EXPERIMENT 1

**AIM:** To find refractive index of the given liquid samples and find Molar refraction and specific refraction.

**APPARATUS:** Abbe’s refractometer, temperature controller, light source and samples.

**THEORY:**

**Abbe’s Refractometer:** The Abbe instrument is the most convenient and widely used refractometer, Fig(1) shows a schematic diagram of its optical system. The sample is contained as a thin layer (~0.1mm) between two prisms. The upper prism is firmly mounted on a bearing that allows its rotation by means of the side arm shown in dotted lines. The lower prism is hinged to the upper to permit separation for cleaning and for introduction of the sample. The lower prism face is rough-ground: when light is reflected into the prism, this surface effectively becomes the source for an infinite number of rays that pass through the sample at all angels. The radiation is refracted at the interface of the sample and the smooth-ground face of the upper prism. After this it passes into the fixed telescope. Two Amici prisms that can be rotated with respect to another serve to collect the divergent critical angle rays of different colors into a single white beam, that corresponds in path to that of the sodium D ray. The eyepiece of the telescope is provided with crosshairs: in making a measurement, the prism angle is changed.
until the light-dark interface just coincides with the crosshairs. The position of the prism is then established from the fixed scale (which is normally graduates in units of $n_D$). Thermosetting is accomplished by circulation of water through the jackets surrounding the prism.

The refractive index of a substance is ordinarily determined by measuring the change in direction of collimated radiation as it passes from one medium to another.

\[
\frac{n_2}{n_1} = \frac{\nu_1}{\nu_2} = \frac{\sin \theta_1}{\sin \theta_2}
\]  

(1)

Where $v_1$ is the velocity of propagation in the less dense medium $M_1$ and $v_2$ is the velocity in medium $M_2$; $n_1$ and $n_2$ are the corresponding refractive indices and $\theta_1$ and $\theta_2$ are the angles of incidence and refraction, respectively Fig 2.

When $M_1$ is a vacuum, $n_1$ is unity because $v_1$ becomes equal to $c$ in equation (1). Thus,

\[
\frac{n_2}{\nu_2} = n_{vac} = \frac{c}{\nu_2} = \frac{\sin \theta_1}{\sin \theta_2}
\]  

(2)

Where $n_{vac}$ is the absolute refractive index of $M_2$. Thus $n_{vac}$ can be obtained by measuring the two angles $\theta_1$ and $\theta_2$Abbe’s refractometer is used to measure the refractive index of the given organic liquid. Using a particular monochromatic light source, the apparatus is calibrated with water as the liquid. Adjust the micrometer screw to focus the boundary between the bright and dark regions. Adjust the refractometer scale to place the cross wire of the telescope exactly on the boundary between the bright and dark regions. Repeat the same process for different organic liquids after the equipment is calibrated.

PROCEDURE:
1. Clean the surface of prism first with alcohol and then with acetone using cotton and allow it to dry.
2. Using a dropper put 2-3 drops of given liquid b/w prisms and press them together
3. Allow the light to fall on mirror.
4. Adjust the mirror to reflect maximum light into the prism box
5. Rotate the prism box by moving lever until the boundary b/w shaded and bright parts appear in the field of view.
6. If a band of colors appear in the light shade boundary make it sharp by rotating the compensator.
7. Adjust the lever so that light shade boundary passes exactly through the centre of cross wire
8. Read the refractive index directly on the scale
9. Take 3 set of readings and find the average of all the readings.

OBSERVATIONS:

Room temp. = _degrees

<table>
<thead>
<tr>
<th>Sr.No.</th>
<th>LIQUID</th>
<th>REFRACTIVE INDEX</th>
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<tbody>
<tr>
<td>1.</td>
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<td>2.</td>
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<td>3.</td>
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</table>

Specific refraction, \( R = \frac{(n^2 - 1)}{(n^2 + 2)} \times \frac{1}{d} \)

Molar refraction, \( R_m = R \times M \) (molecular mass of liquid)
EXPERIMENT 2

Aim: Determine the heat of neutralization of strong acid (HCl) with a strong base (NaOH) calorimetrically.

Theory:

Heat of neutralization may be defined as the amount of heat evolved when one gram equivalent of the acid is completely neutralized with one gram equivalent of the base or vice versa in dilute solutions. According to Arrhenius theory, strong acid and base are completely ionized in aqueous solutions; hence the neutralization of a strong acid (HCl) and strong base may be defined as

\[(\text{H}^+ + \text{Cl}^-) + (\text{Na}^+ + \text{OH}^-) \rightarrow \text{Na}^+ + \text{Cl}^- + \text{H}_2\text{O} + 57.3\text{KJ}\]

Apparatus:

Thermos flask with tight fitted cork, two 250ml beakers, two 100ml measuring flasks, 1/10 degree thermometer.

Chemicals:

0.2M HCl, 0.2 NaOH, distilled water.

Procedure:

To determine the water equivalent of calorimeter

1. Clean the calorimeter, take 100ml of water in it and note its temperature
2. In a separate beaker take 100ml of water and heat it to 15-20°C higher than the room temp.

