Experiment No. 1:

To determine the strength of commercial hydrochloric acid, 10 ml of which have been dissolved per litre of the given solution.

Apparatus required: Burette, pipette, conical flasks, stand, clamps, funnel etc.

Chemicals required: Sodium carbonate solution, HCl solution, methyl orange solution.

Theory: Commercial HCl is an aqueous solution of HCl gas. In order to determine the strength of HCl, it is titrated against aqueous sodium carbonate solution. This is an example of neutralization titration.

Methyl orange is used as an indicator during the titration. It changes colour from yellow to red at the end point.

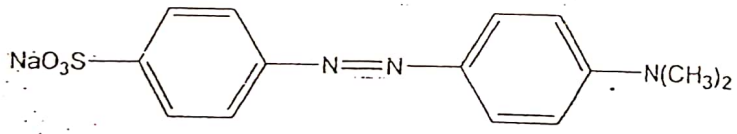
Reactions: $HCl + Na_2CO_3 \longrightarrow NaHCO_3 + NaCl$

$NaHCO_3 + HCl \longrightarrow NaCl + H_2O + CO_2$

Ionically: $CO_3^{-2} + H^+ \longrightarrow HCO_3^-$

$HCO_3^- + H^+ \longrightarrow H_2O + CO_2$

End point: Yellow to red (acid solution in the burette).



Methyl Orange

Procedure:

(i) Rinse and fill the burette with given acid solution.

(ii) Remove the air bubbles and set it at zero mark.

(iii) Pipette out 10 mL of the standard Na_2CO_3 solution into the titration flask and add 2 or 3 drops of methyl-orange.

(iv) Titrate with the HCl to a sharp colour change i.e., till red colour is obtained.

(v) Repeat the titration with every 10 mL of the Na_2CO_3 solution until three concordant readings are obtained.

Observations:

Volume of Na_2CO_3 solution taken for each titration = 10 mL

Burette readings: Titration of Na_2CO_3 vs HCl

Scanned by CamScanner

Department of Applied Chemistry
Delhi Technological University

Table: Titration of Na_2CO_3 vs HCl

S. No.	Initial Reading	Final Reading	Vol. of HCl

Calculations:

Normality of Na_2CO_3 solution = N_{Na2CO3}

To calculate normality of dil HCl solution, apply normality equation

$$N_{HCl} \times V_{HCl} = N_{Na2CO3} \times V_{Na2CO3}$$

or

$$N_{HCl} = N_{Na2CO3} \times V_{Na2CO3} / V_{HCl}$$

$$= N_{Na2CO3} \times 10 / V_{HCl}$$

Strength of dil HCl solution = $N_{HCl} \times 36.5$ g/L (strength = Normality × Eq. wt.)

Since, commercial HCl was diluted 100 times (10 mL in 1000 mL), the strength of commercial HCl can be obtained by multiplying the strength of dil. HCl by 100.

Hence, strength of commercial HCl = $N_{HCl} \times 36.5 \times 100$ g/L

Result:

Strength of commercial HCl is ..... g/L

Precautions:

a) Shake the titration flask continuously during adding the solution from the burette.
b) Keep your eye in level with the liquid surface while taking the burette reading or while reading the pipette or measuring flask etc.
c) Always read lower meniscus in case of colourless solution.

Scanned by CamScanner

Experiment No. 2

To determine the strength of Sodium hydroxide (NaOH) and Sodium carbonate (Na_2CO_3) from given water sample. Given standard N/40 Sodium carbonate to standardize given HCl solution.

Apparatus required: Burette, pipette, conical flask, funnel, conical flask, measuring cylinders.

Material required: Sodium carbonate (N/40) solution, HCl solution, Phenolphthalein (P), Methyl orange (M) and water sample.

Theory

The ionic equations involved for this reaction are:

(i) $OH^- + H^+ \longrightarrow H_2O$] Phenolphthalein

(ii) $CO_3^{-2} + H^+ \longrightarrow HCO_3^-$]

(iii) $HCO_3^- + H^+ \longrightarrow H_2O + CO_2$] Methyl orange

The titration is based upon the specific pH range for each indicator. The phenolphthalein indicator gives pink colour in pH range 8.2 – 10 while methyl orange gives pink colour in pH range 4.4 – 3.0. It has been observed that the presence of sodium hydroxide and sodium carbonate always provide a pH higher than 8.2 to the solution. Initially when standard hydrochloric acid is added, the OH^- ions are first converted to H_2O and then CO_3^{-2} ions are converted to HCO_3^-. When first two steps [(i) and (ii)] completed the pH of solution becomes less than 8.2 and the pink colour of phenolphthalein disappears. At this point, methyl orange is added as indicator to find the complete neutralization. On addition of further acid, the HCO_3^- ions are converted to CO_2 and H_2O. It means step (iii) is completed. When all HCO_3^- ions are used up the pH of solution becomes less than 4.4 instantaneously. At this point methyl orange gives pink colour.

Scanned by CamScanner

Procedure:

I. ***Standardization of given HCl solution***

(i) Take 10 mL of standard Na_2CO_3 with the help of pipette into a conical flask. Add 1-2 drops of methyl orange.

(ii) Run the acid solution from the burette into conical flask drop wise with constant shaking of solution. Find the end point when light yellow colour solution turns pink. Note volume of acid used. Repeat this step 4-5 times till you get at least two concordant readings.

II. ***Titration of given water sample with HCl solution***

(i) Pipette out 10 mL of given water sample into a conical flask. Add 2-3 drops of phenolphthalein indicator. The solution turns pink.

(ii) Add HCl solution from burette into the conical flask with constant shaking till the pink colour disappears. Note the reading on the burette (V_1).

(iii) After discolouration, add 1-2 drops of methyl orange into the solution.

(iv) Titrate now again with acid till yellow coloured solution turns pink again. Note the volume of acid used (V_2). Repeat the steps to get concordant readings.

Observation Table: Standardization of given HCl solution

S.No.	Volume of Na_2CO_3 taken (mL)	Burette Readings		Volume of HCl used (mL)	Concordant reading [illegible] (mL)
		Initial	Final		

Observation Table: Titration of given water sample with HCl

S.No.	Volume of Sample taken (mL)	Burette Readings (mL)		[illegible]	[illegible] (V_0) (mL)
		Initial	[illegible]		
					... (V_1)
					= .... (V_2)

Scanned by CamScanner

Calculations

Normality of given HCl solution (N_{HCl}) = (N_{Na2CO3} × V_{Na2CO3})/V_{HCl}

V_1 = Volume of HCl when whole of NaOH and half of carbonate is neutralized

≈ $[OH^-]$ + ½$[CO_3^{2-}]$ and V_3 ≈ ½$[CO_3^{2-}]$

Hence, Volume of HCl required for the neutralization of NaOH = V_1-V_3

Volume of HCl required for the neutralization of Na_2CO_3 = $2V_3$

Normality of NaOH in given solution (N_{NaOH}) = (N_{HCl} × V_1-V_3)/10

Normality of Na_2CO_3 in given solution (N_{Na2CO3}) = (N_{HCl} × $2V_3$)/10

Strength of NaOH in given solution = N_{NaOH} × Equivalent wt of NaOH g/L

Strength of Na_2CO_3 in given solution = N_{Na2CO3} × Equivalent wt of Na_2CO_3 g/L

Result:

Strength of NaOH in given solution = ... g/L

Strength of Na_2CO_3 in given solution = ... g/L

Precautions:

(i) Before starting the experiment, the glass apparatus must be perfectly cleaned.

(ii) For each titration, the initial reading of burette should always be the same.

