SRM VALLIAMMAI ENGINEERING COLLEGE

(An Autonomous Institution)

SRM Nagar, Kattankulathur – 603 203

DEPARTMENT OF CHEMISTRY

GE3121-Chemistry Laboratory Manual

Regulations 2023

Semester I

(2025-26)

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GENERAL INSTRUCTIONAL POLICIES TO BE FOLLOWED IN THE CHEMISTRY LABORATORY

1. Students must wear a knee length (41-42 inch) laboratory coat while entering into the laboratory.
2. Students must wear closed leather shoe (black or brown colour) for safety purpose.
3. Students must always come to the chemistry lab with lab manual, observation note, and record note.
4. Students should bring waste cloth for cleaning their hands.
5. Students may wear chemical safety goggles, if it is required.
6. Students should report to the concerned chemistry lab as per the time-table schedule.
7. Students, who turn up late to the lab, will not be permitted to do the experiment.
8. Students are required to prepare thoroughly to perform the experiment before coming to chemistry laboratory
9. After completion of the experiment, students must get the signature of the concerned staff in-charge in the observation note.
10. The record note must be submitted to get signature of the previous week experiment to the concerned staff while coming to lab.
11. When the experiment is completed, students should clean the apparatus carefully and should return all the reagent bottles/components/instruments taken for the experiment.
12. Any damages to apparatus occurred during the experiment, should be brought to the notice of lab in-charge. The cost of the repair or new apparatus should be brought by the students.
13. Students should be present in the lab till the completion of allotted hours of lab.
14. Students should not sit on the work tables or keep hands for rest on tables for their own safety.

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15. All doors and windows must be kept open while working in the laboratory. Fans and lights should be switched off, if they are not required.

16. Long hair should be tied when in the laboratory so that it will not catch on fire or come into contact with chemicals.

17. Do not enter the laboratory until your lab instructor is present.

18. While leaving the lab ensure that all gas tap connections, water taps, electrical switches such as and fans, lights shall be kept in off mode. All instruments must be properly shut down as per instructions of the teacher in charge.

19. Do not eat, drink and chew gum or smoke in the laboratory.

20. Do not perform unauthorized experiments. If you see someone else doing something you think may be dangerous, tell him or her to stop and/or report the incident to your lab instructor.

21. In case of emergency, do not get panicky and be brave enough to handle the situation. Medical attention should be requested immediately. If skin comes into contact with acid solutions, it should be washed with water and sodium bicarbonate solution alternately and continuously till skin is smoothened. Similarly exposure to caustic alkalis may be treated with dilute acetic or boric acid solutions and water. Fire scar can be washed thoroughly with water in a tap in the sink and a little oil or pipette be applied. If eyes come into contact with chemicals, wash them with plenty of water and clean them with a dry fresh cloth.

22. Fire buckets containing sand and water or fire extinguishers should always be available in the lab in easily accessible places.

Vision of the Chemistry Department

Bridge the engineering professionals in recent advancements in chemistry for the developments of new devices and materials for the lives of individuals becoming different and complex global society.

Mission of the Chemistry Department

To draw up outstanding educators, employers and researchers and to make progress in the profession of education through teaching and learning and art of research on chemical science towards engineering & technology.

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GE3121 **PHYSICS AND CHEMISTRY LABORATORY**

(First Semester- Common to all branches)

CHEMISTRY LABORATORY: (Any five experiments to be conducted)

OBJECTIVES

- To inculcate experimental skills to test basic understanding of water quality parameters, such as acidity, alkalinity, hardness, DO, TDS, chloride, and chlorine.
- To familiarise the students with electroanalytical techniques like conductometry and flame photometry to determine the impurities in aqueous solution.
- To find the various characteristics of domestic water.
- To understand the Pseudo first-order kinetics reaction.
- To make the students know the synthesis of nanoparticles.

1. Determination of chloride content of water sample by Argentometric method.
2. Determination of total, temporary & permanent hardness of water by EDTA method.
3. Determination of DO content of water sample by Winkler's method.
4. Preparation of Na_2CO_3 as a primary standard and estimation of acidity of a water sample using the primary standard.
5. Determination of types and amount of alkalinity in water samples.
6. Estimation of available chlorine in bleaching powder solution.
7. Conductometric titration of barium chloride against sodium sulfate (Precipitation titration).
8. Estimation of sodium /potassium present in water using a flame photometer.
9. Estimation of TDS of a water sample by gravimetry.
10. Preparation of nanoparticles (Ag/Au/TiO_2/ZnO/CuO).
11. Pseudo first-order kinetics- ester hydrolysis.

TOTAL: 30 PERIODS

OUTCOMES

At the end of the course, the student should be able:

1. To infer the quality of water samples for alkalinity, hardness, DO, TDS, chloride, and chlorine.
2. To apply the knowledge on the estimation of metal ions, acidity and its precipitation nature towards their process.
3. To recognize the threshold limit for various characteristics of domestic water.
4. To identify the simple method of synthesis of nanoparticles.
5. To understand the pseudo first-order kinetics reaction from ester hydrolysis.

