**LIST OF EXPERIMENTS**

1. To determine Ca\(^{++}\) and Mg\(^{++}\) hardness of water using EDTA solution.

2. To determine alkalinity of given water sample.

3. To Prepare Phenol-formaldehyde (P-F) resin

4. To prepare Urea-formaldehyde (U-F) resin.

5. To find out saponification number of Oil

6. To determine strength of HCl solution by titrating against NaOH solution conductometerically.

7. To determine viscosity of lubricant by Red Wood Viscosity (No. 1 & No. 2).

8. To determine dissolved oxygen (D.O.) in the given water sample.

9. To determine of total iron in an ore solution by internal indicator method.

10. To determine TDS of Water samples of different sources.
DO’S

The Chemistry laboratory must be a safe place in which to work and learn about Chemistry. Most of these involve just using common sense.

1. Use protective clothing all the time (e.g. lab coat and safety glasses).
2. Be familiar with your lab assignment before you come to lab. Follow all written and verbal instructions carefully. Observe the safety alerts in the laboratory directions. If you do not understand a direction or part of a procedure, ask the teacher before proceeding.
3. Wash acid, base, or any chemical spill off of yourself immediately with large amounts of water. Notify your teacher of the spill.
4. Clean up spills immediately. If you spill a very reactive substance such as an acid or base, notify the people in the area and then obtain assistance from your teacher. Acid spills should be neutralized with baking soda, base spills with vinegar before cleaning them up.
5. If chemical substances get in your eye, wash the eye out for 15 minutes. Hold your eye open with your fingers while washing it out.
6. Place the reagents in a systemic manner.
7. If you burn yourself on a hot object, immediately hold the burned area under cold water for 15 minutes. Inform your teacher.
8. Observe good housekeeping practices. Work areas should be kept clean and tidy at all times. Only lab notebooks or lab handouts should be out on the table while performing an experiment. Books and book bags should not be on the lab table. Passageways need to be clear at all times.
9. Always add acid to water and stir the solution while adding the acid. Never add water to an acid.

10. Report all accidents to your teacher.

11. Thoroughly clean your laboratory work space at the end of the laboratory session. Make sure that all equipments are clean, and returned to its original place.

**DON’T**

1. Work in the laboratory without an instructor present. Work only with your lab partner(s). Do not venture to other lab stations for any reason.

2. Wear bulky or dangling clothing.

3. Eat or drink in the laboratory. Don’t chew on the end of a pen which was lying on the lab bench.

4. Use Mobile Phones.

5. Directly touch any chemical with your hands. Never taste materials in the laboratory.

6. Waste the reagents.

7. When entering the lab/classroom, do not touch any equipment, chemicals, or other materials without being instructed to do so. Perform only those experiments authorized by the instructor.

8. When weighing never place chemicals directly on the balance pan. **Never weigh a hot object.**

9. Smell anything in the laboratory unless your teacher tells you it is safe. Do not smell a substance by putting your nose directly over the container and inhaling.
Instead, waft the vapors toward your nose by gently fanning the vapors toward yourself.

10. Absolutely no running, practical jokes, or horseplay is allowed in the laboratory.

11. Allow the reagent bottles to accumulate on the bench.
Experiment No. 1

**Aim:** To determine the Ca\(^{++}\) and Mg\(^{++}\) hardness of given water sample using EDTA solution.

**Apparatus/reagent required:** Burette, beaker, conical flask, measuring cylinder, standard hard water, EDTA solution, ammonium buffer solution of \(\text{NH}_4\text{Cl + NH}_4\text{OH}\) (pH-10), calcium precipitating buffer solution of \(\text{NH}_4\text{Cl + NH}_4\text{OH + (NH}_4)_2\text{C}_2\text{O}_4\).

**Indicator:** Eriochrome black–T (EBT).

**IUPAC Name:** Sodium 1-(1-hydroxy-2-naphthylazo)-6-nitro-2-naphthol-4-sulphonate

**End point:** Colour changes from wine red to pure blue.

**Theory**

For finding the total hardness of water, a known volume of water sample, buffered to a pH around 10 with ammonical buffer solution of \(\text{NH}_4\text{Cl + NH}_4\text{OH}\), is titrated with standard EDTA solution using EBT as indicator.

EBro-T indicator is an organic dye having two phenolic ionisable hydrogen atoms (Fig. 1). It can have different forms of depending upon pH:

\[
\begin{align*}
H_2I_n^- & \quad \text{pH} > 6.3 \\
HI_n^{2-} & \quad \text{pH} > 11.5 \\
I_n^{3-} & \quad 
\end{align*}
\]

When indicator EBT is added to hard water at a pH around 10 maintained with ammonical buffer, it forms unstable wine red coloured complexes with bivalent metal ions of water as:
Ethylenediaminetetraacetic acid (EDTA) (Fig 2) is taken in the form of its disodium salt due to higher solubility.

For the simplicity, it can be represented as $H_2Y^2\text{-}$. It ionises in aqueous solution to give a strong chelating ion. It forms complexes with $Ca^{++}$ and $Mg^{++}$.

EDTA when added to wine red solution, it combines with free metal ions of hard water to form their respective soluble complexes. These are more stable than these are more stable than metal-indicator complexes.