(iii) Always read lower meniscus of solution level in burette.

(iv) Near the end point, add acid solution dropwise and after addition of each drop, see the colour against white background.

(v) Do not blow last drop of solution from pipette. Just tap the tip of pipette to the walls of the flask.

Scanned by CamScanner

Department of Applied Chemistry
Delhi Technological University

Experiment No. 3

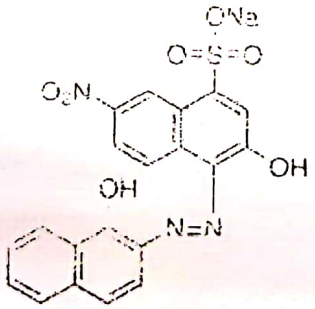
To determine the amount of permanent, temporary and total hardness of given water sample using standard N/50 EDTA solution.

Apparatus Required: Standard flasks, burette, conical flasks, pipette, etc.

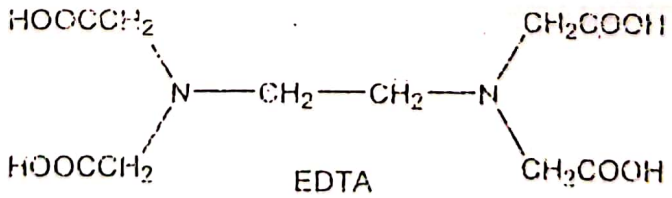
Chemicals Required: Water, EDTA solution, Eriochrome black T, buffer.

Theory: Water that doesn't produce lather with soap solutions rather produces white precipitate or scum is known as hard water. Hardness of water is due to the presence of calcium and magnesium salts in the form of bicarbonate, chloride and sulphate in water. There are two types of hardness present in water: temporary hardness and permanent hardness. Temporary hardness can be removed by boiling the water or by the addition of lime (or calcium hydroxide).

The amount of permanent, temporary and total hardness of water is easily determined by complexometric titration using disodium salt of EDTA solution. Eriochrome black T (EBT) is used as indicator.



EBT



EDTA

Procedure:

Total Hardness of the Water Sample

1. Pipette 20 mL of the water sample into a 250 mL conical flask. Add 2-3 mL of pH 10 ammonical buffer and 1 drop of EBT indicator. You will get a wine red color solution.

Scanned by CamScanner

2. Titrate the water sample with EDTA solution until the color changes from wine red to blue. Record the volume.
3. Repeat this two more times till you get concordant readings.

Permanent Hardness

1. Transfer a 100 mL water sample into a 250 mL beaker. Gently boil the water for 30 minutes. Allow the boiled water to cool to room temperature.
4. Filter the water directly into a clean 100 mL volumetric flask. Fill the volumetric flask to the mark with distilled water.
5. Pipet 20 mL of the filtered sample into a flask and add 2-3 mL of pH 10 ammonical buffer and 1 drop of EBT indicator.
6. Titrate the water sample with EDTA solution until the color changes from wine red to blue. Record the volume.
7. Repeat this two more times till you get concordant readings.

Observation Table Titration of given water sample against EDTA solution for total hardness

S. No.	Initial burette reading = V_1 mL	Final burette reading = V_2 mL	Volume of given Na_2EDTA solution used = (V_2-V_1) mL

Observation Table

Titration of given water sample against EDTA solution for permanent hardness

S. No.	Initial burette reading = V_1 mL	Final burette reading = V_2 mL	Volume of standardized EDTA solution used = (V_2-V_1) mL

Calculations:

The hardness can be calculated using the normality equation:

$$N_{water} \times V_{water} = N_{EDTA} \times V_{EDTA}$$

N_{EDTA} = Normality of Na_2EDTA solution = N/50

V_{EDTA} = Volume of Na_2EDTA solution used = From observation table.

V_{water} = Volume of the given water sample to be analyzed = 10 mL

Substituting these values in the normality equation, we can calculate the normality of the given water sample.

Scanned by CamScanner

Total Hardness or hardness of water is calculated with respect to the $CaCO_3$ only. Thus, hardness of given water sample is N_{water} × Eq. Wt of $CaCO_3$ = ...g/L

= N_{water} × Eq. Wt of $CaCO_3$ × 1000 = mg/L or ppm

For permanent hardness, repeat the calculation taking volume of Na_2EDTA solution used from observation table.

Result:

(i) Total Hardness = ... g/L or ppm

(ii) Permanent Hardness = ... g/L or ppm

(iii) Temporary Hardness = Total hardness – Permanent hardness = ... g/L or ppm.

Precautions:

(i) Before starting the experiment, the glass apparatus must be perfectly cleaned.

(ii) Always read lower meniscus of solution level in burette.

(iii) Near the end point, add EDTA solution drop wise and after addition of each drop, see the colour change.

(v) Do not blow last drop of solution from pipette. Just tap the tip of pipette to the walls of the flask.

Scanned by CamScanner

Experiment No. 4

To determine the strength of free chlorine in given water sample. Given standard N/40 copper sulphate ($CuSO_4$) solution to standardize sodium thiosulfate solution.

Apparatus required: Burette, Pipette, Measuring flask, glass rod

Reagents required: Potassium iodide solution (10%), N/40 Hypo solution ($Na_2S_2O_3$), starch solution (freshly prepared).

Theory: Chlorine is widely used for disinfection of potable and municipal water supplied to remove bacteria, fungus and other pathogenic micro–organisms, and for deodourization, since it is a powerful oxidizing agent and is cheaply available. Chlorination is done with the help of bleaching powder or chlorine gas or chlorine dissolved in water in the form of concentrated solution or with chloramines. The sterilizing action of chlorine is supposed to be due to its reaction with water producing hypochlorous acid and nascent oxygen both of which have powerful germicidal properties.

The principle involved in the estimation of free chlorine in water is that when a measured quantity is treated with excess of potassium iodide, the free chlorine present in the water oxidizes the corresponding amount of potassium iodide to iodine. The liberated iodine is estimated by titrating against standard hypo solution, using starch as indicator.

$$Cl_2 + 2KI \longrightarrow 2KCl + I_2$$

$$I_2 + \underset{\text{Sodium thiosulphate}}{2Na_2S_2O_3} \longrightarrow \underset{\text{Sodium tetrathionate}}{Na_2S_4O_6} + 2NaI$$

$$I_2 + \text{Starch indicator} \longrightarrow \text{Blue colour}$$

Procedure:

Part I: Standardization of hypo solution with given copper sulphate solution

(i) Take 10 ml of given copper sulphate solution ($CuSO_4$) from pipette in conical flask.

(ii) Add 1 ml of potassium iodide (KI) solution and titrate with the hypo solution till faint yellow colour develops. At this point, add 1-2 drop of starch as indicator. The solution will turn blue in colour. Titrate further with hypo to colourless as end point. Note the concordant volume of hypo solution as V mL.

Scanned by CamScanner

Part II: Titration of given water sample with hypo solution

(i) Take 10 ml of given water sample from pipette in a conical flask.

(ii) Add 1 ml of potassium iodide (KI) solution and titrate with the hypo solution till faint yellow colour develops. At this point, add 1-2 drop of starch as indicator. The solution will turn blue in colour. Titrate further with hypo to colourless as end point. Note the concordant volume of hypo solution as V mL.