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TEXTBOOKS

1. Vogel's Textbook of Quantitative Chemical Analysis (8th Edition, 2014).
2. Suchi Tiwari, Engineering Chemistry Lab Manual, Scitech Publications (India) Pvt. Ltd. (2nd Edition, 2013).
3. Pushpendra Kumar, Laboratory Manual for Engineering Chemistry, Reyansh Authortopic Pvt. Ltd., (1st Edition, 2022).

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Titration 1: Standardization of silver nitrate

Standard NaCl vs $AgNO_3$

Sl.No	Volume of std. NaCl (ml)	Burette reading (ml)		Volume of $AgNO_3$ (ml)	Concordant value (ml)	Indicator
		Initial	Final			
1.	20	0				K_2CrO_4
2.	20	0				

Calculation of the strength of silver nitrate

Volume of standard NaCl $(V_2) = 20$ ml

Strength of standard NaCl $(N_2) = 0.01$ N

Volume of unknown $AgNO_3$ $(V_1) =$_____ml

Strength of unknown $AgNO_3$ $(N_1) = ?$

According to volumetric formula, $V_1N_1 = V_2N_2$

$N_1 = V_2 \times N_2/V_1$

$N_1 = 20 \text{ ml} \times 0.01 \text{ N}/V_1$

Strength of $AgNO_3$ (N_1) = _____ N

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1. DETERMINATION OF CHLORIDE CONTENT OF WATER SAMPLE BY ARGENTOMETRIC METHOD (MOHR'S METHOD)

Expt. No.	

Date			

AIM

To estimate the amount of chloride ion present in the water sample by Argentometric method (Mohr's method). You are provided with standard NaCl solution of strength 0.01 N and a link solution of $AgNO_3$ (approximately 0.01N).

CHEMICALS REQUIRED

Standard NaCl solution, $AgNO_3$ solution, Potassium chromate indicator.

PRINCIPLE

Natural water contains chloride ions in the form of NaCl, KCl, $CaCl_2$, $MgCl_2$. The concentration of chloride ions more than 250 ppm is not desirable for drinking purposes. This determination is based on precipitation titration. When $AgNO_3$ solution is added to the water sample, in presence of K_2CrO_4, the chlorides present in it are precipitated first as AgCl.

$$AgNO_3 + NaCl \longrightarrow AgCl \downarrow + NaNO_3$$

In water White ppt.

When all the Cl^- ions are removed, $AgNO_3$ added from the burette will react with K_2CrO_4 to give a reddish brown colour due to silver chromate (Ag_2CrO_4). This is the end point.

$$2\,AgNO_3 + K_2CrO_4 \rightarrow Ag_2CrO_4 \downarrow + 2KNO_3$$

Yellow Reddish brown

PROCEDURE

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Titration 1: Standardisation of $AgNO_3$

The burette is washed well with distilled water and rinsed with small amount of $AgNO_3$ solution. The pipette is washed with distilled water and rinsed with small amount of standard NaCl solution. 20ml of this solution is pipetted out into a clean conical flask. 1 ml or 3 drops of K_2CrO_4 indicator solution is added and titrated against $AgNO_3$ solution taken in the burette. The end point is the change of colour from yellow to reddish brown. The titration is repeated for concordant values.

Titration 2: Estimation of Chloride

Water sample vs Standard $AgNO_3$

Sl.No	Volume of std. water sample (ml)	Burette reading (ml)		Volume of $AgNO_3$ (ml)	Concordant value (ml)	Indicator
		Initial	Final			
1.	20	0				K_2CrO_4
2.	20	0				

Calculation of the normality of water sample (Chloride ion)

Volume of unknown water sample (V_1) = 20 ml

Strength of unknown water sample (N_1) = ?

Volume of standardized $AgNO_3$ (V_2) =_____ml

Strength of standardized $AgNO_3$ (N_2) =_____N

According to volumetric formula, $V_1N_1 = V_2N_2$

$N_1 = V_2 \times N_2/20$

Strength of water sample (N_1) =_____N

Calculation of amount of chloride

Amount of chloride ion present in 100 ml given water sample = Eq. wt. of chloride ion x Normality of chloride ion x 100/1000 g

= 35.46 x Normality of chloride ion x 100/1000 g

= ……..g

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Titration 2: Estimation of Chloride ion

The given water sample is made up into 100 ml in a standard flask using distilled water. 20ml of this solution is pipetted out into a clean conical flask and 1ml of 2% K_2CrO_4 indicator solution is added. It is then titrated against standard $AgNO_3$ solution taken in the burette. The end point is the change of colour from yellow to reddish brown. The titration is repeated for concordant values.

RESULT

The amount of chloride ion present in 100 ml of the given water sample =..... g.

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Titration 1: Determination of Total Hardness

Sample hard water Vs Standard EDTA

Sl.No	Volume of hard water sample (ml)	Burette reading (ml)		Volume of EDTA (ml)	Concordant value (ml)	Indicator
		Initial	Final			
1.	20	0				Eriochrome Black-T
2.	20	0				

Calculation of the total hardness of water sample

Volume of unknown hard water sample (V_1) = 20 ml

Strength of unknown hard water sample (N_1) = ?

