1. INTRODUCTION

1.1 GENERAL

1.1.1. Heterocyclic compounds

Heterocyclic chemistry is a very important branch of organic chemistry accounting for nearly one-third of modern publications. In fact, two third of organic compounds are heterocyclic compounds. A cyclic organic compound containing all carbon atoms in ring formation is referred to as a carbocyclic compound. If at least one atom other than carbon forms a part of the ring system then it is designated as a heterocyclic compound. Nitrogen, oxygen and sulfur are the most common heteroatoms but heterocyclic rings containing other hetero atoms are also widely known. Heterocycles are an important class of compounds and are present in a wide variety of drugs, most vitamins, many natural products, biomolecules and biologically active compounds including antitumor, antibiotic, anti-inflammatory, antidepressant, antimalarial, anti-HIV, antibacterial, antifungal, antiviral, antidiabetic, herbicidal, fungicidal and insecticidal agents. Also, they have been frequently found as a key structural unit in synthetic pharmaceuticals and agrochemicals. Some of these compounds exhibit a significant solvatochromic, photochromic and biochemiluminescence properties.

1.2. IMIDAZOLE

Imidazole is an organic compound with the formula C₃H₄N₂. Imidazole (1) is a cyclic, planar molecule that consists of a five-membered ring containing three carbons and two nitrogens, with the nitrogens arranged in the 1st and 3rd positions. The nitrogen in
the 1\textsuperscript{st} position is a “pyrrole” type nitrogen and the nitrogen in the 3\textsuperscript{rd} position is a “pyridine” type nitrogen.\textsuperscript{2}

\begin{center}
\begin{tikzpicture}
% Diagram here
\end{tikzpicture}
\end{center}

1.2.1. DISCOVERY OF IMIDAZOLE

Imidazole was first reported by Heinrich Debus\textsuperscript{3} in 1858, fully developed by Radziszewski the beginning of 1882 and further modified by Weidenhagen in 1935. It is the synthesis of an imidazole derivative by the condensation of $\alpha$-dicarbonyl compound (e.g. glyoxal), an aldehyde and two equivalents of dry ammonia in alcohol. Therefore, this reaction is generally known as Radziszewski reaction and occasionally called as Radziszewski synthesis, Weidenhagen synthesis or Deus-Radziszewski imidazole synthesis\textsuperscript{4-6} (Scheme 1).

\begin{center}
\textbf{Scheme 1}
\end{center}
1.3. TETRASUBSTITUTED IMIDAZOLE DERIVATIVES

1.3.1. Synthesis

The synthesis of 1,2,4,5-tetrasubstituted imidazoles is carried out by a four-component condensation of 1,2-diketone, α-hydroxyketone or α-ketomonoxime with an aldehyde, primary amine and ammonium acetate using heteropolyacid,\textsuperscript{7} BF\textsubscript{3}.SiO\textsubscript{2},\textsuperscript{8} silica gel/NaHSO\textsubscript{4},\textsuperscript{9} HClO\textsubscript{4}–SiO\textsubscript{2},\textsuperscript{10} ionic liquids,\textsuperscript{11} L-proline,\textsuperscript{12} ZrCl\textsubscript{4},\textsuperscript{13} InCl\textsubscript{3}.3H\textsubscript{2}O,\textsuperscript{14} K\textsubscript{5}CoW\textsubscript{12}O\textsubscript{40}.3H\textsubscript{2}O,\textsuperscript{15} molecular iodine,\textsuperscript{16} silica sulfuric acid,\textsuperscript{17} NiCl\textsubscript{2}.6H\textsubscript{2}O/Al\textsubscript{2}O\textsubscript{3},\textsuperscript{18} Yb(OTf)\textsubscript{3},\textsuperscript{19} Selectfluor,\textsuperscript{20} H\textsubscript{2}SO\textsubscript{4}–SiO\textsubscript{2},\textsuperscript{21} CAN,\textsuperscript{22} (Yb(OPf)\textsubscript{3}),\textsuperscript{23} Zr(acac)\textsubscript{4},\textsuperscript{24} poly(4-vinylpyridinium tribromide) or citric acid,\textsuperscript{25} p-dodecylbenzenesulfonic acid,\textsuperscript{26} FeCl\textsubscript{3}.6H\textsubscript{2}O,\textsuperscript{27} copper acetate,\textsuperscript{28} trifluoroacetic acid,\textsuperscript{29} zeolite-supported reagents,\textsuperscript{30} mercaptopropyl silica (MPS),\textsuperscript{31} brønsted acidic ionic liquid,\textsuperscript{32} p-TsOH,\textsuperscript{33} DABCO,\textsuperscript{34} silica-bonded propylpiperezine-N-sulfamic acid\textsuperscript{35} and tetrabutylammonium bromide.\textsuperscript{36} In addition, they can also be accessed by the cycloaddition reaction of mesoionic 1,3-oxazolium-5-olates with N-(arylmethylene)-benzenesulfonamides\textsuperscript{37} hetero-Cope rearrangement,\textsuperscript{38} condensation of a 1,2-diketone with an aryl nitrile and primary amine under microwave irradiation\textsuperscript{39} and by N-alkylation of trisubstituted imidazoles.\textsuperscript{40}

