PART - II

SYNTHESIS OF THIOPYRIMIDINES, PYRAZOLE & CHROMONES
SECTION - A

SYNTHESIS OF 4-(2-HYDROXY PHENYL)-6-(1,3-DIPHENYL-1H-PYRAZOL-4-YL) PYRIMIDINE-2(1H)-THIONES
2.1.1. INTRODUCTION:

Pyrimidine & thio pyrimidine derivatives have acquired unique importance in recent past. Pyrimidine is the parent substance of a large group of heterocyclic compounds. Compounds belonging to this group were known as breakdown products of uric acid at very early date in the history of organic chemistry.

Pyrimidine forms an integral part of large number of therapeutically important compounds like thiamine, riboflavin, purine bases, sulfadiazine etc. The role of pyrimidine derivatives in biochemical processes is now known with reasonable accuracy. The researchers in this field are regularly adding to our knowledge of chemistry & biochemistry of pyrimidine derivatives.

Most of the pyrimidine derivatives have been found to have physiological activity and are used as drugs. Pyrimidine derivatives like pyridoxine, thiamine, phenobarbital, pentobarbital, mysoline etc. are also well known in pharmacology. Many synthetic members of this group are also important as synthetic drugs e.g. barbituric acid and its derivatives.

Gabriel & coloman first isolated pyrimidine in 1899. Though pyrimidine does not exist in nature but substituted pyrimidine moiety are found as a part of more complex system and are widely distributed. Pyrimidines are considered to be important not only because of they are integral part of the genetic material viz. DNA & RNA as nucleosides & nucleotides but they also impart numerous biological activities such as bactericides, fungicides, vermicides & insecticides. They have found applications in agricultural & industrial chemicals.
In recent years pyrimidine derivatives have received significant attention owing to their diverse range of biological activities, particularly being calcium channel blockers\(^{(1)}\).

Girardet found 4-amino 5-oxopyrido [2,3-\(d\)] pyrimidine riboside was very potent inhibitor of cancer cell proliferation \(^{(2)}\). Recently Kidwai M. and co-workers have studied the anti-malarial properties of 2,4 di-amino -5-parachlorophenyl -6-ethyl pyrimidines\(^{(3)}\).

Pyrimidines & their derivatives are studied by Wichman J. and Tsuji K. for their biological activities\(^{(4-5)}\). Machon & Krystyna have studied analgesic and anti-inflammatory activities of pyrimidine derivatives\(^{(6)}\).

Pyrimidin-2-ones or pyrimidin-2-thiones are important class of heterocycles possessing a wide range of biological activities\(^{(7)}\).

The pyrimidines used as an anti-malarial & antibacterial agents are

![Uracil](image)

![Pyrimethamine](image)
Schmidt and Genter\(^{(8)}\) have studied synthesis and anti-malarial properties of 2,4 diamino-5-para chloro phenyl-6-ethyl pyrimidine.

\[
\begin{align*}
\text{NH}_2 & \quad \text{NH}_2 \\
\text{H}_2\text{N} & \quad \text{N} \\
\text{N} & \quad \text{CH}_2\text{CH}_3 \\
\end{align*}
\]

Grimaux\(^{(9)}\) condensed urea with malonic acid in presence of \(\text{POCl}_3\) and obtained barbituric acid.

\[
\begin{align*}
\text{H}_2\text{N} & \quad \text{NH}_2 \\
\text{COOH} & \quad \text{COOH} \\
\end{align*}
\]

Later this method was modified by Michael\(^{(10)}\) by condensing malonic ester with urea using sodium alkoxide as a catalyst. This process finds application in the preparation of barbiturate drugs.\(^{(11)}\) Traube\(^{(12)}\) in 1900 found that the alkali-catalyzed addition of an amino to cyano group is convenient process. Rupe et al and Bergmann\(^{(13)}\) have synthesized 4-amino 2,6-dihydroxy pyrimidine from cyano acetylurea.
Biginelli\textsuperscript{(14)} synthesized dihydro-pyrimidines by the condensation of urea, \(\beta\)-ketoester and aldehyde.

