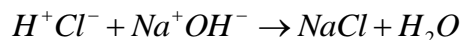


Ex. No. 1**Date:****ESTIMATION OF MIXTURE OF ACIDS BY CONDUCTOMETRY****AIM:**

To estimate the amount of acids present in the whole of the given solution. You are provided with standard 0.2 N sodium hydroxide solution.

PRINCIPLE:

Initial conductance of acid mixture is due to the presence of fast moving H^+ ions. Upon titration with NaOH, the fast moving H^+ ions are replaced by slow moving Na^+ ions. This results in decrease of conductance till the entire HCl is neutralized.



Further addition of NaOH increases the conductance value due to the formation of sodium acetate.



After the complete neutralization of acetic acid, any further addition of NaOH increases the conductance value rapidly.

A plot between volumes of NaOH added versus the conductance of the solution will give two intersection points. The first point corresponds to the titration between strong acid vs strong base. The second point corresponds to the titration between weak acid vs strong base.

APPARATUS & REAGENTS:

Conductivity meter, beaker, burette, pipette, glass rod, sodium hydroxide, hydrochloric acid etc.

PROCEDURE:

Makeup the given acid solution into a clean 100 ml standard flask with distilled water. Pipette out 20 ml of the made up acid solution into a clean 100 ml beaker. Pipette out 20ml of distilled water into the beaker. Rinse the conductivity cell thoroughly with distilled water and immerse into the beaker. Stir the solution gently and carefully with a glass rod. Measure the initial conductance of acid solution.

Now add 1 ml of NaOH from the burette. Stir the solution carefully with the glass rod and note the conductance.

Continue the titration by adding NaOH (in an aliquot of 1 ml) from the burette. When we add NaOH the conductance initially decreases to certain volume of NaOH. After that, the conductance starts increasing slightly for the addition of NaOH. After a particular volume the conductance increases rapidly. Plot a graph between the conductance values and volume of NaOH added as shown in the model graph. The first point of intersection of the two lines in the graph gives the first end point of the titration. Consider this value as V_1 . Consider the second point of intersection of the two lines in the graph as V_3 . $V_3 - V_1$ gives the second end point of the titration.

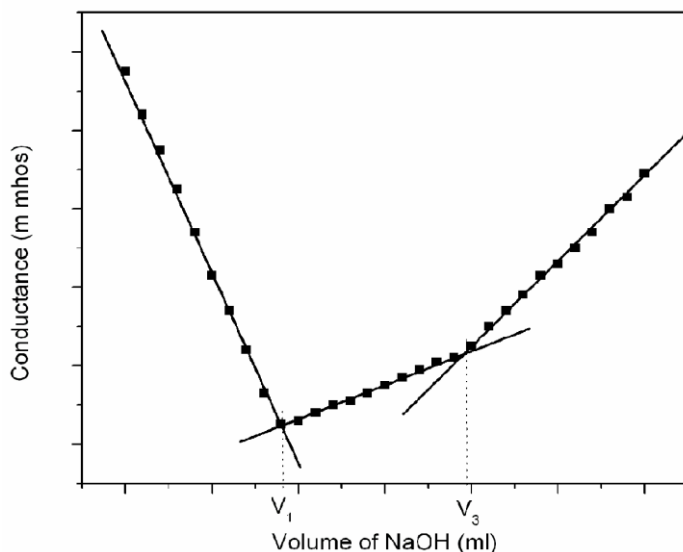
RESULT:

1. Amount of HCl present in the whole of the given solution = g
2. Amount of CH_3COOH present in the whole of the given solution = g

OBSERVATION:

S. No.	Volume of NaOH added (ml)	Conductance (mMho)
1	1	
2	2	
3	3	
4	4	
5	5	
6	6	
7	7	
8	8	
9	9	
10	10	
11	11	
12	12	
13	13	
14	14	
15	15	
16	16	
17	17	
18	18	
19	19	
20	20	
21	21	
22	22	
23	23	
24	24	
25	25	
26	26	
27	27	
28	28	
29	29	
30	30	

MODEL GRAPH:



CALCULATION

- Volume of NaOH (V_1) = ml
 Normality of NaOH (N_1) = 0.2 N
 Volume of Mixture of acids (V_2) = 20 ml
 Normality of HCl = $\frac{V_1 \times 0.2}{20}$ [V_1 - Refer graph]

The amount of HCl present in one litre = Normality of HCl \times Equivalent weight of HCl (36.5)