Observations

Table: Titration of hypo solution versus given $CuSO_4$ solution

S.No.	Volume of $CuSO_4$ solution (mL)	Burette Readings		Volume of hypo used (mL)	Concordant reading (V) (mL)
		Initial	Final		

Table: Titration of hypo solution versus given $CuSO_4$ solution

S. No.	Volume of water sample (mL)	Burette Readings		Volume of hypo used (mL)	Concordant reading (V) (mL)
		Initial	Final		

Calculations

Part I: Normality of $CuSO_4$ solution (N_1) = N/40

Volume of $CuSO_4$ solution (V_1) = 10 ml

Normality of hypo solution (N_2) = ?

Volume of hypo solution (V_2) = burette reading

$N_1 V_1 = N_2 V_2$ will give the normality of hypo solution (N_2)

Part II: Volume of water sample taken for each titration (V_3) = 10 mL

Normality of water sample (equivalent to dissolved chlorine) (N_3) = ?

Normality of hypo solution (N_2) = ?

Volume of hypo solution (V_2) = burette reading

$N_2 V_2 = N_3 V_3$ will give the normality of water sample = N_3

Total chlorine residuals = N_3 Equivalent [illegible] .X35.5 g/L

Convert g/L into ppm.

Result: Amount of total residual [illegible] water = ... ppm

Scanned by CamScanner

Precautions:

1. The solution being unstable should be titrated immediately after its preparation.
2. The solution should be well shaken before each aliquot is withdrawn for titration.
3. Chlorine vapours being harmful, the solution should not be sucked into the pipette with the mouth.

Scanned by CamScanner

Experiment No. 5

To determine the concentration of dissolved oxygen from the given water sample using standard N/40 $Na_2S_2O_3$ solution.

Apparatus required: Burette, Pipette, Measuring flask, glass rod

Chemicals required: Potassium iodide solution (10%), N/40 Hypo solution ($Na_2S_2O_3$), starch solution (freshly prepared).

Theory:

The experiment is based on the oxidation of potassium iodide by the dissolved oxygen. The liberated iodine is titrated against a standard sodium thiosulphate solution using starch as final indicator. Since, oxygen dissolved in water remains present in molecular state; it is not capable of reacting with potassium iodide. An oxygen carrier such as manganese hydroxide is, therefore, used to bring about the reaction. Manganese hydroxide is produced in situ by the action of potassium hydroxide and manganous sulphate. The chemical reactions taking place are as follows:

$$MnSO_4 + 2KOH \rightarrow Mn(OH)_2 + K_2SO_4$$

$$2Mn(OH)_2 + O_2 \rightarrow 2MnO(OH)_2$$

[Basic manganese oxide]

$$MnO(OH)_2 + H_2SO_4 \rightarrow MnSO_4 + 2H_2O + [O]$$

$$2KI + H_2SO_4 + [O] \rightarrow K_2SO_4 + H_2O + I_2$$

$$I_2 + 2Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$$

(Sodium tetrathionate)

Procedure: Take 100 mL water sample in iodometric flask; avoiding, as far as possible, contact with air. Immediately add 1 mL of manganous sulphate solution from the burette and 1 mL of alkaline iodide from the other burette. Insert the stopper and shake several times. Allow the precipitate to settle half way and mix again. Repeat this process of shaking and settling at least three times. Add 1 mL of conc. sulphuric acid. Insert the stopper and shake again. Allow the yellow solution to stand for 5 min. Withdraw 100 mL of the solution. Titrate it against the standard N/40 sodium thiosulphate solution till the colour of the solution becomes faint yellow. At this stage add a few drops of starch solution. The colour turns to deep blue due to the formation of starch – iodide. Continue the titration till the solution becomes colourless.

Scanned by CamScanner

Observations:

Volume of water for each titration = 100 mL

Volume of N/40 sodium thiosulphate solution = V mL

Calculations:

100 mL of water sample ≅ V mL of N/40 $Na_2S_2O_3$ solution

Normality = V/ 100 × 40

Strength = V × 8 / 100 × 40 gm. of O_2/ litre

V × 8 / 100 × 4 × 1000 ppm

Amount of dissolved oxygen = ... ppm

Result: Dissolved oxygen = ... ppm

Precautions:

1. The solution in the iodine flask should be shaken carefully while keeping the thumb on the lid.
2. The solution should be well shaken before each aliquot is withdrawn for titration.
3. The solution in the iodine flask should not be sucked into the pipette with the mouth.

Scanned by CamScanner

Department of Applied Chemistry
Delhi Technological University

Experiment No. 6 and 7

To analyze and interpret the given IR spectrum.

Requirements: IR spectrum

Theory:

The qualitative aspects of infrared spectroscopy are one of the most powerful attributes of this diverse and versatile analytical technique. The vibrational spectrum of a molecule is considered to be a unique physical property and is characteristic of the molecule. The infrared spectrum can be used as a fingerprint for identification by the comparison of the spectrum from an "unknown" with previously recorded reference spectra. This is the basis of computer-based spectral searching. In the absence of a suitable reference database, it is possible to effect a basic interpretation of the spectrum from first principles, leading to characterization, and possibly even identification of an unknown sample.

This first principles approach is based on the fact that structural features of the molecule, whether they are the backbone of the molecule or the functional groups attached to the molecule, produce characteristic and reproducible absorptions in the spectrum. This information can indicate whether there is backbone to the structure and, if so, whether the backbone consists of linear or branched chains. Next it is possible to determine if there is unsaturation and/or aromatic rings in the structure. Finally, it is possible to deduce whether specific functional groups are present.

Interpretation:

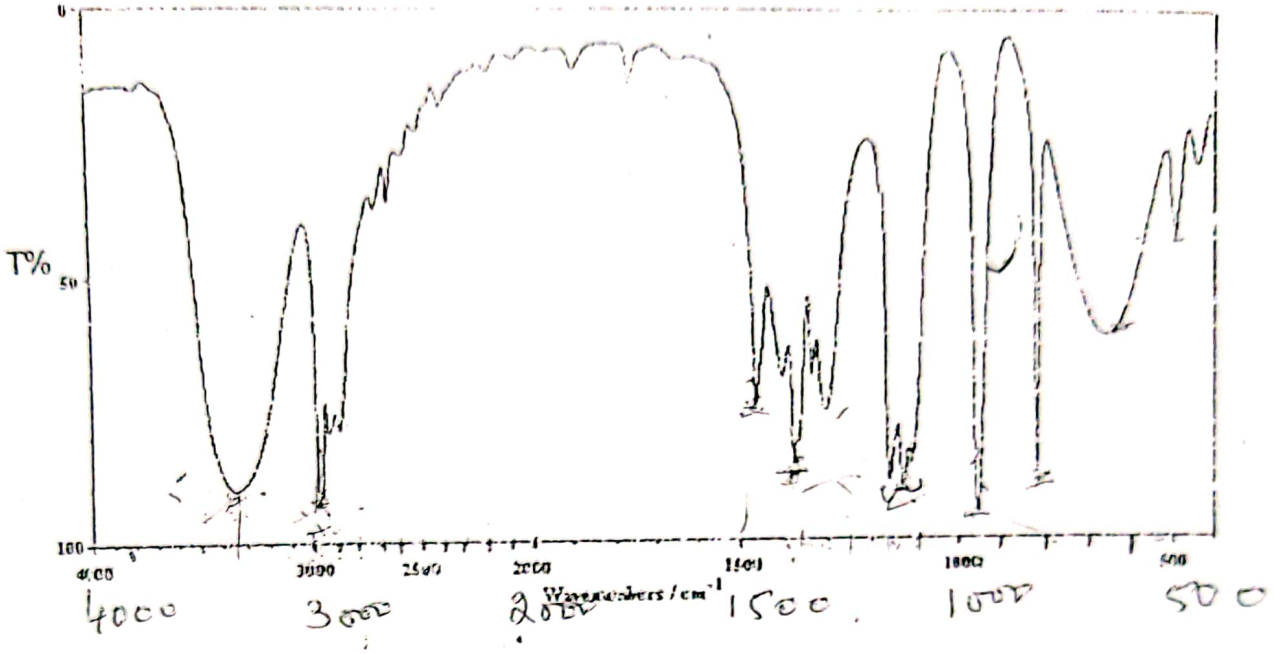
Table for Spectroscopic Data

S. No.	Wave number	Possible group	Explanation, if any

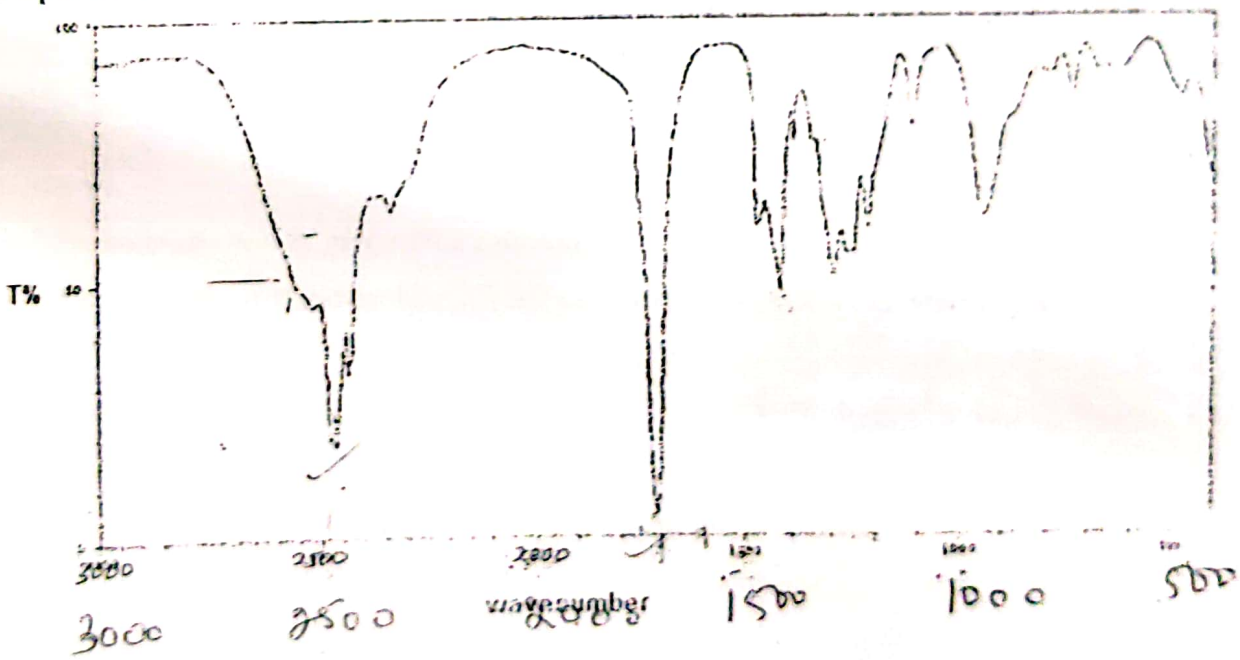
Result:

Scanned by CamScanner

IR Spectra 1



IR Spectra 2



Scanned by CamScanner

Experiment No. 8:

To determine the total alkalinity of the given water sample using methyl orange as indicator. Given standard N/40 Sodium carbonate to standardize HCl solution.

Apparatus required: Burette, pipette, conical flask, funnel, conical flask, measuring cylinders.

Chemicals required: Sodium carbonate (N/40) solution, HCl solution, Methyl orange (M) and water sample.

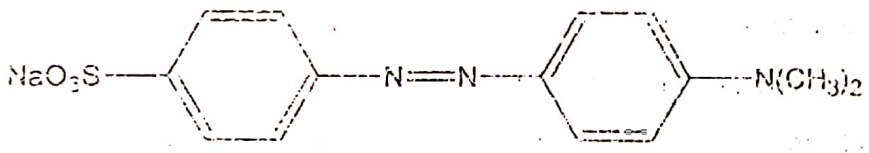
Theory

The ionic equations involved for this reaction are:

(iv) $OH^- + H^+ \longrightarrow H_2O$

(v) $CO_3^{-2} + H^+ \longrightarrow H_2O + CO_2$

This is an example of neutralization titration. The titration is based upon the specific pH range of the indicator. Methyl orange gives red colour in pH range 4.4 - 3.6.



Methyl Orange

Procedure:

I. ***Standardization of given HCl solution***

(i) Take 10 mL of standard Na_2CO_3 with the help of pipette into a conical flask. Add 1-2 drops of methyl orange.

(ii) Run the acid solution from the burette into conical flask drop wise with constant shaking of solution.

(iii) Find the end point when light yellow colour solution turns red. Note volume of acid used. Repeat the steps till you get at least two concordant readings

II. ***Titration of given water sample with HCl solution***

(i) Pipette out 10 mL of given water sample into a conical flask. Add 2-3 drops of methyl orange indicator

(ii) Add HCl solution from burette into the conical flask with constant shaking.

Scanned by CamScanner

(iii) Titrate with acid till yellow coloured solution turns red (end point)

Observations:

Volume of Na_2CO_3 solution taken for each titration = 10 mL

Observation table is similar to that for Exp No 01.

Table: Burette readings for the titration of given water sample vs HCl

S. No.	Initial Reading	Final Reading	Vol. of HCl used

Calculations:

Normality of Na_2CO_3 solution = N_{Na2CO3}

Calculation of Normality of HCl is similar to Exp No 01.

To calculate normality of given water solution, apply normality equation

$$N_{HCl} \times V_{HCl} = N_{water\ sample} \times V_{water\ sample}$$

or

$$N_{water\ sample} = N_{HCl} \times V_{HCl} / V_{water\ sample}$$

Result:

Total alkalinity of the given solution (in terms of $CaCO_3$) .....

Precautions:

a) Shake the titration flask continuously during adding the solution from the burette.

b) Keep your eye in level with the liquid surface while taking the burette reading or while reading the pipette or measuring flask etc.

c) Always read lower meniscus in case of colourless solution.

Scanned by CamScanner

Experiment No. 9

To determine the number of molecules of water of crystallization in washing soda ($Na_2CO_3.nH_2O$) provided 0.1 N HCl.

Apparatus required: Burette, Pipette, Measuring flask, glass rod

Chemicals required: Sodium carbonate solution, HCl solution Methyl orange.

Theory:

Similar to exp 1 to 3.

Reactions: $Na_2CO_3.nH_2O + 2HCl \rightarrow 2NaCl + (n+1)H_2O + CO_2$

Ionically: $CO_3^{2-} + 2H^+ \rightarrow H_2O + CO_2$

Indicator: Methyl orange

End point: Yellow to red (acid solution in the burette)

Procedure

(i) Prepare a standard solution of $Na_2CO_3.nH_2O$.

(ii) Rinse and fill the burette with given 0.1N HCl solution.

(iii) Pipette out 20 ml of the $Na_2CO_3.nH_2O$ solution into a conical flask and add 1-2 drops of methyl orange indicator.

(iv) Add HCl solution from the burette till a permanent red colour is obtained. Repeat till three concordant reading are obtained.

Observations:

Volume of Na_2CO_3 solution taken for each titration = 20 mL

Table: Burette readings for the titration of Na_2CO_3 vs HCl

S. No.	Initial Reading	Final Reading	Vol. of HCl used

Calculations:

Let V be the volume of HCl required to neutralize 20 ml of washing soda solution.

