CHAPTER-2

CHARACTERIZATION TECHNIQUES
Scope of the chapter

This chapter gives a brief introduction of spectroscopy and highlights the general methodology that can be adopted in many ways to extract the information regarding the structures. It also gives a brief description of the theoretical spectral values of polyimides structures.
Analysis of data is a process of inspecting, transforming, and modeling data. The aim of data analysis is to highlight useful information, to suggest conclusions, and to support decision making. It has multiple facets and approaches, encompassing diverse techniques.

### 2.1. ELEMENTAL ANALYSIS

The majority of organic compounds are composed of a relatively small number of elements. The most important are: carbon, hydrogen, oxygen, nitrogen, sulphur, etc. Elementary quantitative analysis is used to determine the content of these elements in the molecule of an organic compound. Elemental analysis was carried out on a Thermofinigan Flash 1101EA (Italy).

### 2.2. SPECTRAL ANALYSIS

Spectroscopy is a general methodology that can be adapted in many ways to extract the information regarding energy of electronic, vibrational and rotational states; structure and symmetry of molecules and other dynamic information. It is also the study of the interaction of matter and light (or other electromagnetic radiation).

Spectroscopic techniques have major importance in many areas of chemistry and physics and are essential to the laboratory polymer chemist as it can be used to determine unknown molecular structures. In this chapter, we have analysed the obtained data with the help of reputed published books and research articles.

#### 2.2.1. Infrared analysis

Infrared spectroscopy is a technique based on the vibrations of the atoms of a molecule. An infrared spectrum is commonly obtained by passing infrared radiation through a sample and determining the amount of incident radiation which is absorbed at a particular energy.

The absorption measurement of different IR frequencies has been carried out by positioning a sample through an IR beam. The chemical functional groups can be identified by this technique. Various functional groups were absorbing
characteristic infrared frequencies. Infrared spectroscopy is especially significant and talented tool for structural explication and compound recognition.

### 2.2.1.1. IR frequency range and spectrum presentation

The IR region is classified into: near IR, mid IR and far IR. The far IR region is used for the analysis of organic, inorganic, and organometallic compounds including heavy atoms, which offers valuable information about the structural characterization e.g. conformation and lattice dynamics of samples. Near IR spectroscopy requires negligible or no sample preparation. It provides high-speed quantitative analysis without consumption of a specimen. Now, the instruments can be collective with UV-visible spectrometer and attached with fiberoptic devices for remote analysis. Near IR spectroscopy is commonly used in the process control applications.

In the present thesis, frequency of mid IR region, between 4000–400 cm\(^{-1}\) (2.5 to 25 \(\mu\)m) was used.

<table>
<thead>
<tr>
<th>IR region</th>
<th>near IR (13,000-4,000) cm(^{-1})</th>
<th>mid IR (4,000-200) cm(^{-1})</th>
<th>far IR (200-10) cm(^{-1})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wavenumber</td>
<td>(0.78-2.5) (\mu)m</td>
<td>(2.5-50) (\mu)m</td>
<td>(50-1,000) (\mu)m</td>
</tr>
</tbody>
</table>

### 2.2.1.2. Fourier Transform spectrometers

Fourier transform spectrometers have been used instead of the dispersive instruments due to their excellent speed and sensitivity. They have been implemented in many regions that are tough to examine by dispersive instruments. In Fourier Transform Infrared (FT–IR) spectroscopy all frequencies are analysed at the same time while in dispersive IR spectrometer, each component frequency is observed consecutively.

### 2.2.1.3. Analytical information

Infrared spectroscopy is mainly employed in two ways: compound identification and structural elucidation.
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**Compound Identification:** Every molecule has unique infrared spectrum. In order to indentify the unknown organic compound, one must have infrared spectrum of reference compound that matches the unknown compound. A huge number of reference spectra are available. The spectral libraries accumulated by Sadtler and Aldrich are the most popular collections. Computer tools are useful in matching process. In many cases the spectrum of unknown material does not match exactly with the reference compound.

**Structural Elucidation:** IR absorption of the functional groups can differ due to complex interactions between atom-atom in the molecule. It has been noted that, numerous functional groups offer characteristic IR absorption at explicit and narrow frequency ranges in spite of their relationship with another molecule. Comprehensive tables of the positions along with intensities of absorption bands have been established and utilized to verify the functional groups present/absent in a specimen.

