

Date:

Engineering Chemistry Laboratory (CHY1001)

Lab Manual

AY-2024-2025

Name: _____

Registration No: _____ Section: _____

Department of Chemistry

Manipal University Jaipur

List of Experiments:

| NO. | EXPERIMENT | TYPE | PAGE |
|-----|---|--------------------------|------|
| 1 | To determine the strength of given unknown ferrous ammonium sulphate solution by titrating against standard $K_2Cr_2O_7$ using diphenylamine as an internal indicator | Redox titration | 07 |
| 2 | To determine the strength of given unknown ferrous ammonium sulphate solution by titrating against standard $K_2Cr_2O_7$ using potassium ferricyanide as an external indicator. | Redox titration | 10 |
| 3 | To determine the total, permanent and temporary hardness of given water sample by complexometric titration using <i>EDTA</i> solution. | Complexometric titration | 13 |
| 4 | To determine the strength of sodium carbonate and sodium hydroxide in given alkali mixture using methyl orange and phenolphthalein as indicators. | Acid-base titration | 16 |
| 5 | To determine the strength of ferrous ammonium sulphate by titrating against $KMnO_4$ solution. | Redox titration | 19 |
| 6 | To prepare urea formaldehyde resin. | Synthesis | 22 |
| 7 | To determine the strength of given <i>HCl</i> solution using a standard <i>NaOH</i> solution by performing a pH-metric titration. | pH-metric titration | 24 |
| 8 | To determine the strength of a given <i>HCl</i> solution using a standard <i>NaOH</i> solution by performing a conductometric titration. | Conductometric titration | 28 |
| 9 | To determine the strength of given CH_3COOH solution using a standard <i>NaOH</i> solution by performing a pH-metric titration. | pH-metric titration | 31 |
| 10 | To determine the pK_a values of phosphoric acid solution by titrating with 0.2N <i>NaOH</i> solution pH metrically and verify that it is a tribasic acid. | pH-metric titration | 33 |
| 11 | To determine the strengths of acids in a mixture of <i>HCl</i> and CH_3COOH solution using a standard <i>NaOH</i> solution by performing a pH-metric titration. | pH-metric titration | 35 |
| 12 | To determine the strength of given <i>HCl</i> solution using a standard <i>NaOH</i> solution by performing a conductometric titration. | conductometric titration | 38 |
| 13 | To determine the viscosity of a given lubricating oil at various temperatures using Redwood Viscometer. | Analysis | 40 |
| 14 | To determine the cloud point and pour point of a given sample of lubricating oil using cloud and pour point apparatus. | Analysis | 43 |
| 15 | To determine the water equivalent of benzoic acid by Using Bomb calorimeter. | Analysis | 45 |
| 16. | Softening of hard water using Lime-soda process | | 48 |

General Instructions for the Chemistry Laboratory

- ❖ **Be punctual:** Late comers will not be entertained in the lab.
- ❖ **Come prepared:** Check the experiment you are going to perform.
- ❖ **Take safety measures:** Students must wear apron and safety goggles before entering the lab.
- ❖ **No gadgets:** Do not use mobile phones and other electronic devices in the lab.
- ❖ **Be careful with the apparatus:** The glass apparatus and other instruments should be handled with care and responsibility. Please report any damage of glassware and/or equipment to the faculty/lab instructor/lab technician as soon as possible.
- ❖ **No littering and wastage:** Do not throw solid waste into the sink. They must be thrown into the waste bin. Keep the water and gas taps closed except when these utilities are needed.
- ❖ **Be organized:** Do not move reagents and chemicals from their own place. Replace the bottles immediately after use. Do not keep reagent bottles on the work bench to avoid contamination.
- ❖ **Keep record:** Update the records and lab manuals session wise. Please get your data verified and signed by the faculty before leaving the lab.
- ❖ **Be honest:** Never cook the result by recording false observations or by making manipulated calculations.
- ❖ **In case of any emergency, immediately report to the faculty/lab instructor/lab technician for proper care.**

Miscellaneous Policies and Procedures

Laboratory notebook:

- i. The one essential tool for any laboratory worker in any field is the laboratory notebook. Its main purpose is to record observations, variations in procedures, experimental results, conclusions and supplementary information from texts, handbooks, and other printed source.
- ii. Never attempt to remove the pages from the notebook nor to erase the entries. Simply cross out neatly any entry you wish to delete and give the page reference for the correction or type in the correction.
- iii. Label the notebook on the outside and inside the front cover with your name, roll number, batch number.
- iv. Leave the first one or two pages blank for a Table of Contents which you must keep up to date each week.
- v. Always read the upcoming experiments carefully and thoroughly, being sure to understand of the directions before entering the lab.
- vi. It is essential that you come to the laboratory with a schedule of operations planned in advance, and with all tables, equations, etc. completed. Tables for the recording of observations should be clearly and neatly set-up advance.

- vii. Take data during lab. Not after lab, on the assumption that it will be neater. Put data directly in your lab book rather than transcribing from another source (e.g., notebook or lab partner).

Homework and Lab Report Due Dates:

Lab reports are due one week from the date of performance, at the beginning of the lab period. Only a verifiable illness (Doctor's note) or prior permission of the instructor counts as excused absences. Unexcused lab reports will receive a **grade of zero**.

Lab Partners:

For those experiments where students are to work in pairs, lab partners will be assigned randomly as announced by the instructor at the beginning of the lab period. You may not exchange lab partners. Both lab partners must be present for the entire experiment.

Copying:

All lab reports are to be your own. Lab partners are to independently produce their lab reports. It is very easy for the grader to spot identical work among two or more students. In the event of copying, all students involved will receive a grade of zero; therefore do not give a copy of your lab report to another student.

Safety Instructions for Chemistry Laboratory

1. You must dress appropriately for the laboratory. Lab coats are required to be worn. Bare feet, sandals, or other open-toed shoes are not permitted in the laboratory. Shorts and short skirts are likewise not permitted; legs must be covered to below the knees.
2. State-approved safety goggles must be always worn in the laboratory.
3. Long hair should be tied back.
4. Learn the location and operation of the safety showers, emergency eyewashes and fire extinguishers in the laboratory. In the case of spill onto a person or clothing, the immediate action should be to flush with water and lots of it.
5. The fire extinguishers should only be used for real emergencies since the chemicals they contain can cause considerable damage.
6. Become familiar with all the exits from the laboratory.

7. Never attempt any unauthorized or unassigned experiments. Follow the experimental procedures explicitly, checking and double-checking the identity of all reagents before you use them.
8. Clean up spills immediately. The next person to come along has no way of knowing if the clear liquid or white powder on the lab bench is innocuous or hazardous. Neutralize acid spills with sodium bicarbonate (baking soda) before cleaning them up. Spills of sulfuric acid solutions are particularly hazardous since only the water will evaporate, thereby making the solution more concentrated upon standing.
9. Never return unused reagents to their storage container. If you take more than you need, dispose of the excess in the appropriate manner. Use the reagents sparingly—they are expensive and time-consuming to prepare.
10. Do not pick up hot objects. Be sure your apparatus is cool before picking it up. Do not point the open end of a test tube or other vessel containing a reaction mixture toward yourself or anyone else. If the procedure calls for you to observe the odor of the contents of a vessel, hold it upright in front of you, gently fan some of the vapors toward your nose and sniff cautiously. Most chemical vapors are at least irritating, and many are quite toxic. Please do not taste any chemicals.
11. Do not eat, drink or smoke in the laboratory.
12. **Playing of radios, tapes, CDs is not permitted.** This includes small portable devices used with earphones or headsets.
13. Keep coats, backpacks and other non-essential materials away from areas where people are working.
14. Dispose of all broken glassware and other sharp objects in the cardboard glass disposal boxes. Custodial personnel will stop collecting trash after they find broken glass in the trashcans.
15. Wash hands often when working in lab, and always wash thoroughly before leaving.
16. Use the hood for evaporation of anything other than water. The vapors from your procedure alone may not present a problem but those from all the students in the lab could combine to create a hazard.
17. Do not leave a Bunsen burner or other heated apparatus unattended. The person working next to you may not know what is involved with your setup and may be working with a flammable material. Turn off open flames if you must leave your area. Make sure the gas taps are completely off whenever the Bunsen burner is not lit.

18. Waste Disposal: Dispose of chemical reagents and other materials properly. The proper disposal of chemical wastes is essential to the health and safety of Institute faculty, staff, students and the surrounding community.
19. Chemical wastes must be managed and discarded in the most responsible and environmentally sound method available.
20. Never use reagents from an unmarked bottle.
21. If a fire is occurred in a beaker or some other container, cover it with a glass dish or other flame-retardant item.
22. Notify instructor immediately in case of an accident.

Safety measures in accidental case

1. Eye accidents:

Flood your eyes immediately with water. For an acid, use dilute Sodium bicarbonate solution; for an alkali, use dilute boric acid solution.

2. Burns:

Acid burns: wash immediately with large quantity of water, then with dilute (8%) sodium bicarbonate solution. If burn is severe, wash again with water and apply the acriflavine.

Alkali burns: wash immediately with water and 1% acetic acid solution.

Bromine burns: wash immediately with ample supply of petrol, when the bromine will be completely removed from skin.

Organic substance: wash immediately with soap and warm water.

3. Cuts:

Wash the wound with sterile gauze, soap and water. Disinfect with an antiseptic and apply a bandage.

4. Reagents in mouth:

If the reagents is in the mouth and not swallowed then spit out at once, wash the mouth out repeatedly with water.

If the substance like acid or alkali is swallowed, dilute by drinking much water. Then for acids follow by drinking much lime water. Milk may be given but not emetics.

For salts of heavy metals give milk or white of egg.

For arsenic or mercury compounds give emetic without any delay.

5. Gas poisoning:

Remove patient to fresh air and loosen clothing at neck. If breathing has stop give artificial respiration until the doctor arrives.

EXPERIMENT 1: Estimation of FAS using internal indicator (diphenylamine)

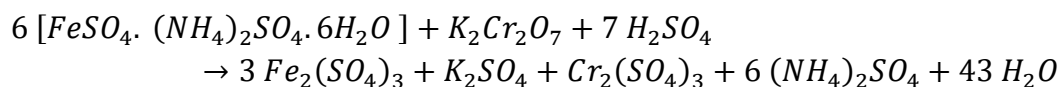
Objective: To determine the strength of an unknown ferrous ammonium sulphate solution by titrating against standard $K_2Cr_2O_7$ using diphenylamine as an internal indicator.

Principle: Ferrous ammonium sulphate (FAS, also known as Mohr's salt) is a complex salt with the chemical formula $[FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O]$.

