

KSK COLLEGE OF ENGINEERING AND TECHNOLOGY

KUMBAKONAM – 612 702

Department of Chemistry

Laboratory Manual (2021 – 22)



BS3171 – Chemistry Lab

(Regulations - 2021)

COMMON TO ALL BRANCHES



INDEX

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General Instructions to the Students

- ❖ Attend all the practical classes and closely follow the instructions of your Master.
- ❖ Students should enter the lab with uniform and necessary things compulsorily (i.e., Calculator, match box, waste cloth, cleaning powder etc.)
- ❖ Come prepared for all the practical classes.
- ❖ Before performing an experiment, go through the procedure thoroughly, understand the theory behind the reactions and then proceed. Incomplete knowledge may land you in danger.
- ❖ Systematic approach is a must in the laboratory. Record your observations as and when you proceed.
- ❖ Do not contaminate the reagents in any way. Turn off gas tap and water tap whenever they are not in use.
- ❖ In the case of any accident like swallowing of chemicals, solutions or splashing of acids and other chemicals in the eyes or on the body, report to the concerned staff immediately.
- ❖ Never handle the chemicals with your finger, always use a spatula or dropper. Use optimum amount of chemicals and reagents, it helps to get better results.
- ❖ Keep the work bench and sink neat and clean. Put waste things such as filter paper, broken bits of glasses, sticks of match, etc in the dustbin. Keep the apparatus clean and arrange them properly on the work bench.
- ❖ Replace the reagent bottles in their respective places at once after use and see that they are closed. Never remove more than one reagent bottle at a time.
- ❖ Report honestly the breakage of apparatus to the concerned authority.
- ❖ Don't hesitate to clarify your doubts.

Toxic and Hazardous chemicals

| Toxic chemicals | Effects |
|---|--|
| Acids, Alkalies, Bromine, Phenol | Severe burn |
| Acetic Anhydride, Acetyl chloride | Irritant to eyes |
| Alcohols, Benzene, Toluene, CS ₂ and Ethers | Highly inflammable (catches fire easily) |
| Diazo compounds, Peroxides, Azides | Explosives |
| Silver nitrate, Mercuric chloride and CuSO ₄ | Poisonous (If taken internally) |
| Sodium metal | Explosive in water. Can be destroyed in methanol |

First Aid procedures in Chemical Laboratory**Eye Accidents**

Flood your eyes immediately with water

If glass has entered the Eyes:

- ❖ Remove the loose glass pieces gently, washing the eyes with lots of water without rubbing.
- ❖ If glass has penetrated inside the eyes, do not rub, keep the eye open as far as possible and consult a doctor immediately.

Acid in the Eye:

- ❖ Wash repeatedly with 1% solution of Sodium bicarbonate and finally with Fresh water.

Alkalies in the Eye:

- ❖ Wash with 1% boric acid solution and finally with fresh water.

Bromine in the Eye:

- ❖ Wash thoroughly with water then immediately with 1% Sodium bicarbonate solution.

Cuts

- ❖ For minor cuts, allow the bleeding to occur for few seconds, remove pieces if any, wash with water, apply Dettol and Bandage. For severe cuts, cotton soaked in rectified spirit or tincture Benzoin may be used.

Burns

| Accident | Remedy |
|--|--|
| Burns caused by dry heat (by touching the flame of hottest tube etc.,) | Apply Burnol or Furacin ointment. |
| Acids on the skin (phenols also) | Wash immediately with liberal quantity of water and saturated sodium bicarbonate solution and finally apply disinfectant and Burnol. |
| Alkalies on the skin | Wash immediately with 1% solution of Acetic acid or lemon juice and with water. For serious burns apply disinfectant and Burnol. |
| Bromine on the skin | Cover the affected part liberally with glycerol and rub it gently. Wipe the area dry and apply Burnol. (or) Wash the affected part immediately with sufficient quantity of petrol and then apply Burnol. (or) If Glycerol and petrol are not available wash with water then with sodium bicarbonate solution and apply Burnol. |
| Sodium on the skin | Remove the pieces of sodium metal if any, |

| | |
|-------------------------------|---|
| | wash with water and then with 1% solution of acetic acid finally apply Olive oil or Burnol. |
| Organic substance on the skin | Wash freely with rectified spirit then with soap and warm water. |

Poisons (Reagents in mouth)

| If swallowed | Remedy |
|--------------------------------|---|
| Acids (including Oxalic acids) | Dilute the acid strength by drinking much water followed by lime water or sodium bicarbonate solution. Milk may be given but no emetics. |
| Caustic alkalies | Dilute by drinking much water followed by vinegar, lemon or orange juice or solution of lactic acid or citric acid. Milk may be given but no emetics. |
| Arsenic or Mercury compounds | Give an emetic immediately (like table salt solution). |

Note: For all the above cases, it is safe to consult a Doctor.

CONTENT

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Short Procedure

| Content | Titration – I Std. NaCl Vs. AgNO ₃ | Titration – II Std. AgNO ₃ Vs. Water Sample |
|------------------|--|--|
| Burette solution | AgNO ₃ Solution | Std. AgNO ₃ Solution |
| Pipette solution | Std. NaCl Solution | Water Sample |
| Indicator | 5 % Potassium Chromate | 5 % Potassium Chromate |
| End point | Colour change from yellow to reddish brown | Colour change from yellow to reddish brown |

Equivalent Weight of Chloride ion = 35.46 g

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

EXPT NO: 1

DATE:

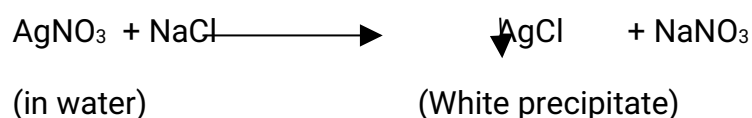
AIM

To determine the amount of chloride present in the given water sample, being supplied with a standard solution of sodium chloride of strength 0.01N and an approximately N/10 solution of silver nitrate.

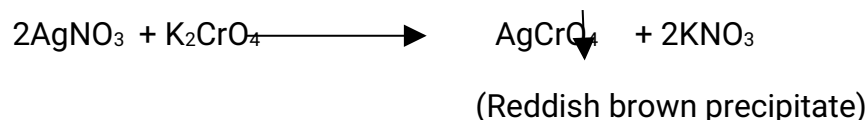
PRINCIPLE

Generally water contains chloride ions (Cl^-) in the form of NaCl , KCl , CaCl_2 and MgCl_2 . Water containing more than 250 ppm of chloride ions in water is not desirable for drinking purpose. The total chloride ions can be determined by argentometric method (Mohr's method).

In this method, Cl^- ion solution is directly titrated against AgNO_3 using potassium chromate (K_2CrO_4) as an indicator.



At the end point, when all the Cl^- ions are removed. The yellow colour of chromate changes into reddish brown due to the following reaction.