2.2.1.4. IR spectral properties of polymers

Hausdroff [1] and Kagarise and Weinberger [2] had published collections of infrared spectra of technical polymers and resins. Krimm [3] studied the infrared spectra of eight different polymers. He made a complete vibrational analysis of these compounds and proposed possible structures.

As a consequence of the sensitivity of IR to change the dipole moments of the vibrating groups under examination, it is considered as one of the useful method for providing information regarding chemical and physical nature of the polymers.

IR spectroscopy furnishes the qualitative and quantitative information of structural units, type and degree of branching and end groups present in a polymer. However, the quality of IR spectroscopy of a polymer primarily depends upon the correct sampling of polymer; otherwise a considerable amount of information may be lost or misinterpreted. Hence, the thorough knowledge of sampling techniques for different forms of polymers, such as films, solid, solution, powder, pellet, granules, fiber etc. is very essential [4,5].
The polymer samples which are insoluble in common solvents do not give good IR Spectra i.e. fine IR spectrum is not observed. Only few bands are easily detectable and in the major part of the spectrum it becomes difficult to observe the characteristic bands. In such cases, the IR spectral data do not serve the purpose of a comparative study. The reliability of IR spectral data can be further improved by using computer supported IR spectral data, which can be easily added or subtracted from the selected data [6].

The most probable structure and chemical nature of the polymer of interest may be readily obtained from the certain characteristic group frequencies and their comparison with the IR data published in IR library. Henniker [7] published the IR spectral data for many industrial polymers. These data can be used as references for identification of new polymers and plastics. An infrared library including monomers and polymers is commercially available from the Sadtler Research Lab [8].

The features of the IR spectra are usually observed in the region from 3500–2900 cm\(^{-1}\) for X–H stretching, in the region from 1800 to 1500 cm\(^{-1}\) for X=Y stretching. In X–H stretching region, bands are observed in the order of decreasing frequency as OH > NH > CH. Among C–H bands; SP C–H, SP\(^2\) C–H and SP\(^3\) C–H bands can be distinguished because of their different placements. In certain cases, bands due to C–H stretching of –CO-CH\(_2\)– are observed and can be distinguished [9].

The characteristic absorption of ether is due to C–O–C stretching asymmetric vibrations in the region 1300 cm\(^{-1}\) to 1000 cm\(^{-1}\) and C–O–C stretching symmetric vibrations in the region 1055 cm\(^{-1}\) to 870 cm\(^{-1}\). The strong absorption of the keto (>C=O) stretching of conjugated aryl appears at 1720 to 1660 cm\(^{-1}\) [8]. The nature of groups decides the position of >C=O band in the IR spectrum.

The skeletal stretching vibrations due to –C=C– of the aromatic rings are observed in the region around 1580 to 1450 cm\(^{-1}\). They are diagnostic due to the presence of the aromatic system. Generally four bands are observed around 1615, 1580, 1465 and 1450 cm\(^{-1}\). The latter band is often mixed up with the aliphatic C–H bending vibration band. The region from 1200–1000 cm\(^{-1}\)
comprises many bands including the bands due to C–O and C–N stretching vibrations. The decision of the assignment of a band due to C–O and C–N stretching is difficult because the position of the band is variable and a band may get mixed up with other bands in the fingerprint region. The region from 1010 to 610 cm\(^{-1}\) contains certain bands.

Generally the spectra of all the compounds containing aromatic systems exhibit easily discernible bands in the region from 820 to 710 cm\(^{-1}\). These bands are of particular interest due to the characteristic substitution pattern of the aromatic nucleus. It is a usual practice to examine this part of the spectrum to decide the presence of an aromatic system and to get indications about the substitution pattern in organic system.

**2.2.2. NMR analysis**

Atom with an odd mass or atomic number having a nuclear spin can be studied by NMR. The NMR spectrum provides valuable information about the determination of an organic structure revealing the hydrogen and carbon framework.

Without using an applied magnetic field, protons are spinning haphazardly and producing a magnetic field (called the magnetic moment). Nevertheless, once an external magnetic field is applied, the protons are either parallel or anti parallel to it. The parallel (alpha spin) orientation has energy lower than that of the anti parallel (beta spin).

While applying a stronger magnetic field, the energy difference (ΔE) between the parallel and anti parallel states is increases. Hence, the strength of the magnetic field decides the energy essential for a nuclear spin flip. The energy difference (ΔE) between the ground and excited states is roughly 0.02 cal. mol\(^{-1}\) which correlates to the radio wave photons. When the radio wave photons are supplied, the NMR signal is created which match with the ΔE of the nucleus.

