

**KING FAISAL UNIVERSITY**  
**College Of Engineering**

**DEPARTMENT OF CIVIL & ENVIRONMENTAL**  
**ENGINEERING**

**CEE350: ENVIRONMENTAL ENGINEERING**

**“Lab Manual”**



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## Major Topics covered and schedule in weeks:

Topic	Week #	Courses Covered
Introduction to the Lab.	1	CEE350_Lab
Lab regulations & safety rules.	2	CEE350_Lab
Determination of pH Water.	3	CEE350
Determination of EC of Water.	4	CEE350
Determination of Acidity in Water.	5	CEE350
Determination of Alkalinity in Water.	6	CEE350
Determination of Total Hardness	7	CEE350
Determination of Calcium Hardness	8	CEE350
Determination of Chloride	9	CEE350
Determination of Nitrate	10	CEE350
Colorimetric Analysis	11	CEE350
Determination of Turbidity of Water.	12	CEE350
Determination of Solids in Water.	13	CEE350
Air Quality	14	CEE452
Final Exam	15	

### Specific Outcomes of Instruction (Lab Learning Outcomes):

1. Perform common environmental experiments relating to water and wastewater quality, and know which tests are appropriate for given environmental problems. [1]
2. Statistically analyze and interpret laboratorial results. [6]
3. Apply the laboratorial results to problem identification, quantification, and basic environmental design and technical solutions. [1]
4. Demonstrate the ability to write clear technical laboratorial reports. [6]
5. Demonstrate the ability to work in groups. [5]

### Student Outcomes (SO) Addressed by the Lab:

z	Outcome Description	Contribution
	General Engineering Student Outcomes	
1.	an ability to identify, formulate, and solve complex engineering problems by applying principles of engineering, science, and mathematics	L
2.	an ability to apply engineering design to produce solutions that meet specified needs with consideration of public health, safety, and welfare, as well as global, cultural, social, environmental, and economic factors	
3.	an ability to communicate effectively with a range of audiences	
4.	an ability to recognize ethical and professional responsibilities in engineering situations and make informed judgments, which must consider the impact of engineering solutions in global, economic, environmental, and societal contexts	
5.	an ability to function effectively on a team whose members together provide leadership, create a collaborative and inclusive environment, establish goals, plan tasks, and meet objectives	L
6.	an ability to develop and conduct appropriate experimentation, analyze and interpret data, and use engineering judgment to draw conclusions	L
7.	an ability to acquire and apply new knowledge as needed, using appropriate learning strategies	

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## Experiment 1: Measurement of pH of Water

### I. Objective:

To determine the pH of the given samples of water using the pH meter.

### II. Test Standard:

Test procedure is in accordance to: APHA Standard Methods for examination of Water and Wastewater – 23<sup>rd</sup> Edition. Method **4500-H<sup>+</sup> B**.

### III. Theory:

pH as defined by Sorenson is negative logarithm of hydrogen ion concentration. At a given temperature, the acidic or basic character of a solution is indicated by pH or hydrogen ion activity. Actually the alkalinity and acidity of the water is something different. The alkalinity and acidity are the acid and base neutralizing capacities of water and are usually expressed as milligrams of CaCO<sub>3</sub> per liter. The pH is important in every phase of environmental engineering practice. In water treatment process it is a factor that is to be considered in chemical coagulation, disinfection, water softening and corrosion control. Coagulation means the mixing of alum like chemicals to make flocks and to increase the settlement of colloidal particles in water purification. The efficiency of the chemical coagulant like alum depends upon the pH of water and it is most efficient in the pH range of 6.5 to 8.5. Similarly, chlorine is added to water to kill the bacteria and other microorganism and this process is known as disinfection. The efficiency of chlorine is also dependent on the pH of water. So the determination and then the required adjustment of pH is a must for the efficient use of coagulant and disinfectants.

The pure water dissociates to yield a concentration of hydrogen ions equal to about 10<sup>-7</sup> mole/l.



The amount of hydroxyl ions is equal to the hydrogen ions, so 10<sup>-7</sup> mole of hydroxyl ion is produced simultaneously. The equilibrium equation gives

$$k = \frac{[H^+][OH^-]}{[H_2O]}$$

As the concentration of water is so extremely large and is diminished so much little by the slight degree of ionization it may be considered as constant and the above equation can be written as:

$$k_w = [H^+][OH^-]$$

For pure water at 25 °C:  $\{H^+\} \{OH^-\} = 10^{-7} \times 10^{-7} = 10^{-14}$

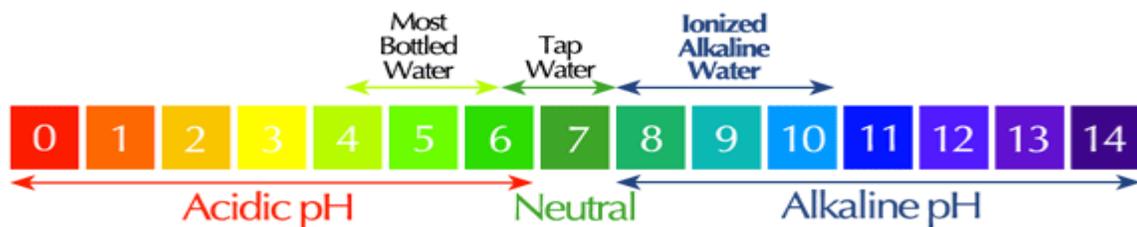
This is known as the ion product or ionization constant for water.

When an acid is mixed in water, it ionizes in the water and the H ion activity increases. Consequently, the OH ion activity must decrease according to the ionization constant. For example if acid is added to increase the {H<sup>+</sup>} to 10<sup>-2</sup>, the {OH<sup>-</sup>} must decrease to 10<sup>-12</sup> (10<sup>-2</sup> × 10<sup>-12</sup> = 10<sup>-14</sup>)

Similarly if a base is added to increase the {OH<sup>-</sup>} to 10<sup>-3</sup>, the {H<sup>+</sup>} decreases to 10<sup>-11</sup>. However the {H<sup>+</sup>} or the {OH<sup>-</sup>} can never be reduced to zero no matter how basic or acidic the solution may be.

Designating the hydrogen ion concentration in terms of molar concentration is cumbersome and to overcome this difficulty, Sorenson gave such value in terms of their negative logarithms as pH, so

$$pH = -\log[H^+] = \log \frac{1}{[H^+]}$$



The pH scale is represented as ranging from 0 to 14 with pH 7 at 25°C designating absolute neutrality. pH lesser than 7 is acidic and more than 7 is a basic solution.

#### Significance:

- 1 In the field of water supplies, it is a factor that must be considered in chemical coagulation, disinfection, water softening and corrosion control.
- 2 In wastewater treatment employing biological processes, pH must be controlled within a range favorable to the particular organisms involved.
- 3 In chemical processes used to coagulate wastewaters, dewater sludge's, or oxidize substances, such as cyanide ion, require that the pH be controlled within rather narrow limits.

#### Principle:

pH is measured by pH meter using a glass electrode which generates a potential varying linearly with the pH of the solution in which it is immersed. Calomel or Ag / AgCl / KCl reference electrode is usually used for determination.

#### IV. Apparatus:

1. pH meter with electrodes
2. Buffer solution
3. 50 ml Erlenmeyer flask & washing bottle with distilled water



## V. Procedure:

1. Calibrate the electrodes with two standard buffer solutions of pH 4.0 and 9.2 (Normally calibration is not required since it will be done periodically)
2. Remove the pH electrode from storage bottle. Rinse with distilled water to remove any impurities.
3. Press ON to switch meter on
4. Press MODE key to select pH measurement mode.
5. Dip the probe into the sample for which the pH measurement is required. Also dip the conductivity cell also as it contains temperature sensor. Take care to see that the glass bulb of the electrode is completely immersed in the sample. Stir gently to create homogeneous sample.
6. Allow time for the reading to stabilize. Note the reading on the display after ready indicator is appears.
7. Follow the same procedure (5-6) for different samples. Rinsing the electrode before immersing in the next solution.

### *For Calibration:*

1. Prepare Buffer solution by dissolving the content of buffer powder or tablet in 100ml of water to get the required buffer solution.
2. Press MODE key to select pH mode
3. Rinse the probe in distilled water. Dip the probe in the buffer solution.
4. Press CAL/MEAS to enter pH calibration mode.
5. Wait for the measured pH value to stabilize. And READY indicator will appear. Press ENTER to confirm calibration. The meter is now calibrated to the current buffer
6. Follow the same steps for the second buffer.

pH can also be found out using pH paper or universal indicator. Follow the instructions printed on the respective products.

## VI. Experimental Work:

Sample No.	Temperature	pH reading
1		
2		
3		

*[Note: you need to present your results on bar chart diagram]*

### **Drinking water standards for pH:**

*Acceptable: 7.0-8.5*

*Cause for rejection: < 6.5 or > 9.2*

## Experiment 2: Measurement of Conductivity of Water

### I. Objective:

To determine the EC of the given sample of water using the EC meter.

