Physical Practical

CEMA-CC-3-5-P

Experiment 1 : Conductometric titration of an acid (strong, weak/monobasic, dibasic and acid mixture) against strong base

Theory :

The strength of an acid (or base) can be accurately obtained from conductometric titration.

The conductance of an electrolytic solution taken in a conductivity cell (having a fixed value for cell constant) is directly proportional to the specific conductance of the solution (κ), which in turn depends on (i) concentration of ions and (ii) respective speed of ions/ionic mobilities.

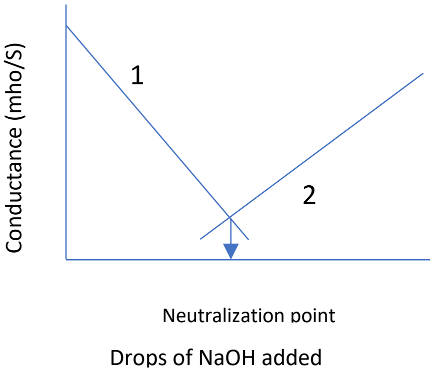
A known volume of an acid is taken in a conductivity cell and initial conductance of the acid solution (caused by the ions present in the acid solution) is measured. The alkali should have strength much higher than that of the acid (in order to avoid error in conductance due to dilution effect, termed dilution error) is added from a burette in drops and conductance measured after each addition. The observed conductance values are then plotted against volume of titrant added.

Strong acid(HCl) against Strong base (NaOH):

Strong acid like HCl is completely dissociated in solution. Neutralization means formation of water, hence replacement of H^+ ions is done by an equal number of Na^+ ions.

$$H^+(aq) + Cl^-(aq) \xrightarrow{Na^+ + OH^-} Na^+(aq) + Cl^-(aq) + H_2O$$

Ionic mobility of H^+ ions is much higher than that of Na^+ ions. As a result, conductance of the solution decreases sharply on gradual addition of NaOH. When all the H^+ ions are thus removed, conductance of the solution reaches the lowest limit. Further addition of NaOH after this point, increases the number of Na^+ ions as well as OH^- ions in solution. Ionic mobility of OH^- is much higher than that of Na^+. Hence the conductance of the solution again increases sharply. The nature of the plot will be



The point of intersection of the two lines correspond to the neutralization point. Strength of HCl is determined using the law of equivalence.

$$V_{HCl} \times S_{HCl} = V_{NaOH} \times S_{NaOH} \ldots\ldots\ldots\ldots\ldots(1)$$

Weak acid (HCl) against Strong base (NaOH):

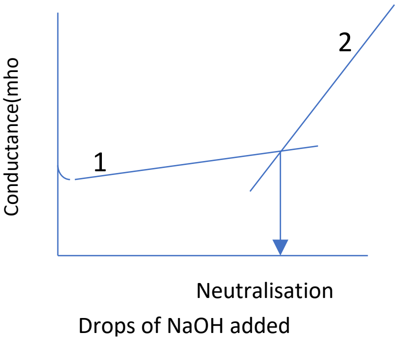
For a weak acid like CH_3COOH, the degree dissociation is low, hence it exists in the following equilibrium,

$$CH_3COOH \rightleftharpoons CH_3COO^- + H^+$$

The concentration of H^+ ion being low, conductance of this solution is appreciably low. As NaOH is just added, initially there is a slight dip in the conductance value due to the formation of

CH_3COONa, which retards the dissociation of CH_3COOH acid, due to common ion effect of acetate ions.

As we continue adding NaOH, OH^- ions remove the H^+ ions, hence to maintain the equilibrium, fresh H^+ ions are produced from the undissociated acid. Hence conductance of the solution increases gradually due to the presence of Na^+ ions, until neutralization point is reached. After neutralization, further addition of NaOH increases the Na^+ ions as well as the faster moving OH^-, hence conductance again increases sharply. The plot of conductance against drops of NaOH will be



The point of intersection of the two lines correspond to the neutralization point. Strength of CH_3COOH is determined using the law of equivalence.

$$V_{CH_3COOH} \times S_{CH_3COOH} = V_{NaOH} \times S_{NaOH} \text{ .... (2)}$$

Mixture of strong and weak acid (HCl + CH_3COOH) against Strong base (NaOH):

When NaOH is added to a mixture of HCl + CH_3COOH, the following reactions take place

$$H^+(aq) + Cl^-(aq) \xrightarrow{Na^+ + OH^-} Na^+(aq) + Cl^-(aq) + H_2O$$

$$H^+(aq) + CH_3COO^-(aq) \xrightarrow{Na^+ + OH^-} Na^+(aq) + CH_3COO^-(aq) + H_2O$$

The strong acid is completely dissociated, hence initially the conductance of the mixture is entirely due to the strong acid, since the ionisation of the weak acid is suppressed due to the common ion effect by the H^+ ions.

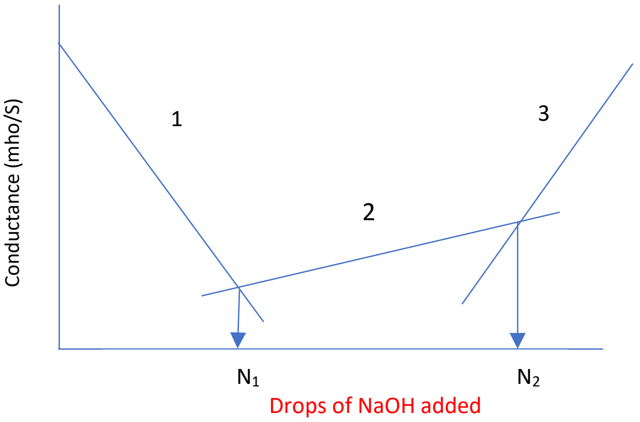
Since ionic mobilities (now present in solution) follow the order,

$$u_{H^+} > u_{OH^-} \gg u_{Na^+} > u_{CH_3COO^-}$$

When NaOH is gradually added, the H^+ ions are replaced by Na^+ ions, hence conductance of the solution decreases, as H^+ ions and OH^- ions combine to form unionized H_2O ions.

When neutralization of strong acid is just complete, (say volume of NaOH corresponds to N_1 drops) on further addition of NaOH, neutralization process of the weak acid starts, forming fully ionized CH_3COONa and unionized H_2O.

After neutralization of the weak acid, (say volume of NaOH corresponds to N_2 drops) the solution contains CH_3COO^- ions, Na^+ ions and OH^- ions. Ionic mobility of OH^- being highest, the conductance of the



solution again sharply increases.

The point of intersection of the 1st and 2nd lines correspond to the neutralization point of strong acid (N_1). Strength of HCl is determined using the law of equivalence.

$$V_{\text{Acid mixture}} \times S_{\text{HCl}} = V_{N_1} \times S_{\text{NaOH}} \text{ ....(3)}$$

Again, the point of intersection of the 2nd and 3rd line correspond to the neutralization point of the weak acid (N_2). Strength of CH_3COOH is determined using the law of equivalence.

$$V_{\text{Acid mixture}} \times S_{CH_3COOH} = (V_{N_2} - V_{N_1}) \times S_{\text{NaOH}} \text{ .... (4)}$$

Dibasic acid (Oxalic acid) against Strong base (NaOH):

If a dibasic acid like oxalic acid is titrated, a curve similar to that of mixed acid will be obtained.