**2.2.2.1. Structural information from NMR spectrum**
Number of signals

Identical protons (which are having same magnetic environment/chemically equivalent) have the same magnetic force, will produce similar and overlapping signals in the NMR spectrum. The number of signals appeared in the NMR spectrum match to the number of equivalent sets of protons. Protons associated diagonally a plane of symmetry e.g. benzene.

Position of signals:

The magnetic field experienced by a proton is a combination of the earth magnetic field, the magnetic field of NMR spectrometer and the local magnetic field produced by the electrons and other nuclei. TMS is used as a reference standard. So, the factor that decides the position of a signal is the magnetic field created by other nuclei and electrons present in the molecule. The chemical shift explains the energy difference relative to the reference protons.

Relative intensity of signals:

Some additional structural information was obtained from the relative intensity of each signal on the NMR spectrum. As per the Beer’s Law, the amount of energy absorbed is directly proportional to the amount of matter present. Likewise, the area covered by each signal is proportional to the number of equivalent protons. Hence, by incorporating this area, the spectrometer gives relative numbers of protons. These relative integral values can be used to find out the number of equivalent protons.

Splitting of NMR signals:

In the NMR spectrum, splitting of a signal observed is known as spin-spin coupling. The interaction between the nuclear spins gives more than one energy state for the system. The magnetic field of two non equivalent protons is when close under the influence of each other, are known as magnetically coupled. Due to spin-spin splitting (a reciprocal property), magnetically coupled proton split the other's signal.
2.2.3. CMR analysis [12]

Besides the PMR spectroscopy, the CMR spectroscopy is also a precise method to determine the structure of organic molecules. Considerably greater sensitivity is required for $^{13}$C than for $^1$H due to low natural abundance of $^{13}$C and the lower magnetic moment compared to that of the proton. However, greater resolution is possible with $^{13}$C.

In contrast to $^1$H spectra, it is not possible to determine the relative ratio of carbon atoms in a compound by integration of the peak areas in the $^{13}$C FT-NMR spectrum. There are two reasons. The first reason results from the different relaxation times of carbon atoms in different environments. This means that some atoms with long relaxation times may still be partly saturated when the next pulse of radiation is received, and the resulting absorption peak areas will not be proportional to the number of different carbon atoms.

Carbon atoms without hydrogen attached, have longer relaxation times and are therefore likely to give rise to peaks of lower intensity in the spectrum. The second reason is due to the Nuclear Overhauser Effect (NOE). This is the enhancement of some signals in the $^{13}$C spectrum as a result of the spin-decoupling process which is used to produce the normal, noise-decoupled spectrum by removing the interaction between carbon and hydrogen nuclei.

The NOE is not the same for all nuclei. The maximum effect is for carbon atoms with hydrogen attached. The consequence is that carbon atoms without hydrogen attached appear without any NOE enhancement. As a result of these two effects, it is often possible to identify by inspection, as a result of their lower intensity, those peaks in the $^{13}$C spectrum which result from carbon atoms not attached to hydrogen, including those in aromatic rings which carry a substituent.

A considerable amount of the data is available which correlates the position of absorptions in the $^{13}$C NMR spectrum with the structure of an organic molecule, and it is imperial correlations which provide the main basis for the use of the technique in structure determination. The values for the chemical shifts are
normally related to the tetramethylsilane carbon absorption, with positive values increasing to lower field. The majority of absorptions fall in a range of 200 ppm between the carbonyl absorptions at low field and the methyl absorptions at high field.

Hybridization of the carbon atom has a significant effect on the chemical shifts. sp3-hybridized carbon absorbs at high field (0-60 ppm downfield from TMS), sp2-carbon at low field (80-200 ppm) and sp-carbon at intermediate values. The precise position of absorption of a particular atom is largely determined by the electronic effects of any substituent, and the fact that these are approximately additive enables fairly accurate predictions of chemical shifts to be made, provided that similar compounds of known structure are available for reference purposes.

Only one of the three items (i.e. chemical shift, coupling constant and relative numbers of absorbing nuclei) of information normally available from PMR spectra is routinely available from the $^{13}$C spectrum, and that is the chemical shift. Quantitative coupling constants are not normally obtained, and relative numbers of nuclei cannot usually be derived from measurement of peak areas. The large $^{13}$C-$^1$H coupling constant (125-200 Hz for directly bonded protons) results in multiplets which overlap to a considerable extent and in the absence of decoupling makes the spectrum difficult to analyze. Spectra are therefore normally spin-decoupled and each absorption appears as a sharp singlet; this technique is known as wide-band or noise decoupling. Although the sensitivity is thus increased, all the information normally available from spin-spin splitting pattern is lost.

