.

## 6.3 PRINCIPLE

The acidity of water can be determined by titration of the water sample with Sodium hydroxide of known values of pH, volume and concentrations. Based on stoichiometry of the reaction and number of moles of Sulphuric acid needed to reach the end point, the concentration of alkalinity in water is calculated.

The color change of phenolphthalein indicator is close to pH 8.3 at 25ºC corresponds to stoichiometric neutralization of carbonic acid to bicarbonate.

## 6.4 Materials Required

### 6.4.1 Apparatus required

1. Burette
2. Pipette
3. Conical flask
4. Measuring cylinder

### 6.4.2 REAGENTS

1. Sodium hydroxide
2. Phenolphthalein indicator
3. Methyl orange indicator

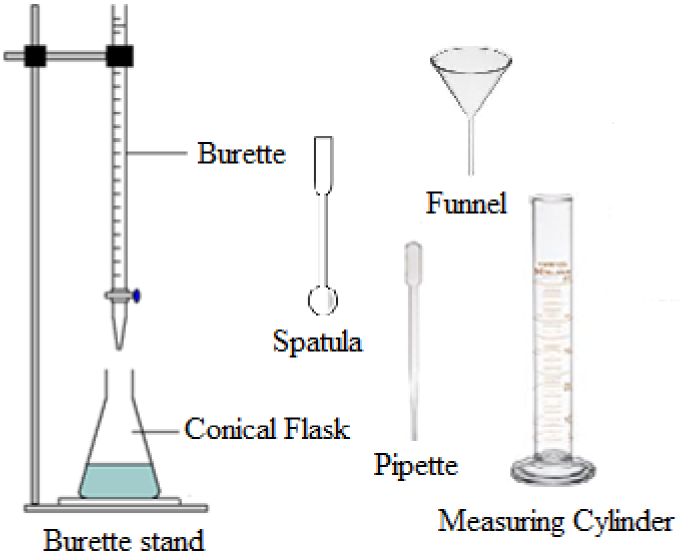


**Figure 6.25: Apparatus required for Acidity experiment**

## 6.5 PROCEDURE

1. Rinse the burette with 0.02N sodium hydroxide and then discard the solution.
2. Fill the burette with 0.02N sodium hydroxide and adjust the burette.
3. A sample size does not exceed 20mL of the titrant. For highly concentrated samples, dilute the sample. Usually, take 100 mL of a given sample in a conical flask using pipette.
4. Add few drops of methyl orange indicator in the conical flask.
5. The color changes to orange. Now titrate the sample against the 0.02N sodium hydroxide solution until the orange color faints.
6. Note down the volume (V1) consumed for titration 0.4mL. This volume is used for calculating the mineral acidity.
7. To the same solution in the conical flask add few drops of phenolphthalein indicator.
8. Continue the titration, until the color changes to faint pink color.
9. Note down the total volume (V2) consumed for titration. This volume is used for calculating the total acidity.

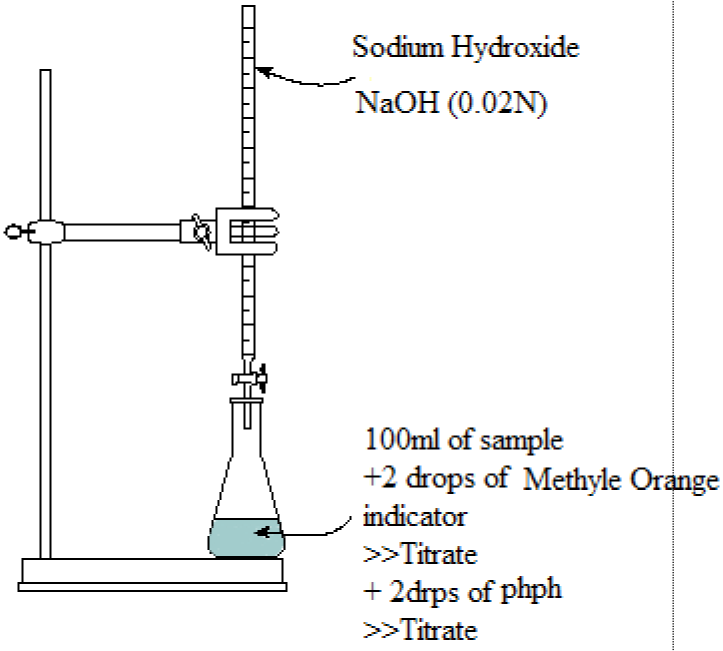


**Figure 6.26: Procedure of Acidity experiment**

***First End point: Changing color from Orange to yellow**

***Second End point: Changing color to Pink**

## 6.6 DATA SHEET

DETERMINATION OF Calcium Hardness DATA SHEET

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Date Tested: August 30, 2010 Tested By: CEM Class, Group A

Project Name: CEM, NITTTR Lab Sample Number: BH1 Sample Location: Perungudi (Lat 12' 57'' 31.74 & Long 80'14'' 8.82) Sample Description: Surface water

Table - 1 for Mineral Acidity:

| Sl.No. | Volume of Sample (mL) | Burette Reading (mL) | | Volume of NaOH (mL) |
|---|---|---|---|---|
| | | Initial | Final | |
| 4. | 100 | 0 | 0.5 | 0.5 |
| 5. | 100 | 0 | 0.4 | 0.4 |
| 6. | 100 | 0 | 0.4 | 0.4 |

Table - 2 for Total Acidity:

| Sl.No. | Volume of Sample (mL) | Burette Reading (mL) | | Volume of NaOH (mL) |
|---|---|---|---|---|
| | | Initial | Final | |
| 1. | 100 | 0 | 2.2 | 2.2 |
| 2. | 100 | 0 | 2.3 | 2.3 |
| 3. | 100 | 0 | 2.3 | 2.3 |

Model Calculation:

Volume of NaOH for Mineral Acidity ($V_1$) = 0.5 mL
Volume of NaOH for Total Acidity ($V_2$) = 8.3 mL
Normality of Sulphuric Acid = 0.02 N
Volume of Sample = 100.0 mL
Equivalent weight of $CaCO_3$ = 50

$$\text{Mineral Acidity} = \frac{\text{Volume of NaOH } (V_1) * N * 50 * 1000}{\text{Volume of sample taken}}$$

$$= 0.4 \times 0.02 \times 50 \times 1000/100 = 4 \text{ mg/L as } CaCO_3 \text{ equivalent}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L

$$\text{Total Acidity} = \frac{\text{Volume of NaOH } (V_2) * N * 50 * 1000}{\text{Volume of sample taken}}$$

$$= 2.3 \times 0.02 \times 50 \times 1000/100 = 23 \text{ mg/L as } CaCO_3 \text{ equivalent}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L

# EXPERIMENT #7: DETERMINATION OF CONDUCTIVITY

## 7.1 AIM

To determine the conductivity of given water sample.

## 7.2 INTRODUCTION

Conductivity of a substance is the ability or power to conduct or transmit heat, electricity or sound’. When an electrical potential difference is placed across a conductor, its movable charges flow, giving rise to an electric current. This property is called conductivity.

The electrical conductivity (G) can be expressed as mhos (Reciprocal of ohms) or as Siemens (S). The inverse of resistivity (R) is determined from to Ohm’s law. I.e. R=V/I then, G=1/R=I/V.

In most water, the conductivity is very low, so mS or μS are used as units for water conductivity. The water Suitable for irrigation has conductivity of 2 mS/cm. The conductivity of a solution is proportional to its ion concentration such as chloride, sodium and ammonium.

In coastal regions, conductivity data can be used to decide the extent of intrusion of sea water into ground water. is useful in determining the suitability of water and wastewater for disposal on land. Suitable for irrigation have 2 mS/cm. The total dissolved solids are about 70% of the conductivity

## 7.3 PRINCIPLE

Conductivity is measured with a probe and a meter. A voltage is applied between the two electrodes in the probe immersed in the sample water. Resistance of the water causes drop in voltage. The meter converts the probe measurement to micro mhos per centimeter and displays the result for the user.

## 7.4 Materials Required

### 7.4.1 Apparatus required

1. Conductivity Meter with probe
2. Measuring cylinder
3. Beaker

### 7.4.2 REAGENTS

1. Potassium Chloride
2. Distilled Water

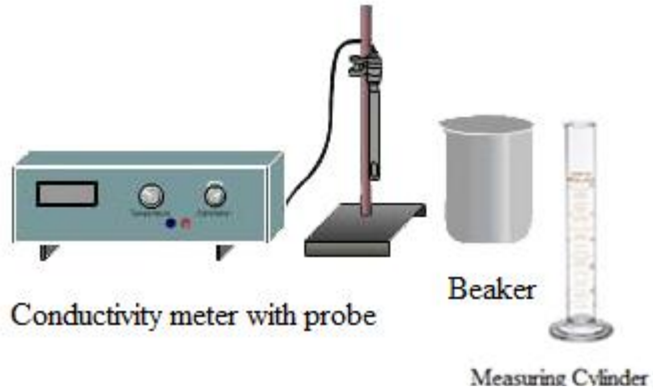

**Figure 7.1: Apparatus of Conductivity experiment**

## 7.5 PROCEDURE

1. Calibration of Conductivity Meter by takes 0.1N Potassium Chloride in a beaker. Switch on the magnetic stirrer and place the beaker on the stirrer. Insert the magnetic bead in the beaker. Place the electrode inside the solution. Select the calibration button and using up and down key adjusts the conductivity of the 0.1N potassium chloride solution to 14.12 mS/cm at 30°C. Now the meter is ready for the measurement of samples.
2. Rinse the electrode totally with deionized water and wipe with a tissue paper carefully.
3. Measure 200mL of water sample and transfer it to a beaker and place it on the magnetic stirrer.
4. Dip the electrode into the sample solution taken in a beaker and wait for a steady reading. Make sure that the instrument is giving stable reading.
5. Note down the reading in the display directly, which is expressed in millisiemens.

