

LABORATORY MANUAL

SEMESTER - SECOND

B.TECH

**CHEMISTRY AND ENVIRONMENTAL
ENGINEERING LAB**

Name of the Student _____

Registration No. _____ Batch No. _____ Branch _____



School of Aeronautics (Neemrana)

I-04, RIICO Industrial Area, Neemrana, Dist. Alwar, Rajasthan

(Approved by Director General of Civil Aviation, Govt. Of India , All India Council for Technical Education, Ministry of HRD, Govt of India & Affiliated to Rajasthan Technical University. Kota, Rajasthan)

School of Aeronautics (Neemrana)



CERTIFICATE

This is to certify that Mr./ Ms. _____

Registration Number _____

of B.Tech () has satisfactorily completed the term of the subject, Chemistry and Environmental Engineering Lab, prescribed by Rajasthan Technical University, Kota.

Dated: _____

Signature of Faculty

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EXPERIMENT - 1

HCL METHOD

OBJECT

To determine the hardness of a given water sample by HCl method.

Glasswares

Beaker, funnel, burette, pipette, conical flask, measuring flask.

Reagents

N/50 HCl solution, Na₂CO₃ solution (N/50), water sample, distilled water.

Indicator

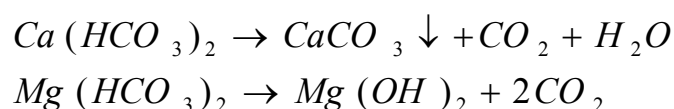
Methyl orange.

End Point

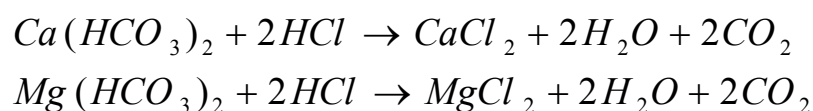
Yellow to cherry red.

THEORY

Hardness of water is the property of water which resists the formation of lather when soap is mixed. It also defined as the soap consuming capacity of water in giving lather. There are two types of hardness of water, viz temporary and permanent hardness. Temporary hardness is caused by the presence of bicarbonates, of Ca and Mg which can be removed by prolonged boiling of water because of it decomposed to carbonate of Ca and hydroxide of Mg with elimination of CO₂



Temporary hardness in water arises due to bicarbonate salts of calcium and magnesium. These are alkaline in nature. Thus temporary hardness can also be determined by estimating the alkalinity of water sample before and after boiling it. For this purpose a standard solution of acid is used in the presence of indicator methyl orange. Before boiling the alkalinity is present and following chemical reactions occurs.



PROCEDURE

1. Wash all the apparatus with distilled water.
2. Rinse and fill the burette with N/50 HCl solution and note the initial reading.
3. Pipette out 20 ml of Na_2CO_3 solution in conical flask and add 2-3 drops of Methyl Orange indicator.
4. Now add HCl from the burette drop wise with constant stirring till color change from yellow to cherry red (pink). This is the end point and burette reading is noted.
5. Repeat the titration till two concordant readings are obtained.
6. Pipette out 20 ml water sample in conical flask and add 2-3 drops of indicator, titrate against HCl solution till color change from yellow to cherry red (pink).
7. Cool the resulting solution, Filter off the precipitate.
8. Now filtrate is make up to 250 ml with distilled water.
9. Take 20ml of filtrate water sample in conical flask and 2-3 drops of methyl orange indicator.
10. Titrate it against N/50 HCl till color changes from yellow to red (pink).
11. Repeat the titration till two concordant readings are obtained.

OBSERVATION TABLES

1. Titration of N/50 Na_2CO_3 solution and N/50 HCl solution

S.L. No.	Volume of FAS Na_2CO_3 (ml)	(Burette reading) IBR (ml) FBR (ml)	Volume of HCl Solution used(ml)	Concordant reading (ml)
1	10			
2	10			
3	10			

2. Titration Before Water Sample v/s N/50 HCl Solution

S.L. No.	Volume of Water Sample ml (Before Boiling)	(Burette reading) IBR (ml) FBR (ml)	Volume of HCl Solution used(ml)	Concordant reading (ml)
1	10			V_2 ml
2	10			
3	10			

3. Titration After Water Sample v/s N/50 HCl Solution

S.L. No.	Volume of Water Sample ml (After Boiling)	(Burette reading) IBR (ml) FBR (ml)	Volume of HCl Solution used (ml)	Concordant reading (ml)
1	10			
2	10			
3	10			V_2 ml

Calculations

1. Calculation for standardization of HCl solution.

HCl solution	Na_2CO_3 solution
$N_1 V_1$	$N_2 V_2$

Where

N_1 = Normality of HCl solution = $N/50$

V_1 = Volume of HCl solution

N_2 = Normality of Na_2CO_3 solution

V_2 = Normality of Na_2CO_3 solution

2. Calculation for the determination of temporary hardness.

Water Sample	HCl solution
$N_3 V_3$	$N_4 V_4$

Where

V_3 = Volume of water sample

N_4 = Normality of HCl solution

$V_4 = (V_2 - V_3)$ Volume of HCl solution

$V_4 = (V_2 - V_3)$ (Volume of HCl used by before boiling water - volume of HCl used by after boiling water)

3. Calculation for strength of water sample or temporary hardness.

= $N_3 \times$ equivalent weight of temporary hardness producing compound

= $N_3 \times 50$ gm/liter

= $N_3 \times 50 \times 1000$ mg/L or ppm Volume of HCL solution

= $N_3 \times 50 \times 1000$ mg/L or ppm

Result

The temporary hardness of given water sample is ppm.

PRECAUTIONS

1. All the glassware should be washed with distilled water.
2. The same amount of indicator solution must be added in each titration.
3. End point must be noted correctly.
4. Titration should be performed slowly near end point.

QUESTIONS

1. What is titration?

2. What is end point ?

3. Define Indicator.

4. Differentiate internal and external indicator.

5. Why burette and pipette must be rinsed with the solution for which they are to be used ?

6. Define titrant.

7. What do you mean by hardness of water?.

8. Write compounds that causes hardness.

9. Hardness in water can be eliminated by which methods?

10. In which compound's term the hardness is measured and why ?

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EXPERIMENT - 2

EDTAMETHOD

OBJECT

To determine the Temporary and Permanent hardness of a given water sample by EDTA method.

Apparatus and Reagents

Beaker, funnel, burette, pipette (50ml), Pipette (20ml), Conical flask, Volumetric flask (100ml), standard hard water sample, EDTA Solution, Ammonia buffer (pH=9-10).

Indicator

Eriochrome black-T indicator.

End Point

Wind red to blue.

Theory

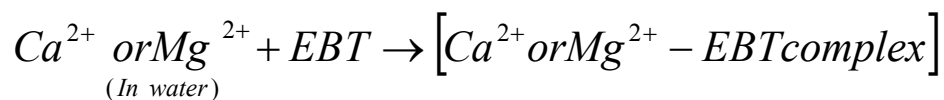
Ethylene Diamine Tetra Acetic Acid (EDTA) is a well known powerful complexing agent.