MATERIALS REQUIRED

1. Std. NaCl solution
2. AgNO₃ Solution
3. 2% K₂CrO₄
4. Burette, pipette, Conical flask etc.,

TITRATION-I**STANDARDISATION OF SILVER NITRATE SOLUTION***Std. NaCl Vs. AgNO₃*

| S. No | Volume of NaCl (V ₁) (ml) | Burette Readings(ml) | | Concordant Volume of AgNO ₃ (V ₂) (ml) | Indicator |
|-------|---------------------------------------|----------------------|-------|---|---------------------------------|
| | | Initial | Final | | |
| 1. | 20 | 0 | | | K ₂ CrO ₄ |
| 2. | 20 | 0 | | | |

Calculation of the Normality of Silver Nitrate solution:Volume of Sodium Chloride solution, V₁ = 20mlStrength of Sodium Chloride solution, N₁ = 0.01NVolume of Silver Nitrate solution, V₂ = mlStrength of Silver Nitrate solution, N₂ = ?

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 \times N_1 / V_2$$

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

$$\text{Strength of Silver Nitrate solution} = \dots\dots\dots \text{N}$$

PROCEDURE**TITRATION –I*****Standardization of silver nitrate solution***

The burette is washed well with distilled water and then rinsed with the small amount of AgNO_3 solution. It is then filled with the same solution upto the zero mark without any air bubbles.

The pipette is washed with distilled water and then rinsed with the small amount of standard NaCl solution. 20ml of this made up solution is pipetted out into a clean conical flask. 1ml of 2% K_2CrO_4 indicator solution is added and then 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 consecutive concordant titre values.

STEP II: DETERMINATION OF CHLORIDE IONS**TITRATION – II****Water sample Vs. Std. AgNO_3**

| S. No | Volume of water sample (V_1) (ml) | Burette Readings(ml) | | Concordant Volume of AgNO_3 (V_2) (ml) | Indicator |
|-------|---------------------------------------|----------------------|-------|---|--------------------------|
| | | Initial | Final | | |
| 1. | 20 | 0 | | | K_2CrO_4 |
| 2. | 20 | 0 | | | |

Calculation of Normality of the water sample (Chloride ions)

Volume of the water sample, V_1 = 20ml

Strength of the water sample (Chloride ions), N_1 =?

Volume of Silver nitrate solution, V_2 =ml

Strength of silver nitrate solution, N_2 = N

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

$$N_1 = V_2 \times N_2 / V_1$$

$$= \text{.....ml} \times \text{..... N} / 20\text{ml}$$

Strength of the water sample (Chloride ions) =N

Calculation of amount of the chloride

Amount of the chloride ions
Strength present in 1 litre (1000ml) of the water sample
1000

} = Equivalent weight of chloride ions X
Strength of the chloride ions X

$$= 35.46 \times \dots\dots\dots N \times 1000$$

$$= \dots\dots\dots \text{ppm}$$

Step II

TITRATION II

Determination of chloride ions

20 ml of the given water sample is pipetted out into a clean conical flask and 1 ml of 2% K_2CrO_4 indicator solution is added. It is then titrated against standardized 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 consecutive concordant titre values.

RESULT

Amount of chloride ions present in the 1000 ml of the given water sample = ppm

Short Procedure

| Content | Titration – I Std. Hard water Vs. EDTA | Titration – II Std. EDTA Vs. Water sample | Titration – III Std. EDTA Vs. Boiled Water sample |
|------------------------|--|---|--|
| Burette solution | EDTA | Std. EDTA | Std. EDTA |
| Pipette solution | Std. Hard water | Water sample | Boiled Water sample |
| Indicator | Eriochrome black - T | Eriochrome black - T | Eriochrome black - T |
| End point | Colour change from wine red to steel blue | Colour change from wine red to steel blue | Colour change from wine red to steel blue |
| Additional solution | 5 ml of ammonia buffer | 5 ml of ammonia buffer | 5 ml of ammonia buffer |
| Temperature | Room temperature | Room temperature | Room temperature |

Equivalent Weight of $\text{CaCO}_3 = 50\text{g}$

2. DETERMINATION OF TOTAL, PERMANENT AND TEMPORARY HARDNESS OF WATER SAMPLE (EDTA METHOD)

EXPT NO: 2

DATE:

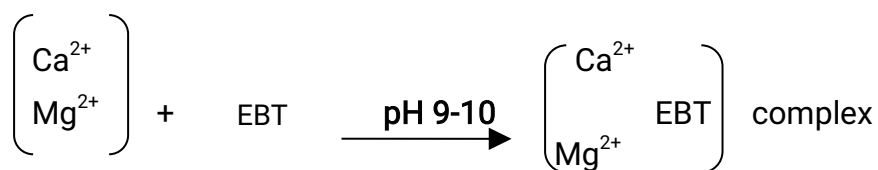
AIM

To determine the amount of total, permanent and temporary hardness of the given hard water sample. You are provided with a standard hard water of known hardness and a link solution of Ethylene Diamine Tetra Acetic acid (EDTA).

PRINCIPLE

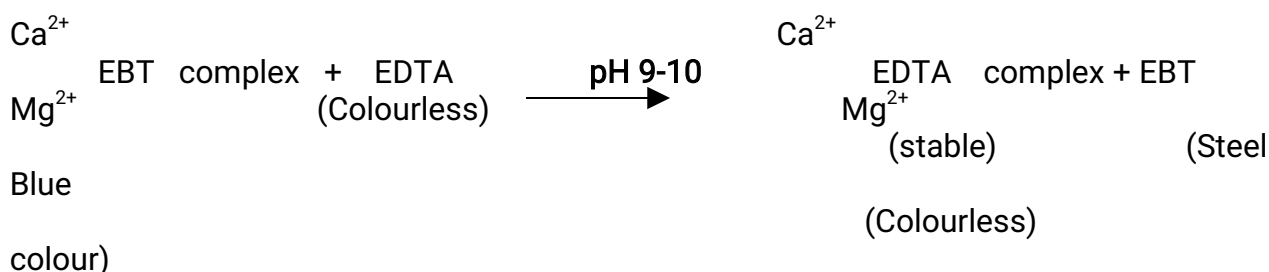
Disodium salt of Ethylene Diamine Tetra Acetic acid (EDTA) is a well-known complexing agent. Its structure is shown in the figure I.

Disodium salt of EDTA is used to determine the various hardness of the given hard water containing Ca^{2+} and Mg^{2+} ions. When EDTA is added to hard water, it reacts with calcium and magnesium ions present in hard water to form stable EDTA metal complexes. From the volume of EDTA consumed the hardness can be calculated. Eriochrome Black –T is used as an indicator. The indicator forms a weak complex with the metal ions present in the hard water and gives wine red colour.



When EDTA is added with the hard water, the metal ions form a stable metal

complex with EDTA by leaving the indicator. When all the metal ions are taken by EDTA from the indicator metal ion complex, the wine red colour changes into steel blue, which denotes the end point. The metal EDTA complex is stable at pH 8-10. This pH range can be maintained by adding ammonia buffer ($\text{NH}_4\text{Cl} + \text{NH}_4\text{OH}$).