When all free metal ions of hard water have complexed with EDTA, a slight excess of EDTA removes metal ions from weak metal indicator complexes to form stronger metal-complexes. This releases the indicator in free form which is blue in colour. This is end point of titration i.e. wine red to blue.

Reaction occurs according to scheme 1.
After determination of total hardness in water sample, Ca\(^{2+}\) ions in hard water are precipitated as calcium oxalate by adding calcium precipitation solution \(\text{i.e.} \, \text{NH}_4\text{Cl} + \text{NH}_4\text{OH} + (\text{NH}_4)_2\text{C}_2\text{O}_4\). The solution is filtered to remove white ppt. Titration of filtrate against standard EDTA solution solution using EBT indicator gives Mg\(^{2+}\) hardness in water sample. Hardness due to Ca\(^{2+}\) is obtained by subtracting Mg\(^{2+}\) hardness from total hardness.

**Procedure**

**Step 1. Standardization of EDTA solution**

Fill burette with EDTA solution. Pipette out 20 mL of Standard Hard Water (SHW) in to a conical flask. To this add 5 mL ammonium buffer solution. On addition of 3-4 drops of Erio-T indicator to this wine red colour will appear. Titrate wine red colour solution against EDTA solution till the end point \(\text{i.e.} \) colour change from wine red to blue. Repeat for taking concordant readings. Let \(V_1\) mL of EDTA solution be used.
Step 2. Total hardness in water samples

Titrate 20 mL of given hard water sample against EDTA as in step 1. Let volume of EDTA used be $V_2$ mL.

Step 3. Determination of Mg$^{2+}$ hardness in sample

Pipette out 100 mL of sample in a 250 beaker. Add 25 mL calcium precipitating buffer solution to this with constant stirring of solution by glass rod. Keep as such for about 30 minute for settling of white ppt formed. Filter the solution.

Titrate 20 mL of given hard water sample against EDTA as in step 1. Let volume of EDTA used be $V_3$ mL.

General Calculations:

Step 1. Standardization of EDTA solution

1 mL of SHW = 1 mg of CaCO$_3$ (given)

Volume of SHW taken for titration = 20 mL

Concordant volume of EDTA solution used = $V_1$ mL

20 mL of SHW = $V_1$ mL of EDTA solution

1 mL of EDTA solution = $20/ V_1$ mL of SHW

1 mL of EDTA solution = $20/ V_1$ mL of CaCO$_3$

Step 2. Total hardness in water samples

Volume of hard water sample taken for titration = 20 mL

Concordant volume of EDTA solution used = $V_2$ mL

20 mL of hard water = $V_2$ mL of EDTA solution = $V_2\times(20/ V_1)$ mg of CaCO$_3$

So 1000 mL of hard water = $(V_2\times(20/ V_1)\times1000)/20$ mg of CaCO$_3$

Total hardness = $(V_2/ V_1)\times1000$ ppm of CaCO$_3$
Step 3. Determination of Mg\(^{2+}\) hardness in sample

Amount of hard water sample taken for titration = 100 mL

Amount of calcium precipitating buffer added = 25 mL

Hardness of 125 mL prepared solution = Mg\(^{2+}\) hardness of 100 mL hard water sample

So, 20 mL filtrate = \((100 \times 20)/125\) = 16 mL of hard water

Concordant volume of EDTA solution used = \(V_3\) mL

So 16 mL of hard water = \(V_3\) mL of EDTA solution = \(V_3 \times (20/ V_1)\) mg of CaCO\(_3\)

1000 mL of hard water = \((V_3 \times (20/ V_1) \times 1000)/16\) mg of CaCO\(_3\)

Mg\(^{2+}\) hardness in water sample = \((V_3 \times (20/ V_1) \times 1000)/16\) ppm in terms of CaCO\(_3\)

Ca\(^{2+}\) hardness in water sample = Total hardness - Mg\(^{2+}\) hardness in water sample

Results

Hardness due to Ca\(^{2+}\) in water sample = …… ppm in terms of CaCO\(_3\)

Hardness due to Mg\(^{2+}\) in water sample = …… ppm in terms of CaCO\(_3\)

Precautions

1. All solutions should be freshly prepared.
2. Distilled water should be checked with care before use.
3. The same amount of the indicator must be added to each time.
4. The reaction mixture must be shaken briskly during the reaction.
5. The end point must be observed during the titration.
6. pH = 10 should be maintained during the titration.
Experiment no. 2

Aim: To determine alkalinity of given water sample.

Apparatus/reagent required: Burette, pipette, conical flask, measuring flask, N/10 H₂SO₄

Indicator: Phenolphthalein and methyl orange indicator

End point: (i) Pink to colourless with Phenolphthalein

   (ii) Colour change from light yellow to reddish orange with methyl orange

Alkalinity

It is the total amount of those substances present in water which tend to increase the concentration of OH⁻ ions either due to dissociation of water or due to hydrolysis. Water becomes alkaline due to presence of HCO₃⁻, HCO₃⁻, CO₃²⁻ ions of salts of weak organic acids which consume H⁺ ions of water leading to increase in concentration of OH⁻ ions.