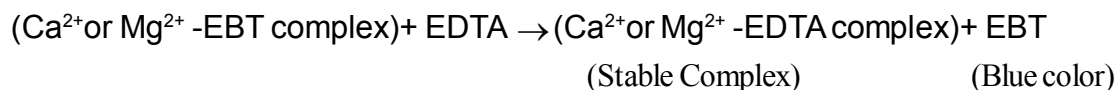
EDTA form complexes with Ca^{2+} and Mg^{2+} ions as well as with many other metal cations. These complexes have the general formula as



EDTA form complexes with Ca^{2+} and Mg^{2+} ions as well as with many other metal cations. These complexes have the general formula as (Ca-EDTA) or (Mg-EDTA).



When hard water titrated against EDTA solution then or its sodium salt forms stable complex with Ca^{2+} or Mg^{2+} ions in water.



When indicator is added to hard water which is buffered at pH 9.7 then it combines with Ca^{2+} , Mg^{2+} ions to form weak complex of wine red color, when an excess of EDTA is added then color change to blue to free EBT. Thus change of wine red color to distinct marks the end point of titration.

PROCEDURE

A. Standardization of EDTA solution with standard Hard water

Pipette out 20ml of standard hard water into 250 ml conical flask. Add 2 ml of buffer solution and 4 drops of eriochrome black-T indicator. A wine red color appears. Titrate it against EDTA solution (taken in burette) to a color change from wine red to pure blue. Record the volume of EDTA used as A ml.

B. Determination of total Hardness

Pipette out 20ml of standard hard water sample into conical flask. Add 2 ml of buffer solution and 4 drops of the indicator. Titrate it against EDTA till the wine red color changes to pure blue. Record the volume of EDTA used as B ml. This corresponds to the total hardness of the water sample.

C. Determination of Mg^{++} Hardness

Measure out 100 ml the hard water sample into a 500 ml dry beaker and add 20 ml of the calcium precipitating buffer solution while constantly stirring the mixture with a glass rod. Allow the precipitate to settle down for about 1 hour and filter through a dry funnel fitted with two pieces of whatman filter paper no. 42 in to a dry flask. Measure 20 ml of the filtrate in to a conical flask, 2ml ammonia buffer (ph-10) and 4 drops of the eriochrome black T indicator and titrate against EDTA solution. The volume of EDTA used (c ml) corresponds to magnesium hardness.

D. Determination of calcium hardness

Calcium hardness is obtained by subtracting magnesium hardness from the total hardness.

OBSERVATION AND CALCULATIONS

1. Titration of Standard Hard Water Sample v/s Solution

S.L. No.	Volume of Water Sample (ml)	(Burette reading) IBR (ml) FBR (ml)	Volume of EDTA Solution used (ml)	Concordant reading (ml)
1	20			V ₂ ml
2	20			
3	20			

2. Titration of Standard Hard Water Sample v/s Solution

S.L. No.	Volume of Hard Water Sample (ml)	(Burette reading) IBR (ml) FBR (ml)	Volume of EDTA Solution used (ml)	Concordant reading (ml)
1	20			V ₂ ml
2	20			
3	20			

3. Titration of Boiling Hard Water Sample v/s EDTA Solution

S.L. No.	Volume of Standard Hard Water Sample (ml)	(Burette reading) IBR (ml) FBR (ml)	Volume of EDTA Solution used (ml)	Concordant reading (ml)
1	20			V ₂ ml
2	20			
3	20			

Calculations

$$1 \text{ ml SHW} = 1 \text{ mg CaCO}_3$$

$$V_1 \text{ ml EDTA} = 20 \text{ ml SHW}$$

$$= 20 \text{ mg CaCO}_3$$

$$1 \text{ ml EDTA} = 20/v_1 \text{ mg CaCO}_3$$

Total Hardness

$$20 \text{ ml sample of hard water consumed} = V_2 \text{ ml EDTA solution}$$

$$= (V_2/V_1) \times (20/V_1 \text{ mg CaCO}_3)$$

$$1 \text{ ml sample of hard water consumed} = V_2/V_1 \text{ mg Ca CO}_3$$

$$1000 \text{ ml of hard water sample} = (V_2/V_1) \times 1000 \text{ mg CaCO}_3$$

$$\text{Total hardness of water} = V_2/V_1 \times 1000 \text{ mg/L}$$

Permanent Hardness

$$20 \text{ ml sample of hard water consumed} = V_3 \text{ ml EDTA solution}$$

$$(V_3) \times (20/V_1 \text{ mg CaCO}_3)$$

$$1 \text{ ml sample of hard water consumed} = V_3/V_1 \text{ mg CaCO}_3$$

$$1000 \text{ ml of hard water sample} = (V_3/V_1) \times 1000 \text{ mg CaCO}_3$$

$$\text{Permanent hardness of water} = V_3/V_1 \times 1000 \text{ mg/L}$$

Temporary Hardness

$$\text{Temporary hardness of water sample} = (\text{Total hardness} - \text{permanent hardness})$$

$$= (V_2/V_1 \times 1000) - (V_3/V_1 \times 1000) \text{ mg/L}$$

$$= (V_2/V_3) \times 1000/V_1 \text{ mg/L}$$

$$= 1000 \times (V_2/V_3)/V_1 \text{ ppm}$$

$$\text{Strength of EDTA solution} = \frac{\text{Strength SHW} \times \text{Volume of SHW}}{\text{Volume of EDTA solution}}$$

$$= \frac{1000 \times X \times 20}{A} = X \text{ ppm}$$

$$\text{Total hardness (CaCO}_3\text{)} = \frac{\text{Strength of EDTA} \times \text{Volume of EDTA}}{\text{Volume of water sample}}$$

$$= \frac{x \times b}{20} = y \text{ ppm}$$

$$\text{Magnesium hardness} = \frac{x \times c}{20} = z \text{ ppm}$$

$$\text{Calcium hardness} = y - z = n \text{ ppm}$$

Results

The hardness of a given water sample is

1. Total hardness =ppm
2. Permanent hardness =ppm
3. Temporary hardness =ppm

PRECAUTIONS

1. All the solution should be freshly prepared.
2. Distilled water should be checked with care before use.
3. The same amount of the indicator must be added each time.
4. The reaction mixture should be briskly shaken during the titration.
5. The end point should be observed correctly.
6. pH-10 should be maintained during the titration.

QUESTIONS

1. What are different methods by which hardness of water can be determined?

2. Why is EDTA method preferred over soap solution method ?

3. In which form EDTA is used ?

4. Which indicator is used in complexometric titration ?

5. Why do hardness occurs in water ?

6. How many types of hardness is present in water ?

7. What is the full form of EDTA ?

8. How many grams of NaOH should be dissolved per liter to get 1N solution ?

9. What do you mean by standardization of a solution.

10. Define Normality, Molarity, Molality of solution ?

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EXPERIMENT- 3

DISSOLVED CO₂

OBJECT

To determine the dissolved CO₂ in given water sample.

APPARATUS REQUIRED

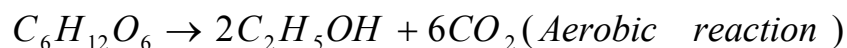
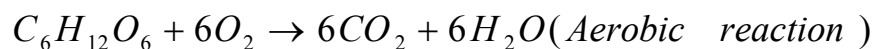
Burette (50ml), Pipette (20ml), Measuring flask (50ml), Conical flask (250ml), Beaker, Funnel, NaOH Solution (N/40), Standard solution of Oxalic acid(N/40), water sample.