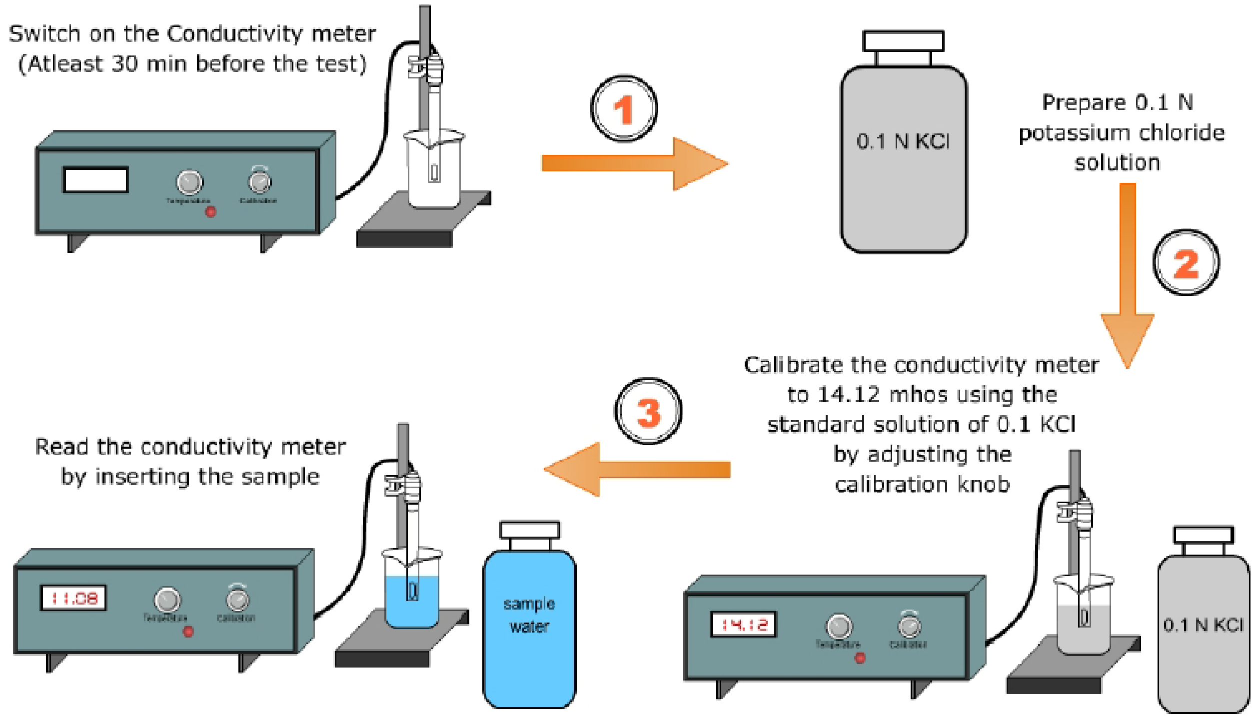

**Figure 7.2: Procedure of Conductivity experiment**

Result:-

The conductivity of the given sample 1 = 349.54 µmho (Surface water)

The conductivity of the given sample 2 = 102.88 µmho (Ground water)

The conductivity of the given sample 3 = 44.864 µmho (Sea water)

# EXPERIMENT #8: DETERMINATION OF TURBIDITY

## 8.1 AIM

To determine the turbidity of the given water sample.

## 8.2 INTRODUCTION

Turbidity is the cloudiness of a solution and it is a qualitative. Turbidity often indicates the presence of dispersed and suspended solids like clay, organic matter, silt, algae and other microorganisms.

Turbidity is caused by suspended materials which absorb and scatter light. These colloidal and finely dispersed turbidity-causing materials do not settle and are difficult to remove by sedimentation.

Measurement of turbidity in settled water prior to filtration is useful in checking on faulty filter operation, useful to determine the optimum dosage of coagulants to treat domestic and industrial wastewaters and it is used to evaluate the performance of water treatment plants.

## 8.3 PRINCIPLE

The turbidity of the sample is thus measured from the amount of light scattered by the sample. The turbidity of the sample is measured using the Turbidity meter which is a nephelometric instrument that senses the quantity of light-reflecting particles in water.

A beam of light is focused and passed through the sample solution (suspended particles) directly and the amount of scattered radiation is measured generally at 90° angle and displays the results in NTU (Nephelometric Turbidity Units). The higher the intensity of scattered light the higher is the turbidity.

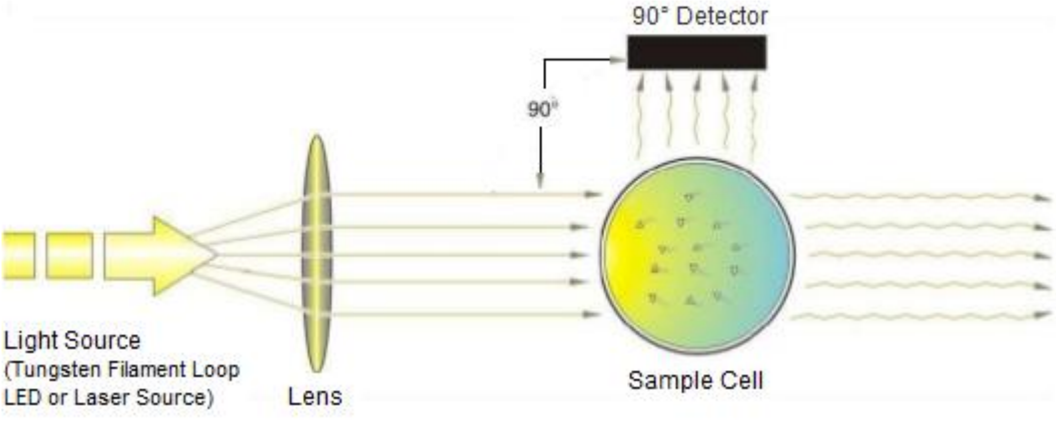

**Figure 8.1: Schematic view of a Nephelometric instrument.**

## 8.4 Materials Required

### 8.4.1 Apparatus required

1. Turbidity Meter
2. Sample cells
3. Wash Bottle
4. Tissue Papers

**Figure 8.2: Apparatus for Turbidity experiment**

### 8.4.2 REAGENTS

1. Distilled Water
2. Samples with known conductivity value 0.0, 20, 100, 400 NTU

## 8.5 PROCEDURE

### 8.5.1 Calibration:

1. Press “POWER” to turn on the meter, and the display will show “STBY”
2. Gently and slowly invert the calibration solution bottles five times but do not shake the bottles as air bubbles can affect the readings. If there is suspended material in the solution, please shake the bottles for 10seconds, and wait for 5minutes before calibration.
3. Press and hold “**CAL**” to enter calibration mode.
4. After entering calibration mode, the display will show “**0.00 NTU**” and “**CAL 1**”. Place the CAL 1 standard solution into the sample well, and:
    a. press “READ” for calibration, and the “CAL” icon will be flashing, or
    b. Press “DOWN ARROW/STO” to skip CAL 1calibration.
    c. After The display will then show “**20.0 NTU**” and “**CAL 2**”.
5. Repeat the above steps to calibrate (or skip) CAL 2 ~ CAL 4.
6. After CAL 4, the display will show “SA” and “END”, and then the meter will return to measurement mode.
7. Check corresponding numbers at the lower left of the display (calibration status indicator) to see which calibrations are done correctly.
8. If any of the calibration fails, the corresponding number icons will be flashing. For example, “1” icon will be flashing if CAL 1 is not completed correctly or the standard solution is out of range.

### 8.5.2 Measurement:

1. Fill the sample solution into the glass bottle.
2. Place the bottle into the sample well.
3. Press “READ” and wait for around 10 seconds to get the reading.