1.3.2. LITERATURE REVIEW OF TETRASUBSTITUTED IMIDAZOLE DERIVATIVES

Safari \textit{et al.}\textsuperscript{41} have synthesized 1,2,4,5-tetrasubstituted imidazoles under ultrasonic irradiation in the presence of ionic liquid, 1-methyl-3-(3-trimethoxysilylpropyl)imidazolium chloride immobilized on Fe\textsubscript{3}O\textsubscript{4} nanoparticles at room temperature (Scheme 2).
Mukhopadhyay et al.\textsuperscript{42} have reported the potassium hydrogen sulfate in the presence of dibenzo-18-crown-6 (DB18C6) turns out to be a very efficient and effective catalyst for the facile synthesis of a wide variety of tetrasubstituted imidazoles in aqueous medium at moderate temperature (Scheme 3).

Hasaninejad et al.\textsuperscript{43} have reported the catalyst-free synthesis of 1,2,4,5-substituted imidazoles under conventional heating and microwave irradiation using 1-butyl-3-methylimidazolium bromide [(Bmim)Br], as a neutral reaction media is described in Scheme 4.
Dinesh Kumar et al.\textsuperscript{44} have reported the selective formation of tetrasubstituted imidazole in the presence of HBF$_4$–SiO$_2$ catalyst (Scheme 5).

\begin{center}
\textbf{Scheme 5}
\end{center}

Acke et al.\textsuperscript{45} using a modified Radziszewski reaction have optimized a procedure for the generation of tetrasubstituted imidazoles via microreactor technology (Scheme 6).

\begin{center}
\textbf{Scheme 6}
\end{center}

Chen et al.\textsuperscript{46} have reported an expedient and metal-free synthetic route for the construction of tetrasubstituted imidazole derivatives via an acid promoted multicomponent reaction methodology (Scheme 7).

\begin{center}
\textbf{Scheme 7}
\end{center}
Samanta et al.\textsuperscript{47} have reported NiCl\textsubscript{2}.6H\textsubscript{2}O and Ni(OAc)\textsubscript{2}.4H\textsubscript{2}O as efficient catalysts for C-H activation of benzyl and aliphatic amines for the unprecedented multi C-N bond forming cyclization with 1,2-diketones under refluxing toluene to furnish highly substituted and polycyclic imidazoles (Scheme 8).

![Scheme 8](image)

R\textsuperscript{1} = Ph, 4-Br-Ph, CH\textsubscript{3}, CH\textsubscript{2}CH\textsubscript{3},
R\textsuperscript{2} = Ph, 4-OCH\textsubscript{3}-Ph, 4-CH\textsubscript{3}-Ph, pyridine

**Scheme 8**

Wu et al.\textsuperscript{48} have reported the synthesis of various poly-substituted 2-[(pyridin-2-yl)imidazoles from 2-cyanopyridine, corresponding aromatic aldehydes and NH\textsubscript{4}OAc/primary amine (Scheme 9).

![Scheme 9](image)

R\textsuperscript{1} = 4-Cl, 4-OCH\textsubscript{3},
R\textsuperscript{2} = H, 4-CH\textsubscript{3}, 4-Cl, 4-Br, 4-OCH\textsubscript{3}

**Scheme 9**

Tlahuext-Aca et al.\textsuperscript{49} have reported Ni(0)-catalyzed dehydrogenation of benzylic-type imines to yield asymmetrical tetra-substituted imidazoles (Scheme 10).

