ENVIRONMENTAL ENGINEERING 1

LAB MANUAL

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Contents

EXPERIMENT NO. # 01

"TO DETERMINE THE ALKALINITY OF WATER"

ALKALINITY:

The measure of buffering capacity of water.

OR

Alkalinity is a measure of the ability of a solution to neutralize acid without changing the pH. The term acid neutralization capacity is some times also used (ANC).

BUFFER SOLUTIONS:

Buffer solutions are the solutions which resist change in their pH.

REASONS FOR ALKALINITY:

The alkalinity of natural waters is due to the presence of salts of weak acids, weak or strong bases may also contribute. The bicarbonates represent the major form of alkalinity. Few organic salts may also contribute towards the alkalinity of the fresh waters. The salts of weak acids like acetic acid, propionic acid would also contribute towards the alkalinity. All these substances acts as buffers to resist the change in pH.

MAJOR CONTRIBUTOR TOWARDS ALKALINITY:

Alkalinity of water is due to the presence of one or more of a number of ions. These ions include hydroxides, carbonates and bicarbonates. Hydroxide ions are always present in water, even if the concentration is extremely small. However, significant concentrations of hydroxides are unusual in natural water supplies, but may be present after certain types of treatment. Small amounts of carbonates are found in natural water supplies in certain sections of the country, rarely exceeding 3 or 4 (gpg). In most natural water bodies the buffering system is carbonate-bicarbonate. The presence of calcium carbonate or other compounds such as magnesium carbonate contribute carbonate ions to the buffering system. Alkalinity is often related to hardness because the main source of alkalinity is usually from carbonate rocks (limestone) which are mostly $CaCO_3$. If $CaCO_3$ actually accounts for most of the alkalinity, hardness in $CaCO_3$ is equal to alkalinity. Since hard water contains metal carbonates (mostly $CaCO_3$) it is high in alkalinity. Conversely, unless carbonate is associated with sodium or potassium which don't contribute to hardness, soft water usually has low alkalinity and little buffering capacity. So, generally, soft water is much more susceptible to fluctuations in pH from acid rains or acid contamination. Phosphates and silicates are rarely found in natural supplies in concentrations significant in the home. Compounds containing these ions may be used in a variety of water treatment processes. Moderate concentrations of alkalinity are desirable in most water supplies to balance the corrosive effects of acidity. However, excessive quantities cause a number of problems. These ions are, of course, free in the water, but have their counterpart in cations such as calcium, magnesium and sodium or potassium.

* Hydroxides, carbonates and bicarbonates causes alkalinity in water
* The ability of a water to neutralize an acid due to presence of carbonate, bicarbonate and hydroxide ions.
* Any process that removes or reduces alkalinity of water is called dealkalization

M.O.A = Measure of total alkalinity of an aqueous sol. determined through titration with a M.O colour indicator.

P-alkalinity = Alkalinity determined by titration with H_2SO_4 to pH 8.3, indicated by colour change of phenolphthalein and expressed as mg/L of $CaCO_3$

ters ability to resist the effect of acid rain.

METHOD OF DETERMINING ALKALINITY:

METHODOLOGY:

Alkalinity is an electrometric measurement which is performed by the computer aided titrimeter (CAT) and the pH electrode. A potentiometric titration is taken to an end-point reading of pH 4.5. The amount of acid required to reach a pH of 4.5 is expressed in milliliters. The calcium ions (CO_3) neutralize the acid in this reaction, and show the buffering capacity of the sample. From the amount of acid used, a calculation will indicate the amount of carbonate (CO_3) involved in the reaction. This then is expressed as mg of CaCO3/L even though actually part of the alkalinity may be contributed by $MgCO_3$, Na_2CO3 or K_2CO_3. Alkalinity is reported in terms of $CaCO_3$ equivalent. For samples where pH is above 8.3 the titration is made in two steps in first step the titration is conducted until pH is lowered to 8.2. The point at which phenolphthalein indicator turns from pink to colourless. The second phase of the titration is conducted until pH is lowered to methyl orange endpoint. When the pH of titration is reaches of 4.5.

(margin notes: versio carbonate → bicarbonate ions)
(margin notes: Conversion of bicarbonates → carbonic acid)

INFERFERENCES:

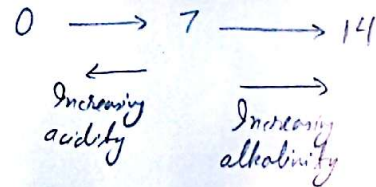Chlorides (The are removed by adding $Na_2S_2O_3$ (0.1N)

REAGENTS:

Standard sulfuric acid 0.02N

Methyl orange indicator

Phenolphthalein indicator

Sodium thiosulphate 0.1N (if residual chlorine is present)

(margin diagram: 0 ⟶ 7 ⟶ 14, Increasing acidity ←, Increasing alkalinity →)

PROCEDURE:

(A) PHENOLPHTHALEIN ALKALINITY

1. Take 50ml sample.

2. Add 2-3 drops of phenolphthalein in a titration flask if pink colour present.

3. Titrate over a white surface with O.02N H_2SO_4 to a colourless endpoint.

(B) METHYL ORANGE ALKALINITY

1. In the same flask add 2-3 drops of methyl orange indicator.

2. Titrate with O.02N H_2SO_4 until the colour changes to orange.

3. Take at least three readings for A and B and calculate the alkalinity by the formula given below:

Alkalinity (mg/l) = (A x N x 50000)/ (ml of sample)

Where,

    A = Volume of Titrant used

    N = Normality of Acid (0.02) or N/50 H_2SO_4

    Ml of sample used = 50 ml

ALKALINITY READINGS:

    Total Alkalinity = Phenolphthalein alkalinity + Methylorange alkalinity

READINGS:

SAMPLE 1 (LAB PREPARED SAMPLE)

    1st Reading = 20 ml

    2nd Reading = 20.5 ml

    3rd Reading = 22 ml

        Average = 20.83

    Phenolphthalein Alkalinity = (A x N x 50 x 1000)/ (ml of sample)

    Phenolphthalein Alkalinity = (20.83 x 0.02 x 50000)/50

    Phenolphthalein Alkalinity = 416.6 mg/l

SAMPLE 2 (LAB PREPARED SAMPLE)

PHENOLPHTHALEIN ALKALINITY

    1st Reading = 12.7 + 8.1 =   (Phenolphthalein alkalinity + Methylorange alkalinity)

    2nd Reading = 13 + 11.1 =   (Phenolphthalein alkalinity + Methylorange alkalinity)

    3rd Reading = 13.2 + 10 =   (Phenolphthalein alkalinity + Methylorange alkalinity)

    Phenolphthalein Average = 12.97

    Phenolphthalein Alkalinity = (A x N x 50000)/ (ml of sample)

    Phenolphthalein Alkalinity = (12.97 x 0.02 x 50000)/50

    Phenolphthalein Alkalinity = 259.4 mg/l

METHYLORANGE ALKALINITY

    1st Reading = 8.1 ml

    2nd Reading = 11.1 ml

3rd Reading = 10 ml

$$\text{Average} = 9.73 \text{ ml}$$

Phenolphthalein Alkalinity = (A x N x 50000)/ (ml of sample)

Phenolphthalein Alkalinity = (9.73 x 0.02 x 50000)/50

Phenolphthalein Alkalinity = 194.6 mg/l

TOTAL ALKALINITY

Total Alkalinity = Phenolphthalein alkalinity + Methylorange alkalinity

Total Alkalinity = 259.4 + 194.6

Total Alkalinity = 454 mg/l

SAMPLE 3 (LAB PREPARED SAMPLE)

1st Reading = 14 ml

2nd Reading = 14.5 ml

3rd Reading = 14.3 ml

$$\text{Average} = 14.27 \text{ ml}$$

Phenolphthalein Alkalinity = (A x N x 50000)/ (ml of sample)

Phenolphthalein Alkalinity = (14.27 x 0.02 x 50000)/50

Phenolphthalein Alkalinity = 285.4 mg/l

COMMENTS:

Highly alkaline waters are usually unpalatable and upper limits with respect to phenolphthalein alkalinity and total alkalinity are required to be specified.