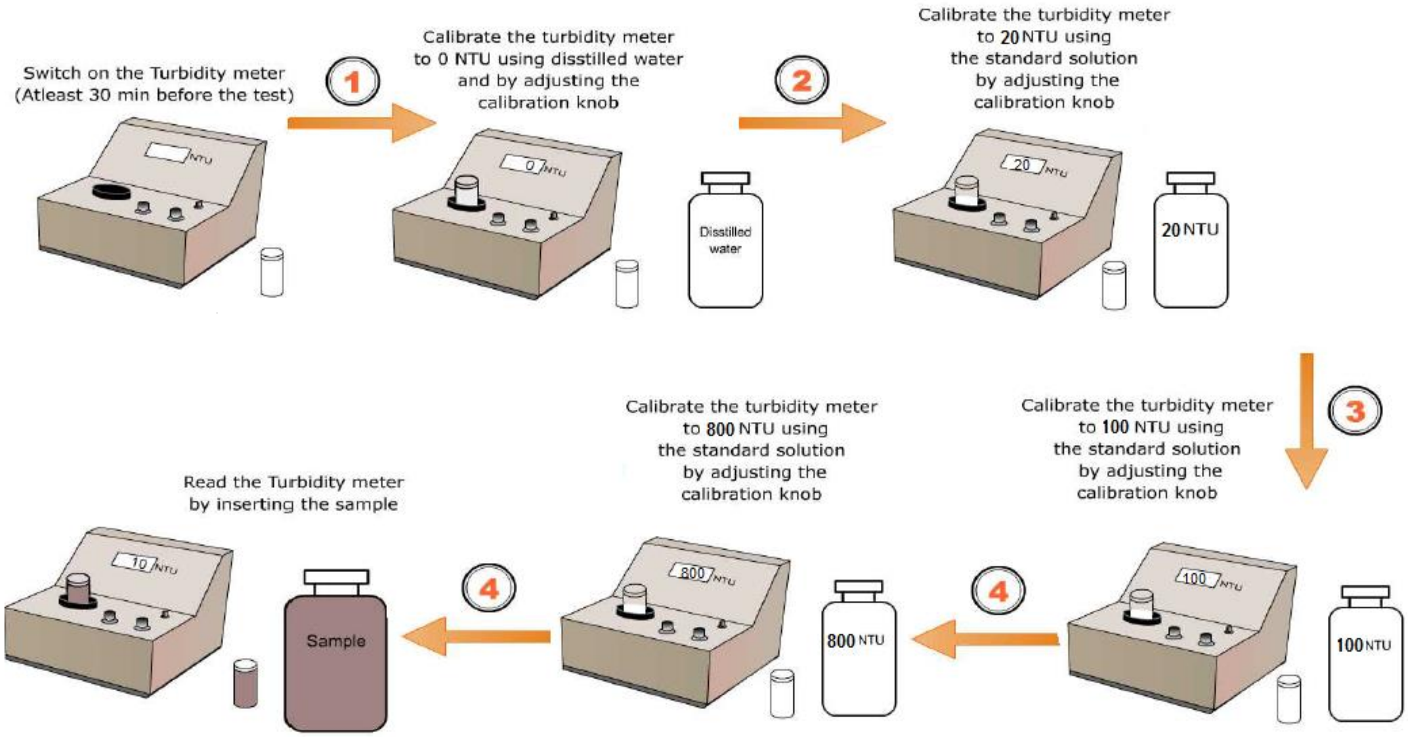

**Figure 8.3: Procedure of Conductivity experiment**

Result:-

The turbidity of the given sample 1 = 8.0 NTU

The turbidity of the given sample 2 = 2.3 NTU

The turbidity of the given sample 3 = 44 NTU

# EXPERIMENT #9: DETERMINATION OF TOTAL SOLID

## 9.1 AIM

To determine the total solids in the given water sample.

## 9.2 INTRODUCTION

Total Solids is the term applied to the material residue left in the vessel after evaporation of a sample, TS measured the combination of total dissolved solids and total suspended solids.

The term “solids” is generally used when referring to any material suspended or dissolved in water or wastewater that can be physically isolated either through filtration or through evaporation. Measurement of Solids can be made in different water samples (industrial, domestic and drinking water) and it is defined as residue upon evaporation of free water.

Total solids measurements come from runoff from construction, agricultural practices, logging activities, sewage treatment plant discharges, and other sources.

High levels of total solids will reduce the clarity of the water. This decreases the amount of sunlight able to penetrate the water, thereby decreasing the photosynthetic rate. Reduced clarity also makes the water less aesthetically pleasing. When the water is cloudy, sunlight will warm it more efficiently because the suspended particles in the water absorb the sunlight which, in turn, warms the surrounding water. This leads to other problems associated with increased temperature levels.

**Effects of High Total Solids**

- Can be harmful to aquatic organisms
- Reduce water clarity
- Aesthetically unpleasing
- Decrease photosynthetic rate
- Increase water temperature

## 9.3 PRINCIPLE

The sample is evaporated in a weighed dish on a steam bath and is dried to a constant mass in an oven either at 103-105°C or 179-181°C. Total solids/residue is calculated from increase in mass.

## 9.4 Materials Required

### 9.4.1 Apparatus required

1. Evaporating Dish
2. Drying Oven
3. Desiccators
4. Analytical Balance
5. Dish Tongs

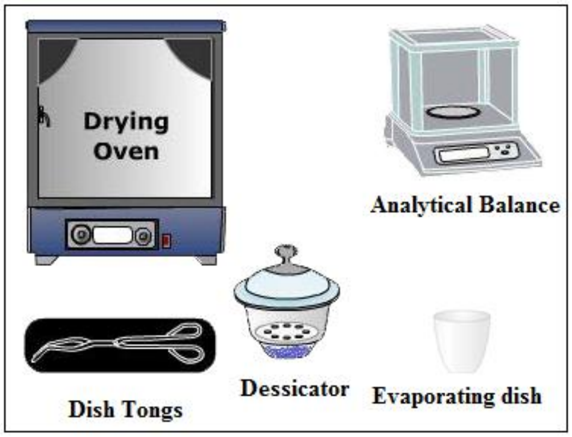

**Figure 9.1: Apparatus for Turbidity experiment**

## 9.5 PROCEDURE

1. Take a clean porcelain dish which has been washed and dried in a hot air oven at 105°C for one hour.
2. Weigh the empty evaporating dish in analytical balance. Let’s denote as (W1)
3. Using pipette transfer 75mL of unfiltered sample in the porcelain dish.
4. Switch on the oven and allowed to reach 105°C.
5. Place the evaporating dish in the hot air oven for 1 to 2 hours to eliminate necessity of checking for constant mass.
6. Cool the container in a desiccator to the room temperature. A dessicator will keep the samples from absorbing any water from the air that would increase their mass.
7. Weigh the dish as soon as it has cooled to avoid absorption of moisture. Let’s denote as (W1)

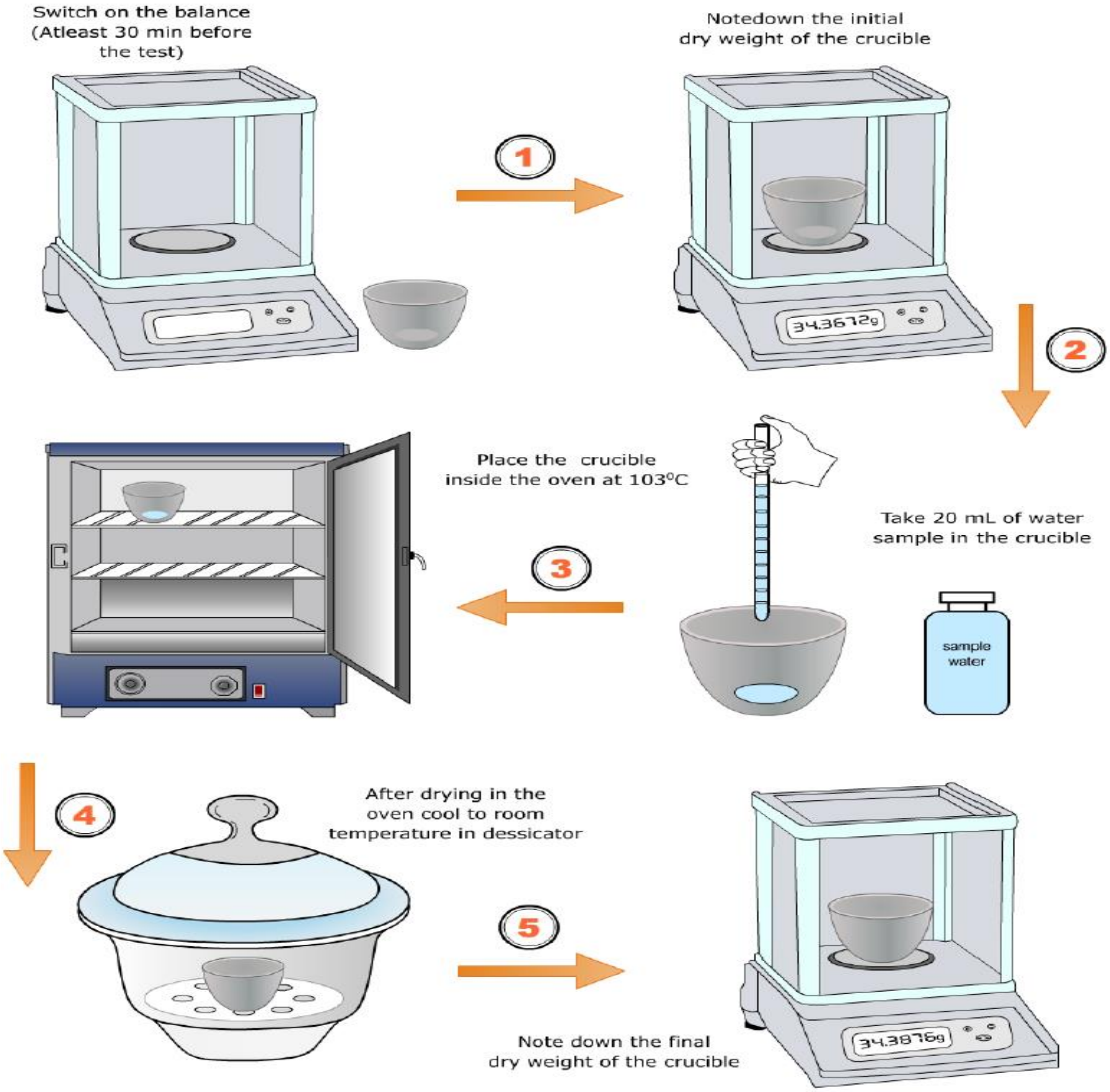

Figure 9.2: Procedure of Conductivity experiment

## 9.6 DATA SHEET

DETERMINATION OF Calcium Hardness DATA SHEET

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Date Tested: August 30, 2010

Tested By: CEM Class, Group A

Project Name: CEM, NITTTR Lab

Sample Number: BH1

Sample Location: Perungudi (Lat 12’ 57’’ 31.74 & Long 80’14’’ 8.82)

Sample Description: Surface water

| Description | | Weight (g) |
|---|---|---|
| Initial Weight of the Crucible (g) | $W_1$ | 35. 4323 |
| Final Weight of the Crucible + sample (g) | $W_2$ | 35. 4739 |
| Weight of residue(g) | W | 0. 0416 |
| Volume of the Sample (mL) | V | 75. 0 |
| **Total Solids (mg/L)** | **TS** | **555.0** |

<u>Specimen Calculation:</u>

$W_1$ = 35. 4323 g

$W_2$ = 35. 4739 g

V = 75. 0 mL

Weight of residue (g) W = $W_2$ - $W_1$

= 35. 4739 - 35. 4323 = 0. 0416 g

Weight of residue in mg (To convert W (g) to W (mg), multiply W (g) with 1000)

W (mg) = 0.0416 x 1000 = 41.6mg

Multiply the weight of the dry solids (in mg) by 1,000 mL/L to convert the sample size from mL to L.