Applying normality equation, the normality of washing soda = $0.1 \times V/20 = 0.005V$

Molecular-weight of the washing-soda = 106+18n

Equivalent weight of $Na_2CO_3.nH_2O$ = (106+18n)/2

Strength of washing soda solution = normality × Eq. Wt.

Scanned by CamScanner

$$= 0.005V\ (53+9n)\ g/L$$

But the actual strength of washing soda = $4(W_2-W_3)/53$ g/L (from weighing)

$$\therefore 0.005V\ (53+9n) = 4(W_2-W_3)/53$$

From this equation 'n' can be calculated.

Result: The number of molecules of water of crystallization in washing-soda = ...

Precautions:

a) Shake the titration flask continuously during adding the solution from the burette.

b) Keep your eye in level with the liquid surface while taking the burette reading or while reading the pipette or measuring flask etc.

c) Always read lower meniscus in case of colourless solution.

Scanned by CamScanner

Experiment No. 10

To determine the molecular weight of given polymer by Viscometer.

Apparatus required: Viscometer, Measuring flask, glass rod

Chemicals required: Polyvinyl alcohol, Water

Theory: The viscosity of a polymer solution is related to the molecular weight, M, of the polymer by the equation:

$$\eta_{sp} / C = \eta / \eta_0 - 1 = KM^{\alpha} \quad \text{.....(1)}$$

Where η_{sp} is the specific viscosity of the solution, η_0 and η the viscosities of solvent and solution respectively, C the weight of polymer (g) in 100 mL of the solution and K and α are constants. This equation is only valid for low concentrations of a linear polymer, dissolved in a good solvent, where there is no association. It is thus necessary to extrapolate the curve of C against η_{sp} / C to C = 0; the intercept is the intrinsic viscosity $|\eta|$. Thus, in the limit, eq (1) becomes

$$|\eta| = KM^{\alpha} \quad \text{.....(2)}$$

In general, the value of K depends on the type of polymer, the solvent and the temperature while α is a function of the geometry of the molecule. The viscometric method of determining the molecular weight is very convenient and gives accurate result if K and α are known. These are obtained from the samples of polymers of known molecular weight, in which the m. wt. has been determined by alternative methods.

Reagents and apparatus: Viscometer (Ostwald's) stop watch, PVA, water.

Procedure:

1. Prepare a solution containing amount 0.8 gm of PVA in 100 mL of water. This is 0.8% solution of PVA.
2. Similarly, prepare the solution of different conc. by diluting the above solution.
3. Pipette out 20 mL of pure water in the bulb B of the viscometer which should be held vertically.
4. Through a rubber tube attached to upper arm of bulb suck up the solvent until it rises above the mark X. Allow the solvent to fall freely through a capillary upto

Scanned by CamScanner

the mark. Start the stop watch and note the time T1 for the flow of liquid from mark X to mark Y.

5. Repeat to get 3-4 readings and take the mean as the flow time t_o. Remove the solvent.

6. Pipette out the one of the solution prepared above and determine the flow time as above. Repeat to get three readings and take their mean.

7. Similarly determine the flow time for solutions of different concentrations.

Observations:

Solvent used is water.

Value of K = 20×10^{-5}

Value of α = 0.76 at 25 °C

Observation Table: Viscosity Measurements

S. No.	Concentration of Polymer Solution	Time of flow	Relative viscosity $\eta_r = t/t_o$	Specific viscosity $\eta_{sp} = \eta_r - 1$	Reduced viscosity $\eta_{red} = \eta_{sp}/c$
1	Pure solvent	t_o =			
2	0.8%	t_1			
3	0.4%	t_2			
4	0.2%	t_3			
5	0.1%	t_4			
6	0.05	t_5			

Calculations: Plot the graph between η_{red} and concentration and extrapolate the graph to zero concentration.

$[\eta] = KM^{\alpha}$, by substituting the values of $[\eta]$, K and α, M can be calculated

Using logarithm

$\log [\eta] = \log K + \alpha \log M$

$\log M = \log [\eta] - \log K / \alpha$

Scanned by CamScanner

$$M = \text{Antilog}\{\log[\eta] - \log K/\alpha\}$$

Precautions:

1. All measurements should be made using a constant volume of liquid.
2. Viscometer should be kept vertical.
3. Suction should be done carefully.
4. There should not be any air bubbles in the viscometer.

Scanned by CamScanner

Experiment No. 11:

To determine the structure of an unknown compound A using Spectroscopic data.

Given: The Molecular Formula for the unknown compound: $C_9H_{10}O$

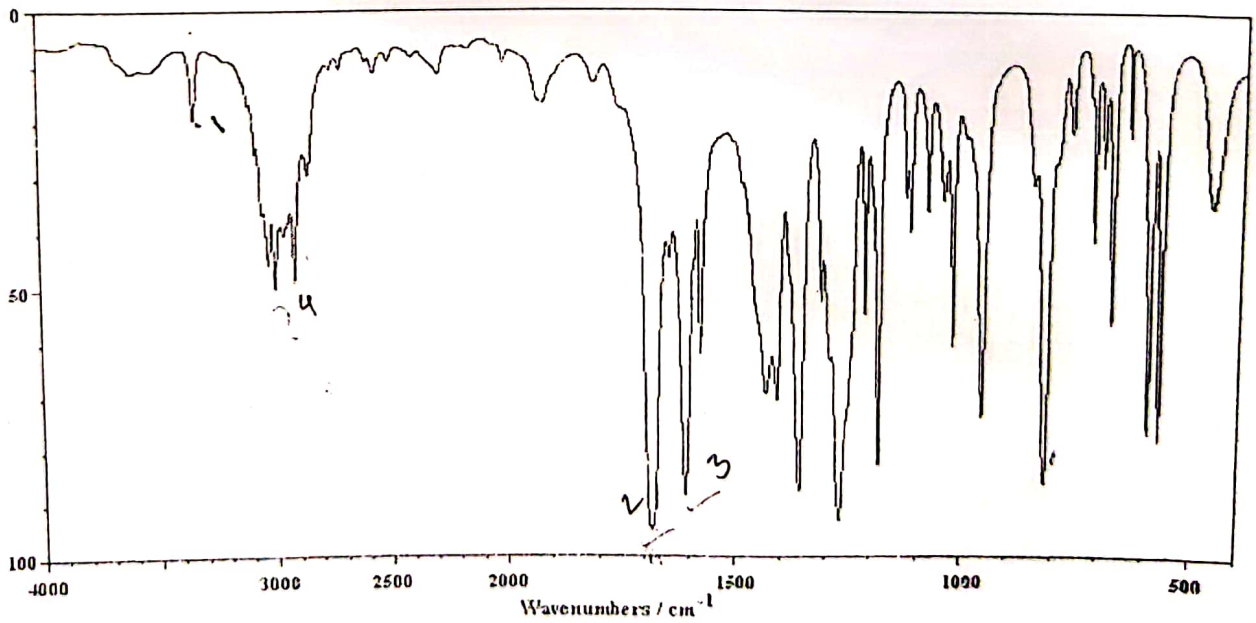
Theory:
In a modern structure analysis, the powerful spectroscopic methods of IR and NMR play a major role. In order to determine the structure of an unknown compound, it may be required to combine a variety of spectroscopic data.

Infra Red (IR) Spectroscopy: Infrared spectrometry is especially useful in identifying the functional groups in unknown compounds.