![Scheme 10](image)

R = Phenyl

**Scheme 10**
Safari et al.\textsuperscript{50} have reported a series of 1,2,4,5-tetrasubstituted imidazole derivatives by using Fe\textsubscript{3}O\textsubscript{4}–PEG–Cu as a highly effective and heterogeneous catalyst (Scheme 11).

![Scheme 11](image)

\textit{Ar} = H, p-OMe, m-OMe, p-Cl, 2-Naphthyl, m-OH, m-NO\textsubscript{2}, m-Br, p-Me, p-OH, p-(Me)\textsubscript{2}-N

Rajaguru et al.\textsuperscript{51} have reported erbium triflate promoted multicomponent synthesis of highly substituted imidazoles (Scheme 12).

![Scheme 12](image)

\( R_1 = H, \text{CH}_3, \text{Br}, \text{Cl}, \text{NO}_2 \); \( R_2 = \text{H,OCH}_3, \text{CH}_3 \); \( R_3 = \text{H,OCH}_3, \text{CH}_3 \); \( R_4 = \text{H, Br} \)

1.3.3. Pharmacological studies

Imidazole ring system exhibited a variety of pharmaceutical activities\textsuperscript{52–54} and plays a vital role in biochemical processes. The core structure of imidazole is found as a structural part of many important biological molecules like histidine, histamine and biotin as well as several drug moieties\textsuperscript{55} such as Trifenagrel (2), Eprosartan (3) and Losartan (4). It is known that clinically useful drugs such as miconazole (5), econazole (6) and oxiconazole (7) containing imidazole moiety exhibit strong antifungal activity. Imidazole derivatives have novel therapeutic activities, inhibitors of p38 MAP kinase,\textsuperscript{56} herbicides\textsuperscript{57} and plant-growth regulators.\textsuperscript{58} Furthermore, recent reports indicate that imidazoles are cytotoxic,\textsuperscript{59}
anticonvulsant,\textsuperscript{60} anti-inflammatory,\textsuperscript{61} analgesic\textsuperscript{62} and potent inhibitors of protein–protein interactions.\textsuperscript{63}

Malhotra \textit{et al.}\textsuperscript{64} have reported a series of novel substituted imidazole derivatives. The compounds 8-15 have shown good hypotensive \& bradycardiac responses. Compounds 10, 11, 12 and 15 have shown better activity than reference drug clonidine.
Gleave et al. have reported a series of tetrasubstituted imidazoles as P2X7 antagonists. Compound 16 was identified as a potent P2X7 antagonist with reduced in vitro metabolism and high solubility.

Iradyan et al. have reported a series of thiosemicarbazides and hydrazonohydrazides of 4-nitroimidazole-5-thioacetic acid derivatives. All the compounds exhibited antimutagen properties. Some compounds exhibited moderate antitumor activity.
Saberi et al.\textsuperscript{67} have reported a series of tetrasubstituted imidazoles. All the compounds 28-33 showed good antibacterial and antifungal activities.

Liverton et al.\textsuperscript{68} have reported a novel potent and selective diaryl imidazoles (34 and 35) as inhibitors of p38 MAP (mitogen-activated protein) kinase which have shown activity in both cell-based assays of tumor necrosis factor-\(\alpha\) (TNF-\(\alpha\)) release and an animal model of rheumatoid arthritis.
Ozkay et al. have synthesized many novel imidazole-(Benz) azole and imidazole-piperazine derivatives. Anticancer activity screening results revealed that 36, 37 and 38 were the most active compounds in the series. Cisplatin was used as the reference drug in that study.

Jayabharathi et al. have synthesized a series of substituted imidazoles (39-42). These synthesized compounds have exhibited better structure activity and thus in future these compounds may be used as templates to generate better drugs to fight against bacterial and fungal infections.
1.4. 2-(PIPERAZIN-1-YL)ETHANAMINE