Volume of standard EDTA (V_2) =_____ml

Strength of standard EDTA (N_2) = 0.01 N

According to volumetric formula, $V_1N_1 = V_2N_2$

$N_1 = V_2 \times N_2/20$

=

Strength of hard water sample (N_1) =_____N

Total hardness of the hard water sample = N_1 x 50 x 1000

= ---------- ppm

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2. DETERMINATION OF TOTAL, TEMPORARY & PERMANENT HARDNESS OF WATER BY EDTA METHOD

Expt. No.	

Date			

AIM

To estimate the amount of total hardness, permanent hardness and temporary hardness of a given hard water sample by EDTA method. You are provided with a standard solution of calcium of strength 0.01 N.

CHEMICALS REQUIRED

EDTA, Eriochrome black-T, water sample, ammoniacal buffer solution.

PRINCIPLE

The amount of calcium and magnesium ions present in a given sample of water is estimated by titration with standard EDTA. The hardness is expressed in terms of calcium carbonate equivalence.

Hardness

It is one of the important characteristics of water. Hardness of water is due to the presence of calcium and magnesium salts. Lather is not produced until the cations, Ca^{2+} and Mg^{2+} are completely precipitated out in the form of insoluble salts. For example,

$$\underset{\text{Sodium stearate}}{2C_{17}H_{35}COONa} + \underset{\text{(in water)}}{CaSO_4} \longrightarrow \underset{\text{(ppt)}}{(C_{17}H_{35}COO)_2Ca} + Na_2SO_4$$

$$\underset{\text{Sodium stearate}}{2C_{17}H_{35}COONa} + \underset{\text{(in water)}}{MgCl_2} \longrightarrow \underset{\text{(ppt)}}{(C_{17}H_{35}COO)_2Mg} + 2NaCl$$

Temporary hardness

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It is due to bicarbonates of calcium and magnesium. It can be removed by boiling. The salts are precipitated as carbonates and hydroxides respectively, which can be removed by filtration leaving only permanent hardness producing salts.

Permanent hardness

It is due to the sulphates and chlorides of calcium and magnesium.

Titration 2: Determination of permanent hardness

Boiled hard water vs Standard EDTA

Sl.No	Volume of hard water (ml)	Burette reading (ml)		Volume of EDTA (ml)	Concordant value (ml)	Indicator
		Initial	Final			
1.	20	0				Eriochrome Black-T
2.	20	0				

Calculation of the permanent hardness of water sample

Volume of unknown hard water sample (V_1) = 20 ml

Strength of unknown hard water sample (N_1) = ?

Volume of standard EDTA (V_2) =_____ml

Strength of standard EDTA (N_2) = 0.01 N

According to volumetric formula, $V_1N_1 = V_2N_2$

$N_1 = V_2 \times N_2/20$

=

Strength of hard water sample (N_1) =_____N

Permanent hardness of the hard water sample = N1 x 50 x 1000

= ---------- ppm

Temporary hardness = Total hardness - Permanent hardness

= ---------- ppm

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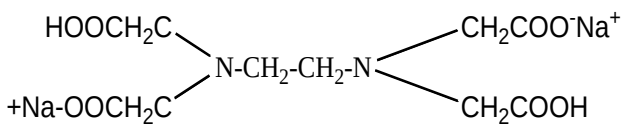
Hardness is expressed in terms of $CaCO_3$ equivalence

The reason for choosing $CaCO_3$ as the standard for calculating hardness of water is due to its molecular weight being 100, which makes the calculation easier. It is the most insoluble salt, thus can be easily precipitated in water treatment processes.

Determination of hardness

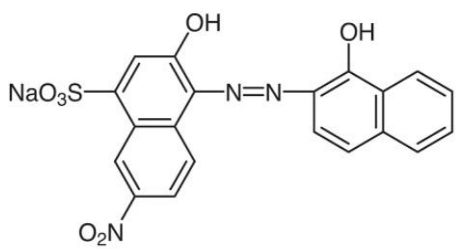
The hardness of water can be determined by complexometric titration. EDTA is used as a complexing agent. The Ca^{2+} and Mg^{2} present in water are titrated with EDTA using Erichrome Black T as indicator.

The Chemical structure of EDTA (Ethylene diamine tetra acetic acid) is represented as below:



The indicator Eriochrome Black-T (EBT), forms a wine red colored unstable complex with calcium and magnesium ions in hard water at a pH of 8 – 10.

STRUCTURE OF EBT



$$Mg^{2+}/\ Ca^{2+} \quad + \quad EBT \longrightarrow (Mg/Ca - EBT)$$

(in hard water) unstable complex (wine red)

As this solution is titrated against EDTA, the Ca^{2+} and Mg^{2} ions from the indicator complex form stable metal ion - EDTA complex leaving the indicator free. The free form of indicator is steel blue in colour.

The metal – EDTA complex is stable at pH 8-10. This pH range can be maintained by using ammoniacal buffer ($NH_4Cl + NH_4OH$).

$$Mg/Ca\ - EBT \ + \ EDTA \longrightarrow (Mg/Ca - EDTA) \quad + \quad EBT$$

(wine red complex) colourless stable complex steel blue

The amount of EDTA used corresponds to the hardness of water.