The amount of HCl present in the whole of the given solution

$$= \frac{\text{Normality of HCl} \times 36.5 \times 100}{1000} \text{ grams}$$

- Volume of NaOH (V_4) = $(V_3 - V_1)$ ml
 Normality of NaOH (N_1) = 0.2 N
 Volume of mixture of acids (V_2) = 20 ml
 Normality of CH_3COOH = $\frac{(V_3 - V_1) \times 0.2}{20}$ [V_1 and V_3 - Refer graph]

The amount of CH_3COOH present in one litre

$$= \text{Normality of } \text{CH}_3\text{COOH} \times \text{Equivalent weight of } \text{CH}_3\text{COOH} (60)$$

The amount of CH_3COOH present in the whole of the given solution

$$= \frac{\text{Normality of } \text{CH}_3\text{COOH} \times 60 \times 100}{1000} \text{ grams}$$

Ex. No. 2**Date:****ESTIMATION OF FERROUS ION BY POTENTIOMETRY****AIM:**

To estimate the amount of ferrous ions present in the whole of the given solution. You are provided with a standard 0.1 N potassium dichromate solution.

PRINCIPLE:

For a ferrous – ferric system: $Fe^{2+} \leftrightarrow Fe^{3+} + e^-$

The electrode potential is determined by combining platinum electrode immersed in $Fe^{2+} - Fe^{3+}$ system with saturated calomel electrode to form a cell and it is measured as emf. The cell is represented by:



and E_{cell} is given by:

$$E_{cell} = E_{R.H.S} - E_{L.H.S}$$

by applying the Nernst equation,

$$E_{cell} = \left[E_{Fe^{3+}/Fe^{2+}}^o + \frac{0.0591}{n} \log \frac{Fe^{3+}}{Fe^{2+}} \right] - 0.2422$$

Where E is the emf of the electrode, $E_{Fe^{3+}/Fe^{2+}}^o$ is the standard electrode potential for ferrous-ferric system, n is the number of electrons involved in the reaction, F is the Faraday's constant, R is the gas constant and T is the temperature.

Ferrous ions are oxidized to ferric ions by adding potassium dichromate which simultaneously increases the emf of the cell with a sharp increase in the emf. at the end point which is due to complete conversion of ferrous to ferric ions.

APPARATUS & REAGENTS:

Potentiometer, standard calomel electrode, platinum electrode (indicator electrode), beaker, burette, pipette, glass rod, standard flask, potassium dichromate, ferrous solution and sulphuric acid

PROCEDURE:

Transfer the given ferrous ion solution into a clean 100 ml standard flask. Make up the solution with distilled water. Pipette out 20 ml of the made up solution into a 100 ml beaker and add 20

ml of dil. H_2SO_4 . Immerse the platinum and calomel electrodes in the solution and connect them to the potentiometer.

Stir the solution and note the initial constant emf of the solution. Then add 0.5 ml aliquots of $\text{K}_2\text{Cr}_2\text{O}_7$ solution from the burette and note the emf. Initially the difference in emf will be small followed by a large difference in the emf values near the end point. After the end point again the difference in emf values will be small. By calculating ΔE , ΔV and $\Delta E / \Delta V$, a graph is plotted by taking volume of $\text{K}_2\text{Cr}_2\text{O}_7$ solution (V) on the X-axis and $\Delta E / \Delta V$ on the Y-axis. From the graph, the volume of $\text{K}_2\text{Cr}_2\text{O}_7$ solution consumed is determined.

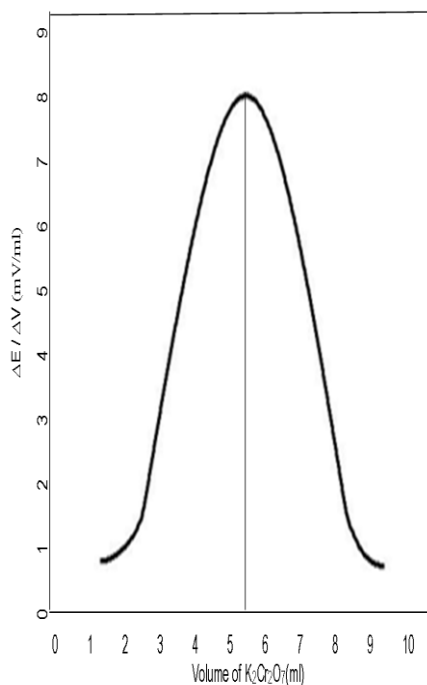
RESULT:

Amount of ferrous ion present in the whole of the given solution = _____ g.