1.4.1. Pharmacological studies

A majority of bioactive molecules are heterocyclic compounds. Among them, piperazine and its derivatives have played a significant role in medicinal chemistry. Piperazine is a small molecule with a rigid backbone with numerous biological activities such as anticancer,71,72 calcium channel blockers,73,74 antimalarial,75 antihistamine,76 antimicrobial,77,78 antidepressant,79 antioxidant,80 antiallergic,81 antiviral,82 antipsychotic83 and antiparasitic.84,85 Many potent marketed drugs like Prozosin (Anti-Hypertensive Drug) 43, flunarizine (Calcium Channel Blocker) 44, cinnarizine, lomerizine, fluphenazine, ciprofloxacin (Antibiotic) 45, Merck HIV protease, crixivan, etc. have a piperazine nucleus in their structure. The various biological activities of piperazine nuclei are due to easy modification, proper alkalinity, water solubility, capacity for formation of hydrogen bonds and adjustment of molecular physicochemical properties.86
1.4.2. LITERATURE REVIEW ON 2-(PIPERAZIN-1-YL)ETHANAMINE

Shingade *et al.*\(^87\) have reported a series of novel aryl-3-(2-piperazin-1-ylethyl)-1,3-thiazolidin-4-ones \(46-49\). The compounds \(46-49\) exhibited significant antibacterial as well as antifungal activities.

![Chemical Structure](image)

Sondhi *et al.*\(^88\) have tested a series of mono and bis-Schiff's bases. Compound \(50\) exhibited good anticancer activity.

![Chemical Structure](image)

Demirci *et al.*\(^89\) have reported a series of bi- and tri-heterocyclic azoles. The antimicrobial screening suggests that among the newly synthesized compounds, \(51\) exhibited excellent activity against most of the test microorganisms.
Karagoz et al.\textsuperscript{90} have synthesized a series of $N$-ethylpiperazine substituted thioureas and their copper (II) complexes (52-54). All the compounds were tested for their anticancer activity against MCF-7 and L1210 cell lines. In addition, synthesized compounds showed remarkable ferrous ion chelating and radical scavenging activities on DPPH and ABTS radicals. These compounds (52-54) have shown higher antioxidant activity than standard antioxidant trolox.
1.5. 2-MORPHOLINOETHANAMINE

1.5.1. Pharmacological studies

$N$-Alkylmorpholines play an important role in the production of many chemical compounds including surfactants, dyes, pesticides, preservatives, herbicides, rubber accelerator, lubricants and medicinal intermediates.$^{91-93}$ For example, $N$-alkylmorpholines can be used as important blocks in many pharmaceutical compounds such as moclobemide 55 for depression, fominoben 56 for antitussive, morphocycline for infection and the diagnosis of lung cancer, bimolane 57 for psoriasis and so on. Given its versatility, exploring more efficient synthesis of $N$-alkylmorpholines is still attracting much attention.$^{94,95}$

\[ \text{\includegraphics[width=\textwidth]{molecules.png}} \]

1.5.2. LITERATURE REVIEW ON 2-MORPHOLINOETHANAMINE

A series of novel $N$-hydroxyalkyl-2-aminophenothiazines has been developed by Takácsa et al.$^{96}$ MDR inhibition studies on rat hepatocyte cell culture revealed that compound 58 exhibit marked biological efficacy exceeding that of the standard verapamil.

\[ \text{\includegraphics[width=\textwidth]{molecules.png}} \]
Kumar et al.\textsuperscript{97} have reported a series of piperazine-2,6-dione and 4-\((1H\text{-}\text{indole-2-carbonyl})\text{piperazine-2,6-diones. These compounds were screened for anticancer activity against five human cancer cell lines. Compounds 59 and 60 exhibited good anticancer activity.}

\begin{center}
\includegraphics[width=0.5\textwidth]{images}
\end{center}

Nama et al.\textsuperscript{98} have synthesized aminoalkyl-substituted coumarin derivatives. Compound 61 did not affect the general behavior of mice.

\begin{center}
\includegraphics[width=0.3\textwidth]{images}
\end{center}

Sahin et al.\textsuperscript{99} have synthesized 1,2,4-triazole derivatives containing morpholine. Among the newly synthesized compounds, 62 showed excellent antimicrobial activity against most of the tested microorganisms. Compounds 63 and 64 exhibited better activity against the Gram-positive bacteria. Compounds 65-67 showed antibacterial activity against Gram-positive and Gram-negative bacterial strains and mycobacterium smegmatis.
Sambrekar et al.\textsuperscript{100} have synthesized a series of 3-substituted-4-amino-5-mercapto-4(\(H\))-1,2,4-triazoles. Compound 68 has shown significant anti-convulsant activity in METS method by using carbamazepine as the standard.

