Chapter II

Instruments and Methods

This chapter discusses the instrumentation and methods used in performing all the study, starting with voltammetry and ending at magnetic resonance. The chapter is a detailed explanation of all the instruments used in the study.
2.1 Introduction

This chapter is the discussion of the instruments and methods used in the experiments performed. The explanation and diagrammatic representation of instruments is also provided. The methods regarding voltammetry and instrumentation of spectrophotometry, pH metery has been explained. FT-IR instrumentation and NMR instrumentation has been explained in with theoretical explanation.

2.2 Polarography

Polarography is an electro-analytical technique developed by Jaroslav Heyrovsky in 1922 for which he received Noble Prize in Chemistry. It is an electrolytic method in which a dropping mercury electrode (dme) is used as indicator electrode/ working electrode and saturated calomel electrode as reference electrode. In this method a current voltage relationship curve is obtained by varying the potential and observing the current which is known as Polarogram.

2.2.1 Polarogram and its significance:

Polarogram is the graphical representation of current voltage relationship for a specific redox system. The polarograms consist of three main parts as indicated in the figure

![A Typical Polarogram](image)

*Figure 2.1 A Typical Polarogram [2]*
2.2.2 Migration current:

As indicated in the figure the portion of graph represented by (A) is known as migration current. This current is due to migration of electroactive species to indicator electrode. These species may be present as impurities in the solution and when potential is applied, the primary current present is known as migration current. This current is not due to species to be analyzed, it is only due to impurities which quickly get reduced at dropping mercury electrode.

2.2.3 Diffusion current:

This current is due to diffusion of the electroactive species through the solution medium. This current is represented in the Polarogram by steep increase in current as seen in figure 2.1 the letter (B) indicates the diffusion current. When the potential is increased for the analyzing solution the current goes on increasing a little and a time reaches when current increases rapidly. The rapid increasing current is diffusion current. This current is due to the electroactive substance in solution medium which drift towards cathode while potential is applied it diffuses through the solution medium gets reduced at the dropping mercury electrode. These electroactive substances either form an amalgam with mercury or settle at the bottom of the cell or the ions get reduced at electrode and get back in to the solution.

\[ M^{n+} + n \text{e}^- \leftrightarrow M(\text{Hg}) \]

This diffusion current is used for the quantitative analysis of the species and is given by the Ilkovic equation

\[ I_d = 0.732 \, n \, F \, C \, D^{1/2} \, m^{2/3} \, t^{1/6} \]

Where (m) is the mass of mercury flow per second, (D) is diffusion coefficient, (t) is drop time, (C) is molar concentration and (F) is Faraday constant.

2.2.4 Limiting current:

In the Figure 2.1 letter (C) represents the limiting current when the potential is applied to the solution and current goes on increasing and a stage reaches when there is no further increase in the current. At that point all the electroactive species are diffuse to
the cathode and further increase in potential does not affect the diffusing electroactive species.

2.2.5 Electrodes used in polarography:

There are two main types of electrodes used in polarography that is working/ indicator electrode and reference electrode. The reference electrode may be internal which is in direct contact with the analyzing solution. The external reference electrode is mainly connected to the solution cell through a salt bridge. The electrodes used in polarography are briefly explained as under.

2.2.6 Dropping mercury electrode (dme):

In this electrode, mercury is allowed to flow through a thin capillary from a reservoir on a height and drops of mercury falling continuously through the orifice of capillary. The drop rate is regulated by adjusting the height of reservoir. The electrode is used as a working electrode in classical D.C polarography and the drop rate is regulated between 20-22 drops per minute. There are many advantages of using this electrode as working electrode since, each time a new surface is available for the electroreduction /oxidation and the hydrogen over potential for mercury is high more than 1.2V hence it can be used to analyze the solutions at higher potentials.

2.2.7 Hanging drop mercury electrode:

This is also a type of mercury electrode and is used as an indicator electrode. In this electrode a mercury drop is allowed to hang from a thin capillary, the drop is in contact with solution to be analyzed. The hanging drop in this electrode is mixed with amalgamated form of gold or silver to increase the analyzing properties.

