LAB MANUAL

Soil and Water Quality Lab

CENTRE OF EXCELLENCE IN WATER RESOURCES ENGINEERING

UET, LAHORE

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Experiment #01

Title

To find the pH value of a given solution.

Objective/Significance:

It represents the acidity or basicity of water sample.

PH scale ranges from 0-14.

1
2
3 Acidity increases (H^+ ions are more)
4 Basicity decreases (OH^- ions are less)
5
6
7 Neutral point (at temperature 25 ^{0}C)
8
9
10 Basicity increases (OH^- ions are more)
11 Acidity decrease (H^+ ions are less)
12
13
14

pH is important because biological and chemical reactions take place in very narrow range of pH. If pH is not appropriate, animals and plants will be harmed.

Apparatus: pH meter, Beaker, Distill water. (Buffer Solution)

Reagent: No reagent is used.

Procedure: Dip the pH meter probe in sample, stir it and note the reading until stable.

Experiment #02

Title

To find out the Alkalinity of the given solution.

Objective/Significance:

Alkalinity is a measure of water's ability to neutralize acids or it shows buffering capacity of water.

Alkalinity is mainly due to:

- Carbonate ion CO_3^{-2}
- Bi-Carbonate ion HCO_3^{-1}
- Hydroxyl ion OH^{-1}

Alkalinity determination is needed for calculating the coagulant dose and lime and soda ash requirements in water softening process. Alkalinity is total concentration of base in water.

Apparatus:

Burette, Pipettes, Titration flask, Measuring cylinder, beakers.

Reagents:

Phenolphthalein Indicator, Methyl Orange Indicator, H_2SO_4(0.02N) Titrant.

Procedure:

Take 50 ml of water sample in titration flask. Add few drops (2-5) of phenolphthalein indicator. If water becomes pink, titrate it by H_2SO_4 until pink color disappears. Measure the amount of H_2SO_4 used. Then add few drops (3-5) of methyl orange in the sample. The solution will become yellow. Titrate it by H_2SO_4 until yellow color changes into orange or pinkish orange. Measure the H_2SO_4 used. Sum up the volumes of H_2SO_4 used in both titrations and calculate total alkalinity by this formula.

$$\text{Total Alkalinity (in mg/lit)} = \frac{(\text{ml of titrant}) \times (N) \times (50) \times (1000)}{\text{Sample vol. in ml}}$$

Where, N= Normality of H_2SO_4i.e. (0.02).

Note: If phenolphthalein does not change the color of water to pink, directly go for methyl orange alkalinity

Experiment# 03

Title

To find out the Acidity of the given solution

Objective/Significance:

It is measure of the acids present in water sample. In natural unpolluted waters, acidity is due to CO_2 which reacts with H_2O to form carbonic Acid. In polluted water some acidity is due to acetic or organic acids. If acidity is more (i.e. pH low) then it harmful for giving organisms in water and for drinking purpose.

Apparatus:

Burette, Pipettes, Titration Flask, Measuring cylinder, Beakers.

Reagents:

Sodium Hydroxide (NaOH)0.02N solution, phenolphthalein indicator.

Procedure:

Take 100 ml of water sample in titration flask. Add 2 drops of a phenolphthalein indicator and Titrate it with sodium hydroxide solution till light pink color appears.

Calculate acidity by following formula:

$$\text{Total Acidity (in mg/lit)} = \frac{(\text{ml of titrant}) \times (N) \times (50) \times (1000)}{\text{Sample vol. in ml}}$$

Where

N = Normality of NaOH i.e. (0.02).

OBSERVATION AND CALCULATIONS:

No. of Observations	Initial reading	Final reading	Actual volume used	Mean
1				
2				
3				

Result:...

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Experiment #04

Title

To find out the dissolved oxygen of a given sample.

Objective/Significance:

Dissolved oxygen (DO) in water comes from air and also produced by plants living in water. DO is required for respiration by all living organisms of water. If DO is less or absent the living organism may not be survive. High temperature, more dissolved solids and organisms matter cause decrease in DO levels.

Apparatus:

. Burette, Pipettes, Measuring Cylinder, Titration flask, beaker, Stirrer

Reagents:

$MnSO_4$ (Manganese sulphate), Alkali iodide Azide, $Na_2S_2O_3$(Sodium Thio-sulphate 0.025N). Conc. H_2SO_4.Starch Indicator.

Procedure:

Take 300 ml water sample in titration flask. Add 2 ml $MnSO_4$ and 2 ml alkali iodide Azide into a simple. Shake very well and settle the contents for 3 minutes. Add 2 ml H_2SO_4 and Shake. Take 200 ml of the bottle content in a flask. Titrate against $Na_2S_2O_3$until a pale straw color (pale yellow color) appears. Now add 3 drops of starch indicator. The solution becomes blue. Now titrate it with 0.025N Na_2So_3 until blue color disappears.

Calculate dissolved oxygen by formula:

$$\underset{\text{(In mg/lit)}}{\text{Dissolved Oxygen}} = \frac{(\text{ml of titrant}) \times (8) \times (0.025) \times (1000)}{\text{Sample vol. In ml}}$$

Observations and Calculations:

No. of Observations	Initial reading	Final reading	Actual volume used	Mean
1				
2				
3				

Result:..