A dibasic acid like oxalic acid has two dissociations:

$$H_2A \rightarrow H^+ + HA^- \quad ; \quad HA^- \rightleftharpoons H^+ + A^{2-}$$

The first dissociation is like that of a strong acid and the second dissociation is like that of a weak acid, with respective to the dissociation constant values of the two dissociations.

Hence the nature of the curve is similar to that of a mixed acid solution, but neutralisation of acid would correspond to N_2 drops

$$V_{N_2} \times S_{\text{NaOH}} = V_{\text{Oxalic acid}} \times S_{\text{Oxalic acid}} \text{......................................(5)}$$

The strength of NaOH can be determined by this method, from known strength of Standard Oxalic acid solution. Or if we have a standardized NaOH solution, we can determine the strength of a dibasic acid with unknown concentration.

Procedure:

1. Prepare 100 ml of standard (N/5) oxalic acid solution by accurate weighing.

2. Prepare 100 ml of ~(N/2) NaOH solution.

3. Standardise the NaOH solution using Standard Oxalic Acid solution prepared.

4. For Dibasic acid - base titration:

   Take 10 ml of the prepared oxalic acid solution in a 100 ml beaker and dip the conductivity electrode. Add little distilled water to just cover the electrode (conductivity cell).

4) Measure the conductance of the solution and note down in proper units.

5) Add ~(N/2) NaOH solution dropwise from a burette, homogenise the solution by stirring the

solution after each addition and also note conductance value after each addition.

6) Continue process (5), until conductance value reaches close to initial value.

7) <u>For Strong acid – base titration:</u>

Take 10 ml of the supplied strong acid solution in a 100 ml beaker and dip the conductivity electrode. Add little distilled water to just cover the electrode.

8) Repeat steps (4), (5) and (6).

9) <u>For Weak acid – base titration:</u>

Take 10 ml of the supplied weak acid solution in a 100 ml beaker and dip the conductivity electrode. Add little distilled water to just cover the electrode.

10) Repeat step (5) and continue until conductance value shows sharp change for 7 – 8 consecutive readings.

11) <u>For Mixed acid – base titration:</u>

Take 10 ml of the supplied mixed acid solution in a 100 ml beaker and dip the conductivity electrode. Add little distilled water to just cover the electrode.

12) Repeat steps (4), (5) and (6).

13) Measure volume of NaOH corresponding to 40/50 drops from burette and calculate volume for 1 drop.

14) Enter data for each set into Excel spread sheet and do the calculations.

<u>Calculations to be done in Excel Spreadsheet: (Not to be copied in practical note book)</u>

a. Enter Conductance data for each acid solution.
b. Plot conductance vs Drops of NaOH using 'scattered plot' option.
c. Add 'trendline' for each linear portion of plot (1, 2, 3 as applicable) along with equation and R^2 displayed.
d. Determine neutralization point by solving the respective equations:
    (i) Equation 1 and 2 for strong and also weak acid.

    (ii) Equation 1 & 2 to obtain N_1 for the strong acid and 2 & 3 to obtain N_2 for weak acid in 'mixture of strong and weak acid'.

    (iii) Equation 2 & 3 for N_2 in case of dibasic acid.

e. <u>Calculation of strength of Strong acid:</u>

Using S_{NaOH} obtained, calculate $S_{strong\ acid}$ using equivalence relation (1)

g. Calculation of strength of weak acid:
Using S_{NaOH} obtained, calculate $S_{weak\ acid}$ using equivalence relation (2)

h. Calculation of strength of weak and strong acid in acid mixture:
Calculate $S_{strong\ acid}$ using equivalence relation (3) and $S_{Weak\ acid}$, using equivalence relation (4).

i. Calculation of strength of dibasic acid:

Experimental Data:

a) Temperature during experiment:

b) Preparation of standard (N/5) oxalic acid solution:
Wt to be taken to make 100 ml (N/5) oxalic acid solution = …….. gm

Initial Wt (W_1) gm	Final Wt (W_2) gm	Wt taken (W_1 – W_2) gm	$S_{oxalic} = \frac{Wt\ taken}{Wt\ to\ be\ taken}\left(\frac{N}{10}\right)$

c) Standardisation of NaOH solution:

No.of obs	Vol of Oxalic Acid taken (ml)	Burette Reading for NaOH (ml)		Mean volume of NaOH reqd(ml)	S_{NaOH}
		Initial	Final		

d) Conductometric Titration of Strong acid against (~N/2) NaOH solution

No of obs	**Drops of NaOH added**	**Total Drops of NaOH added**	**Conductance (C) (proper unit, µS/mS)**

e) Conductometric Titration of Weak acid against (~N/2) NaOH solution

No of obs	**Drops of NaOH added**	**Total Drops of NaOH added**	**Conductance (C) (proper unit, µS/mS)**

f) Conductometric Titration of Dibasic acid against (~N/2) NaOH solution

No of obs	**Drops of NaOH added**	**Total Drops of NaOH added**	**Conductance (C) (proper unit, µS/mS)**

g) Conductometric Titration of Acid mixture against (~N/2) NaOH solution

No of obs	**Drops of NaOH added**	**Total Drops of NaOH added**	**Conductance (C) (proper unit, µS/mS)**

Calculation and Result:

Strong acid: $\frac{V_{NaOH}\ (at\ eq\ pt)\times S_{NaOH}}{V_{acid}}$

Weak acid : $\frac{V_{NaOH}\ (at\ eq\ pt)\times S_{NaOH}}{V_{acid}}$

Dibasic Acid: $\frac{V_{NaOH}\ (at\ eq\ pt)\times S_{NaOH}}{V_{acid}}$

Acid Mixture:

Strong acid in mixture: $\frac{V_{NaOH}\ (at\ eq\ pt, for\ N1)\times S_{NaOH}}{V_{acid}}$

Weak acid in mixture: $\frac{V_{NaOH}\ (at\ eq\ pt, for\ (N2-N1))\times S_{NaOH}}{V_{acid}}$

Experiment 2 : Study of saponification reaction conductometrically

Theory :

When an ester (R^1COOR^2) derived from a monocarboxylic acid (R^1COOH) and a monohydric alcohol (R^2OH) is treated with a caustic alkali (NaOH), the ester is hydrolysed to produce the alcohol and sodium salt of the acid:

$$R_1COOR_2 + NaOH \rightleftharpoons R_1COO^-Na^+ + R_2OH$$

Such alkaline hydrolysis of an ester is called **saponification**.