Indicator : Phenolphthalein indicator.

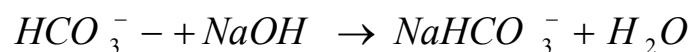
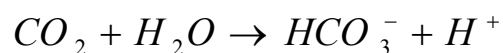
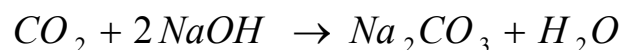
End Point : Pink color.

THEORY

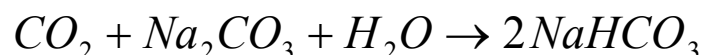
Dissolved CO₂ in water may be due to the absorption of atmospheric CO₂ or by biological oxidation of organic matter present in water.



This free CO₂ which gets dissolved in water, is determined by titrating with standard solution of NaOH using phenolphthalein as an indicator.



Free CO₂ react with alkali and produce NaHCO₃. At end point pink color appears. Na₂CO₃ solution can be used in place of NaOH.



PROCEDURE

1. Wash all the apparatus with the distilled water.
2. Rinse and fill the burette with NaOH solution and note the initial reading.
3. For standardization of intermediate NaOH solution by N/40 standard solution of Oxalic acid.
4. Pipette out 20 ml of N/40 Oxalic acid solution in conical flask and 2-3 drops of indicator (Phenolphthalein).
5. Repeat the titration till two concordant readings are obtained.
7. Pipette out 20 ml of given water sample in a conical flask and add 2-3 drops of phenolphthalein indicator.
8. Titrate the water sample against NaOH solution. The appearance of pink color indicates end point. Note down the burette reading.
9. Repeat the titration to get the concordant reading.

Observation Table
For standardisation of NaOH

S.L. No.	Volume of Oxalic acid (ml)	(Burette reading) IBR (ml) FBR (ml)	Volume of NaOH Solution used (ml)
1	20		V_2 ml
2	20		
3	20		

For Titration of Water v/S NaOH solution

S.L. No.	Volume of Water Sample (ml)	(Burette reading) IBR (ml) FBR (ml)	Volume of NaOH Concordant used (ml)
1	20		V_2 ml
2	20		
3	20		

Calculations**1. Calculation for standardization of NaOH solution.**

NaOH solution

Oxalic acid solution

$$N_1V_1 = N_2V_2$$

 N_1 = Normality of NaOH Solution V_1 = Volume of NaOH Solution N_2 = Normality of Oxalic acid Solution V_2 = Volume of Oxalic acid solution**2. Calculate for determination of free CO₂ in water sample.**

Water sample

NaOH solution

$$N_3V_3 = N_4V_4$$

 N_3 = Normality of water sample V_3 = Volume of water sample N_4 = Normality of NaOH Solution V_4 = Volume of NaOH Solution

3. Strength of CO₂ in water Sample =N₃X equivalent weight of CO₂

$$=N_3 \times 22\text{gm/L}$$

$$=N_3 \times 22 \times 1000 \text{ mg/L or ppm}$$

Results

Amount of CO₂ in a given sample of water ismg/L

Precautions

1. The solution should be well shaken before each titration.
2. Fixed amount of indicator should be added during each titration.
3. End point should be noted properly.

QUESTIONS

1. What is the range of phenolphthalein indicator ?

2. How CO_2 get dissolved in water?

3. What should be the standard value for CO_2 in water.

4. How dissolved CO_2 can be removed from water ?

5. Name the gases, dissolved in water that causes corrosion.

6. How standardization of NaOH takes place ?

7. What is the end point of CO₂ titration ?

8. Phenolphthalein gives which colour with base.

9. How to prepare $\frac{N}{10}$ oxalic acid and $\frac{N}{10}$ NaOH solution.

10. Write reactions of CO₂ production (aerobic and anaerobic reaction).

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EXPERIMENT - 4

pH METER

OBJECT

To determine the pH of a given sample by pH meter.

APPARATUS REQUIRED

pH meter, Combined pH glass electrode.

REAGENTS REQUIRED

Standard buffer solutions of pH 4 and pH7, 100 ml beaker, magnetic stirrer.

THEORY

pH meter is an instrument which is used to determine the pH of the samples. It is a solid state high resistance field effect transistor. The amplified current of the device is further calibrated in terms of potential in mV or as pH.

In the measurement of pH, a specially designed glass electrode is used. A typical glass having low melting point and high electrical conductivity containing Na₂O 21.4%, CaO 6.4%, and SiO₂ 72.2% is used to make the glass electrode. The glass electrode has a thin glass bulb filled with 0.1 N HCl in which Ag/AgCl electrode as a wire is dipped. This glass electrode works as working electrode which is coupled with a calomel reference electrode to complete the EMF cell and used to determine the pH of samples along with a pH meter. Now a day, combined pH glass electrodes are available having both the electrodes.

The cell may be represented as:

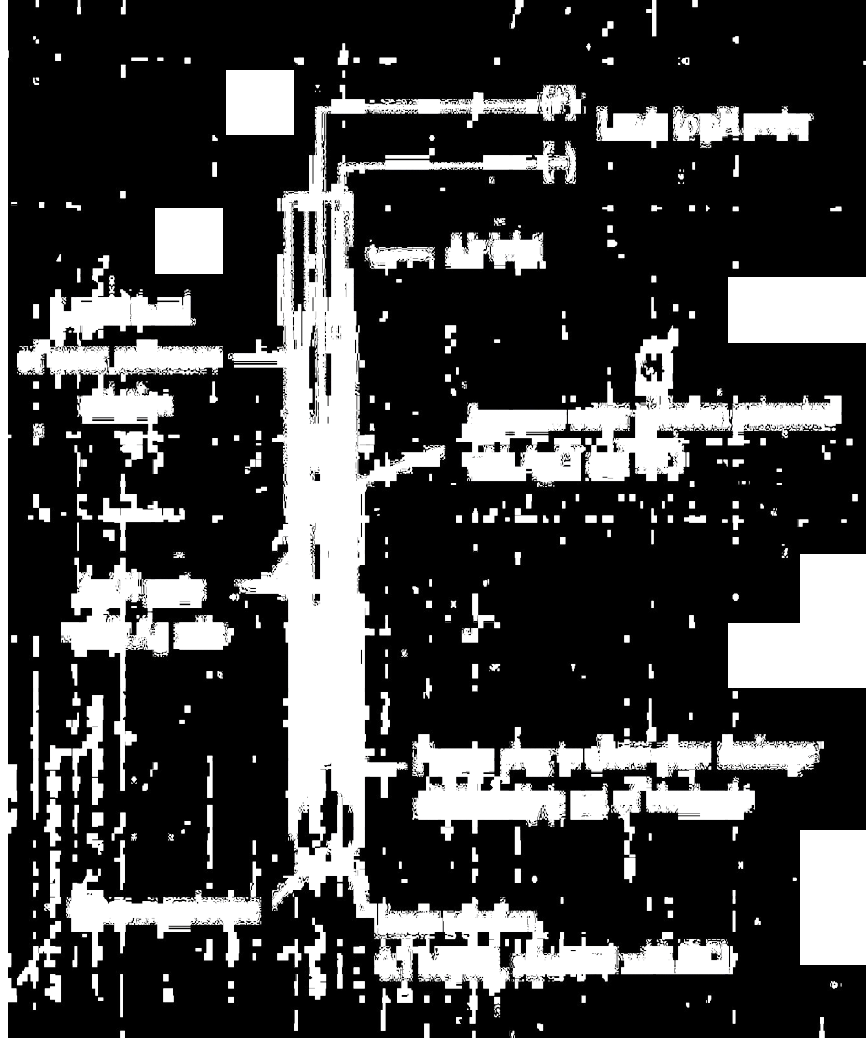
Glass Electrode/Sample Solution/Calomel Electrode
(Ag/AgCl, HCl(0.1N))