<u>Total Solids (mg/L)</u>

V = Volume of the sample (mL) (To convert mL to L, multiply by 1000)

=41.6 mg/75 mL = 0.555 mg/mL

= 0.555 mg/mL x 1,000 mL/L = 555 mg/L

# EXPERIMENT #10: JAR TEST EXPERIMENT

## 10.1 AIM

To determine the optimum dosage of coagulant to remove small or charged particles present inside water.

## 10.2 INTRODUCTION

The two basic terms which can exactly explain the happenings of this experiment are "Coagulation" and "Flocculation".

1. **Coagulation**: It is the process of addition of a chemical to de-stabilize a stabilized charged particle.

2. **Flocculation**: It is a slow mixing technique which promotes agglomeration and helps the particles to settle down. So the "slow mixing" step is needed in the present experiment.

Generally we encounter **very fine and charged** clay like particles in water treatment which should be removed before we continue for further processes. These impurities **do not settle by gravity** when the water is passed through a sedimentation tank. The reason being that these are **charged particles, they repel each other** and just stay.

## 10.3 PRINCIPLE

When Coagulant like alum solution is added to water, the molecules dissociate to yield positive and negative charges like $SO_4^{2-}$ and $Al^{3+}$ in alum. These charged species combine with the charged colloidal particles to neutralize the charge of the component producing turbidity, and then these components are conglomerated and settled out fig (7.1).

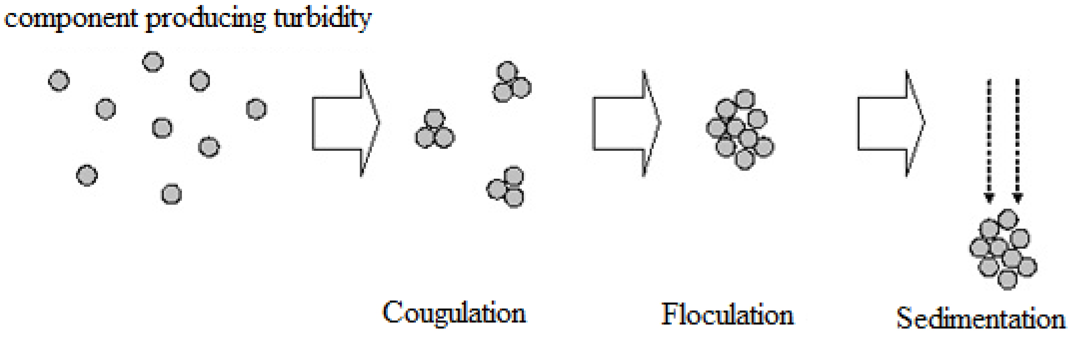


Figure 10.1: Coagulation-Sedimentation Diagram

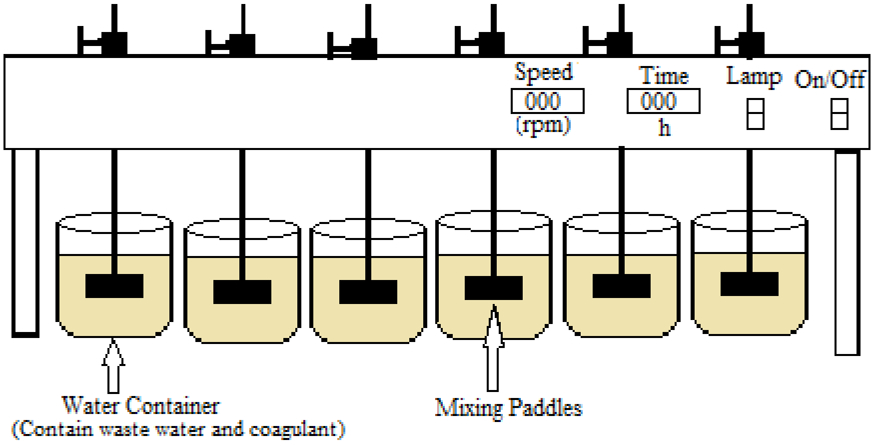
## 10.4 Materials Required

### 10.4.1 Apparatus required

1. Jar testing apparatus
2. Turbidity meter
3. 6-Beaker (1L)

### 10.4.2 REAGENTS

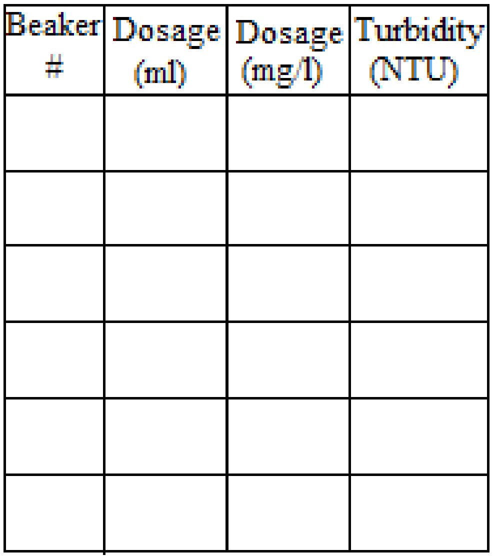
1. Alum solution (1L containing 10g of alum)
2. Distilled Water



## 10.5 PROCEDURE

1. Take 1000 ml of given sample in a 6-beakers.
2. Add 1 ml, 2 ml, 4 ml, 8 ml, 10 ml, 12 ml of alum respectively in each one of the beakers.
3. Now insert the paddle of the jar testing apparatus inside the beakers and start it.
4. Initially maintain a speed such that the paddles rotate at an angular velocity of 100 rpm for a time of 1 minute.
5. Now adjust the speed such that the paddles rotate at 40 rpm/min for a time of 10 minutes.
6. Now allow the beakers to settle down for 10 minutes.
7. Make an observation as of which of the 6 beakers is most clearer.
8. Measure the turbidity of each beaker using a turbidity meter and tabulate your results.
9. Plot a graph "Turbidity" Vs. "Coagulant dosage".

## 10.6 RSULT AND GRAPH:



| Beaker # | Dosage (ml) | Dosage (mg/l) | Turbidity (NTU) |
|---|---|---|---|
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

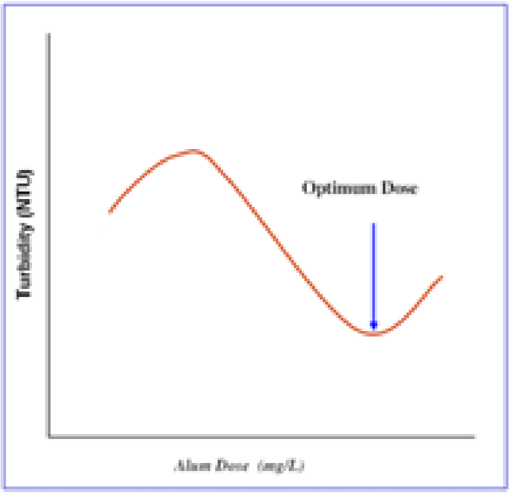


Figure7.3: Turbidity vs Coagulant dosage

# EXPERIMENT #11: CHEMICAL OXYGEN DEMAND (COD)

## 11.1 AIM

To determine chemical oxygen demand in the given water sample

## 11.2 INTRODUCTION

Chemical oxygen demand (COD) test is commonly used to indirectly measure the amount of organic compounds in water, making COD a useful measure of water quality. It is expressed in (mg/L), which indicates the mass of oxygen consumed per liter of solution.

COD determines the quantity of oxygen required to oxidize the organic matter in water or waste water sample, under specific conditions of oxidizing agent ($K_2Cr_2O_7$), temperature ($150^{\circ}$ C), and time (2hr). This method covers the determination of COD in ground and surface waters, domestic and industrial wastewaters. The applicable range is (3-900) mg/L.

The ratio of BOD to COD is useful to assess the amenability of waste for biological treatment. Ratio of BOD to COD greater than or equal to 0.8 indicates that wastewater highly polluted and amenable to the biological treatment.

BOD value is always lower than COD value. For domestic and some industrial wastewater, COD value is about 2.5 times BOD value

## 11.3 PRINCIPLE

The organic matter present in sample oxidized completely by potassium dichromate ($K_2Cr_2O_7$) in the presence of sulphuric acid ($H_2SO_4$), silver sulphate ($AgSO_4$) and mercury sulphate ($HgSO_4$) to produce $CO_2$ and $H_2O$. The excess potassium dichromate ($K_2Cr_2O_7$) is determined by titration against ferrous ammonium sulphate abbreviation (FAS), using ferroin as an indicator. The dichromate consumed by the sample is equivalent to the amount of $O_2$ required to oxidize the organic matter.