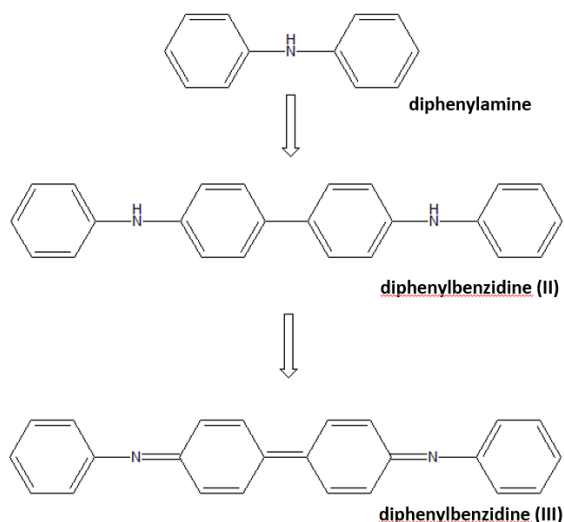
To determine the strength of FAS, we can use **redox titration** to estimate the amount of ferrous ions (Fe^{2+}) present in it. The amount of ferrous ions (Fe^{2+}) can be estimated by oxidizing them to ferric (Fe^{3+}) ions.

Potassium dichromate ($K_2Cr_2O_7$) acts as an oxidizing agent in acidic medium and it can oxidize ferrous ions (Fe^{2+}) to ferric ions (Fe^{3+}).

The complete reaction is



To properly identify the endpoint, an indicator is required. In this experiment, the redox indicator *diphenylamine* is used. The color exhibited by diphenylamine change from greenish to purple at the end point.



A mixture of dil. H_2SO_4 and dil H_3PO_4 is used. This mixture ensures that the proper redox potential of the solution is maintained, and the product remains in diphenylbenzidine (III) form which is purple in colour. If the redox potential is not maintained, the colour change would not be prominent to detect by eyes.

Procedure:**Standardization of $K_2Cr_2O_7$ solution:**

1. Wash all the apparatus with distilled water.
2. Rinse and fill the burette with $K_2Cr_2O_7$ solution.
3. Pipette out 10 ml N/40 FAS solution into conical flask.
4. Add 5 ml of acid mixture [dil. $H_2SO_4 + H_3PO_4$] into conical flask.
5. Add 2-3 drops of diphenylamine indicator to the solution.
6. Titrate the FAS solution with $K_2Cr_2O_7$ solution from the burette.
7. At the end point, color of the solution will turn to purple from light green.
8. Note the burette reading and repeat the titration two more times.

Estimation of FAS:

(Follow the same procedure for the unknown FAS solution; replace Step-3 with unknown FAS solution)

Observations and calculations:**Standardization of $K_2Cr_2O_7$:**

| Sr. No. | Volume of FAS solution taken (ml) | Burette reading | | Volume of $K_2Cr_2O_7$ consumed (ml) | Average burette reading (ml) |
|---------|-----------------------------------|-----------------|-------|--------------------------------------|------------------------------|
| | | Initial | Final | | |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

Standardization of $K_2Cr_2O_7$ solution:

N_1 = Normality of known FAS solution = _____

V_1 = Volume of FAS solution = _____

N_2 = Normality of $K_2Cr_2O_7$ solution = (unknown)

V_2 = Volume of $K_2Cr_2O_7$ solution (burette reading) = _____

$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_1V_1}{V_2} = \text{_____} N$$

Date:

Estimation of unknown FAS solution:

| Sr. No. | Volume of FAS solution taken (ml) | Burette reading | | Volume of $K_2Cr_2O_7$ consumed (ml) | Average burette reading (ml) |
|---------|-----------------------------------|-----------------|-------|--------------------------------------|------------------------------|
| | | Initial | Final | | |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

Estimation of Ferrous ammonium sulphate:

N_3 = Normality of unknown FAS solution = (unknown)

V_3 = Volume of FAS solution = _____

N_4 = Normality of $K_2Cr_2O_7$ solution = _____

V_4 = Volume of $K_2Cr_2O_7$ solution = _____

$$N_3V_3 = N_4V_4$$

$$N_3 = \frac{N_4V_4}{V_3} = \text{_____} N$$

Strength = $N_3 \times \text{Equivalent weight of FAS g/L}$

$$= N_3 \times 392 \text{ g/L}$$

$$= \text{_____} \text{ g/L}$$

Note: The equivalent weight of FAS = 392

Result:

The strength of given unknown ferrous ammonium sulphate solution = _____ g/L

EXPERIMENT 2: Estimation of FAS using external indicator (potassium ferricyanide)

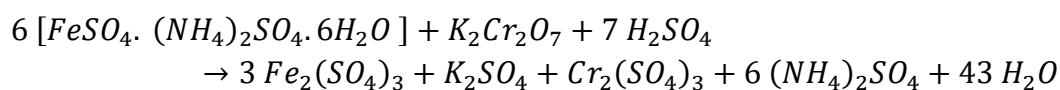
Objective: To determine the strength of given unknown ferrous ammonium sulphate solution by titrating against standard $K_2Cr_2O_7$ using potassium ferricyanide as an external indicator.

Principle: Ferrous ammonium sulphate (FAS, also known as Mohr's salt) is a complex salt with the chemical formula $[FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O]$.

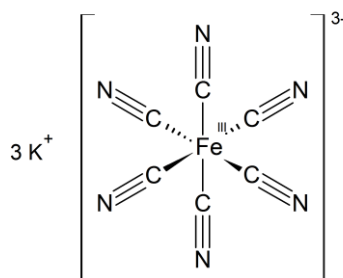
To determine the strength of FAS, we can use **redox titration** to estimate the amount of ferrous ions (Fe^{2+}) present in it. The amount of ferrous ions (Fe^{2+}) can be estimated by oxidizing them to ferric (Fe^{3+}) ions.

Potassium dichromate ($K_2Cr_2O_7$) acts as an oxidizing agent in acidic medium and it can oxidize ferrous ions (Fe^{2+}) to ferric ions (Fe^{3+}).

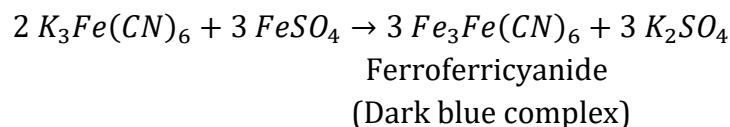
The complete reaction is



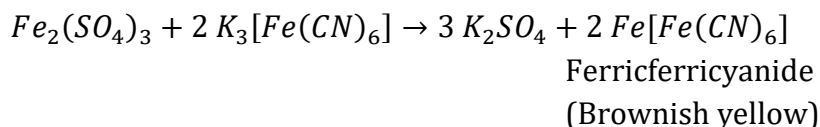
To properly identify the endpoint, an indicator is required. In this experiment, freshly prepared 0.1% solution of potassium ferricyanide acts as external indicator (freshly prepared potassium ferricyanide indicator is used because with time, it becomes contaminated with potassium ferrocyanide).



Here the indicator cannot be added to the solution to be titrated, but is placed in the depressions of a white glazed tile. As long as ferrous ion is present in the titration mixture, the indicator will turn blue on the transfer of solution. The appearance of blue color is due to the formation of ferroferricyanide (Turnbull's blue). The following reaction happens:



At the end point, all Fe^{2+} ions are oxidized to Fe^{3+} , and the color of the indicators drop becomes light brownish yellow due to the formation of brown colored ferricferricyanide complex.

**Procedure:****Standardization of $K_2Cr_2O_7$ solution:**

1. Wash all the apparatus with distilled water.
2. Rinse and fill the burette with $K_2Cr_2O_7$ solution.
3. Pipette out 10 ml N/40 FAS solution into conical flask.
4. Add 5 ml of dil. H_2SO_4 in the conical flask.
5. Put a series of drops of the indicator (potassium ferricyanide solution) white glazed tile.
6. Titrate the FAS solution with $K_2Cr_2O_7$ solution from the burette.
7. In regular interval, take a drop of titration mixture and put it on one of the indicator-drops on tile and notice the color. At the beginning of the experiment, the drop will turn dark blue.
8. Continue the titration and repeat the process at regular intervals. When the color of indicator drop does not turn blue, the end point is reached.
9. Note the burette reading and repeat the titration two more times.

Estimation of FAS:

(Follow the same procedure for the unknown FAS solution; replace Step-3 with unknown FAS solution)

Observations and calculations:**Standardization of $K_2Cr_2O_7$:**

| Sr. No. | Volume of FAS solution taken (ml) | Burette reading | | Volume of $K_2Cr_2O_7$ consumed (ml) | Average burette reading (ml) |
|---------|-----------------------------------|-----------------|-------|--------------------------------------|------------------------------|
| | | Initial | Final | | |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

Standardization of $K_2Cr_2O_7$ solution:

N_1 = Normality of known FAS solution = _____

V_1 = Volume of FAS solution = _____

Date:

N_2 = Normality of $K_2Cr_2O_7$ solution = (unknown)

V_2 = Volume of $K_2Cr_2O_7$ solution (burette reading) = _____

$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_1V_1}{V_2} = \text{_____} N$$

Estimation of unknown FAS solution:

| Sr. No. | Volume of FAS solution taken (ml) | Burette reading | | Volume of $K_2Cr_2O_7$ consumed (ml) | Average burette reading (ml) |
|---------|-----------------------------------|-----------------|-------|--------------------------------------|------------------------------|
| | | Initial | Final | | |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

Estimation of Ferrous ammonium sulphate:

N_3 = Normality of unknown FAS solution = (unknown)

V_3 = Volume of FAS solution = _____

N_4 = Normality of $K_2Cr_2O_7$ solution = _____

V_4 = Volume of $K_2Cr_2O_7$ solution = _____

$$N_3V_3 = N_4V_4$$

$$N_3 = \frac{N_4V_4}{V_3} = \text{_____} N$$

Strength = $N_3 \times \text{Equivalent weight of FAS g/L}$

$$= N_3 \times 392 \text{ g/L}$$

$$= \text{_____} \text{ g/L}$$

Note: The equivalent weight of FAS = 392

Result:

The strength of given unknown ferrous ammonium sulphate solution = _____ g/L

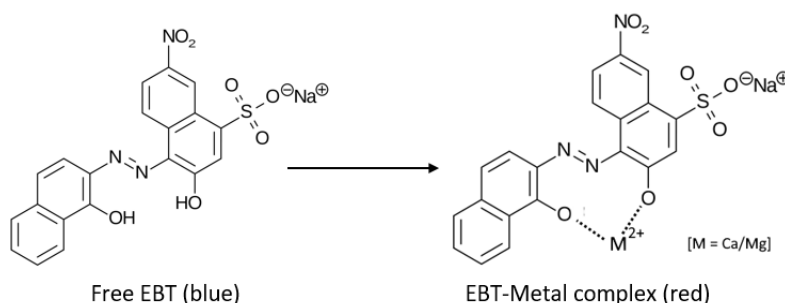
EXPERIMENT 3: Estimation of the hardness of water

Objective: To determine the total, permanent and temporary hardness of given water sample by complexometric titration using EDTA solution.