The EMF of the cell at 298 K is

$$E_{\text{cell}} = E_{\text{Calomel Electrode}} - E_{\text{Glass Electrode}}$$

$$E_{\text{cell}} = E_{\text{Calomel Electrode}} - (E^0_{\text{Glass Electrode}} + 0.0591 \text{pH})$$

$$\text{Therefore, } pH = \frac{(E^0_{\text{Calomel Electrode}} - E^0_{\text{Glass Electrode}})}{0.0591}$$



PROCEDURE

(A) Calibration of combined pH glass electrode

1. Connect the pH electrode to socket of the pH meter.
2. Keep the function switch in the stand by position.
3. Switch on the pH meter for about 15 minutes for its warm up.
4. Clean the bulb of pH electrode with distilled water and dry the surface with soft tissue paper.
5. Measure the temperature of the sample solution and adjust the temperature knob of pH meter accordingly.
6. The function switch is changed to pH mode.
7. Dip the pH electrode in the standard buffer of pH7.
8. Adjust the Calibrate control knob of the pH meter to display reading 7.00.
9. Wash the pH electrode again as mentioned in step 4 and then dip it into the standard buffer of pH4.
10. Adjust the 'Slope' knob of pH meter to display reading 4.00.
11. Remove the pH electrode wash again as step 4 for the analysis of pH of sample solution.
12. Always keep the function switch to the stand by mode after measuring the pH of sample.

(B) pH measurement

1. Always calibrate the pH meter as mentioned above.
2. Dip the properly washed and dried pH electrode in the sample solution.
3. Allow the reading to be stabilized.
4. The display shows the pH of the sample under test.

Observation Table
The pH Value of the water sample

S.L. No.	Sample of Water	pH Value	Nature

Result:

The pH of the water sample.....

PRECAUTIONS

1. The pH electrode must be properly washed and dried before its use.
2. Always keep the function switch of pH meter on stand by position after taking the pH readings.
3. Temperature knob should be set according to the temperature of the pH readings.
4. Magnetic stirrers should be used while measuring the pH for accurate measurement.

QUESTIONS

1. Define the term pH.

2. What is the effect of temperature and pressure on pH.

3. What is the possible pH of pure water at 65°C?

4. What is effect of dilution of pH of an acidic solution ?

5. What chemicals are required to make a buffer of pH (I) 5 (II) 10 ?

6. Name three electrodes, which are usually. Employed to measure pH of a solution, Which one is most suitable ?

7. What is combined electrode not generally used in pH measurement ?

8. Why hydrogen electrode not generally used in pH measurement ?

9. Define reference electrode. Why its necessary to measure pH of drugs?

10. Glass electrode is preferred to quinhydrone electrode in measuring pH of solution. Give reasons.

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EXPERIMENT - 5

WATER SAMPLE IODOMETRICALLY

OBJECT

To determine the residual free chlorine in a given water sample iodometrically.

Glasswares: Breaker, funnel, burette, pipette, conical flask.

REAGENTS REQUIRED

10% of KI solution, N/40 sodium thiosulphate solution, Starch solution, concentrate H_2SO_4 solution, potassium dichromate solution (N/40), water sample, distilled water.

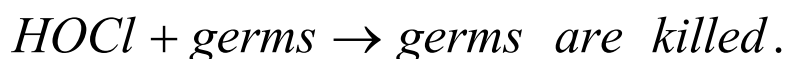
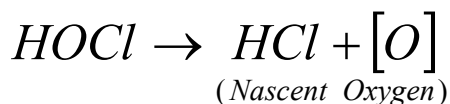
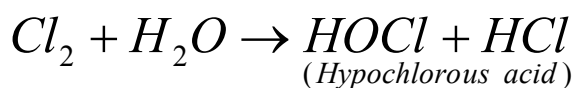
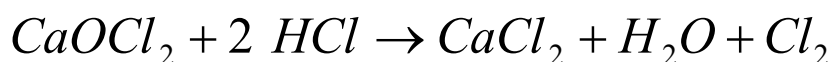
Indicator : Freshly prepared starch solution

End Point : Blue color disappears and milky white color appears.

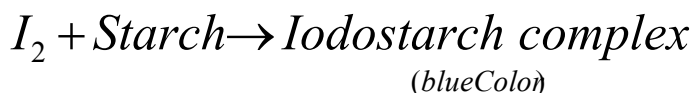
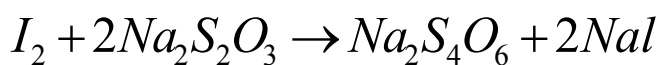
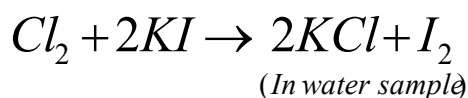
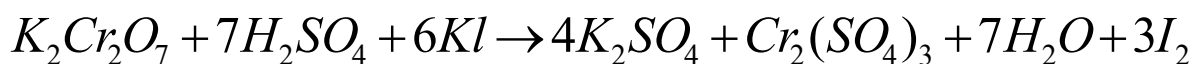
THEORY

It is an Iodometric titration method. The various methods used for disinfection of water are boiling, by adding bleaching powder, by using chloramines, U.V. Radiation, sterilization by ozone, etc.

Chlorine is widely used for disinfection and chlorination is done with the help of bleaching powder or chlorine gas or chlorine dissolved in water in the form of concentrated solution or with chloramines. The sterilizing action of chlorine is due to formation of hypochlorous acid and nascent oxygen.



When measured quantity of water sample is treated with solid potassium iodide or its standard solution then free chlorine present in water oxidizes the corresponding amount of potassium iodide to iodine. This liberated iodine is estimated by titrating with standard hypo solution, using starch as an indicator.



PROCEDURE

1. Fill the burette with hypo solution.
2. Take 20 ml of $K_2Cr_2O_7$ solution in conical flask.
3. Add 5 ml distilled H_2SO_4 and 10 ml of KI solution. Shake it vigorously and cover it with watch glass for 5 minutes.
4. Solution become brown due to the liberation of iodine.
5. Titrate this solution with hypo solution from burette.
6. When brown color fades, add 2-3 drops of freshly prepared starch indicator.
7. The color of solution become blue.
8. Again add hypo solution from burette until the blue color disappears and milky white color appears.
9. This is the end point.
10. Repeat this same procedure with water sample and get the concordant reading from burette.