### II. Test Standard:

Test procedure is in accordance to: APHA Standard Methods for examination of Water and Wastewater – 23<sup>rd</sup> Edition. Method **2510 B** & **2520 B**.

### III. Theory:

Salinity of water can be determined by measuring its electrical conductivity. The conductivity of a solution is a measure of its ability to carry an electrical current, and varies both with the number and with the type of ions the solution contains. Most dissolved inorganic substances in water supplies are in the ionized form (like  $\text{Cl}^-$ ,  $\text{Na}^+$ ,  $\text{Ca}^{2+}$  and  $\text{SO}_4^{2-}$ ) and so contribute to the specific conductance. The electrical conductivity (EC) of water is used as a rapid measure replacing total dissolved solids (TDS) concentration. The EC measurements give a practical estimate of the variation in dissolved minerals content of a specific water supply. Also, by the use of an empirical factor, specific conductance can allow a rough estimate to be made of the dissolved mineral content of water samples.

The electrical conductivity can be expressed as Micro-Siemens per centimeter ( $\mu\text{S}/\text{cm}$ ). Values for salinity are also reported as TDS in  $\text{mg}/\text{l}$ . For most agricultural irrigation purposes, the values for EC and TDS are related to each other and can be converted within an accuracy of about 10% using:

$$\text{TDS (mg/l)} \approx \text{EC } (\mu\text{S/cm}) \times 0.64$$

The used conversion factor of 0.64 can vary in the range (0.55 to 0.7)

Salinity is an important parameter in the analysis of drinking water, irrigation water, industrial water and seawater. Salinity can also be reported as parts per thousands (ppt) [gram of solids/ kg of solution; gram of solids/ 1000 gram of solution].

**Conductivity Measurements:** The conductivity is measured in the laboratory in term of resistance measured in ohms. The electric resistance of a conductor is inversely proportional to its cross sectional area and directly proportional to its length. The magnitude of the resistance measured in an aqueous solution therefore depends on

the characteristics of the conductivity cell used. Specific resistance is the resistance of a cube of 1cm. In aqueous solutions, such a measurement is seldom made because of the difficulties in fabrication of electrode. Actually the electrodes measure a given fraction of the specific resistance known as the cell constant C

$$C = \text{Measured resistance, } R_m / \text{Specific resistance, } R_s$$

The reciprocal of resistance is conductance. It measures the ability to conduct a current and is expressed in reciprocal of ohms i.e. mhos. In water analysis generally micromhos is used. Knowing the cell constant the measured conductance is converted to the specific conductance or conductivity,  $K_s$ , as the reciprocal of the specific resistance.

$$K_s = 1/R_s = C/R_m$$

**Health effects:** High levels of total dissolved solids may impart an objectionable taste to drinking water. Chloride, in particular, has a low taste threshold. Sodium sulfate and magnesium sulfate levels above 250 mg/l in drinking water may produce a laxative effect. Excess sodium may affect those restricted to low sodium diets and pregnant women suffering from toxemia. The limit of TDS in drinking water is less than 500 mg/l.

**Irrigation effects:** Salinity of irrigation water determined by measuring its electrical conductivity is the most important parameter in determining the suitability of water for irrigation. The presence of salts affects plant growth in three ways: (1) osmotic effects, caused by the total dissolved salt concentration in the soil water; (2) specific ion toxicity caused by the concentration of individual ions, and (3) soil particle dispersion, caused by high sodium and low salinity. With increasing salt salinity in the root zone, plants expend more of their available energy on adjusting the salt concentration within the tissue (osmotic adjustment) to obtain needed water from the soil. The influence of the salt on the crops depends on the type of the plant as some plants are less sensitive to high salts concentration (known as salt tolerant). The limit of TDS in irrigation water is in generally in the range 500 – 1000 mg/l (dependent upon crop sensitivity).

**Industrial effects:** Dissolved solids may corrode metallic surfaces. Salt in intake water may interfere with chemical processes within the plant (factory). Also, high salts may affect the taste of beverages. The limit of TDS in industrial water depends on the type of industry, for instance, for the production of fine paper TDS should be less than 200 mg/l; for Ground wood paper less than 850 mg/l. Industry can de-ionize water to meet requirements; economics\ is the limiting factor.

#### IV. Apparatus:

(a) Conductivity meter: This is an instrument consisting of a source of alternating current, a Wheatstone bridge, a null indicator and a conductivity cell. Generally an instrument capable of measuring conductivity with an accuracy of 1% or 1  $\mu$  mhos/cm is used. A thermometer capable of reading up to 0.1 °C within a range of 15 to 30 °C is used.

(b) Conductivity Cell: Platinum-electrode type conductivity cells containing platinized electrodes are used depending upon the expected range of conductivity. Non platinum-electrode type conductivity cells containing electrodes constructed from durable metals like stainless steel are used for continuous monitoring systems.

#### Reagents:

(a) Conductivity water: Pass distilled water through a mixed bed deionizer and discard first liter. Conductivity should be less than 1  $\mu$  mhos/cm.

(b) Standard Potassium Chloride Solution (KCl, 0.01M), Dissolve 745.6 mg of anhydrous KCl in conductivity water and dilute to 1000 ml at 25 °C. This is the standard reference solution having a conductivity of 1413  $\mu$  mhos/cm at 25 °C, useful for the cell constants between 1 and 2.



#### V. Procedure:

(i) *Determination of Cell Constant:*

Wash the conductivity cell with 0.01 M KCl solution. Adjust the temperature of the standard KCl at 25± 0.1°C. Measure resistance of the KCL and note the temperature. The Cell Constant,  $C = (0.001413) (R_{KCl}) [1+0.0191(t-25)]$ .

(ii) *Conductivity Measurement*

Rinse cell with the sample. Adjust temperature of the sample to 25± 0.1°C. Measure sample resistance or conductivity and the temperature. If the temperature deviates from 25°C, the corrected conductivity shall be as follows

$$K = (K_m) C / (1+0.019(t-25))$$

$K_m$  is the measured conductivity at  $t$  (°C).

#### VI. Experimental Work:

Water sample No.	Temperature	EC (dS/cm)	EC ( $\mu$ mhos/cm)	Total dissolved solids (TDS) in mg/l= EC x 0.64

[Note: you need to present your results on bar chart diagram]

#### Comments:

(i) Knowing the conductivity the total dissolved solids can be calculated.

(ii) Continuous monitoring of the conductivity of a flowing stream of water or waste water reflects any sudden change and the probable cause can be detected.

## Experiment 3: Determination of Water Acidity

### I. Objective:

To determine the acidity of the given samples of water.

### II. Test Standard:

Test procedure is in accordance to: APHA Standard Methods for examination of Water and Wastewater – 23<sup>rd</sup> Edition. Method **2310 B**

### III. Theory:

Acidity of water is its quantitative capacity to neutralize a strong base to a designated pH. Strong minerals acids, weak acids such as carbonic and acetic acid and hydrolyzing salts such as ferric and aluminum sulfates may contribute to the measured acidity. According to the method of determination, acidity is important because acid contributes to corrosiveness and influences certain chemical and biological processes. It is the measure of the amount of base required to neutralize a given sample to the specific pH.

Hydrogen ions present in a sample as a result of dissociation or hydrolysis of solutes are neutralized by titration with standard alkali. The acidity thus depends upon the end point pH or indicator used. Dissolved CO<sub>2</sub> is usually the major acidity component of unpolluted surface water. In the sample, containing only carbon dioxide-bicarbonate-carbonate, titration to pH 8.3 at 25°C corresponds to stoichiometric neutralization of carbonic acid to carbonate. Since the color change of phenolphthalein indicator is close to pH 8.3, this value is accepted as a standard end point for the titration of total acidity. For more complex mixture or buffered solution fixed end point of pH 3.7 and pH 8.3 are used. Thus, for standard determination of acidity of wastewater and natural water, methyl orange acidity (pH 3.7) and phenolphthalein acidity (pH 8.3) are used. Thus, in determining the acidity of the sample, the volumes of standard alkali required to bring about color change at pH 8.3 and at pH 3.7 are determined.

#### Significance of Acidity:

1. Acidity present due to free carbon-di-oxide has no significance from public health viewpoint, but water containing mineral acidity are unacceptable.
2. Acidic waters pose corrosion problem and interfere in water softening and water treatment process.
3. For biological treatment of wastewater, pH must be between 6 and 9.5. Hence, highly acidic waters require adjustment of pH before treatment.

#### IV. Apparatus:

Both carbon-di-oxide and mineral acidity can be measured by means of standard solutions of alkaline reagents like sodium hydroxide.