PROCEDURE:

Titration 1: Estimation of total hardness of sample water

20 ml of the given hard water sample is pipetted out into a clean conical flask. 5ml of ammoniacal buffer solution and 2 drops of EBT indicator are added. The solution turns wine red in colour. The solution is then titrated against EDTA solution taken in the burette. The change of colour from wine red to steel blue is the end point. The titration is repeated for concordant values. Let the titre value be V ml. This gives the total hardness of the sample water.

Titration 2: Estimation of permanent hardness

100 ml of the given sample water is taken in a clean 250 ml beaker and boiled for 10-15 minutes until it is reduced to $1/5^{th}$ of the original volume. It is then cooled and filtered. The filtrate is collected in a 100 ml standard flask and made upto the mark using distilled water. 20 ml of this made up solution is pipetted out into a clean conical flask and the titration is repeated in the same way as in titration 1. Let the titre value be V ml. This gives the permanent hardness of the sample water.

Estimation of temporary hardness

Temporary hardness of the water sample is calculated by subtracting permanent hardness from total hardness.

Temporary hardness = Total hardness – Permanent hardness.

RESULT

1. The total hardness of the given water sample = ……..ppm
2. The permanent hardness of the given water sample= ……..…..ppm
3. The temporary hardness of the given water sample =…….ppm

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Table. 1 Titre values and different alkalinities

S.No.	Value of [P] & [M]	Alkalinity due to			Nature of alkalinity
		OH^-	CO_3^{2-}	HCO_3^-	
1	P = 0	0	0	M	Only HCO_3^- ions present
2	P = ½M	0	2P	0	Only CO_3^{2-} ions present
3	P = M	P = M	0	0	Only OH^- ions present
4	P< ½ M	0	2P	M-2P	HCO_3^- , CO_3^{2-} ions present
5	P> ½ M	2P-M	2M – 2P	0	Only OH^-, CO_3^{2-} ions present

Table. 2 Water sample 1 vs Standard acid

Sl.No	Volume of water sample (ml)	Burette reading (ml)			Volume of the acid (ml)	
		Initial	[P]	[M]	[P]	[M]
1.	20	0				
2.	20	0				

Calculation

If the data satisfies the condition P> 1/2M

(i) Volume of HCl required for OH^- Alkalinity = 2P- M = ……ml

(ii) Volume of HCl required for CO_3^{2-} Alkalinity = 2M – 2P = …..……ml

(iii) HCO_3^- absent

Calculation of OH^- alkalinity

Volume of standardized HCl (V1) = ……ml

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Strength of standardized HCl (N1) = 0.1 N

Volume of unknown water sample (V2) = 20 ml

Strength of unknown water sample (N2) = ?

According to volumetric formula, $V_1N_1 = V_2N_2$

N2 = …… V1ml x0.1N /20

=…….N

3. DETERMINATION OF TYPES AND AMOUNT OF ALKALINITY IN WATER SAMPLES

Expt. No.	

Date			

AIM

To determine the type and amount of alkalinity present in the given water sample. You are provided with standard solution of hydrochloric acid of strength of 0.1N.

CHEMICALS REQUIRED

0.1N HCL, phenolphthalein and methyl orange.

PRINCIPLE

The alkalinity of water is normally due to the presence of bicarbonates, carbonates and hydroxides of sodium, potassium, calcium and magnesium. The knowledge of alkalinity is important in calculating the amounts of reagents required in water softening and in internal conditioning of boiler feed waters.

The alkalinity in water can be present due to the presence of the above mentioned ions present alone or in combination as

- Hydroxide only (OH^-)
- Carbonates only (CO_3^{2-})
- Bicarbonates only (HCO_3^-)
- Hydroxide and carbonates ($OH^- + CO_3^{2-}$)
- Carbonate and bicarbonates ($CO_3^{2-} + HCO_3^{2-}$)

The possibility of all the three ions existing together is ruled out because the hydroxides and bicarbonate ions combine together to form CO_3^{2-} ions

$$OH^- + HCO_3^- \longrightarrow CO_3^{2-} + H_2O$$

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The alkalinity of the water sample is determined by titrating against the standard acid using phenolphthalein and methyl orange indicators. The following reactions take place.

$$OH^- + H^+ \longrightarrow H_2O \quad \text{……...(1)}$$

$$CO_3^{2-} + H^+ \longrightarrow HCO_3^- \quad \text{……...(2)}$$

$$HCO_3^- + H^+ \longrightarrow H_2CO_3 \longrightarrow H_2O + CO_2 \quad \text{……..…...(3)}$$

Amount of OH^- present in 1 litre of water sample

in terms of $CaCO_3$ equivalent = strength of water sample x Eq. wt. of $CaCO_3$

i.e OH^- alkalinity in terms of $CaCO_3$ equivalent = …….N x 50 x 10^3 ppm

Alkalinity due to OH^- ion = ……..ppm

Calculation of CO_3^{2-}alkalinity

Volume of standardized HCl (V1) = ……..ml

Strength of standardized HCl (N1) = 0.1 N

Volume of unknown water sample (V2) = 20 ml

Strength of unknown water sample (N2) = ?