Observation Tables

1. Titration of hypo solution and $K_2Cr_2O_7$ solution.

S.L. No.	Volume of $K_2Cr_2O_7$ (ml)	(Burette reading) IBR (ml) FBR (ml)	Volume of Hypo solution Consumed	Concordant Reading(ml)
1	20			
2	20			
3	20			

Observation Tables
2. Titration of hypo solution and water sample

S.L. No.	Volume of $K_2Cr_2O_7$ (ml)	(Burette reading) IBR (ml) FBR (ml)	Volume of Hypo solution Consumed	Concordant Reading(ml)
1	20			
2	20			
3	20			

Calculations:

Standardization of hypo solution

Calculation for determination of free chlorine

Hypo solution

 $K_2Cr_2O_7$ solution

Hypo solution

Water sample

$$N_1V_1 =$$

$$N_2V_2$$

$$N_3V_3 =$$

$$N_4V_4$$

Where,

 N_1 = Normality of hypo solution N_3 = Normality of hypo solution V_1 = Volume of hypo solution V_3 = Volume of hypo solution N_2 = Normality of $K_2Cr_2O_7$ solution N_4 = Normality of water sample V_2 = Volume of $K_2Cr_2O_7$ solution V_4 = Volume of water sample**Strength of free chlorine**

$$= N_4 \times \text{Equivalent weight of chlorine.}$$

$$= N_4 \times 35.5 \text{ gm/liter}$$

$$= N_4 \times 35.5 \times 1000 \text{ mg/liter or ppm.}$$

Result :

The total free chlorine in given water sample isppm.

PRECAUTIONS

1. All the glass wares should be washed with the distilled water.
2. The same amount of indicator solution must be added in each titration.
3. The solution is titrated immediately as possible.
4. End point must be noted correctly.
5. The volumetric flask must be covered with watch glass, otherwise there will not be complete liberation of iodine.
6. The solution should be well shaken before each titration.

QUESTIONS

1. Write the reactions involved in the residual chlorine determination.

2. Why water is chlorinated ?

3. What is break point chlorination ?

4. What is the utility of colour change at end point ?

5. Why the colour change at end point ?.

6. What oxidizing agents are generally present in natural water ?

7. How does chloramine water act as disinfectant .

8. Why chloramine is preferred over chorion for disinfection purpose ?

9. What is the formula for bleaching powder ?

10. How $K_2Cr_2O_7$ act as oxidizing agent. What is its equivalent weight.

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EXPERIMENT - 6

DISSOLVED O₂ IN GIVEN WATER SAMPLE

OBJECT

To determine the dissolved O₂ in given water sample.

APPARATUS REQUIRED

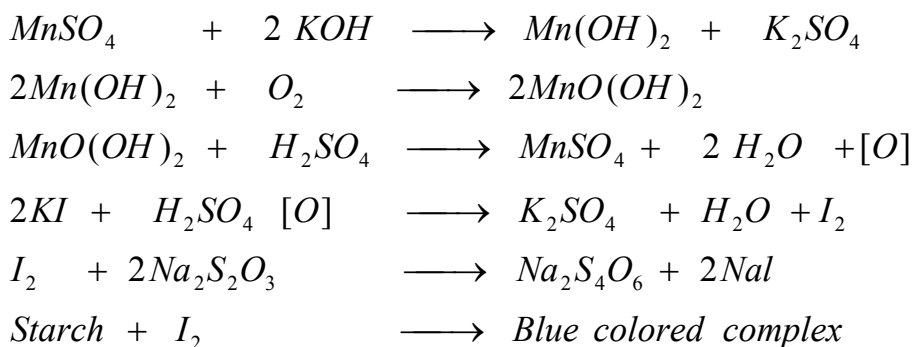
Manganous sulphate solution (4.8%) alkaline potassium iodide, conc. Hcl acid, standard thio sulphate solution (0.01N), freshly prepared starch solution, pipette, burette, glass bottle, glass rod, conical flask, measuring flask and measuring cylinder.

THEORY

In presence of good amount of dissolved oxygen, aerobic bacteria lead to oxidation of organic compound present in water this is called aerobic oxidation. If water is polluted with large amount of organic compound a large amount of oxygen is rapidly used up in biological aerobic oxidation. This decreases the dissolve oxygen which in turn decreases the pollution of aquatic life. The dissolved oxygen tests is applied mainly for determining the DO of polluted water and industrial effluents and constitute a method of controlling pollution of water sources.

The solubility of dissolved oxygen decreases with increase with concentration of salt at one atmosphere pressure the solubility of dissolve oxygen in water at 30C is about 7-8 ppm the solubility is less saline water and at given temperature decreases with increases in conc. Of impurities.

Dissolve oxygen in determine by winkler's method it is based on the fact that dissolve oxygen oxidizes KI to I₂. The liberated iodine is titrated against standard sodium thiosulphate solution using starch as indicator. Since dissolved oxygen is present in the molecular state, it as such can't oxidize KI. So manganese hydroxide generated by the action of KOH on manganese sulphate is used as an oxygen carrier to bring about the reaction between KI and oxygen.



PROCEDURE

1. Take 250 ml of the water in a bottle avoiding as far as possible contact with air.
2. To it add 2 ml of manganous sulphate solution and 2 ml of alkaline KI solution.
3. Stopper the bottle and shake the content thoroughly.
4. When the precipitates are settled, add 2 ml of conc. HCl. Shake the bottle until the precipitates have completely dissolved.
5. Allow the solution to stand for 5 minutes.
6. Take 100 ml of this solution add a drop of starch, blue color appears and titrate it against N/100 hypo solution till blue color disappears. Note down the volume of hypo solution during titration.
7. Repeat the titration to get the concordant value of hypo used.

Observation :

Normality of hypo solution (N_2) = $IV / 100$

Volume of sample water taken (V_1) = 100 ml

Observation Tables

S.L. No.	Volume of sample solution taken (ml)	(Burette reading)		Volume of Hypo solution Used $V_2 = \text{ml}$
		Initial	Final	
1				
2				
3				Concordant volume (V_2) =

Calculation:

$$\begin{aligned}
 \frac{N_1 V_1}{\text{(water)}} &= \frac{N_2 V_2}{\text{(hypo)}} \\
 N_1 \times 100\text{ml} &= 1 \times V_2 / 100 \text{ ml} \\
 N_1 &= V_2 / 10,000 \\
 \text{Normality} \times \text{Strength of oxygen} &= N_1 \times 8 \text{ gm / lit} \\
 &= V_2 \times / 10,000 \text{ gm / lit.} \\
 &= V_2 \times 8 \times / 10,000 \text{ gm / lit.} \\
 &= 0.8 V_2 \text{ mg / lit.}
 \end{aligned}$$

Result :

The amount of dissolved oxygen in the given sample of water is.....

PRECAUTIONS

1. Contact of water with oxygen / air should be avoided.
2. No bubble should be present in the bottle as it will interfere with the reaction and will lead to higher concentration of oxygen.

QUESTIONS

1. What is the optimum value for dissolved O_2 in water ?

2. Is dissolved oxygen necessary in water ?

3. What is the meaning of fixation of dissolved oxygen ?

4. Why starch indicator is added near to end point ?

5. How dissolved oxygen can be removed from water ?

6. Why Iodine is not used as self indicator instead of starch ?

7. Why starch is not used in strongly acidic medium ?

8. What is the formula of hypo ?How the stock solutions are prepared?

9. Why only hypo is used in this titration?

10. Can dissolved oxygen be removed by adding sodium sulphite ? Give reaction.

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EXPERIMENT - 7

GIVEN SAMPLE BY CONDUCTIVITY METER

OBJECT

To determine conductivity of a given sample by conductivity meter.