^{1}H-NMR Spectroscopy: One of the most important pieces of information in a ^{1}H NMR spectrum is the chemical shift of the various kinds of protons in the sample. Usually, it is difficult or even impossible to decipher structural information from a ^{1}H NMR spectrum without paying close attention to the chemical shifts of the NMR peaks.

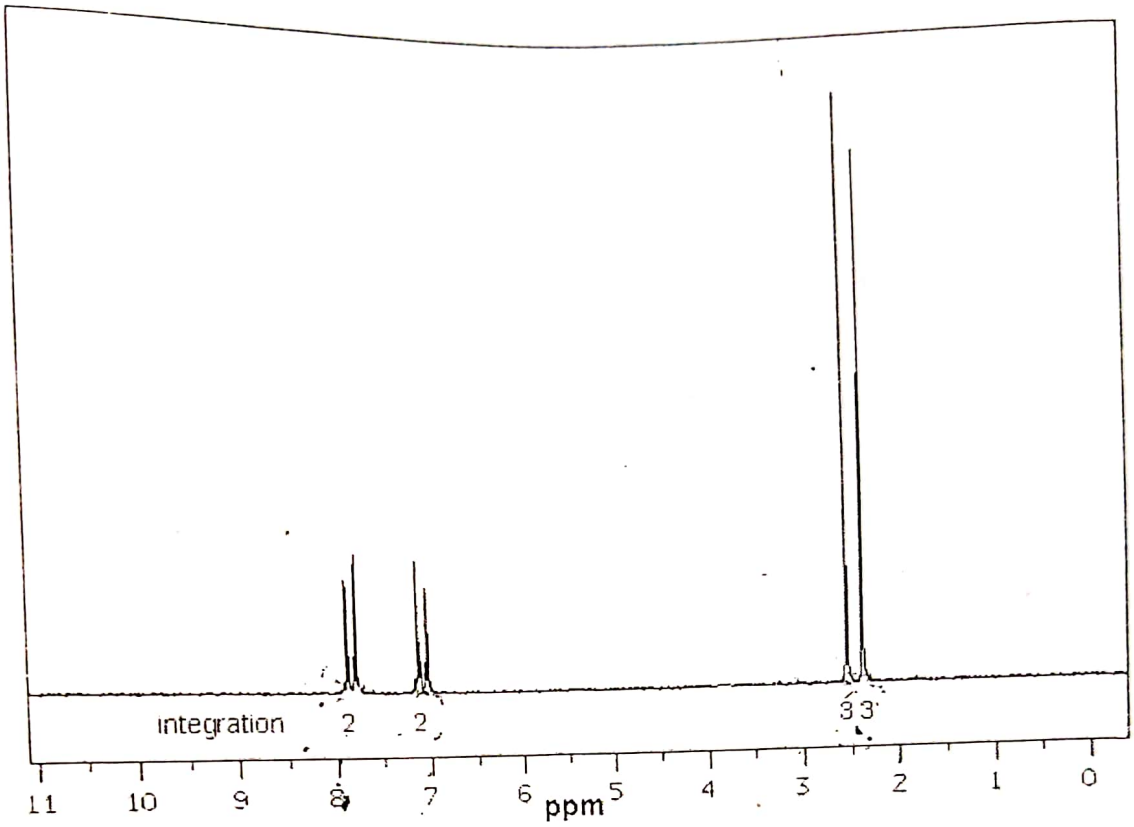
Even with NMR analysis, however, it can be useful to see if your conclusions fit with other observations, such as those from infrared and mass spectroscopy and chemical reactivity. For example, if your NMR spectrum suggests that the unknown may be a carboxylic acid, the exceptionally broad OH and the sharp, intense C=O stretching vibrations in its infrared (IR) spectrum can provide important confirming evidence.

IR Spectrum



Scanned by CamScanner

N.M.R. Spectrum



Observations:

Table: Spectroscopic Data

IR Spectrum		NMR Spectrum	
1.		1.	
2.		2.	
3.		3.	
4.		4.	

Analysis & Interpretation

Result:

Scanned by CamScanner

Department of Applied Chemistry
Delhi Technological University

Experiment No. 12:

To determine the structure of an unknown compound **B** using Spectroscopic data.

Given: The Molecular Formula for the unknown compound: $C_8H_{10}O_2$

Theory:

In a modern structure analysis, the powerful spectroscopic methods of IR and NMR play a major role. In order to determine the structure of an unknown compound, it may be required to combine a variety of spectroscopic data.

Infra Red (IR) Spectroscopy: Infrared spectrometry is especially useful in identifying the functional groups in unknown compounds.

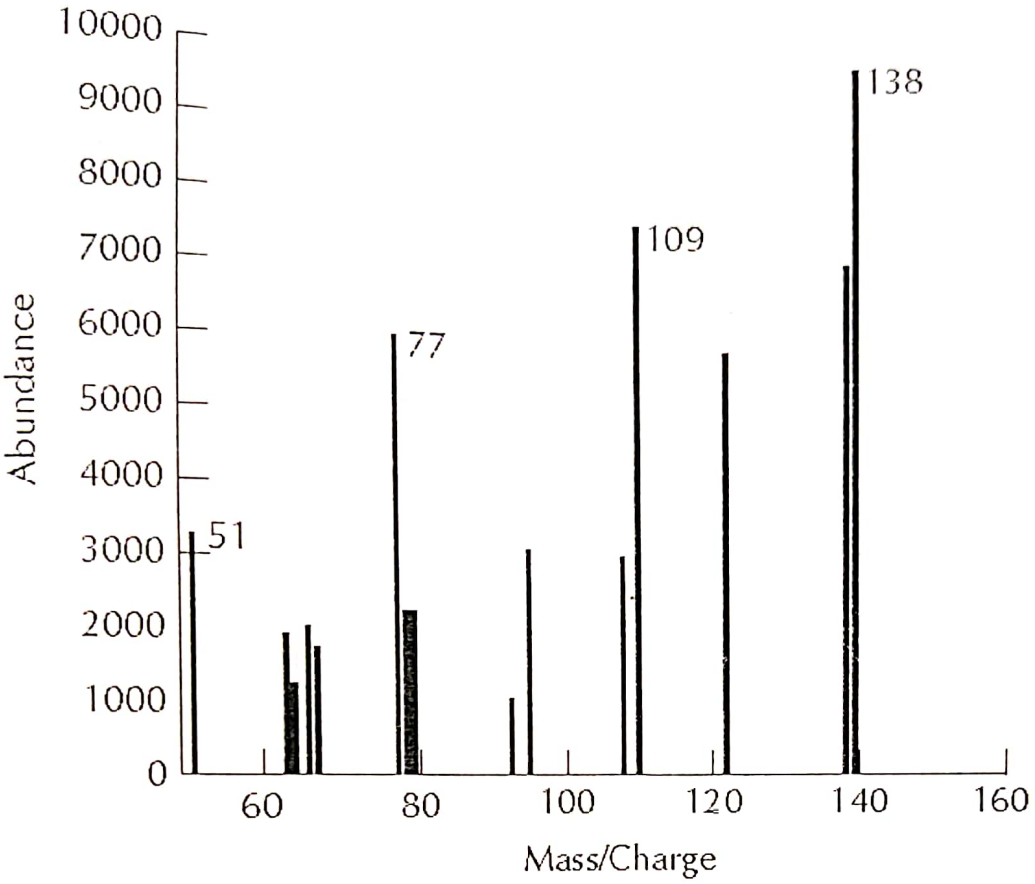
^{1}H-NMR Spectroscopy: One of the most important pieces of information in a ^{1}H NMR spectrum is the chemical shift of the various kinds of protons in the sample. Usually, it is difficult or even impossible to decipher structural information from a ^{1}H NMR spectrum without paying close attention to the chemical shifts of the NMR peaks.