Types

Alkalinity of water in due to presence of

(i) Hydroxide ions (OH⁻ ions)
(ii) Carbonates ions (CO₃²⁻)
(iii) Bicarbonates ions (CO₃²⁻)

These may be present alone or in combination e.g. OH⁻ + CO₃²⁻ and CO₃²⁻ + HCO₃⁻. The possibility of OH⁻ + HCO₃⁻ is ruled out due to following reaction
OH\(^-\) + HCO\(_3\)^- → CO\(_3\)^{2-} + H\(_2\)O

Hence, all three cannot be present together.

**Theory**

The type and amount of alkalinity in water can be determined by titrating a known volume of it against standard acid using two indicator namely phenolphthalein and methyl orange in the same order.

Volume of acid used up to phenolphthalein end point (A mL) neutralizes all OH\(^-\) ions and converts CO\(_3\)^{2-} ions into HCO\(_3\)^- ions (i.e. half neutralization of CO\(_3\)^{2-}). The end point is disappearance of pink colour at a pH around 8.3. The acid used in further titration using methyl orange as an indicator neutralizes all HCO\(_3\)^- ions whether present originally or obtained from CO\(_3\)^{2-} ions. The end point is colour change from light yellow to reddish orange and comes at a pH range of 3-4.5. The total acid used in titration (A+B mL) gives total alkalinity of water. The reactions involved are:

(i) OH\(^-\) + H\(^+\) → H\(_2\)O  \[P\]

(ii) CO\(_3\)^{2-} + H\(^+\) → HCO\(_3\)^-  \[M\]

(iii) HCO\(_3\)^- + H\(^+\) → CO\(_2\) + H\(_2\)O

P = Phenolphthalein alkalinity = OH\(^-\) + ½ CO\(_3\)^{2-}

M = Total or methyl orange alkalinity = OH\(^-\) + CO\(_3\)^{2-} + HCO\(_3\)^- (due to all ions)

M-P = Alkalinity due to HCO\(_3\)^- ions

From the calculated values of P and M, following alkalinity correlations can be made using following table:

<table>
<thead>
<tr>
<th>Case</th>
<th>Alkalinity (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>OH(^-)</td>
</tr>
<tr>
<td>P = 0</td>
<td>0</td>
</tr>
<tr>
<td>P = M</td>
<td>P or M</td>
</tr>
<tr>
<td>P = ½M</td>
<td>0</td>
</tr>
<tr>
<td>P &gt; 1/2M</td>
<td>2P-M</td>
</tr>
<tr>
<td>P &lt; ½M</td>
<td>0</td>
</tr>
</tbody>
</table>
Procedure

1. Rinse and fill the burette with standard acid and note down its initial reading.
2. Pipette out 20 mL of water sample in a conical flask and add 1-2 drops of phenolphthalein indicator and pink colour will appear.
3. Titrate pink solution against acid till colour disappears and note down the final reading.
4. Now add 1-2 drops of methyl orange indicator to this and colour of solution becomes light yellow.
5. Titrate it further against acid till colour changes to light orange. Note the final burette reading again. Repeat for making concordant reading.

Observations

Table: Titration of water vs acid

<table>
<thead>
<tr>
<th>Sr. no.</th>
<th>Initial burette reading (x)</th>
<th>Final burette reading with phenolphthalein (y)</th>
<th>Final burette reading with methyl orange (z)</th>
<th>Vol. of acid used (mL) up to phenolphthalein end point (y-x)</th>
<th>Vol. of acid used (mL) up to methyl orange (z-x)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>2.</td>
<td></td>
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<td></td>
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<tr>
<td>3.</td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

Concordant reading of acid used upto phenolphthalein end point = y-x mL = A mL
Vol. of acid used up to methyl orange = Total volume consumed = z-x mL = B mL

General calculations

For finding the phenolphthalein alkalinity (P)

Concordant volume of acid used = A mL

Applying Normality equation

\[ N_1 V_1 = N_2 V_2 \]

(water) (acid)
\[ N_1 \times 20 = \frac{1}{10} \times A \]

\[ N_1 = \frac{A}{200} \]

Strength in terms of \( \text{CaCO}_3 \) = Normality \times \text{Eq. wt. of CaCO}_3

\[ = \frac{A}{200} \times 50 \text{ gL}^{-1} \]

So, phenolphthalein alkalinity \( (P) = \frac{A}{200} \times 50 \times 1000 \text{ mgL}^{-1} = \frac{A}{200} \times 50 \times 1000 \text{ ppm} \]

\[ = 250 \times A \text{ ppm} \]

**For finding the methyl orange alkalinity (M)**

Concordant volume of acid used = \( T \) mL

Applying Normality equation

\[ N_3 V_3 = N_4 V_4 \]

(water) (acid)

\[ N_3 \times 20 = (1/10) \times T \]

\[ N_3 = A/200 \]

Strength in terms of \( \text{CaCO}_3 \) = Normality \times \text{Eq. wt. of CaCO}_3

\[ = (T/200) \times 50 \text{ gL}^{-1} \]

So, methyl orange alkalinity \( (P) = (T/200) \times 50 \times 1000 \text{ mgL}^{-1} = (T/200) \times 50 \times 1000 \text{ ppm} \]

\[ = 250 \times A \text{ ppm} \]

Since, \( P \) is related to \( M \) as ……., the water samples contains following alkalinity:

(i) Alkalinity due to \( \text{OH}^- \) = …… ppm
(ii) Alkalinity due to \( \text{CO}_3^{2-} \) = …… ppm
(iii) Alkalinity due to \( \text{HCO}_3^- \) = …… ppm
Results

(i) Phenolphthalein alkalinity = ..... ppm
(ii) Methyl orange alkalinity = ........ppm
(iii) Alkalinity due to OH⁻ = ........ppm
(iv) Alkalinity due to CO₃²⁻ = ..........ppm
(v) Alkalinity due to HCO₃⁻ = ..........ppm
Experiment No. 3

Aim: To prepare Phenol formaldehyde (P-F) resin.