Folker & Johnson\textsuperscript{(15)} have synthesized 2-keto-4-benzyl-5-phenyl-1-2-3-4-tetra hydro-pyrimidines by condensing phenyl acetaldehyde, acetophenone, and urea in absolute ethyl alcohol in presence of conc.HCl.

Condensation of \(\beta\)-diketone with an aldehyde & urea\textsuperscript{(16)} by conc.HCl proceeded smoothly by Biginelli reaction.
Similarly thio-urea undergoes all cyclisation reactions of urea with considerably greater extent.

Mc.Casland et al \(^{(17)}\) has synthesized various 1,4-dihydro-2-pyrimidine thiols from chalcones and ammonium thiocynate (NH4SCN) under reflux.

Chemische et al\(^{(18)}\) prepared various 2-thio-4-hydroxy tetra hydro pyrimidines by refluxing \(\alpha,\beta\)-unsaturated ketone with thiourea using sodium & methanol.
Zimmermann & et al\textsuperscript{(19)} condensed various \( \beta \)-hydroxy and \( \alpha,\beta \)-unsaturated ketones with thiourea in the presence of alkali & alcohol to prepare corresponding pyrimidines.

The various pharmacologically active 1-alkyl, 4,6-diphenyl, 2-(1H) pyrimidinones & pyrimidine thiones have been reported by Hardtmann et al\textsuperscript{(20)} Several thiopyrimidines and sedatives are prepared by condensation of N-acetyl thiourea with chalcones in alcoholic KOH.

Durganath Dhar et al\textsuperscript{(21)} have carried out the reaction of thiourea with chalcones in acidic medium and isolated various 2-oxo-1,3-thiazines.
Kaname Takagi, Michel Hubert-Habert & collaborators have done extensive work on pyrimidine & thio-pyrimidines. They have synthesized pyrimidine derivatives from oxygen containing heterocycles by reaction with urea & thiourea.

Benzofuran condensed with urea gives pyrimidines.

Condensation of thiourea with 3-cyano or 3 carbonyl benzofuran in presence of sodium ethoxide yield the corresponding 2-thio pyrimidines.\textsuperscript{(22-25)}
Musante & Fattuma (28) carried out the reactions of guanidine on Khellin & obtained the corresponding 2-amino pyrimidines.

Hubert-Habert & et al (27) have reported reactions of guanidine, thiourea, urea & cyano guanidine (28) on various chromones in presence of sodium ethoxide & obtained corresponding pyrimidine derivatives.
Hubert-Habert & coworkers\(^{(29)}\) have also reported pyrimidines from chromones without substituent at 2-position. They have reported two isomeric products.

Out of these two structures (I) is observed in higher amount.

C.H. Gill & Thakar\(^{(30)}\) carried out reaction of thiourea on 2-phenyl chromones in alcoholic KOH.
2.1.2. PRESENT WORK :-

In the present investigation the 4(2-hydroxy phenyl),6-(1,3,di phenyl -1H-pyrazol-4-yl) 1,2, dihydro-2-pyrimidine-thiones 2 are prepared by the reaction of thiourea on 2(1,3,di phenyl -1H -pyrazol -4-yl) chromones, in alcoholic KOH.

![Reaction Scheme]

Reflux Alco.KOH

1

2
2.1.3. EXPERIMENTAL :-

General procedure for synthesis of 4(-2-hydroxy phenyl) 6-(1,3-diphenyl -1H- pyrazol -4-yl) 1,2-dihydro- 2-pyrimidine thiones.