Mineral acidity is measured by titration to a pH of about 4.5 using methyl orange as indicator. The carbon-di-oxide acidity present in the sample can be neutralized completely by titration to pH of 8.3 using phenolphthalein end point which measures both mineral and CO<sub>2</sub> acidity

#### Reagents:

- i) Standard sodium hydroxide (0.02N).
- ii) Indicators – Methyl orange & Phenolphthalein.



#### V. Procedure:

##### For Mineral acidity:

- i) Take 50ml of sample in a conical flask and add 2 drops of methyl orange indicator.
- ii) If the solution turns **yellow**, the methyl orange acidity is absent. If it turns **faint orange** color, titrate with 0.02N NaOH, which is in the Burette.
- iii) At the end point color changes **from faint orange to yellow**.
- iv) Note down the volume of titrant (NaOH).

$$\text{Mineral acidity} = \frac{\text{Average volume of NaOH} \times 1000}{\text{Volume of sample}} \text{ (units in mg/l as CaCO}_3\text{)}$$

*Note: After adding the Methyl orange if faint orange or pink color fails to appear, then, Mineral acidity is absent.*

##### For Total acidity:

- a) Take 50ml of sample in a conical flask and add 2 drops of phenolphthalein indicator.
- b) Initially the solution will be **colorless**.
- c) Titrate with 0.02N NaOH until the appearance of **pink color**.
- d) Note down the volume of NaOH.

$$\text{Total acidity} = \frac{\text{Average volume of NaOH} \times 1000}{\text{Volume of sample}} \text{ (units in mg/l as CaCO}_3\text{)}$$

$$\text{CO}_2 \text{ acidity} = \text{Total acidity} - \text{mineral acidity (units in mg/l as CaCO}_3\text{)}$$

## VI. Experimental Work:

Calculation and tabulation:

### 1- For Mineral Acidity

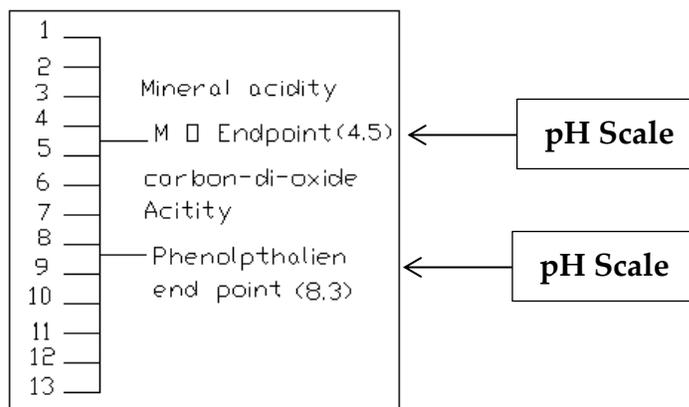
Sample	Trial No	Initial reading (in ml)	Final reading (in ml)	Average vol. Of NaOH added (in ml)
Lab sample	1			
	2			
Tap water	1			
	2			

### 2- For Total Acidity:

Sample	Trial No	Initial reading (in ml)	Final reading (in ml)	Average vol. Of NaOH added (in ml)
Lab sample	1			
	2			
Tap water	1			
	2			

### 3- Summarized Results:

sample	Total acidity mg/l as CaCO <sub>3</sub>	Mineral acidity mg/l as CaCO <sub>3</sub>	CO <sub>2</sub> acidity mg/l as CaCO <sub>3</sub>
Lab sample			
Tap water			



## Experiment 4: Determination of Water Alkalinity

### I. Objective:

To determine the alkalinity of the given sample of water.

### II. Test Standard:

Test procedure is in accordance to: APHA Standard Methods for examination of Water and Wastewater – 23<sup>rd</sup> Edition. Method **2320 B**

### III. Theory:

The alkalinity of water is a measure of its capacity to neutralize acids. It is primarily due to salts of weak acids, although weak or strong bases may also contribute. Alkalinity is usually imparted by bicarbonate, carbonate and hydroxide. It is measured volumetrically by titration with 0.02 N sulfuric acid and is reported in terms of  $\text{CaCO}_3$  equivalent. For samples whose initial pH is above 8.3, the titration is conducted in two steps. In the first step, the titration is conducted until the pH is lowered to 8.2, the point at which phenolphthalein indicator turns from pink to colorless. This value corresponds to the points for conversion of carbonate to bicarbonate ion. The second phase of titration is conducted until the pH is lowered to 4.5, corresponds to methyl orange end point, which corresponds to the equivalence points for the conversion of bicarbonate ion to carbonic acid.

#### Sanitary significance:

1. Highly alkaline water is usually non-palatable from consumers' point of view.
2. Alkalinity values provide guidance in applying proper doses of coagulants in water and wastewater treatment processes particularly in coagulation, softening and operational control of anaerobic digestion.

### IV. Apparatus:

Alkalinity can be measured by nitrating the sample with standard HCl or  $\text{H}_2\text{SO}_4$ . Titration to pH 8.3 will result in decolorization of Phenolphthalein indicator and will be complete neutralization of hydroxides and half of carbonates, while to pH 4.5 a sharp change from yellow to pink or orange will indicate neutralization of total alkalinity i.e.,  $\text{OH}^-$ ,  $\text{CO}_3^{2-}$  and  $\text{HCO}_3^-$ .

#### Reagents:

- 1) Standard HCl 0.02N
- 2) Indicators (Phenolphthalein & Methyl orange)



## V. Procedure:

### i) Phenolphthalein or Partial alkalinity (P)

Pipette out 50ml of sample in a conical flask and add 2-3 drops of Phenolphthalein indicator. Immediately **pink color** develops due to high alkalinity. Titrate with 0.02 HCl until the **color disappears**. Note the volume of HCl added.

$$\text{Phenolphthalein alkalinity} = \frac{\text{Average volume of HCl}}{\text{Volume of sample}} \times 1000 \text{ (units in mg/l as CaCO}_3\text{)}$$

*Note: After adding the Phenolphthalein if **pink color fails to appear**, then, Phenolphthalein alkalinity is absent.*

### ii) Methyl Orange or Total alkalinity (T)

Pipette out 50ml of sample in a conical flask and add 2-3 drops of Methyl orange indicator. Titrate with 0.02 HCl until the appearance of **orange pink color**. Note the volume of HCl added.

$$\text{Total alkalinity} = \frac{\text{Average volume of HCl}}{\text{Volume of sample}} \times 1000 \text{ (units in mg/l as CaCO}_3\text{)}$$

iii) Methyl orange alkalinity = Total alkalinity – Phenolphthalein alkalinity.

## VI. Experimental Work:

### i) Phenolphthalein Alkalinity Data Table:

Sample	Trial No	Initial reading (in ml)	Final reading (in ml)	Average vol. of HCl run down in (ml)
Lab sample	1			
	2			
Tap water	1			
	2			

### ii) Total Alkalinity Data Table:

Sample	Trial No	Initial reading (in ml)	Final reading (in ml)	Average vol. of HCl run down in (ml)
Lab sample	1			
	2			
Tap water	1			
	2			

**Different Alkalinities:**

Results	Caustic alkalinity (OH <sup>-</sup> )	Carbonates CO <sub>3</sub> <sup>2-</sup>	Bi-Carbonates HCO <sub>3</sub> <sup>-</sup>
If P = 0	0	0	T
If P < 1/2 T	0	2P	T-2P
If P = 1/2 T	0	2P	0
If P > 1/2 T	2P - T	2(T-P)	0
If P = T	T	0	0

**Results (all units in mg/l as CaCO<sub>3</sub>):**

Sample No.	Partial Alk.	Total Alk.	Caustic alkalinity (OH <sup>-</sup> )	Carbonates CO <sub>3</sub> <sup>2-</sup>	Bi-Carbonates HCO <sub>3</sub> <sup>-</sup>
Lab sample					
Tap water					

**Drinking water standards for Alkalinity:**

Acceptable: **200 mg/l as CaCO<sub>3</sub>**

Cause for rejection: **600 mg/l as CaCO<sub>3</sub>**

## Experiment 5: Determination of Water Hardness

### I. Objective:

To determine total hardness, calcium hardness and magnesium hardness of tap water.

### II. Test Standard:

Test procedure is in accordance to: APHA Standard Methods for examination of Water and Wastewater – 23<sup>rd</sup> Edition. Method **2340 C** & **3500-Ca B**

### III. Theory:

Originally, the hardness of water was understood to be a measure of the capacity of water for precipitating soap. Soap is precipitated chiefly by the calcium and magnesium ions commonly present in water, but may also be precipitated by ions of other polyvalent metals, such as aluminum, iron, manganese, strontium and zinc, and by hydrogen ions. Because, all but the first two are usually present in insignificant concentrations in natural waters, hardness is defined as a characteristic of water, which represents the total concentration of just the calcium and the magnesium ions expressed as calcium carbonate. However, if present in significant amounts, other hardness producing metallic ions should be included.