Chemicals required: Phenol (2g), 40% aq formaldehyde solution or formalin (2.5 mL), glacial acetic acid (5 mL) and conc. HCl (8mL).

Theory: Phenol formaldehyde resin or P-F resin or phenolic resins (also called phenoplasts) are important class of polymers which are formed by condensation polymerization of phenol and formaldehyde in acidic or alkaline medium. Following steps are involved:

Step 1: Formation of methylol phenol derivative

Initially the monomers combine to form methylol phenol derivative depending upon phenol to formaldehyde ratio.

Step 2: The phenol formaldehyde derivatives react among themselves or with phenol to give a linear polymer or a higher cross linked polymer.
(a) Linear polymer (Novolac)

(b) Cross linked polymer (Bakelite)

A highly cross linked thermosetting polymer called Bakelite may be formed by further condensation of novolac or methylol derivative.

It was first prepared by Backeland. It is easily formed if curing agent hexamethylene tetramine is added during synthesis.
**Procedure**

1. Place 5 mL of glacial acetic acid and 2.5 mL of 40% aq formaldehyde solution in a 100 mL beaker. Add 2 g phenol safely.
2. Wrap the beaker with a wet cloth or place it in a 250 mL beaker having small amount of water in it.
3. Add conc. HCl drop wise with vigorous stirring by a glass rod till a pink coloured gummy mass appears.
4. Wash the pink residue several times with to make it free from acid.
5. Filter the product and weigh it after drying in folds of a filter or in an oven. Report the yield of polymer formed.

**Observation**

Weight of empty watch glass = $W_1$ g

Weight of watch glass + polymer formed = $W_2$ g

Weight of polymer formed = $W_2 - W_1$ g

**Result**

Weight of phenol formaldehyde resin = $W$ g
Experiment No. 4

Aim: To prepare Urea formaldehyde (U-F) resin.

Chemical required: Urea (2g), 40% aq formaldehyde solution or formalin (5 mL), conc. H₂SO₄ (3-4 drops).

Theory

Urea formaldehyde resins are formed by condensation of urea and formaldehyde in acidic medium in following steps:

Step 1. Formation of methylol urea derivative

Initially urea and formaldehyde react to form methylol urea derivatives depending upon forsmaldehyde (U/F ratio).

\[ \text{H} \quad \text{N} \quad \text{H} \quad + \quad \text{CH}_2\text{O} \quad \xrightarrow{\text{H}^+} \quad \text{H} \quad \text{N} \quad \text{CH}_2\text{OH} \]

\[ \text{U/F}=1:1 \]

\[ \text{C} \quad \text{= O} \]

Monomethylol urea

\[ \text{H} \quad \text{N} \quad \text{H} \]

N,N'-Dimethylol urea

\[ \text{H} \quad \text{N} \quad \text{CH}_2\text{OH} \]

\[ \text{U/F}=1:2 \]

\[ \text{C} \quad \text{= O} \]

Step 2: Polymerization of methylol urea

Several molecules of methylol urea derivatives condense with loss of water molecules to form a highly cross linked urea formaldehyde resin.
Procedure

Take a 5 mL of 40% aqueous formaldehyde solution in a 100 mL beaker. To this add 2 g urea powder. Stir with a glass rod to make a saturated solution. Add a few drops of conc. H$_2$SO$_4$ and stir vigorously till a white solid mass is formed. Filter the residue and wash it several times with distilled water to remove any acid. Dry the residue in folds of filter paper or in an oven and weigh. Report the yield of urea formaldehyde polymer formed.

Observation

Weight of empty watch glass = $W_1$ g

Weight of watch glass + polymer formed = $W_2$ g

Weight of polymer formed = $W_2 - W_1$ g

Result

Weight of urea formaldehyde resin = $W$ g
Experiment No. 5

Aim: To find out saponification number of oil.

Chemical required: Standard alc. $\frac{N}{2}$ KOH solution, standard alc. $\frac{N}{2}$ HCl solution, ethyl methyl ketone as solvent.

Indicator: Phenolphthalein

End point: Disappearance of pink colour

Theory

Saponification is process of alkaline hydrolysis of oils (vegetable or animal) and fats giving soap.

Saponification number is defined as number of milligrams of KOH required to saponify 1 mg of a fatty oil.