2.2.8 Platinum electrode:

This electrode is used an internal reference electrode as its hydrogen overvoltage potential is low and cannot be used as working electrode at higher potential it is mainly a platinum wire attached to circuit providing potential.

2.2.9 Saturated calomel electrode (SCE):

This electrode is vastly used as a reference electrode. It has various shapes commonly available. The bottle type laboratory made saturated calomel electrode is made by
taking pure mercury in a bottle and having top layer of calomel. The bottle is filled with saturated solution of KCl, and connected to a salt bridge and a connecting for contact. The electrode mainly serves as an external reference electrode.

2.2.10 Carbon electrodes:

These electrodes are solid in nature and are used as working electrodes. They have high over potentials and low resistances hence are good materials for the electrode surface which easily provide a reproducible surface. The electrodes in this category are glassy carbon electrode, carbon paste electrode and graphite electrode. These electrodes are also used in other voltammetric methods also.

2.2.11 Half-wave reduction potential and its significance:

Diffusion current is an important aspect in Polarographic estimation as the current obtained is used for quantitative estimation by the Ilkovic equation. The half-wave reduction potential is the potential at which diffusion current is half (I_d/2). This is obtained current voltage polarograms. This potential has important qualitative applications and is used to identify the different compounds in solution medium. This potential is specific for each electro reducible species.

2.2.12 Polarographic instrumentation:

Classical D.C Polarographic instrumentation consists of a polarographic cell with working electrode as dropping mercury electrode and a saturated calomel electrode as the reference electrode. The potential is applied between these electrodes by a D.C battery, and a rheostat is used to vary the potential. The instrumentation also consists of moving coil galvanometer to observe the current deflection and a scale to measure diffusion limited nanocurrent. The modern Polarographs are mostly digitally built they only consists of polarographic cell with electrodes and a digital screen with knobs to adjust potential and other various parameters.

2.2.13 Polarographic analysis:

Analysis by polarography is performed by preparing the analyzing solution in a 100 fold supporting, whose main function is to carry migration current. The solution is also added with 0.004-0.04% gelatin which suppresses the polarographic kinks arising due to streaming motion of the solution.
The solution prepared is kept in a closed polarographic cell and nitrogen gas is passed for at least 10 minutes so as to remove the dissolved oxygen which may interfere in the analysis process by generating oxidation curves in polarograms. Electrodes are inserted in the polarographic cell and potential is varied. The diffusion limited nanocurrent is measured digitally in a polarograph.

2.3 Hydrodynamic voltammetry

It is an allied technique of polarography in this method platinum electrode is rotated at high speed by a motor which puts solution is motion. The rotating platinum electrode replaces dropping mercury electrode in classical polarography. The saturated calomel electrode is used as reference electrode and the assembly is applied with small potential between electrodes, the electro active species present in solution get oxidized and the current generated is observed as deflections by a moving coil galvanometer light spot on scale. The reactions occurring at rotating platinum electrode are as

\[
\begin{align*}
X_2 + 2e^- & \rightarrow \quad 2X^- & \text{At the RPE} \\
2Hg + 2Cl^- & \rightarrow \quad Hg_2Cl_2 + 2e^- \quad \text{At the SCE} \\
X_2 + 2Hg + 2Cl^- & \leftrightarrow \quad 2X^- + Hg_2Cl_2
\end{align*}
\]

2.3.1 Hydrodynamic voltammetry instrumentation:

The instrumentation is almost same as that of polarography with only few differences. The instrumentation includes rotating platinum electrode (RPE) attached to a pure A.C motor and saturated calomel electrode (SCE) as reference electrode. The other parts are moving coil galvanometer, scale and a D.C battery. The potential applied between two electrodes is about 0.01-0.02 V and is varied by rheostat, the RPE is rotated at 650 rpm and the deflection of moving coil galvanometer is controlled by shunt.