## 11.4 Materials Required

### 11.4.1 Apparatus required

4. COD Digester
5. COD vials with stand
6. Pipette
7. Burette
8. Conical flask
9. Funnel

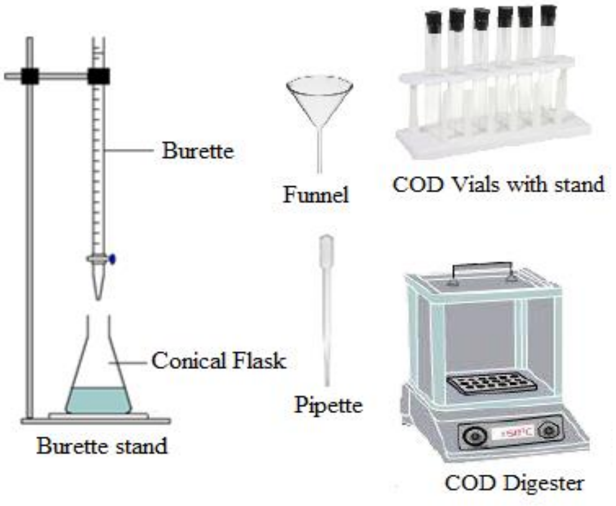


Figure 11.1: Apparatus required for COD

## 11.4.2 REAGENTS

1. Potassium dichromate ($K_2Cr_2O_7$)
2. Sulfuric acid ($H_2SO_4$)
3. Ferrous ammonium sulphate
4. Ferroin indicator
5. Organic free distilled water

## 11.5 PROCEDURE

1. Take three COD vials with stopper (two for the sample and one for the blank).
2. Add 2.5 mL of the sample to each of the two COD vials and the remaining COD vial is for blank; to this COD vial add distilled water.
3. Add 1.5 mL of potassium dichromate reagent to each of the three COD vials.
4. Add 3.5 mL of sulphuric acid reagent to each of the three COD vials.
   *CAUTION: COD vials are hot now.
5. Cap tubes tightly. Switch on the COD Digester and fix the temperature at 150º C and set the time at 2 hours.
6. Place the COD vials into a block digester at 150°C and heat for two hours.
7. The digester automatically switches off. Then remove the vials and allow it to cool to the room temperature.
8. Fill the burette with the ferrous ammonium sulphate (FAS) solution.
9. Transfer the contents of the blank vial to conical flask.
10. Add few drops of ferroin indicator. The solution becomes bluish green in color.
11. Titrate it with the FAS until reddish brown color appearance
12. Note down the volume of FAS solution added for the blank (A) is 14.1mL.
13. Transfer the contents of the sample vial to conical flask.
14. Add few drops of ferroin indicator. The solution becomes green in color.
15. Titrate it with the FAS until reddish brown color appearance
16. Note down the volume of FAS solution added for the sample (B) is 13.2mL.

12.***End point: appearance of the reddish brown color**

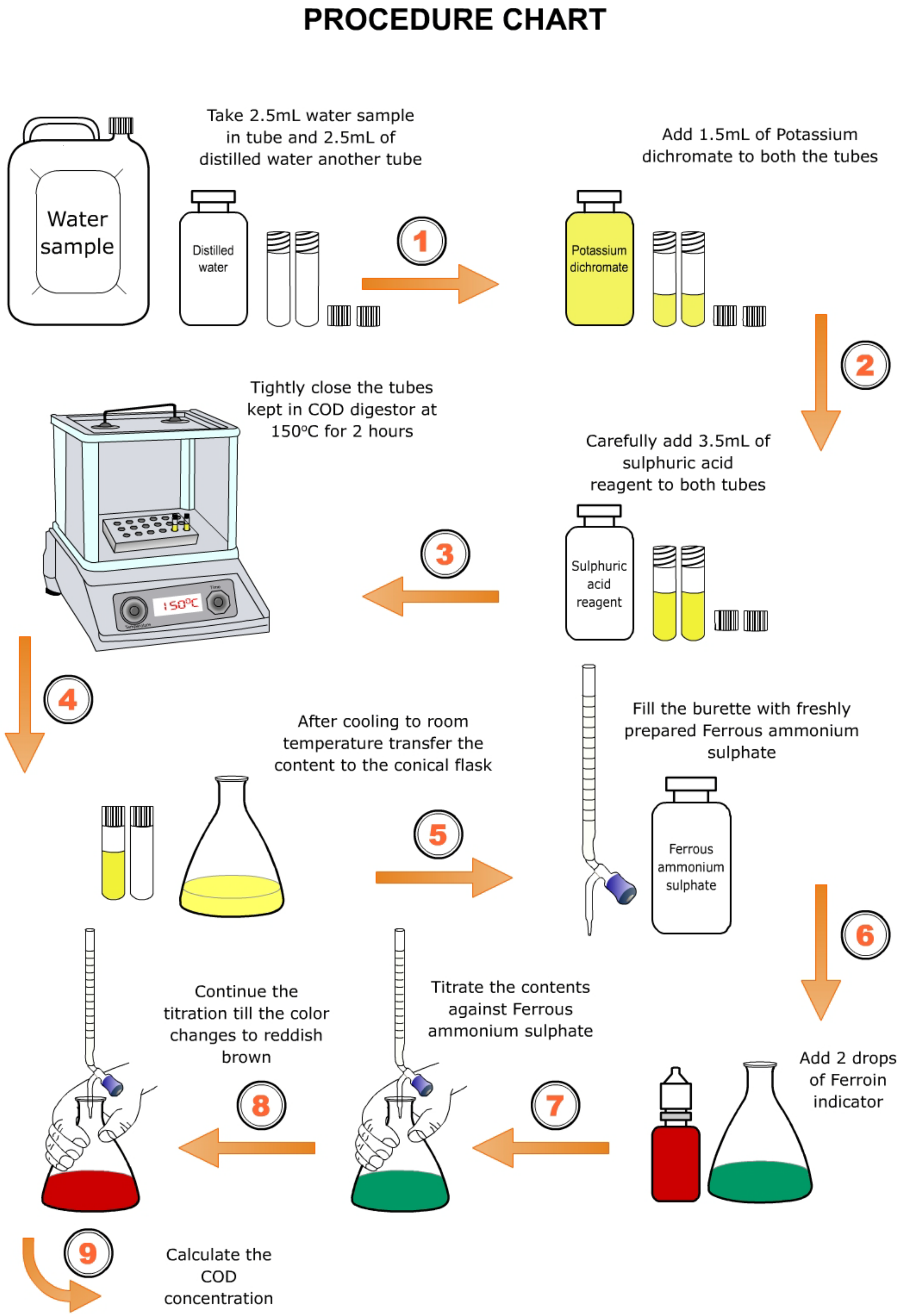


Figure 11.2: Procedure for COD experiment

## 11.6 DATA SHEET

DETERMINATION OF RESIDUAL CHLORINE DATA SHEET

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Date Tested: August 30, 2010

Tested By: CEM Class, Group A

Project Name: CEM, NITTTR Lab

Sample Number: BH1

Sample Location: Perungudi (Lat 12’ 57’’ 31.74 & Long 80’14’’ 8.82)

Sample Description: Surface water

### TABULATION

| Sl No. | Sample | Volume of Sample (mL) | Burette Reading (mL) | | Volume of 0.1 N FAS (mL) |
|---|---|---|---|---|---|
| | | | Initial | Final | |
| 4. | Blank | 2.5 | 0 | 14.1 | 14.1 |
| 5. | Sample 1 | 2.5 | 0 | 13.2 | 13.2 |
| 6. | Sample 2 | 2.5 | 0 | 13.2 | 13.2 |

Specimen Calculation:

Volume of Ferrous Ammonium sulphate for blank (A) = 14.1 mL
Volume of Ferrous Ammonium sulphate for Sample (B) = 13.2 mL
Normality of Ferrous Ammonium sulphate N = 0.1 N
Volume of Sample V = 2.5 mL

Chemical Oxygen Demand =

$$\frac{(A - B * N * 8 * 1000)}{\text{Volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L to convert the sample size from mL to L.

Residual Chlorine (mg/L) = (14.1 - 13.2) x 0.1 x 8 x 1000/2.5

= 288 mg/L

# EXPERIMENT #12: DETERMINATION OF DISSOLVED OXYGEN (DO)

## 12.1 AIM

To determine the dissolved oxygen (DO) in the given water sample.

## 12.2 INTRODUCTION

Dissolved Oxygen is used to describe the amount of oxygen dissolved in a unit volume of water and it’s measure of the ability of water to maintain aquatic life.

The dissolved oxygen content of water is influenced by the source, raw water temperature, treatment and chemical or biological processes taking place for samples.

The presence of oxygen in water is a good sign. Depletion of dissolved oxygen in water supplies can encourage the microbial reduction of nitrate to nitrite and sulfate to sulfide. It can also cause an increase in the concentration of ferrous iron in solution, with subsequent discoloration at the tap when the water is aerated.

Dissolved oxygen comes from the atmosphere and from photosynthesis by aquatic plants, and its removed from water by respiration and decomposition of organic matter. The solubility of oxygen decreases as water temperature increases. Its solubility is about 14.6 mg/l for pure water at 0°C under normal atmospheric pressure and it drops to 7 mg/l at 35°C.

The wastes serve as the food for certain aerobic bacteria so the concentration of bacteria increases and their population increase causes a decline in the amount of DO.