Zheng et al.\textsuperscript{101} have synthesized a novel series of pyrazolo [1,5-a]pyrazin-4(5\(H\))-ones. Compounds 69, 70 and 71 displayed significant effects on the growth of A549 and H322 cells.
1.6. PYRIDIN-4-YLMETHANAMINE

1.6.1. Pharmacological studies

Among the nitrogen-containing heterocycles, pyridine derivatives constitute one of the most important classes of compounds as they widely occur as key structural subunits in numerous natural products that exhibited various types of biological activities *viz.* antimicrobial, antibacterial, antimycobacterial, anticonvulsant, antitumoral, cytotoxic, antimalarial, antidiabetic and pesticidal. These derivatives possess a large spectrum of biological activities like anti-prion, anti-hepatitis B virus, anticancer, antimicrobial, anticonvulsant, antiviral, anti-HIV, antifungal and antimycobacterial activities. Pyridine ring also plays important role in antidiabetic activity of some drugs pioglitazone and rosiglitazone.

![Chemical structure of pioglitazone (72) and rosiglitazone (73)]

1.6.2. LITERATURE REVIEW OF PYRIDIN-4-YLMETHANAMINE

Mitchell *et al.* have reported a series of imidazo[1,2-a]pyrazine diarylureas. The most active molecules and were found to have potent activity (<100 nM) against the angiogenesis-related kinases VEGFR2 and Tie2.
Mokale et al.\textsuperscript{129} have reported a series of substituted imidazol-5-ones. It has been observed from the \textit{in vitro} screening that newly synthesized compounds possess RT inhibitory activity. Among the synthesized compounds 77, 78, 79 and 80 showed more significant RT inhibitory activity and their IC\textsubscript{50} values were 3.9, 3.5, 2.5 and 3.8 lM, respectively.
Kumar et al.\textsuperscript{97} have tested a series of 4-\textit{\{1H-indole-2-carbonyl\}piperazine-2,6-dione}. Compound \textbf{81} exhibited good anticancer activity.

Kumar et al.\textsuperscript{130} have synthesized isoindole, pyrrolopyrazine, benzimidazoisoindole and benzimidazopyrrolopyrazine derivatives. Compound \textbf{82} exhibited good anti proliferative activity while the compound \textbf{83} exhibited good anti-inflammatory activity.
1.7. SULPHATED METAL OXIDE CATALYST

More than 90% of chemical processes employ catalysts. Among these processes, acid catalysts play a substantial role in organic synthesis and transformations. Currently, most of these acid-catalyzed reactions are catalyzed by conventional acids such as H$_2$SO$_4$, HNO$_3$ and HF or Lewis acids such as AlCl$_3$ and BF$_3$, which exhibit significant disadvantages in handling, containment and disposal because of their toxic and corrosive nature. Now-a-days sulphated metal and mixed metal oxides gained much more recognition for their significant catalytic activity than metal and mixed metal oxides due to their higher number of acid sites and larger surface area, which resulted in enhanced catalytic activity.\textsuperscript{131-135} The additional advantages of sulphated metal oxides are non toxicity, non corrosiveness, easily handling, low cost and easy to recover and recycling.

1.7.1. LITERATURE REVIEW OF SULPHATED METAL OXIDE CATALYST

Reddy \textit{et al.}\textsuperscript{136} have reported sulfated zirconia as an efficient catalyst for organic synthesis and transformations Schemes\textsuperscript{13-17}. Simple work up procedure, mild reaction conditions and shorter reaction time are some of the advantages associated with the sulfated zirconia catalyzed processes.

\begin{equation}
R_1\text{-CHO} + \text{Me}\text{-C}\text{O}\text{-CO}\text{-OR}_2 + \text{H}_2\text{N} \text{-NH}_2 \xrightarrow{\text{solvent-free}} R_2\text{-C}\text{O}\text{-NH}_2
\end{equation}

\textbf{Scheme-13}

\begin{equation}
X = O,S
\end{equation}
Kahandal et al.\textsuperscript{137} have reported sulphated mixed metal oxides as an efficient catalyst for the synthesis of 5,6-unsubstituted 1,4-dihydropyridines (\textbf{Scheme 18}).