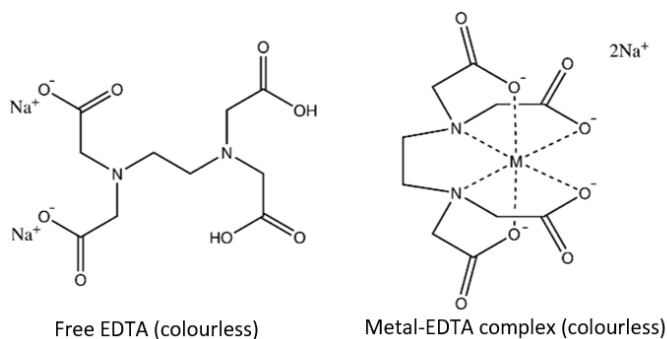
Principle: The presence of Ca^{2+} and Mg^{2+} ions (among many others e.g., iron, manganese, aluminium etc.) in water introduces hardness. Hard water consumes a lot of soap or detergent before it starts foaming. If hard water is used in a boiler, it results in excessive scaling and sludging which results in loss of efficiency and safety.

If Ca^{2+} and Mg^{2+} ions are present as bicarbonate salts, they can be easily removed by boiling as boiling decomposes bicarbonates to insoluble carbonate salts ($CaCO_3$ and $MgCO_3$). It is said to be temporary hardness. When Ca^{2+} and Mg^{2+} ions are present in any other form (e.g., chlorides, nitrates, sulphates etc.) it is called permanent hardness of water as this type of hardness cannot be removed easily.

To determine the hardness of water, we perform a complexometric titration. Eriochrome Black-T (EBT) is used as an indicator and the titration is performed with ethylene diamine tetraacetic acid (EDTA) solution.



When EBT is added to hard water, the indicator molecule forms a semi-stable complex with the hardness producing metal ions. The complex is wine-red in colour. When EDTA is added to this mixture, EDTA forms a colourless stable complex with the metal atoms and as a result, the indicator becomes free. Hence the end point of this titration identified by the colour change of wine red to blue.



EDTA is a chelating agent which behaves as a hexadentate ligand in the basic medium. In basic medium, the tetra-basic form of EDTA forms complexes with virtually all metal ions. Each of the acidic oxygen and each of the amine nitrogen can donate one pair of electrons to form a one-to-one complex with the metal ions.

The effectiveness of the complexing agent EDTA is strongly affected by pH. The electron pair of the carboxylic acid groups of EDTA are only available to the metal ion when the acid is dissociated. At low pH EDTA will be un-dissociated will not be an effective complexing agent. Additionally, many metal ions form complexes with hydroxide ions. Hydroxide ions compete with the chelating agent for coordination sites in the metal ion. Therefore, the effectiveness of the complexing agent will also be reduced at high pH.

Therefore, suitable buffer solution is added to keep pH of the solution nearly constant at pH = 10. A mixture of NH_4Cl and NH_4OH is used as a buffer solution

Procedure:**Total Hardness:**

1. Clean the burette and fill with standard EDTA solution and note the initial reading.
2. Pipette out 10 ml of hard water into a clean conical flask.
3. Add 5 ml of buffer solution (pH=10) and 2-3 drops of EBT indicator.
4. Titrate it against standard EDTA solution.
5. At the end point color changes from wine red to clear blue.
6. Note the burette reading and repeat it two more times to get the reading corresponding to the total hardness.

Permanent and temporary hardness:

7. Take 250 ml hard water in to 500 ml beaker and boil it gently for an hour, filter the solution in to a 250 ml measuring flask and make the solution up to 250 ml with distilled water and shake it thoroughly.
8. Repeat steps 1-5 as mentioned above. Replace Step-3 with the boiled and filtered water sample to get the permanent hardness.
9. Temporary hardness can be obtained by subtracting the value of permanent hardness from the value of total hardness.

Date:

Observations:

Estimation of total hardness:

| Sr. No. | Volume of hard water (ml) | Burette reading | | Volume of EDTA consumed (ml) | Average burette reading (ml) |
|---------|---------------------------|-----------------|-------|------------------------------|------------------------------|
| | | Initial | Final | | |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

$$\text{Total hardness} = \frac{BR \times \text{molarity of EDTA} \times MW \text{ of } CaCO_3(100) \times 1000}{\text{Volume of hard water}}$$

Estimation of permanent hardness:

| Sr. No. | Volume of boiled hard water (ml) | Burette reading | | Volume of EDTA consumed (ml) | Average burette reading (ml) |
|---------|----------------------------------|-----------------|-------|------------------------------|------------------------------|
| | | Initial | Final | | |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

$$\text{Total hardness} = \frac{BR \times \text{molarity of EDTA} \times MW \text{ of } CaCO_3(100) \times 1000}{\text{Volume of hard water}}$$

Result: The hardness of given water sample is

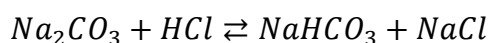
- Total hardness = _____ ppm
- Permanent hardness = _____ ppm
- Temporary hardness = _____ ppm

EXPERIMENT 4: Titration of alkali mix

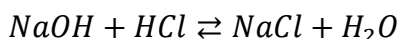
Objective: To determine the strength of sodium carbonate and sodium hydroxide in given alkali mix.

Principle: Determination of $NaOH$ and Na_2CO_3 in a mixture, involves the double indicator method.

The principle involved in this method is that when Na_2CO_3 solution is titrated with HCl the neutralization occurs in two stages. The first step is hydrogen carbonate step:

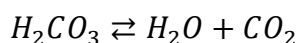
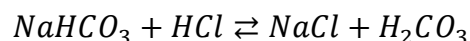


At the same time, all $NaOH$ gets titrated.



The equivalence for the first step on ionization of carbonic acid is at pH 8.3. This can be detected by phenolphthalein indicator which works in the pH range of 8-10.

The second step corresponds to the displacement of all carbonic acid. If the titration is continued with methyl orange, the neutralization of the bicarbonate to the carbonic acid takes place:



Hence, when the endpoint is observed with phenolphthalein, all of $NaOH$ and half of Na_2CO_3 is titrated.

$$[P] = NaOH + \frac{1}{2} Na_2CO_3$$

And when the endpoint is observed with methyl orange, all of $NaOH$ and all of Na_2CO_3 is titrated.

$$[M] = NaOH + Na_2CO_3$$

Procedure:

1. Standardization of HCl

- i) Pipette out 10 ml of standard sodium carbonate solution and transfer it into a conical flask.
- ii) Add 2-3 drops of methyl orange indicator into the conical flask
- iii) Titrate it against HCl solution until a sharp color change from yellow to red indicates the end point.

- iv) Repeat the procedure for two more times.
2. **Estimation of phenolphthalein end point:**
- Pipette out 10 ml of alkali mix and transfer it into a conical flask.
 - Add 2 – 3 drops of phenolphthalein indicator, pink color appears in the conical flask.
 - Titrate it against the HCl solution until the pink color disappears i.e. phenolphthalein end point [P]
 - Repeat the procedure for two more times.
3. **Estimation of phenolphthalein end point:**
- Pipette out 10 ml of alkali mix and transfer it into a conical flask.
 - Now add 2-3 drops of methyl orange indicator into the conical flask and continue the titration.
 - A sharp color change from yellow to red occurs indicates the end point. This titrate value is known as methyl orange end point [M]
 - Repeat the procedure for two more times.

Observation and results:**Standardization of HCl:**

| Sr. No. | Volume of Na_2CO_3 (ml) | Burette reading | | Volume of HCl consumed (ml) | Average burette reading (ml) |
|---------|---------------------------|-----------------|-------|-----------------------------|------------------------------|
| | | Initial | Final | | |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

 $N_1 = \text{Normality of known } Na_2CO_3 \text{ solution} = \underline{\hspace{2cm}}$
 $V_1 = \text{Volume of } Na_2CO_3 \text{ solution} = \underline{\hspace{2cm}}$
 $N_2 = \text{Normality of HCl} = (\text{unknown})$
 $V_2 = \text{Volume of HCl (burette reading)} = \underline{\hspace{2cm}}$

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{N_1 V_1}{V_2} = \underline{\hspace{2cm}} N$$

Date:

Analysis of alkali mixture:

| Sr. No. | Volume of alkali mixture (ml) | Burette reading for phenolphthalein | | Burette reading for methyl orange | |
|---------|-------------------------------|-------------------------------------|-------------|-----------------------------------|-------------|
| | | Readings | Average (P) | Readings | Average (M) |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

Calculation for Na_2CO_3 :

- Volume of HCl corresponding to Na_2CO_3 (V_1) = $2 [M - P] =$ _____ ml
- Normality of HCl (N_1) = _____ N
- Volume of alkali (V_2) = _____ ml
- Normality of Na_2CO_3 (N_2) = $\frac{N_1 V_1}{V_2} =$ _____ N
- Equivalent weight of $Na_2CO_3 = 53$
- Strength of $Na_2CO_3 = normality \times eq. wt =$ _____ g/lit

Calculation for $NaOH$:

- Volume of HCl corresponding to $NaOH$ (V_1) = $[2 P - M] =$ _____ ml
- Normality of HCl (N_1) = _____ N
- Volume of alkali (V_2) = _____ ml
- Normality of $NaOH$ (N_2) = $\frac{N_1 V_1}{V_2} =$ _____ N
- Equivalent weight of $NaOH = 40$
- Strength of $NaOH = normality \times eq. wt =$ _____ g/lit

Result: The alkali mixture contains:

- $NaOH =$ _____ g/L
- $Na_2CO_3 =$ _____ g/L

EXPERIMENT 5: Estimation of FAS using self-indicator

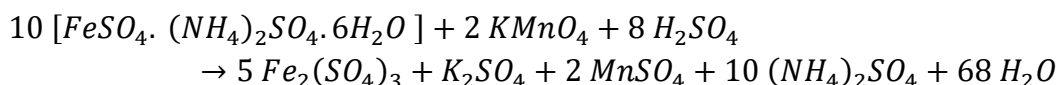
Objective: To determine the strength of an unknown ferrous ammonium sulphate solution by titrating against standard $KMnO_4$ solution.

Principle: Ferrous ammonium sulphate (FAS, also known as Mohr's salt) is a complex salt with the chemical formula $[FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O]$.

To determine the strength of FAS, we can use **redox titration** to estimate the amount of ferrous ions (Fe^{2+}) present in it. The amount of ferrous ions (Fe^{2+}) can be estimated by oxidizing them to ferric (Fe^{3+}) ions.

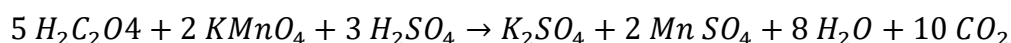
Potassium permanganate ($KMnO_4$) acts as an oxidizing agent in acidic medium and it can oxidize ferrous ions (Fe^{2+}) to ferric ions (Fe^{3+}).

The complete reaction is

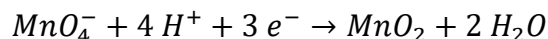


To properly identify the endpoint, an indicator is required. In this experiment, $KMnO_4$ by itself acts as a self-indicator. As $KMnO_4$ is bright purple in colour and when reduced, produced $MnSO_4$ is colourless, the endpoint is marked by the appearance of light pink colour (because of residual $KMnO_4$).