The total alkalinity of all 3 samples is between 200 and 600. This means these samples are suitable for drinking purpose as well.

Coagulation:

It means mixing of alum like chemicals to make flocks and to increase the settlement of colloidal particles in water purification.

- Below pH of 4.5

All carbonate species are present as H_2CO_3 and alkalinity is -ve due to H^+

- PH = 8.3

Most Carbonate species present as HCO_3^- and alkalinity is HCO_3^-

- PH > 12.3

All carbonate species are present as CO_3^{-2} alkalinity equals to CO_3^{2-}, OH^-.

Questions:

1) Why alkalinity is measured as CaCO₃ Eq?

Ans) So that measurements of different samples can be compared to each other. By convention, alkalinity is not expressed in molarity units but rather in mg/L as $CaCO_3$

2) Write applications of alkalinity measurement?

i) To maintain alkalinity of water. The desirable limit of alkalinity of drinking water is 200mg/lit. The Permissible limit in the absence of alternate source is 600 mg/lit.

ii) Alkalinity or acidity measures the total resistance to the pH change or buffering capacity.

iii) It is significant in many uses and treatments of natural waters and waste waters.

iv) Alkalinity in excess of alkaline earth metal concentrations is significant in determining suitability of water for irrigation.

3) Why alkailinty is know as buffering capacity of water?

Ans) Because alkalinity serves as a measure of buffering capacity. The greater the alkalinity, the greater the buffering capacity.
Alkalinity is the water's capacity to resist changes in pH that would make the water more acidic. This capacity is commonly known as buffer capacity.

4) What is the importance of alkalinity in chemical coagulation?

Ans) Alum used in coagulation is an acid salt which when added in small quantity to natural water, reacts with alkalinity present to form flocs. If insufficient alkalinity is present to react with all the alum, coagulation will be incomplete and soluble alum will be left in water. It is necessary to add alkalinity in the form of soda ash to complete the coagulation.

5) Write down application of alkalinity data in water softening.

Ans) A knowledge of the kinds of alkalinity present in water and their magnitudes is important in calculating the amounts of lime $[Ca(OH)_2]$ and soda $[Na_2 CO_3]$ needed for water softening.

Experiment #2

HARDNESS

TITLE

To determine the total Hardness, Calcium Hardness, Magnesium Hardness of different water samples by EDTA (Ethyle Diamine Tetra Acetic Acid) Method.

Hard Water

The water is applied to soap; if it gives lather جاگ then it is soft water. But if it precipitates and does not make enough lather then it is hard water. Mineral deposits on cooking dishes or rings of insoluble soap scum in bathtub are signs of hard water from the municipal water supply.

Hard water is water that contains divalent cations with a charge (+2), especially Ca^{2+}, Mg^{2+}, Mn^{2+}, Fe^{2+}, Sr^{2+}. Normally hardness is due to Ca^{2+}, Mg^{2+}. More degree of ions present in water more is hardness. Due to presence of these ions water precipitate soap instead of lather. In hard water more soap is consumed. These cations can combine with the anions (HCO_3^{1-}, Cl^{1-}, SO_4^{2-}, NO_3^{1-} and SiO_3^{2-}) in water.

These ions do not pose any health threat, but they can engage in reactions that leave insoluble mineral deposits. These deposits can make hard water unsuitable for many uses, and so a variety of means have been developed to "soften" hard water; i.e. remove the calcium and magnesium ions. In fresh waters, the principal hardness-causing ions are calcium and magnesium; strontium, iron, barium and manganese ions also contribute.

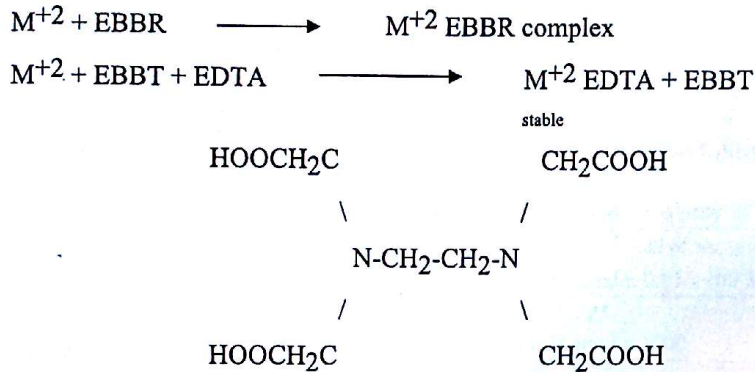| CATIONS CAUSING HARDNESS	ANIONS CAUSING HARDNESS
	HCO_3^-
Ca^{2+}	
	SO_4^{-2}
Mg^{2+}	
	Cl^-
Sr^{2+}	
	NO_3^-
Fe^{2+}	
	SiO_3^{-2}
Mn^{2+}	

Hardness can be measured by the reaction of polyvalent metallic ions in a water sample with a chelating agent such as ethylenediaminetetra-acetic acid (EDTA) and is commonly expressed as an equivalent concentration of calcium carbonate. Hardness can also be estimated by determining the concentrations of the individual components of hardness and expressing their sum in terms of an equivalent quantity of calcium carbonate.

TITRATION THEORY:

When a small amount of dye EBBT is added to the solution containing hard water, the indicator form its complex with metal ions and gives wine red colour. But EDTA is capable of

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forming more stable complex with EBBT by the addition of EDTA. EDTA replaces EBBT ions and again the blue colour of solution is obtained due to free indicator ions.

EBBT = Erich Rome Blue Black R

M^{+2} + EBBR $\longrightarrow$ M^{+2} EBBR complex

M^{+2} + EBBT + EDTA $\longrightarrow$ M^{+2} EDTA + EBBT

stable

HOOCH$_2$C   CH$_2$COOH

\   /

N-CH$_2$-CH$_2$-N

/   \

HOOCH$_2$C   CH$_2$COOH

EDTA = Ethylene Di-amine tetra acetic acid

LABORATORY TEST PROCEDURE

Hardness can be determined by Complexo-metric titration.

1. For Total Hardness

1. Take 25 ml of water sample in Titration Flask.
2. Add 25 ml Distilled water in it to dilute the water solution (to avoid formation of $CaCO_3$). Distilled water does not participate in ion exchange in reaction.
3. Add 1 - 2 ml of Buffer Solution (Ammonia Buffer NH_4Cl + NH_4OH) to maintain pH in solution 7 - 9.
4. Add small amount of EBT (Erichrome Black T) as indicator. As a result of addition of EBT, solution of color changes to wine red.
5. Titrate it against 0.01N EDTA solution. Add EDTA (Ethyle Diamine Tetra Acetic Acid) from burette until color changes to blue.
6. Test three samples and determine mean volume of Titrant used.
7. Total Hardness (mg/lit) as $CaCO_3$ is calculated from the following formula.

$$\text{Total Hardness in mg/l as } CaCO_3 = \frac{\text{mean vol of titrant used x molarity of EDTA x MW of } CaCO_3 \text{ x 1000}}{\text{vol of sample in ml}}$$

$$\text{Total Hardness in mg/l as } CaCO_3 = \frac{\text{mean vol of titrant used x 0.01 x 100 x 1000}}{25}$$

$$\text{Total Hardness in mg/l as } CaCO_3 = \frac{\text{mean vol of titrant used x 1000}}{25}$$