For determination of saponification number of an oil, a known weight of oil is refluxed with a known excess of standard alc. KOH in a suitable solvent. During refluxing saponification of oil takes place.
\[
\begin{align*}
\text{CH}_2\text{O} - & \text{C} - \text{R}_1 \\
\text{CH}_2\text{O} - & \text{C} - \text{R}_1 \quad + 3 \text{ KOH} \\
\text{CH}_2\text{O} - & \text{C} - \text{R}_1
\end{align*}
\]

\[
\begin{align*}
\text{CH}_2\text{OH} & \quad \text{R}_1\text{COOK} \\
\text{CH} - & \text{OH} + \quad \text{R}_2\text{COOK} \\
\text{CH}_2\text{OH} & \quad \text{R}_3\text{COOK}
\end{align*}
\]

(R₁, R₂, R₃ may be same or different)

Amount of unreacted KOH left behind is determined by titration of mixture against 
standard HCl using phenolphthalein indicator. Disappearance of pink colour of solution marks 
the end point.

\[
\text{H}^+ + \text{OH}^- \rightarrow \text{H}_2\text{O}
\]

A blank experiment without oil is also performed. From the volume of HCl used in two 
titrations, saponification of oil can be obtained.

**Procedure**

1. Take two 250 mL conical flasks and label them as I and II. Weigh accurately about 1-2 
gm of given oil in a weighing bottle and transfer it to flask I. Weigh the empty bottle also.
2. Add 25 mL of ethyl methyl ketone and 25 mL of alc. \( \frac{N}{2} \) KOH solution to both flasks.
3. Put an air condenser in mouth of each bottle of each flask and keep them for refluxing on 
a water bath for 45 minutes.
4. Remove the flask from water bath. Wash the inner walls of each condenser with some 
distilled water into the respective flasks. Remove both condenser and cool both the flasks 
at room temperature.
5. Add 7-8 drops of phenolphthalein indicator to each flask. Titrate the solution of each 
flask against N/2 HCl taken in a burette till pink colour just disappears. Note burette 
reading in both titrations.

**Observations**

Weight of weighing bottle + oil sample = \( W_1 \) g
Weight of empty weighing bottle = $W_2$ g

Weight of oil taken for experiment = $W_1 - W_2$ g

Volume of ethyl methyl ketone added to each flask = 25 mL

Volume of N/2 alc. KOH added to each flask = 25 mL

**Table: Titration of unreacted KOH vs N/2 HCl**

<table>
<thead>
<tr>
<th></th>
<th>Initial burette reading (mL)</th>
<th>Final burette reading (mL)</th>
<th>Volume of HCl used (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>For flask I</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>For flask II</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Volume of N/2 HCl used by unreacted KOH in flask I = A mL

Volume of N/2 HCl used by unreacted KOH in flask II = B mL

Vol. of N/2 HCl is equivalent to volume of N/2 KOH used for saponification of $W_1 - W_2$ g oil = (B - A) mL

Saponification number of oil = \[
\frac{\text{Vol. of KOH used} \times \text{normality of KOH} \times \text{Eq wt of KOH}}{\text{wt of oil sample}}
\]

\[
= \frac{(B-A) \times \frac{1}{2} \times 56}{W_1-W_2} = \frac{(B-A) \times 28}{W_1-W_2} = X
\]

**Result:** Saponification number of given oil is $X$. 
Experiment No. 6

Aim: Determination of strength of HCl solution by titrating it against NaOH solution conductometrically.

Apparatus required: conductivity meter, conductivity cell

Chemical required: $\frac{N}{100}$ KCl, $\frac{N}{10}$ NaOH, $\frac{N}{100}$ HCl and distilled water.

Theory

End point of a volumetric analysis can also be found by conductometric titration which involves measurement of conductance of solution during titration. The principle of these titrations is that electrolytic conductance varies during the course of titration as it depends upon ions in solution and their mobility. The end point is found to from a plot of conductance of values against volume of titrant added which gives two lines intersecting each other. The point of intersection gives the end point of titration.

Titration of HCl vs NaOH (strong acid vs strong base) is studied by titrating it a known volume of acid against standard alkali and measuring conductance of solution at different times. Initially the conductance is high as HCl is strong electrolyte and is highly ionized. On adding NaOH from burette, the conductance decreases, it is because fast moving $\text{H}^+$ ions of HCl are neutralized and replaced by slow moving $\text{Na}^+$ ions.

\[
\text{H}^+ + \text{Cl}^- + \text{Na}^+ + \text{OH}^- \rightarrow \text{Na}^+ + \text{Cl}^- + \text{H}_2\text{O}
\]

![Plot of conductance vs volume of NaOH added](image)

Fig. 1 plot of conductance vs volume of NaOH added
After complete neutralization, added NaOH increases the conductance of solution due to addition of highly mobile OH\textsuperscript{-} ions, conductivity is minimum at equivalent point. On plotting conductance vs vol. of NaOH added, a V-shaped graph is obtained (Fig 1). The point of intersection of two lines gives end point. From volume of NaOH used at end point, the strength of HCl solution can be determined.

Procedure

1. Calibrate the conductivity meter using N/10 KCl.
2. Pipette out 50 mL of given HCl solution in a 100 mL beaker.
3. Dip conductivity cell in a HCl solution.
4. Note down the conductance value of solution.
5. Add 1 mL of N/10 NaOH solution from burette. Stir solution with a glass rod and note down the conductance of solution when it becomes stable.
6. Add NaOH solution in 1 mL lots and note the corresponding conductance values till conductance becomes constant.
7. Plot a graph between conductance (Y-axis) and volume of added NaOH solution (along X-axis).
8. Find out the volume of NaOH used at end point of intersection of two lines in graph.