$KMnO_4$ is standardised by titrating it against oxalic acid ($H_2C_2O_4$). The complete reaction is:



For this reaction, the temperature has to be maintained at $60^\circ C$ to $70^\circ C$. Else, MnO_2 will be formed and the solution will become turbid. The endpoint will be hard to detect if the temperature and acidity is not maintained.



Procedure:

Standardization of $K_2Cr_2O_7$ solution:

1. Wash all the apparatus with distilled water.
2. Rinse and fill the burette with $KMnO_4$ solution.
3. Pipette out 10 ml N/10 oxalic acid solution into conical flask.
4. Add 5 ml of dilute H_2SO_4 into the conical flask.
5. Heat the mixture to $60^\circ C$ to $70^\circ C$ on a hot water bath.
6. Titrate the FAS solution with $KMnO_4$ solution from the burette.
7. At the end point, color of the solution will turn to faint purple/pink from colourless.

Date:

8. Note the burette reading and repeat the titration two more times.

Estimation of FAS:

(Follow the same procedure for the unknown FAS solution; replace Step-3 with unknown FAS solution and skip Step-5)

Observations and calculations:

Standardization of $KMnO_4$:

| Sr. No. | Volume of oxalic acid solution taken (ml) | Burette reading | | Volume of $KMnO_4$ consumed (ml) | Average burette reading (ml) |
|---------|---|-----------------|-------|----------------------------------|------------------------------|
| | | Initial | Final | | |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

Standardization of $KMnO_4$ solution:

N_1 = Normality of known oxalic acid solution = _____

V_1 = Volume of oxalic acid solution = _____

N_2 = Normality of $KMnO_4$ solution = (unknown)

V_2 = Volume of $KMnO_4$ solution (burette reading) = _____

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{N_1 V_1}{V_2} = \text{_____} N$$

Date:

Estimation of unknown FAS solution:

| Sr. No. | Volume of FAS solution taken (ml) | Burette reading | | Volume of $KMnO_4$ consumed (ml) | Average burette reading (ml) |
|---------|-----------------------------------|-----------------|-------|----------------------------------|------------------------------|
| | | Initial | Final | | |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

Estimation of Ferrous ammonium sulphate:

N_3 = Normality of unknown FAS solution = (unknown)

V_3 = Volume of FAS solution = _____

N_4 = Normality of $KMnO_4$ solution = _____

V_4 = Volume of $KMnO_4$ solution = _____

$$N_3 V_3 = N_4 V_4$$

$$N_3 = \frac{N_4 V_4}{V_3} = \text{_____} N$$

Strength = $N_3 \times \text{Equivalent weight of FAS g/L}$

$$= N_3 \times 392 \text{ g/L}$$

$$= \text{_____} \text{ g/L}$$

Note: The equivalent weight of FAS = 392

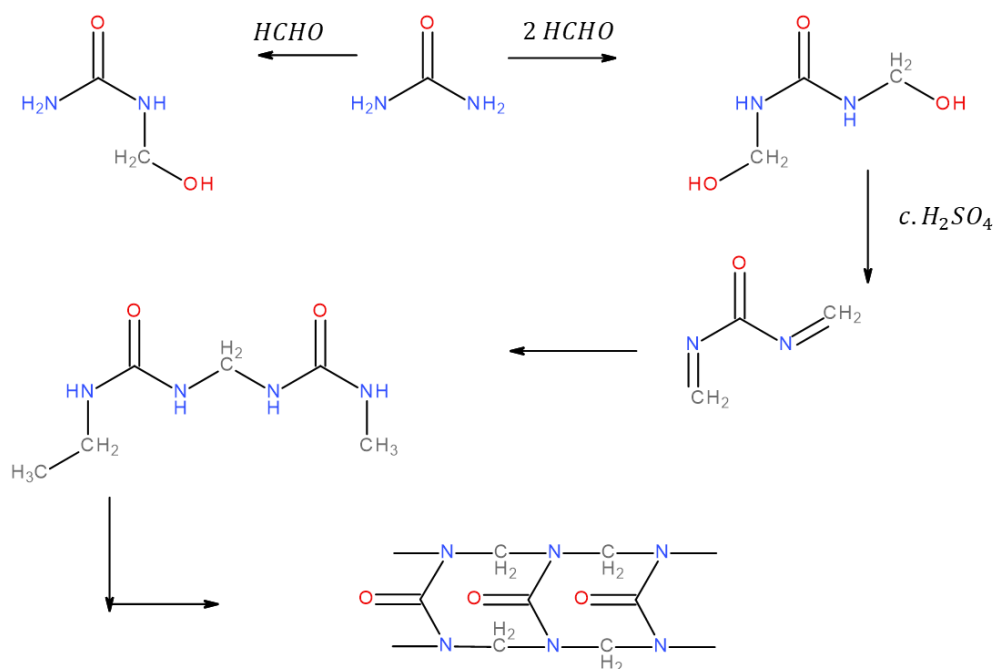
Result:

The strength of given unknown ferrous ammonium sulphate solution = _____ g/L

EXPERIMENT 6: Synthesis of urea formaldehyde resin.

Principle: Amino resins are obtained by condensation reaction of urea or melamine with formaldehyde. Such resins find uses in packaging, water tumblers, unbreakable dishes, buttons etc. They are also used in paper industry to improve the strength of paper.

Urea formaldehyde resin is formed by a condensation reaction. Urea and formaldehyde are mixed to form the precursor molecule and concentrated H_2SO_4 is added to remove water and grow a three-dimensional network structure. The reaction is as follows:



Reagents: formaldehyde (40%), urea, conc. H_2SO_4 , distilled water.

Apparatus: 100 ml beaker, measuring cylinder, glass rod, watch glass, funnel, filter paper

Procedure:

1. Take 2 gm of urea in a 100 ml beaker and add 5 ml of 40% formaldehyde solution in it with constant stirring.
2. Add 2/3 drops of conc. H_2SO_4 to the reaction mixture with constant stirring.
3. White voluminous mass appears in the beaker.
4. When the reaction is complete, wash the residue with distilled water, and filter it.
5. Dry the product in between folds of filter paper and put in a hot air oven.
6. Calculate the yield of the resin formed.

Date:



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Observations:

- Weight of empty watch glass = $W_1 = \text{_____}g$
- Weight of watch glass with product = $W_2 = \text{_____}g$
- Weight of the product = $(W_2 - W_1) = \text{_____}g$

EXPERIMENT 7: pH-metric titration of a strong acid (HCl) with a strong base ($NaOH$)

Objective: To determine the strength of given HCl solution using a standard $NaOH$ solution by performing a pH-metric titration.

Principle: The negative logarithm (to the base of 10) of the hydrogen ion concentration of the given solution is defined as the pH of the solution.

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

Similarly, the alkalinity of a solution can be expressed in terms of pOH, where

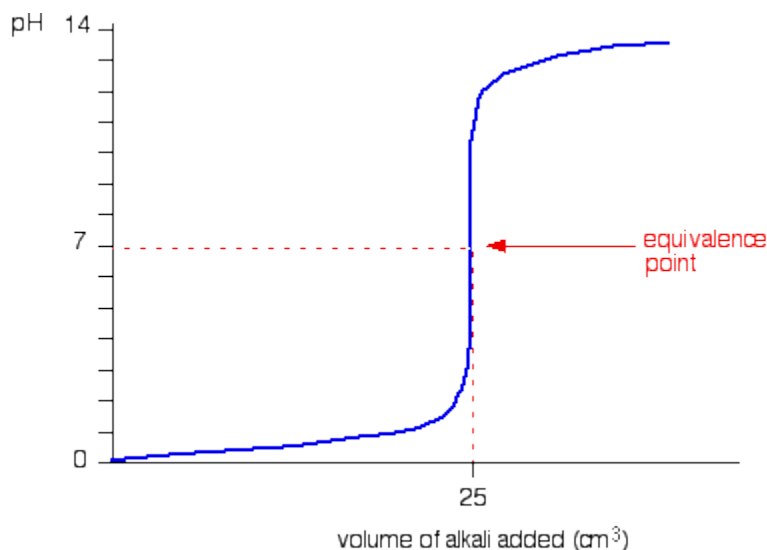
$$pOH = -\log [OH^-]$$

For pure or neutral solution: $pH = 7$ i.e., $[H^+] = [OH^-] = 1 \times 10^{-7}$ g/lit of ions.

For an acidic solution: $pH < 7$ i.e., $[H^+] > 1 \times 10^{-7}$ g/lit

For a basic solution: $pH > 7$ i.e., $[H^+] < 1 \times 10^{-7}$ g/lit

In this experiment, the pH of a solution is utilized as an indicator for determining the endpoint of a strong acid-strong base titration. The base solution is standard while the concentration of acid is unknown. A fixed quantity of solution of strong acid is taken in a beaker and its initial pH is recorded using a pH meter. To this, if we start adding a strong base solution, we find that the pH of the reaction mixture follows a graph of the sigmoid shape:



When a base is added to an acid, initially the pH rises slowly, and then it changes rapidly near the end point. Ionization of the base is responsible for the shape of the inflexion point and symmetry of the curve. If we consider the volume of alkali added corresponding to the value of pH 7, we can find the end point.

Date:

Procedure:

1. Clean and rinse all the equipment and apparatus.
2. Calibrate the pH meter using the supplied buffer solution.
3. Fill the burette with NaOH solution.
4. Take out 50 ml of given HCl solution into a 100 ml conical flask.
5. Dip the pH electrode into the beaker and measure the initial pH of the acid solution.
6. From the burette, start adding the NaOH solution in 0.2 ml increments with constant stirring. Note down the pH after each increment.
7. Continue Step 6 until the pH goes above 12.
8. Plot a graph between the observed pH value (y axis) and the volume of NaOH solution added (x axis).
9. The end point in this reaction comes as $\text{pH} = 7$. Check the volume of NaOH added at $\text{pH} = 7$ from the graph.
10. Calculate the strength using the data obtained.

Observation:

| S. No. | Volume of NaOH added | pH |
|--------|----------------------|----|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |
| 6 | | |
| 7 | | |
| 8 | | |
| 9 | | |
| 10 | | |
| 11 | | |
| 12 | | |
| 13 | | |
| 14 | | |
| 15 | | |

| S. No. | Volume of NaOH added | pH |
|--------|----------------------|----|
| 16 | | |
| 17 | | |
| 18 | | |
| 19 | | |
| 20 | | |
| 21 | | |
| 22 | | |
| 23 | | |
| 24 | | |
| 25 | | |
| 26 | | |
| 27 | | |
| 28 | | |
| 29 | | |
| 30 | | |

Date:

Calculations:

$$N_1 V_1 = N_2 V_2$$

N_1 = Normality of acid (unknown), V_1 = Volume of acid (pipette reading)

N_2 = Normality of NaOH (known), V_2 = Volume of NaOH added (from the graph)

$$N_1 = \frac{N_2 V_2}{V_1} = \text{_____} N$$

Strength = $N_1 \times$ Equivalent weight of HCl

$$= \text{_____} \times 36.5 \text{ g/L} = \text{_____} \text{ g/L}$$

Result:

The strength of the given HCl solution = _____ g/L

EXPERIMENT 8: Conductometric titration of HCl against NaOH

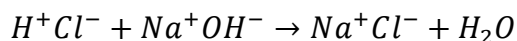
Objective: To determine the strength of a given HCl solution using a standard NaOH solution by performing a conductometric titration.