When the hardness is numerically greater than the sum of the carbonate alkalinity and the bicarbonate alkalinity, the amount of hardness, which is equivalent to the total alkalinity, is called carbonate hardness; the amount of hardness in excess of this is called non-carbonate hardness. When the hardness is numerically equal to or less than the sum of carbonate and bicarbonate alkalinity all of the hardness is carbonate hardness and there is no noncarbonated hardness. The hardness may range from zero to hundreds of milligrams per liter in terms of calcium carbonate, depending on the source and treatment to which the water has been subjected. "Ethylene diamine tetraacetic acid" and its sodium salts (EDTA) form a chelated soluble complex when added to a solution of certain metal cations. If a small amount of a dye such as **Eriochrome black T** is added to an aqueous solution containing calcium and magnesium ions at a pH of  $10 \pm 0.1$ , the solution will become wine red. If EDTA is then added as a titrant, the calcium and magnesium will be complexed. After sufficient EDTA has been added to complex all the magnesium and calcium, the solution will turn from wine red to blue. This is the end point of the titration.

The degree of hardness of drinking water has been classified in terms of the equivalent  $\text{CaCO}_3$  concentration as follows:

<b>Soft</b>	0 – 60 mg/l
<b>Medium</b>	60 – 120 mg/l
<b>Hard</b>	120 – 180 mg/l
<b>Very hard</b>	>180 mg/l

**Hardness may be classified:**

- a) Carbonate and non-carbonate hardness
- b) Calcium and magnesium hardness
- c) Temporary and permanent hardness.

**Significance:**

Hardness in water causes scale formation in boilers. It is also objectionable for laundry and domestic purposes since it consumes large quantity of soap. Hardness of water is an important consideration in determining the suitability of water for domestic and industrial use. Hardness value serve as a basis for routine control of softening process.

**IV. Apparatus:**

This method involves the use of “Ethylene Diamine Tetra Acetic acid” (EDTA) as the titrant. In the alkaline condition, EDTA reacts with calcium, magnesium and other divalent ions causing hardness and form a stable complex.

Calcium and magnesium ions develop wine red color with Eri-chrome black ‘T’ indicator under alkaline conditions. When EDTA is added as a titrant the Ca, mg, divalent ions get complexes with EDTA resulting in sharp change from wine red to blue color. The P<sup>H</sup> for this titration has to be maintained at 10.0 ± 0.1.

At higher pH, i.e., at about 12, the magnesium ion precipitates and calcium ion remains in solution. At this pH murex or murexide indicator forms pink color with Ca<sup>++</sup>, when EDTA is added Ca<sup>++</sup> ion gets complexes resulting in a change from pink to purple which indicates end point of the titration.

**Reagents:**

- Hardness buffer solution
- Sodium hydroxide solution (1N)
- Eri chrome Block ‘T’ indicator
- Murex indicator
- Standard EDTA Solution (0.01M)



## V. Procedure:

### 1- Total Hardness:

- i) Measure out 50 ml of given sample of water in a conical flask
- ii) Add 2 ml of Hardness buffer solution (to maintain a pH of  $10 \pm 0.1$ ) and 2 drops of Eri-chrome Black 'T' indicator.
- iii) Titrate with standard EDTA solution until color changes **from wine red to blue**.

Volume of EDTA used for titration-'A' ml.

$$\text{Total Hardness} = \frac{A \times 1000}{50} \text{ mg/l as CaCO}_3$$

### 2- For calcium hardness:

- i) Measure out 50 ml of given sample of water in a conical flask
- ii) To the sample 2ml of NaOH (1 N) solution was added which acts as a buffer solution.( pH will rise to  $12 \pm 0.1$ )
- iii) To it a pinch of murex indicator was added so that the color changes to pink after murex dissolves properly.
- iv) Burette is filled with EDTA solution.
- v) Titrate with EDTA till the color changes **from pink to purple**.
- vi) Volume of EDTA used for titration-'B' ml.

$$\text{Calcium Hardness} = \frac{B \times 1000}{\text{Volume of sample}} \text{ mg/l as CaCO}_3$$

## VI. Experimental Work

Sample No.	Trial No	Initial Reading in ml	Final reading in ml	Average vol. Of EDTA Rundown in ml
Lab sample	1			
	2			
Tap water	1			
	2			

**Magnesium Hardness (as mg/l of CaCO<sub>3</sub>):** [Total Hardness (as mg/l as CaCO<sub>3</sub>) – Calcium harness (as mg/l of CaCO<sub>3</sub>)]

**Amount of Calcium:** (as mg/l of  $\text{Ca}^{++}$ ) =  $\frac{\text{Calcium hardness (as mg/l as CaCO}_3\text{)}}{2.5}$

**Amount of Magnesium:** (as mg/l of  $\text{Mg}^{++}$ ) =  $\frac{\text{Magnesium hardness (as mg/l as CaCO}_3\text{)}}{4.116}$

**Carbonate (Temporary) Hardness and non-carbonate (permanent) Hardness:**

These can be determined from the values of Total Alkalinity ( $T_A$ ) and Total Hardness ( $T_H$ ) of the given sample of water.

Case	Carbonate (Temporary) Hardness	Non-Carbonate (Permanent) Hardness
$T_H > T_A$	$T_A$	$T_H - T_A$
$T_H \leq T_A$	$T_H$	0

**Results (all units in mg/l as  $\text{CaCO}_3$  , in mg/l  $\text{Ca}^{++}$  & in mg/l of  $\text{Mg}^{++}$ ):**

Sample No.	Total Hardness	Calcium Hardness	Magnesium Hardness	Amount of Calcium	Amount of Magnesium	Temporary Hardness	Permanent Hardness
1							
2							
Tap water							

**Drinking water standards for Hardness:**

Acceptable: **200 mg/l as  $\text{CaCO}_3$**

Cause for rejection: **600 mg/l as  $\text{CaCO}_3$**

**Drinking water standards for Calcium:**

Acceptable: **75 as mg/l as  $\text{Ca}^{++}$**

Cause for rejection: **200 mg/l as  $\text{Ca}^{++}$**

**Drinking water standards for Magnesium:**

Acceptable:  **$\leq 75$  mg/l as  $\text{Mg}^{++}$**

Cause for rejection: **150 mg/l as  $\text{Mg}^{++}$**

## Experiment 6: Determination of Chloride in Water

### I. Objective:

Determination of Chloride concentration for water samples

### II. Test Standard:

Test procedure is in accordance to: APHA Standard Methods for examination of Water and Wastewater – 23<sup>rd</sup> Edition. Method **4500-Cl- C**

### III. Theory:

Chlorides occur in all natural waters in widely varying concentrations. Upland and mountain streams are usually low in chloride concentration. Chloride gain access in many ways. Solvent power of water dissolves salts from top soil, invasion of fresh water during tides. Underground formations and seepage of human excreta, industrial wastewater and sea water incrustation etc. Chlorides more than accepted limits indicate sewage contamination

### Principle:

Chloride is determined by titration with standard silver nitrate. Silver nitrate reacts with chloride to form a slightly soluble precipitate of AgCl. At the endpoint when all the chlorides gets precipitates, free silver ions react with chromate to form silver chromate of reddish brown color.

### IV. Apparatus:

#### Reagents:

Potassium chromate indicator

N/35.5 Silver nitrate solution.

### V. Procedure:

1. Take 50ml of sample in a conical flask.
2. Place the same quantity of chloride free distilled water in another flask (Blank).

3. Add to both 0.5ml of potassium chromate indicator light yellow color if formed.
4. Titrate both with N/35.5 silver nitrate solution, till color changes to reddish brown
5. Note the amount of titrant used for both sample and the blank.

Chloride as Cl=

$$\frac{(\text{ml of AgNO}_3 \text{ for sample} - \text{ml of AgNO}_3 \text{ for blank}) * N \text{ of AgNO}_3 * 35.5 * 1000}{\text{Volume of sample}} \text{ mg/l}$$

## VI. Experimental Work:

S. No.	Trial No.	Initial Reading (ml)	End Reading (ml)	Volume of EDTA rundown (ml)	Average of EDTA (ml)	Average amount of Chloride (mg/L) as Cl
1	1					
	2					
2	1					
	2					
Tap Water	1					
	2					
Distilled Water (Blank)	1					
	2					

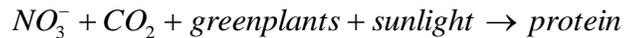
### Drinking water standards for Chlorides:

*Acceptable: 200 mg/l as Cl*

*Cause for rejection: 1000 mg/l as Cl*



carried to the earth in the rain. Nitrates are also produced by direct oxidation of nitrogen or of ammonia in the production of commercial fertilizers. The nitrates serve to fertilize plant life and are converted to proteins. Through the **nitrogen assimilation** mechanisms nitrate is taken up by plants and microorganisms and then converted to nitrite and then to ammonium, and ends up as protein.



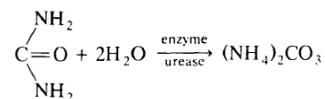
In addition, ammonia and ammonium compounds are applied to soils to supply plants with ammonia for further production of proteins.