OBSERVAION:

S. No.	Volume of K ₂ Cr ₂ O ₇ , V (ml)	Observed emf, E (mV)	ΔE	ΔV	$\Delta E / \Delta V$ (mV/ml)
1	0				
2	0.5				
3	1				
4	1.5				
5	2				
6	2.5				
7	3				
8	3.5				
9	4				
10	4.5				
11	5				
12	5.5				
13	6				
14	6.5				
15	7				
16	7.5				
17	8				
18	8.5				
19	9				
20	9.5				
21	10				
22	10.5				
23	11				
24	11.5				
25	12				
26	12.5				
27	13				
28	13.5				
29	14				
30	14.5				
31	15				

MODEL GRAPH:



CALCULATION

$$\text{Normality of } Fe^{2+} \text{ ion solution} = \frac{V_1 \times 0.1}{20} \quad [V_1 - \text{Refer graph}]$$

$$\begin{aligned} \text{The amount of } Fe^{2+} \text{ ion present in one litre} \\ = \text{Normality of } Fe^{2+} \text{ ion} \times \text{Equivalent weight of } Fe^{2+} \text{ ion (55.85)} \end{aligned}$$

$$\begin{aligned} \text{The amount of } Fe^{2+} \text{ ion present in the whole of the given solution} \\ = \frac{\text{Normality of } Fe^{2+} \text{ ion} \times 55.85 \times 100}{1000} \text{ grams} \end{aligned}$$

Ex. No. 4

Date:

ESTIMATION OF IRON BY PHOTOCOLORIMETRY

AIM:

To estimate the amount of Fe^{3+} ion present in the given water sample using spectrophotometer.

PRINCIPLE:

When a monochromatic light passes through a homogeneous coloured solution, a portion of incident light is reflected, a portion is absorbed and the remaining is transmitted.

$$I_0 = I_r + I_a + I_t$$

Where

I_0	=	Intensity of light incident before entering the solution
I_r	=	Intensity of incident light reflected
I_a	=	Intensity of incident light absorbed
I_t	=	Intensity of incident light transmitted

I_r is usually eliminated and hence $I_0 = I_a + I_t$. The mathematical statement of Lambert- Beer's Law is given by

$$T = I/I_0 = 10^{-kcl}$$

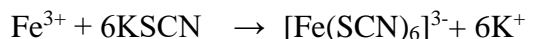
Where

T	=	Transmittance of solution
I	=	Intensity of light leaving the solution
I_0	=	Intensity of light incident on the solution
k	=	Molar absorption coefficient
c	=	Concentration of the solution in moles/ litre
l	=	Length (path thickness) of the absorbing solution

(or) $A = \log (I_0/I) = k c l$

where A is the absorbance or optical density of solution i.e when a ray of monochromatic light passes through an absorbing medium, its density decreases exponentially as the concentration of absorbing substance and the width or thickness increases independently. Keeping the path length constant (say $l = 1 \text{ cm}$), the variation is only due to concentration, c .

Fe^{3+} ion does not give any colour in solution. However, it develops a red colour when it reacts with potassium thiocyanate solution.

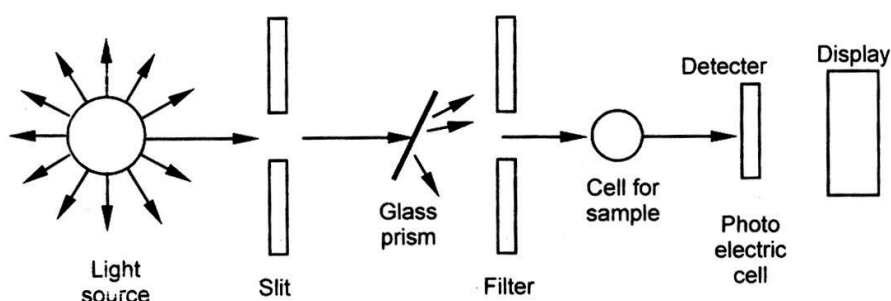


Red colour complex

Further, this colour is in the blue region, ($\lambda=480$ nm). Spectrophotometer has a wide range of adaptability that allows selection of monochromatic light of any wavelength in the visible spectrum.

INSTRUMENTATION:

The light source is an ordinary light bulb and monochromatic light is obtained by using either a glass prism or a diffraction grating. The monochromatic light then passes through the filter and is directed through a cell containing the sample. The incident light passes through the solution and hits the photoelectric cell and the output of this can be seen in the display.