Structure of disodium salt of EDTA

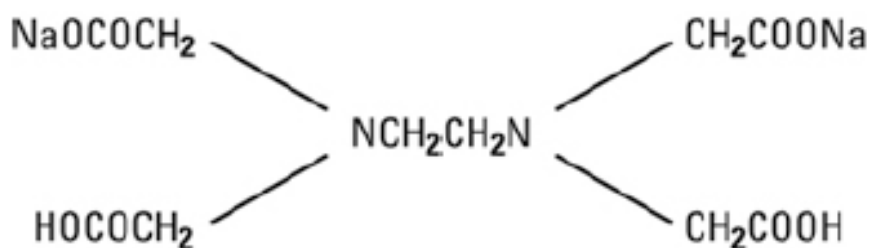


Fig. 1

STANDARDISATION OF EDTA

TITRATION-I

Standard Hard water Vs. EDTA

| S. No | Volume of Standard Hard water (ml) | Burette readings (ml) | | Concordant volume of EDTA(V_1) (ml) | Indicator |
|-------|--|--------------------------|-------|---|-----------|
| | | Initial | Final | | |
| 1. | 20 | 0 | | | EBT |
| 2. | 20 | 0 | | | |

Calculation

Volume of Standard hard water = 20 ml

Volume of EDTA solution consumed, V_1 =ml

1ml of standard hard water contains 1mg of CaCO_3

20ml of standard hard water contains 20mg of CaCO_3

20ml of standard hard water consumes V_1 ml of EDTA

ie., V_1 ml of EDTA solution = 20mg of CaCO_3

\therefore 1ml of EDTA solution = $20/V_1$ mg of CaCO_3 equivalent

Temporary hardness can be removed by boiling and filtering the given hard water sample. During boiling, soluble bicarbonates are converted into insoluble carbonates.



The filtrate is collected in the conical flask. EBT indicator and buffer solution are added. It is then titrated against the EDTA.

Temporary hardness = Total hardness – Permanent hardness

MATERIALS REQUIRED

1. EDTA solution
2. Standard hard water
3. EBT indicator
4. Buffer solution
5. Burette, pipette, conical flask, 250 ml beaker, 100ml std. flask
6. Sample hard water

PROCEDURE

TITRATION –I

Standardization of EDTA

The burette is washed well the distilled water and rinsed with a little amount of the given EDTA solution. It is then filled with the same EDTA solution upto the zero level without any air bubbles. 20ml of standard hard water solution is pipetted out into a conical flask. 5ml of ammonia buffer solution and 2 drops of Eriochrome Black –T indicator are added. The Solution turns wine red in colour and it 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 final reading in the burette is noted. The titration is repeated to get concordant values.

Let the volume of EDTA be V_1 ml.

TITRATION-II

DETERMINATION OF TOTAL HARDNESS OF HARD WATER SAMPLE

Hard water Sample Vs. Std. EDTA

| S. No | Volume of Sample Hard water (ml) | Burette readings (ml) | | Concordant volume of EDTA(V_2) (ml) | Indicator |
|-------|---|--------------------------|-------|---|-----------|
| | | Initial | Final | | |
| 1. | 20 | 0 | | | EBT |
| 2. | 20 | 0 | | | |

Calculation of the total hardness of hard water

Volume of hard water = 20 ml

Volume of EDTA consumed, V_2 =ml (Titre value)

20 mL of the given hard water sample consumes V_2 ml of EDTA

i.e., 20 ml of the given hard water sample contains $\frac{20 \times V_2}{V_1}$ mg of CaCO_3

\therefore 1000 ml of the given hard water sample contains = $20 \times V_2 \times 1000$ mg of CaCO_3

$$\begin{aligned}
 &= \frac{20 \times V_2}{V_1} \times \frac{1000}{20} \text{ mg of CaCO}_3 \\
 &= \frac{1000 \times V_2}{V_1} \text{ mg of CaCO}_3 \\
 &= 1000 \times \text{—————} \text{ mg of CaCO}_3 \\
 &= \text{.....ppm}
 \end{aligned}$$

∴ Total hardness of the given sample of hard water =ppm

TITRATION-II

Determination of Total Hardness of Hard water Sample

20ml of the given hard water sample is pipetted out into a clean conical flask. 5ml of ammonia buffer solution and 2 drops of Eriochrome Black –T indicator are added. The solution turns wine red in colour. This solution is titrated against standardized EDTA solution taken in the burette. The change of colour from wine red into steel blue c is the end point. The titration is repeated to get concordant values.

Let the volume of EDTA be V_2 ml

Determination of permanent Hardness

100 ml of the given sample of hard water is taken in a clean 250 ml beaker and boiled for 10 – 15 minutes. It is then cooled and filtered. The filtrate is collected in a 100 ml standard flask. 5 ml of ammonia buffer solution and 2 drops of Eriochrome Black – T indicator are added. The solution turns wine red in colour . This solution is titrated against the EDTA taken in the burette. The change of colour from wine red into steel blue c is the end point. The titration is repeated to get concordant values.

Let the volume of EDTA be V_3 ml

Determination of Temporary Hardness

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

Temporary hardness = Total hardness – Permanent hardness

TITRATION-III**DETERMINATION OF PERMANENT HARDNESS OF HARD WATER SAMPLE*****Boiled hard water Sample Vs. Std. EDTA***

| S. No | Volume of <i>Boiled hard water</i> (ml) | Burette readings (ml) | | Concordant volume of EDTA(V_3) (ml) | Indicator |
|-------|---|--------------------------|-------|---|-----------|
| | | Initial | Final | | |

| | | | | | |
|----|----|---|--|--|-----|
| 1. | 20 | 0 | | | EBT |
| 2. | 20 | 0 | | | |

Calculation of the total hardness of hard water

Volume of boiled hard water = 20 ml

Volume of EDTA consumed, V_3 =ml (Titre value)

20 ml of the given boiled hard water sample consumes V_3 ml of EDTA

i.e., 20 ml of the given boiled hard water sample contains $\frac{20 \times V_3}{V_1}$ mg of CaCO_3

$$\begin{aligned} \therefore 1000 \text{ ml of the given hardwater sample contains} &= \frac{20 \times V_3 \times 1000}{V_1 \times 20} \text{ mg of } \text{CaCO}_3 \\ &= \frac{20 \times V_3}{V_1} \times \frac{1000}{20} \text{ mg of } \text{CaCO}_3 \\ &= \frac{1000 \times V_3}{V_1} \text{ mg of } \text{CaCO}_3 \end{aligned}$$

mg of
 CaCO_3 equivalent

=ppm

\therefore Permanent hardness of the given sample of hardwater =ppm

Calculation of the temporary hardness of the hard water

Temporary hardness of the } = Total hardness – Permanent hardness
given sample of water }

= -

= ppm.

RESULT

1. Amount of total hardness of the given sample of water _____ =
ppm

2. Amount of permanent hardness of the given sample of water = _____ ppm



Short Procedure

| Content | Titration – I Std. HCl Vs Water sample | Titration – II Std. HCl Vs Water Sample |
|------------------|---|--|
| Burette solution | Std. HCl | Std. HCl |
| Pipette solution | Water sample | Water sample |
| Indicator | Phenolphthalein | Methyl Orange |
| End point | Disappearance of pink colour | Colour change from yellow to red orange |

Equivalent Weight of $\text{CaCO}_3 = 50\text{g}$

Table - I

Titre values of different alkalinities

| S. No | Result of titration of [P] and [M] | Hardness causing ions | | |
|-------|---------------------------------------|-----------------------|--------------------|------------------|
| | | OH^- | CO_3^{2-} | HCO_3^- |
| 1. | [P] = 0 | 0 | 0 | [M] |
| 2. | [P] = [M] | [P] or [M] | 0 | 0 |
| 3. | [P] = $\frac{1}{2}$ M | 0 | 2 [P] or [M] | 0 |
| 4. | [P] $> \frac{1}{2}$ M | 2[P] – [M] | 2 [M] – 2 [P] | 0 |
| 5. | [P] $< \frac{1}{2}$ M | 0 | 2 [P] | [M – 2 [P]] |

3.DETERMINATION OF ALKALINITY IN WATER SAMPLE

EXPT NO: 3

DATE:

AIM

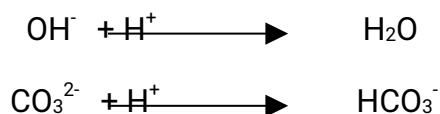
To determine the types and amount of alkalinity present in the given water sample. A standard solution of 0.1 N hydrochloric acid is provided.