Molarity of EDTA = 0.01 Mole
Volume of sample used = 25 ml
MW of $CaCO_3$ = 100

2. For Calcium Hardness

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1. Take 25 ml of water sample in Titration Flask.
2. Add 25 ml Distilled water in it to dilute the water sample.
3. Add 2 - 3 ml of NaOH Buffer Solution.
4. Add small amount of EBBR (Eerichrome Blue Black R) as indicator. As a result of addition of EBBR, color of solution changes to wine red.
5. Titrate it against 0.01N EDTA solution. Add EDTA (Ethyle Diamine Tetra Acetic Acid) from burette until color changes to blue.
6. Test three samples and determine mean volume of Titrant used.
7. Calcium Hardness (mg/lit) as $CaCO_3$ is calculated from the following formula

$$\text{Calcium Hardness in mg/l as } CaCO_3 = \frac{\text{mean vol of titrant used x molarity of EDTA x MW of } CaCO_3 \text{ x 1000}}{\text{vol of sample in ml}}$$

$$\text{Calcium Hardness in mg/l as } CaCO_3 = \frac{\text{mean vol of titrant used x 0.01 x 100 x 1000}}{25}$$

$$\text{Calcium Hardness in mg/l as } CaCO_3 = \frac{\text{mean vol of titrant used x 1000}}{25}$$

Molarity of EDTA = 0.01 Mole
Volume of sample used = 25 ml
MW of $CaCO_3$ = 100

3. For Magnesium Hardness

Total Hardness = Calcium Hardness + Magnesium Hardness
Magnesium Hardness = Total Hardness - Calcium Hardness

1. For Calcium Ions

Procedure is same as above for Calcium Hardness
Calcium Ions (mg/lit) is calculated from the following formula

$$\text{Calcium Ions in mg/l} = \frac{\text{mean vol of titrant used x molarity of EDTA x MW of Ca x 1000}}{\text{vol of sample in ml}}$$

$$\text{Calcium Ions in mg/l} = \frac{\text{mean vol of titrant used x 0.01 x 40.08 x 1000}}{25}$$

$$\text{Calcium Ions in mg/l} = \frac{\text{mean vol of titrant used x 400.08}}{25}$$

Molarity of EDTA = 0.01 Mole
Volume of sample used = 25 ml
MW of Ca = 40.08

OBSERVATIONS AND CALCULATIONS

TOTAL HARDNESS

Total Hardness

Sample No.	Initial Reading	Final Reading	Volume of Titrant used (ml)	Mean Volume (ml)	Volume of sample (ml)	Total Hardness as CaCO$_3$ (mg/l)
1	12.7	15.4	2.7	2.7	25	108
2	12.4	15.6	3.2	3.2	25	128
3	13.1	15.1	2	2	25	80

. Total Hardness as CaCO$_3$ (mg/l) = (Volume of Titrant used x 1000)/Volume of sample

CALCIUM HARDNESS, MAGNESIUM HARDNESS AND CALCIUM IONS

Sample No.	Initial Reading	Final Reading	Volume of Titrant used (ml)	Mean Volume (ml)	Volume of sample (ml)	Calcium Hardness as CaCO$_3$ (mg/l)	Magnesium Hardness as CaCO$_3$ (mg/l)	Calcium ions (mg/l)
1	4.6	6.3	1.7	1.7	25	68	40	27.25
2	4.1	6.7	2.6	2.6	25	104	24	41.68
3	4.8	6	1.2	1.2	25	48	32	19.24

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* Temporary Hardness is due to Carbonates and bicarbonates of calcium and magnesium. It can be removed by boiling the water or by adding lime to water.

* Permanent hardness is due to sulphates, chlorides and nitrates of calcium and magnesium. It requires special methods for water softening.
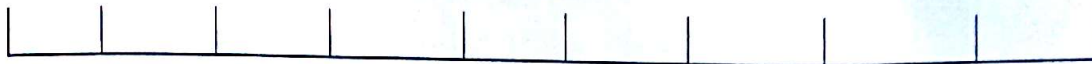
* For drinking water

   Total hardness (as $CaCO_3$) mg/l = 300 - 600

* Magnesium hardness with sulphate ion causes Laxative effect

* The hard water forms scales

* Hardness upto 50 ppm is soft water

Calcium Hardness as $CaCO_3$ (mg/l) = (Volume of Titrant used x 1000)/Volume of sample

Magnesium Hardness as $CaCO_3$ (mg/l) = Total Hardness as $CaCO_3$ - Calcium Hardness as $CaCO_3$

Calcium Ions (mg/l) = (Volume of Titrant used x 400.8)/Volume of sample

Comments:

1) It is important consideration in determining the suitability of water for domestic and industrial use.

2) The environmental engineer uses this value as a basis for recommending the need for softening process.

Questions:

1). what is pseudo hardness?

Ans) The waters containing high concentrations of sodium that interferes with normal behaviour of soap.

2). Why hardness is always calculated in terms of $CaCO_3$?

Ans) Because molar weight of $CaCO_3$ is 100g/mol which is easy to calculate and moreover it is insoluble in water.

3). What causes hardness in water?

Ans) The hardness is due to presence of carbonates, bicarbonates, chlorides. and sulphates of Calcium and Magnesium in dissolved form. It is due to divalent cations i.e Ca^{2+} 1's

4). Why NaOH is used in the determination of Calcium hardness?

Ans) To determine $[Ca^{2+}]$, it is necessary to eliminate the Mg^{2+} ions in solution. This is done by adding NaOH. The Mg^{2+} will precipitates as very insoluble magnesium hydroxide which will not react with EDTA and calcium alone can be titrated. NaOH is not buffer but it acts only to ppt. $Mg(OH)_2$

5) The EDTA titration is known as complexometric titration Why?.

Ans) Complexometric titration is a form of volumetric analysis in which the formation of a colored complex is used to indicate the end point of titration. This titration is useful for the determination of mixture of different metal ions in solution.
In EDTA titration voilet color is appeared at the end of reaction.
Hence it is complexometric titration.

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Experiment #3

CHLORIDES

TITLE

Determination of chloride concentration in different water samples.

Chlorides

Chloride is a salt compound resulting from the combination of the gas chlorine and a metal. Some common chlorides include sodium chloride ($NaCl$) and magnesium chloride ($MgCl_2$) and calcium chloride ($CaCl_2$). Chlorine alone as Cl_2 is highly toxic, and it is often used as a disinfectant. In combination with a metal such as sodium it becomes essential for life. Small amounts of chlorides are required for normal cell functions in plant and animal life.

Chlorides in Water

Almost all natural waters contain chloride and sulfate ions. Their concentrations vary considerably according to the mineral content of the earth in any given area. In small amounts they are not significant. In large concentrations they present problems. Usually chloride concentrations are low. Sulfates can be more troublesome because they generally occur in greater concentrations. Low to moderate concentrations of both chloride and sulfate ions add palatability to water. In fact, they are desirable for this reason. Excessive concentrations of either, of course, can make water unpleasant to drink.

The EPA Secondary Drinking Water Regulations recommend a maximum concentration of 250 mg/lit for chloride ions and 250 mg/lit for sulfate ions (expressed as Cl^- and SO_4^{-2}, not as $CaCO_3$). Chlorides give water a salty taste. At what concentrations this becomes noticeable again depends upon the individual. In large concentrations chlorides cause a brackish, briny taste that definitely is undesirable. Although chlorides are extremely soluble, they possess marked stability. This enables them to resist change and to remain fairly constant in any given water unless the supply is altered by dilution or by industrial or human wastes. Both chlorides and sulfates contribute to the total mineral content of water. As indicated above, the total concentration of minerals may have a variety of effects in the home. High concentrations of either sulfate or chloride ions add to the electrical conductivity of water.

Chlorides and sulfates can be substantially removed from water by reverse osmosis. Deionization (demineralization) or distillation will also remove chlorides and sulfates from water, but these methods are less suitable for household use than reverse osmosis.

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Methods of determination of Chlorides

1. Mohr's method or Argentometric method
2. Mercuric Nitrate method
3. Potentiometeric method

MERCURIC NITRATE METHOD

General Discussion

a. Principle: Chloride can be titrated with mercuric nitrate, $Hg(NO_3)_2$ because of the formation of soluble, slightly dissociated mercuric chloride. In the pH range 2.3 to 2.8, diphenyl carbazone indicates the titration end point by formation of a purple complex with the excess mercuric ions. Xylene cyanol FF serves as a pH indicator and end-point enhancer. Increasing the strength of the titrant and modifying the indicator mixtures extend the range of measurable chloride concentrations.

b. Interference: Bromide and iodide are titrated with $Hg(NO_3)_2$ in the same manner as chloride. Chromate, ferric, and sulfite ions interfere when present in excess of 10 mg/lit.