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Experiment #05

Title

To find out the Carbon Dioxide in a given sample.

Objective/Significance:

It comes from air and is also produced by all living organisms. CO_2 is necessary for food production in plants. If it is not present, plants will die. So, all organisms will be harmed. If CO_2 is more, it will increase acidity which is harmful.

Apparatus:

Burette, Pipettes, Measuring Cylinder, Titration Flask and Beaker.

Reagents:

Na_2CO_3(sodium carbonate, 0.045N), Phenolphthalein indicator.

Procedure:

Take 50ml water sample in titration flask. Add 4 drops of phenolphthalein indicator. If sample become pink, then there is no free CO_2.Do not titrate it. If Sample is colorless, it means that free $CO_2$is present. Titrate it with Na_2CO_3 (0.045N). When very light pink color appears, it means that it is end point. Note the ml of titrant used and calculate free CO_2 by the following formula:

$$\underset{\text{(In mg/lit)}}{C0_2} = \frac{\text{(ml of titrant)} \times (0.045) \times (22) \times (1000)}{\text{Sample vol. in ml.}}$$

Observations and Calculations:

No. of Observations	Initial reading	Final reading	Actual volume used	Mean
1				
2				
3				

Result:

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Experiment #06

Title

To find out the Total hardness in a given sample.

Aim:

To estimate the amount of total hardness in the collected sample of water.

Principle:

Hardness in water is due to the presence of dissolved salts of calcium and Magnesium. It is unfit for drinking, bathing, washing and it also forms scales in boilers. Hence it is necessary to estimate the amount of hardness producing substances present in the water sample. Once it is estimated, the amount of chemicals required for the treatment of water can be calculated.

The estimation of hardness is based on complexometric titration. Hardness of water is determined by titrating with a standard solution of ethylene diamine tetra acetic acid (EDTA) which is a complexing agent.

Total hardness:

Total hardness is due to the presence of bicarbonates, chlorides and sulphates of calcium and magnesium ions.

Apparatus:

Burette, Pipettes, Measuring Cylinder, Titration flask, Beakers, Conical flask.

Reagents:

Ethylene diamine tetra acid (EDTA)0.01M Erichrome Black-T, Buffer solution (pH 9.5)

1 ml of 0.01M EDTA solution =1.00mg of $CaCO_3$

Procedure:

- Take 50 ml water sample in titration flask.
- Add 5 ml of Ammonia buffer and 2 drops of Erichrome Black-T indicator.
- Titrate it against 0.01M EDTA until the blue color is appeared.
- Note the ml of titrant used and calculate the hardness of the sample by the following formula:

$$\text{Hardness as mg/l } CaCO_3 = \frac{\text{Vol. of EDTA used for titration} \times 1000}{\text{Sample vol.in ml.}}$$

Observations and Calculations:

No. of Observations	Initial reading	Final reading	Actual volume used	Mean
1				
2				
3				

Result:

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Experiment #07

Title

To find out the permanent hardness in a given sample.

Permanent hardness:

Permanent hardness is due to the presence of chlorides and sulphates of calcium and magnesium ions. This type of hardness cannot be removed by boiling**.**

Apparatus:

Burette, Pipettes, Measuring Cylinder, Titration Flask, Beaker, Conical Flask.

Reagents:

EDTA0.01M Erichrome Black-T, Buffer solution (pH 9.5).

Procedure:

Take 100 ml of water sample in titration flask. Boil it for 20 minutes and then cool it. Add 5ml of Ammonia buffer and 2 drops of Erichrome Black-T indicator. Titration it against 0.01M EDTA until the light blue color is appeared. Note the ml of titrant used and calculate the permanent hardness of the sample by the following formula:

$$M_1V_1 = M_2V_2$$

Observations and Calculations:

No. of Observations	Initial reading	Final reading	Actual volume used	Mean
1				
2				
3				

Result:

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Experiment #8

Title

To find out the total suspended solids in the given sample:

Objectives/Significance:

Environmental waters may contain a variety of solid or dissolved impurities. In quantifying levels of these impurities, suspended solids are the term used to describe particles in the water column. Practically, they are defined as particles large enough to not pass through the filter used to separate them from the water. Smaller particles, along with ionic species, are referred to as dissolved solids. In considering waters for human consumption or other uses, it is important to know the concentrations of both suspended and dissolved solids. The most common pollutant in the world is “dirt” in the form of TSS.

TSS can include a wide variety of material, such as silt, decaying plant and animal matter, industrial wastes, and sewage. High concentrations of suspended solids can cause many problems for stream health and aquatic life.

Total

Apparatus:

Crucibles (Evaporating dish), Measuring Cylinder, oven. Furnace, Desiccators balance.

Reagents:

No reagent is used.