PRINCIPLE

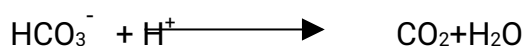
Alkalinity is caused by the presence of hydroxide, carbonate and bicarbonate. There are five types of alkalinity conditions possible in a given sample of water. They are hydroxide only, carbonate only, bicarbonate only, combination of carbonate and hydroxide, carbonate and bicarbonate. The various alkalinities can be determined by titrating with standard acid using phenolphthalein and methyl orange indicators successively.

1.Phenolphthalein end point

When alkaline water is titrated with acid using phenolphthalein indicator, hydroxide alkalinity is completely neutralized and carbonate alkalinity is partially neutralized.

**2. Methyl orange end point**

After the phenolphthalein end point, methyl orange indicator is added to water sample and titrated with acid. Bicarbonate neutralization occurs.



From the two titre values, the alkalinities can be calculated.

STEP I: Std. HCl Vs water sample

| S.No | Volume of water sample (V_1) (ml) | Burette Readings (ml) | | Concordant Volume of Std. HCl (ml) | |
|------|---------------------------------------|-------------------------------|-----------------------------|------------------------------------|-----|
| | | Phenolphthalein end point [P] | Methyl orange end point [M] | [P] | [M] |
| 1. | 20 | | | | |
| 2. | 20 | | | | |
| 3. | 20 | | | | |

Calculation

The data of [P] and [M] satisfy the condition of $[P] < 1/2[M]$

Hence, water sample will contain carbonate & bicarbonate ions.

Carbonate alkalinity

$$\text{Volume of N/10 HCl for CO}_3^{2-} \text{ alkalinity, } V_1 = 2[P]$$

$$= 2 \times \dots\dots\dots \text{mL}$$

$$= \dots\dots\dots \text{mL}$$

$$\text{Normality of N/10 HCl, } N_1 = 1/10$$

$$\text{Volume of water sample, } V_2 = 20 \text{ mL}$$

$$\text{Normality of water sample, } N_2 = ?$$

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 N_1 / V_2 = \dots\dots\dots \text{mL} \times 1/10 = \dots\dots\dots \text{N}$$

20

$$\begin{aligned}\text{Alkalinity of CO}_3^{2-} \text{ alkalinity present in 1 Litre of water sample} &= \text{.....N} \times 50 \times 1000 \\ &= \text{.....ppm}\end{aligned}$$

MATERIALS REQUIRED

1. EDTA solution
2. Standard hard water
3. EBT indicator
4. Buffer solution
5. Burette, pipette, conical flask, 250 ml std. flask
6. Sample hard water.

PROCEDURE**TITRATION – I****With phenolphthalein indicator**

20 mL of the water sample is pipetted out in a clean conical flask. A drop of phenolphthalein indicator is added. Pink colour is observed. This solution is titrated against the standard HCl, already taken in the burette, until pink colour is disappeared. The end point is noted. This titre value corresponds to phenolphthalein end point (P).

TITRATION – II**With methyl orange indicator**

Few drops of methyl orange indicator are added to the same solution after the phenolphthalein end point. The titration is continued until the solution becomes red orange. The total titre value is noted. This value corresponds to methyl orange end point (M). The titration is repeated for concordant values.

From the titre values the amount of each alkalinity present in given water sample is calculated.

Bicarbonate alkalinity

$$\text{Volume of N/10 HCl for HCO}_3^- \text{ alkalinity, } V_1 = M - 2 [P]$$

$$= \text{.....} - 2 \times \text{..... mL}$$

$$= \text{..... mL}$$

$$\text{Normality of N/10 HCl, } N_1 = 1/10$$

$$\text{HCl, } N_1$$

$$\text{Volume of water sample } V_2 = 20 \text{ mL}$$

$$V_2$$

$$\text{Normality of water sample } N_2 = ?$$

$$N_2$$

$$V_1 N_1 = V_2 N_2$$

$$N_2 = V_1 N_1 / V_2 = \frac{\text{..... mL} \times 1/10}{20} = \text{..... N}$$

$$20$$

$$\text{Alkalinity of HCO}_3^- \text{ alkalinity present in 1 Litre of water sample} = \text{..... N} \times 50 \times 1000$$

$$= \text{..... ppm}$$

RESULT

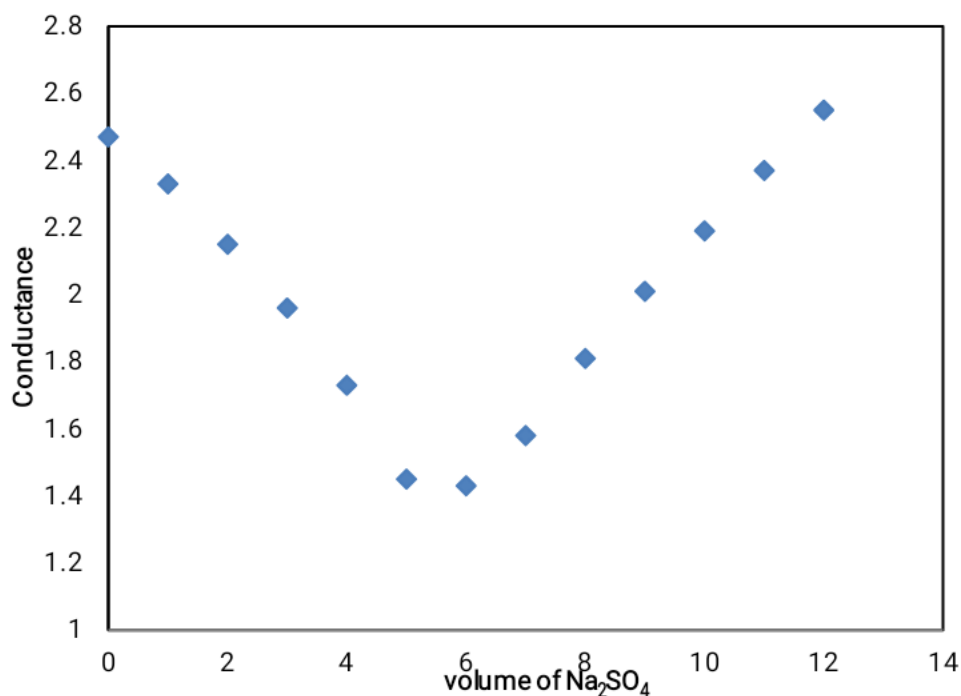
Water sample contains

1. Hydroxide alkalinity =.....ppm
2. Carbonate alkalinity =.....ppm
3. Bicarbonate alkalinity =.....ppm
4. Total alkalinity =.....ppm

Short procedure

| Content | Titration |
|------------------|----------------------------|
| Burette solution | Na_2SO_4 |
| Pipette solution | BaCl_2 |
| Electrode | Conductivity cell |
| Measurement | Conductance |
| Instrument | Digital conductivity meter |
| Temperature | Room Temperature |

Equivalent weight of $\text{BaCl}_2 = 122.14\text{g}$ **Model graph**



4. CONDUCTOMETRIC PRECIPITATION TITRATION USING (BaCl_2 - Na_2SO_4)

EXPT NO: 4

DATE:

AIM

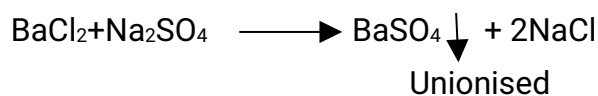
To determine the amount of BaCl_2 present in whole of the given solution by conductometric titration using standard solution of 0.05N Na_2SO_4 .