Even with NMR analysis, however, it can be useful to see if your conclusions fit with other observations, such as those from infrared and mass spectroscopy and chemical reactivity. For example, if your NMR spectrum suggests that the unknown may be a carboxylic acid, the exceptionally broad OH and the sharp, intense C=O stretching vibrations in its infrared (IR) spectrum can provide important confirming evidence.

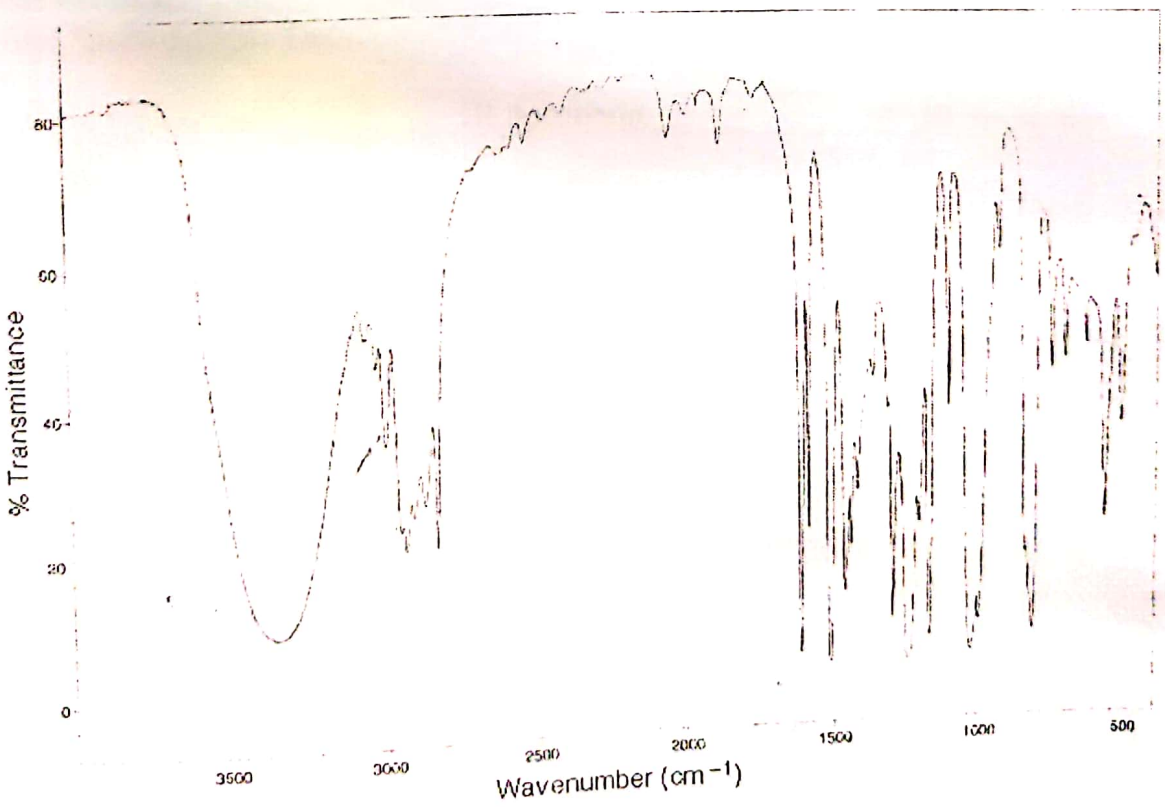
Mass Spectrometry: The most obvious information from a compound's mass spectrum is its molecular mass. The molecular ion peak is often the peak at the greatest mass in the spectrum. In practice, however, it can be tricky to rely on this generalization, because impurities in the spectrometer may produce small peaks of higher molecular mass. Then it becomes difficult to know exactly what the molecular mass of your compound really is. In some compounds, the molecular ion peak can be negligibly small because of its extensive fragmentation in the electron beam.

Scanned by CamScanner

Mass Spectrum

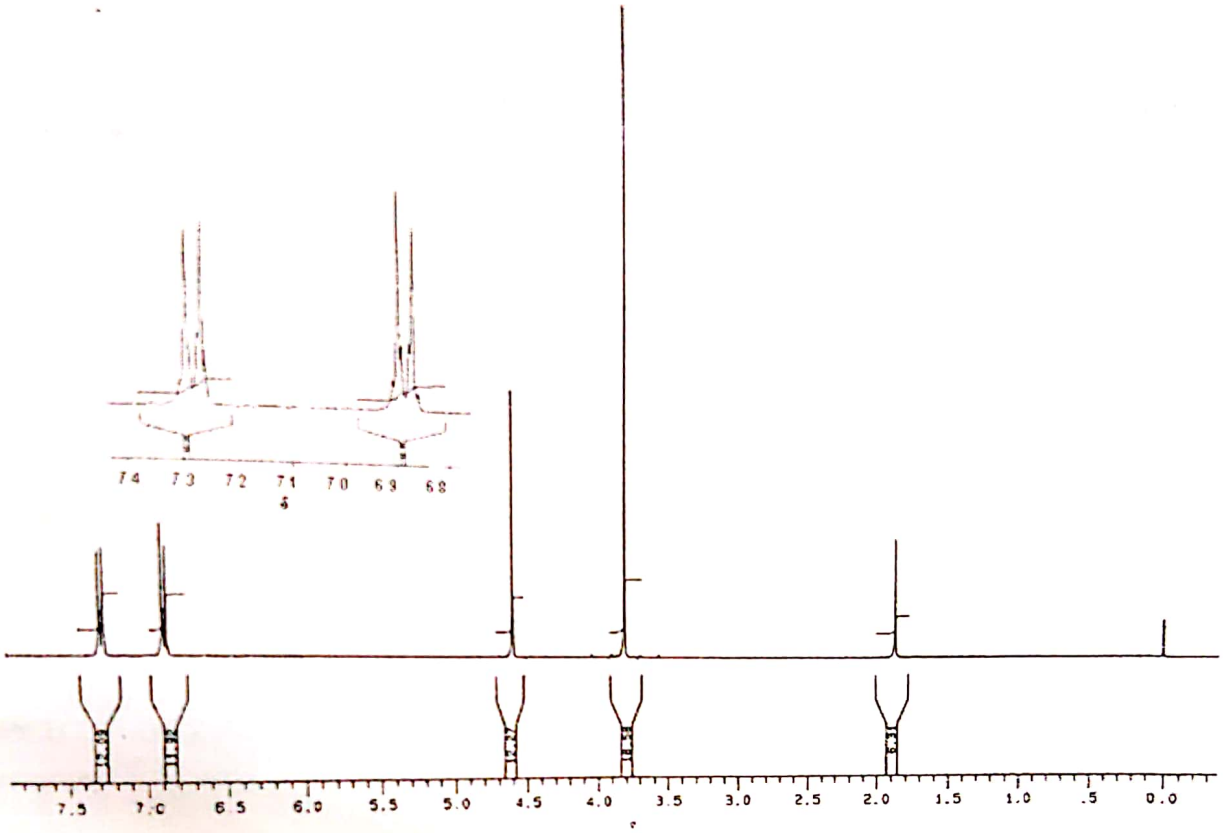


IR Spectrum



Scanned by CamScanner

NMR Spectrum



Observations:

Table: Spectroscopic Data

Mass Spectrum		IR Spectrum		NMR Spectrum	
1.		1.		1.	
2.		2.		2.	
3.		3.		3.	
4.		4.		4.	