Procedure

a. for Titration of chloride concentrations less than 100 mg/lit:

i. Take a 100-ml of chloride containing solution in titration flask (100ml chloride containing sample is taken because of this sample contains less amount of chlorides)

ii. Add 1.0 ml indicator-acidifier reagent. It is a mixture of diphenyl carbazone and Xylene cyanol. if

pH<2.30	then the color of the solution will be green
pH=2.30 to 3.80	then the color of the solution will be bluish green
pH>3.80	then the color of the solution will be blue

iii. Titrate this sample with 0.0141N $Hg(NO_3)_2$ titrant till a definite purple end point color achieved.

iv. We can use high strength 0.141N $Hg(NO_3)_2$ instead of 0.0141N $Hg(NO_3)_2$ as a titrant but after few drops the definite purple end point color will be achieved. So it requires very care.

v. Take three readings and determine mean volume.

vi. Take 100-ml distilled water in titration flask.

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vii. Add indicator (diphenyl carbazone + Xylene cyanol) + 10mg Na_2CO_3.

viii. Titrate this sample with 0.0141N $Hg(NO_3)_2$ titrant till a definite purple end point color achieved.

ix. Take only reading and determine volume of the titrant $Hg(NO_3)_2$ used

x. Chloride ions concentration can be determined from following formula

$$Cl^- \text{ in mg/l} = \frac{(A-B) \times N \times EqW \text{ of } Cl^- \times 10000}{\text{vol of sample in ml}}$$

$$Cl^- \text{ in mg/l} = \frac{(A-B) \times N \times 35.4 \times 10000}{100}$$

where:

A = mean volume of $Hg(NO_3)_2$ in ml used to titrate the sample

B = mean volume of $Hg(NO_3)_2$ in ml used to titrate the distilled water

N = normality of $Hg(NO_3)_2$ i.e 0.0141N $Hg(NO_3)_2$

b. **for Titration of chloride concentrations greater than 100 mg/lit:**

i. Take a 50-ml of chloride containing solution in titration flask

ii. Add 0.5 ml mixed indicator. It is a mixture of diphenylcarbazone and bromphenol. If

iii. Add few drops of 0.1N HNO_3 till color become yellow

iv. Titrate this sample with high strength 0.141N $Hg(NO_3)_2$ titrant till a definite purple end point color achieved

v. Take three readings and determine mean volume. .

vi. Take 50-ml distilled water in titration flask.

vii. Add indicator

viii. Titrate this sample with 0.0141N $Hg(NO_3)_2$ titrant till a definite purple end point color achieved.

ix. Take only reading and determine volume of the titrant $Hg(NO_3)_2$ used

x. Chloride ions concentration can be determined from following formula

$$Cl^- \text{ in mg/l} = \frac{(A-B) \times N \times MW \text{ of } Cl^- \times 10000}{\text{vol of sample in ml}}$$

$$Cl^- \text{ in mg/l} = \frac{(A-B) \times N \times 35.4 \times 10000}{50}$$

where:

A = mean volume of $Hg(NO_3)_2$ in ml used to titrate the sample

B = mean volume of $Hg(NO_3)_2$ in ml used to titrate the distilled water

N = normality of $Hg(NO_3)_2$ i.e 0.10N $Hg(NO_3)_2$

OBSERVATIONS AND CALCULATIONS

a) For Cl⁻ conc. <100mg/l

Sample No.	Initial Reading	Final Reading	Volume of Titrant used (ml)	Mean Volume (ml)	Volume of sample (ml)	Cl⁻ Concentration (mg/l)
1						
2						
Blank						

b) For Cl⁻ conc. >100mg/l

Sample No.	Initial Reading	Final Reading	Volume of Titrant used (ml)	Mean Volume (ml)	Volume of sample (ml)	Cl⁻ Concentration (mg/l)
1	0 0 0	44 43 44	44 43 44	43.67	50	4335.65
2	0 0 0	19.4 19.4 19.4	19.4 19.4 19.4	19.4	50	1909.41
3	0 0 0	4.6 4.5 4.3	4.6 4.5 4.3	4.46	50	415.87
Blank	0	0.3	0.3			

Cl⁻ (mg/l) = ((A - B) x N x 35.40 x 1000) / Volume of sample

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where

A = mean volume of titrant used for sample

B = volume of titrant used for distilled water

N = normality of titrant used

4. Comments:

1) The chloride determination of water gives the idea about salinity of water. Saline water is not fit for drinking and irrigation purpose.

2) The acceptable limit is 250mg/L

3) High concentration of chloride ions is harmful for metallic pipes, irrigation, human beings.

Questions:

1) What is the WHO guideline value for chlorides?

Chloride Salts	Solubility in cold water (g/l)	Solubility in hot water (g/l)
NaCl	357	391
KCl	344	567
CaCl₂	745	1590

According to BIS 10500:1991 specification for drinking water, the chlorides (as Cl) mg/lit = 250

2) What is the significance of chlorides test for construction purposes?

Ans)) 1) If Cl^-, SO_4^{2-} concentration is much more in water which is used in construction, then it will react with cement and causes cracks in cement.

2) Chlorides when present in reinforced concrete can cause very severe corrosion of steel.

3) How chlorides gain access to natural waters?

Ans) Chloride is one of the major anions to be found in water and sewage. Its presence in large amounts may be due to natural processes such as the passage of water through natural salt formations in earth or it may be an indication of pollution from sea water intrusion, industrial or domestic waste or deicing operations. Anthropogenic sources of chloride include fertilizers, road salt, human and animal waste, these sources can result in significant concentrations of chloride in shallow ground water.

4) What would be the role of mixed indicator in this titration?

Ans) Mixed indicator is added in acid-base titration because a mixture of two indicators is used to give sharper end point colour change.

5) Why different procedures are used depending upon chlorides concentration?

There are two differences in the procedures depending upon chlorides concentration which are described below

1) Composition of indicator:

Two different composition of indicators are used because when $Cl^- > 100 mg/L$ the (Diphenyl carbazone + xylene cyanol) indicator will not give sharp colour of solution, hence (Diphenyl carbazone + Bromophenol) is used.

2) Titrant:

For $Cl^- < 100 mg/L$, we use $0.0141 N Hg(NO_3)_2$ because if we use greater normality $Hg(NO_3)_2$, the solution will be titrated in just few drops and it will be very difficult to note the exact reading. Hence, less normality $Hg(NO_3)_2$ is used for $Cl^- < 100 mg/L$ and $0.141 N Hg(NO_3)_2$ is used for $Cl^- > 100 mg/L$.