Principle: The specific electrical conductivity and the electrical conductance are measures of the ability of a solution to conduct electrical current. In solutions, the current is carried by cations and anions

“Conductance is the reciprocal of the resistance” or $C = \frac{1}{R}$. The unit of R is ohm and the unit of C is ohm^{-1} or mhos or siemens. Electrolytic conductors obey ohm’s law which states $I = \frac{E}{R}$ where I = current, E = potential difference between electrode, and R = resistance

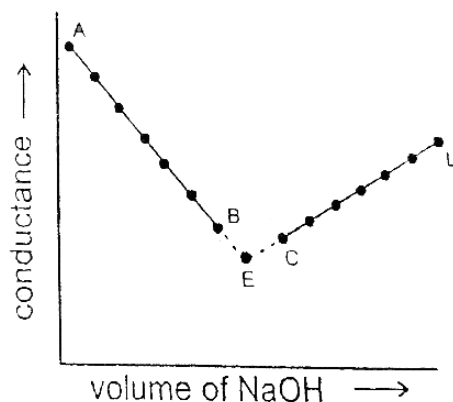
In this experiment, the conductivity of a solution is utilized as an indicator for determining the endpoint of a strong acid-strong base titration. The base solution is standard while the concentration of acid is unknown.

A fixed quantity of the solution of a strong acid (HCl) is taken in a beaker and its initial conductivity is recorded. Being a strong electrolyte, the conductivity value will be large (due to the presence of the fast H^+ ions). To this, if we start adding a strong base solution, we find that the value of conductivity falls linearly (as H^+ ions combine with OH^- ions and form a non-conducting molecule – water).



As soon as the equivalence point is reached, the added ions of the strong base remain free in solution and hence, beyond this point, further addition of base leads to a sharp rise in the conductivity of the solution (because of the increase of the OH^- ions).

To determine the end point, the observed conductivity of the solution is plotted against the volume of base added. Conductivity values follow two distinct linear trends before and after the equivalence point, as can be seen in the following schematic diagram:



Procedure:

1. Calibrate the conductivity meter.
2. Wash all the glass wares with distilled water.
3. Fill the standard NaOH (0.2N) solution into the burette and note the initial volume level in the burette.
4. Take 50 ml of given unknown HCl solution in 250 ml beaker.
5. Dip the conductivity cell into the solution of HCl acid and note the initial conductance of HCl solution.
6. From the burette, start adding the strong base solution in 0.2 ml increments.
7. Shake the solution properly and note down the conductivity after each increment.
8. Continue with the previous step till you reach a minimum value, and then the value increases up to the value you started with (before adding any base).
9. Plot a graph between volumes of NaOH added verses observed conductivity value. Locate the end point as the intersection of the two lines (point E in the figure).

Observation:

| S. No. | Volume of NaOH added | Conductance |
|--------|----------------------|-------------|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |
| 6 | | |
| 7 | | |
| 8 | | |
| 9 | | |
| 10 | | |
| 11 | | |
| 12 | | |
| 13 | | |
| 14 | | |
| 15 | | |

| S. No. | Volume of NaOH added | Conductance |
|--------|----------------------|-------------|
| 16 | | |
| 17 | | |
| 18 | | |
| 19 | | |
| 20 | | |
| 21 | | |
| 22 | | |
| 23 | | |
| 24 | | |
| 25 | | |
| 26 | | |
| 27 | | |
| 28 | | |
| 29 | | |
| 30 | | |

Date:

Calculations:

$$N_1 V_1 = N_2 V_2$$

N_1 = Normality of acid (unknown), V_1 = Volume of acid (pipette reading)

N_2 = Normality of NaOH (known), V_2 = Volume of NaOH added (from the graph)

$$N_1 = \frac{N_2 V_2}{V_1} = \text{_____} N$$

Strength = $N_1 \times$ Equivalent weight of HCl

$$= \text{_____} \times 36.5 \text{ g/L} = \text{_____} \text{ g/L}$$

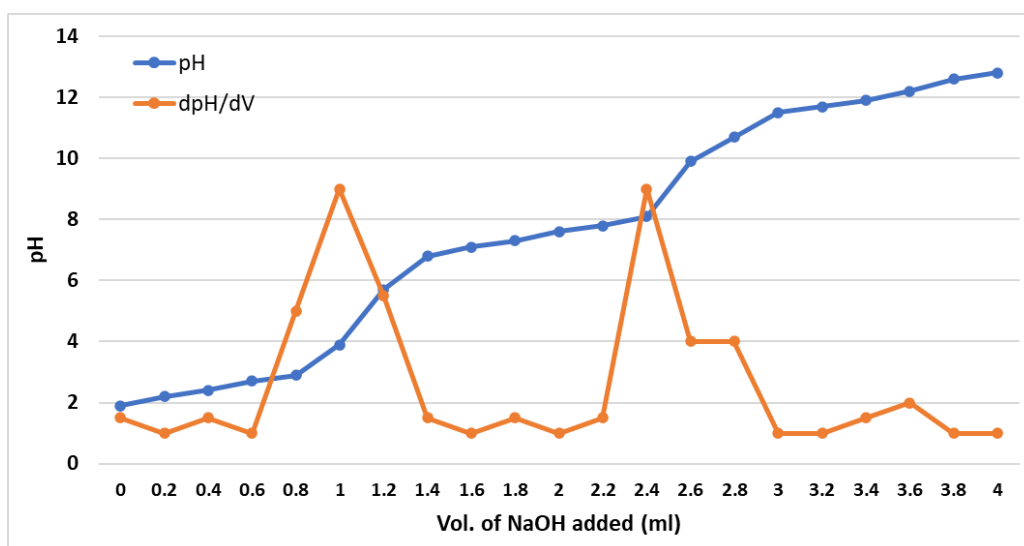
Result:

The strength of the given HCl solution = _____ g/L

Experiment 09: pH-metric titration of a weak acid (CH_3COOH) with a strong base (NaOH)

Objective: To determine the strength of given HCl solution using a standard NaOH solution by performing a pH-metric titration.

Theory: The basic theory of this experiment is similar to that of Expt. 7. However, in this case, we cannot interpolate the end point by referencing the data for $\text{pH} = 7$ as the titration happens at a pH slightly higher than 7. To solve this problem, we draw a $\frac{dpH}{dV}$ plot along with the pH plot. This graph indicates the change of pH with addition of base. As at the endpoint, there will be huge change in pH, which will be captured in this graph. We'll use the peak position to get our required burette reading.



Procedure: -

- Calibrate the pH meter first with the provided buffer solution.
- Wash the electrode by distilled water properly and wipe it with tissue paper,
- Fill the burette with standard NaOH solution.
- Take 50 ml of unknown acid solution in a 100 ml beaker and dip the electrode in it. Note down the reading displayed on the pH meter.
- Now add 0.2 ml standard NaOH solution from the burette to the unknown solution and note the pH after each addition.
- Plot two graphs: i) between pH value and the volume of NaOH, and ii) between $\frac{dpH}{dV}$ and the volume of NaOH to find out the equivalence point.

Date:

Observation:

| S. No. | Volume of NaOH added | pH | $\frac{d\text{ pH}}{dV}$ |
|--------|----------------------|----|--------------------------|
| 1 | | | |
| 2 | | | |
| 3 | | | |
| 4 | | | |
| 5 | | | |
| 6 | | | |
| 7 | | | |
| 8 | | | |
| 9 | | | |
| 10 | | | |
| 11 | | | |
| 12 | | | |
| 13 | | | |
| 14 | | | |
| 15 | | | |

| S. No. | Volume of NaOH added | pH | $\frac{d\text{ pH}}{dV}$ |
|--------|----------------------|----|--------------------------|
| 16 | | | |
| 17 | | | |
| 18 | | | |
| 19 | | | |
| 20 | | | |
| 21 | | | |
| 22 | | | |
| 23 | | | |
| 24 | | | |
| 25 | | | |
| 26 | | | |
| 27 | | | |
| 28 | | | |
| 29 | | | |
| 30 | | | |

Calculations:

$$N_1V_1 = N_2V_2$$

N_1 = Normality of acid (unknown), V_1 = Volume of acid (pipette reading)

N_2 = Normality of NaOH (known), V_2 = Volume of NaOH added (from the graph)

$$N_1 = \frac{N_2V_2}{V_1} = \text{_____} N$$

Strength = $N_1 \times$ Equivalent weight of HCl

$$= \text{_____} \times 36.5 \text{ g/L} = \text{_____} \text{ g/L}$$

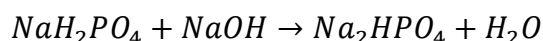
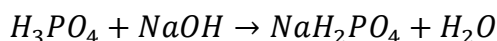
Result:

The strength of the given HCl solution = _____ g/L

Experiment No.10: To determine the pK_a values of phosphoric acid

Objective: To determine the pK_1 and pK_2 values of a polyprotic acid (phosphoric acid).

Theory: Neutralization of phosphoric acid takes place in three steps -



In the titration curve as shown in the Figure 1 below three inflections will be observed. First and second inflection points will be clearly seen but the third will not be observed so clearly. The solution remains acidic after the first neutralization step but owing to the salt hydrolysis, it becomes mildly alkaline after the second neutralization step and strongly alkaline after the third neutralization step.

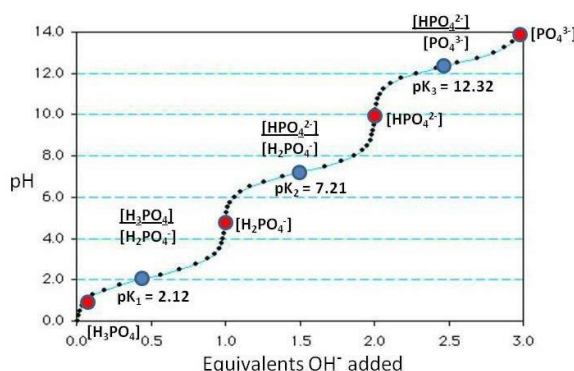


Figure 1

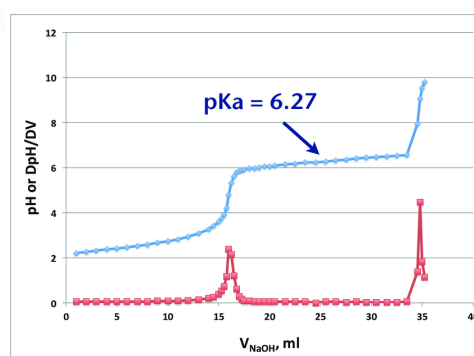


Figure 2

Given the sensitivity of the experiment, the pH vs. v_{NaOH} graph will not be enough to point out the inflection points and hence, we have to draw a $\frac{d(pH)}{dv}$ vs. v graph. This graph indicates the change in pH with the addition of $NaOH$. If the readings are represented as $pH_1, pH_2, pH_3 \dots$ then the $\frac{d(pH)}{dv}$ value can be calculated as $\frac{pH_2 - pH_1}{\text{Volume of NaOH added}}$. The plot will look like the Figure 2 shown above.