A mixture of 1 (0.003 mole) & thiourea (0.005 mole) was dissolved in 10 ml ethanol. To this reaction mixture KOH pellets (0.005 mole) was added. The reaction mixture was heated under reflux for three hours. After completion of heating, reaction mixture was cooled to room temperature & then poured over crushed ice and neutralized with acetic acid. Resulting product was separated by filtration and crystallized from ethanol. Their m.p.& percentage yield are given in table.5

<table>
<thead>
<tr>
<th>Compound no.</th>
<th>R1</th>
<th>R2</th>
<th>R3</th>
<th>R4</th>
<th>% Yield</th>
<th>M.P. °C</th>
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<tbody>
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<td>H</td>
<td>H</td>
<td>Cl</td>
<td>H</td>
<td>55</td>
<td>285</td>
</tr>
<tr>
<td>2b</td>
<td>CH₃</td>
<td>H</td>
<td>CH₃</td>
<td>H</td>
<td>49</td>
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</tr>
<tr>
<td>2c</td>
<td>Cl</td>
<td>H</td>
<td>Cl</td>
<td>H</td>
<td>59</td>
<td>210</td>
</tr>
<tr>
<td>2d</td>
<td>H</td>
<td>H</td>
<td>F</td>
<td>H</td>
<td>55</td>
<td>235</td>
</tr>
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<td>H</td>
<td>CH₃</td>
<td>Cl</td>
<td>H</td>
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<td>299</td>
</tr>
<tr>
<td>2f</td>
<td>H</td>
<td>H</td>
<td>CH₃</td>
<td>H</td>
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<td>262</td>
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<td>CH₃</td>
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<td>H</td>
<td>56</td>
<td>252</td>
</tr>
<tr>
<td>2h</td>
<td>Cl</td>
<td>H</td>
<td>Cl</td>
<td>CH₃</td>
<td>60</td>
<td>295</td>
</tr>
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</table>
2.1.4. DISCUSSION OF SPECTRA : -

I.R. spectra:

I.R. spectra of representative compounds of this series were scanned by using Perkin Elmer FT spectrophotometer.

I.R spectrum (KBr disc.) of (2a) showed following characteristic absorption bands.

3444 cm\(^{-1}\) due to O-H stretching

1635 cm\(^{-1}\) due to C= N stretching

1460 cm\(^{-1}\) due to C- N stretching
NMR spectra:

The NMR spectra of representative compounds of this series were scanned on Varian 300 MHz spectrophotometer using DMSO-$d_6$ and CDCl$_3$ as solvent and TMS as an internal standard.

Compound (2a) exhibited following characteristic NMR signals in δ ppm

| 14.4 | 1H singlet | OH |
| 13.9 | 1H singlet | N-H |
| 9.3  | 1H singlet | Pyrazole-H |
| 7.2-8.0 | 13H multiplet | Ar- H |
| 7.0  | 1H singlet | Pyrimidine-H |
2.1.5. REFERENCES :-


12) Traube. *Ber.* 33,1371,3035 (1900).


PART - II

SECTION - B

SYNTHESIS OF 2(5-(1,3-DIPHENYL-1H-PYRAZOL-4-YL)-1H-PYRAZOL-3-YL) PHENOLS
2.2.1. INTRODUCTION :-

In heterocyclic chemistry pyrazole derivatives find wide applications\(^1{\text{-}2}\). Pyrazole ring is an important constituent of various natural & synthetic products. Example is Lonazolac,\(^3\) Fezolamin,\(^4\) Defenamizole,\(^5\) and Mepirizole.\(^6\) Lonazolac are new anti-inflammatory drugs\(^7\).
Some naturally occurring biologically active compounds containing pyrazole ring are 4,5-dihydro-3-phenyl-6H-pyrrolo[1-2-b]pyrazole\(^\text{®}\) pyrazomycine \(^\text{®}\) and (S)-3-pyrazolyl alanine\(^\text{°}\).

\[
\begin{align*}
\text{Ph} & & \text{OH} \\
\text{NH}_2 & & \text{COON} \\
\text{NH} & & \text{OH} \\
\text{Ph} & & \text{NH}_2 \\
\end{align*}
\]

4,5-dihydro-3-phenyl 6H-pyrrolo(1,2-b) pyrazole  

\[
\begin{align*}
\text{HO} & & \text{NH}_2 \\
\text{OH} & & \text{OH} \\
\text{OH} & & \text{OH} \\
\end{align*}
\]

pyrazomycin

\[(S) \text{ 3-Pyrazolylalanine}\]

Lesopitron is a new nonbenzodiazepine anxiolytic without side effects which is currently in phase II trial \(^\text{\textsuperscript{11}}\).