APPARATUS REQUIRED

Conductivity meter, Conductivity cell, Thermometer, beaker.

REAGENTS REQUIRED

Standard KCl solution, Distilled water.

THEORY

Conductivity is a good indication of water quality. Conductivity is widely used in the ultra pure water industry where no ions are desired in the water. Conductivity levels of .055 micro s/cm are very common in this application. For this application, the term resistivity is commonly used instead of conductivity due to the low conductivity values. Resistivity is the inverse of conductivity. The units are Megohms. Ultra pure water typically has values of 18.3 Megohms.

S.L. No.	Type of water	Conductivity
1	Ultra Pure	0.05 $\mu\text{S/cm}$
2	Deionized water	0.10 $\mu\text{S/cm}$
3	Tap water	100 $\mu\text{S/cm}$
4	Sea water	10.0 m S/cm
5	NaOH	1 S/cm

Conductivity is a numerical expression of the ability of a water sample to carry an electrical current and varies with the number and types of ions the solution contains. Most dissolved inorganic substances in water are in the ionized form and hence contribute to conductance. Conductance G is defined as the reciprocal of resistance R.

$$G = \frac{1}{R} \quad \text{Where, R in ohm and G in ohm}^{-1} \text{ (sometimes written as mho)}$$

Conductance of a solution is measured between two spatially fixed chemically inert electrodes. To avoid polarization at the electrode surface, the conductance measurement is made with an alternating current signal.

The conductance (G) of a solution is directly proportional to the surface area (A, cm²) and inversely proportional to the distance between the electrode (L, cm). The constant of proportionality (k) is such that

$$G = k \frac{A}{L}$$

k is called “conductivity” (preferred to “specific conductance”). The unit of k is 1/ohm-cm or mho per centimeter. It can be defined as the conductance of conductor 1 cm in length and 1 cm² in cross-sectional area. The specific conductance depends on the nature of the conductor (the solution between the electrodes), the ion concentration and pressure.

The conductance of a solution measured by conductivity cell depends on cell parameters, A and L. Although theoretically one could calculate the specific conductance from measurements of G (or R) and values of A and L, it is easier and more accurate to use a solution of known specific conductance, measure G and calculate the ratio A/L. The specific conductance of a solution is calculated from

$$\text{Specific conductance, } k = G \times K_c$$

Where K_c is equal to L/A and is known as the cell constant. Typical values of cell constant ranges from 0.10 to 2cm⁻¹.

Therefore, the cell constant (K_c , cm⁻¹) is calculated as follows:

$$K_c = \frac{\text{Conductivity of standard KCl solution, in mho/cm}}{\text{Meter conductance}}$$

It should be noted that, measured and true conductivity is expressed in same unit.

The temperature of the solution is critical, because electrical conductivity increase with temperature at a rate of approximately 1.9% per °C.

1. Switch on the conductivity meter for about 15 to 20 minutes.
2. Set the function switch to check position.
3. Display must read 1.000 otherwise set it with CAL control knob of conductivity meter.
4. Put the function switch to cell constant position & adjust it to the value of conductivity cell.
5. Set the temperature control to the actual temperature of the solution under test.
6. Dip the washed and dried conductivity cell in the breaker containing sample solution and connect the cell to the input socket of the conductivity meter.
7. Change the function switch to conductivity position and bring the range switch at a position of maximum resolution.
8. Note display reading as a conductivity of sample solution.

It is highly recommended to calibrate the cell constant of the conductivity cell before measuring the conductivity of any sample. Following steps are to used for calibrating the cell constant :

1. Dip the conductivity cell into the standard KCl solution whose conductivity is also known at a particular temperature.
2. Put the function switch to normal position and adjust the range switch to an appropriate range.
3. Set the reading in the display to the conductivity value of standard KCl solution by a cell constant knob.
4. The function switch is changed to CAL position and display shows the calibrated cell constant of the conductivity cell.

Observation Table

S.L. No.	Sample	Conductivity
1	Sample No. 1	
2	Sample No. 2	
3	Sample No. 3	
4	Sample No. 4	
5	Sample No. 5	

Result :

The conductivity of given sample is.....

Precautions

1. Always calibrate the cell constant of conductivity cell.
2. Temperature of the sample should be carefully noted as conductivity varies with the temperature.
3. Conductivity cell should be completely dipped in the solution.
4. After completion of the experiment, the conductivity cell should be properly washed and dipped into distilled water.

QUESTIONS

1. Define conductance. Give its units.

2. Define Ohm's law.

3. What is cell constant? Give its units.

4. What is specific conductance and What are its units.

5. What is equivalent conductance?

6. What are units of equivalent conductance ?What are different methods by which turbidity can be reduced?

7. What is molecular conductance ? Give units.

8. Why ordinary water is unsuitable for conductivity measurements? What is the conductivity water.

9. State the principle on which the conducto meter is based ?

10. What is the effect of dilution on conductance ?

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EXPERIMENT - 8

FLUORIDE IN WATER

OBJECT

To determine the fluoride in water.

Spectrophotometric measurement of fluoride.

APPARATUS REQUIRED

Pipettes, Measuring flasks, Beakers, Spectrophotometer, Quartz cuvette of cm light path.

REAGENTS REQUIRED

SPADNS [Sodium -2 (p-sulfophenylazol)- 1,8-dihydroxy-3, 6-naphthalene disulfonate], Zirconyl chloride octahydrate, Concentrated HCl, Sodium fluoride, distilled water.

THEORY

Spectrophotometric measurement of fluoride is also called SPADNS method. In this method absorbance or transmittance is measured between 550 to 580 nm for the fluoride present in the sample solution. The color is developed as per the concentration of fluoride because of reaction of fluoride and zirconium ions in acidic medium using SPADNS dye.

Procedure

- 1. Preparation of stock and standard fluoride solution :** Prepare the solutions of NaF as mentioned in the potentiometric determination of fluoride.
- 2. Preparation of SPADNS solution :** Dissolve 958mg of SPADNS in distilled water and dilute it to 500 ml. Solution is stable for one year if protected from direct sunlight.
- 3. Preparation of acid zirconyl solution :** Dissolve 133 mg of zirconyl chloride in 25 ml of distilled water. Add 350 ml of concentration HCl and dilute upto the 500 ml with distilled water.
- 4. Preparation of acid zirconyl SPADNS solution :** Mix equal volumes of SPADNS solution and acid zirconyl solution. This solution is stable for about 2 years.
- 5. Preparation of sodium arsenite solution :** Dissolve 5.0 gm NaAsO₂ and dilute it to litre with distilled water. This solution is toxic, so use it carefully.
- 6. Preparation of reference solution :** Take 10 ml of SPADNS solution and add 100 ml of distilled water. Take 7 ml of conc. HCl and it to 10 ml with distilled water and this to SPADNS solution. This solution will be used for setting the instrument the reference point (zero) and is stable for about 1 year.