Methyl acetate (CH_3COOCH_3)on alkaline hydrolysis produces methanol (CH_3OH) and acetate (CH_3COO^-):

$$CH_3COOCH_3 + OH^- \rightleftharpoons CH_3COO^- + CH_3OH$$

t=0	a	a	0	0
t=t	(a-x)	(a-x)	x	x
t=∞	0	0	a	a

The overall reaction is kinetically of second order, being first order with respect to each of the reactants, the ester and hydroxyl ions (OH^-). The rate of the overall reaction may be expressed as:

$$rate = \frac{d[ester]}{dt} = k[ester][OH^-]\text{...}(1)$$

where k is the rate constant in and [ ] stands for concentration terms . If the initial concentrations of both ester and alkali be **a** and those after time *t* be **(a–x)** where, **x** is the amount of alkali/ester consumed, then,

$$\frac{dx}{dt} = k.(a-x)^2 \dots\dots\dots\dots\dots\dots\dots\dots (2)$$

Integration of equation (2), for x=0, when, t=0 yields,

$$k = \frac{1}{t}.\frac{x}{a-x} \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (3)$$

The progress of the reaction can be monitored by measuring the electrolytic conductance of the reaction mixture, since the highly conducting OH^- ions λ_0 =198.5 ohm^{-1} cm^2 g eq^{-1}) are replaced by weakly conducting CH_3COO^-ions(λ_0 = 40.9 ohm^{-1} cm^2 g eq^{-1}). If C_0, C_t and C_∞ be the conductances of the reaction mixture at the times t=0, t, and at the completion of the reaction (infinite time, t=∞), then,

$$a \propto (C_0 - C_\infty) \dots\dots\dots\dots\dots\dots\dots . (4a)$$

$$x \propto (C_0 - C_t) \dots\dots\dots\dots\dots\dots\dots . (4b)$$

$$(a-x) \propto (C_t - C_\infty) \dots\dots\dots\dots . (4c)$$

Equation (3) is then transformed to:

$$\frac{(C_0 - C_t)}{(C_t - C_\infty)} = k.a.t \dots\dots\dots\dots\dots\dots .. (5)$$

A plot of $\frac{(C_0 - C_t)}{(C_t - C_\infty)}$ versus *t* will be a straight line passing through the origin with a positive slope = **ka**. Thus, k may be evaluated from the relation,

$$k = \frac{slope}{a}$$

Procedure:

1. Prepare 100 ml of standard (~N/10) oxalic acid solution by accurate weighing.

2. Prepare 250 ml of (~N/10) NaOH solution and standardize the same against standard (N/10) oxalic acid using phenolphthalein indicator. Prepare 100 ml of **exact** (N/60) NaOH solution by accurate dilution of the standardized NaOH solution in a 100 ml volumetric flask.

3. Prepare 250 ml of (~N/10) acetic acid and standardize the same against the standardized NaOH solution using phenolphthalein indictor. Prepare 100 ml of **exact** (N/60) acetic acid solution by accurate dilution of the standardized acetic acid solution in a 100 ml volumetric flask.

4. Pipette out 25 ml (N/60) NaOH solution in a clean dry 100 ml beaker, add 25 ml conductivity water using the same pipette. Mix thoroughly and measure its conductance (C_0).
5. Prepare an exact (N/120) solution of sodium acetate, $CH_3COO^-Na^+$, by mixing equal volumes of (N/60) CH_3COOH and (N/60) NaOH solutions. Measure the conductance of this solution (C_∞).

6. Prepare a standard solution of the ester by taking 1 ml of it (using a graduated pipette) ~ 50 ml with water taken in a 100 ml volumetric flask, and make up the volume upto the mark of the volumetric flask.

7. Note the density of ester (mentioned on the bottle) and calculate density of ester taken at working temp(t) using the relation

$$0.932 - (t-20)\times 1.25 \times 10^{-4} \text{ gm/ml.}$$

Hence calculate the strength of the solution (taking MW=74). From this stock solution prepare, by accurate dilution, 100 ml of **exact** (N/60) solution.

8. In a clean dry 100 ml beaker, take 25 ml of (N/60) methyl acetate, and add 25 ml of (N/60) NaOH to it from a pipette, dip the conductivity cell and start stop watch at the time of half-discharge. Homogenise the solution and measure the conductance (C_t) of the reaction mixture at intervals of 1 minute. Take at least 15 such readings. Record the conductance vs. time data in a tabular form.

9. Enter the data in an Excel worksheet, calculate $(C_0 - C_t)$, $(C_t - C_\infty)$, and $\frac{(C_0 - C_t)}{(C_t - C_\infty)}$.

Plot $\frac{(C_0 - C_t)}{(C_t - C_\infty)}$ vs time on a scatter plot, add trendline with equation, R^2 and zero intercept.

Calculate k from k = slope/a . Here $a = \frac{1}{120}$ (N)

Experimental Data:

a) Temperature during experiment:

b) Preparation of standard (N/10) oxalic acid solution:
Wt to be taken to make 100 ml (N/10) oxalic acid solution = …….. gm

Initial Wt (W_1) gm	Final Wt (W_2) gm	Wt taken ($W_1 - W_2$) gm	$S_{oxalic} = \frac{Wt\ taken}{Wt\ to\ be\ taken}\left(\frac{N}{10}\right)$

c) Preparation of 250 ml (~N/10) NaOH solution.
d) Preparation of 250 ml of (~N/10) acetic acid solution.

e) Standardisation of (~N/10) NaOH solution:

No.of obs	Vol of Oxalic Acid taken (ml)	Burette Reading for NaOH (ml)		Mean volume of NaOH reqd(ml)	S_{NaOH}
		Initial	Final		

f) Standardisation of (~N/10) Acetic acid solution:

No.of obs	Vol of Acetic Acid taken (ml)	Burette Reading for NaOH (ml)		Mean volume of NaOH reqd(ml)	$S_{Acetic\ acid}$
		Initial	Final		

g) Preparation of 100 ml **exact** (N/60) NaOH solution:

h) Preparation of 100 ml **exact** (N/60) Acetic acid solution:

i) Calculation of strength of methyl acetate solution prepared:

j) Preparation of 100 ml exact (N/60) Methyl acetate solution:

k) Recording of conductance values for (N/60) NaOH + (N/60) Methyl acetate solution:

Time recorded		Conductance (mS)
Min	Sec	

Calculation and Result:

Experiment No 3 : Verification of Ostwald's dilution law and determination of ionisation constant (Ka) of a weak acid by conductometric method.

Theory :

A monobasic weak acid HA, is partially ionised in aqueous solution as:

$$\begin{array}{llll} & HA\,(aq) \rightleftharpoons & H^{+}(aq) + & A^{-}(aq) \\ \text{At eqbm} & c(1-\alpha) & c\alpha & c\alpha \end{array} \quad \text{...(1)}$$

The ions are aquated and the degree of ionisation (α) increases with dilution.

The degree of ionisation at a particular molar concentration (c) of the weak electrolyte, HA may be well approximated by the ratio $\frac{\Lambda}{\Lambda_0}$, where, Λ is the equivalent conductance of HA at concentration c and Λ_0 is its equivalent conductance at infinite dilution. Activity Ionisation constant (K_a) of the weak acid, HA, may be defined as,

$$K_a = \frac{a_{H^+}.a_{A^-}}{a_{HA}} \text{...(2)}$$

where, **a** stands for the activity of the respective species. Again, **a = f.c**, where **f** represents the ionic activity coefficient and c is the molar concentration in gm-moles/L.