The *Henderson – Hasselbach equation* provides the relationship between pH and pK_a value for an acid, HA as

$$pH = pK_a + \log \frac{[A^-]}{[HA]}$$

This indicates that at the point $pH = pK_a$ the concentrations of the acid and the conjugate base form are equal. We would use this fact to determine the pK_1 and pK_2 values.

Apparatus and Reagents required: pH meter with electrodes, 100 ml beaker, burette, pipette, phosphoric acid solution, 0.2N NaOH solution, buffer solution.

Procedure:

1. Set the switch on pH mode in the pH meter and set the temperature at room temperature.
2. Calibrate the pH meter against the supplied buffer solution.
3. Fill the burette with 0.2N NaOH.
4. Take 50 ml of phosphoric acid (of unknown strength) in 100 ml beaker and immerse the electrode in the solution and note down the reading (reading 1).
5. Add NaOH solution in batch of 0.2 ml into the acid solution with constant stirring and note the pH meter reading.
6. pH will appreciably change near the neutralization point as, small amount of NaOH added.
7. Continue the procedure till the pH of the solution is 12.

Observation Table:

| S. No. | Volume of NaOH added | pH | $\frac{d pH}{dV}$ |
|--------|----------------------|----|-------------------|
| 1 | | | |
| 2 | | | |
| 3 | | | |
| 4 | | | |
| 5 | | | |
| 6 | | | |
| 7 | | | |
| 8 | | | |
| 9 | | | |
| 10 | | | |
| 11 | | | |
| 12 | | | |
| 13 | | | |
| 14 | | | |
| 15 | | | |

| S. No. | Volume of NaOH added | pH | $\frac{d pH}{dV}$ |
|--------|----------------------|----|-------------------|
| 16 | | | |
| 17 | | | |
| 18 | | | |
| 19 | | | |
| 20 | | | |
| 21 | | | |
| 22 | | | |
| 23 | | | |
| 24 | | | |
| 25 | | | |
| 26 | | | |
| 27 | | | |
| 28 | | | |
| 29 | | | |
| 30 | | | |

Date:

Calculation:

Let's suppose the first and the second inflection points occur at volumes V_1 and V_2 , respectively.

$$pK_1 \text{ of } H_3PO_4 \text{ is } = \frac{V_1}{2} =$$

$$pK_2 \text{ of } H_3PO_4 \text{ is } = \frac{V_2 - V_1}{2} =$$

Result:

The pK_1 and pK_2 values of phosphoric acid are _____ and _____ .

In the titration curve, three inflections are observed. It verifies all the three steps in the neutralization of phosphoric acid i.e., ortho phosphoric acid is a tribasic acid.

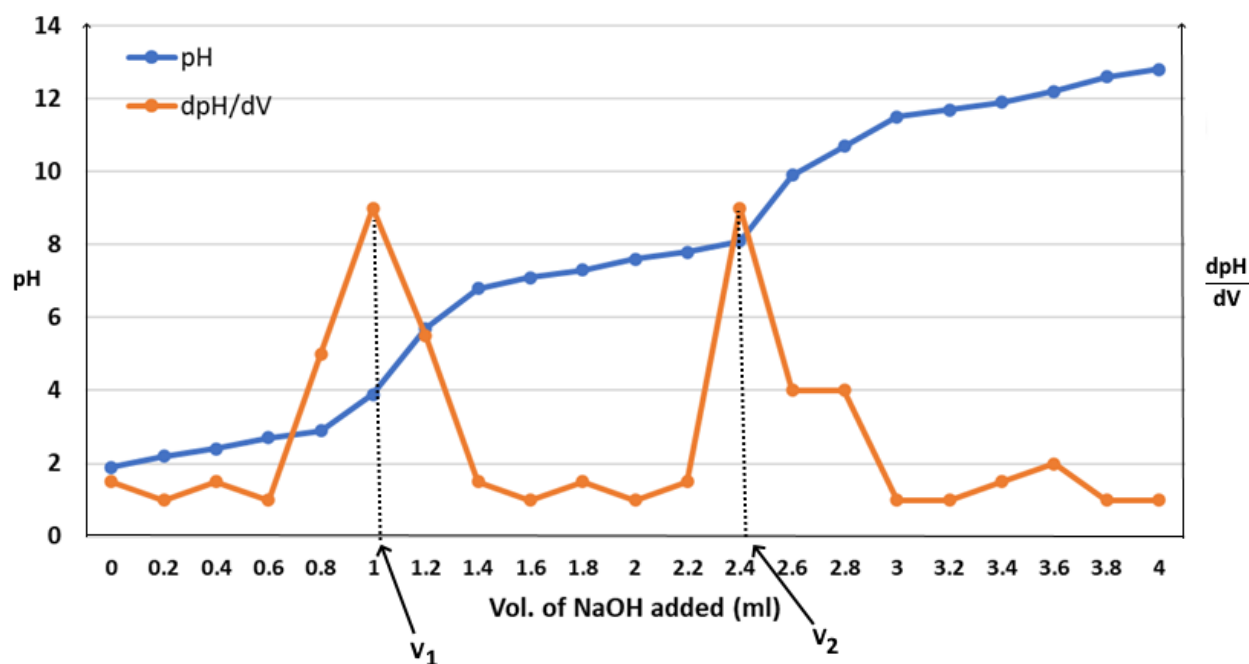
Experiment No.11

Objective: pH-metric titration of a mixture of a strong acid (HCl) and a weak acid (CH_3COOH).

Theory: The basic theory of this experiment is similar to that of Expt. 7. However, in this case, we cannot interpolate the end point by referencing the data for $pH = 7$ as there is a mixture of acids involved.

CH_3COOH , being a weak acid, does not dissociate if there is a strong acid present in the mixture. Hence, when we titrate the acid mixture with $NaOH$, the strong acid – HCl gets titrated first and once it is completely titrated, the weak acid – CH_3COOH gets titrated.

We draw a $\frac{dpH}{dV}$ plot along with the pH plot. This graph indicates the change of pH with addition of the base. As at the endpoints, there will be large change in pH, which will be captured in the graph in the form of two peaks in the value of $\frac{dpH}{dV}$. The volume v_1 corresponds to $NaOH$ and the volume $(v_2 - v_1)$ corresponds to CH_3COOH .



Procedure: -

- i. Calibrate the pH meter first with the provided buffer solution.
- ii. Wash the electrode by distilled water properly and wipe it dry with a paper towel.
- iii. Fill the burette with standard $NaOH$ solution.

Date:

- iv. Take 50 ml of unknown acid solution in a 100 ml beaker and dip the electrode in it. Note down the reading displayed on the pH meter.
- v. Now add 0.2 ml standard NaOH solution from the burette to the unknown solution and note the pH after each addition.
- vi. Plot two graphs: i) between pH value and the volume of NaOH , and ii) between $\frac{dpH}{dV}$ and the volume of NaOH to find out the equivalence points.

Observation:

| S. No. | Volume of NaOH added | pH | $\frac{d pH}{dV}$ |
|--------|-------------------------------|----|-------------------|
| 1 | | | |
| 2 | | | |
| 3 | | | |
| 4 | | | |
| 5 | | | |
| 6 | | | |
| 7 | | | |
| 8 | | | |
| 9 | | | |
| 10 | | | |
| 11 | | | |
| 12 | | | |
| 13 | | | |
| 14 | | | |
| 15 | | | |

| S. No. | Volume of NaOH added | pH | $\frac{d pH}{dV}$ |
|--------|-------------------------------|----|-------------------|
| 16 | | | |
| 17 | | | |
| 18 | | | |
| 19 | | | |
| 20 | | | |
| 21 | | | |
| 22 | | | |
| 23 | | | |
| 24 | | | |
| 25 | | | |
| 26 | | | |
| 27 | | | |
| 28 | | | |
| 29 | | | |
| 30 | | | |

Calculations:

$$N_1V_1 = N_2V_2$$

N_1 = Normality of acid (unknown), V_1 = Volume of acid (pipette reading)

N_2 = Normality of NaOH (known), V_2 = Volume of NaOH added (from the graph)

Date:

Strength of HCl : [the volume corresponding to the base is (v_1) from the graph]

$$N_1 = \frac{N_2 V_2}{V_1} = \text{_____} N$$

Strength = $N_1 \times$ Equivalent weight of HCl

$$= \text{_____} \times 36.5 \text{ g/L} = \text{_____} \text{ g/L}$$

Strength of CH_3COOH : [the volume corresponding to the base is ($v_2 - v_1$) from the graph]

$$N_1 = \frac{N_2 V_2}{V_1} = \text{_____} N$$

Strength = $N_1 \times$ Equivalent weight of CH_3COOH

$$= \text{_____} \times 60 \text{ g/L} = \text{_____} \text{ g/L}$$

Result:

The strength of the given HCl solution = _____ g/L

The strength of the given CH_3COOH solution = _____ g/L

EXPERIMENT 12: To determine the equivalent conductance of an electrolyte

Objective: To determine the equivalent conductance of an electrolyte.

Theory: - Equivalent conductance is the power of all the ions produced by one gm of electrolyte. Equivalent conductance of an electrolyte at any concentration and represented by λ_c .

$$\Lambda_c = k \cdot V = 1000k / C \text{ ohm}^{-1}\text{cm}^2\text{gm equiv}^{-1}$$

Where k = specific conductivity; V = volume in which one gm. of equivalent is present.

Specific conductance $k = l/a \times \text{observed conductance}$ (Where l/a = cell constant)

Cell constant = specific conductance / observed conductance

Required apparatus and reagents: Conductivity meter with electrode, wash bottle, beakers, electrolyte solutions.

Procedure:

1. Connections are made according to the manual of the instrument.
2. Prepare 0.1N/0.01N KCl solution by dissolving an accurate amount of pure and dry KCl in conductivity water.
3. Wash the conductivity cell with distilled water and rinse the conductivity cell with KCl solution.
4. When equilibrium temperature has been attained, measure the conductivity. Wait for some time and again measure the conductivity of the solution
5. Prepare the stock solution of 0.1 N of electrolyte (HCl) and determine the equivalent conductivity by quantitative dilution of the solutions of 0.05, 0.02, 0.01, 0.001, 0.002 and 0.005N are prepared from stock solution.
6. Now wash the electrode with distilled water and rinse it with 0.1 N KCl solution, dip the conductivity cell in to 0.1 N HCl solution and measure the conductivity of the solution.
7. Now measure the conductivity of the other solutions in similar way, care should be taken that every time cell must be washed and rinsed properly with respective solutions.
8. The conductivity cell should be washed and kept in to the distilled water after each reading.