Observation

Temperature of HCl solution = …..°C
Volume of HCl taken = 50 ml
Normality of NaOH taken = N/10

Table: Titration of HCl vs NaOH solution

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Vol. of NaOH added (mL)</th>
<th>Observed conductance (mho)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>.</td>
<td>.</td>
<td></td>
</tr>
</tbody>
</table>

General calculations

Let the volume of NaOH used at end point (from graph) = V mL
Applying normality equation

\[ N_1 V_1 = N_2 V_2 \]

(\text{HCl}) \quad (\text{NaOH})

\[ N_1 \times 50 = \frac{1}{10} \times V \]

\[ N_1 = \frac{V}{500} \]

Strength of HCl solution = normality \times \text{equivalent wt of HCl}

\[ = \frac{V}{500} \times 36.5 = X \text{ g L}^{-1} \]

\textbf{Result:} Strength of HCl solution is \ldots \text{ g L}^{-1}
Experiment No. 7

Aim: Determination of viscosity of lubricant by Redwood viscometer (No. 1 & 2).

Apparatus required: Redwood viscometer (No. 1 & 2), stop watch, thermometer, given lubricant oil and distilled water.

Theory

Viscosity is defined as the internal friction offered by internal friction offered by the layers of fluid of its flow. Viscosity is a measure of flow ability of a liquid at a definite temperature. It determines the performance of oil under operating conditions. Higher the viscosity of fluid lesser will be its flow.

Coefficient of viscosity is called absolute viscosity is defined as tangential force per unit area required to maintain a unit velocity gradient between two parallel layers a unit apart. It is denoted by \( \eta \) (eta).

Mathematically, \( \eta = \frac{F}{A \frac{dv}{dx}} \)

Where \( F \) = force
\( A \) = Area
\( \frac{dv}{dx} \) = velocity gradient.

Units: In C.G.S. system poise = dyne cm\(^2\) s
In SI system N m\(^{-2}\) s.

The absolute viscosity of lubricant is determined by measuring the time of flow of the oil through a capillary of definite dimensions at uniform temperature. The viscosity is can be measured by a Redwood viscometer.

Description of redwood viscometer:

It is available in two sizes. These are:

(i) \( RW_1 \) or Redwood No. 1 (Universal)
(ii) \( RW_2 \) or Redwood No. 2 (Admirality)

<table>
<thead>
<tr>
<th>Viscometer</th>
<th>Diameter of capillary</th>
<th>Length of jet</th>
</tr>
</thead>
<tbody>
<tr>
<td>( RW_1 )</td>
<td>1.62 mm</td>
<td>10 mm</td>
</tr>
<tr>
<td>( RW_2 )</td>
<td>3.8 mm</td>
<td>50 mm</td>
</tr>
</tbody>
</table>
The rate of discharge of oil through RW₂ is nearly 10 times faster than the discharge through RW₁ because receiving flask of RW₂ has a wider mouth.

Construction

It consists of a standard brass oil cup fitted with an agate jet of specific dimension in the middle of base and open at the upper end (Fig. 1). The jet can be opened or closed by a polished ball valve which controls the flow of oil. A pointer is provided in the oil cup to indicate the level up to which oil shall be filled in it. The lid of the cup is provided with a thermometer to note the temperature of oil. Surrounding the oil cup is cylindrical vessel made of copper. This vessel is filled with water and serves as a water bath to maintain the oil at a desired temperature of oil. It is heated by means of heating coils.

The water bath is provided with stirrer having four blades to maintain uniform temperature of bath. A thermometer is fitted in a water bath to know the temperature of water at its base. A 50 mL flask called Kohlrausch flask is provided below the jet to collect the liquid flowing out of the jet. The apparatus is also provided with leveling screws for its leveling.

Fig. 1. Redwood apparatus no. 1 & 2.
Procedure

1. Level the viscometer with the help of leveling screws. Fill the outer bath with water and connect to the electric mains. Clean the oil cup and discharge jet with a suitable jet with a suitable solvent like hexane, carbon tetrachloride etc. and properly dry it.
2. Place the ball valve on agate jet to close it. Pour the test oil in the cup carefully up to the pointer. Insert a thermometer and stirrer and cover the lid.
3. Adjust the temp. of water bath until the oil attains the desired temperature. In this period keep water in water bath and oil in oil cup.
4. Place a clean and dry Kohlrausch flask immediately below and directly in the line with the discharge jet
5. Remove the ball when oil attains desired temperature with one hand and start stop watch with the other hand. Allow the oil to flow till the flask is filled up to 50 mL mark. Stop the stop watch and note the time of flow in seconds.
6. Repeat the experiment 3-4 times and record the reading.
7. Report the mean value in Redwood seconds and also mentioning the viscometer used and the test temperature.