The solutions used being very dilute, the ionic strength of the medium is very low. Hence the numerical values for **f** are almost close to unity, by Debye-Hückel Limiting law. Under this condition, according to Ostwald's dilution law, K_a may be written as:

$$K_a = \frac{[H^+][A^-]}{[HA]} = \frac{c\alpha . c\alpha}{c(1-\alpha)} = \frac{c\alpha^2}{(1-\alpha)} \quad \ldots\ldots\ldots\ldots(3)$$

Substituting $\alpha = \frac{\Lambda}{\Lambda_0}$ in equation (3), one obtains,

$$K_a = \frac{c\left(\frac{\Lambda}{\Lambda_0}\right)^2}{1-\frac{\Lambda}{\Lambda_0}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(4)$$

which on rearrangement gives,

$$\frac{1}{\Lambda} = \frac{1}{\Lambda_0} + \left(\frac{1}{K_a . (\Lambda_0)^2}\right) . \Lambda c \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(5)$$

If a series of solutions of the weak acid (HA) of different concentrations are prepared and their equivalent conductances are determined by measuring their conductances in a cell of known cell constant, then by plotting $\frac{1}{\Lambda}$ against Λc, we may obtain a straight line, with a positive intercept of $\frac{1}{\Lambda_0}$ and a positive slope of $\left(\frac{1}{K_a . (\Lambda_0)^2}\right)$.

Hence K_a may be calculated using the relation:

$$K_a = \frac{(\text{intercept})^2}{\text{slope}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(6)$$

provided Λ_0 is determined with sufficient accuracy. Therefore, by this method, the ionisation constant (K_a) as well as the equivalent conductance at infinite dilution Λ_0 of a weak electrolyte (HA) can be determined.

Procedure:

1. Prepare 100 ml of standard (~N/10) oxalic acid solution by accurate weighing.

2. Prepare 250 ml of (~N/10) NaOH solution and standardize the same against standard (N/10) oxalic acid using phenolphthalein indicator.

3. Prepare 250 ml of (~N/10) acetic acid and standardize the same against the standardized NaOH solution using phenolphthalein indictor. Prepare 100 ml of **exact** (N/50) acetic acid solution by quantitative dilution of the standardized acetic acid solution with **conductivity water** in a 100 ml volumetric flask.
4. Prepare 250 ml a standard KCl solution (strength slightly higher than N/10). From this solution prepare 100 ml of an **exact** (N/10) KCl solution by accurately diluting the prepared standard (N/10) KCl solution.
5. From this **exact** (N/10) KCl solution prepare 100 ml of an **exact** (N/100) KCl solution by quantitative dilution.

6. Rinse a 100 ml beaker and the conductivity cell with the exact (N/100) KCl solution and then pour sufficient volume of this solution into the beaker so that the electrodes are completely immersed in the solution. Record the conductance. Repeat this procedure with the exact (N/10) KCl solution.
7. Calculate the cell constant from the measured conductance values of these two solutions, and using the literature values for specific conductance of KCl solutions at these concentrations (κ), at the same temperature. Then calculate mean cell constant form these two values of cell constant obtained.
8. Measure the conductance of the conductivity water used for this experiment.
9. In a clean dry 100 ml beaker take 50 ml of the (N/50) acetic acid solution using a 25 ml pipette. Dip the clean dry conductivity cell into this solution, stir well and record the conductance. Carefully pipette **out** 25 ml of (N/50) acetic acid solution using a 25ml pipette labelled 'acid' and pour in exactly 25 ml conductivity water into the cell using another pipette labelled 'water'. Homogenise the solution well and record the conductance of this (N/100) acetic acid solution.

10. Follow the same procedure of progressive dilution to obtain (N/200), (N/400) and (N/800) acetic acid solutions and record their conductances.

11. Enter all conductance data into an Excel spreadsheet.

12. Subtract the value of conductance of water from recorded conductance of all solutions to obtain the corrected value for conductance.

13. Calculate the equivalent conductance values of all the acetic acid solutions using the mean value of the cell constant using the relation: $\Lambda = 1000\kappa/c$.

14. Plot $(1/\Lambda)$ versus Λc using a scatter plot and add trendline, with equation and R^2 values displayed. Find Λ_o from the intercept of the equation obtained from trendline.

15. Estimate K_a from the slope and intercept using equation (6).

Note:

Compare the experimental value of Λ_o obtained from the intercept with the calculated value of Λ_o of acetic acid using the known ion conductance (literature values) for hydrogen ion and acetate ion with proper temperature correction.

$$\Lambda_0(\mathrm{H^+}) = 349.82 \text{ and } \Lambda_0(\mathrm{CH_3COO^-}) = 40.9 \text{ ohm}^{-1}\,\text{cm}^2\text{gm} - \text{eq}^{-1} \text{ at } 25^{\circ}\text{C}$$

$$\Lambda_t = \Lambda_{25}\,[1 + \alpha\,(T - 25)], \text{ T temp in } ^{\circ}\text{C}$$

$$\alpha = 0.0142 \text{ for } (\mathrm{H^+}) \text{and } 0.02 \text{ for } (\mathrm{CH_3COO^-})$$

Utilize the literature value of Λ_o to calculate K_a and compare with that of Λ_0 obtained graphically.

Experimental Data:

a) Temperature during experiment:

b) Preparation of standard (N/10) oxalic acid solution:
Wt to be taken to make 100 ml (N/10) oxalic acid solution = …….. gm

Initial Wt (W_1) gm	Final Wt (W_2) gm	Wt taken ($W_1 - W_2$) gm	$S_{oxalic} = \frac{Wt\ taken}{Wt\ to\ be\ taken}\left(\frac{N}{10}\right)$

c) Preparation of 250 ml (~N/10) NaOH solution.

d) Preparation of 250 ml of (~N/10) acetic acid

e) Standardisation of (~N/10) NaOH solution:

No.of obs	Vol of Oxalic Acid taken (ml)	Burette Reading for NaOH (ml)		Mean volume of NaOH reqd(ml)	S_{NaOH}
		Initial	Final		

f) Standardisation of (~N/10) Acetic acid solution:

No.of obs	Vol of Acetic Acid taken (ml)	Burette Reading for NaOH (ml)		Mean volume of NaOH reqd(ml)	$S_{Acetic\ acid}$
		Initial	Final		

g) Preparation of 100 ml exact (N/50) Acetic acid solution. solution:

h) Preparation of standard (N/10) KCl solution, exact (N/10) KCl solution and exact (N/100) KCl solution.

i) Measurement of conductance of KCl solutions and determination of cell constant:

Strength of KCl solution	Conductance (G)	Specific conductance at T^oC (κ)	Cell constant(l/a)	Mean cell constant

j) Conductance of conductivity water = ………

k) Measurement of conductance of Acetic acid solutions:

Strength of Acetic acid solution	Measured conductance (μS/mS)

Calculation:

Result:

SPECIFIC CONDUCTANCES OF STANDARD SOLUTIONS OF KCl

Temp. of the solution in ^{0}C	**(N/10) KCl (Ohm^{-1}cm^{-1})**	**(N/100) KCl (Ohm^{-1}cm^{-1})**
15	0.01048	0.001147
16	0.01072	0.001173
17	0.01095	0.001199
18	0.01119	0.001225
19	0.01143	0.001251
20	0.01167	0.001278
21	0.01191	0.001305
22	0.01215	0.001332
23	0.01239	0.001359
24	0.01264	0.001386
25	0.01288	0.001413
26	0.01313	0.001441
27	0.01337	0.001468
28	0.01362	0.001496
29	0.01388	0.001524
30	0.01412	0.001552
31	0.01437	0.001582
32	0.01462	0.001610
33	0.01487	0.001638
34	0.01512	0.001666
35	0.01538	0.001696
36	0.01563	0.001724
37	0.01588	0.001752
38	0.01614	0.001782
39	0.01640	0.001810
40	0.01666	0.001838

Experiment No. 4: Determination of E° of Fe3+/Fe2+ couple in the hydrogen scale by potentiometric titration of ferrous ammonium sulfate solution using K2Cr2O7.