6

Experiment #4

SOLIDS

TITLE

Determination of the Solids in water

INTRODUCTION

Water and waste water consists of pure water along with various <u>dissolved, colloidal and suspended contaminants</u>. The suspended contaminants in turn may be settle able or non settle able. These may be either organic or inorganic in nature. Strictly speaking, all matter except the water contained in liquid materials is classified as solid matter
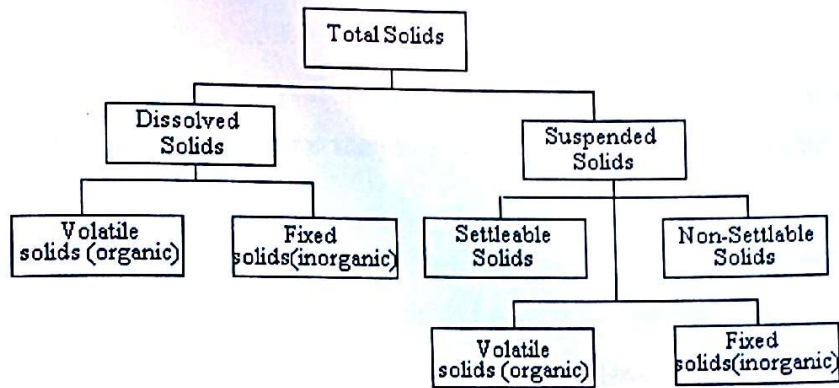
DEFINITION

The usual definition of solids however refers to the matter that remains as residue upon evaporation and drying at 103- 105^{0}C

EXPLANATION

All materials that exert significant vapor pressure at such temperatures are, of course, lost during the evaporation and drying procedures. The residue or solids remaining represent only those materials present in a sample that have a negligible vapor pressure at 105 ^{0}C. In the same way that a solid holds its shape, the atoms inside of a solid are not allowed to move around too much. This is one of the physical characteristics of solids. Atoms and molecules in liquids and gases are bouncing and floating around, free to move where they want. The molecules in a solid are stuck. The atoms still spin and the electrons fly around, but the entire atom will not change position. Solids can be made up of many things. They can have pure elements or a variety of compounds inside. When you get more than one type of compound in a solid it is called a mixture. Most rocks are mixtures of many different compounds. Concrete is a good example of a manmade mixture.

CLASSIFICATION OF SOLIDS

```
                          ┌─────────────┐
                          │ Total Solids│
                          └──────┬──────┘
              ┌──────────────────┴──────────────────┐
        ┌─────────────┐                       ┌─────────────┐
        │  Dissolved  │                       │  Suspended  │
        │    Solids   │                       │    Solids   │
        └──────┬──────┘                       └──────┬──────┘
         ┌─────┴─────┐                     ┌──────────┴──────────┐
   ┌──────────┐ ┌──────────┐        ┌──────────┐        ┌──────────────┐
   │ Volatile │ │  Fixed   │        │Settleable│        │ Non-Settlable│
   │  solids  │ │  solids  │        │  Solids  │        │    Solids    │
   │(organic) │ │(inorganic)│       └──────────┘        └──────────────┘
   └──────────┘ └──────────┘          ┌──────────────┴──────────────┐
                                ┌──────────┐             ┌──────────────┐
                                │ Volatile │             │    Fixed     │
                                │  solids  │             │    solids     │
                                │(organic) │             │ (inorganic)  │
                                └──────────┘             └──────────────┘
```

1. DETERMINATION OF TOTAL SOLIDS (TS)

Definition

It refers to the material residue left in a dish or vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature. Total solids include total suspended solids and total dissolved solids.

Purpose

During TSS determination if the suspended material clogs the filter and prolongs filtration we can estimate the TSS by calculating the difference between the total solids and total dissolved solids. For this purpose total solids are measured. In addition to this, this method can be applied for the determination of moisture content and total solid material in a wastewater, sludge or any other sample.

Scope

This procedure is applicable to waters of wide range of quality including surface water, industrial and domestic effluents, treated wastewaters etc. In addition to these samples, this method can also be applied for the analysis of moisture content in sludges, soil etc.

Interferences

- Sampling, sub-sampling, and pipeting two-phase samples may introduce serious errors. Homogenization of sample before sampling and use of wide-mouth pipette eliminates the error;

- Exclude large floating particles or submerged agglomerates of non-homogeneous materials from the sample if it is determined that their inclusion is not representative;

- Highly mineralized waters containing hygroscopic constituents may interfere. Prolonged drying and rapid weighing may reduce this error;

- Excessive solids may form water-trapping crust, which can be avoided by limiting the sample to produce 0.2 g residue;

- To aid in the quality assurance, analyze samples in duplicate.

Sampling and Storage

- Obtain an appropriate quantity of sample in a glass or plastic bottle;

- If suspended matter adheres to the container walls prefer glass container;

- Analyze sample as soon as possible. If preservation required, store it at 4^0C to avoid biological degradation;

- Preferably analyze with 24 hours and in no case after 7 days.

- Bring sample to room temperature before analysis.

Apparatus

- Muffle furnace for operating at 550 ^{0}C;

- Desiccator provided with a desiccant containing a color indicator of moisture concentration;

- Drying oven, for operation at 180 ^{0}C;

- Analytical balance capable of weighing 0.1 mg;

- Magnetic stirrer

- Wide-mouth pipettes;

- Evaporating dishes of 100 mL capacity;

- Graduated cylinder

- Low-form beaker

Procedure

- If volatile solids are to be measured, ignite cleaned evaporating dish at 550 ^{0}C for 1 hour in a muffle furnace;

- If only total dissolved solids are to be measured, heat clean dish to 180^{0}C for 1 hour in an oven;

- Store and cool ~~the~~ in desiccator until needed. Weigh immediately before use.

- Choose sample volume to yield between 2.5 and 200 mg dried residue.

- Pipette a measured volume of well-mixed sample to a pre-weighed evaporating dish;

- Evaporate to dryness in the drying oven. If necessary add successive sample portions to the same dish after evaporation;

- Dry evaporated sample for at least 1 hour in an oven at 103 to 105 ^{0}C;

- Cool in a desiccator to balance temperature, and weigh;

- Repeat drying cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until weight loss is less than 4% of previous weight or 0.5 mg, whichever is less;

- When weighing dried sample, be alert to change in weight due to air exposure and/or sample degradation.

Calculation

$$TS, mg/L = \frac{(A - B) \times 1000}{\text{Sample volume, mL}}$$

Where:

A = weight of dried residue + dish, g

B =   weight of dish, g

2. DETERMINATION OF TOTAL SUSPENDED SOLIDS (TSS)

Definition

It refers to the solid material in water or wastewater sample, which is retained by a filter of 2.0 µm (or smaller) nominal size under specified conditions.

Purpose

TSS is an important parameter in wastewater treatment. The data obtained from this test is used in the design of wastewater treatment plants. In addition to this, TSS is a parameter mentioned in NEQS, which is to be reported by industries under SMART.

Scope

This pr0Cedure is applicable to waters of wide range of quality including surface water, industrial and domestic effluents, treated wastewaters etc.

Interferences

- Sampling, sub-sampling, and pipeting two-phase samples may introduce serious errors. Homogenization of sample before sampling and use of wide-mouth pipette eliminates the error;

- Exclude large floating particles or submerged agglomerates of non-homogeneous materials from the sample if it is determined that their inclusion is not representative;

- Excessive solids may form water-trapping crust, which can be avoided by limiting the sample to produce 0.2 g residue;

- For samples with high TDS thoroughly wash the filter to ensure removal of dissolved material;

- To aid in the quality assurance, analyze samples in duplicate.

Sampling and Storage

- Obtain an appropriate quantity of sample in a glass or plastic bottle;
- If suspended matter adheres to the container walls prefer glass container;

- Analyze sample as soon as possible. If preservation required, store it at 4 ^{0}C to avoid biological degradation;
- Preferably analyze with 24 hours and in no case after 7 days.
- Bring sample to room temperature before analysis.

Apparatus

- Muffle furnace for operating at 550^{0}C;
- Desiccator provided with a desiccant containing a color indicator of moisture concentration;
- Drying oven, for operation at 103 to 105 ^{0}C;
- Analytical balance capable of weighing 0.1 mg;
- Magnetic stirrer.
- Wide-mouth pipettes;
- Glass fiber filter disks without organic binder;
- Filtration apparatus;
- Suction flasks sufficient capacity for sample size selected;
- Aluminum weighing dishes.