Observations

Table: For time of flow

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>Temperature (°C)</th>
<th>Time of flow (RW seconds)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Result: Viscosity of given lubricant is ……RW seconds.
**Experiment No. 8**

**Aim:** Determination of dissolved oxygen (D. O.) in given water sample.

**Chemicals required:** 48% Manganous sulphate solution (MnSO$_4$), alkaline potassium iodide (KI+KOH), conc. H$_2$SO$_4$, standard sodium thiosulphate or hypo solution (N/20 Na$_2$S$_2$O$_3$).

**Indicator:** freshly prepared starch solution added near the end point.

**End point:** Disappearance of blue colour (Hypo in burette).

**Theory:**

**Dissolved oxygen (D.O.):** It is defined as the amount of dissolved oxygen in milligram present in per litre of water. Its amount is about 8 ppm at room temperature.

DO can be determined by Winkler’s iodometric titration method. In this method DO is made to oxidize iodide ion to iodine (I$_2$) indirectly in the presence of manganous sulphate, KOH and sulphuric acid. Fixation of DO gives manganous oxide which on acidifying releases nascent oxygen required for oxidizing iodide ions. The liberated iodine is titrated against standard sodium thiosulphate solution using freshly prepared starch solution as indicator near the end point. Following reactions are involved:

\[
\begin{align*}
\text{MnSO}_4 + 2\text{KOH} &\rightarrow \text{Mn(OH)}_2 + \text{K}_2\text{SO}_4 \\
2\text{Mn(OH)}_2 + \text{O}_2 &\rightarrow 2\text{MnO} (\text{OH})_2 \\
&\text{(DO)} \\
2\text{MnO} (\text{OH})_2 + \text{H}_2\text{SO}_4 &\rightarrow \text{MnSO}_4 + \text{H}_2\text{O} + [\text{O}] \\
&\text{(Nascent oxygen)} \\
2\text{KI} + \text{H}_2\text{SO}_4 + [\text{O}] &\rightarrow \text{K}_2\text{SO}_4 + \text{H}_2\text{O} + \text{I}_2 \\
\text{I}_2 + 2\text{Na}_2\text{S}_2\text{O}_3 &\rightarrow 2\text{NaI} + \text{Na}_2\text{S}_4\text{O}_6 \\
&\text{sodium thiosulphate} \\
&\text{Sod. tetrathionate}
\end{align*}
\]

From the vol. of hypo used in the titration, DO of water can be determined.

**Procedure**

1. Rinse and fill the burette with hypo solution and note its IBR. Fill the DO bottle with water sample avoiding air bubbles.
2. Add 1 mL of 48% MnSO$_4$ solution and alkaline KI solution by keeping tip of pipette inside water.
3. Put a stopper on the flask and shake well. Keep the bottle undisturbed for some time to allow the brown ppt of basic oxide to settle.
4. Add 2 mL conc. H$_2$SO$_4$. Put a stopper on the flask again and shake well to dissolve the ppt.
5. Transfer 100 mL of brown solution to a conical flask. Titrate slowly against hypo solution till colour becomes pale yellow. Add 2 mL starch solution so that colour becomes blue. Titrate further against hypo till blue colour disappears. Note FBR at end point. Repeat to get concordant reading.

Observation

Volume of solution taken for each titration = 100 mL

<table>
<thead>
<tr>
<th>S. No.</th>
<th>IBR (mL)</th>
<th>FBR (mL)</th>
<th>Vol.of hypo solution used (mL)</th>
</tr>
</thead>
</table>

General calculation

Volume of hypo solution used in titration = $V$ mL

Normality of hypo solution = $N/20$

Applying normality equation

$$N_1V_1 = N_2V_2$$

(water) (Hypo)

$$N_1 \times 100 = (1/20) \times V$$

$$N_1 = \frac{V}{2000}$$

Strength of dissolved oxygen = Normality $\times$ Equivalent wt of oxygen

$$= \frac{V}{2000} \times 8 \text{ g L}^{-1} = \frac{V}{2000} \times 8 \times 1000 \text{ mg L}^{-1} = 4V \text{ ppm}$$

Result: The amount of dissolved oxygen in water sample is ….ppm
Experiment No. 9

Aim: To determine the total iron in an ore solution by internal indicator method.

Chemicals required: 5% stannous chloride solution (SnCl₂), saturated mercuric chloride solution (HgCl₂), standard dichromate solution (N/10 K₂Cr₂O₇), conc. HCl and dil. HCl

Indicator: Sodium diphenylamine sulfonate

End point: A blue-green, through a greyish tinge to the first permanent violet

Theory

An iron ore solution is prepared by treating the ore with acid. It contains both ferrous (Fe²⁺) and ferric (Fe³⁺) ions. The amount of iron in ore can be estimated by its titration with an oxidizing agent like standard pot. dichromate solution in acidic medium.