Chavan et al.\textsuperscript{138} have reported sulphated SnO\textsubscript{2} as efficient catalyst in facile \textit{trans} esterification of ketoesters (\textbf{Scheme 19}).
Kahandal *et al.*\(^{139}\) have reported that the sulphated yttria–zirconia efficiently catalyzed the epoxide ring opening with a variety of alcohols under solvent free conditions to give corresponding β-alkoxy alcohols *(Scheme 20).*

![Scheme 20](image)

### 1.8. YTTRIUM OXIDE

Yttrium oxide, also known as yttria (Y\(_2\)O\(_3\)), is an air-stable, white solid substance. Yttria is a promising high temperature stable ceramic material which has high melting point and low thermal expansion coefficient.\(^{140,141}\) A dense, nano-grained yttria exhibits transparency over a wide wavelength region and low optical emissive property. Dense yttria finds applications as chemically stable high temperature substrates, crucibles for melting reactive metals, nozzles for jet-casting of molten rare earth iron magnetic alloys, cutting tools, IR windows in rockets and luminous pipes.\(^{142,143}\) It is also known as a recognized host matrix material for phosphors to be used in televisions. Synthesis of nanocrystalline yttria is extremely important because they exhibit high surface area and offer low temperature sintering for achieving nano-grained sintered microstructures.\(^{144}\)

### 1.9. SPECTROSCOPY

#### 1.9.1. IR Spectroscopy

Infrared (IR) radiation refers broadly to the part of the electromagnetic spectrum between the visible and microwave region. The greater practical use to the field of organic chemistry is
the limited portion between 4000 and 400 cm$^{-1}$. Absorption bands in the spectrum result from energy changes due to molecular vibrations of the stretching and bending modes of a bond. This absorption is quantized; vibrational spectrum appears as bands rather than a line because a single vibrational energy change is accompanied by a number of rotational energy changes. Band positions in infrared spectra are presented as wavenumber ($\nu$) or wavelength ($\lambda$).

Even a very simple organic molecule can give extremely complex infrared spectrum. The organic chemist takes advantage of this complexity when one matches the spectrum of an unknown compound against that of an authentic sample. A peak-by-peak correlation is an excellent evidence for same infrared spectrum. Since the structural elucidation/identification do not solely dependent on infrared spectrum, a detailed analysis of the spectrum will not be required. In the present study, IR spectra are utilized in conjugation with other spectral data to determine the molecular structure.

1.9.2. NMR Spectroscopy

1.9.2.1. 1D-NMR spectroscopy

Nuclear magnetic resonance (NMR) spectroscopy is a well established technique for providing information about structural diagnosis of organic molecules. It involves transition of a nucleus from one spin state to another state with the resultant absorption of electromagnetic radiation in the radio wave frequency region by spin active nuclei when they are placed in a magnetic field. The energy associated with NMR experiment is incapable of disrupting even the weakest chemical bond in a molecule. One dimensional NMR spectrum constitutes a plot of the frequencies of the absorption peaks $versus$ peak intensities.
1.9.2.2. Coupling constant

Vicinal coupling constant is a $^1$H NMR parameter, most frequently employed in configurational and conformational studies. In a saturated system the vicinal coupling constant between two protons depends on the dihedral angle between them. Karplus using valence bond calculations,\textsuperscript{146} showed that the coupling constant describes an asymmetrical U shaped curve on increasing the torsional angle between vicinal protons from 0 to $180^\circ$ and also pointed out that vicinal coupling constant depends upon other factors \textit{viz.} the electro negativity of groups attached to carbon, C-C bond length and H-C-C angle.

1.9.2.3. 2D-NMR Spectroscopy

In one dimensional NMR spectra, intensities against frequencies of absorption are plotted. In recording 1D–NMR spectroscopy, during the detection of signals, time is varied. The magnification along Y-axis is plotted as a function of time and this plot is called Free Induction Decay (FID). Generally, NMR spectrum is plotted after measuring several FIDs. Moreover, it is possible to collect FIDs by using various time intervals between two pulse sequences. Thus, each FID is recorded for different time intervals. Hence, the data are acquired as a function of two time parameters $t_1$ and $t_2$. After Fourier Transformation, the data can be obtained as a function of two different frequencies. Thus, intensity is plotted as a function of two frequencies. Both may be for $^1$H and another may be for $^{13}$C. Several types of 2D NMR spectra can be recorded. Different kinds of information such as nuclei involved in coupling and protons involved in NOe can be obtained from 2D NMR spectra.
1.9.2.3.1. **HSQC**

This two dimensional NMR technique correlates $^{13}$C nuclei with directly attached protons. The sensitivity is very high since the experiment is proton detected $^1$H-$^{13}$C correlation. Only one bond couplings ($^1J_{C,H}$) are detected. Two and three bond carbon-hydrogen couplings are eliminated.