2.3.2 Kinetic investigation by hydrodynamic voltammetry:

Hydrodynamic voltammetry is an important technique to study fast reaction kinetics. The techniques uses voltammetric principles. The rotating platinum electrode is used
as a working electrode for the observation of the electro active species. To observe the kinetics of fast reaction at least one of the reactants or products must be electroactive.

a) Calibration; the electroactive reactant solution is prepared in varying concentrations and galvanometer deflection is noted for each solution, after taking it in a H.V cell. The data obtained is used to plot the calibration curve which is further used to obtain the unknown concentration at any galvanometer deflection.

b) The reactant solutions of the reaction to be studied are prepared and the electroactive reactant is taken in the H.V cell and the deflection is adjusted by the shunt to a maximum value for the higher concentration. To this, the other reactant is, the reactants start reacting and varying galvanometer deflections are noted with time. The data obtained is used to calculate the specific reaction rates and other various kinetic parameters.

2.4 pH metery

pH is an abbreviation of “pondus Hydrogenii”. This term was first coined by S.P.L Sorensen in 1909 to represent small hydrogen ion concentration. Most natural processes in this world are pH dependent the chemical process, biological process and many more. The effect of pH is also pronounced on the color observation, the dyes which are mostly dependent on the pH have varying color observations that mean these dyes are showing a unique color in one pH solution and same dye shows different color in another pH solution. The pH of blood is regulated at 7.4 by the help of natural blood buffers and any change in the natural blood buffer system may give rise to diseases.

pH is the measure of acidity or basicity of aqueous solutions. The acidity is due to presence of [H+] ions in the solution and basicity is due to presence of [OH⁻] ions in the solution. Acidic substances furnish [H⁺] ions in the solution and bases furnish OH⁻ in the solution

\[
\text{Acid; } \quad HA \quad \rightarrow \quad H^+ + A^- \\
\text{Base; } \quad BOH \quad \rightarrow \quad OH^- + B^+
\]
Where $A^-$ is an anion and $B^+$ is a cation. The pH of the solution is given by the negative logarithm of activity of hydrogen ions

$$pH = - \log_{10} [a_{H^+}]$$

Which can be also be written as hydrogen ions concentration in the solution

$$pH = - \log_{10}[H^+]$$

The pH of the neutral solution is 7 meaning that the solution is neither acidic nor basic any change above or below the pH scale of 7 will make solution basic or acidic. The increase in the pH of the solution above 7 makes solution basic due increase in the $OH^-$ ions in the solution and decrease in the pH below 7 makes the solution acidic due to $H^+$ ions.

2.4.1 pH measurement:

The measurement of pH is done by pH sensitive electrode mainly by the glass electrode. The potential is developed between glass electrode and reference electrode due to acidity of the solutions and this potential difference is represented in the form of pH. The potential of most pH meters is 0 (mV) at 7 pH.

The glass electrode acts as electrochemical sensing for pH and has been successful in determining the pH of the solutions. This electrode consists of a very thin glass bulb up to 0.1mm in thickness. The electrode is filled with Ag/AgCl electrolyte solution and excess of $Cl^-$ is filled to maintain constant potential as this acts as reference electrode.

Glass is an amorphous mixture of SiO$_2$ and when this electrode is inserted in the solution some groups of SiO$^-$ get converted as

$$SiO^- + H_3O^+ \rightarrow SiOH^+ + H_2O$$

The exchange of hydronium ions on the surface puts the potential barrier on the glass bulb surface. This potential difference is represented by

$$E_{ext} - E_{int} = RT / 2.303 F \times \log a[H3O]^+$$

Where R is gas constant having value of 8.314 J mol$^{-1}$K$^{-1}$
T is temperature in Kelvin.