For finding total iron, the ore solution is first reduced with slight excess of SnCl₂ in medium acidified with conc. HCl.

\[
\begin{align*}
\text{Sn}^{+2} & \quad \text{Sn}^{+4} + 2e^- & \quad \text{(oxidation)} \\
2\text{Fe}^{+3} + 2e^- & \quad 2\text{Fe}^{+2} & \quad \text{(reduction)} \\
\text{Sn}^{+2} + 2\text{Fe}^{+2} & \quad \text{Sn}^{+4} + 2\text{Fe}^{+2} \\
\end{align*}
\]

Sn⁺² left in excess is destroyed by adding saturated mercuric chloride solution in one lot.

\[
\begin{align*}
\text{Sn}^{+2} & \quad \text{Sn}^{+4} + 2e^- \\
2\text{Hg}^+ + 2e^- + 2\text{Cl}^- & \quad \text{Hg}_2\text{Cl}_2 \\
2\text{Hg}^+ + 2\text{Sn}^{+2} + 2\text{Cl}^- & \quad \text{Sn}^{+4} + \text{Hg}_2\text{Cl}_2 \\
\end{align*}
\]

(Silky white ppt)

Total iron present as Fe²⁺ in reduced solution is titrated against dichromate solution in acidic medium using sodium diphenyl diammine sulphonate indicator.

\[
\begin{align*}
\text{Cr}_2\text{O}_7^{2-} + 14\text{H}^+ + 6e^- & \quad 2\text{Cr}^{+3} + 7\text{H}_2\text{O} \\
\text{Fe}^{+2} & \quad \text{Fe}^{+3} + e^- \times 6 \\
\text{Cr}_2\text{O}_7^{2-} + 14\text{H}^+ + 6\text{Fe}^{+2} & \quad 2\text{Cr}^{+3} + 6\text{Fe}^{+3} + 7\text{H}_2\text{O} \\
\end{align*}
\]

At the end point when all Fe⁺² ions get oxidised by dichromate, an excess drop of dichromate oxidises the indicator.

From the vol. of dichromate solution used in titration, total iron in ore solution can be calculated.
Procedure

1. Rinse and fill the burette with dichromate solution. Note the initial burette reading (IBR).
2. Pipette out 10 mL of solution in a conical flask.
3. Add 3 mL conc. HCl into it and boil on flame to get yellowish colour solution.
4. Add 5% SnCl₂ solution dropwise till yellow colour disappears. Add 2-3 mL drops in excess.
5. Cool the solution to room temperature under tap water,
6. Add 3 mL saturated H₂Cl₂ solution in one lot and shake it till silky white ppt are obtained.
7. Add 10 mL dil. HCl, 7-8 drops of indicator and titrate against dichromate solution till end point.
8. Note final burette reading, repeat the titration to record concordant value.

Observations

Table: Titration of reduced ore solution vs K₂Cr₂O₇ solution

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>IBR (mL)</th>
<th>FBR (mL)</th>
<th>Vol. of K₂Cr₂O₇ solution used (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

General calculation:

Volume of ore solution taken for titration = 10 mL
Concordant vol. of K₂Cr₂O₇ solution used = V mL
Normality of K₂Cr₂O₇ solution = N/10
Applying normality equation

\[ N_1 V_1 = N_2 V_2 \]
\[ (\text{ore}) \quad (\text{K}_2\text{Cr}_2\text{O}_7) \]
\[ N_1 \times 10 = \frac{1}{10} \times V \]
\[ N_1 = \frac{V}{100} \]
Strength of dissolved oxygen = normality \times \text{equivalent wt of iron}

\[
\frac{V}{100} \times 56 \text{ g L}^{-1} = \frac{V}{100} \times 56 \times 1000 \text{ mg L}^{-1}
\]

= A \text{ ppm}

\textbf{Result:} The total iron in given ore solution contains A ppm.
Experiment No. 10

Aim: To determine total dissolved solids (TDS) in given water samples.

Requirements: conductivity meter, conductivity cell, \(\frac{N}{100}\) KCl, distilled water and water samples

Theory

Total Dissolved Solids (TDS) is total amount of soluble mineral and salts impurities present in water. TDS is measured in parts per million (ppm). For example, drinking water should be less than 500 ppm, water for agriculture should be less than 1200 ppm, and high tech manufactures often require impurity free water.

One way to measure impurities in water is to measure the electric conductivity of water. A conductivity sensor measures how much electricity is being conducted through a centimeter of water. Specific conductivity is expressed as mhos per centimeter (M/cm), sometimes called siemens per centimeter (S/cm). Because a mho (or siemen) is a very large unit, the micromho (microsiemen) or millimho (millisiemen) typically is used (mS/cm). To convert the electric conductivity of a water sample (mS/cm) into the approximate concentration of total dissolved solids (ppm), the mS/cm is multiplied by a conversion factor. The conversion factor depends on the chemical composition of the TDS and can vary between 0.54 – 0.96. A value of 0.67 is commonly used as an approximation if the actual factor is not known.

\[
\text{TDS in ppm} = \text{Conductivity (μS/cm)} \times 0.67
\]

Procedure

1. Calibrate the conductivity meter using N/100 KCl and find out its cell constant.
2. Pipette out 50 mL of given water sample in a 100 mL beaker.
3. Dip conductivity cell in water sample.
4. Note down the conductance value of sample.
5. Repeat steps 2-4 for other water samples.

Observation

Conductance of given water samples are…….. μS.

Calculations

Conductivity of water sample = Conductance \times \text{Cell constant} \quad \text{μScm}^{-1}

\[
\text{TDS in ppm} = \text{Conductivity (μScm}^{-1}) \times 0.67
\]

Result: TDS of water samples are ……..ppm.