An alternative method of decoupling (off resonance decoupling) does however allow coupling of directly bonded carbon and hydrogen to be observed, although the separation of the peaks of the multiplets produced by this method is not equal to the true $^{13}$C-$^1$H coupling constant. It is thus possible to identify carbon atoms associated with methyl, methylene and methine groups since the absorptions appear as quartets, triplets and doublets respectively provided that the bonded hydrogen are equivalent.
2.3. THERMOGRAVIMETRIC ANALYSIS (TGA)

2.3.1. Introduction

Thermal analysis techniques include thermogravimetric analysis (TGA) which is utilized to show a broad diversity of materials. The amount and rate of change in the mass of a sample is measured by this technique as a function of temperature or time in a controlled atmosphere. The thermal, oxidative stabilities and compositional properties can be resolving using these measurements initially. It is particularly helpful in the study of polymer materials, like thermoplastics, thermosets, elastomers, fibers, composites, films, coatings and paints.

TGA measurements offer precious information which is used to choose materials for definite end-use applications that forecast the presentation of product and develop good quality of product. The technique is particularly useful for the following types of measurements:

- Compositional analysis of multi-component materials or blends
- Purity and thermal stabilities
- Oxidative stabilities
- Estimation of product lifetimes
- Decomposition kinetics
- Effects of reactive atmospheres on materials
- Filler content of materials
- Moisture and volatile content

2.3.2. Methodology

The thermogravimetric analysis (TGA) of the prepared samples has been carried out by using Perkin - Elmer Pyres 1 TGA Instrument, at a heating rate 10 °C / min in a slow stream of air and a temperature range between 30 and 750 °C. The boat prepared from the platinum foil holds the polymer sample that is to be analyzed.

It was properly washed and dried. It was suspended on the quartz rod in the TGA balance. The sample was placed in the boat and is covered by quartz tube in which the flow of air was maintained. The weight of the sample was noted on the
TGA balance. The whole assembly was brought down in the furnace. It was ascertained that the boat was hanging on quartz rod. The experiment was started by heating the system at a constant rate of 10 °C min⁻¹ and simultaneous change in the weight was recorded automatically.

TGA is a process that utilizes heat and stoichiometry ratios to determine the percent by mass of a solute. Test is done by increasing the temperature of the specimen slowly and drawing a graph of weight (percentage) against temperature. The temperature is raised upto 700 °C or greater. To search the correct points of inflection, curve smoothing and other functions are carried out after getting the result.

A hi-resolution TGA method is frequently used to get a more precision in the areas where the derivative curve rises. In this method, as temperature increases slowly the weight loss increases. When a peak is obtained the exact temperature is more accurately identified.

2.4. NUMBER AVERAGE MOLECULAR WEIGHT \((\bar{M}_n)\)

Many physical properties are confirmed by polymer molecular weight. Various definitions of the molecular weight of a polymer have been formulated depending on the method employed in the measurement of the molecular weight of the polymer.

To evaluate the average molecular weight of the polymer, a common standard is used to verify number average molecular weight \((\bar{M}_n)\), weight average molecular weight \((M_w)\) or viscosity average molecular weight \((M_v)\). Their definitions are as follows [10–12]. To get valuable commercial applications of the polymer material, the molecular weight should be very low so that transition temperatures and the mechanical properties will also be very low hence it is used for waxes or liquids.

- **Number average molecular weight \((\bar{M}_n)\)**

Number average molecular weight \((\bar{M}_n)\) follows the conventional definition for the mean value of any statistical quantity. To get a formula for Number average
molecular weight, the molecular weight distribution discontinues the function of M. Fairly, only discrete values of M are accepted. The values of M are diverse multiples of the monomer molecular weight - M_o. Monomer molecular weight is the weight per monomer that shows in the polymer chain. During the condensation reaction, water molecules are characteristically misplaced from the monomers, where M_o is monomer molecular weight.

The probable values of M frame a set of numbers through distinct standards tagged Mi.