Procedure:

- Note down the initial dry weight of filter paper.
- Filter 50ml of the field water sample using whattman filter paper
- Take the filter paper and place it on the evaporating dish
- Place the evaporating dish inside the oven at 103^{o}C
- After drying in the oven cool to room temperature inside desiccator
- Note down the final dry weight of the filter paper

Calculations:

Weight of filter paper W_1= ……………….g

Weight of filter paper + residue (W_2) = …………g

$$\text{Total solids (in mg/lit)} = \frac{(W_2 - W_1) \times 1000}{\text{Sample vol. in ml}}$$

Experiment #09

Title:

To find out the volatile solids in a given solution

Aim:

To determine total organic (Volatile Solids) and inorganic (Fixed Solids) solids in the given water sample.

Introduction:

The term total volatile solids refer to materials that are completely volatilized from water at higher temperature (550ºC). These solids are often referred to the organic content of the water, and the term total fixed solids can be referred to materials which are not volatilized from water at higher temperature (550ºC). These solids are often referred to the inorganic content of the water.

Apparatus:

Crucibles (Evaporating dish), Measuring Cylinder, Oven Furnace, Desiccators and Balance

Reagents:

No reagent is used.

Procedure:

- To measure the total volatile solids, take a clean silica crucible which has been washed and dried in a hot air oven at 105º C for one hour and ignited at 550ºC to remove all organic materials present in it.
- Now weigh the empty silica crucible in analytical balance. Let's denote the weight measured as W1.
- Using pipette transfer 75ml of unfiltered sample in porcelain dish.
- Switch on the oven and allowed to reach 105ºC
- Place the silica crucible in the hot air oven and care should be taken to prevent of splattering of sample during evaporating or boiling
- Dry the sample to get the constant mass
- Cool the container in desiccator.
- Note the weight with residue as W_2.
- Switch on the furnace and allow it to reach 550ºC
- Place the silica crucible in the furnace
- Note the weight as W_3 .

Calculations:

Initial weight of the evaporating dish + sample **(W_1)** **=…………..g**

Final weight of the evaporating dish + sample after drying at 103^{o}C **(W_2) =…………g**

Final weight of the evaporating dish + sample after drying at 550^{o}C **(W_3) =…………g**

Weight of Volatile Substance = $W_2 - W_3$

$$\text{Total volatile solids (in mg/lit)} = \frac{(W_2 - W_3)\times 1000}{\text{Sample vol.in ml}}$$

Experiment #10

Title:

To find out settle-able solids in a given solution.

Objective:

The settle able solids test is the measurement of the volume of solids in one liter of sample that will settle to the bottom of an Imhoff cone during a specific time. The test indicates the volume of solids removed by settling in sedimentation tanks, clarifiers, or ponds. The settle able solids test indicates whether the primary and secondary processes are functioning properly.

Apparatus:

Imhoff Cone, Measuring Cylinder, Beaker.

Reagents:

No reagent is used

Procedure:

- Mix the waster sample and pour 1000ml into Imhoff cone.
- Place the cone in the support rack allow the sample to settle for 45 minutes.
- Spain the cone and allow the sample to settle for another 15 minutes.
- Record the volume of settle-able matter (ml/lit which has accumulated in the bottom of the cone.

Experiment #11

Title

To find out the total solids in the given solution.

Aim:

To determine the total solids in the given water sample

Introduction:

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.

Solids can be classified as either filterable or non-filterable. Filterable solids may either be settleable or non-settleable. Solids can also be classified as organic or inorganic.

Total solids in term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying out in an oven at a defined temperature. Dissolved solids may lead to scaling in boiler, corrosion and degraded quality of the product. Estimation of total dissolved solids is useful to determine whether the water is suitable for drinking purpose, agriculture and industrial purpose.

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.

Environmental Significance:

Total Solids measurements can be useful as an indicator of the effects of runoff from construction, agriculture practices, logging activities, sewage treatment plant discharge and other sources.

Total solids also affect water clarity. Higher solids decrease the passage of light through water. Water will heat up affect aquatic life that has adapted to a lower temperature regime.

As with turbidity, concentration often increases sharply during rainfall. Especially in developed watersheds.

Apparatus:

Crucible (Evaporating dish), Measuring Cylinder, Oven Furnace, Desiccators, Balance

Reagents:

No reagent is used.

Procedure:

- Put the crucible in furnace at 103^0C temperature for 30 minutes.
- Cool the crucible in desiccator.
- Weigh the crucible. This is represented by D.
- Now add a measured amount of water sample in crucible.
- Place the crucible inside the oven at 103^0C for 60 minutes.
- After this take crucible out and cool it in desiccator.
- After cooling, weigh the crucible with solids.
- This weight is represented by R.
- Calculate the total solids by the following formula:

$$\text{Total solids (in mg/lit)} = \frac{(R - D) \times 1000}{\text{Sample vol. in ml}}$$

Precautions:

- Water or wastewater samples which contain high concentrations of calcium, chloride, magnesium or sulphate can readily absorb moisture from the air. Such samples may need to be dried for a longer period of time, cooled under proper desiccation and weighed rapidly in order to achieve a reasonable constant weight.
- Non representative particles such as leaves, sticks etc. should be excluded from the sample.