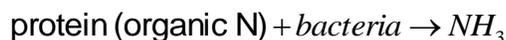
Animals and human beings are incapable of utilizing nitrogen from the atmosphere or from inorganic compounds to produce proteins. They are dependent upon plants, or other animals that feed upon plants, to provide protein. Atmospheric nitrogen is also converted to proteins by “**nitrogen-fixing**” bacteria. **Nitrogen fixation** is driven by an enzyme called nitrogenase.



Within the animal body, proteins are used largely for growth and repair of muscle tissue. Some may be used for energy purposes. In any event, nitrogen compounds are released in the waste products of the body during life. At death the proteins stored in the body become waste matter for disposal. Afterwards, the protein undergoes the process of **nitrogen mineralization or ammonification**, through which organic nitrogen is transformed to inorganic forms ( $NH_4^+$ ). This process is driven by a wide variety of microorganisms, e.g. bacteria, fungi, etc. The urine contains the nitrogen resulting from the metabolic breakdown of proteins. The nitrogen exists in urine principally as urea which is hydrolyzed rather rapidly by the enzyme urease to ammonium carbonate:



The feces of animals contain appreciable amounts of unassimilated protein (organic nitrogen). It and the protein remaining in the bodies of dead animals and plants are converted in large measure to ammonia by the action of heterotrophic bacteria, under aerobic or anaerobic conditions:

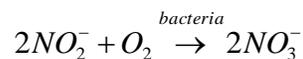


Some nitrogen always remains in nondigestible matter, and as such it becomes part of the residues in nature, e.g. in the sediments.

The ammonia released by bacterial action on urea and proteins may be used by plants directly to produce plant nitrogen. If it is released in excess of plant requirements, the excess is oxidized by autotrophic nitrifying bacteria. This process is called **nitrification**. The *Nitrosomonas* group of nitrifying bacteria, known as the nitrite formers, convert ammonia under aerobic conditions to nitrites and derive energy from the oxidation:



The nitrites are oxidized by the *Nitrobacter* group of nitrifying bacteria, which are also called the nitrate formers.



The nitrates formed may serve as fertilizer for plants. Nitrates produced in excess of the needs of plant life are carried away in water percolating through the soil because the soil does not have the ability to hold them. This frequently results in relatively high concentrations of nitrates in groundwater, and is an extensive problem in Gaza and some localities in the West bank.

Under **anaerobic** conditions **nitrates** and **nitrites** are both reduced by a process called **denitrification**. Presumably nitrates are reduced to nitrites, and then reduction of nitrites occur. Nitrites reduction is carried by bacteria to nitrogen gas, which escapes to the atmosphere. This constitutes a serious loss of **fertilizing** matter in soils when anaerobic conditions develop. The formation of nitrogen by reduction of nitrates is sometimes a problem in the activated sludge process of wastewater treatment. Prolonged detention of activated sludge problem in final settling tanks allows formation of sufficient nitrogen gas to buoy the sludge, if nitrates are present in adequate amounts. This is often referred to as the “rising” sludge problem.

Advantage is taken of denitrification in one proposed scheme for removing nitrogen from wastes where this is required to prevent undesirable growths of algae and other aquatic plants receiving waters, and also for the protection of groundwater. Ammonia and organic nitrogen are first biologically converted to nitrites and nitrates by aerobic treatment. The waste is then placed under anoxic conditions, where denitrification converts the nitrites and nitrates to nitrogen gas, which escapes to the

atmosphere. For denitrification to occur, organic matter must be present, and is oxidized for energy while nitrogen is being reduced.

**Forms of nitrogen and analysis forms:**

**Total nitrogen** consists of organic nitrogen, ammonia, nitrite and nitrate.

**Organic nitrogen**, as Protein

**Ammonia nitrogen**  $\text{NH}_4^+ - \text{N}$

**Nitrite nitrogen**  $\text{NO}_2^- - \text{N}$

**Nitrate nitrogen**  $\text{NO}_3^- - \text{N}$

**Kjeldahl nitrogen:** organic nitrogen and ammonia

The  $\text{NH}_4^+ - \text{N}$ ,  $\text{NO}_2^- - \text{N}$ , and  $\text{NO}_3^- - \text{N}$  are inorganic nitrogen

**Determination of Nitrates in Water:** Generally the ground water has high nitrate concentration because of the percolating sewage, industrial waste, chemical fertilizers, leaches from solid waste landfills, septic tank effluents etc. Whatever may be the reason the high concentration of nitrate is harmful to human beings, particularly for infants. The low acidity in the infants' intestine permits the growth of nitrate reducing bacteria that converts the nitrate to nitrite that is then absorbed in the blood stream. The nitrite has a great affinity for hemoglobin than the oxygen and it replaces oxygen in the blood. The deficiency of oxygen causes suffocation. The color of the skin of the infants becomes blue so it is termed as blue baby disease. The medical name is 'methemoglobinemia'. This disease is a fatal disease and it takes place when the concentration of nitrates is more than 45 ppm. So it is important to find the amount of nitrate in drinking water though it is a difficult task and requires spectrophotometer also.

**IV. Apparatus:**

- (i) Spectrophotometer with a range of 300 – 700 nm.
- (ii) Nessler tubes capacity 100 ml



**Reagents:**

- (i) Standard silver sulfate
- (ii) Phenol disulfonic acid
- (iii) Ammonium hydroxide
- (iv) Stock nitrate solution: Dry potassium nitrate ( $\text{KNO}_3$ ) in an oven at  $105^\circ\text{C}$  for 24 hours. Dissolve 0.1631 g in water and dilute to 1000 ml. 1.0 ml = 100 micro gram  $\text{NO}_3^- - \text{N}$ . Preserve with 2 ml  $\text{CHCl}_3/\text{l}$

(v) Standard nitrate solution

### **V. Procedure:**

- (i) Take 50 ml of filtered sample in a flask.
- (ii) Add an equivalent amount of silver sulfate to remove chlorides. So chloride determination is done prior to the nitrates.  $1 \text{ mg/l Cl} = 1 \text{ ml Ag}_2\text{SO}_4$  solution.
- (iii) Slightly warm and filter the precipitated AgCl.
- (iv) Evaporate the filtrate in a porcelain dish to dryness.
- (v) Cool and dissolve the residue in 2 ml phenoldisulphonic acid and dilute to 50 ml.
- (vi) Add 10 ml of liquid ammonia to develop a yellow color. Actually nitrate reacts with disulfonic acid and produces a nitro- derivative that in alkaline medium produces a yellow color..
- (vii) Observe the color developed at 410 nm with a light path of 1 cm.
- (viii) Calculate the concentration of nitrate from the standard curve.
- (ix) Prepare the standard curve using suitable aliquots of standard nitrate solution in the range of 5 to 500 mg  $\text{NO}_3$  by following the above procedure.

### **VI. Experimental Work:**

The observed value of nitrates in the given sample of water = .....mg/l

#### **Comments**

- The determination of nitrates is important from health point of view that is safe against the diseases produced
- It is also used to assess the self-purification capacity of water bodies and the nutrient balance in surface waters and soil.
- It is useful to find out state of decomposition of organic matter in sewage.

The observed value of nitrates in the given sample of water indicates that.....

## Experiment 8: Colorimetric Analysis

### I. Objective:

Determination of copper in unknown solution using colorimeter based on Beer Lambert's law, by preparing standard curve from the test results of the standard solutions, and to test the absorbance of a  $\text{CuSO}_4$  solution of unknown molar concentration and determine its concentration using our calibration curve.

### II. Test Standard:

Test procedure is in accordance to: APHA Standard Methods for examination of Water and Wastewater – 23<sup>rd</sup> Edition. Method **XXXX**

### III. Theory:

The measure or matching of color may frequently furnish a convenient method of quantitative determination of materials which are themselves colored or can be made to form colored compounds by suitable chemical reactions. Variation of the color of a system with changes in concentration of some component forms the basis of colorimetric analysis. Therefore, colorimetric analysis is concerned with determination of the concentration of a substance based on relative absorption of light with respect to a known concentration of a substance.

Most test substances in water are colorless and undetectable to the human eye. To test for their presence we must find a way to "see" them. A colorimeter or spectrophotometer can be used to measure any test substance that is itself colored or can be reacted to produce a color. In fact a simple definition of colorimetry is "the measurement of color" and a colorimetric method is "any technique used to evaluate an unknown color in reference to known colors". In a colorimetric chemical test the intensity of the color from the reaction must be proportional to the concentration of the substance being tested.

To avoid these sources of error, a colorimeter or spectrophotometer can be used to photoelectrically measure the amount of colored light absorbed by a colored sample in reference to a colorless sample (blank). A colorimeter is generally any tool that characterizes color samples to provide an objective measure of color characteristics. In chemistry, the colorimeter is an apparatus that allows the absorbance of a solution at a particular frequency (color) of visual light to be determined. Colorimeters hence make it possible to ascertain the concentration of a known solute, since it is proportional to the absorbance.