According to volumetric formula, $V_1N_1 = V_2N_2$

N_2 = …… V1ml x0.1N /20

=…….N

Amount of CO_3^{2-} present in 1 litre of water sample

in terms of $CaCO_3$ equivalent = strength of water sample x Eq. wt. of $CaCO_3$

i.e CO_3^{2-} alkalinity in terms of $CaCO_3$ equivalent = …….N x 50 x 10^3 ppm

Alkalinity due to CO_3^{2-} ion = ……..ppm

Table. 3 Water sample 2 vs Standard acid

Sl.No	Volume of water sample (ml)	Burette reading (ml)			Volume of the acid (ml)	
		Initial	[P]	[M]	[P]	[M]
1.	20	0				
2.	20	0				

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Calculation

If the data satisfies the condition P< 1/2M

(i) Volume of HCl required for CO_3^{2-} Alkalinity = 2P =…….ml

(ii) Volume of HCl required for HCO_3^- Alkalinity = M – 2P = …….ml

(iii) OH^- ion absent

The volume of acid used upto phenolphthalein end point corresponds to the reaction (1) and (2), i.e, complete neutralization of OH^- ions and neutralization of CO_3^{2-} ion up to HCO_3^- stage.

The total volume of the acid used upto methyl orange end point corresponds to all the reactions (1), (2), and (3), i.e a complete neutralization of OH^- , CO_3^{2-} and HCO_3^- ions.

Thus from the respective volumes of acid used the strength of various ions can be determined.

PROCEDURE

For water sample 1

Titration I: Phenolphthalein indicator

The burette is washed, rinsed and filled with the given std. hydrochloric acid. 20 ml of the water sample (1) is pipetted into a clean conical flask. 2-3 drops of phenolphthalein indicator is added. Pink color is observed. This is titrated against the standard acid until the pink color disappears. This titre value is noted which corresponds to the phenolphthalein end point (P).

Titration II: Methyl orange indicator

Few drops of methyl orange indicator is added to the same solution after the phenolphthalein end point. The solution turns yellow in color. The titration is continued until the yellow color changes to reddish orange color. The titre value corresponds to methyl orange end point (M). The titration is repeated for concordant values. Using the titre values the type and amount of alkalinity is calculated.

For water sample 2

The given water sample (2) is make up in a 100 ml standard flask with distilled water. 20ml of the made up solution is pipetted out in to a clean conical flask. The titration is repeated as above by adding phenolphthalein indicator followed by methyl orange indicator. From the titre value the alkalinity is found out.

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Calculation of CO_3^{2-} alkalinity

Volume of standardized HCl (V1) = ……ml

Strength of standardized HCl (N1) = 0.1 N

Volume of unknown water sample (V2) = 20 ml

Strength of unknown water sample (N2) = ?

According to volumetric formula, $V_1N_1 = V_2N_2$

N_2 =……V1ml x0.1N /20

=…….N

Amount of CO_3^{2-} present in 1 litre of water sample

in terms of $CaCO_3$ equivalent = strength of water sample x Eq. wt. of $CaCO_3$

i.e CO_3^{2-} alkalinity in terms of $CaCO_3$ equivalent = …….N x 50 x 10^3 ppm

Alkalinity due to CO_3^{2-} ion = ……..ppm

Calculation of HCO_3^- alkalinity

Volume of standardized HCl (V1) = ……ml

Strength of standardized HCl (N1) = 0.1 N

Volume of unknown water sample (V2) = 20 ml

Strength of unknown water sample (N2) = ?

According to volumetric formula, $V_1N_1 = V_2N_2$

N_2 =…….V1ml x0.1N /20

=…….N

Amount of HCO_3^- present in 1 litre of water sample

in terms of $CaCO_3$ equivalent = strength of water sample x Eq. wt. of $CaCO_3$

i.e HCO_3^- alkalinity in terms of $CaCO_3$ equivalent = …….N x 50 x 10^3 ppm

Alkalinity due to HCO_3^- ion = ……..ppm

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RESULT

The given water sample (1) contains

1. Hydroxide alkalinity = ……..ppm
2. Carbonate alkalinity = ……...ppm
3. Bicarbonate alkalinity = ……ppm

The given water sample (2) contains

1. Carbonate alkalinity = ……...ppm
2. Bicarbonate alkalinity = ……ppm
3. Hydroxide alkalinity = ……..ppm

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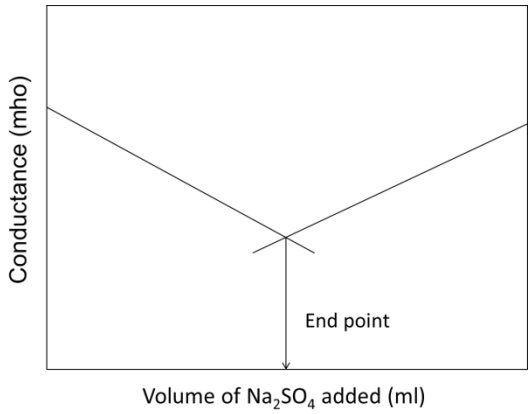
Titration of mixture of $BaCl_2$ Vs Na_2SO_4

S.No	Volume of Na_2SO_4 added (ml)	Conductance (mho)
1. 2. 3. . . . 20.		