\[
\begin{align*}
\text{Cl} & & \text{Cl} \\
\text{N} & & \text{N} \\
\text{N} & & \text{N} \\
\text{N} & & \text{N} \\
\end{align*}
\]

Lesopitron

Following isopyrazoles are found to be associated with cytotoxic activity\(^\text{\textsuperscript{12}}\).
Nitropyrazole derivatives such as 4-nitro pyrazole, 1-methyl, 4-nitropyrazole and 4,4-dinitro 1-methylene dipyrazole are known as antiparasitic agents. These are more effective than penicillin, levomycetin, & polymyxin.

Pyrazole & their derivatives are also widely used in agro chemistry as insecticides, herbicides & fungicides. A powerful fungicidal composition for the protection of plants from phytopathogenic fungi containing 4-chloro,3 (3,5-dichlorophenyl) -1H-pyrazole has been patented.

Pyrazoles are usually prepared by condensation of hydrazine derivative & 1,3 dicarbonyl compounds or by 1,3 dipolar cycloaddition of diazoalkanes or nitrite imines to olefins or acetylenes. 3(dimethylamino) propionates are actually masked by 1,3-dicarbonyl compounds, they can be transformed into substituted pyrazoles upon treatment with hydrazine derivatives.
The reaction of 2-phenylchromones with hydrazines has been thoroughly studied by Kallay et al.\textsuperscript{(17-20)} The reactions of various, diaryl 1,3 diketones with hydrazine hydrate in absolute ethanol led to the formation of corresponding pyrazole derivatives.

![Reaction diagram]

Reaction of chromone with hydrazine has been reported to give 5(3) (ortho hydroxy phenyl) pyrazole.\textsuperscript{(18)}
Reaction of 2- methyl chromone with hydrazine has been reported to give 3 - (ortho-hydroxy phenyl) -5- methyl pyrazole\(^{(19)}\).

\[
\text{\begin{tikzpicture}[baseline=(current  baseline)]
  \node (a) at (0,0) [circle,draw] {$\text{CH}_3$};
  \node (b) at (2.5,0) [circle,draw] {OH};
  \node (c) at (5,0) [circle,draw] {N}
  \node (d) at (7,0) [circle,draw] {CH}_3;
  \draw (a) -- (b);
  \draw (b) -- (c);
  \draw (c) -- (d);
\end{tikzpicture}}
\]

However treatment of 2-methyl chromones with monosubstituted hydrazine can in principle, generate two isomeric products. Reaction of 2-methyl chromone with phenyl hydrazine was first investigated by Alberati\(^{(20)}\) who assigned the structure to the products as having methyl group located at 5-position.

\[
\text{\begin{tikzpicture}[baseline=(current  baseline)]
  \node (a) at (0,0) [circle,draw] {$\text{CH}_3$};
  \node (b) at (2.5,0) [circle,draw] {OH};
  \node (c) at (5,0) [circle,draw] {N}
  \node (d) at (7,0) [circle,draw] {N}
  \node (e) at (7,2) [circle,draw] {Ph};
  \draw (a) -- (b);
  \draw (b) -- (c);
  \draw (c) -- (d);
  \draw (d) -- (e);
\end{tikzpicture}}
\]

Nawrot Modranka studied the reaction of chromones with CH\(_3\)-NH-NH\(_2\).\(^{(21)}\)
2.2.2. PRESENT WORK :-

In the present investigation 2 (1,3-diphenyl -1H- pyrazol -4- yl) chromones 1 were treated with hydrazine hydrate in ethanol to afford corresponding derivatives 2-(5 (1,3-diphenyl -1H- pyrazol -4- yl )-1H-pyrazol-3-yl) phenols 3.
2.2.3. EXPERIMENTAL:

General procedure for synthesis of 2(5-(1-3-diphenyl -1H -pyrazol- 4- yl ) - 1H- 3-pyrazolyl ) phenols.