PRINCIPLE

Solution of electrolytes conducts electricity due to the presence of ions. Since specific conductance of the solution is proportional to the concentration of ions in it, conductance of the solution is measured during titration.

In the precipitation titration the ions are converted into insoluble precipitate, which will not contribute to the conductance.

When Na_2SO_4 is added slowly from the burette to the solution of BaCl_2 , BaSO_4 gets precipitated while the chloride ions remain unchanged.



The Ba^{2+} ions in the solution are replaced by free Na^+ ions. Since the mobility of Na^+ ions are less than that of Ba^{2+} ions, the conductance of the solution decreases. After the end point, when all the Ba^{2+} ions are replaced, further addition of Na_2SO_4 increases the conductance. This is due to the increase of Na^+ and SO_4^{2-} ions in the solution.

MATERIALS REQUIRED

1. Digital conductivity meter
2. Conductivity cell
3. 100 ml beaker
4. Standard Na_2SO_4 solution
5. Given BaCl_2 solution
6. Burette, pipette, glass rod etc.,
7. Distilled water

TABLE-I: BaCl_2 Vs Na_2SO_4

| S.No. | Volume of Na_2SO_4 added(ml) | Conductance (mho) | Remarks |
|-------|--|-------------------|---------|
| 1. | | | |
| 2. | | | |
| 3. | | | |
| 4. | | | |
| 5. | | | |
| 6. | | | |
| 7. | | | |
| 8. | | | |
| 9. | | | |
| 10. | | | |
| 11. | | | |
| 12. | | | |
| 13. | | | |
| 14. | | | |
| 15. | | | |
| 16. | | | |
| 17. | | | |

| | | | |
|-----|--|--|--|
| 18. | | | |
| 19. | | | |
| 20. | | | |

Calculation

Volume of BaCl₂ solution, $V_1=40\text{ml}$

Strength of the BaCl₂ solution, $N_1=.....?$

Volume of Na₂SO₄ solution, $V_2=$

Strength of the Na₂SO₄ solution, $N_2=0.05\text{N}$

According to the law of volumetric analysis $V_1N_1= V_2N_2$

$$N_1= V_2N_2/ V_1 ; \quad =.....\text{ml} \times 0.05\text{N}/40$$

Strength of the BaCl₂ $=.....\text{N}$

The amount of BaCl₂ present in 1 litre of the given solution = Strength of the BaCl₂ ×
Eq.wt.of

BaCl₂

$$=.....\text{N} \times 122.14$$

$$=.....\text{gms/L}$$

The amount of BaCl₂ present in 100ml of the given solution =
 $=.....\text{N} \times 122.14 \times 100/1000$
 $=.....\text{g}$

PROCEDURE

The burette is washed well with water and rinsed with the Na₂SO₄ solution. It is then filled with the same Na₂SO₄ solution up to zero level. The given BaCl₂ solution is made up to 100ml in a standard flask. 40ml of the made up solution is pipetted out into a clean 100ml beaker. The conductivity cell is placed in it and the two terminals of the cell are connected with a digital conductivity meter.

Now 0.5ml of Na₂SO₄ from the burette is added to the solution taken in the beaker, stirred, and then conductance is measured. This is continued up to the end point. (conductance is going on decreasing up to the end point). After the end point, again Na₂SO₄ is gradually added and few more readings are noted and tabulated.

Now the graph is plotted between the volume of Na₂SO₄ and conductance. The

end point is the intersection of the two curves. From the volume of Na_2SO_4 , the strength and hence the amount of BaCl_2 present in 100 ml is calculated.

RESULT

The amount of BaCl_2 present in 100 ml of the given solution =

Short Procedure

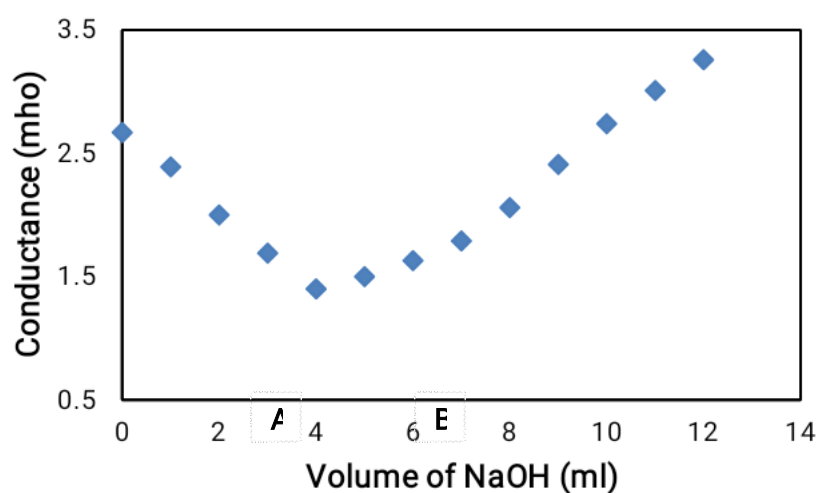
| Content | Titration Std. Sodium hydroxide Vs. Mixture of acids |
|------------------|---|
| Burette solution | Std.Sodium hydroxide solution |
| Pipette solution | Mixture of acids |
| Electrode | Conductivity cell |
| Measurement | Conductance |

| | |
|-------------|----------------------------|
| Instrument | Digital conductivity meter |
| Temperature | Room Temperature |

Equivalent weight of HCl = 36.45 g

Equivalent weight of CH_3COOH = 60 g

Model graph: Volume of NaOH Vs. Conductance



5. DETERMINATION OF STRENGTH OF ACIDS IN A MIXTURE USING CONDUCTIVITY METER

EXPT NO: 6

DATE

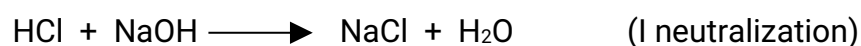
AIM

To determine the strengths and amounts of a mixture containing a strong acid and a weak acid (HCl and CH_3COOH) present in 100 ml of the given mixture of acid solution by conductometric titration. You are provided with a standard NaOH solution of 0.5N strength.

PRINCIPLE

Solutions of electrolytes conduct electricity due to the presence of ions. Since specific conductance of a solution is proportional to the concentration of ions in it, conductance of the solution is measured during titration.