Model Graph



Calculation of strength of $BaCl_2$

Volume of unknown $BaCl_2$ (V_1) = 10 ml

Strength of unknown $BaCl_2$ (N_1) =……...…N

Volume of standard Na_2SO_4(V_2) = …….ml (titre value)

Strength of standard Na_2SO_4(N_2) = 0.1 N

Strength of unknown $BaCl_2$ (N_1) = V_2N_2 / V_1

= V_2 x 0.1 / 10

Strength of unknown $BaCl_2$ (N_1) =……...…N

The amount of barium chloride present in 1 litre of the given solution = strength of $BaCl_2$ x Equivalent weight of $BaCl_2$

= ……..N x 122.14

= ……..g/L

4. CONDUCTOMETRIC TITRATION OF BARIUM CHLORIDE AGAINST SODIUM SULFATE (PRECIPITATION TITRATION)

Expt. No.	

Date			

AIM

To estimate the amount of $BaCl_2$ present in the given solution. You are provided with standard solution of Na_2SO_4 of strength 0.1 N.

MATERIALS REQUIRED

Conductivity meter, conductivity cell, micro burette, pipette, standard 0.1 N Na_2SO_4, $BaCl_2$ solution.

PRINCIPLE

Conductivity of the solution depends upon number of ions in solution. During the addition of Na_2SO_4 to $BaCl_2$ taken in the beaker. Ba^{2+} ions are removed as $BaSO_4$ precipitate (which produce negligible amount of ions).

$$BaCl_2 + Na_2SO_4 \longrightarrow BaSO_4 + Cl^- + Na^+$$

Hence the conductance decreases sharply. When all the Ba^{2+} ions are removed as $BaSO_4$, further addition of Na_2SO_4 produces $2Na^+$ and SO_4^{2-} ions. Hence conductivity starts increasing sharply. The intersection point corresponds to the end point of the titration.

PROCEDURE

Sodium sulphate is taken in the burette. 10ml of the given $BaCl_2$ solution is pipetted out into a clean beaker. Conductivity cell is placed in it and diluted with conductivity water.

1 ml of Na_2SO_4 solution is added to the solution, stirred and then conductance is measured. This is continued up to the end point. The conductance decreases up to the end point. After the end point, conductance increases due to the addition of excess of Na_2SO_4.

A graph is plotted between the volume of Na_2SO_4 and conductance. From the graph end point is noted and the amount of $BaCl_2$ present in 1 litre of the solution is calculated.

RESULT

The amount of $BaCl_2$ present in 1 litre of the given solution = …….g/L

Titration 1: Determination of acidity of water sample 1

Sample water Vs Standard Na_2CO_3

Sl.No	Volume of water sample 1 (ml)	Burette reading (ml)		Volume of Standard Na_2CO_3 (ml)	Concordant value (ml)	Indicator
		Initial	Final			
1.	20	0				Methyl orange
2.	20	0				

Calculation of acidity of water sample

Volume of water sample $(V_1) = 20$ ml

Strength of water sample $(N_1) = ?$

Volume of standard Na_2CO_3 $(V_2) =$_____ml

Strength of standard Na_2CO_3 $(N_2) = 0.01$ N

According to volumetric formula, $V_1N_1 = V_2N_2$

$N_1 = V_2 \times N_2/20$

Strength of water sample 1 $(N_1) =$_____N

Amount of acidity present in 1 litre of water sample in terms of $CaCO_3$ equivalent = strength of water sample x Eq. wt. of $CaCO_3$ x 1000

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= …….N x 50 x 10^3 ppm

The acidity of given water sample 1 = ……..ppm

5. PREPARATION OF Na_2CO_3 AS A PRIMARY STANDARD AND ESTIMATION OF ACIDITY OF A WATER SAMPLE USING THE PRIMARY STANDARD

Expt. No.	

Date			

AIM

To determine the acidity of water sample.

MATERIALS REQUIRED

Apparatus:

100 mL beaker, 100 mL standard flask, 250 mL conical flask, 20 mL pipette, burette, retort stand

Chemicals:

Na_2CO_3, concentrated HCl, Methyl orange indicator

PRINCIPLE

Acidity of a water is its quantitative capacity to react with a strong base to a designated pH. The measured value may vary significantly with the end-point pH used in the determination. Acidity is a measure of aggregate property of water and can be interpreted in terms of specific substances only when the chemical composition of the sample is known. Strong minerals acids, weak acids such as carbonic and acetic, and hydrolyzing salts such as iron or aluminium sulfate may contribute to the measured acidity according to the method of determination.

Pure sodium carbonate is a non-hygroscopic primary standard reagent. A standard solution prepared by dissolving a known amount of the solid Na_2CO_3 in a fixed volume can be used to standardize other acid solutions. The aim of the experiment is to prepare a standard solution of exact concentration which can later be used to standardize other solutions.