Theory :

When the Fe^{3+}/Fe^{2+} redox system is coupled with a saturated calomel electrode (SCE) as the reference electrode, the following electrochemical cell is produced:

$$(Pt)\ Hg(l)/Hg_2Cl_2(S)/KCl\ (saturated)\ (aq)\ ||\ Fe^{2+}(aq), Fe^{3+}(aq)\ |(Pt)$$

where the symbol || stands for agar–KNO_3 salt bridge which minimizes the liquid junction potential. The half-cell reactions at the electrodes are:

L. H. E : $2Hg(l) + 2Cl^-(aq) \rightleftharpoons Hg_2Cl_2(s) + 2e$

R. H. E: $Fe^{3+}(aq) + e \rightleftharpoons Fe^{2+}(aq)$

Overall cell reaction is,

$2Hg(l) + 2Cl^-(aq) + 2Fe^{3+}(aq) \rightleftharpoons Hg_2Cl_2(s) + 2Fe^{2+}(aq)$…….(1)

The e.m.f of the cell (E_{cell}) is given by,

$$E_{cell} = E_R - E_L$$
$$= E^o{}_{Fe^{3+}/Fe^{2+}} + 0.059\log\frac{[Fe^{3+}]}{[Fe^{2+}]} - E_{SCE} \text{.....................................}(2)$$

Since E_{SCE} remains unchanged, if the temperature remains unchanged, the e.m.f of the cell (E_{cell}) varies with variation of the ratio, $[Fe^{3+}]/[Fe^{2+}]$. If an oxidant $K_2Cr_2O_7$ is added to a solution of Fe^{2+} in acid medium, concentration of Fe^{2+} will fall and that of Fe^{3+} will rise due to the reactions:

$$Cr_2O_7{}^{2-} + 14\,H^+ + 6Fe^{2+} = 2Cr^{3+} + 7H_2O + 6Fe^{3+} \ldots\ldots..(3)$$

With the addition of oxidant, the ratio ($[Fe^{3+}]/[Fe^{2+}]$) progressively increases, consequently E_{cell} increases.

The standard reduction potential of an electrode may be defined as the emf of the cell produced by coupling standard hydrogen electrode on the left and the electrode under consideration on the right with all reactants and products at unit activities. Plot of E_{cell} versus volume or number of drops of the oxidant added is generated. The standard electrode potential of Fe^{3+}/Fe^{2+} system can be determined from the graph as follows:

At the half equivalence point, exactly half of the Fe^{2+} originally present is converted to Fe^{3+}, and the ratio ($[Fe^{3+}]/[Fe^{2+}]$) becomes unity. At this point,

$$E_{cell} = E_{\frac{1}{2}} = E^o{}_{Fe^{3+}/Fe^{2+}} - E_{SCE}$$

$$\therefore \quad \mathrm{E^o}_{\mathrm{Fe^{3+}/Fe^{2+}}} = E_{\frac{1}{2}} + E_{SCE} \text{ or } E_{cell} + E_{SCE} \text{......................(4)}$$

E_{SCE} is obtainable from literature using,

$$\mathrm{E_{SCE}} \text{ at } \mathrm{T^oC} = [0.2415 - 0.00076(\mathrm{T} - 25)] \text{ volt}$$

At any other point, it follows equation (2), hence

$$0.059 \log\frac{[\mathrm{Fe^{3+}}]}{[\mathrm{Fe^{2+}}]} = \mathrm{E^o}_{\mathrm{Fe^{3+}/Fe^{2+}}} - (E_{cell} + E_{SCE}) \text{ ....(5)}$$

Thus $\mathrm{E^o}_{\mathrm{Fe^{3+}/Fe^{2+}}}$ at temperature of experiment can be obtained from the intercept of the plot of $\log\frac{[\mathrm{Fe^{3+}}]}{[\mathrm{Fe^{2+}}]}$ against $(E_{cell} + E_{SCE})$.

Note:

Strictly speaking, this is the formal potential for the couple. This is due to the fact that $[\mathrm{Fe^{3+}}]= [\mathrm{Fe^{2+}}]$ does not necessarily imply $\mathrm{a_{Fe^{3+}}} = \mathrm{a_{Fe^{2+}}}$, because of the difference of the activity coefficients of the two ions.

Procedure:

1. Prepare 100 ml of standard (N/2) $K_2Cr_2O_7$ solution by accurate weighing using a digital balance.
2. Prepare 100 ml of (~N/20) solution of Mohr salt, $(NH_4)_2SO_4$. $FeSO_4$. $6H_2O$ in ~2(N) H_2SO_4.
3. Standardize the potentiometer with the built-in Weston Cadmium standard cell at 1.018V cell.
4. Take the $K_2Cr_2O_7$ solution prepared in a burette. Determine the volume of 50 drops from your burette and hence, determine the volume for 1 drop of $K_2Cr_2O_7$ solution.
5. Take an aliquot of 10 ml of the prepared (~N/20) Mohr salt solution in a 100 ml beaker. Add sufficient amount of (~2N) H_2SO_4 and dip a clean platinum electrode in the solution. Connect this half-cell with a saturated calomel electrode through an agar-KNO_3 salt bridge.
6. Measure the EMF of the experimental cell.
7. Add the (N/2) $K_2Cr_2O_7$ solution from the burette drop wise into the Mohr salt solution taken in the beaker and measure the EMF each time. Add 2 drops initially for 10 readings, then 1 drop at a time till the equivalence point is reached, which is indicated by a sharp increase of E_{cell}. Take at least 4 readings after the end point.

8. Enter the data in to an Excel spreadsheet.

9. Plot EMF versus the number of drops of $K_2Cr_2O_7$ solution added, using the scatter plot option with smooth curve.
10. Calculate $|\frac{\Delta E}{\Delta n}|$ and mean drops of $K_2Cr_2O_7$ solution (n_{mean}) added and from maximum value for $\left|\frac{\Delta E}{\Delta n}\right|$, identify drops of $K_2Cr_2O_7$ solution, which corresponds to this maximum value. This value represents its volume at the equivalence point.
11. Determine the mean drops of $K_2Cr_2O_7$ solution, which represents its volume at the equivalence point.
12. Calculate C_{Mohr} using the formula $C_{Mohr} \times V_{Mohr} = C_{Dichromate} \times V_{Dichromate}$, for volume of dichromate corresponding to equivalence point, in accordance to step 10.