PROCEDURE:

Prepare a series of standard solution containing 2-10 ppm of iron by adding 1 ml of 1:1 HCl and 1 ml of 10% potassium thiocyanate solution. The red solution is made up to 100 ml in a standard flask. Switch on the photocolormeter and warm it up for about 10 minutes. Adjust the monochromator at the wavelength $\lambda = 480$ nm. The blank is distilled water with the reagents used for the experiment. Keep the blank solution in the cuvette and adjust the instrument to transmittance corresponding to 100 or absorbance is zero. Similarly, place the various standard concentrations of nickel solutions in the cuvette one by one and measure the corresponding absorbance. Also, measure the absorbance of the unknown solution. Draw the calibration graph to determine the concentration of the unknown solution.

RESULT:

Amount of iron present in the given solution = _____ ppm.

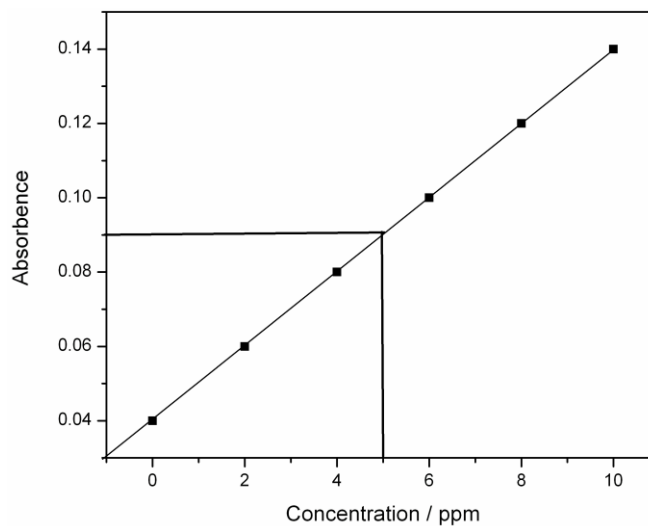
OBSERVATION:

Wavelength, $\lambda = 480 \text{ nm}$

Distilled water (Blank) = zero absorbance

Sl. No.	Concentration (ppm)	Absorbance
1	Blank	0
2	2	
3	4	
4	6	
5	8	
6	10	
7	Unknown	

MODEL GRAPH:



Ex. No. 5

Date:

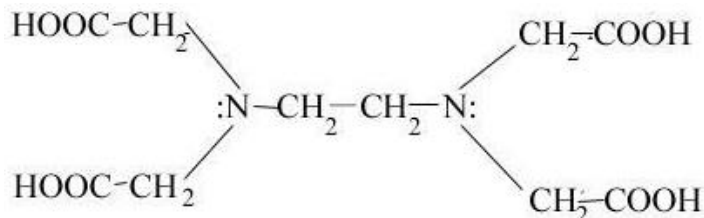
ESTIMATION OF HARDNESS OF WATER BY EDTA METHOD

AIM:

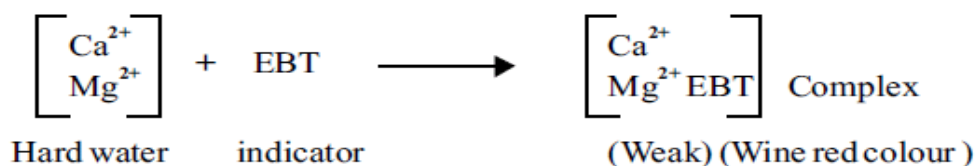
To estimate the total hardness of the given sample of water by EDTA method

PRINCIPLE:

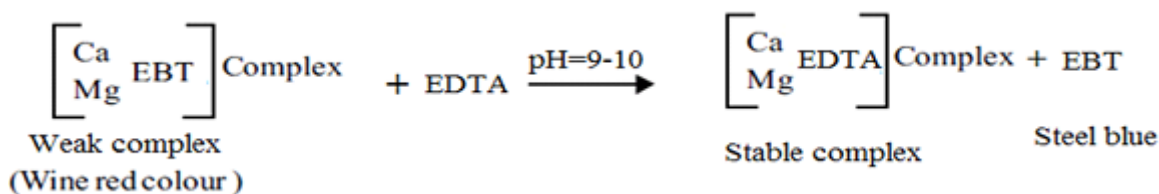
This estimation is based on complexometric titration. Ethylene diamine tetra acetic acid (EDTA) forms stable complexes with calcium and magnesium ions present in the hard water.