When the sodium hydroxide solution is added slowly from the burette to the mixture solution, HCl (strong acid) gets neutralized first. Since the fast moving H^+ ions are replaced by slow moving Na^+ ions, decrease in conductance take place until the end point is reached.



After the complete neutralisation of all HCl, the neutralization of CH_3COOH starts,

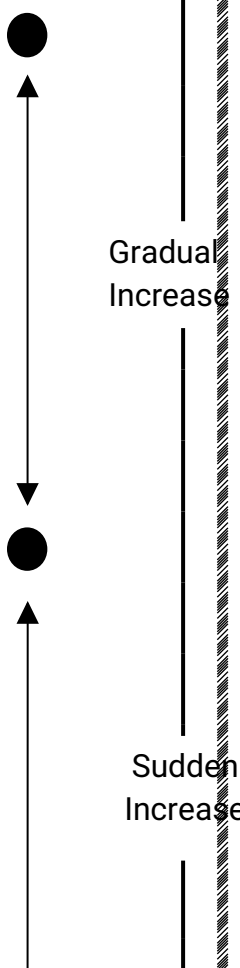


Since CH_3COONa is stronger electrolyte than CH_3COOH , conductivity slowly increases until all CH_3COOH is completely neutralized. When the end point is reached, further addition of NaOH will cause sudden increase in the conductance. This is due to the presence of fast moving OH^- ions.

TABLE

TITRATION OF MIXTURE (HCl+CH₃COOH) Vs. NaOH

| S.NO | Volume of NaOH added(ml) | Conductance(mho) | Remarks |
|------|--------------------------|------------------|-----------------|
| 1. | 0 | | Sudden Decrease |
| 2. | 0.5 | | |
| 3. | 1.0 | | |
| 4. | 1.5 | | |
| 5. | 2.0 | | |

| | | | |
|-----|------|--|--|
| 6. | 2.5 | |  |
| 7. | 3.0 | | |
| 8. | 3.5 | | |
| 9. | 4.0 | | |
| 10. | 4.5 | | |
| 11. | 5.0 | | |
| 12. | 5.5 | | |
| 13. | 6.0 | | |
| 14. | 6.5 | | |
| 15. | 7.0 | | |
| 16. | 7.5 | | |
| 17. | 8.0 | | |
| 18. | 8.5 | | |
| 19. | 9.0 | | |
| 20. | 9.5 | | |
| 21. | 10.0 | | |

MATERIALS REQUIRED

1. Digital conductivity meter
2. Conductivity cell
3. 100 ml Beaker
4. Standard NaOH
5. Given HCl & CH₃COOH mixture
6. Burette, pipette, glass rod etc.,
7. Distilled water.

STEP I***CALCULATION OF STRENGTH OF HCl SOLUTION***

Volume of the mixture (HCl) solution, V_1 = 40 ml

Strength of the mixture (HCl), N_1 = ?

Volume of the NaOH solution, V_2 = ml (A) (I titre value)

Strength of the NaOH solution, N_2 = 0.5

According to the law of volumetric analysis, V_1N_1 = V_2N_2

$$N_1 = V_2N_2 / V_1$$

$$N_1 = \dots\dots\dots \times 0.5 / 40$$

Strength of HCl solution =..... N

CALCULATION OF AMOUNT OF HCl

The amount of HCl present in

1000 ml of the given solution

$$= \text{Strength of HCl} \times \text{Eq. wt. of HCl}$$

$$= \dots\dots\dots N \times 36.45$$

$$= \dots\dots\dots \text{ g}$$

The amount of HCl present in

100 ml of the given solution

$$= \text{Strength of HCl solution} \times \text{Eq. wt. of HCl} \times 100/1000$$

$$= \dots\dots\dots N \times 36.45 \times 100/1000$$

$$= \dots\dots\dots \text{ g}$$

PROCEDURE

The given mixture of acids (HCl + CH₃COOH) is transferred into 100 ml standard flask and made upto 100 ml using distilled water. 40 ml of this made up solution is pipetted out into a clean 100 ml beaker. The conductivity cell is placed in it. The two terminals of the cell are connected with a digital conductivity meter

The burette is washed well with water and rinsed with the given standard NaOH solution. It is then filled with NaOH solution upto the zero level.

Now 0.5 ml of NaOH from the burette is added to the solution, taken in the beaker, stirred for some times and then conductivity is measured. (The conductivity is going on decreasing upto the end point). This process is repeated until at least five readings are taken after the end point (A) has been reached.

After the end point, again NaOH is gradually added, which causes increase in conductance. This increase in conductance is observed until the end point (B) is reached.

After the second end point, sudden increase in conductance is observed on further addition of NaOH. The readings (conductivity) are continuously measured for each addition of NaOH and are tabulated. Now the graph is plotted between the volume of NaOH added Vs. conductance. From the graph, the first end point (A) and the second end point (B) are noted. From the end points the strengths and amounts of HCl and CH₃COOH present in 100 ml of the mixture of solution are calculated.

STEP II

CALCULATION OF STRENGTH OF CH₃COOH SOLUTION

Volume of mixture (CH₃COOH), V_1 = 40ml

Strength of the mixture (CH₃COOH), N_1 = ?

Volume of the NaOH solution, V_2 = (B-A) ml (II titre value)

Strength of NaOH solution, N_2 = 0.5 N

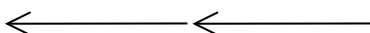
According to the law of volumetric analysis, $V_1N_1 = V_2N_2$

$$N_1 = V_2N_2 / V_1$$

Strength of CH_3COOH =N

The amount of CH_3COOH present in

The amount of CH_3COOH present in



RESULT

1. The amount of HCl present in 100 ml the given solution =g
2. The amount of CH₃COOH present in 100 ml of the given solution = g

Short Procedure

| Content | Titration Std. Sodium hydroxide Vs. Hydrochloric acid |
|------------------|---|
| Burette solution | Std. Sodium hydroxide solution |
| Pipette solution | Hydrochloric acid |
| Electrode | Glass electrode |
| Measurement | pH |
| Instrument | Digital pH meter |

Temperature

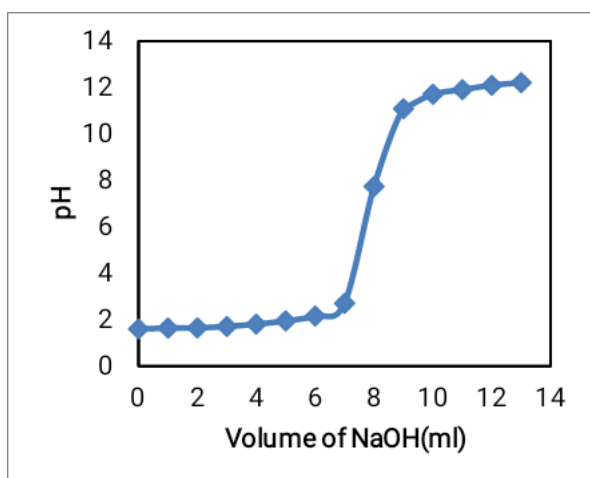
Room Temperature

Equivalent weight of HCl = 36.45 g

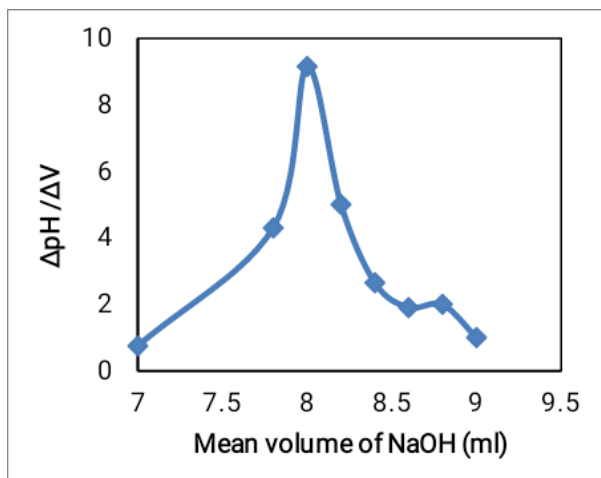
Model graph:

Graph: 1

Volume of NaOH Vs. Conductance



Graph: 2

Mean Volume of NaOH Vs. $\Delta pH / \Delta V$ 

6. pH METRY- DETERMINATION OF STRENGTH OF HCl by NaOH

EXPT NO: 7

DATE

AIM

To determine the strength of given HCl by pH metry, a standard solution of NaOH of

strength 0.5N is provided.

PRINCIPLE

The pH of the solution is related to the H^+ ion concentration by the following formula.

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

Measurement of pH of the solution gives the concentration of H^+ ions in the solution. When NaOH is added slowly from the burette to the solution of HCl, the fast moving H^+ ions are progressively replaced by slow moving Na^+ ions. As a result pH of the solution increases.



The increase in pH takes place until all the H^+ ions are completely neutralized (upto the end point). After the end point, further addition of NaOH increases the pH sharply as there is an excess of fast moving OH^- ions.

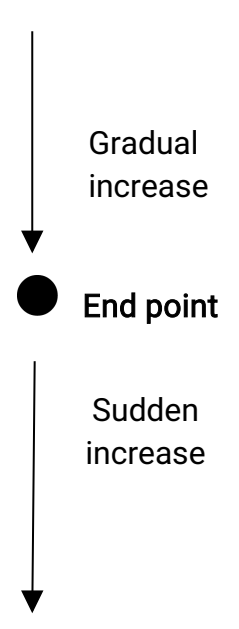
MATERIALS REQUIRED

1. Digital pH meter,
2. pH electrode,
3. 100 ml Beaker
4. Standard NaOH,
5. Given HCl,
6. Burette, pipette, Glass rod etc.,
7. Distilled water.

TABLE-I (Titration-I)

HCl (40ml) Vs. Std. NaOH

| S. NO | Volume of NaOH (ml) | pH | Remarks |
|-------|---------------------|----|---------|
|-------|---------------------|----|---------|

| | | | |
|-----|------|--|---|
| 1. | 0 | |  <p>Gradual increase</p> <p>● End point</p> <p>Sudden increase</p> |
| 2. | 1.0 | | |
| 3. | 2.0 | | |
| 4. | 3.0 | | |
| 5. | 4.0 | | |
| 6. | 5.0 | | |
| 7. | 6.0 | | |
| 8. | 7.0 | | |
| 9. | 8.0 | | |
| 10. | 9.0 | | |
| 11. | 10.0 | | |

PROCEDURE**TITRATION - I**

The given hydrochloric acid solution is transferred into 100ml standard flask and made up to the zero mark using distilled water. 40ml of this made up solution is pipetted out into a clean 100ml beaker. The glass electrode is dipped in it and connected with a pH meter.

The burette is washed well with water and then rinsed with a small amount of given NaOH solution. It is then filled with the same solution upto zero mark. Titration is carried out by adding std. NaOH solution in portions of 1ml from the burette to the HCl solution taken in the beaker and pH of the solution is noted for each addition. This process is continued until at least 5 readings are taken after the end point, and the range at which the end point lies is found out by plotting volume of NaOH added against pH (graph I).

TABLE-II (Titration II)
Titration of HCl (40ml) Vs. NaOH

| S.NO | Volume of NaOH (ml) | pH | Δ pH | Δ V (ml) | Δ pH / Δ V | Mean volume of NaOH (ml) |
|------|---------------------|----|-------------|-----------------|--------------------------|--------------------------|
| 1. | | | | | | |
| 2. | | | | | | |
| 3. | | | | | | |
| 4. | | | | | | |
| 5. | | | | | | |
| 6. | | | | | | |
| 7. | | | | | | |
| 8. | | | | | | |
| 9. | | | | | | |
| 10. | | | | | | |
| 11. | | | | | | |
| 12. | | | | | | |
| 13. | | | | | | |
| 14. | | | | | | |
| 15. | | | | | | |
| 16. | | | | | | |

Calculation***Calculation of strength of HCl***

Volume of HCl solution,

 $V_1 = 40\text{ml}$

Strength of the HCl solution,

 $N_1 = ?$

Volume of the NaOH solution,

 $V_2 = \dots\dots\dots$ (titre value)

Strength of the NaOH solution,

 $N_2 = 0.5\text{ N}$ According to the law of volumetric analysis, $V_1N_1 = V_2N_2$; $N_1 = V_2 \times N_2 / V_1$ $= \dots\dots\dots \times 0.5\text{ N} / 40\text{ ml}$

Strength of HCl present in the given solution

 $= \dots\dots\dots\text{ N}$ ***Calculation of amount of HCl***The amount of HCl present in 1000ml of the given solution = $\dots\dots\dots\text{ N} \times 36.45$ $= \dots\dots\dots\text{ g/L}$ The amount of HCl present in 100ml of the given solution = $\dots\dots\dots\text{ N} \times 36.45 \times 100 / 1000$ $= \dots\dots\dots \times 100 / 1000$ $= \dots\dots\dots\text{g}$

TITRATION - II

Another titration is carried out by adding std. NaOH solution in portions of 0.2 ml near the end point and pH of the solution is noted after each addition. The addition of NaOH is continued even after the end point for further 1ml. The accurate end point is found out by plotting $\Delta\text{pH}/\Delta V$ against volume of NaOH added (graph II). From the end point, the strength of HCl solution and hence the amount of HCl is calculated.

RESULT

Amount of HCl present in 100 ml of the solution =g.