    ($V_{Dichromate}$ = Mean drops × volume for 1 drop of $K_2Cr_2O_7$ solution)
13. **Determination of $E^o_{Fe^{3+}/Fe^{2+}}$:**

By equation (5), intercept of plot of $\log\frac{[Fe^{3+}]}{[Fe^{2+}]}$ against $(E_{cell} + E_{SCE})$ on y-axis gives $E^o_{Fe^{3+}/Fe^{2+}}$.

$E^o_{Fe^{3+}/Fe^{2+}} = E_{cell} + E_{SCE}$, where $\frac{[Fe^{3+}]}{[Fe^{2+}]} = 1$ and $\log\frac{[Fe^{3+}]}{[Fe^{2+}]} = 0$ (for half-neutralisation point)

Experimental Data:

a) Temperature during experiment:

b) Preparation of standard (N/2) $K_2Cr_2O_7$ solution:
Wt to be taken to make 100 ml (N/2) $K_2Cr_2O_7$ solution = …….. gm

Initial Wt (W₁) gm	Final Wt (W₂) gm	Wt taken (W₁ – W₂) gm	$S_{K_2Cr_2O_7} = \frac{Wt\ taken}{Wt\ to\ be\ taken}\left(\frac{N}{2}\right)$

c) Preparation of (~N/10) Mohr salt solution in 2N H_2SO_4:

d) 50 drops of $K_2Cr_2O_7$ solution = ………ml

e) Potentiometric titration of Mohr solution against $K_2Cr_2O_7$ solution:

Drops of $K_2Cr_2O_7$ added	Total drops of $K_2Cr_2O_7$ solution added	EMF of solution (V)

Calculation:

Result:

Experiment No. 5: Determination of K_{sp} for AgCl by potentiometric titration of $AgNO_3$ solution against standard KCl solution.

Theory :

When an $Ag^+/Ag(s)$ redox electrode is coupled with a saturated calomel electrode (SCE) as the reference electrode, the following electrochemical cell is produced:

$$(Pt)Hg(l)\ |\ Hg_2Cl_2(s)\ |KCl\ (saturated)(aq)||\ Ag^+(aq)|Ag(s)$$

where the symbol || stands for agar–KNO_3 salt bridge which minimizes the liquid junction potential. The half-cell reactions at the electrodes are:

L. H. E : $2Hg(l) + 2Cl^-(aq) \rightleftharpoons Hg_2Cl_2(s) + 2e$

R. H. E: $Ag^+(aq) + e \rightleftharpoons Ag\ (s)$

Overall cell reaction is,

$$2Hg(l) + 2Cl^-(aq) + 2Ag^+(aq) \rightleftharpoons Hg_2Cl_2(s) + 2Ag\ (s) \ .....(1)$$

The e.m.f of the cell (E_{cell}) is given by,

$$\begin{aligned} E_{cell} &= E_R - E_L \\ &= E_{Ag^+/Ag} - E_{SCE} \\ &= E^o_{Ag^+/Ag} + \frac{RT}{nF}\ln\frac{[Ag^+]}{[Ag]} - E_{SCE} \text{..............................}(2) \end{aligned}$$

Ag(s) being in the standard state, its activity is taken as unity. For a dilute solution, activity of Ag ion (a_{Ag^+}) may be replaced by the numerical value of concentration of Ag ($[Ag^+]$).

$Ag^+ \rightleftharpoons Ag$ being a 1 electron transfer, $n = 1$.

Equation (2) may be rewritten as

$$E_{cell} = E^o_{Ag^+/Ag} + \frac{RT}{F}\ln[Ag^+] - E_{SCE} \text{...................}(3)$$

Since $E^o_{Ag^+/Ag}$ and E_{SCE} remains unchanged, if the temperature remains unchanged, the e.m.f of the cell (E_{cell}) depends only on $[Ag^+]$.

When a solution of KCl is added to $AgNO_3$ solution, the following reaction takes place:

$$AgNO_3(aq) + KCl\ (aq) = KNO_3(aq) + AgCl(s) \downarrow$$

AgCl begins to precipitate, and, $[Ag^+]$ decreases as more and more of KCl is added, AgCl being a sparingly soluble salt.

$$Ag^+\ (aq) + Cl^-\ (aq) = AgCl\ (s)$$

As a consequence, E_{cell} decreases with increase in the number of drops of KCl added.

Near the equivalence point, addition of small amount (1 drop) of KCl practically removes all the Ag^+ ion from solution, hence E_{cell} decreases abruptly and $|\frac{\Delta E_{cell}}{dn}|$ value becomes very large (n=number of drops).

At equilibrium point, Ag^+ ions present now come from the dissociation of sparingly soluble salt AgCl.

$$\mathrm{AgCl(aq)} \rightleftharpoons \mathrm{Ag^+(aq)} + \mathrm{Cl^-(aq)}$$

The activity solubility product K_{sp} of AgCl is given as:

$$K_{sp} = a_{Ag^+} \times a_{Cl^-}$$

The activities a_{Ag^+} and a_{Cl^-} are identical.

$$K_{sp} = [Ag^+] \times [Cl^-] \,.\, f_{Ag^+} f_{Cl^-} = [Ag^+] \times [Cl^-] .\, f^2{}_{\pm}$$

The solution being dilute, $f_{\pm}$ is taken as unity, hence activity terms are replaced by concentration terms. Hence,

$$K_{sp} = [Ag^+] \times [Cl^-] = [Ag^+]^2 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(4)$$

$$[Ag^+] = \sqrt{K_{sp}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(5)$$

Substituting (5) in equation (3), E_{cell} at equivalence point becomes,

$$E_{cell}{}^{eq} = E^o{}_{Ag^+/Ag} + \frac{RT}{F}.\frac{1}{2}.\ln K_{sp} - E_{SCE}$$

Or, $$E_{cell}{}^{eq} = E^o{}_{Ag^+/Ag} + \frac{2.303\ RT}{2F}.\log K_{sp} - E_{SCE}$$

{substituting, values of R, F and T = 298K, $\frac{2.303\ RT}{F} = 0.059$}

$$E_{cell}{}^{eq} = E^o{}_{Ag^+/Ag} + \frac{0.059}{2}.\log K_{sp} - E_{SCE}$$

$$K_{sp} = Antilog\left[\frac{\left\{E_{cell}{}^{eq} + E_{SCE} - E^o{}_{Ag^+/Ag}\right\}}{0.0295}\right] \ldots\ldots\ldots\ldots(6)\ \text{(at 298K)}$$

After equivalence point addition of more KCl solution will lower $[Ag^+]$ further, due to common ion effect of Cl^- ions, hence E_{cell} will decrease further. ***

If we plot E_{cell} against drops of KCl added, or $|\frac{\Delta E_{cell}}{dn}|$ against 'mean drops' of KCl added, it is possible to find the drops (hence volume) of KCl to completely precipitate the Ag^+ ions present in solution, corresponding to the equivalence point. The strength of $AgNO_3$ solution can be determined using the relation,

$$V_{AgNO_3} \times S_{AgNO_3} = V_{KCl} \times S_{KCl}$$

Hence, from $E_{cell}{}^{eq}$, E_{SCE} at the room temperature and standard literature value of $E^o{}_{Ag^+/Ag}$, K_{sp} of AgCl can be determined.