7. Preparation of calibration curve:

- i) Prepare fluoride standard solutions in the range of 0 to 1.40 mg/L.
- ii) Take 50 ml of standard solution.
- iii) Take 10 ml acid zirconyl-SPADNS solution & mix thoroughly with each standard solution.
- iv) Switch on the spectrophotometer for sufficient time before taking the reading for proper warm up.
- v) Set the spectrophotometer to zero absorbance with reference solution.
- vi) Take absorbance reading for standard solutions of F at 550 nm.
- vii) Prepare the standard curve of absorbance against concentration of F.
- viii) Take absorbance reading for unknown sample solution of fluoride.

Observation Table

SL. No.	Concentration (mg/litre)	Reading of UV-Vis spectrometer at 550nm
1	Reading with standard solution	
2		
3		
4		
5		
6	Reading of unknown sample	

Calculation

Actual concentration of F^- in the sample can be obtained by converting the potential difference reading from the standard curve.

Result

Fluoride content in given water sample is ppm.

PRECAUTIONS

1. The glass used should be thoroughly washed with distilled water and dried.
2. Take proper care while using quartz cuvette.
3. Before and after the use clean the electrode properly with distilled water and dry it gently with tissue paper.
4. Prepare standard solution accurately.

QUESTIONS

1. Give basic principle of spectrophotometer.

2. Give acceptable limit of fluoride in water.

3. How fluoride can be estimated in water?

4. In spectrophotometric method at which wavelength fluoride absorbs ?

5. Give name of SPANDS.

6. Other than spectrophotometer, by which method fluoride can be measure in water?

7. How the fluoride standard stock solution is prepared ?

8. Give preparation of reference solution.

9. What is buffer, How it is prepared ?

10. What is the meaning and utility of calibration curve ?

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EXPERIMENT - 9

NITRATE IN WATER

OBJECT

Measurement of nitrate in water.

APPARATUS REQUIRED

Spectrophotometer (3000-700 nm), Porcelain dish, pipettes, measuring flask, distilled water, phenol disulphonic acid, Ammonium hydroxide, standard nitrate solution.

THEORY

Nitrate in contact with sulphuric acid which in dry conditions (in presence of excess concentrated H_2SO_4) brings about nitration of phenol disulphonic acid. This nitrophilic product gives intense yellow color in alkaline medium which is measured through colorimeter. The reaction must proceed in cold otherwise nitric acid may be lost by volatilization).

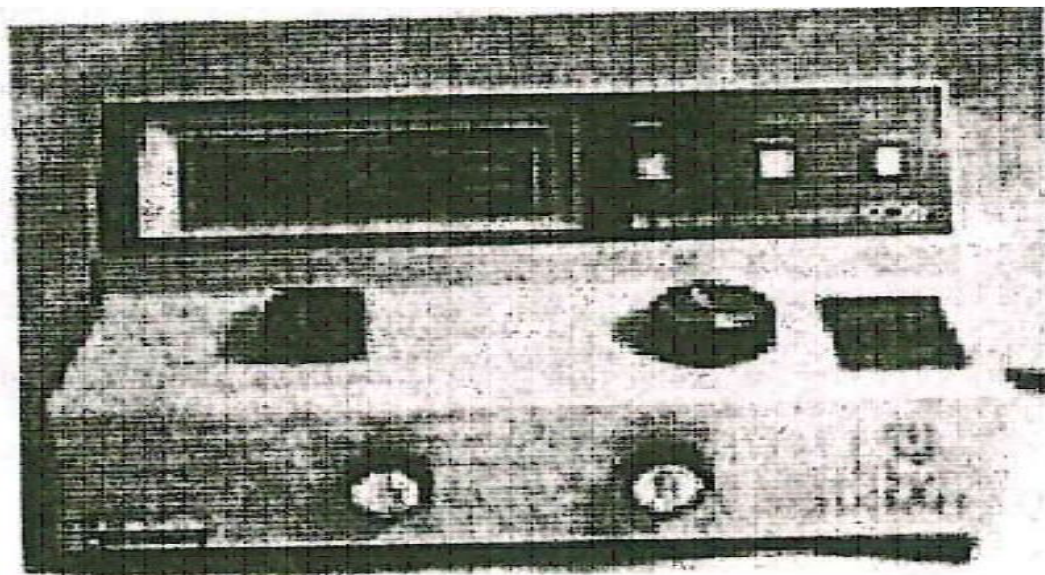


Fig. Spectrophotometer

PROCESS

1. Take 50 ml of sample in porcelain dish and evaporate it to dryness on a hot water bath.
2. Add 3 ml of phenol disulphonic acid to the residue and dissolve the latter by rotating the dish.
3. After 10 minutes ml of distilled water is added and stirred with a glass rod on cooling the contents are washed down in to 100ml volumetric flask.
4. Add ammonia (1.1) slowly with mixing till the solution is alkaline as indicated by the development of yellow color due to the presence of nitrate. Then add another 2 ml of ammonia and the volume made up (100) with distilled water.
5. Intensity of yellow color is read in the color is read in the colorimeter at 420 nm (blue filter).
6. Calculated the concentration of nitrate from the standard curve.
7. Prepare the standard curve using suitable aliquots of standard nitrate solution in the range of 5 to 500 mg No 3 by following the above procedure.

Preparation of standard curve for nitrate

A stock containing 100 ppm nitrate (NO_3^-) is prepared by dissolving 0.7215 g of AR grade potassium nitrate (Oven-dried and cooled) in distilled water and making the volume to one litre.

This is diluted ten times to give are 10 ppm NO_3^- solution. Aliquots (2,5,10,15,20&25 ml) are evaporated on boiling water bath to dryness in small porcelain dishes (or beakers).

When cool, 3 ml of phenol disulphuric acid is added and the yellow coulor is developed and read .A blank, (without nitrate) must be run and correction made by adjusting the colorimeter to zero with blank.

A calibration curve is drawn between concentration of NO_3^- and colorimeter reading (absorbance) of sample of different concentrations.

Calculations

$\text{mg of NO}_3^- / \text{litre} = \text{mg of NO}_3^- \text{ from standard curve} / \text{ml of sample.}$

Result

The observed value of nitrate in the given sample of water ismg/L

PRECAUTIONS

1. Analysis should be made as soon as possible.
2. If analysis can be made within 24 hours the sample should be preserved by refrigeration at 4°C.
3. The glass wares should be thoroughly washed with distilled water and dried.
4. Take proper care while using quartz cuvette.
5. Clean it before and after the experiment and wipe it gently with soft tissue paper.

QUESTIONS

1. How nitrogen can be estimated ?

2. At which wave length nitrate can be analysed in spectrophotometric analysis.

3. Give formula for calculating Nitrate concentration.

4. Name disease caused by excess amount of Nitrate present in water.

5. Differentiate metallic and electrolytic conductors ?

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EXPERIMENT -10

SULPHATE IONS

OBJECT

To determine the amount of Sulphate ions present in given water sample.

APPARATUS REQUIRED AND REAGENTS REQUIRED

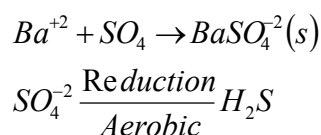
Drying oven, Analytical Balance, Muffle furnace, desiccator, Glass water like funnel, filter paper, steam bath, Methyl Red indicator solution, silver nitrate, nitric acid reagent, Hydrochloric acid.

Indicator: Methyl Red.

THEORY

Sulphate is widely distributed in water and may be present in natural water to concentration ranging from a few to several thousand milligrams/ litre.