3. Add this hot water quickly to the cold water in the calorimeter. Replace the stopper, stir with the stirrer and note the temp of the mixture.

To determine the heat of neutralization of HCl with NaOH

4. Take 100ml of 0.2m HCl in a clean calorimeter and note its temp

5. Take 100ml of 0.2m NaOH. The temperature must be equal to that of HCl, otherwise wait for sometime till both the solution attain same temp.

6. Pour 100ml of 0.2m NaOH solution quickly into the HCl sol in the calorimeter taking care to avoid splashing. Stir well and note the highest temp attained.

**Observations:**

**For water equivalent of calorimeter**

Volume of cold water taken=100ml  
Temp of cold water \(=t^0_c\)  
Volume of hot water=100ml  
Temp of hot water\(=t_1^0C\)  
Temp of the mixture\(=t_2^0C\)  
Density of water=1 g/ml
For heat of neutralization of HCl with NaOH

Volume of 0.2m HCl taken=100ml
Volume of 0.2 m NaOH taken=100ml
Initial temp of HCl and NaOH=t_3^\circ C
Final temp after neutralization=t_4^\circ C

Calculations

To calculate the water equivalent
Rise in the temp of cold water=(t_2-t_0)^\circ C
Fall in temp of hot water=(t_1-t_2)^\circ C
Let W be the water equivalent of the calorimeter and its contents

Acc to principle of calorimetry
Heat lost by hot water=heat gained by calorimeter and cold water

\[ W = \frac{[100(t_1-t_2)/(t_2-t_0) -100]}{g} \]

To calculate the enthalpy of neutralization

Initial temp of HCl and NaOH before mixing=
Final temp after mixing=t_4^\circ C
Rise in temp=(t_4-t_3)^\circ C
Total volume of mixture in the calorimeter=200ml
Heat of neutralization of 1000ml of 1M HCl=

\[ (200+W)\times(t_4-t_3)^\circ C \times 4.184/0.2 \times 100 \text{ kJ} = Q \text{ KJ} \]

Enthalpy of neutralization of HCl with NaOH=q KJ/mol
Result
Heat of neutralization of HCl and NaOH=……..kJ

    Actual value= -57.3kJ

%error =………

Precautions
1. Use very accurate 1/10th degree thermometer.
2. The bulb of the thermometer should not touch the bottom of the calorimeter.
3. The solution should be thoroughly stirred while noting the temp.
4. The solution should not splash out when poured from beaker in to flask.
EXPERIMENT 3

Aim: Determine the heat of neutralization of weak acid (CH₃COOH) with a strong base (NaOH) calorimetrically.

Theory:

Heat of neutralization may be defined as the amount of heat evolved when one gram equivalent of the acid is completely neutralized with one gram equivalent of the base or vice versa in dilute solutions. However if the acid involved in neutralization is weak, then the heat evolved is less than 57.3 KJ because some amount of heat is used in dissociation of weak acid.

\[(\text{CH}_3\text{COO}^- + \text{H}^+) + (\text{Na}^+ + \text{OH}^-) \rightarrow \text{Na}^+ + \text{CH}_3\text{COO}^- + \text{H}_2\text{O}\]

Apparatus:

Thermos flask with tight fitted cork, two 250ml beakers, two 100ml measuring flask, 1/10 degree thermometer.

Chemicals:

0.2M CH₃COOH, 0.2 NaOH, distilled water.

Procedure:

To determine the water equivalent of calorimeter

1. Clean the calorimeter, take 100ml of water in it and note its temperature
2. In a separate beaker take 100ml of water and heat it to 15-20°C higher than the room temp.
3. Add this hot water quickly to the cold water in the calorimeter. Replace the stopper, stir with the stirrer and note the temp of the mixture.

To determine the heat of neutralization of CH₃COOH with NaOH

4. Take 100ml of 0.2m CH₃COOH in a clean calorimeter and note its temperature.
5. Take 100ml of 0.2m NaOH. The temperature must be equal to that of HCl, otherwise wait for sometime till both the solution attain same temp.
6. Pour 100ml of 0.2m NaOH solution quickly into the sol CH₃COOH in the calorimeter taking care to avoid splashing. Stir well and note the highest temp attained.