Global Water's colorimeters pass a colored light beam through an optical filter, which transmits only one particular color or band of wavelengths of light to the colorimeter's photodetector where it is measured. The difference in the amount of monochromatic light transmitted through a colorless sample (blank) and the amount of monochromatic light transmitted through a test sample is a measurement of the amount of monochromatic light absorbed by the sample. In most colorimetric tests the amount of monochromatic light absorbed is directly proportional to the concentration of the test factor producing the color and the path length through the sample.

However, for a few tests the relationship is reversed and the amount of monochromatic light absorbed is inversely proportional to the concentration of the test factor.

The choice of the correct wavelength for testing is important. It is interesting to note that the wavelength that gives the most sensitivity (lower detection limit) for a test factor is the complementary color of the test sample. For example the Nitrate-nitrogen test produces a pink color proportional to the nitrate concentration in the sample (the greater the nitrate concentration, the darker the pink color). A wavelength in the green region should be selected to analyze this sample since a pinkish-red solution absorbs mostly green light.

**Principle:**

Colorimetry is simply the measurement of color. In this experiment we will be measuring the concentration of copper sulfate solutions. However, due to the light blue color of copper sulfate, the color fades out by the time we'll have made a 10% concentrated solution. So we add Ammonium solution to make it visible. In general, a colorimetric procedure involves adding some type of chemical (or "reagent") to a sample of water. The reagent then reacts with the substance we are trying to measure, causing a color change in the sample. In the case of the Copper Sulfate test, we add ammonia solution to the sample. There is a chemical reaction that makes the water cloudy (the color change!) Since we not only want to know if  $\text{CuSO}_4$  is present in our samples, but also how much of it is present, the degree of color change due to the chemical reaction must be related to the concentration of chloride in the sample. The color change may be visible to our eyes, and we can use the colorimeter to make a precise measurement of the degree of color change.

A colorimeter passes light of a particular "wavelength" through the sample. Using a detector, the colorimeter can measure how much of the light has been absorbed by the sample. The amount of light absorbed by the sample is related to the color change caused by the chemical reaction. The amount of the absorption can then be used to determine the concentration of the chemical of interest (in this case  $\text{Cu}^{2+}$ ).

The following are applications of Colorimetric analysis:

- i. Determination of presence and quantity of impurities in water.
- ii. Determination of presence and amounts of metals in metal ores.
- iii. Urinary malondialdehyde determination.

The chief advantage of calorimetric analysis is that it provides a simple means of determining minute quantities of substances. However, the disadvantage is that the prevalence of errors due to personal characteristics of each observer, the visual methods of calorimetric analysis is gradually being replaced by the use of photoelectric cells.

#### IV. Apparatus:

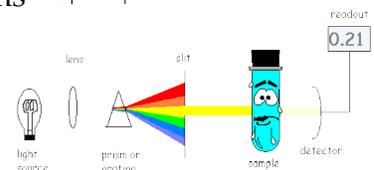
Colorimeter, 2 Cuvettes, five 20 × 150 mm test tubes, labeled 1-5, test tube rack or large beaker for holding test tubes, two 100 ml beakers, stirring rod, 250 ml volumetric flask, tissues (preferably lint-free), a pipette, an electronic beam balance and a 10ml measuring cylinder. Copper Sulfate and Concentrated Ammonia solution.

#### V. Procedure:

1. Although we were supposed to measure 1.1925g to make 0.02M  $\text{CuSO}_4$  solution, we measured 1.255g to account for the presence of 0.5% impurities in  $\text{CuSO}_4$  into a 250 $\text{cm}^3$  volumetric flask, added 50 $\text{cm}^3$  of concentrated Ammonia solution, and then topped it up with distilled water. We used this as our stock solution.
2. Using a 10 $\text{cm}^3$  measuring cylinder and distilled water, we prepared 80%, 60%, 40% 20% and 10% concentrations of  $\text{CuSO}_4$  From the stock solution.
3. Using the 80% concentration of the standard solution and the colorimeter, we scanned the region of the spectrum between 400 and 700nm, then selected the wavelength filter that gave the highest absorbance of the radiation.
4. Using the selected maximum wavelength ( $\lambda_{\text{max}}$ ) we measured the absorbance of the series of concentrations starting from 10%, 20%, 40%, 60% and 80%.
5. We plotted the calibration curve for the absorbance against concentrations.
6. The laboratory technician prepared a sample of  $\text{CuSO}_4$  of unknown concentration to us.



Spectrophotometer



We measured the concentration of the solution and recorded its absorbance.

The results of the experiment were recorded on the signed result slip attached unto this report.

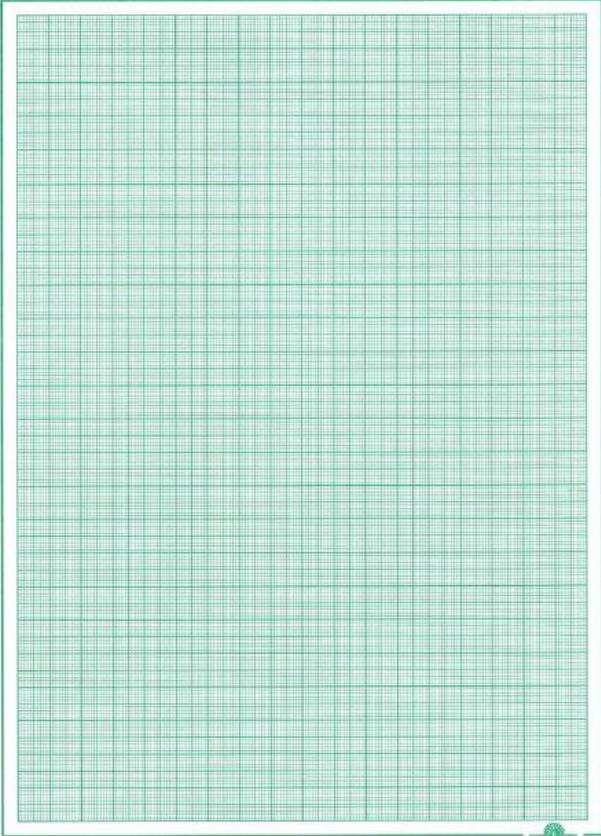
### **VI. Experimental Work:**

Using the results recorded on the result slip, we plotted a graph of absorbance against concentration of the  $\text{Cu}^{2+}$  in the solution.

As demonstrated by the graph, the following were respective absorbance of every concentration:

Concentration	Absorbance.
80%	
60%	
40%	
20%	
10%	
0%	
Unknown Con.(      )	

Batch No..... DATE : □□□□□□



MAYUR

## Experiment 9: Turbidity Test

### I. Objective:

To measure the turbidity of tap water and given samples.

### II. Test Standard:

Test procedure is in accordance to: APHA Standard Methods for examination of Water and Wastewater – 23<sup>rd</sup> Edition. Method **2130 B**

### III. Theory:

Turbidity in water is due to suspended particles like clay, silt and finely divided organic matter and microscopic organisms. The method presented below is based on a comparison of the intensity of light scattered by the sample in specific conditions with the intensity of light scattered by standard reference suspension under the same condition. The higher the intensity of scattered lights, higher the turbidity. Formazine polymer, which has gained acceptance as the turbidity standard reference suspension is used as a reference turbidity standard suspension for water. It is easy to prepare and is more reproducible in its lights scattering properties than the clay or turbid natural water standards previously used. The turbidity of a given concentration of Formazine has an approximate turbidity of 100 NTU, when measured on candle turbidity meter. Nephelometric turbidity units based on Formazine preparation will have approximate units derived from Jackson candle Turbidimeter but will not be identical to them.

#### Significances:

- i) Turbid water is undesirable from aesthetic point of view. Consumers will hesitate to drink the turbid waters.
- ii) Turbid waters pose problems in water treatment plants. Efficiency of the filters is reduced.
- iii) Disinfections is also affected since pathogenic organisms will take shelter between particles and will not get destroyed.

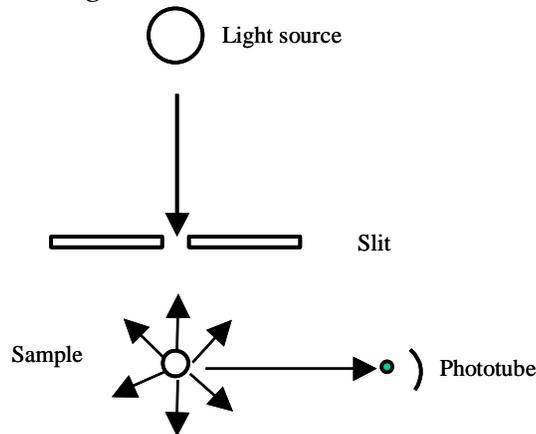
#### Drinking water standards:

- 1) Turbidity should not exceed 10 mg/lit on silica scale
- 2) Turbidity should be less than 2.5 JTU

Instrumental method: (Nephelometric method)

**Principle:**

When light is passed through a sample having turbidity, some of the light is scattered by the particles. The scattering of the light is generally proportional to the turbidity. The turbidity of a sample is thus measured from the amount of light scattered by the sample taking a reference with standard turbidity suspension.