In a healthy body of water such as a lake, river, or stream, the dissolved oxygen is about 8 ppm. The minimum DO level of 4 to 5 ppm is desirable for survival of aquatic life.

## 12.3 PRINCIPLE

Dissolved Oxygen can be measured either by titrimetric or electrometric method.

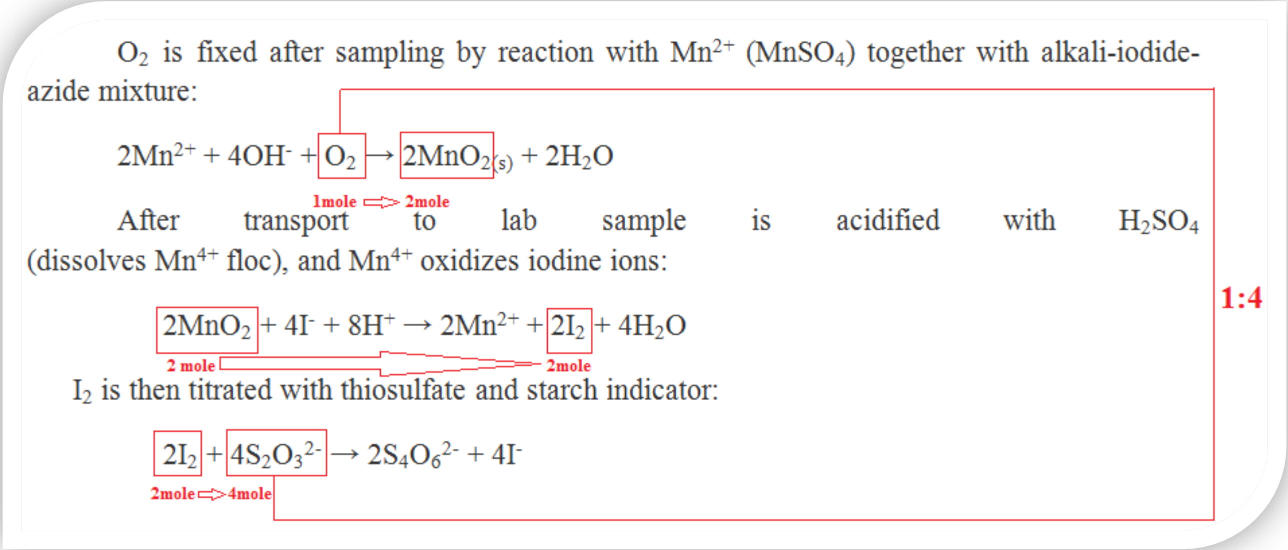
### (1) Titrimetric Method

Titrimetric method is based on the oxidizing property of DO while the electrometric method (using membrane electrodes) is based on the rate of diffusion of molecular oxygen across a membrane. There are different titrimetric methods based on the type of sample to be tested.

a) Winkler Method : Explained below in this experiment
b) Azide Modification: The interference caused by nitrate is removed effectively.
c) Alum Flocculation Modification: For sample contains suspended solids.
d) Permanganate Modification: For sample contains iron ($Fe^{2+}$) ions

## The Winkler Method for DO Determination:

Oxygen in the water sample oxidizes iodide ion **($I^-$)** to iodine **($I_2$)** quantitatively. The amount of iodine generated is then determined by titration with a standard thiosulfate ($S_2O_3^{-2}$) solution. The endpoint is determined by using starch as a visual indicator. The amount of oxygen can then be computed from the titration **1mole of $O_2$** reacts with **4moles of thiosulfate**.



$O_2$ is fixed after sampling by reaction with $Mn^{2+}$ ($MnSO_4$) together with alkali-iodide-azide mixture:

$$2Mn^{2+} + 4OH^- + \boxed{O_2} \rightarrow \boxed{2MnO_2}_{(s)} + 2H_2O$$

After transport to lab sample is acidified with $H_2SO_4$ (dissolves $Mn^{4+}$ floc), and $Mn^{4+}$ oxidizes iodine ions:

$$\boxed{2MnO_2} + 4I^- + 8H^+ \rightarrow 2Mn^{2+} + \boxed{2I_2} + 4H_2O$$

$I_2$ is then titrated with thiosulfate and starch indicator:

$$\boxed{2I_2} + \boxed{4S_2O_3^{2-}} \rightarrow 2S_4O_6^{2-} + 4I^-$$

### (2) Electrometric Method

Dissolved oxygen can be measured by a special sensor kept in an electrochemical cell, the cell consists of a sensing electrode, a reference electrode and a supporting electrolyte, a semi-permeable membrane, which served dual function, It separates the water sample from the electrolyte, and at the same time, permits only the DO to diffuse from the water sample through the membrane into the supporting electrolyte.

**Figure 12.1: Dissolved Oxygen meter**

Simply place the probe in the sample and allow the meter time to settle the final measurement value. The diffusion current created by migration of oxygen through a permeable membrane is linearly proportional to the concentration of molecular oxygen in the sample.

## 12.4 Materials Required

### 12.4.1 Apparatus required

1. Burette
2. Pipette
3. Conical flask
4. Measuring cylinder
5. Funnel

### 12.4.2 REAGENTS

1. Manganese sulfate
2. alkali-iodide-azide
3. concentrated sulfuric acid
4. starch solution
5. Sodium thiosulfate

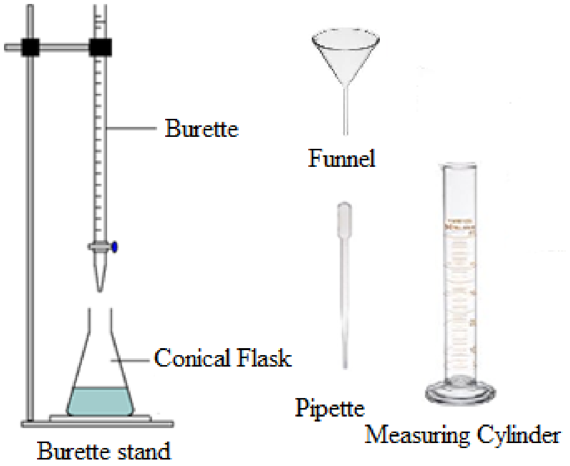


**Figure 12.27: Apparatus required for DO**

## 12.5 PROCEDURE

1. Take two 300-mL glass stoppered BOD bottle and fill it with sample to be tested. Avoid any kind of bubbling and trapping of air bubbles. Remember – no bubbles!
2. Add 2mL of manganese sulfate to the BOD bottle by inserting the calibrated pipette just below the surface of the liquid.
3. Add 2 mL of alkali-iodide-azide reagent in the same manner.
4. Squeeze the pipette slowly so no bubbles are introduced via the pipette (The pipette should be dipped inside the sample while adding the above two reagents. If the reagent is added above the sample surface, you will introduce oxygen into the sample).
5. If oxygen is present, a brownish-orange cloud of precipitate or floc will appear.
6. Allow it to settle for sufficient time in order to react completely with oxygen.
7. Add 2 mL of concentrated sulfuric acid just above the surface of the sample.
8. Carefully stopper and invert several times to dissolve the floc.
9. Stored the Sample for up to 8 hours if kept in a cool, dark place.
10. Rinse the burette with sodium thiosulphate and then fill it with sodium thiosulphate. Measure out 203 mL of the solution from the bottle and transfer to an conical flask. Titration needs to be started immediately after the transfer to conical flask.
11. Titrate it against sodium thiosulphate using starch as indicator. (Add 3 - 4 drops of starch indicator solution) until disappearance of the blue color.
12. Note down the volume of sodium thiosulphate solution added which gives the dissolved oxygen in 7.9 mL
13. Repeat the titration for concordant values.