Procedure

- Preparation of glass fiber filter disk: Insert disk with wrinkled side up into filtration apparatus;
- Apply vacuum and wash disk with three successive 20 mL volumes of distilled water. Continue suction to remove all traces of water and discard washings;
- Remove filter from filtration apparatus and transfer to an inert aluminum weighing dish;
- Dry the glass fiber filter disk in an oven at 103 to105 ^{0}C for 1 hour. If volatile solids are to be measured, ignite at 550 ^{0}C for 15 minutes in a muffle furnace;
- Cool in a desiccator to balance temperature and weigh;
- Repeat cycle of drying or igniting, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% of the previous weighing or 0.5 mg, whichever is less. Store in a desiccator until needed;

- Choose sample volume to yield between 2.5 and 200 mg dried residue. If volume filtered fails to meet minimum yield, increase the sample volume up to 1 L. If more than 10 minutes are required for complete filtration, increase the filter size or decrease sample volume;

- Assemble filtering apparatus and filter and begin suction. Wet filter with a small volume of distilled water to seat it;

- Pipette a measured volume of well-mixed sample onto the seated glass fiber filter disk (pre-dried and weighed) with applied vacuum;

- Wash with three successive 10 mL volumes of distilled water, allowing complete drainage between washings, and continue suction for about 3 minutes after filtration is complete. Samples with high TDS may require additional washings;

- Carefully remove filter from filtration apparatus and transfer to an aluminum weighing dish;

- Dry for at least 1 hour at 103 to105 0C in an oven, cool in a desiccator to balance temperature and weigh;

- Repeat drying cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until weight loss is less than 4% of previous weight or 0.5 mg, whichever is less.

Calculation

$$TSS, mg/L = \frac{(A - B) \times 1000}{Sample\ volume,\ mL}$$

Where:

A = weight of dried residue + filter, g
B = weight of filter, g

3. DETERMINATION OF TOTAL DISSOLVED SOLIDS (TDS)

Definition

It refers to the portion of solids present in water or wastewater that passes through a filter of 2.0 µm (or smaller) nominal pore size under specified conditions. The principal ions contributing to TDS are carbonate, bicarbonate, chloride, sulfate, nitrate, sodium, potassium, calcium and magnesium. TDS in water may originate from natural sources, sewage effluent discharges, urban

runoff, or industrial effluent discharges.

Purpose
TDS is measured in waters and wastewaters because they influence other qualities of water, such as taste, hardness, corrosion and etc. Its amount in water is a consideration for the suitability of that water for drinking and other purposes.

Scope
This procedure is applicable to waters of wide range of quality including surface water, industrial and domestic effluents, treated wastewaters etc.

Interferences

- Sampling, sub-sampling, and pipeting two-phase samples may introduce serious errors. Homogenization of sample before sampling and use of wide-mouth pipette eliminates the error;
- Highly mineralized waters containing hygroscopic constituents may interfere. Prolonged drying and rapid weighing may reduce this error;
- Excessive solids may form water-trapping crust, which can be avoided by limiting the sample to produce 0.2 g residue;
- To aid in the quality assurance, analyze samples in duplicate.

Sampling and Storage

- Obtain an appropriate quantity of sample in a glass or plastic bottle;
- Analyze sample as soon as possible. If preservation required, store it at 4 0C to avoid biological degradation;
- Preferably analyze with 24 hours and in no case after 7 days.
- Bring sample to room temperature before analysis.

Apparatus

- Muffle furnace for operating at 550 ^{0}C;
- Desiccator provided with a desiccant containing a color indicator of moisture concentration;
- Drying oven, for operation at 180 ^{0}C;
- Analytical balance capable of weighing 0.1 mg;

- Magnetic stirrer with TFE stirring bar;
- Wide-mouth pipettes;
- Glass fiber filter disks without organic binder;
- Filtration apparatus;
- Suction flasks sufficient capacity for sample size selected;
- Evaporating dishes of 100 mL capacity.

Procedure:

- Preparation of glass fiber filter disk: Insert disk with wrinkled side up into filtration apparatus;
- Apply vacuum and wash disk with three successive 20 mL volumes of distilled water. Continue suction to remove all traces of water and discard washings;
- If volatile solids are to be measured, ignite cleaned evaporating dish at 550 ^{0}C for 1 hour in a muffle furnace. If only total dissolved solids are to be measured, heat clean dish to 180^{0}C for 1 hour in an oven;
- Transfer into a desiccator and store there until needed. Weigh immediately before use.
- Choose sample volume to yield between 2.5 and 200 mg dried residue. If more than 10 minutes are required for complete filtration, increase the filter size or decrease sample volume;
- Pipette a measured volume of well-mixed sample onto the seated glass fiber filter disk with applied vacuum;
- Wash with three successive 10 mL volumes of distilled water, allowing complete drainage between washings, and continue suction for about 3 minutes after filtration is complete;
- Transfer total filtrate to a pre-dried and weighed evaporating dish and evaporate to dryness in a drying oven;
- If filtrate volume exceeds dish capacity add successive portions to the same dish after evaporation;
- Dry after evaporation for at least 1 hour in an oven at 180 ^{0}C, cool in a desiccator to balance temperature, and weigh;
- Repeat drying cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until weight loss is less than 4% of previous weight or 0.5 mg, whichever is less.

Calculation

$$TDS, mg/L = \frac{(A - B) \times 1000}{\text{Sample volume, mL}}$$

Where:

A = weight of dried residue + dish, g
B = weight of dish, g

4. DETERMINATION OF SETTLE ABLE SOLIDS (SS)

Definition

"Settleable solids" is the term applied to the material settling out of suspension within a defined period.

Purpose

This parameter gives us information about the amount of suspended mater on surface water, domestic and industrial wastewater which is settle able under gravitational force. In other words, this is the portion of solids in a water sample which can be removed from water under gravitational force.

Scope

This procedure is applicable to waters of wide range of quality including surface water, industrial and domestic effluents, treated wastewaters etc.

Interferences

Sampling and sub-sampling may introduce serious errors. Homogenization of sample before sampling eliminates the error.

Sampling and Storage:

- Obtain an appropriate quantity of sample in a glass or plastic bottle;
- If suspended matter adheres to the container walls prefer glass container;
- Analyze sample as soon as possible. If preservation required, store it at 4 °C to avoid biological degradation;
- Preferably analyze with 24 hours and in no case after 7 days.
- Bring sample to room temperature before analysis.

Apparatus:

Imhoff cone

Procedure:

- Fill an Imhoff cone to the 1-L mark with a well mixed sample;
- Settle for 45 min, gently agitate sample near the sides if the cone with a rod or by spinning;
- Settle 15 min longer and record volume of settleable solids in the cone as mL/L;
- If the settled matter contains pockets of liquid between large settled practical, estimate volume of these and subtract from volume of settled solids
- The practical lower limit of measurement depends on sample composition and generally is in the range of 0.1 to 1.0 mL/L;
- Where a separation of settle able and floating materials occurs, do not estimate the floating material as settle able matter;
- Replicates usually are not required.

5. DETERMINATION OF FIXED AND VOLATILE SOLIDS (F&VS)

Definition

"Fixed solids" is the term applied to the residue of total, suspended or dissolved solids after ignition for a specified time at a specified temperature; And "Volatile solids" is the term used for the weight lost on ignition of above sample.

Purpose

In wastewater treatment technologies Volatile Suspended Solids (VSS) are considered to depict the biomass of wastewater. Hence, its determination is helpful in the design of a secondary wastewater treatment system;

Fixed solids are considered to be the inorganic constituents of wastewater. Thus, the determination of fixed solids in a wastewater gives an estimate of inorganic constituents of wastewater.

Scope

This procedure is applicable to waters of wide range of quality including surface water, industrial and domestic effluents, treated wastewaters etc.

Interferences

Determinations of total, fixed and volatile solids do not distinguish precisely between inorganic and organic matter because the loss on ignition is not confined to organic matter. It includes losses due to decomposition or volatilization of some mineral salts.

Sampling and Storage

Obtain an appropriate quantity of sample in a glass or plastic bottle;

If suspended matter adheres to the container walls prefer glass container;

Analyze sample as soon as possible. If preservation required, store it at 4 °C to avoid biological degradation;

Preferably analyze with 24 hours and in no case after 7 days.

Bring sample to room temperature before analysis.

Procedure

Ignite residue to constant weight in a muffle furnace at a temperature of 550 °C;

Ignite a blank glass fiber filter along with samples;

Usually, 15 to 20 minute ignition is required for 200 mg residue.