The hard water is buffered to a pH of about 9-10 and Eriochrome black T indicator is added. The indicator combines with some of Ca^{2+} and Mg^{2+} ions in the hard water to form an unstable complex of wine red in colour.



When the EDTA is added, the free Ca^{2+} and Mg^{2+} ions form complex with EDTA. These complexes are more stable than the indicator metal ion complex. When all the hardness causing ions are complexed by EDTA the indicator is set free which gives steel blue colour



APPARATUS & REAGENTS:

Burette, pipette, Conical flask, EDTA, Standard hard water, NH_4OH - NH_4Cl buffer solution, EBT indicator

PROCEDURE:

TITRATION I: Standardization of EDTA solution

Pipette out 20 ml of the standard hard water into a clean conical flask. Add about 5ml of (NH_4Cl + NH_4OH) buffer solution and a small quantity of Eriochrome Black T indicator. The solution turns to wine red in colour. Titrate against EDTA solution taken in the burette until the solution turns to steel blue colour. Repeat the titration for concordant values. Let V_1 be the volume of EDTA consumed by standard hard water.

TITRATION II: Estimation of total hardness of given water sample

Pipette out 20 ml of sample hard water into a clean conical flask. Add about 5 ml of (NH_4Cl + NH_4OH) buffer solution and a small quantity of Eriochrome Black T indicator. The solution turns to wine red in colour. Titrate against EDTA solution taken in the burette until the solution turns to steel blue colour. Repeat the titration for concordant values. Let V_2 be the volume of EDTA consumed by the sample hard water.

RESULT: Total hardness of the given sample of water = ppm (parts of CaCO_3 per million parts of water).

OBSERVATION AND CALCULATION:

Titration I: Standardization of EDTA solution

Burette : EDTA solution
 Pipette solution : 20 ml of std. hard water + 5ml buffer solution
 Indicator : Eriochrome Black T
 End point : Wine red to steel blue

Sl. No.	Volume of Std. hard water	Burette Reading		Volume of EDTA	Concordant Value
		Initial	Final		

1 ml of standard hard water = 1 mg of CaCO_3 equivalent hardness(1)

20 ml of standard hard water = 20 mg of CaCO_3 equivalent hardness(2)

20 ml of standard hard water consumes = V_1 ml of EDTA(3)

From equation (1) and (2):

V_1 ml of EDTA = 20 mg of CaCO_3 equivalent hardness

1 ml of EDTA = $\frac{20}{V_1}$ mg of CaCO_3 equivalent hardness

Titration II: Estimation of total hardness of water

Burette : Standardised EDTA solution
 Pipette solution : 20ml water sample + 5ml buffer solution
 Indicator : Eriochrome Black T
 End point : Wine red to steel blue

S. No.	Volume of Given Water Sample	Burette Reading		Volume of EDTA	Concordant Value
		Initial	Final		

20 ml of sample hard water consumes = V_2 ml of EDTA

20 ml of sample hard water = $V_2 \times 1$ ml of EDTA

20 ml of sample hard water = $V_2 \times \frac{20}{V_1}$ mg of CaCO_3 equivalent hardness

1000 ml of sample hard water = $V_2 \times \frac{20}{V_1} \times \frac{1000}{20}$ mg of CaCO_3 equivalent hardness

Total hardness of given water sample = $1000 \times \frac{V_2}{V_1}$ ppm.

Ex. No. 7

Date:

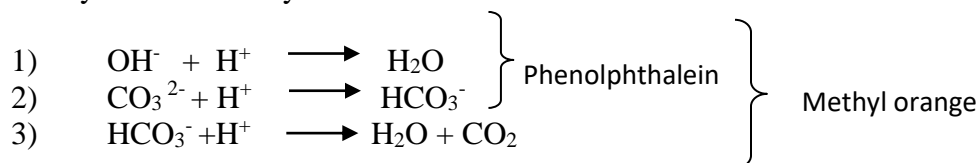
DETERMINATION OF ALKALINITY OF WATER

AIM:

To determine the types and amount of alkalinity present in the given sample of water. You are provided with a solution of Na_2CO_3 of strength 0.01N and a link solution of HCl solution are provided.

PRINCIPLE:

Alkalinity in water is due to the presence of soluble hydroxides, bicarbonates and carbonates. Determination of the various types and amounts of alkalinity is easily carried out by titration with standard HCl employing the indicators like phenolphthalein and methyl orange independently or successively.