HCl is not a primary standard. Thus, after a dilute HCl solution is prepared, it has to be standardized with a primary standard solution in order to determine its concentration accurately. In this experiment a standard Na_2CO_3 solution and a dilute HCl solution will be prepared. The HCl solution will later be standardized against the Na_2CO_3 solution. The reaction between HCl and Na_2CO_3 is as follows.

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$$Na_2CO_3\,(aq) + 2HCl\,(aq) \rightarrow 2NaCl\,(aq) + CO_2\,(g) + H_2O(l)$$

$$CO_3^{2-}(aq) + 2H^+\,(aq) \rightarrow CO_2\,(g) + H_2O\,(l)$$

These are the hydrochloric acid and sodium carbonate equations. In acid-base titrations, the amount of the acid becomes chemically equivalent to the amount of base present. The solution becomes neutral in the event of a strong acid and a strong base titration of the solution.

Titration 2: Determination of acidity of water sample 2

Sample water Vs Standard Na_2CO_3

Sl.No	Volume of water sample 2 (ml)	Burette reading (ml)		Volume of Standard Na_2CO_3 (ml)	Concordant value (ml)	Indicator
		Initial	Final			
1.	20	0				Methyl orange
2.	20	0				

Calculation of acidity of water sample

Volume of unknown water sample $(V_1) = 20$ ml

Strength of unknown water sample $(N_1) = ?$

Volume of standard Na_2CO_3 $(V_2) =$…….ml

Strength of standard Na_2CO_3 $(N_2) = 0.01$ N

According to volumetric formula, $V_1N_1 = V_2N_2$

$$N_1 = V_2 \times N_2/20$$

Strength of water sample 2 $(N_1) =$.....N

Amount of acidity present in 1 litre of water sample in terms of $CaCO_3$ equivalent = strength of water sample x Eq. wt. of $CaCO_3$ x 1000

= …….N x 50 x 10^3 ppm

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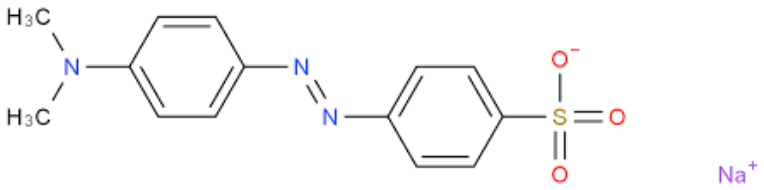
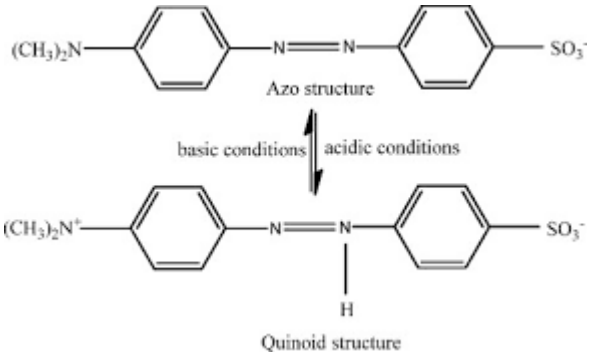
The acidity of given water sample 2 = ……..ppm

The chemical structure of methyl orange is represented as below:



The indicator **methyl orange** (MeO), forms a red colored complex with acidic water sample a pH of 3-5.



As this solution is titrated against Na_2CO_3, the indicator forms a stable complex leaving the indicator free. The free form of indicator is red in colour.

PROCEDURE

Preparation of standard Na_2CO_3 solution

1. Weigh accurately about 1.33 g of Na_2CO_3. Record the exact mass of the Na_2CO_3.
2. Dissolve the Na_2CO_3 in about 50 mL water in a 100 mL beaker.
3. Dissolve the Na_2CO_3 in about 50 mL water in a 100 mL beaker.
4. Transfer the solution into a 250 mL volumetric flask. Rinse the beaker with distilledwater.
5. Add distilled water to the mark. Stopper the flask. Shake it by turning it upside down afew times to make the solution homogeneous.

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Titration: Determination of acidity of water sample 1

The burette is washed well with distilled water and rinsed with small amount of Na_2CO_3 solution. The pipette is washed with distilled water and rinsed with small amount of water sample. 20ml of water sample is pipetted out into a clean conical flask. 1 ml or 3 drops of methyl orange indicator is added and titrated against standard Na_2CO_3 solution taken in the burette. The end point is the change of colour from red orange to yellow. The titration is repeated for concordant values. From the titre value, the acidity is found out.

Titration: Determination of acidity of water sample 2

The given water sample is made up into 100 ml in a standard flask using distilled water. 20ml of the made up water sample is pipetted out in to a clean conical flask. This is titrated against the standard Na_2CO_3 until the indicator colour changes from red orange to yellow. The titration is repeated for concordant values. From the titre value, the acidity is found out.

RESULT

1. The acidity of given water sample 1 = ……ppm
2. The acidity of given water sample 2 = ……ppm

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CALCULATION:

Subtract the initial weight (in grams) of the empty container from the weight of the container with the dried residue to obtain the increase in weight.