Short Procedure

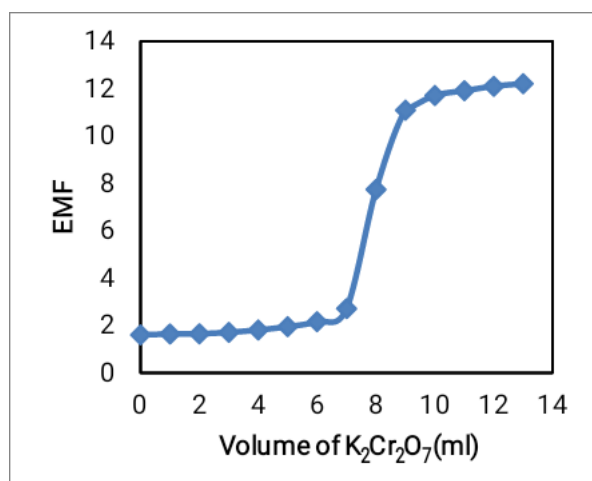
| Content | Titration Std. $K_2Cr_2O_7$ Vs. Ferrous ion solution |
|------------------|---|
| Burette solution | Std. $K_2Cr_2O_7$ |
| Pipette solution | Ferrous ion solution |
| Electrode | Platinum and calomel electrode |
| Measurement | EMF |
| Instrument | Digital potentiometer |
| Temperature | Room Temperature |

Equivalent weight of ferrous ion = 55.85 g

Model graph:

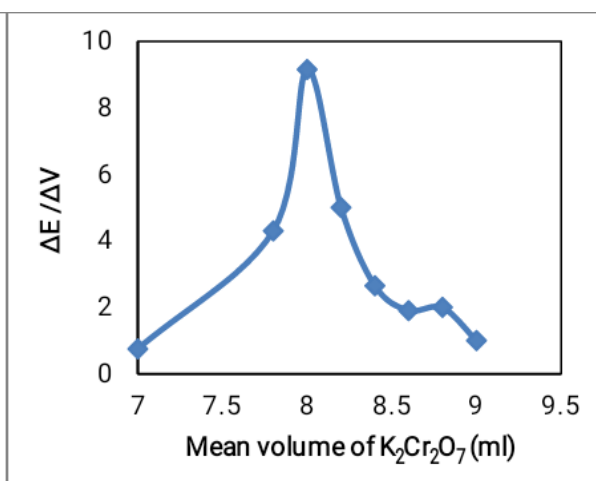
Graph: 1

Volume of $K_2Cr_2O_7$ Vs. EMF



Graph: 2

Mean Volume of $K_2Cr_2O_7$ Vs. $\Delta E / \Delta V$



7. ESTIMATION OF FERROUS ION BY POTENTIOMETRIC TITRATION

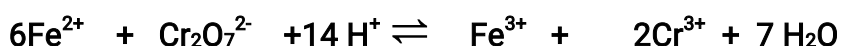
EXPT NO: 8

DATE: **AIM**

To estimate the amount of ferrous ion (Fe^{2+}) present in 100 ml of the given solution potentiometrically. A standard solution of potassium dichromate of strength 0.25 N is provided.

PRINCIPLE

Potentiometric titrations depend on measurement of EMF between reference electrode and an indicator electrode. When a solution of ferrous ion is titrated with a solution of potassium dichromate, the following redox reaction takes place.



During this titration Fe^{2+} ions are converted into Fe^{3+} , whose concentration increases. At the end point, there will be a sharp change due to sudden removal of all Fe^{2+} ions.

The cell is set up by connecting this redox electrode with a calomel electrode as shown below.



A graph between EMF measured against the volume of $\text{K}_2\text{Cr}_2\text{O}_7$ added is drawn and the end point is noted from the graph.

MATERIALS REQUIRED

1. Digital Potentiometer
2. Pt Electrode
3. Saturated Calomel Electrode
4. Std. $\text{K}_2\text{Cr}_2\text{O}_7$
5. Given ferrous ion solution
6. Burette and pipette

7. Dil.H₂SO₄

TABLE-I (Titration-I)

Std. K₂Cr₂O₇ Vs. Ferrous ion solution (20 ml)

| S.NO. | Volume of std. K ₂ Cr ₂ O ₇ (ml) | Emf (Volts) |
|-------|--|-------------|
| 1. | 0 | |
| 2. | 1.0 | |
| 3. | 2.0 | |
| 4. | 3.0 | |
| 5. | 4.0 | |
| 6. | 5.0 | |
| 7. | 6.0 | |
| 8. | 7.0 | |
| 9. | 8.0 | |
| 10. | 9.0 | |
| 11. | 10.0 | |

PROCEDURE

The given ferrous ion solution is transferred into 100ml standard flask and made up to the zero mark using distilled water. 20ml of this made up solution is pipetted out into a clean 100ml beaker. About 20 ml of dil. H_2SO_4 is added in it. A platinum electrode is dipped into the solution. This electrode is then coupled with a saturated calomel electrode and then connected to Digital potentiometer.

TITRATION-I

The burette is washed well with water and rinsed with the given $\text{K}_2\text{Cr}_2\text{O}_7$ solution. It is then filled with the same up to zero mark and titrated against the ferrous ion solution taken in the beaker.

The addition is carried out by adding Std. $\text{K}_2\text{Cr}_2\text{O}_7$ solution in portions of 1 ml and the EMF of the cell is measured after each addition. The addition of $\text{K}_2\text{Cr}_2\text{O}_7$ is continued even after the end point and the range at which end point lies is found out by plotting volume of $\text{K}_2\text{Cr}_2\text{O}_7$ added against EMF (graph-I).

TITRATION-II

Another titration is carried out by adding Std. $\text{K}_2\text{Cr}_2\text{O}_7$ solution in portions of 0.2 ml near the end point and the EMF of the cell is measured after each addition. The addition of $\text{K}_2\text{Cr}_2\text{O}_7$ is continued even after the end point for further 1 ml. The accurate end point is determined by plotting $\Delta E / \Delta V$ Vs. mean volume of $\text{K}_2\text{Cr}_2\text{O}_7$ added (graph-2). From the end point, the strength of ferrous ion solution and hence its amount is calculated.

TABLE-II (Titration II)
Std. K₂Cr₂O₇ Vs. Ferrous ion solution (20 ml)

| S.NO | Volume of K ₂ Cr ₂ O ₇ (ml) | Emf (Volts) | ΔE (Volts) | ΔV (mL) | $\Delta E / \Delta V$ Volts/ml | Mean Volume of K ₂ Cr ₂ O ₇ (ml) |
|------|--|-------------|--------------------|-----------------|--------------------------------|---|
| 1. | | | | | | |
| 2. | | | | | | |
| 3. | | | | | | |
| 4. | | | | | | |
| 5. | | | | | | |
| 6. | | | | | | |
| 7. | | | | | | |
| 8. | | | | | | |
| 9. | | | | | | |
| 10. | | | | | | |
| 11. | | | | | | |
| 12. | | | | | | |
| 13. | | | | | | |
| 14. | | | | | | |
| 15. | | | | | | |
| 16. | | | | | | |

Calculation***Calculation of strength of ferrous iron solution***Volume of ferrous ion solution $V_1 = 20\text{ml}$ Strength of ferrous ion solution $N_1 = ?$ Volume of the K₂Cr₂O₇ solution $V_2 = \dots\dots\dots \text{ml}$ Strength of the K₂Cr₂O₇ solution $N_2 = 0.25 \text{ N}$

According to the law of volumetric analysis, $V_1 N_1 = V_2 N_2$; $N_1 = V_2 \times N_2 / V_1$
 $= \dots\dots\dots \times 0.25\text{N}/20\text{ml}$

Strength of ferrous ion solution, $N_1 = \dots\dots\dots \text{N}$ ***Calculation of amount of ferrous iron***The amount of Fe²⁺ ion present in 1000ml of the given solution = $\dots\dots\dots \text{N} \times 55.85$

= g/L

The amount of Fe^{2+} ion present in 100ml of the given solution =N \times 55.85 \times 100/1000

= g/L

RESULT

The amount of ferrous iron present in 100ml of the given solution = g.