**Observations:**

**For water equivalent of calorimeter**

- Volume of cold water taken=100ml
- Temp of cold water =t₀°C
- Volume of hot water=100ml
- Temp of hot water=t₁₀°C
- Temp of the mixture=t₂₀°C
- Density of water=1 g/ml
For heat of neutralization of CH$_3$COOH with NaOH

Volume of 0.2 m CH$_3$COOH taken=100ml
Volume of 0.2 m NaOH taken=100ml
Initial temp of CH$_3$COOH and NaOH=$t_3^\circ$C
Final temp after neutralization=$t_4^\circ$C

Calculations
To calculate the water equivalent
Rise in the temp of cold water=$\left(t_2-t_0\right)^\circ$C
Fall in temp of hot water=$\left(t_1-t_2\right)^\circ$C
Let $W$ be the water equivalent of the calorimeter and its contents

Acc to principle of calorimetry
Heat lost by hot water=heat gained by calorimeter and cold water

\[
W = \frac{100\left(t_1-t_2\right)}{t_2-t_0} - 100 \text{ g}
\]

To calculate the enthalpy of neutralization
Initial temp of CH$_3$COOH and NaOH before mixing=
Final temp after mixing=$t_4^\circ$C
Rise in temp=$\left(t_4-t_3\right)^\circ$C
Total volume of mixture in the calorimeter=200ml
Heat of neutralization of 1000ml of 1M CH$_3$COOH=
\[
(200+W)\times\left(t_4-t_3\right)\times 4.184/0.2\times 100 \text{ kJ} = Q \text{ KJ}
\]

Enthalpy of neutralization of CH$_3$COOH with NaOH=$q$ KJ/mol
Result
Heat of neutralization of CH$_3$COOH and NaOH=……kJ

Precautions
1. Use very accurate 1/10$^{th}$ degree thermometer.
2. The bulb of the thermometer should not touch the bottom of the calorimeter.
3. The solution should be thoroughly stirred while noting the temp.
4. The solution should not splash out when poured from beaker in to flask.
EXPERIMENT 4

AIM: Determination of the strength of a solution of hydrochloric acid by a standard solution of sodium hydroxide conductometrically

Theory:
When hydrochloric acid solution (HCl) is titrated with sodium hydroxide solution (NaOH), the highly mobile hydrogen ions ($\lambda_{H}^+ = 350 \text{ ohm}^{-1} \text{ cm}^{-1}$) are progressively replaced by slower moving sodium ions ($\lambda_{Na}^+ = 50 \text{ ohm}^{-1} \text{ cm}^{-1}$) and the conductance of the solution decreases. After the end point, the conductance of the solution rises sharply due to the presence of excess, highly mobile hydroxide ion ($\lambda_{OH}^- = 198 \text{ ohm}^{-1} \text{ cm}^{-1}$). Thus the neutralization of a strong acid by addition of a strong base leads to a minimum conductance at the end points. This is due to the disappearance of $H^+$ ions and their
replacement by slower moving Na\(^+\) ions of the base followed by the presence of highly mobile OH\(^-\) ions after the end point.

Therefore the nature of the plot (conductance of the solution versus volume of base added) will be as given below:

**Procedure:**

1. HCl solution of unknown strength is provided.
2. 0.1 (N) NaOH solution is provided.
3. Calibration of the instrument done at room temperature.

**Figure 1**

The conductivity cell used for this titration should permit stirring by shaking and to which the reagent can be added from burette. A large increase in volume during titration should be avoided.

**Apparatus:** Conductometer, conductivity cell, beaker, pipette, burette, conical flask.

**Chemicals:** Hydrochloric acid (HCl), sodium hydroxide (NaOH), conductivity water.

**Procedure:**

1. HCl solution of unknown strength is provided.
2. 0.1 (N) NaOH solution is provided.
3. Calibration of the instrument done at room temperature.
4. **Conductometric Titration:**

1. Rinse the conductivity cell a number of times with conductivity water or double distilled water.

2. Pipette out 20 mL of HCl in a beaker and dip the conductivity cell in it, so that the cell should dip completely in solution.

3. Note the temperature of the sample solution and accordingly set the temperature control or keep the cell in a thermostat at room temperature.

4. Add NaOH solution (few drops) from burette into HCl in lots of 0.5 ml after each addition stir it and measure the conductance after each addition.

5. Note the conductance till 10 ml of NaOH solution has been added

**Observation and Calculation:**

**Table 1:** Conductometric Titration

<table>
<thead>
<tr>
<th>s.no</th>
<th>Volume of 0.1 N NaOH added (ml)</th>
<th>Conductance</th>
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<td></td>
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<tr>
<td>2</td>
<td>0.50</td>
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<td>3</td>
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<tr>
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</tr>
<tr>
<td>18</td>
<td>8.50</td>
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</tbody>
</table>

Plot a graph between conductance and volume of titrant (NaOH solution). Two intersecting lines will be obtained (as given in the Figure 1) and the points of intersection of these lines represent the equivalent point.

Let, \( V_2 \) be the volume of NaOH at the equivalent point (from graph) for 20 ml 0.1 NHCl

Then \( (\text{HCl}) \ N_1 \ V_1 = N_2 V_2 (\text{NaOH}) \)

10. \( N_1 = (\frac{V_2}{200}) \, (N) \)
Strength = normality × equivalent bond
= \left(\frac{V_2}{200}\right) \times 36.5 \text{ g/L}

**Precautions:**

i) Electrical connection should be made carefully.

ii) Temperature during the experiment should be kept constant as conductance depends on temperature.

iii) Stirring should be done after each addition of titrant.

iv) To avoid the dilution effect, the concentration of the titrant should be 5-10 times more than that of the solution to be titrated.