F is Faraday constant with value 96485.36, a[H30]⁺ is the activity of hydronium ion. At the temperature of 30°C the value of RT / 2.303 F is up to 0.060V. The solution inside the bulb maintains the potential constant and the potential developed on the membrane surface is measured by pH meter. The potential developed on the glass membrane is given by

$$E_{glass\ electrode} = E^0 + \frac{RT}{2.303} \times \log a[H30]^+$$

$E^0$ represents the sum of potentials inside the bulb, potential change on the glass surface is about 0.60 mV for one unit change in pH.

The glass electrode is still the best electrode for the determination of pH but certain limitations still persist. The problems in determining strong alkalis can damage the electrode surface, sodium and potassium ions interference cannot be neglected, they have small size and exchange with proton. The protein determination has also damaging effect on the surface of electrode. To remove these problems modern electrodes are introduced to determine the pH of these solutions.

2.5 UV-Visible Spectroscopy

Spectroscopy is an important tool of modern laboratory and the job of analyzing the samples is done very easily. The different spectrosopes have been developed from time to time and each spectroscopy is based on the absorption of electromagnetic radiations. Various spectroscopes are named on the specific regions of electromagnetic spectrum.

2.5.1 Electromagnetic spectrum:

The natural electromagnetic spectrum with sun as source consists of a broad range of electromagnetic radiations which are arranged into different regions on the basis of their frequency/wavelength. The arrangement of these waves from lower to higher frequencies/wavelengths or vice versa constitutes the electromagnetic spectrum. The spectrum with applications is explained in figure below.
2.5.2 Nature of Electromagnetic waves:

The origin of these waves is due to electric and magnetic fields which are vibrating perpendicular to each other and the wave propagates perpendicular to both these fields. The electromagnetic radiations have the property of rectilinear propagation and can travel in vacuum also. The traveling speed of electromagnetic radiations is $3 \times 10^8$ m/s. These waves can be explained on the basis of both wave and particle nature which is explained below.

2.5.3 Wave nature of light:

The electromagnetic waves are considered as wave and transference occurs in the form of crests and troughs. The length of one crests and trough constitutes the wave length of the wave represented by ($\lambda$) and number of crests and troughs passing at a specific point per second is the frequency of the wave represented by ($\nu$).

2.5.4 Particle nature of light:

Quantum mechanics explains the particle nature of light which is transferred in the form of small packets of energy know as quanta’s. The energy of each quantum is given by

$$ E = h \nu $$

Where $E$ is energy in joules.

Figure 2.2 Electromagnetic spectrum [16]
h is Plank constant having value 6.626×10^{-34} Js

U is the frequency of the wave

The frequency of wave is given by 
\[ \nu = \frac{c}{\lambda} \]

The c is the speed of light which is 3×10^8 m/s and \( \lambda \) wave length inserting the values in the equation above the energy becomes

\[ E = \frac{hc}{\lambda} \]

The equation is used to calculate the energy of electromagnetic radiations.

2.5.5 Laws governing the UV-Visible spectroscopy:

There are two main laws applicable to this spectroscopy one of the laws deals with concentration and another with path length. These laws are explained below

2.5.6 Lambert’s law:

This law states that when a monochromatic light is passed through a transparent medium the rate of fall in intensity with thickness is directly proportional to intensity of light. The law explains that as we pass light through a medium the intensity of light decreases with the thickness, more the thickness of medium, more will be fall in intensity. The law is expressed by differential equation

\[ -\frac{dI}{dl} = k I \]

where \( I \) the intensity of incident light, \( l \) is the thickness of the medium and \( k \) is constant. On integration with limits \( I = I_0 \) and \( l=0 \) we get

\[ \ln \frac{I_0}{I_0} = k l \]

Or

\[ I_0 = I_0 e^{-kl} \]

The ratio of \( I/I_0 \) is the fraction light transmitted through medium \( l \) and is known as transmittance represented by T. The reciprocal \( I0/It \) is the absorbance represented by A and given by

\[ A = \log \frac{I_0}{I_t} \]
2.5.7 Beer’s law:

This law states that as we pass the monochromatic light through a solution, the fall in intensity of incident light is proportional to the concentration of the solution. This law is same as that of Lambert’s, but this deals with concentration of the solution and former with thickness. The law is given by

\[ I_t = I_0 e^{-kc} \]

Or

\[ I_t = I_0 10^{-Kc} \]

Where \( c \) is the concentration and \( k \) is constant combining the equations we get

\[ I_t = I_0 10^{-acl} \]

Or

\[ \log \frac{I_0}{I_t} = a c l \]

This is fundamental equation of spectrophotometery and is known as beer lamberts law. It can be also given as

\[ A = \varepsilon c l \]

Where \( A \) is absorbance, \( \varepsilon \) is molar absorption coefficient,

\( c \) is concentration and \( l \) is thickness in cm.

2.5.8 Instrumentation of UV-Visible spectroscopy:

This is within the range of ultra violet and visible region of the electromagnetic spectrum and the instrumentation is explained below.

![UV-Visible instrumentation](image)

*Figure 2.3 UV-Visible instrumentation [15]*
Figure 2.3 indicated the instrument consists of number of parts starting from source till readout device. The detailed instrumentation is explained below.

2.5.9 Radiation source:

For a spectrophotometer it is important that there must be an illuminating radiation source. The lamp in the figure 2.3 indicates the source position. For the generation of UV light, hydrogen or deuterium lamps are used and for visible region, tungsten lamp is used. In case of the spectrophotometer both the sources are present. The UV source provides radiation from a wave length of 200-400 nm and tungsten lamp provides 400-700 nm.

2.5.10 Collimating lens:

In the figure 2.3 we see there is a collimating lens. The function of the lens is to collimate the radiation source from lamp to monochromator. This lens does not allow dispersing the radiations but collimate them to a specific position.

2.5.11 Monochromator:

This part includes the grating; the function of monochromator is to make the radiations monochromatic. The radiations from the source are polychromatic with the electric and magnetic fields vibrating in different planes. After passing the radiations through monochromator, the radiations came out as monochromatic with one plane of vibrations.

2.5.12 Sample holder:

The function is to hold the sample cell. The solution to be analyzed is put in the sample cell made of quartz. Quartz cuvetts are used because glass blocks the UV radiations passing through it. For the analysis in the visible region, glass cuvetts can be used. The solution cuvetts are put in the sample holder and a monochromatic radiation source is allowed to pass.

2.5.13 Detector:

The function is to detect the radiation coming out of the sample cell. The solution in the cell is absorbing some radiations and these absorbing radiations will be detected by the detector. The detector is a photo multiplier tube which can detect the radiations...
in the electronic form. The other form of detector like semiconductor devices can be also used.

2.5.14 Amplifier and readout devices:

These are placed after detector and their function is to amplify the signal coming out from a photo multiplier tube and the read out device prints the signal obtained from the detector. The readout devices are computer controlled.

2.6 FT-IR Spectroscopy

Infrared spectroscopy is an important tool of laboratories. The advantage of this spectroscopy over others is that all form of the compound can be studied including solid, liquid and gas.

2.6.1 Applications of infrared spectrum:

Infrared spectrum is like the figure print of humans as all the bonds in the molecule have different environment and they cannot absorb the same frequency although they are identical. The difference in the environment of bonds makes the vibrations of these bonds different and so the absorption frequencies. There may be the similarity in the absorption but not exact. Infrared spectrum can identify compounds with similarities and dissimilarities. If the compounds cannot be identified we can check their infrared spectrum. If all the peaks in the infrared spectrum match then they are same.

Other important applications of infrared spectrum is that it can be used in structure determination of the compounds that is if we have to determine the structure of a compound we have to take infrared spectrum and compare the values of absorption to the standard values as these are not exact but an idea of structure can be obtained. For instance the absorption in range of 3000±150 cm\(^{-1}\) is almost due to C-H bonds in the molecule, 1715±100 cm\(^{-1}\) indicates the presence of C=O bonds in the compound.