A- Weight of clean dried container (g)

B- Weight of container and residue (g)

C- Volume of Sample (ml)

Sl.No	**Volume of water sample (ml)**	**Weight of clean dried container (g)**	**Weight of container and residue (g)**	**Weight of residue solids (mg/g)**
1.				
2.				

Dissolved solids, TDS (mg/l) = mg of solids in the beaker / (volume of sample)x 1000

= ((B - A)/ C)* (1000 mg/g) / (1000) ml/L

= _____ mg/L

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6. ESTIMATION OF TDS OF A WATER SAMPLE BY GRAVIMETRY

Expt. No.	

Date			

AIM

To determine the Total dissolved solids (TDS) of given water samples.

MATERIALS REQUIRED

Water sample, electronic balance machine, beaker, measuring cylinder, Whatman filter paper/Gooch crucible or sintered crucible, funnel, dropper, evaporating dish/ceramic dish, desiccator

PRINCIPLE

Introduction:

Water is a good solvent and picks up impurities easily. Pure water - tasteless, colorless, and odorless is often called the universal solvent. Dissolved solids" refer to any minerals, salts, metals, cations or anions dissolved in water. Total dissolved solids (TDS) comprise inorganic salts (principally calcium, magnesium, potassium, sodium, bicarbonates, chlorides, and sulfates) and some small amounts of organic matter that are dissolved in water.

In general, the total dissolved solids concentration is the sum of the cations (positively charged) and anions (negatively charged) ions in the water. Therefore, the total dissolved solids test provides a qualitative measure of the amount of dissolved ions but does not tell us the nature or ion relationships

An elevated total dissolved solids (TDS) concentration is not a health hazard. The TDS concentration is a secondary drinking water standard and, therefore, is regulated because it is more of an aesthetic rather than a health hazard. An elevated TDS indicates the following:

1) The concentration of the dissolved ions may cause the water to be corrosive, salty or brackish taste, result in scale formation, and interfere and decrease efficiency of hot water heaters; and

2) Many contain elevated levels of ions that are above the Primary or Secondary Drinking Water Standards, such as an elevated level of nitrate, arsenic, aluminum, copper, lead, etc.

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PROCEDURE

1. Filter your water sample through a Whatman Filter paper.
2. Collect the filtrate (liquid) and rinse water in a beaker/flask.
3. Take the weight of empty container (beaker/ ceramic dish/ evaporating dish). Make sure the container should be dried.
4. Add the filtrate to the container and allow the sample to stay in the oven at 105°C for 24 hours. If

possible, increase the temperature of the drying oven to 180°C and allow the sample to dry for up to 8 hours.

5. Remove the container - Remember it is very hot. After removing from the drying oven, the sample container should be placed in a desiccator to cool in a dry air environment for at least 3 to 4 hours.
6. After the container cools, reweigh the container at least three times. Then do the following:

RESULT

The dissolved solids, TDS of given water sample = …… (mg/L) or ppm

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7. PREPARATION OF NANOPARTICLES (AgNPs)

Expt. No.	

Date			

AIM

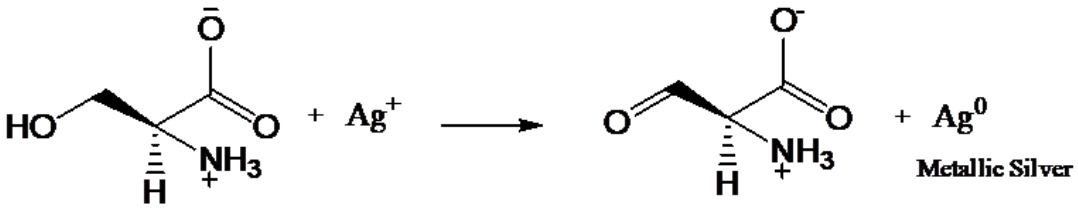
To synthesize the silver nanoparticles (AgNPs) by chemical reduction method..

MATERIALS REQUIRED

Silver nitrate (AgNO3), serine, polyvinyl pyrrolidone (PVP), and NaOH.

PRINCIPLE

The Ag NPs were synthesized by the reduction of Ag+ ions in the presence of microwave radiation using serine (amino acid) as a reducing agent and PVP as a capping agent. There is an oxidation of -OH group of serine into aldehyde group by reducing Ag+ and finally leading to the formation of Ag0 nuclei, which can be stabilized by PVP.



PROCEDURE

Synthesis of silver nanoparticles

A microwave oven was used in the typical synthesis of Ag NPs. 100 mL of 5 mM silver nitrate solution was prepared in 250 mL conical flask using the de-ionized water. 0.0525 g of serine and 0.75 g of PVP were added into the above silver nitrate solution, and then 4 drops of 1 M NaOH were added by altering the pH for the favorable reduction. The above mixed solution was kept in the microwave oven (input power 1200 W, 50 Hz) for 90 s. The solution turned into yellowish brown colour, which confirmed the formation of Ag NPs.

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RESULT

The AgNPs were successfully synthesized by chemical reduction method with help of microwave oven.