Date:

Observations:

| Strength of <i>KCl</i> solution (N) | Conductivity | | Cell constant = specific Conductance /observed conductance |
|-------------------------------------|------------------------------------|----------------------|--|
| | Specific conductance of <i>KCl</i> | Observed conductance | |
| 0.1 | 0.01289 | | |
| 0.01 | 0.001412 | | |

| Strength of solution (N) | Observed conductance | Specific conductance k = cell constant \times observed conductance | Equivalent conductance = $\Lambda_c = 1000 k / C$ |
|--------------------------|----------------------|--|---|
| 0.05 | | | |
| 0.02 | | | |
| 0.01 | | | |
| 0.005 | | | |
| 0.002 | | | |

Calculations:

1. $\lambda_c = 1000 k/0.1$
2. $\lambda_c = 1000 k/0.05$
3. $\lambda_c = 1000 k/0.02$
4. $\lambda_c = 1000 k/0.01$
5. $\lambda_c = 1000 k/0.002$
6. $\lambda_c = 1000 k/0.005$

Result:

1. The cell constant = _____/ cm
2. Equivalent conductivity for different solutions = _____mhos

EXPERIMENT 13: Determination of viscosity using the Redwood viscometer

Objective: To determine the viscosity of a given lubricating oil at various temperatures using Redwood viscometer.

Principle: Viscosity is a measure of the internal resistance to the motion of a fluid and is mainly due to the forces of cohesion between the fluid molecules.

The **absolute Viscosity** is defined as “the tangential force per unit area required to maintain unit velocity gradient between two parallel planes in the fluid at a unit distance apart. It is denoted by η (eta). Its unit in CGS system is *poise*. 1 *poise* is equal in one dyne/second/cm² (dimensions of absolute viscosity are ML⁻¹T⁻¹).

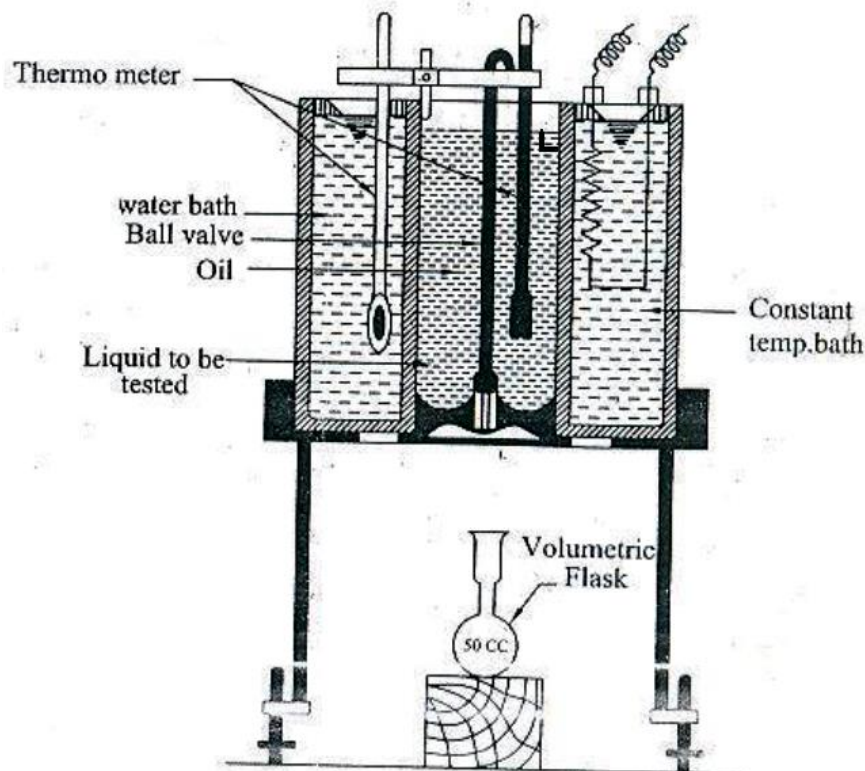
The ratio of absolute viscosity to density for any fluid is known as its **absolute kinematic viscosity**.

Viscosity generally decreases with the increase in temperature. The maintenance of viscosity over the range of temperature is called the *Viscosity Index*. A relatively small change/no change in viscosity with temperature is indicated by high viscosity index whereas low viscosity index shows relatively large change in viscosity with temperature.

The instrument used for measuring the viscosity is known as viscometers. Different types of viscometers are: Saybolt Viscometer, Angler's Viscometer, Ostwald Viscometer, Kinematic Viscometer, Redwood Viscometer etc.

In this experiment, a **Redwood Viscometer** is **used**. It is of two types: Redwood viscometer No.1 and Redwood viscometer No. 2. Both the above viscometers are identical in principle, shape, and mode of operation. The essential differences between the two are:

| | Redwood viscometer No.1 | Redwood viscometer No.2 |
|------------------------------|--|---|
| Dimensions of orifice | Length = 10 mm Diameter = 1.62 mm | Length = 50 mm Diameter = 3.8 mm |
| Kohlrausch flask | Smaller mouth | Wider mouth |
| Useful for | Low viscous oil having flow time between 30s-2000s e.g., Kerosine oil and mustard oil | Higher viscous oils having flow time greater than 2000s e.g., Fuel oil, mobile oil |



The **Redwood viscometer** consists of three parts

1. Oil Cup: It holds the test sample of lubricating oil. The bottom of the cup is fitted with polished-agate discharge tube containing an orifice of specified dimension
2. Water Bath: Oil cup is surrounded by water bath for adjusting the temperature
3. Kohlrausch Flask: It receives the oil from polished-agate discharge tube

Reagents and apparatus: Redwood Viscometer, Thermometer 0-100°C (2 Nos), stopwatch, 50 ml standard narrow necked volumetric flask or Kohlraush's flask, given sample of oil.

Procedure:

1. Select the Redwood viscometer according to the nature of oil (RW₁ for light oil and RW₂ for heavy oil).
2. Level the viscometer using the leveling screws.
3. Clean the cylindrical oil cup and ensure the orifice tube is free from dirt.
4. Seal the orifice using the brass ball and put the receiver (Kohlraush's flask 50 ml) under the jet to collect the pouring oil.
5. Fill the bath with water or oil according to the temperature of the experiment.
6. Maintain the desired temperature and stir the water/oil and the oil in the bath and cup respectively.
7. At a specific temperature lift the ball and simultaneously start the stopwatch.

8. Collect the oil in the 50 ml flask and note the time taken in seconds for the collecting 50 ml of oil.
9. Repeat the whole process at five different temperature and note respective times.

Observations:

| S. No. | Temperature (°C) | Time t (sec) | | | | Viscosity (mean) |
|--------|------------------|--------------|---------|---------|---------|------------------|
| | | Trial 1 | Trial 2 | Trial 3 | Average | |
| 1 | | | | | | |
| 2 | | | | | | |
| 3 | | | | | | |
| 4 | | | | | | |
| 5 | | | | | | |

Calculations:

The ratio of 'absolute viscosity' to the 'density of any fluid' is known as its 'absolute kinematic viscosity'. Because the instruments used are of standard dimensions, the kinematic viscosity of the oil in centistokes can be calculated from the time taken by the oil to flow through the standard orifice of the instrument with the help of the following equation.

The viscosity of the given oil sample is:

$$\eta = At - \frac{B}{t}$$

Where t is the time taken and A & B are instrument constants.

$A = 0.264$ and $B = 190$ for $t = 40-85$ seconds

$A = 0.247$ and $B = 65$ for $t = 85-2000$ seconds

Result:

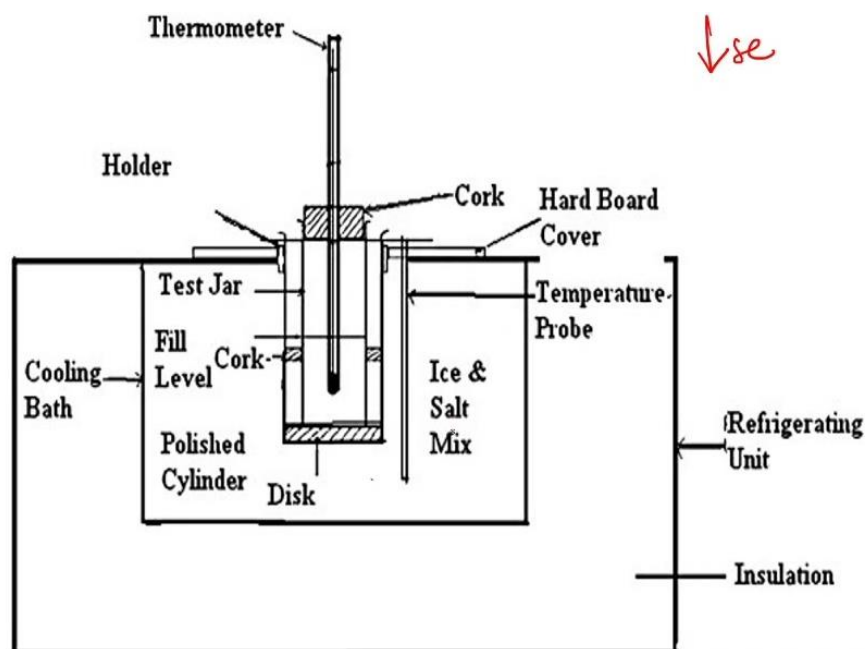
The kinematic viscosity of given oil at different temperatures were determined.

EXPERIMENT 14: Determination of the cloud point and pour point of an oil

Objective: To determine the cloud and pour point of a given sample of lubricating oil using the “cloud and pour point apparatus”.

Principle: Cloud point and pour point are two important physical properties of any liquid. Cloud point, as the name suggests, is the temperature at which a cloud of wax crystals first appears in a liquid fuel when it is cooled under special testing conditions. The cloud point of any petroleum product is an indicator of how well the fuel will perform under cold weather conditions.

Pour point is the opposite of cloud point as it refers to the lowest temperature at which movement of oil is observed and the fuel can be pumped easily.



The cloud point and pour point apparatus consists of the following parts:

- i) Test tube: it is made of glass having flat bottom and standard dimensions.
- ii) Cooling bath: It consists of an insulated jacket of glass or copper containing cooling mixture.
- iii) Thermometers: In given sample and cooling mixture, there are two thermometers fitted.

Reagents and apparatus: lubricating oil, freezing mixtures, cloud and pour point apparatus

Procedure:

1. Thoroughly dry and clean the flat-bottomed glass tube.
2. Fill the oil sample to form a film of 5 cm thickness and fit the cork.
3. Adjust the dipped bulb of thermometer in the oil and in the cooling bath properly.
4. The tube is kept in the freezing mixture.
5. Prepare freezing mixture as mentioned below:

| Mixture | Composition | Temperature range |
|---------|---------------------------------|-------------------|
| A | Ice + water | Upto 10 °C |
| B | Ice + NaCl | Upto -10 °C |
| C | Ice + CaCl ₂ | Upto -25 °C |
| D | Solid CO ₂ + Acetone | Upto -60 °C |

6. The temperature of oil falls on cooling (when cooling bath filled with freezing mixture).
7. Take out the tube after every 1 °C fall in temperature and inspect for a moment. It may restore if the cloudiness has not formed. This step should be completed in 3 Seconds.
8. For cloud point, note the temperature for hazy appearance.
9. And for pour point, continue the cooling process. After every 3 °C fall, the tube is withdrawn and tilted to horizontal position for about 5-10 seconds. When oil stops flowing out of the jar, note the temperature. It is the pour point for the given sample of lubricating oil.