Total Weight of all polymers = \( \sum_{i=1}^{\infty} NiMi \)

Total number of polymer molecules = \( \sum_{i=1}^{\infty} Ni \)

Where,  
\( Mi = \) molecular weight of polymers  
\( Ni = \) number of polymers

As discussed above,

No. Average Mol. weight = \( \frac{\sum_{i=1}^{\infty} NiMi}{\sum_{i=1}^{\infty} Ni} = \frac{\text{Total Weight}}{\text{Number of Polymers}} = \frac{\text{Weight}}{\text{Polymers}} \)

No. Average Mol. weight  Mn = \( \sum_{i=1}^{\infty} XiMi \)

Where,  
\( Ni / \sum Ni = \) number fraction of polymers  
\( Mi = \) molecular weight of polymers  
\( Xi = \) mole fraction

The alternating form is:

\[ M_n = \frac{\sum_{i=1}^{\infty} Ci}{\sum_{i=1}^{\infty} Ci/Mi} = \frac{\text{Total Weight}}{\text{Number of Polymers}} = \frac{\text{Weight}}{\text{Polymers}} \]

Dividing numerator and denominator by \( \sum Ci \) results in

\[ M_n = \frac{1}{\sum_{i=1}^{\infty} \omega_i / Mi} \]

Where, \( \omega_i = \) weight fraction of polymer i or the weight of polymer i divided by the total polymer weight:
The methods employed for the estimation of \( (\overline{M}_n) \) are end group analysis, non-aqueous conductometric titration or the methods based on the estimation of colligative properties of polymer like vapor pressure osmometry (VPO).

**Weight average molecular weight (Mw)**

To obtain the weight average molecular weight, substitute the number of polymers having molecular weight \( i/N_i \) in the number average molecular weight formula with the weight of polymer known as molecular weight \( i \) or \( N_iM_i \). The result is

\[
\text{Number average molecular weight (Mw)} = \frac{\sum_{i=1}^{\infty} N_iM_i^2}{\sum_{i=1}^{\infty} N_iM_i}
\]

**Viscosity average molecular weight (Mv)**

It is determined by viscosity measurements (by Ostwald Fenske Viscometer or Brooke Field Viscometer or Mooney Viscometer) and is to be used in the following equation assuming that the intrinsic viscosity and the molecular weight relationship of the sample follow the Staudinger's equation.

\[
[\eta] = KM^a
\]

Where ‘a’ is a constant dependent upon the polymer–solvent system, \([\eta]\) is a intrinsic viscosity, \(M\) is a molecular weight of polymer.

\[
\overline{M}_n = \sum_{i=1}^{\infty} [WiM_i]^{\frac{1}{2}} \left[ \frac{\sum_{i=1}^{\infty} N_iM_i^{a+1}}{\sum_{i=1}^{\infty} N_iM_i} \right]^{\frac{1}{2}}
\]

Where ‘a’ equals unity, the viscosity average reduces to the weight average. The value of \((M_v)\) is intermediate between \((\overline{M}_n)\) and \((M_w)\). The order is \((M_n) < (M_v) < (M_w)\)

In these relations \(N_i, Wi\) and \(M_i\) respectively are the number of moles, weight fraction and molecular weight of the \(i^{th}\) type of the molecule present in the
polymer sample. The ratio of $M_w / \overline{M_n}$ gives the polydispersity index and is a measure of the polydisperse nature of the polymer sample.

The average molecular weight of a polymer plays a very important role in deciding the specific end application of a polymer and hence its estimation is considered as one of the essential property for the polymer characterization.

Different methods have been developed for the estimation of average molecular weight of a polymer. Individual methods of molecular weight determination can be distinguished qualitatively according to the statistical averages obtained and also according to the relationship between quantity to be measured and molecular weight. A distinction is made between:

- Absolute methods
- Equivalent methods and
- Relative methods

With absolute methods, the quantity to be measured is directly related to the molecular weight without having to make any assumptions about chemical and/or physical structures.

For equivalent methods, the chemical structure must be known. Relative methods measure the properties which depend both on chemical and physical structure, and which is required for evaluation of a calibration relationship between quantity to be measured and molecular weight.