1.9.2.3.2. **DEPT**

As the name implies, sensitivity is increased by polarization transfer from the more sensitive coupled proton(s) to the less sensitive $^{13}$C atom. DEPT spectrum does not show the signals corresponding to quaternary carbons.

1.9.3. **Mass Spectrometry**

The mass spectrometry is also an important tool for the structural elucidation of organic compounds. The substance under identification is bombarded with a high energy electron beam which produces mostly singly charged positive ions. These ions are separated on the basis of mass to charge ratio and the result of electron impacts are recorded on a spectrum. By considering the combination of these ions, the structure of original molecule can be reconstructed.

Generally, in electron impact or chemical ionization mass spectroscopy, the carrier gas (methane gas) is ionized by electron impact. This in turn produces the primary ions followed by secondary ions. The secondary ions usually react with the organic molecule under study thereby produce the ions characteristic for the molecule and its fragments. Two common categories of mass spectrometry are high resolution mass spectrometry (HRMS) and low resolution mass spectrometry (LRMS). Not all mass
spectrometers simply measure molecular weights as whole numbers. High resolution mass spectrometers can measure mass so accurately that they can detect a minute difference in mass between two compounds that, on a regular low-resolution instrument, would appear to be identical. The reason is because atomic masses are not exact multiples of the mass of a proton, as we might usually think. An atom of $^{12}\text{C}$ weighs 12.00000 amu, $^{16}\text{O}$ weighs 15.9949 amu, $^{14}\text{N}$ weighs 14.0031 amu, $^{1}\text{H}$ weighs 1.00783 amu. Thus a high resolution mass spectrometer can supply an exact molecular formula for a compound because of the unique combination of masses that result. In LRMS, the molecular weight is determined to the nearest amu.

In HRMS, the molecular weight in amu is determined to several decimal places. That precision allows the molecular formula to be narrowed down to only a few possibilities. HRMS relies on the fact that the mass of an individual atom does not correspond to an integral number of atomic mass units.

**1.10. SINGLE CRYSTAL X-RAY CRYSTALLOGRAPHY**

Single crystals for the determination of structure were chosen by an examination under polarising microscope. A Polaroid photograph of a good specimen mounted on a three-circle goniometer was taken to ensure the good quality of the crystal. For intensity data collections graphite monochromated Mo$K_\alpha$ and Cu$K_\alpha$ radiations were used. The data were collected School of Chemistry, University of Hyderabad, India (Oxford and Bruker Smart Apex–II CCD diffractometers using graphite monochromated Mo$K_\alpha$ radiation).
1.11. **SCOPE OF THE PRESENT INVESTIGATION**

Heterocyclic ring systems having imidazole have aroused great interest in the past and recent years due to their wide variety of biological properties and their presence in biologically active pharmaceutical ingredients. Piperazine moiety containing heterocyclic compounds have also attracted much attention as they display diverse biological and pharmacological properties. Hence, this eventually formed a new basis and opened up a horizon to synthesize new series of systems *viz.* 1-(2-(2,4,5-triphenyl-1H-imidazol-1-yl)ethyl)piperazines, 1-(2-(4,5-dimethyl-2-phenyl-1H-imidazol-1-yl)ethyl)piperazines, 4-(2-(4,5-dimethyl-2-phenyl-1H-imidazol-1-yl)ethyl)morpholines and 4-((2,4,5-triphenyl-1H-imidazol-1-yl)methyl)pyridines. It was envisaged that the new series of synthesized compounds are expected to endow with broad spectrum of biological properties. Hence, the possible antibacterial and antifungal potency of the synthesized compounds are also explored and discussed.

All the synthesized compounds have been characterized by FT-IR, $^1$H & $^{13}$C NMR and Mass spectra. Single crystal XRD analysis also carried out to confirm the structure of the selected synthesized compounds.