Sulphate are of great concern because they are indirectly responsible for two serious problems after associated with the handling and treatment of waste water odour and corrosion problem result from the reduction of sulphates on addition of barium chloride in presence of hydrochloric acid sulphates precipitate as barium sulphate. The precipitation is carried out near the boiling temperature and offer a period of digestion, the Precipitates are filtered, washed with water until free from chloride, dried and weighted as barium sulphate.



PROCEDURE

1. About 100 ml of water sample is transferred in a breaks.
2. Two drops of Methyl red indicator are added to it.
3. Concentrated Hcl is added to it dropwise, till the colour change to pink.
4. Two drops of conc. Hcl are added in excess.
5. It is heated to nearly boiling, then hot barium chloride solution is added to it with stirring until the formation of white precipitate is complete, 2 drops of hot BaCl₂ are added in excess.
6. The precipitate (BaSO₄) is digested for half hours.

7. It is filtered, is washed several times with distilled water, until the washing are all free from chloride ions.
8. It is filtered using whatman filter paper No. 42 .
9. After it completely drains out the filter paper along with precipitate is transferred carefully into previously weighted crucible and heated at 800-900°C till all traces of filter paper are burnt.
10. The crucible is cooled in a desiccator and finally weighted.

Observations:

1. Weight of empty crucible = W_1 gm
2. Weight of crucible + $BaSO_4$ ppt = W_2 gm
3. Weight of $BaSO_4$ ppt = $(W_1 - W_2) = x$ gm SO_4^{-2}
4. Volume of water sample taken = 100 ml

Calculation:

Molecular weight of $BaSO_4 = 232.4$

Ba = 137.4, S = 32, O₄ = 64

232 gms. Of $BaSO_4$ Contains = 96 gm of SO_4^{-2}

$$1 \text{ gm of } BaSO_4 \text{ Contains} = \frac{96}{232} \text{ gm of } SO_4^{-2}$$

$$x \text{ gm of } BaSO_4 \text{ contains} = \frac{96}{232.4} \times \text{gm } SO_4^{-2}$$

$$100 \text{ ml water sample contains} = x \text{ gm } BaSO_4 = \frac{96}{232.4} \times \text{gm of } BaSO_4$$

$$1 \text{ ml of water sample contains} = \frac{96}{232.4} \times \frac{x}{100}$$

$$100 \text{ ml. Of water sample contains} = \frac{96x \times 1000}{232.4 \times 100 \times \text{mg } BaSO_4} \text{ gm } BaSO_4$$

$$= y \text{ mg or ppm } BaSO_4$$

Result

The amount of sulphate ions in water sample is-----ppm.

PRECAUTIONS

1. Precipitation is carried out in dilute solution and in hot condition.
2. Some dilute HCl must be added before precipitation.
3. Digest the precipitate in boiling water bath till crystalline precipitate settle down.
4. Since, H_2SO_4 is Volatile, so prefer it as precipitating agent.

QUESTIONS

1. How sulphate occurs in water ?

2. What will be the effects if water is contaminated by sulphate?

3. What is post precipitation ?

4. Name the methods by which sulphate ions in water are measured ?

5. Why digestion of precipitate is done ?

6. Define co-precipitation.

7. Why phosphoric acid is added when, diphenyl amine is used as an Indicator.

8. Give structure and formula of diphenylamine.

9. Give commercial name of Na_2CO_3 and NaHCO_3 .

10. Why potassium ferricyanide is not used as an internal Indicator ?

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EXPERIMENT-11

TDS MEASUREMENT

OBJECT

Evaluation of Reverse Osmosis (RO) process by TDS measurement

APPARATUS REQUIRED AND REAGENTS REQUIRED

R.O. silica crucible, beaker, pipette, measuring cylinder, desiccator, distilled water, weighing balance, whatman filter paper No. 44.

THEORY

Natural Osmosis is the process in which flows from a less concentrated solution through semipermeable membrane to a more concentrated saline solution until the concentrations on both sides of the membrane are equal. Reverse Osmosis requires external pressure to reverse natural osmotic flow. Semipermeable membrane rejects bacteria, Pyrogens and about 85-95% of inorganic solids.

This technique is used to purify the water. To test the effectiveness of R.O. process we can check the water by testing its hardness, nitrates and total dissolved solids before and after using the process. Here, we are determining the TDS using gravimetry. The process can be done by using the TDS meter also.

Dissolved solids can be determined by evaporating a pre-filtered sample to dryness, and then finding the mass of the dry residue per liter of sample. The TDS concentration in water body is affected by many different factors. A high concentration of dissolved ions is not, by itself an indication that a stream is polluted or unhealthy.

It is normal for dissolved and accumulate fairly high concentrations of ions from the minerals in the rocks and soils over which they flow. Fertilizers from fields and lawns can add a variety of ions to a stream. Increase in TDS can also result from runoff from roads that have been salted in the winter. Organic matter from wastewater treatment plants may contribute higher levels of nitrate or phosphate ions. Measurement of dissolved solids gives information about degree of pollution in the effluents, sewage and water.

Known volume of well-mixed sample is first filtered through whatman filter paper No. 44, so that all the suspended solids can be removed as residue on the filter paper. The filtrate of the sample has the dissolved solids which is taken in the pre weighed crucible or beaker. Place the filtered sample in at 103-105°C. Cool the crucible or beaker in the desiccator and weigh it for calculating dissolved solids in the sample.

PROCEDURE

1. Mix the water sample thoroughly to homogenize it.
2. Take known volume (V ml) of sample and filter it through whatman filter paper No. 44.
3. Take the filtrate in the dried and pre weighed crucible or beaker (W_1 gm).
4. Evaporate the sample to dryness.
5. Place the sample in the oven for about 1 hour at 103-105°C.
6. Cool the sample in the desiccator to room temperature.
7. Weight the crucible or breaker (W_2 gm) again with residue left in it as dissolved solids.
8. Repeat the process of drying and cooling to get constant weight.

Observations

1. Volume of sample taken = V mL.
2. Weight of empty crucible or beaker dried at 103-105°C = W_1 gm
3. Weight of crucible or breaker residue dried at 103-105°C = W_2 gm
4. Weight of dissolved solids in V mL of sample = $(W_2 - W_1)$ gm

Calculations

Dissolved solids in V mL of sample = $(W_2 - W_1)$ gm

$$\begin{aligned} \text{Therefore, dissolved solids in 1 litre of sample} &= \frac{(W_2 - W_1)}{V} \times 10^3 \text{ gm / L} \\ &= \frac{(W_2 - W_1)}{V} \times 10^6 \text{ gm / L or ppm} \end{aligned}$$

Result

Dissolved solids present in the given water sample = ppm

PRECAUTIONS

1. Heat the sample carefully at specified temperature.
2. Cool the hot dried crucible or beaker in desiccator.
3. Take the weights accurately.
4. Always use clean crucible or beaker.

QUESTIONS

1. What is reverse Osmosis?

2. Give the principle of reverse Osmosis.

3. What is the full form of TDS?

4. Which materials are used for RO membranes?

5. Give details of RO membranes.

6. Is RO process is a green technology (environment Friendly)?

7. Is mineral water better than RO water?

8. Name the minerals essential for human body.

9. What is the adequate amount of turbidity in water?

10. Give proper amount of total dissolved solids in potable water.

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