***End point: Disappearance of the blue color to colorless**

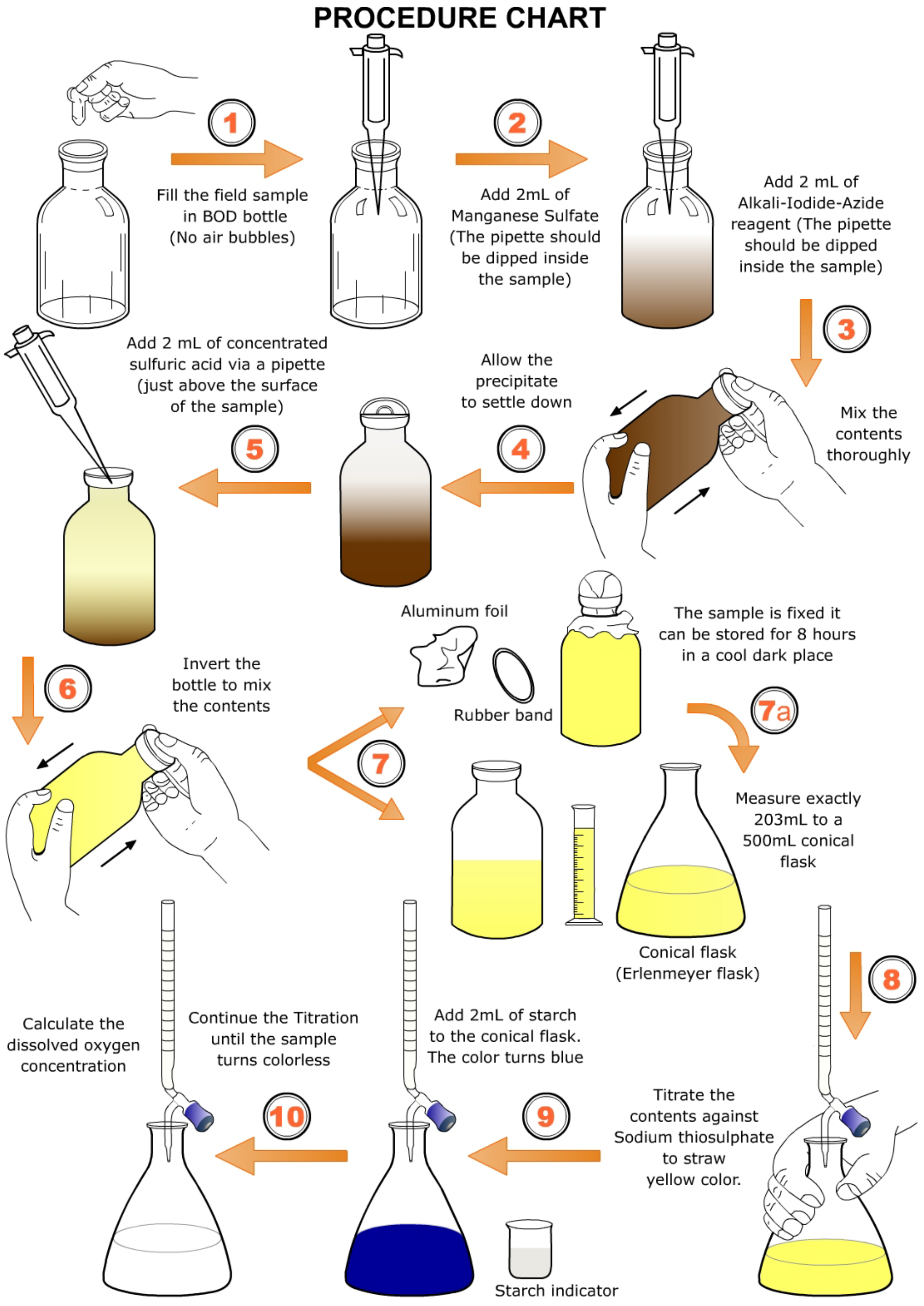


**Figure 12.28: Procedure of DO experiment**

## 12.6 DATA SHEET

DETERMINATION OF Calcium Hardness DATA SHEET

Date Tested: August 30, 2010

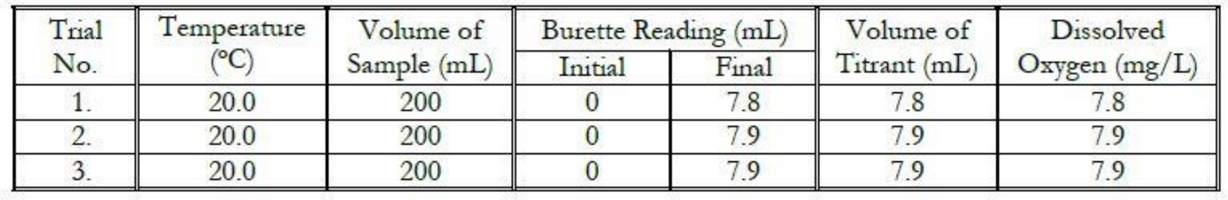
Tested By: CEM Class, Group A

Project Name: CEM, NITTTR Lab

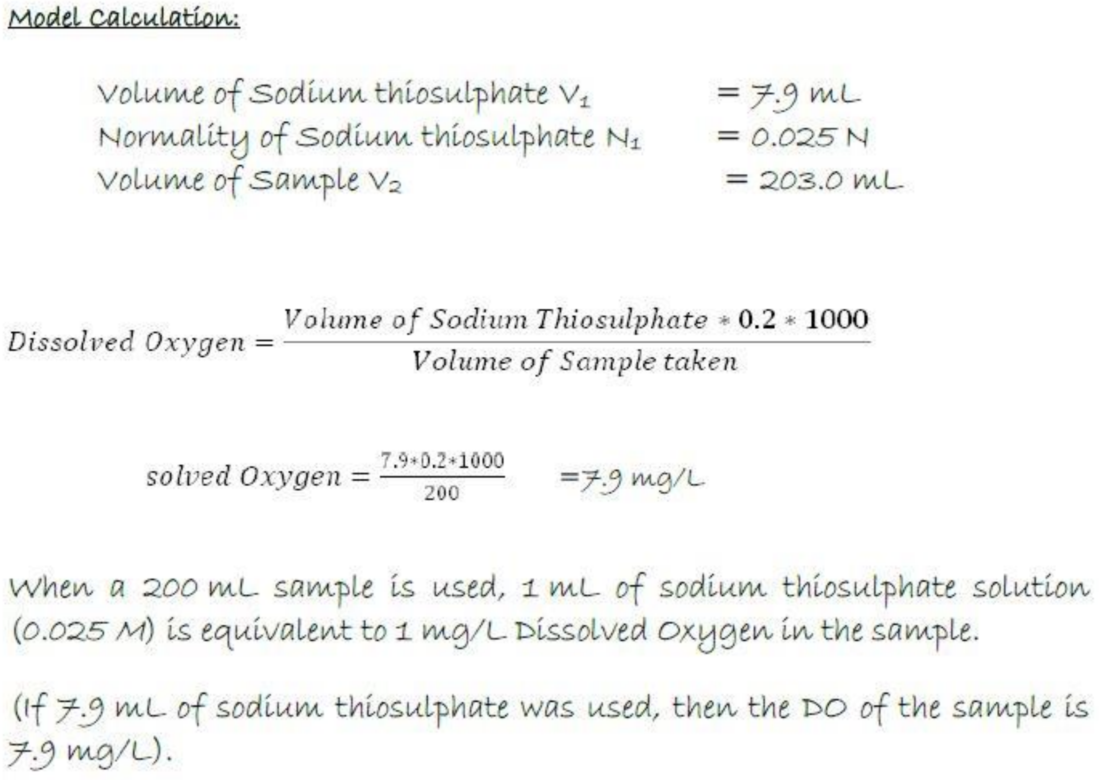
Sample Number: BH1

Sample Location: Perungudi (Lat 12’ 57’’ 31.74 & Long 80’14’’ 8.82)

Sample Description: Surface water



| Trial No. | Temperature (°C) | Volume of Sample (mL) | Burette Reading (mL) | | Volume of Titrant (mL) | Dissolved Oxygen (mg/L) |
|---|---|---|---|---|---|---|
| | | | Initial | Final | | |
| 1. | 20.0 | 200 | 0 | 7.8 | 7.8 | 7.8 |
| 2. | 20.0 | 200 | 0 | 7.9 | 7.9 | 7.9 |
| 3. | 20.0 | 200 | 0 | 7.9 | 7.9 | 7.9 |



Model Calculation:

Volume of Sodium thiosulphate $V_1$ = 7.9 mL

Normality of Sodium thiosulphate $N_1$ = 0.025 N

Volume of Sample $V_2$ = 203.0 mL

$$Dissolved\ Oxygen = \frac{Volume\ of\ Sodium\ Thiosulphate * 0.2 * 1000}{Volume\ of\ Sample\ taken}$$

$$solved\ Oxygen = \frac{7.9*0.2*1000}{200} \quad = 7.9\ mg/L$$

When a 200 mL sample is used, 1 mL of sodium thiosulphate solution (0.025 M) is equivalent to 1 mg/L Dissolved Oxygen in the sample.

(If 7.9 mL of sodium thiosulphate was used, then the DO of the sample is 7.9 mg/L).

# EXPERIMENT #13: DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND (BOD)

## 13.1 AIM

To determine biochemical oxygen demand in the given water sample.

## 13.2 INTRODUCTION

The biochemical oxygen demand (BOD) is the amount of dissolved oxygen needed by aerobic organisms to break the organic materials present in the given water sample at certain temperature (20°C) over a specific period of time (5 days), then determining the change in dissolved oxygen content. Usually the BOD test is used to measure the strength of organic pollution and it is the only parameter that give an idea of the biodegradability of any sample and self-purification capacity of rivers and streams.

Ordinary domestic sewage may have a BOD of 200 mg/L. Any effluent to be disposal of water should have BOD less than 30 mg/L. Drinking water usually has a BOD of less than 1 mg/L. But, when BOD value reaches 5 mg/L, the water is doubtful in purity.

## 13.3 PRINCIPLE

The sample is filled in BOD bottle and diluted with a known amount of dilution water and the dissolved oxygen of the sample is determined before and after five days of incubation at 20°C and the BOD is calculated from the difference between initial and final DO.