- E_{SCE} at t°C = [0.2415 – 0.00076 (t – 25)] volt.
- For experimental temperature other than 25°C (298 K), the factor 0.059 should be modified to $\left(\frac{0.059\,T}{298}\right)$, where T is absolute temperature.
- *** E_{cell} will decrease further with increase in Cl^- according to
  $$E_{cell} = E^o{}_{Ag^+/Ag} + 0.059\log K_{sp} - 0.059\log[Cl^-] - E_{SCE}$$
  $$= E^f{}_{Ag^+/Ag} - 0.059\log[Cl^-] - E_{SCE}$$

where $E^f{}_{Ag^+/Ag}$ represents formal potential of $AgCl(s) + e \rightarrow Ag + Cl^-$.

Procedure:

1. Record the room temperature.
2. Prepare 100 ml of (N/100) $AgNO_3$ solution in distilled water.
3. Prepare 100 ml of a standard (N/10) KCl solution in distilled water by accurate weighing, using a digital balance.
4. Take 10 ml of the prepared $AgNO_3$ solution in a 100 ml beaker and dip the silver electrode in this solution. Add sufficient amount of conductivity water so that the electrode dips properly. This constitutes the experimental electrode Ag^+/Ag.
5. Take the prepared KCl solution in a burette and determine the volume of 50 drops and then calculate the volume of 1 drop of KCl.
6. Standardize the potentiometer with the built-in Weston Cadmium standard cell at 1.018V cell.
7. Connect the experimental cell with the standardized potentiometer.
8. Measure the e.m.f. of the cell (E_{cell}). Add 1 drop of (N/10) KCl solution at a time, homogenise the solution by stirring gently and record the E_{cell}.
9. Repeat the procedure till the equivalence point is reached, which is indicated by a sharp decrease of E_{cell}.
10. Take a few more readings beyond the equivalence point.
11. Enter the data in to an Excel spreadsheet.
12. Plot EMF versus the number of drops of KCl solution added, using the scatter plot option with smooth curve.
13. Calculate $|\frac{\Delta E}{\Delta n}|$ and mean drops of KCl solution (n_{mean}) added, identify maximum value for $|\frac{\Delta E}{\Delta n}|$, determine the mean drops of KCl solution, which corresponds to this maximum value. This value represents its volume at the equivalence point.
14. Calculate C_{AgNO_3} using the formula $C_{AgNO_3} \times V_{AgNO_3} = C_{KCl} \times V_{KCl}$, for volume of KCl corresponding to equivalence point, in accordance to step 13

    (V_{KCl} = Mean drops × volume for 1 drop of KCl solution)

Determination of $\mathbf{K_{sp}}$(AgCl):

a. The value of drops of KCl corresponding to maxima in value of $|\frac{\Delta E}{\Delta n}|$ represents the 'mean drops' of KCl, whose E_{cell} value cannot be measured (being half of a drop, 1 drop being added at a time). Hence calculate this intermediate value of E_{cell} using the FORECAST function in Excel for x-values and y-values just higher and lower than the n_{mean}.

b. Using this value of E_{cell}^{eq}, E_{SCE} at the room temperature and standard literature value of

$E^o_{Ag^+/Ag}$,determine K_{sp} of AgCl using the relation (6).

Experimental Data:

f) Temperature during experiment:

g) N/100 $AgNO_3$ solution was supplied.

h) Preparation of standard (N/10) KCl solution:
Wt to be taken to make 100 ml (N/10) KClsolution = …….. gm

Initial Wt (W$_1$) gm	Final Wt (W$_2$) gm	Wt taken (W$_1$ – W$_2$) gm	$S_{KCl} = \frac{Wt\ taken}{Wt\ to\ be\ taken}\left(\frac{N}{10}\right)$

i) 50 drops of KCl solution = ………ml

j) Potentiometric titration of $AgNO_3$ solution against KCl solution:

Drops of KCl added	Total drops of KCl solution	EMF of solution (V)

Calculation:

Result:

Experiment No. 6: Determination of Heat of Neutralisation of a Strong Acid by a Strong Base

. Theory :

Neutralization of an acid by a base is the formation of water from H^+ ions of the acid and OH^- ions of the base.

The molar heat of neutralisation(ΔH_{neut}) is defined as the enthalpy change associated with the complete neutralisation for 1 mole of H^+ from acid by 1 mole of OH^- ions from the base in dilute aqueous solution.

Let us consider a neutralisation reaction of a strong acid (HA) by a strong base (BOH) in aqueous solution. Since both are completely dissociated in aqueous solution, we can represent the neutralisation reaction as,

$$\mathrm{H^+(aq) + A^-(aq) + B^+(aq) + OH^-(aq) \rightarrow A^-(aq) + B^+(aq) + H_2O}$$

$$\mathrm{or, H^+(aq) + OH^-(aq) \rightarrow H_2O}$$

Therefore, heat of neutralization of a strong acid with a strong base is a constant irrespective of the strong acid or the strong base used.

Measurement of Heat of neutralization (ΔH_{neut}) is done using a calorimeter. The heat given off the neutralization reaction is absorbed by the reaction solution in the calorimeter and **also** by the calorimeter. The change in temperature of the reaction solution in the calorimeter and **also** by the calorimeter is measured using a thermometer.

$$\text{Total heat of neutralisation} = \Delta \mathrm{H_{neut}} = \textit{Heat gained by salt soln} + \textit{Heat gained by calorimeter}.$$

$$= [\text{Mass of salt soln.} \times \text{Sp. Heat capacity of salt soln.} \times \Delta\text{t of salt soln.}]$$

$$+ [\text{Calorimeter constant} \times \Delta\text{t of calorimeter}].$$

$$\therefore \textit{Molar heat of neutralisation} = \frac{\Delta \mathrm{H_{neut}}}{\text{No.of moles}}$$

In this determination, other than determination of temperature change, it is also necessary to evaluate the calorimeter constant (also known as its heat capacity) in J/°C.

Measurement of calorimeter constant:

If one adds a known mass of hot water to a known mass of cold water which is in the calorimeter, Heat (Q), lost by the hot water is absorbed by the cold water and the calorimeter.

Heat lost by Hot water = Heat gained by cold water + Heat gained by calorimeter

$$\therefore \text{Mass of hot water} \times \text{Sp. Heat capacity of water} \times \Delta\text{t of Hot water} =$$

$$[\text{Mass of cold water} \times \text{Sp. Heat capacity of water} \times \Delta\text{t of cold water}]$$

$$+ [\ \underline{(\text{Mass of calorimeter} \times \text{Sp. Heat capacity of calorimeter})\ } \times \Delta\text{t of calorimeter}]$$

where Calorimeter constant [C_k] = [Mass of calorimeter × Sp. Heat capacity of calorimeter]

Procedure:

1. Standardise an ~1(N) NaOH using a standard 1(N) standard oxalic acid solution taking 10 ml of acid (using phenolphthalein indicator).