2.6.2 Infrared absorptions by molecules:

Infrared radiations are absorbed by the molecules having changing dipole moment with vibrations of bonds. Mostly hetero diatomic are active in infrared spectrum and
homo diatomic molecules are inactive and this is the selection rule for infrared spectroscopy.

2.6.3 Modes of vibration:

The most important modes of vibration are stretching and bending. The stretching vibration is the vibration of bond along the internuclear axis either in the same direction or opposite, further these vibrations are classified as symmetric and antisymmetric vibrations. Symmetric vibrations are those vibrations when two molecules either approach to each other simultaneously or moves away, the angle between vibration axis and chemical bond is not altered and asymmetric stretch are stretches when one nuclei of the bond approach to one point and other moves away.

Bending vibrations are due to change in the angle between vibrating bonds. These are due to nuclear motions they are further classified rocking, wagging, scissoring and twisting, but bending vibrations require at least three atoms.

2.6.4 Instrumentation:

The instrumentation for infrared spectrometer is same as that of UV-Visible spectrometers in general but vary in the source. Simple infrared instrumentation is give in the figure below

![Figure 2.4 Detailed instrumentation of IR spectrometer](image)
2.6.5 Source of radiations:

Source of radiations for infrared spectrometer is a tungsten lamp for near IR source and for broad spectrum of IR radiations Nernst glower lamp is used. The Nernst lamp is made of nichrome wire and is mixed with oxides tungsten yttrium and others. The Nernst glower is heated up to $1300^\circ$C and broad range of IR radiations is obtained.

2.6.6 Sample cells:

Different procedures are for preparation of sample for each case of solid, liquid and gases. In case of solids the sample is mainly prepared by dissolving the solid in the organic solvents like carbon tetrachloride and carbon disulphide. The sample solution is injected into the sample cell with cell window made of transparent material to IR radiations. Mostly alkyl halide salts like KBr, KCl and NaCl are used as window material. For those solid samples which are not soluble in these solvents their fine powder is made and dissolved in the mineral oil Nujol for a suspension known as mulls, these mulls are pressed between the KBr or other alkyl halide plated and analyzed.

For the liquid samples the samples are mostly used as neat and IR transparent windows are used to pass the broad range of IR spectrum. The neat liquid is also pressed with the salt sheets and analyzed.

The gas samples are analyzed in the specially designed gas cell. The highly absorbing gas is filled in the cell of 10 cm and low absorbing gas needs larger path length cells than this. The gas samples are filled from inlet value and both the ends are sealed. The broad spectrum of IR radiations are used to analyze the gas.

2.6.7 Monochromator:

In most of IR spectrometer grating is used as Monochromator. The function of grating is to provide the unique set of IR radiations to be used for identification however prism made of alkali salts like KCl, KBr are also used in few instruments but there are several disadvantage of using the prism as Monochromator, these are effect of moisture on the prism and not having good resolution in spectra.
2.6.8 Detector:

The detectors used in the IR spectrometer are thermal; these detectors are based on thermocouple in which the change in temperature is converted into electronic signal and which is detected by readout devices. The semiconductor detectors are also used, they are based on the semiconductor like Hole- electron process which are dependent on the band gap energy levels.

2.6.9 Readout Devices:

After the signal from detector the signal is amplified and chopped then the readout devices prints the record. The read out devices are mainly x-y plotters and few computer controlled devices are also used. The S/N ratio of signal must be high so as to get the good resolution graph.

2.7 Nuclear Magnetic Resonance

The nuclei have magnetic moments associated with them. When an external magnetic field is applied and a radiofrequency is simultaneously applied, then the magnetic field interacts with the field of nuclei and if the frequency of external field matches with the frequency of nuclei, resonance take place and the absorption of external r.f. occurs. The whole process is known as nuclear magnetic resonance. The magnetic resonance uses radiofrequency region of electromagnetic spectrum as explained before. The change in the energy due to absorption is given by

\[ \Delta E = E_0 - E \]

\[ \Delta E = \mu \beta H \]

The nuclear magnetic resonance is possible only with those nuclei having non zero spins including the proton which is widely studied spectroscopy, other nuclei commonly studied are $^{13}$C and $^{31}$P.