**Figure 1: Schematic diagram of a nephelometer**

**IV. Apparatus:**

1. Turbidity Meter
2. Sample Cells
3. Standard Flasks
4. Funnel
5. Wash Bottle
6. Tissue Papers



**V. Procedure:**

1. Switch on the Nephelometer
2. Using the standard solution, calibrate the Turbidity Meter
3. Keep the sample and find out the value of Turbidity in NTU from the display

**VI. Experimental Work:**

Sample No.	Temperature of the Sample (°C)	Turbidity (NTU)

## Experiment 10: Determination of Total Solids

### I. Objective:

To determine the following types of solids in given samples(s) of water

- a) Total solids
- b) Fixed solids
- c) Volatile solids
- d) Suspended solids
- e) Filterable solids
- f) Filterable volatile (organic) solids
- g) Settleable solids

### II. Test Standard:

Test procedure is in accordance to: APHA Standard Methods for examination of Water and Wastewater – 23<sup>rd</sup> Edition. Method **2540 B, C, D, E**

### III. Theory:

The environmental engineer is concerned with the measurement of solid matter in a wide variety of liquid and semiliquid materials ranging from potable waters through polluted waters, domestic and industrial wastes, and sludge produced in treatment processes. Strictly speaking, all matter except the water contained in liquid materials is classified as solid matter. The usual definition of solids, however, refers to the matter that remains as residue upon evaporation and drying at 105 to 110 °C.

#### Significance:

The amount of dissolved solids present in water is considered for suitability for domestic use. In general, water with total solids content of less than 500mg/l are more desirable. In industrial and public water supply the total solids determination is important. In cases where softening is needed, the type of softening procedure may be decided by the total solids content.

The settleable solids determination has two very important applications. (a) It is used extensively in the analysis of industrial waste to determine the need and design of primary settling tanks in plants employing biological treatment process. (b) The test is widely used in sewage treatment plant operations to determine the efficient of sedimentation units.

Suspended and volatile suspended solids determinations are used to evaluate the strength of sewage and industrial waste. It is also used to control aeration of solids in activated sludge process.

The Total- and volatile –solids tests are normally applied to sludge. They are indispensable in the design and operation of sludge-digestion, vacuum filter, thickener and incineration units.

#### **Definitions for solids found in wastewater:**

- **Total solids (TS):** the residue remaining after a wastewater sample has been evaporated and dried at a specific temperature (103 – 105 °C)
- **Total volatile solids (TVS):** the solids that can be volatilized and burned off when the TS are ignited (500 ± 50 °C)
- **Total fixed solids (TFS):** the residue that remains after TS are ignited (500 ± 50 °C)
- **Total suspended solids (TSS):** portion of the TS retained on a filter with a specified pore size, measured after being dried at a specific temperature ((105 °C). the filter used most commonly for the determination of TSS is the Whatman glass fiber filter, which has a nominal size of about 1.58 µm.
- **Volatile suspended solids (VSS):** those solids that can be volatilized and burned off when the TSS is ignited (500 ± 50 °C).
- **Fixed suspended solids (FSS):** the residue that remains after TSS are ignited (500 ± 50 °C).
- **Total dissolved solids (TDS): (TS – TSS):** those solids that pass through the filter, and are then evaporated and dried at specific temperature. It should be noted that what is measured as TDS is comprised of colloidal and dissolved solids. Colloids are typically in the size range from 0.001 to 1 µm.
- **Total volatile dissolved solids (VDS):** those solids that can be volatilized and burned off when the TDS are ignited (500 ± 50 °C).
- **Fixed dissolved solids (FDS):** the residue that remains after TDS are ignited (500 ± 50 °C).
- **Settleable solids:**  
Suspended solids, expressed as milliliters per liter that will settle out of suspension within a specific period of time (one hour under quiescent conditions).

*Note: With the exception of settleable solids, all solids are expressed in mg/l.*

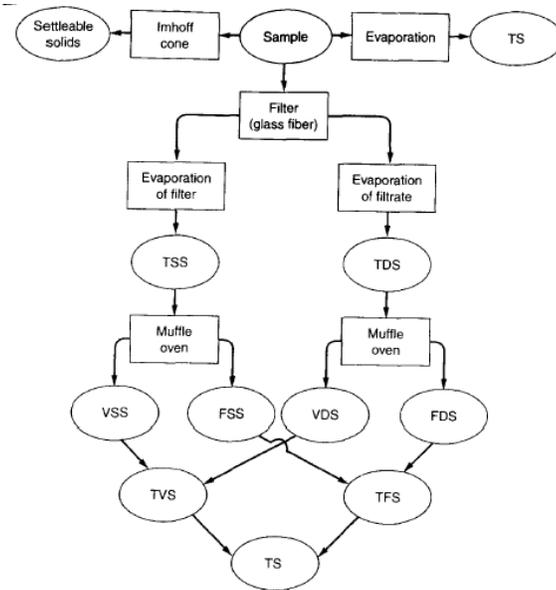


Figure 1. Interrelationships of solids found in water and wastewater.

- TS = total solids
- TSS = total suspended solids
- TDS = total dissolved solids
- VSS = volatile suspended solids
- FSS = fixed suspended solids
- VDS = volatile dissolved solids
- FDS = fixed dissolved solids
- TVS = total volatile solids
- TFS = total fixed solids

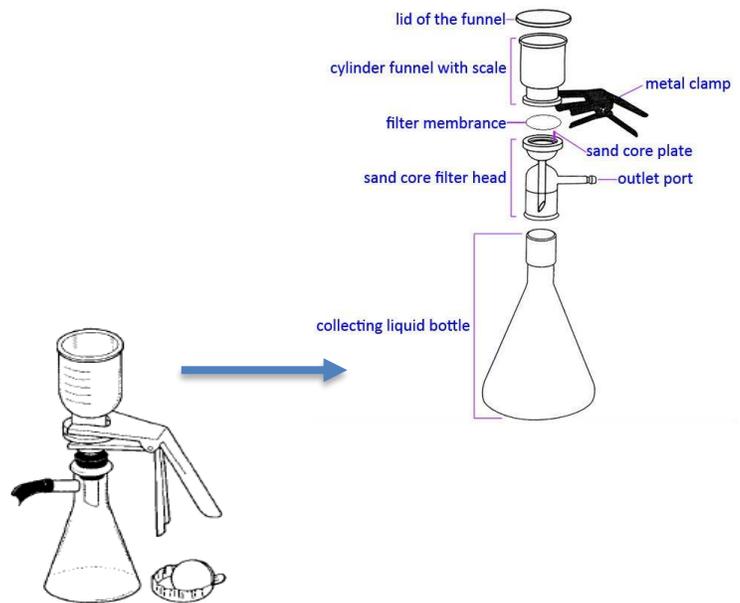


Figure 2. Apparatus used for the determination of total suspended solids. After wastewater sample has been filtered, the pre-weighed filter paper is placed in a dish for drying before weighing.

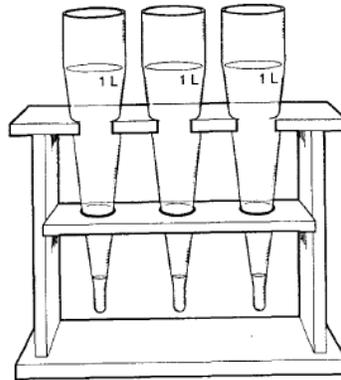


Figure 3. Imhoff cone used to determine settleable solids in wastewater. Solids that accumulate in the bottom of the cone after a specified period of time, and are reported as ml/l.

### Sludge volume index (SVI)

SVI<sub>30</sub>: the volume of 1 gram TSS after 30 minutes settling in Imhoff cone.

$$SVI = \frac{\text{settled volume of sludge, ml/l}}{\text{TSS, g/l}} = \frac{\text{ml}}{\text{g}}$$

### IV. Apparatus:

1. Porcelain evaporating dishes of 150 to 200ml capacity.
2. Steam bath
3. Drying oven
4. Desiccator
5. Balance of 0.1mg capacity
6. Filter paper
7. Electric muffle furnace
8. Imhoff cone.