Result:

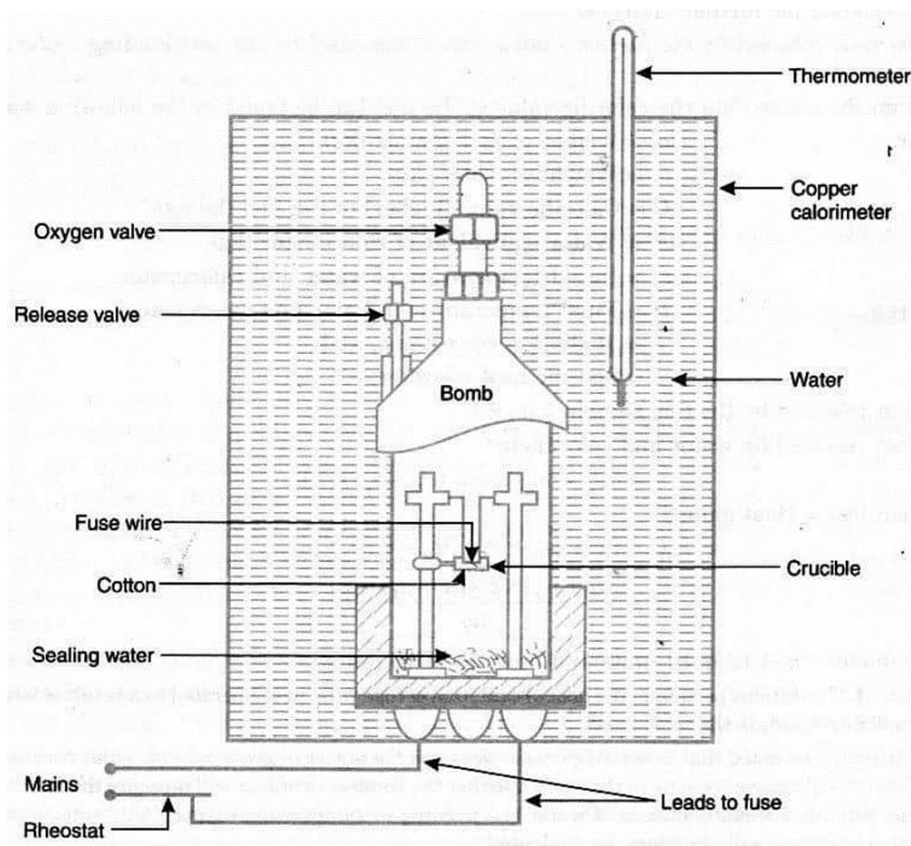
The cloud point of the given sample is _____°C and

The pour point of the given sample is _____°C.

EXPERIMENT 15: To determine the water equivalent of a bomb calorimeter

Objective: To determine the water equivalent of a bomb calorimeter.

Principle: A bomb calorimeter is used for determining the calorific value of solid and liquid fuels.



The bomb calorimeter consists of a strong stainless-steel vessel, called bomb, capable of withstanding high pressure. The bomb is provided with a lid, which can be screwed firmly on the bomb to make a perfect airtight seal. The lid contains inlet for two electrodes and an oxygen inlet valve. One of the electrodes is provided with a ring to accommodate the silica crucible.

The bomb is placed in a calorimeter which contains a known weight of water (W kg). The copper calorimeter in turn, is surrounded by an air jacket to prevent heat loss due to radiation. The calorimeter is also equipped with an electrical stirrer and a thermometer to record the initial (t_1) and final (t_2) temperature.

Once known amount of the fuel (m kg) is ignited in the presence of enough oxygen, complete combustion happens, and the generated heat is passed to the water in the calorimeter. If higher calorific value of the fuel is θ kcal/kg, the fuel generates $m\theta$ kcal of heat.

Date:

The produced heat is absorbed by the water and some heat is also absorbed by the calorimeter. The water equivalent of the calorimeter is w kg. According to the theory of calorimetry,

$$\text{heat generated} = \text{heat absorbed}$$

$$m\theta = (W + w)(t_2 - t_1)$$

The equation can be used to determine the water equivalent (w) of the bomb calorimeter if the HCV of the fuel is known.

Example: HCV of benzoic acid = 6325 kcal/kg and HCV of naphthalene = 9688 kcal/kg

Reagents and apparatus: given solid fuel sample, pellet maker, the bomb calorimeter with control hub.

Procedure:

1. The fuel is weighed, made into a pellet using the pellet-maker, and placed in the silica crucible.
2. A fine magnesium wire touching the fuel pellet is stretched across the electrodes.
3. The bomb is filled with oxygen till the pressure of oxygen inside the bomb reaches 25-30 atm.
4. The calorimeter is filled with measured amount of water (W).
5. Initial temperature of the water in the calorimeter is noted after thorough stirring (t_1).
6. The electrodes are then connected to a 6-volt battery and the circuit is complete.
7. The current is switched on and the fuel in the crucible burns. The heat produced is transferred to the water, which is stirred throughout the experiment with the help of the electric stirrer.
8. Maximum temperature shown by the Beckman's thermometer is recorded (t_2).

Observations:

$$\theta = \text{_____ kcal/kg}$$

$$m = \text{_____ kg}$$

$$W = \text{_____ kg}$$

$$t_1 = \text{_____ } ^\circ\text{C}$$

$$t_2 = \text{_____ } ^\circ\text{C}$$

Date:

Calculations:

$$m\theta = (W + w)(t_2 - t_1)$$

$$w = \frac{m\theta}{t_2 - t_1} - W = \frac{(\quad) \times (\quad)}{(\quad) - (\quad)} - (\quad) = \quad \text{kg}$$

Result:

Water equivalent of the bomb calorimeter is _____ kg

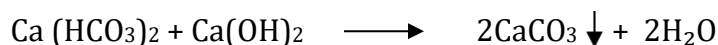
Date:

EXPERIMENT 4: Softening of hard water using Lime-soda process.

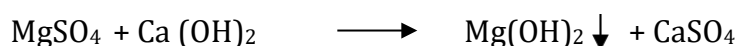
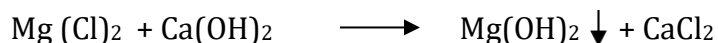
Objective: To estimate the amount of lime and soda required to soften the given hard water sample and to check for the removal of hardness in given sample.

Principle: The lime soda process involves the chemical conversion of all the soluble hardness causing salts by the addition of soda (Na_2CO_3) and lime ($\text{Ca}(\text{OH})_2$) into insoluble precipitates which can be easily removed by settling and filtration. As lime in the form of limewater is added to hard water, the pH of the water sample is raised and the equilibrium of carbonate species present in the water is shifted. Dissolved carbon dioxide (CO_2) is changed into bicarbonate (HCO_3^-) and then carbonate (CO_3^{2-}). This action causes calcium carbonate to precipitate because it exceeds the solubility product. Additionally, magnesium can be precipitated as magnesium hydroxide in a double displacement reaction. In the process both the calcium (and to an extent magnesium) in the hard water as well as the calcium added with the lime are precipitated. The chemical reactions involved in Lime-soda treatment are:

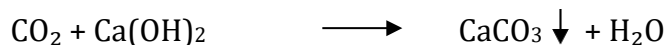
(i) Lime for the removal of temporary hardness:



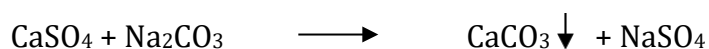
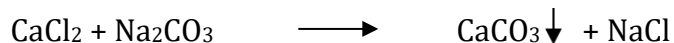
(ii) Lime for the removal of permanent magnesium hardness:



(iii) Lime for the removal of dissolved carbon dioxide:



(iv) Soda for the removal of complete calcium permanent hardness:



Procedure:

1. Wash all the apparatus with distilled water.
2. Prepare standard hard water solution by adding salts of calcium and magnesium in required amounts.
3. Calculate the amount of Lime and soda required to remove the hardness of water sample.

Date:

4. Add the calculated amount of Lime and soda.
5. Heat the mixture for 20 minutes to remove the hardness of water sample.
6. Let the mixture cool to room temperature and then filter the precipitates using Whatman filter paper.
7. Titrate the remaining solution against standard EDTA solution using EBT as indicator and check for the removal of hardness.

Observations and calculation:

Requirement of lime and soda for the treatment of given hard water sample, following calculations need to be done:

100 parts by mass of CaCO_3 are equivalent to:

- (i) 74 part of $\text{Ca}(\text{OH})_2$ and
- (ii) 106 parts of Na_2CO_3

Lime required for softening:

$$= \frac{74}{100} \left[\text{emp. Ca}^{2+} + 2 \times \text{Temp. Mg}^{2+} + \text{Perm. (Mg}^{2+} + \text{Fe}^{2+} + \text{Al}^{3+}) + \text{CO}_2 + \text{H}^+ (\text{HCl or H}_2\text{SO}_4) - \text{NaAlO}_2 + \text{HCO}_3^- \right]$$

Soda required for softening:

$$= \frac{106}{100} \left[\text{Perm. (Ca}^{2+} + \text{Mg}^{2+} + \text{Fe}^{2+} + \text{Al}^{3+}) + \text{H}^+ (\text{HCl or H}_2\text{SO}_4) - \text{HCO}_3^- \right]$$

Calculate the hardness of water sample before and after lime-soda treatment by complexometric titration using EDTA solution. If upon treatment with EDTA solution, the color changes from wine red to blue, it indicates that some extent of hardness persists in the sample, and if no color change is observed, it indicates that the hardness has been completely removed.

Hardness of given water sample before lime-soda treatment

| Sr. No. | Volume of hard water (ml) | Burette reading | | Volume of EDTA consumed (ml) | Average burette reading (ml) |
|---------|---------------------------|-----------------|-------|------------------------------|------------------------------|
| | | Initial | Final | | |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

Before Treatment hardness = $BR \times \frac{\text{molarity of EDTA} \times \text{MW of CaCO}_3(100) \times 1000}{\text{Volume of hard water}}$

Hardness of given water sample after lime-soda treatment

| Sr. No. | Volume of Treated water (ml) | Burette reading | | Volume of EDTA consumed (ml) | Average burette reading (ml) |
|---------|------------------------------|-----------------|-------|------------------------------|------------------------------|
| | | Initial | Final | | |
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |

After treatment hardness = $BR \times \frac{\text{molarity of EDTA} \times \text{MW of CaCO}_3(100) \times 1000}{\text{Volume of treated water.}}$

[Note: Given water contain 100 mg of CaCl₂ and 100 mg of Ca (NO₃)₂]

Result:

Lime required for the given water sample is -----mg/L

Soda required for the given water sample is-----mg/L

Hardness of given water sample before lime-soda treatment ----- mg/L

Hardness of given water sample after lime-soda treatment-----mg/L