2.7.1 Chemical shift:

The nuclei present in the environment of magnetic field may not sense the magnetic field same as that of applied field because the electronic cover surrounding the nuclei generate magnetic currents which interact with the external magnetic field thus
causing a shift in magnetic field sensing. The generation of magnetic current to external field applied is known as shielding. The local change in the magnetic field is given by.

$$\Delta B = -\zeta B_0$$

Where $\zeta$ shielding constant and $B_0$ is applied magnetic field. Shielding constant is unit less and its values are dependent on electronic environment surrounding the magnetic nuclei due to which exact magnetic field sensed by magnetic nuclei cannot be measured and only a shift in magnetic field can be observed which is given by

$$\text{Chemical shift} = (v-v^0/v^0) 10^6$$

$v^0$ is reference frequency and $v$ as resonance frequency also known as Larmor frequency. The chemical shift is measured with respect to reference.

The reference is a chemical substance added with sample and the chemical shift of reference and the magnetic field is observed for the compound with reference to reference added. Most used references are (TMS) Tetramethylsilane whose chemical shift is zero in (ppm) parts per million.

The electronic shielding is not the only thing affecting the chemical shift there are various other parameters which affect the shielding process. The resonance effect is also pronounced which may be due to delocalization of ring electrons causing shielding/deshielding in the environment. The others are paramagnetic/diamagnetic effects, solvent effects and neighboring group participation. These all parameters have the effect on the shielding and hence induce the chemical shift.

2.7.2 Spin-Spin splitting \((n+1)\) rule:

As explained above the chemical shift, here we will explain the important information revealed by NMR spectrum that is providing the information regarding the number and types of protons in the molecule. This will be explained by an example given below

$$\text{C}_6\text{H}_2\text{Br}--\text{C}_2\text{HBr}_2$$

There are two different types of protons on two different carbons so predicting the NMR the compound must give two peaks in the spectrum but the compound gives one
triplet and one doublet, this is due to the phenomena known as spin-spin splitting and can be explained by (n+1) rule, which explains that if there are \( n \) adjacent protons in the neighboring carbon the peaks in the spectrum will \( (n+1) \) as is the case with present molecule the protons present on \( C_\beta \) carbon interacts with the adjacent protons on \( C_\gamma \) which is one and \( C_\beta \) protons give doublet with \( C_\varepsilon \) protons and in turn \( C_\varepsilon \) single proton give triplet with \( C_\beta \) protons according to \( (n+1) \) rule. In the spectrum of this compound there is a triplet and a doublet so validating \( (n+1) \) rule.

2.7.3 Instrumentation:

The detailed instrumentation NMR is given in the figure

![Figure 2.5 Instrumentation of NMR [17]](image)

The basic structure of the NMR spectrometer is given above. NMR samples are dissolved in the deprotonated solvents like heavy water; CDCl\(_3\) and CCl\(_4\) the sample is also added with internal reference mostly TMS Tetramethylsilane and are kept in sample tube as indicated by the figure above.

The sample is kept between the two electromagnets, with spinning the tube at high speed and ensuring the all parts get the same magnetic field. The radiofrequency transmission coils adjust the magnetic field to the sample through magnets and if the sample does not absorb, the receiving coils does not pick up any signal and if the absorption occurs the receiver coils pick the signal and transfer it to the detector. The protons in the different environment observe the resonance at different resonance
frequencies the detector detects the signal and transfer it to recorder, which is computer controlled and provide print record of NMR spectrum for the specific compound.

2.8 References


17. [https://www2.chemistry.msu.edu/faculty/reusch/virttxtjml/spectrpy/nmr/nmr1.htm](https://www2.chemistry.msu.edu/faculty/reusch/virttxtjml/spectrpy/nmr/nmr1.htm)

18. [www.google.com](http://www.google.com)