Let dish or filter disk cool partially in air until most of the heat has been dissipated;

Transfer to a desiccator for final cooling in a dry atmosphere. Do not overload desiccator;

Weigh dish or disk as soon as it has cooled to balance temperature;

Repeat cycle of igniting, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% or 0.5 mg, whichever is less.

Weight loss of the blank filter is an indication of unsuitability of a particular brand or type of filter for this analysis.

Calculation

$$\text{VS, mg/L} = \frac{(A - B) \times 1000}{\text{Sample volume, mL}}$$

$$\text{FS, mg/L} = \frac{(B - C) \times 1000}{\text{Sample volume, mL}}$$

Where:

A = weight of dried residue + dish or filter before ignition, g

B = weight of residue + dish or filter after ignition, g

C = weight of dish or filter, g

OBSERVATIONS AND CALCULATIONS

Total Solids   Sample = 100 ml

B = (Initial wt. of China Dish)	57.5251
A = (Final wt. of China Dish after evaporation, Drying and Cooling)	57.645
Total Solids = (A - B)x1000/ml of sample	1199 mg/L

Suspended Solids   Sample = 100 ml

B = (Weight of Filter Paper)	0.0915 g
A = (Weight of Filter Paper after drying)	0.12142 g
Suspended Solids = (A - B)x1000/ml of sample	299.2 mg/l

Dissolved Solids   Sample = 100 ml

B = (Initial wt. of China Dish)	52.67 g
A = (Final wt. of China Dish after evaporation, Drying and Cooling)	52.76 g
Dissolved Solids = (A - B)x1000/ml of sample	900 mg/l

Volatile Suspended Solids

B = (Weight after ignitian)	
A = (Weight before ignitian)	
Volatile Suspended Solids = (A - B)x1000/ml of sample	

Fixed Suspended Solids

B = (Weight after ignitian)	
C = (Weight of Cruicible)	
Fixed Suspended Solids = (B - C)x1000/ml of sample	

Settleable Solids

Settleable Solids from Imhoff Cone	3.9 g/L

Comments:

* The value of TDS obtained by us is less than 1000 g/l which is acceptable by WHO.
* The presence of high levels of TDS may be objectionless to consumers due to its taste.
* It may be corrosive to water supply system.

Questions:

1) Why sample is evaporated at 180^0c for dissolved solids?

Ans) * Samples high in bicarbonates requires careful and prolonged drying at $180°C$ to insure complete conversion of bicarbonates to carbonates.
* Residues dried at $180°C$ will lose almost all mechanically occluded water.
* Organic matter may be volatiled but not completely.
* At this temp. the values will be more accurate.

2) What is the WHO guideline value for total solids?

Ans) According to WHO, the maximum allowable concentration of total solids is 1200 mg/litre. While the palatability of drinking water is excellent when concentration of TDS is less than 300 mg/L

3) Define volatile solids?

Ans) Volatile solids are those solids in water or other liquids that are lost on ignition of dry solids at ($550°c$). It is a water quality measure obtained from the loss on ignition of Total suspended solids. It has importance in water and waste water treatment.

4) What is a gravimetric method?

Ans) It is a technique through which the amount of the ion being analyzed can be determined through the measurement of mass.
It depends on comparing the masses of two compounds containing analyte.

5) Why some solids settle down after some time?

Ans) Some solids settle down after some time due to gravitational force. Such solids have specific gravity (G_s) greater than G_s of water. Hence they settle down.

EXPERIMENT No. # 15

"DETERMINATION OF PH OF WATER"

RELATED THEORY

PH:

The concept of pH was first introduced by Danish chemist S. P. L. Sørensen at the Carlsberg Laboratory in 1909.

PH is a measure of the acidity or alkalinity of a solution. Aqueous solutions at 25°C with a pH less than seven are considered acidic, while those with a pH greater than seven are considered basic (alkaline). When a pH level is 7.0, it is defined as 'neutral' at 25°C because at this pH the concentration of H_3O^+ equals the concentration of OH^- in pure water. pH is formally dependent upon the activity of hydronium ions (H_3O^+),[1] but for very dilute solutions, the molarity of H_3O^+ may be used as a substitute with little loss of accuracy.[2] (H^+ is often used as a synonym for H_3O^+.) Because pH is dependent on ionic activity, a property which cannot be measured easily or fully predicted theoretically, it is difficult to determine an accurate value for the pH of a solution. The pH reading of a solution is usually obtained by comparing unknown solutions to those of known pH, and there are several ways to do so.

EQUATION:

$$pH = -\log_{10} \alpha_{H^+}$$

Where αH^+ denotes the activity of H^+ ions, and is dimensionless. In solutions containing other ions, activity and concentration will not generally be the same. Activity is a measure of the effective concentration of hydrogen ions, rather than the actual concentration; it includes the fact that other ions surrounding hydrogen ions will shield them and affect their ability to participate in chemical reactions. These other ions change the effective amount of hydrogen ion concentration in any process that involves H^+.

PH OF PURE WATER:

In solution at 25 °C, a pH of 7 indicates neutrality (i.e. the pH of pure water) because water naturally dissociates into H^+ and OH^- ions with equal concentrations of 1×10^{-7} mol/L. A lower pH value (for example pH 3) indicates increasing strength of acidity, and a higher pH value (for example pH 11) indicates increasing strength of basicity. Note, however, that pure water, when exposed to the atmosphere, will take in carbon dioxide, some of which reacts with water to form carbonic acid and H^+, thereby lowering the pH to about 5.7. The pH of water gets smaller with higher temperatures. For example, at 50 °C, pH of water is 6.55 ± 0.01. This means that a diluted solution is neutral at 50 °C when its pH is around 6.55 and that a pH of 7.00 is basic.

PH ADJUSTMENT:

Distilled water has an average pH of 7 (neither alkaline nor acidic) and sea water has an average pH of 8.3 (slightly alkaline). If the water is acidic (lower than 7), lime or soda ash is

added to raise the pH. Lime is the more common of the two additives because it is cheap, but it also adds to the resulting water hardness.

PH MEASUREMENT:

PH can be measured by addition of a pH indicator into the solution under study. The indicator color varies depending on the pH of the solution.

PROCEDURE:

1- Caliberate the Instrument

    a- After using of apparatus for various liquids standard solution p^H change,so in order to perform next experiment we have to make standard solution p^H within neutral range

    b- So for this purpose BUFFER SOLUTIONS are used to neutralize the standard solution used for testing because buffer solutions resist against change in p^H of the solution

    c- BUFFER SOLUTIONS are of two types which are as follows

        i- Acidic Buffer

            Acidic buffer solution is combination of strong base and salt

            EXAMPLE

            CH3COOH and CH3COONa
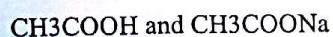
        ii- Basic Buffer

            Basic buffer solution is combination of weak base and salt

2- Switch On the Instrument

3- Take that sample whose P^H is to be determined

4- Insert glass electrode probe of the p^H meter in the solution which was initially present in standard buffer solution and wait for 5-10 minutes when blinking quotation of stabilizing on the screen of p^H meter stops note that reading

5- Take the glass electrode probe out form the solution and placed again in standard Buffer solution

Representative pH values	
Substance	pH
Hydrochloric acid, 10M	-1.0
Lead-acid battery	0.5
Gastric acid	1.5 – 2.0
Lemon juice	2.4
Cola	2.5
Vinegar	2.9
Orange or apple juice	3.5
Tomato Juice	4.0
Beer	4.5
Acid Rain	<5.0
Coffee	5.0
Tea or healthy skin	5.5
Urine	6.0
Milk	6.5
Pure Water	7.0
Healthy human saliva	6.5 – 7.4
Blood	7.34 – 7.45
Seawater	7.7 – 8.3
Hand soap	9.0 – 10.0
Household ammonia	11.5
Bleach	12.5
Household lye	13.5

6- For next experiment repeat the previous procedure

OBSERVATION AND CALCULATIONS:

SAMPLE NAME	pH OF THE SAMPLE	TEMPERATURE °C
pH=4	4	15
pH=7	7.28	15
pH-10	9.32	15
Lab sample 1	3.11	15
Lab sample 2	6.35	15
Lab sample 3	12-15	15

COMMENTS:

1) The acceptable value of pH for drinking water is $6.5 \to 8.5$. Water having pH low or above is rejectable.
2) Higher values of pH reduce the germicidal potential of chlorine.
3) pH below 6.5 starts corrosion in pipes thereby releasing toxic metals like Zn.
4) Chemical coagulation, disinfection, water softening and corrosion control are governed by pH.