## 13.4 Materials Required

### 13.4.1 Apparatus required

1. BOD Incubator
2. 300mL BOD bottles
3. Burette
4. Pipette
5. Conical flask
6. Measuring cylinder
7. Funnel

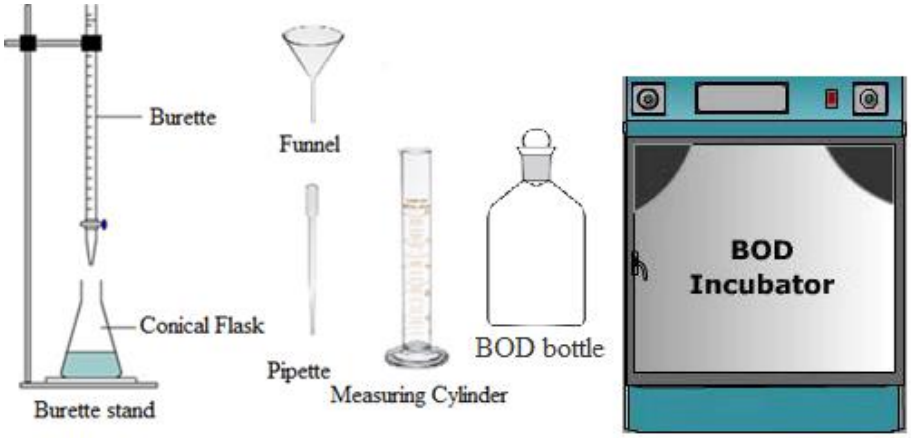


**Figure 12.1: Apparatus required for BOD experiment**

### 13.4.2 REAGENTS

**For diluted water preparation:**

1. Manganese sulfate ($MgSO_4$)
2. Calcium Chloride solution
3. Magnesium Sulphate solution
4. Phosphate buffer solution
5. Ferric chloride solution
6. Distilled water

**For DO determination:**

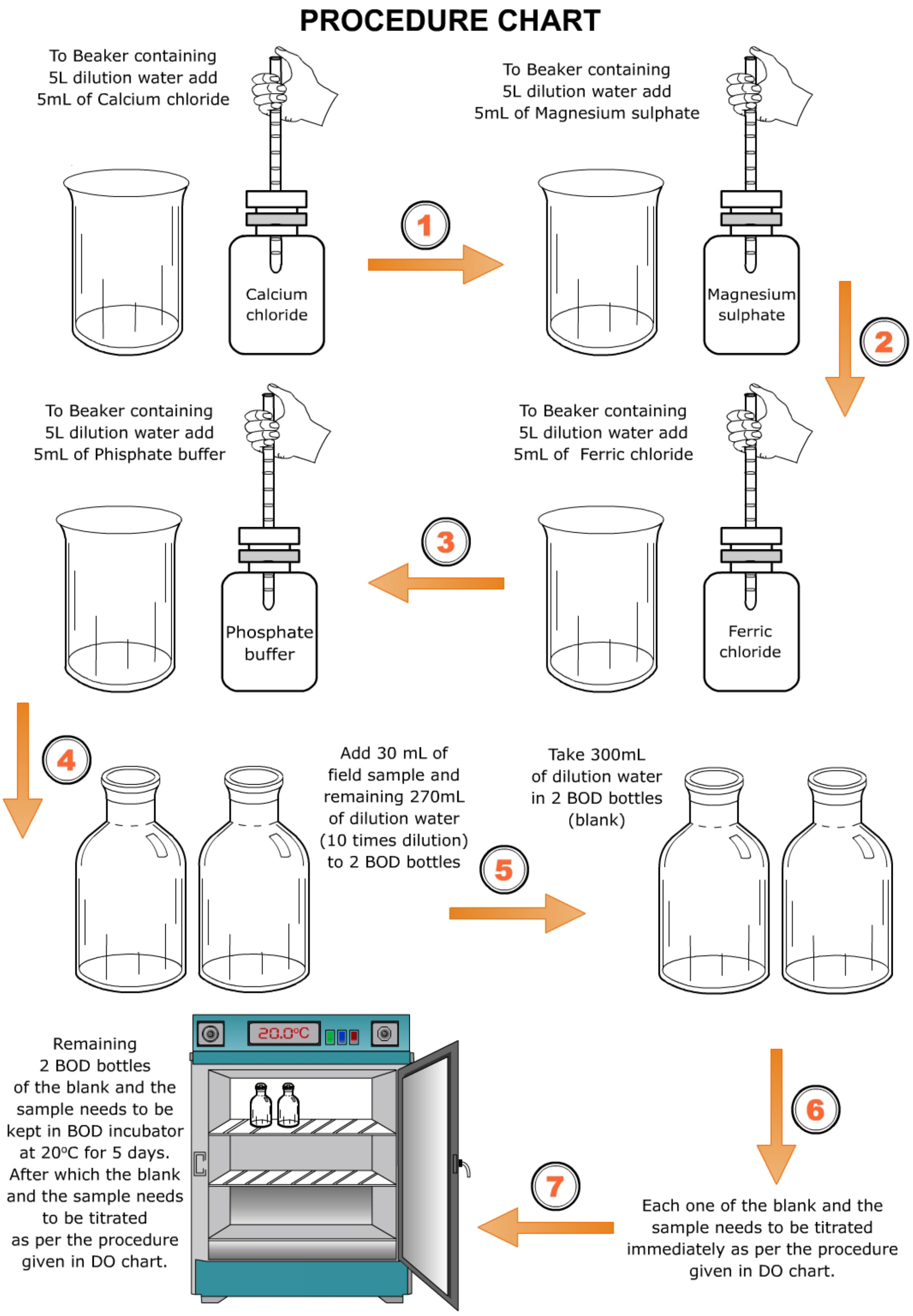
1. Manganese sulfate
2. alkali-iodide-azide
3. concentrated sulfuric acid
4. Sodium thiosulphate solution
5. Starch indicator

### 13.5 PROCEDURE

1. Aerated 5L of distilled water by bubbling compressed air through water.
2. Prepare dilution water by adding 5ml of phosphate buffer, 5ml of magnesium sulphate solution, 5ml of calcium chloride solution and 5ml of ferric chloride solution into aerated distilled water.
3. Take 4-glass BOD bottles 300mL (two for the sample and two for the blank).
4. Dilute the sample with the distilled water and mix the contents well.
5. Take 10 mL of the sample into 2-BOD bottles and the fill the remaining quantity with the dilution water.
6. The remaining 2-BOD bottles are for blank, to these bottles add dilution water alone.
7. Now preserve one blank solution bottle and one sample solution bottle in a BOD incubator at 20ºC for five days.
8. The other 2-bottles (one blank and one sample) needs to be analyzed immediately.
9. Prevent any air entry into the bottles.
10. Add 2mL of manganese sulfate into BOD bottle by inserting the pipette below the surface of the liquid, and then add 2 mL of alkali-iodide-azide reagent in the same manner.
11. Allow it to settle for sufficient time in order to react completely with oxygen.
12. When this floc has settled to the bottom, shake the contents thoroughly
13. Add 2 mL of concentrated sulfuric acid above the surface of the sample.
14. Carefully stopper and invert several times to dissolve the floc.
15. Rinse the burette with sodium thiosulphate and then fill it with sodium thiosulphate. Measure out 203 mL of the solution from the bottle and transfer to a conical flask.
16. Titration needs to be started immediately after the transfer to conical flask.
17. Titrate the solution with standard sodium thiosulphate solution until the yellow color of liberated Iodine is almost faded out. (Pale yellow color)
18. Add 1 mL of starch solution, and titrate until the blue color disappears to colorless.

19. Note down the volume of sodium thiosulphate solution added , which gives the D.O
20. After five days, take out the bottles from the BOD incubator and repeat procedure from 10 to 20 to the sample and the blank to find DO after 5-days.

***End point: Disappearance of the blue color to colorless**



PROCEDURE CHART

To Beaker containing
5L dilution water add
5mL of Calcium chloride

To Beaker containing
5L dilution water add
5mL of Magnesium sulphate

To Beaker containing
5L dilution water add
5mL of Phisphate buffer

To Beaker containing
5L dilution water add
5mL of Ferric chloride

Add 30 mL of
field sample and
remaining 270mL
of dilution water
(10 times dilution)
to 2 BOD bottles

Take 300mL
of dilution water
in 2 BOD bottles
(blank)

Remaining
2 BOD bottles
of the blank and the
sample needs to be
kept in BOD incubator
at 20ºC for 5 days.
After which the blank
and the sample needs
to be titrated
as per the procedure
given in DO chart.

Each one of the blank and the
sample needs to be titrated
immediately as per the procedure
given in DO chart.

Figure 12.2: Procedure of BOD experiment

## 13.6 DATA SHEET

DETERMINATION OF Calcium Hardness DATA SHEET

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Date Tested: August 30, 2010

Tested By: CEM Class, Group A

Project Name: CEM, NITTTR Lab

Sample Number: BH1

Sample Location: Perungudi (Lat 12’ 57’’ 31.74 & Long 80’14’’ 8.82)

Sample Description: Surface water

| Trial No. | Day | Volume of Sample (mL) | Burette Reading (mL) | | Volume of Titrant (mL) | Dissolved Oxygen (mg/L) |
|---|---|---|---|---|---|---|
| | | | Initial | Final | | |
| Blank | 0 | 200 | 0 | 8.2 | 8.2 | 8.2 |
| 3. | 0 | 200 | 0 | 7.9 | 7.9 | 7.9 |
| 4. | 0 | 200 | 0 | 7.9 | 7.9 | 7.9 |
| Blank | 5 | 200 | 0 | 8.0 | 8.0 | 8.0 |
| 1. | 5 | 200 | 0 | 3.2 | 3.2 | 3.2 |
| 2. | 5 | 200 | 0 | 3.2 | 3.2 | 3.2 |

Specimen Calculation:

Initial DO of the diluted sample, $D_0$ = 7.9 mL

DO at the end of 5 days for the diluted sample, $D_5$ = 3.2 mL

Blank correction = $C_0$ - $C_5$, BC = 0.2 mL

Initial DO of the blank, $C_0$ = 8.2 mL

DO at the end of 5 days for the blank, C5 = 8.0 mL

**Biochemical Oxygen Demand**

$$= \frac{\{D0 - D5 - BC\} \times \text{Volume of the diluted sample}}{\text{Volume of sample taken}}$$

**Biochemical Oxygen Demand (mg/L)**

$$= (7.9 - 3.2 - 0.2) \times 200 / 10$$

$$= 4.5 \times 200 / 10$$

$$= 90 \text{ mg/L}$$