Table 2.1 summarizes the most important methods of determining molecular weight and divides them into absolute (A), equivalent (E) and relative methods (R)\[12\].
**Table 2.1: Principle Methods of Determining Molecular Weight [12]**

<table>
<thead>
<tr>
<th>No.</th>
<th>Method</th>
<th>Type</th>
<th>Molecular weight range (g/mole)</th>
<th>Mean values of the molecular weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Membrane osmometry</td>
<td>A</td>
<td>$10^4$–$10^6$</td>
<td>Mn</td>
</tr>
<tr>
<td>2</td>
<td>Ebullioscopy</td>
<td>A</td>
<td>$10^4$</td>
<td>Mn</td>
</tr>
<tr>
<td>3</td>
<td>Cryoscopy</td>
<td>A</td>
<td>$10^4$</td>
<td>Mn</td>
</tr>
<tr>
<td>4</td>
<td>Isothermal distillation</td>
<td>A</td>
<td>$10^4$</td>
<td>Mn</td>
</tr>
<tr>
<td>5</td>
<td>Vapour pressure osmometry</td>
<td>A</td>
<td>$10^4$</td>
<td>Mn</td>
</tr>
<tr>
<td>6</td>
<td>End group determination</td>
<td>E</td>
<td>$10^5$</td>
<td>Mn</td>
</tr>
<tr>
<td>7</td>
<td>Light scattering</td>
<td>A</td>
<td>$10^2$–$10^8$</td>
<td>Mw</td>
</tr>
<tr>
<td>8</td>
<td>Sedimentation equilibrium</td>
<td>A</td>
<td>$10^6$</td>
<td>Mw</td>
</tr>
<tr>
<td>9</td>
<td>Sedimentation equilibrium</td>
<td>A</td>
<td>$10^5$</td>
<td>Mn, Mw</td>
</tr>
<tr>
<td></td>
<td>with density gradient</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Combination of sedimentation / diffusion coefficient</td>
<td>A</td>
<td>$10^3$–$10^8$</td>
<td>Mn, Mw</td>
</tr>
<tr>
<td>11</td>
<td>Solution viscosity</td>
<td>R</td>
<td>$10^2$–$10^8$</td>
<td>Mv</td>
</tr>
<tr>
<td>12</td>
<td>Melt viscosity</td>
<td>R</td>
<td>$10^2$–$10^7$</td>
<td>Mw</td>
</tr>
<tr>
<td>13</td>
<td>Gel permeation chromatography</td>
<td>R</td>
<td>$10^2$–$10^7$</td>
<td>$M_{GPC}$</td>
</tr>
<tr>
<td></td>
<td>(the index GPC does not indicate a recognized argument)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Molecular weight determination by electron microscopy is highly effective where a very dilute polymer solution and the individual molecules are counted. Sedimentation and diffusion of macromolecules in solvents are also dependent on molecular weight and can be measured in an ultracentrifuge and utilized for molecular weight determination.

A simple low cost commonly used method of investigating molecular weights of polymers is that of viscosity measurements [13]. Certain more specific methods have been employed for the estimation of the molecular weight of aromatic and mixed aromatic–aliphatic polymers which are polycarboxylic acids, polyphenols or polyamines. These methods are conductometric or potentiometric titration in...
non-aqueous media [14–16]. In the present thesis, the polyimides are almost insoluble in most of the organic solvents so the colligative properties like molecular weight determination have not been studied by these methods. So the unfair method i.e. by non-aqueous conductometric titration method has been employed for the produced polyadducts. It was carried out in pyridine solution against standard tetra-n-butyl ammonium hydroxide in pyridine solution as titrant [17–19].

2.5. GENERAL REMARKS FOR THE EXPERIMENTAL TECHNIQUES

- Melting points (°C) was measured by open capillary method. All the melting points were uncorrected.
- All solvents used were distilled and dried. The purity of the compounds was checked by TLC. Column chromatography was performed on silica gel (60-120 mesh).
- Micro analytical (C, N, H) data i.e. C, H, N contents of all the compounds were recorded on Thermofinigan 1101 Flash elemental analyzer.
- Infrared spectra (FT-IR) were obtained from KBr pellets in the range of 4000–400 cm\(^{-1}\) with a Nicolet 760D spectrophotometer.
- PMR and CMR spectra were recorded on 400-MHz Bruker NMR spectrophotometer. PMR ad CMR chemical shifts are recorded in δ-value using TMS as an internal standard in CDCl\(_3/\)D\(_6\)-DMSO.
- The thermogravimetric studies were carried out with a model Perkin Elmer thermogravimetry analyzer at a heating rate of 10 °C min\(^{-1}\) in the temperature range 50–700 °C.
- Number average molecular weight (\(\bar{M}_n\)) of coordination polymers was determined by non-aqueous conductometric titration.
REFERENCES