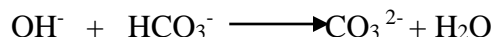


The titration of water sample against a standard acid up to phenolphthalein end point shows the completion of hydroxide alkalinity and one half of carbonate alkalinity present.

On the other hand titration of the sample against a standard acid using methyl orange end point makes the completion of reactions 1 to 3. Total amount of the acid consumed is used to measure the total alkalinity.

The possible combination of ions is OH^- only, HCO_3^- only, CO_3^{2-} only, OH^- and CO_3^{2-} together and CO_3^{2-} and HCO_3^- together.

OH^- and HCO_3^- ions cannot exist together since both the ions combine instantaneously to form CO_3^{2-} .



Similarly CO_3^{2-} , OH^- and HCO_3^- cannot exist together.

PROCEDURE

TITRATION I: Standardization of HCl

Pipette out 20 ml of standard sodium carbonate solution into a clean conical flask. Add 3 drops of methyl orange indicator. The solution turns yellow in colour. Titrate the solution against the HCl solution taken in the burette. The end point is the colour change from **yellow to pale pink**. Repeat the titration for concordant titre value.

TITRATION II: Estimation of alkalinity of water sample

Pipette out exactly 20 ml of the given water sample into a conical flask. Add few drops of phenolphthalein indicator and titrated against the standard hydrochloric acid taken in the burette. The end point is the **disappearance of pink colour** (P ml). Into the same solution add few drops of methyl orange indicator. The solution changes to yellow in colour. Continue the titration till **pale pink colour reappears** by adding the same HCl without break (M ml). Titration is repeated till you get concordant titre values.

RESULT:

The individual amounts of alkalinity in the water sample

- | | |
|---|-------------|
| (a) Hydroxide (OH^-) alkalinity | = ppm |
| (b) Carbonate (CO_3^{2-}) alkalinity | = ppm |
| (c) Bicarbonate alkalinity (HCO_3^-) | = ppm |
| (d) Total Alkalinity | = ppm |

OBSERVATION AND CALCULATION:

TITRATION I: Standardization of HCl

Burette solution: HCl

Pipette solution: 20 ml of std. Na_2CO_3

Indicator: 2-3 drops of methyl orange

End point: Yellow to pale pink

S. No	Volume of Na_2CO_3 (ml)	Burette Reading (ml)		Volume of HCl (ml)	Concordant value (ml)
		Initial	Final		

Volume of Na_2CO_3 solution (V_1) = 20 ml
 Normality of Na_2CO_3 solution (N_1) = 0.01 N
 Volume of HCl solution (V_2) = ml
 Normality of HCl solution (N_2) = ?

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

$$N_2 = \frac{20 \times 0.01}{V}$$

= N

TITRATION II: Estimation of alkalinity of water sample

Burette solution: HCl

Pipette solution: 20 ml of water sample

Indicator 1: 2-3 drops of phenolphthalein

End point 1: Disappearance of pale pink colour (Phenolphthalein End Point: P)

Indicator 2: 2-3 drops of methyl orange

End point 2: Reappearance of pale pink colour (Methyl orange End Point: M)

S. No.	Volume of water sample (ml)	Burette Reading (ml)			Volume of Std. HCl (ml)		Concordant Value (ml)	
		Initial	Final		[P]	[M]	[P]	[M]
			[P]	[M]				

S.No	Case	OH ⁻	CO ₃ ²⁻	HCO ₃ ⁻
1	P=0	Nil	Nil	M
2	P=M	P=M	Nil	Nil
3	P = ½ M	Nil	2P	Nil
4	P > ½ M	2P – M	2(M – P)	Nil
5	P < ½ M	Nil	2P	(M – 2P)

(i) If $P > 1/2 M$, Water sample contains OH⁻ + CO₃²⁻ alkalinity.

$$OH^{-} = \frac{(2P - M) \times \text{Normality of HCl} \times 50 \times 1000}{20}$$

= -----ppm

$$CO_3^{2-} = \frac{2(M - P) \times \text{Normality of HCl} \times 50 \times 1000}{20}$$

= -----ppm

(i) If $P < 1/2 M$, Water sample contains CO₃²⁻ + HCO₃⁻ alkalinity.

$$CO_3^{2-} = \frac{2P \times \text{Normality of HCl} \times 50 \times 1000}{20}$$

= -----ppm

$$HCO_3^{-} = \frac{(M - 2P) \times \text{Normality of HCl} \times 50 \times 1000}{20}$$

= ----- ppm