### V. Procedure:

#### a) Total solids:

1. Note down the empty weight of the dish ( $W_1$ )
  - 1) Pour a measured portion (25 ml) of the well mixed sample into the dish and evaporate the contents by placing the dish on a steam bath or a Bunsen burner at 105°C.
  - 2) Allow the dish to cool in air before placing it in a desiccator to complete the cooling in a dry atmosphere.
  - 3) Weigh the dish as soon as it is cooled ( $W_2$ )
  - 4) Weigh of the total residue (mg/L) =  $\frac{(W_2 - W_1)}{25} \times 10^6$  mg/l

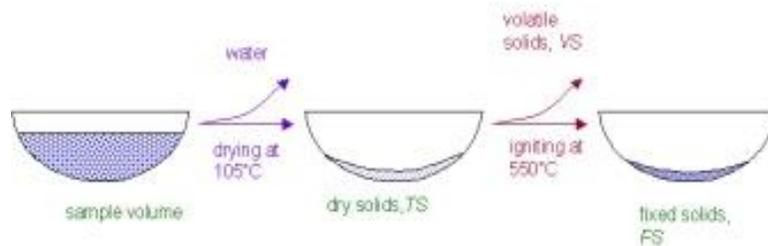
25

**b) Total Fixed solids:**

1. Keep the same dish used for determining the total residue in a muffle furnace for 1 hr. at 550°C
2. Allow the dish to partially cool in air until most of the heat is dissipated, then transfer to a desiccator for final cooling in a dry atmosphere.
3. Weight the dish as soon as it is cooled ( $W_3$ )
4. Weight of the total fixed residue =  $(W_3 - W_1)$ mg
5. Fixed solids (mg/L) =  $\frac{W_3 - W_1}{25} \times 10^6$

25

**Note:**  $W_3$  and  $W_1$  should be expressed in mg.



**c) Total Volatile solids**

$$\text{Total Volatile Solids} = \text{Total Solids} - \text{Total Fixed Solids}$$

**d) Filterable solids:**

1. Filter a measure portion of the mixed sample (25 ml) through a filter paper and collect the filtrate in a previously prepared and weighted evaporating dish.
2. Repeat the steps 3 to 6 as in the total solids procedure.
3. Weight of filterable solid =  $(W_6 - W_4)$  mg.
4. Note:-  $W_4$  = Weight of empty evaporating dish in mg.

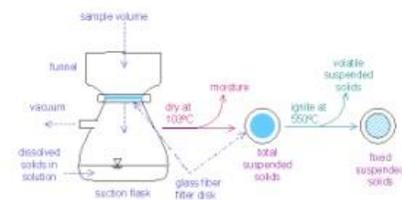
$W_6$  = Weight of empty evaporating dish in mg + Residue left after evaporating the filtrate in mg.

$$\frac{W_6 - W_4}{25} \times 10^6 \text{ mg/L}$$

25

**e) Settleable solids (Optional): (By Volume)**

1. Fill an Imhoff cone to the liter mark with thor
2. Allow 1hr of setting time at lab temperature under quiescent condition.
3. Record the volume of the settleable matter in the cone as ml/l.



## VI. Experimental Work:

Total Solid Determination Experiment (Data)			
Part A		Weights (g)	Heating/Burning Temp.
W1	Weight of the empty <b>dish</b> (g)		N/A
W2	Weight of the empty <b>dish</b> + Residues after <b>heating (105°C)</b> in the <b>Oven</b> (g)		104 ±1°C
W3	Weight of the empty <b>dish</b> + Residues after <b>burning</b> in the <b>Furnace</b> (g)		550 °C
V1	Volume of the Sample in Part A (ml)		N/A
Part B		Weights (g)	Heating/Burning Temp.
W4	Weight of the empty <b>dish</b> (g) (For TDS Calculations)		N/A
W5	Weight of the <b>Filter</b> Paper (g)		N/A
W6	Weight of the empty <b>dish</b> + Residues after <b>heating</b> in the <b>Oven</b> (g)		104 ±1°C
W7	Weight of the <b>Filter</b> paper+ Residues after <b>heating</b> in the <b>Oven</b> (g)		104 ±1°C
W8	Weight of the empty <b>dish</b> + Residues after <b>burning</b> in the <b>Furnace</b> (g)		550 °C
W9	Weight of the <b>Filter</b> paper+ Residues after <b>burning</b> in the <b>Furnace</b> (g)		550 °C
V2	Volume of the Sample in Part B (ml)		N/A
Total Solid Determination Experiment (Results)			
Part A	Results are in milli-gram (mg)	Conc. (mg/L)	(%) From the Total Solids
TSA	<b>Total Solids (mg/L) _ From Part A</b>		
TFS	<b>Total Fixed Solids (mg/L)</b>		
TVS	<b>Total Dissolved Solids (mg/L)</b>		
Part B	Results are in milli-gram (mg)	Conc. (mg/L)	(%) From the Total Solids
TSS	<b>Total Suspended Solids (mg/L)</b>		
FSS	<b>Total Fixed Suspended Solids (mg/L)</b>		
VSS	<b>Total Volatile Suspended Solids (mg/L)</b>		
TDS	<b>Total Dissolved Solids (mg/L)</b>		
FDS	<b>Total Fixed Dissolved Solids (mg/L)</b>		
VDS	<b>Total Volatile Dissolved Solids (mg/L)</b>		
TSB	<b>Total Solids (mg/L) _ From Part B</b>		

## Experiment 11: Air Quality Monitoring

### I. Objective:

To understand operation of air quality monitor and compare air quality (CO<sub>2</sub>, temperature, relative humidity) of indoor and outdoor air environments

### II. Test Standard:

Test procedure is in accordance to: APHA Standard Methods for examination of Water and Wastewater – 23<sup>rd</sup> Edition. Method

### III. Theory:

Indoor Air Quality monitoring with 90% of our time spent indoors, determining the quality of the air we breathe indoors is essential for good health and productivity. The IAQ monitor key indoor air quality indicators including CO<sub>2</sub>, humidity, temperature and CO. Should these measurements fall outside recognized guidelines; further tests can be made to suggest an appropriate course of action. For example, ventilation studies show that as room temperatures rise above 75°F(24°C) the ability of occupants to concentrate can drop by up to 50% and high levels of carbon dioxide will indicate poor ventilation that results in drowsiness and perceived stuffiness. Both situations are very common and seriously affect productivity. Overventilation wastes energy and results in increased building running costs. The Surveyor range has been designed with the user in mind. Minimal training is required to use the instruments as the intuitive menu system and display provide step-by-step guides for each operation that are updated when smart probes are plugged in. Description of the instrument The ambient air conditions measuring instrument, for assessing Indoor Air Quality and tuning and testing VAC systems, stands out on account of its efficient measurement process. The user-friendly measuring instrument has the right measurement engineering for every application and different flow speeds; for example, thermal probes, vane or Pitot tube measurement. Probe-controlled menus and selectable user profiles, e.g. for duct measurement or long-term measurement, ensure the user friendliest operation possible. The new IAQ probe measures Indoor Air Quality by measuring the CO<sub>2</sub> level, air moisture and air temperature. In addition, lux and comfort level probes can be connected to measure draught air. Clear analysis and archiving ensure convenient documentation. Temperature and humidity measurement is built-in in the new thermal probe. The special flow protocol professionally documents duct measurements. It is also possible to connect additional temperature and humidity probes. Readings from up to 3 temperature or humidity probes can be displayed in the measuring instrument; data transmission is by radio, i.e. wireless. • Instrument

memory for 10,000 readings • PC software for analysis, filing and documentation of measurement data • Thermal probes, vane measurement and built-in differential pressure probe for Pitot tube measurement • IAQ probe, lux probe and comfort level probe

#### **IV. Apparatus:**

Indoor air quality monitor (automatic sampler) for carbon monoxide (CO), carbon dioxide (CO<sub>2</sub>), temperature, humidity and pressure.

#### **V. Procedure:**

Measure air quality of three indoor locations and one outdoor location and compare their air quality parameters.

For a given location:

1. Prepare a sampling assembly.
2. Set the time constant depending upon the required averaging period (Instrument can be switch on and it will display concentration).
3. Simultaneously instrument will start recording the concentration values in the memory card. Using data transfer cable to download data from instrument to personal computers.

#### **VI. Experimental Work:**

Compile data for all locations in following manner and compare air quality of different locations. Also comment on air quality parameters.

location	carbon monoxide	carbon dioxide	relative humidity	temperature
indoor 1				
indoor 2				
indoor 3				
outdoor 1				

## References:

- i. *Standard Methods for the Examination of Water and Waste Water*, American Public Health Association (APHA), 23<sup>rd</sup> Edition, 800 / Street, N.W., Washington D.C., 2017.
- ii. *Chemistry for Environmental Engineers*, 5<sup>th</sup> Edition, Sawyer and McCarty, Tata McGraw Hill, 2003
- iii. *International Standards for Drinking Water* – World Health Organization.
- iv. *Introduction to Environmental Engineering*, 5<sup>th</sup> Edition, M.L. Davis, D.A. Cornwell, WCB McGraw-Hill, 2013.
- v. *AQ-5000, indoor air quality monitor manual*, QUEST technology, USA.