Analysis & Interpretation

Result:

Scanned by CamScanner

Infrared Spectroscopy Absorption Table

Last updated: 22:36, 29 Nov 2014

The following table lists **infrared spectroscopy absorptions** by frequency regions.

			4000-3000 cm^{-1}			
3700-3584	medium	sharp	O-H	stretching	alcohol	free
3550-3200	strong	broad	O-H	stretching	alcohol	intermolecular bonded
3500 3400	medium		N-H	stretching	primary amine	
3400-3300 3330-3250	medium		N-H	stretching	aliphatic primary amine	
3350-3310	medium		N-H	stretching	secondary amine	
3300-2500	strong	broad	O-H	stretching	carboxylic acid	usually centered on 3000 cm^{-1}
3200-2700	weak	broad	O-H	stretching	alcohol	intramolecular bonded
3000-2800	strong	broad	N-H	stretching	amine salt	
			3000-2500 cm^{-1}			
3333-3267	strong	sharp	C-H	stretching	alkyne	
3100-3000	medium		C-H	stretching	alkene	
3000-2840	medium		C-H	stretching	alkane	
2830-2695	medium		C-H	stretching	aldehyde	doublet
2600-2550	weak		S-H	stretching	thiol	
			2400-2000 cm^{-1}			
2349	strong		O=C=O	stretching	carbon dioxide	
2275-2250	strong	broad	N=C=O	stretching	isocyanate	
2260-2222	weak		C≡N	stretching	nitrile	
2260-2190	weak		C≡C	stretching	alkyne	disubstituted
2175-2140	strong		S-C≡N	stretching	thiocyanate	
2160-2120	strong		N=N=N	stretching	azide	
2150			C=C=O	stretching	ketene	
2145-2120	strong		N=C=N	stretching	carbodiimide	
2140-2100	weak		C≡C	stretching	alkyne	monosubstituted
2140-1990	strong		N=C=S	stretching	isothiocyanate	
2000-1900	medium		C=C=C	stretching	allene	
2000			C=C=N	stretching	ketenimine	

Scanned by CamScanner

2000-1650 cm^{-1}					
2000-1650	weak	C-H	bending	aromatic compound	overtone
1870-1540 cm^{-1}					
1818 1750	strong	C=O	stretching	anhydride	
1815-1785	strong	C=O	stretching	acid halide	
1800-1770	strong	C=O	stretching	conjugated acid halide	
1775 1720	strong	C=O	stretching	conjugated anhydride	
1770-1780	strong	C=O	stretching	vinyl / phenyl ester	
1760	strong	C=O	stretching	carboxylic acid	monomer
1750-1735	strong	C=O	stretching	esters	6-membered lactone
1750-1735	strong	C=O	stretching	δ-lactone	γ: 1770
1745	strong	C=O	stretching	cyclopentanone	
1740-1720	strong	C=O	stretching	aldehyde	
1730-1715	strong	C=O	stretching	α,β-unsaturated ester	or formates
1725-1705	strong	C=O	stretching	aliphatic ketone	or cyclohexanone or cyclopentenone
1720-1706	strong	C=O	stretching	carboxylic acid	dimer
1710-1680	strong	C=O	stretching	conjugated acid	dimer
1710-1685	strong	C=O	stretching	conjugated aldehyde	
1690	strong	C=O	stretching	primary amide	free (associated: 1650)
1690-1640	medium	C=N	stretching	imine / oxime	
1685-1666	strong	C=O	stretching	conjugated ketone	
1680	strong	C=O	stretching	secondary amide	free (associated: 1640)
1680	strong	C=O	stretching	tertiary amide	free (associated: 1630)
1650	strong	C=O	stretching	δ-lactam	γ: 1750-1700 β: 1760-1730
1670-1600 cm^{-1}					
1678-1668	weak	C=C	stretching	alkene	disubstituted (trans)
1675-1665	weak	C=C	stretching	alkene	trisubstituted
1675-1665	weak	C=C	stretching	alkene	tetrasubstituted
1662-1626	medium	C=C	stretching	alkene	disubstituted (cis)
1658-1648	medium	C=C	stretching	alkene	vinylidene
1650-1600	medium	C=C	stretching	conjugated alkene	
1650-1580	medium	N-H	bending	amine	
1650-1566	medium	C=C	stretching	cyclic alkene	
1648-1638	strong	C=C	stretching	alkene	monosubstituted
1620-1610	strong	C=C	stretching	α,β-unsaturated ketone	
1600-1300 cm^{-1}					
1550-1500 1372-1290	strong	N-O	stretching	nitro compound	
1465	medium	C-H	bending	alkane	methylene group
1450 1375	medium	C-H	bending	alkane	methyl group
1390-1380	medium	C-H	bending	aldehyde	
1385-1380 1370-1365	medium	C-H	bending	alkane	gem dimethyl
1400-1000 cm^{-1}					
1440-1395	medium	O-H	bending	carboxylic acid	
1420-1330	medium	O-H	bending	alcohol	
1415-1380 1200-1185	strong	S=O	stretching	sulfate	
1410-1380 1204-1177	strong	S=O	stretching	sulfonyl chloride	
1400-1000	strong	C-F	stretching	fluoro compound	
1390-1310	medium	O-H	bending	phenol	
1372-1335 1195-1168	strong	S=O	stretching	sulfonate	
1370-1335 1170-1155	strong	S=O	stretching	sulfonamide	
1350-1342 1165-1150	strong	S=O	stretching	sulfonic acid	anhydrous hydrate: 1230-1120
1350-1300 1160-1120	strong	S=O	stretching	sulfone	
1342-1266	strong	C-N	stretching	aromatic amine	

Scanned by CamScanner

Table of IR Absorptions

Functional Group	*Characteristic Absorption(s)* (cm^{-1})	*Notes*
Alkyl C-H Stretch	2950 - 2850 (m or s)	Alkane C-H bonds are fairly ubiquitous and therefore usually less useful in determining structure.
Alkenyl C-H Stretch Alkenyl C=C Stretch	3100 - 3010 (m) 1680 - 1620 (v)	Absorption peaks above 3000 cm^{-1} are frequently diagnostic of unsaturation
Alkynyl C-H Stretch Alkynyl C≡C Stretch	~3300 (s) 2260 - 2100 (v)	
Aromatic C-H Stretch Aromatic C-H Bending Aromatic C=C Bending	~3030 (v) 860 - 680 (s) 1700 - 1500 (m,m)	
Alcohol/Phenol O-H Stretch	3550 - 3200 (broad, s)	See "Free vs. Hyrdogen-Bonded Hydroxyl Groups" in the Introduction to IR Spectra for more information
Carboxylic Acid O-H Stretch	3000 - 2500 (broad, v)	
Amine N-H Stretch	3500 - 3300 (m)	Primary amines produce two N-H stretch absorptions, secondary amides only one, and tetriary none.
Nitrile C≡N Stretch	2260 - 2220 (m)	
Aldehyde C=O Stretch Ketone C=O Stretch Ester C=O Stretch Carboxylic Acid C=O Stretch Amide C=O Stretch	1740 - 1690 (s) 1750 - 1680 (s) 1750 - 1735 (s) 1780 - 1710 (s) 1690 - 1630 (s)	The carbonyl stretching absorption is one of the strongest IR absorptions, and is very useful in structure determination as one can determine both the number of carbonyl groups (assuming peaks do not overlap) but also an estimation of which types.
Amide N-H Stretch	3700 - 3500 (m)	As with amines, an amide produces zero to

Scanned by CamScanner