QUESTIONS:

1) WHAT ARE THE APPLICATIONS OF pH TEST IN ENVIRONMENTAL ENGINGGERIN?

Ans) In water treatment process, it is a factor that is to be considered in chemical coagulation, disinfection, water softening and corrosion control.

2) EXPRESS THE FOLLOWING PH INTO HYDROGEN ION CONCENTRATION PH 2, PH10 AND PH4.5

$$pH = -\log[H^+]$$

$pH = 2 \Rightarrow [H^+] = -100$

$pH = 10 \Rightarrow [H^+] = -1 \times 10^{10}$

$pH = 4.5 \Rightarrow [H^+] = -31622.78$

3) WHAT IS THE GUIDELINE VALUE OF pH FOR DRINKING WATER?

Ans) $pH = 6.5$ to 8.5

According to Drinking standard (BIS 10500 : 1991)

4) WHY pH CHANGES IN THE PRESENCE OF ALGAL BLOOMS?

Ans) A rapidly producing floating colony of algae that may cover a stream, lake, or reservoir, creating a nuisance condition is called algal blooms.

pH changes in the presences of algal blooms is because algal bloom absorbs CO_2 from water and thus it increases the pH of water.

5) DEFINE pH.

Ans) "It is negative logarithm of hydrogen ion concentration."

It refers to hydrogen ion activity.

It can be measured by pH paper or electrometrically by pH electrode.

It is a measure of acidity or alkalinity of solution.

Experiment #6

TURBIDITY

TITLE

Determination of the Turbidity of water

INTRODUCTION

Imagine being in a boat on a sunny day, heading out onto a lake. The clearer the water, the more sunlight can penetrate the surface, and the deeper you can see. If the water is cloudy because of solid particles floating in it, less light can pass through it, and an object submerged beneath the surface will soon be invisible from the boat. Turbidity is a measure of this cloudiness.

There are three major types of particles that contribute to turbidity. The first is algae, which grows in all kinds of lakes and streams. Second, dead organic matter (from algae, plants, bacteria, fungi, etc.) also gets washed into lakes, streams and oceans and adds more particles to the water. Third, silt and sediment from shoreline erosion and from disturbance of the riverbed or lakebed also becomes suspended in the water, making it cloudy.

So, Turbidity of water is due to suspended solids such as clay, plankton, silt, finely divided organic matter, microscopic organisms and similar materials. These solids will deflect (or scatter) light as it passes through the sample. Turbidity is a measurement of the scattered light as compared to the amount of light scattered by a standard. The more light that is deflected the higher the turbidity of the sample.

MEASUREMENT OF TURBIDITY

The measuring device used in today's laboratories is called a nephelometric meter. This type of meter does not measure all of the deflected light, only that which is deflected at a right angle (90°) from the sample and light source. Turbidity is read as nephelometric turbidity units (NTU).

REAGENTS

1. **Turbid free water** - if the turbidity of the laboratory grade water is 0.05 NTU or higher, pass the water through a membrane filter having precision-sized holes of 0.2 m m. Rinse collecting flask twice with filtered water. Discard the next 200 mL of filtered water, then start collecting filtered water to prepare standards. Commercially prepared water can be substituted when its turbidity is lower than what is available in the laboratory.

2. **Stock Turbidity Suspension Solution** – Stock Turbidity Suspension Solution 1 - Weigh 1.00 gram of hydrazine sulfate, $(NH_2)_2 \cdot H_2SO_4$. Dissolve the 1.00 gram of hydrazine sulfate in a 100 mL volumetric flask. Use turbid free water to fill to the 100 mL line on

the volumetric flask Prepare stock solution monthly. Caution: hydrazine sulfate is a known carcinogen: avoid inhalation, ingestion and contact with skin.

3. Stock turbidity Suspension Solution 2 - Weigh 10.00 grams hexamethylenetetramine, $C_6H_{12}N_4$. Dissolve the 10.00 grams of hexamethylenetetramine in a 100 mL volumetric flask. Use turbid free water to fill to the 100 mL mark on the volumetric flask. Prepare stock solution monthly.

STORAGE / PRESERVATION

The turbidity test should be determined on the day the sample is taken. If this is not possible, refrigerate the sample at 4° C for up to 24 hours. Remember to vigorously shake all samples before examination.

PROCEDURE

Calibration

Always follow the manufacturer's instructions for calibration of your particular meter. If the instrument does not have a prepared calibration curve, make one by using various values of turbidity standards. Plot turbidity reading versus the standard concentration to obtain curve.

Check the accuracy of the instrument against bubble-free prepared standards; make adjustments in readings according to manufacturer's instructions. Run a minimum of one standard for each range used during the test, making sure that the meter gives stable readings in all sensitivity ranges used.

Sample Analysis

1. Select the scale.
2. Add the standard solution in turbidimeter cell and placed it in turbidimeter.
3. Calibrate the instrument.
4. Thoroughly shake sample. Wait until air bubbles disappear before pouring sample into turbidity tube. When necessary, immerse turbidity tube in an ultrasonic bath for 1 to 2 seconds to dislodge bubbles. Letting the sample stand for a period of time to allow air bubbles to dissipate will also allow solids to settle thus changing the characteristics of the sample being evaluated.
5. Wipe outside of tube to remove fingerprints, dust dirt, and water droplets. Place tube in turbidimeter.

6. Read turbidity from instrument direct reading scale or convert from calibration curve.

OBSERVATIONS

Sample No.	Source	Turbidity (NTU)
1	Lab sample 1	55
2	Lab sample 2	30
3	Lab sample 3	40
4		
5		
6		
7		
8		
9		
10		

Comments:

1) Higher level of turbidity can protect microorganisms from effects of disinfection, stimulate the growth of bacteria and give rise to a significant chlorine demand.

2) Turbidity of drinking water should be less than 5 NTU.

Questions:

1). Compare Jackson candle and nephelometric method.

Ans) * Turbidity measurements are reported in nephelometric turbidity units (NTU) or Jackson turbidity units (JTU). These two units are roughly equivalent.

* Jackson candle turbidimeter is an historical method and it cannot measure less than 25 JTU. In this method, water is poured in the tube and reading is taken when candle burning under tube can no longer be seen.

* In nephelometer, beam of light passed through water sample. Amount of light scattered at 90° angle is measured.

2). what are the major contributors towards the turbidity of water?

Ans)
1) Algae
2) Dead organic matter (algae, plants, bacteria, fungi)
3) Silt and sediments

3). what would be the effect of turbid water on the disinfectant ability?

Ans) Turbidity reduces the efficiency of disinfecting chemical process. The suspended matter can change the chemical nature of water once the disinfectant is added. Some solids may change the germ killing property of disinfectant.

4). what are the Colloidal particles?

Ans) The sewage consists of filterable solids, colloidal solids and dissolved solids. The colloidal particles consists of finely divided particles of gels, emulsions, grease, oil causing foams. It also consists of fine clay particles with some charge repulsing each other. Because of their size and charge they don't settle by gravitational force.

5). Why scale selection is important for analysis?

Ans) Scale selection is important to get more accurate results. Also each scale has its maximum limit as 0-10 scale will give false result of sample having turbidity more than 10. By naked eye an avg. person can begin to see turbidity starting at around 5NTU or greater. If water appears muddy its turbidity has reached at least 100NTU. So a range of 100NTU can be selected.

If water is completely opaque its turbidity has at least 2000NTU.