2. Calculate the no. of moles of NaOH in 50 ml solution ('n').

Neutralisation experiment:

3. Take 50 ml ~1(N) HCL in a 100 ml beaker using a measuring cylinder and cover with a watch glass.
4. Also take 50 ml of standardised NaOH in a clean and dry coffee cup of the calorimeter using a burette.
5. Record temperature of both solutions every 30 seconds using a digital thermometer until a steady temperature shows for about 2 minutes.
6. If the same digital thermometer is used to measure temperatures of both acid and base solutions, in step (5), then wash and dry the thermometer each time before switching between acid and base solutions.
7. Pour the acid quickly into the NaOH solution and start the stopwatch. Stir the solution mixture well with the probe of the digital thermometer and note the temperature every 30 seconds for about 9/10 minutes from mixing.

Determination of Calorimeter constant:

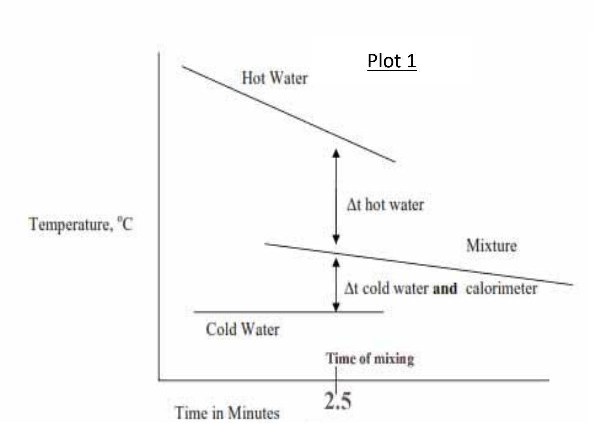
8. Wash and dry the coffee cup of the calorimeter. Take 50 ml distilled water in the same coffee cup using a burette, cover with the lid and record the temperature every 30 seconds until a steady temperature shows for about 2 minutes.

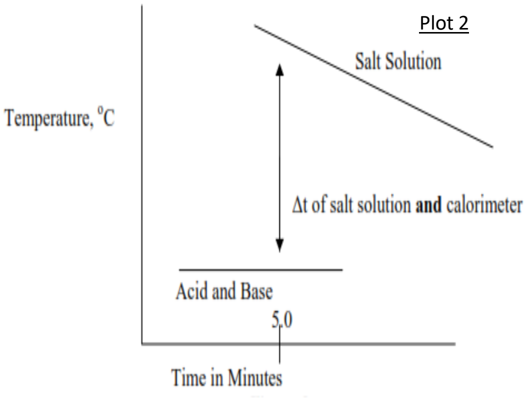
9. Take 50 ml distilled water in a clean and dry 100 ml beaker using measuring cylinder and heat to about 20^{o}C above room temperature in a thermostat. Monitor the temperature with the digital thermometer until the temperature comes down to about 15^{o}C higher than the room temperature.

10. Pour this hot water quickly into the cold water into the coffee cup and start the stopwatch. Stir the solution well with the probe of the digital thermometer and note the temperature for 30 minutes. After recording of data, add one drop of phenolphthalein was added to ensure that the base was limiting.

11. Plot temperature in (°C) in Y axis vs time in seconds in X axis for (a) hot water, (b) mixture of hot and cold water and(c) cold water (constant value over time) in one graph. (Plot 1)

12. Plot temperature in (°C) in Y axis vs time in second in X axis for reaction mixture, for NaOH solution and HCl solution (constant value over time) in one graph. (Plot 2)





Experimental Data:

1. Recording of room temperature:
2. Standardisation of NaOH solution:
3. Calculation of no. of moles of NaOH in 50 ml solution.
4. Recording of temperature of 1(N) HCl solution, prior to mixing:

Obs No.	Time		Time (min)	Temperature (^{o}C)
	Min	sec		

Hence, temperature of acid at time of mixing = ……… ^{o}C.

5. Recording of temperature of 1(N) NaOH solution, prior to mixing:

Obs No.	Time		Time (min)	Temperature (^{o}C)
	Min	sec		

Hence, temperature of base at time of mixing = ……… ^{o}C.

6. Recording of temperature after mixing HCl and NaOH solutions in calorimeter:

Obs No.	Time		Time (min)	Temperature (^{o}C)
	Min	sec		

7. Calibration of Calorimeter:

Recording of temperature of hot water prior to mixing:

Obs No.	Time		Time (min)	Temperature (^{o}C)
	Min	sec		

Hence, temperature of hot water at time of mixing = ……… ^{o}C.

8. Recording of temperature of cold water prior to mixing:

Obs No.	Time		Time (min)	Temperature (°C)
	Min	sec		

9. Hence, temperature of cold water at time of mixing = ……… °C.

10. Recording of temperature after mixing hot and cold water in calorimeter mixing:

Obs No.	Time		Time (min)	Temperature (°C)
	Min	sec		

Calculations:

A. $\mathbf{\Delta H_{neut}} = Q_{neut} = Q_{soln} + Q_{calorimeter}$ ………..(a)

B. $\mathbf{Q_{soln}} = [(\text{Mass of NaOH}) \times (\text{Sp. Heat of water}) \times (\text{Temp. change of NaOH})]$
$+ [(\text{Mass of HCl}) \times (\text{Sp. Heat of water}) \times (\text{Temp. change of HCl})]$………………………(b)

Mass of NaOH (g) = 50
Mass of HCl (g) = 50

Temp. change of NaOH (°C) = ………..
Temp. change of HCl (°C) = …………..

$\therefore Q_{soln}$(J) = …………….. [from (b)]

C. $\mathbf{Q_{calorimeter}}$ = (Calorimeter const. [C_k]) × (Temp. change of calorimeter during heat exchange between hot and cold water in a similar range of temperature)

Calorimeter constant (C_k): Heat capacity of calorimeter in j/°C

During mixing of hot and cold water in the calorimeter,

$$Q_{hot} = Q_{cold} + Q'_{calorimeter}$$

$$\Rightarrow Q'_{calorimeter} = Q_{hot} - Q_{cold}$$

$Q_{hot} = (\text{mass of hot water}) \times (\text{sp. heat of water}) \times (\text{temp change of hot water due to mixing})$

$Q_{cold} = (\text{mass of cold water}) \times (\text{sp. heat of water}) \times (\text{temp change of cold water due to mixing})$

Mass of hot water (g) = 50

Mass of cold water (g) = 50

Temp. change of hot water (°C) = ………

Temp. change of cold water (°C) = …………

Sp. Heat of water (J/ °C) = 4.18

$\therefore$ Calorimeter constant (C_k) = $\frac{Q'_{\text{calorimeter}}}{(temp\ change\ of\ cold\ water)}$……….. J/°C

$\therefore$ $\mathbf{Q_{calorimeter}}$ = (Calorimeter const. [C_k]) × (Temp. change of NaOH)

$\therefore$ $\mathbf{Q_{neut}} = \mathbf{Q_{soln}} + \mathbf{Q_{calorimeter}}$ = …………….J

No. of moles (n) of NaOH in 50 ml solution taken = …………….

$\therefore$ Heat of neutralisation per mole of NaOH by HCl at 33.2°C =

$\frac{\mathbf{Q_{neut}}}{n}$……….. J/mol = ……………. kJ/mol