A mixture of 1 (0.003 mole ) & 0.005 mole of hydrazine hydrate was dissolved in 10 ml ethanol. Reaction mixture was refluxed for 3 hours then reaction mixture was cooled to room temperature and poured over crushed ice and neutralized with acetic acid. Resulting product was separated by filtration and crystallized from ethanol. The compounds synthesized by above procedure are listed in table 6 with their physical constants and percentage yield.

Table 6

<table>
<thead>
<tr>
<th>Compound no.</th>
<th>R1</th>
<th>R2</th>
<th>R3</th>
<th>R4</th>
<th>% Yield</th>
<th>M.P. °C</th>
</tr>
</thead>
<tbody>
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<td>3a</td>
<td>H</td>
<td>H</td>
<td>Cl</td>
<td>H</td>
<td>68</td>
<td>269</td>
</tr>
<tr>
<td>3b</td>
<td>CH₃</td>
<td>H</td>
<td>CH₃</td>
<td>H</td>
<td>48</td>
<td>230</td>
</tr>
<tr>
<td>3c</td>
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<td>H</td>
<td>Cl</td>
<td>H</td>
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<td>3d</td>
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<td>CH₃</td>
<td>H</td>
<td>H</td>
<td>46</td>
<td>247</td>
</tr>
<tr>
<td>3e</td>
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<td>CH₃</td>
<td>H</td>
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<td>H</td>
<td>Cl</td>
<td>CH₃</td>
<td>52</td>
<td>268</td>
</tr>
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2.2.4. DISCUSSION OF SPECTRA :-

I.R. Spectra:

I.R. spectra of representative compounds of this series were scanned by using Perkin Elmer FT spectrophotometer.

I.R. Spectrum (KBr disc) of 3a showed following characteristic absorption bands

3444 cm\(^{-1}\) due to O-H & N-H stretching

1458 cm\(^{-1}\) due to C=N stretching
NMR Spectra:

The NMR spectra of representative compounds of this series were scanned on Varian 300 MHz spectrophotometer using DMSO-d6 and CDCl$_3$ as a solvent and TMS as an internal standard.

Compounds 3a exhibited following characteristic NMR signals in $\delta$ ppm.

<table>
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<th>$\delta$</th>
<th>Multiplicity</th>
<th>\(n)</th>
<th></th>
<th>\(H)</th>
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<td>13.44</td>
<td>singlet</td>
<td>1</td>
<td></td>
<td>OH</td>
</tr>
<tr>
<td>10.9</td>
<td>singlet</td>
<td>1</td>
<td></td>
<td>NH</td>
</tr>
<tr>
<td>8.95</td>
<td>singlet</td>
<td>1</td>
<td></td>
<td>Pyrazole-H</td>
</tr>
<tr>
<td>6.99-8.00</td>
<td>multiplet</td>
<td>14</td>
<td></td>
<td>Ar-H and pyrazole-H</td>
</tr>
</tbody>
</table>
2.2.5. REFERENCES :


5) T. Kameyana and H. Nlvva. Japan ,68 ,06 621 (1968) Chem. Abstra. 69,106704 x


PART- II

SECTION - C

SYNTHESIS OF
2(1,3-DIPHENYL-1H-PYRAZOL -4-YL)
CHROMONES
2.3.1. INTRODUCTION :-

Flavones are usually prepared by demethylation of the corresponding methyl ethers\(^{(1)}\) which are obtained from 2-hydroxy substituted dibenzoyl methanes\(^{(2)}\). The direct conversion of 2-hydroxy substituted dibenzoylmethane to hydroxy flavones has been attempted by heating them with hydroiodic acid \(^{(1)}\). Agarwal & soni\(^{(3)}\) reported a method to synthesize chromones from chalcones.

![Chemical Reaction Diagram]

Makrandi & Surendrakumar\(^{(4)}\) reported synthesis of 2-substituted chromones from ortho hydroxy β-diketone & pyridine hydrochloride.

![Chemical Reaction Diagram]

2-hydroxy chalcones when refluxed in ethanol in presence of ethylene diamine then yield flavones \(^{(5,6)}\).

Chromon-4-ones, 2-aryl chromanones and 3-aryl chroman-4-ones are naturally occurring substances with variety of biological properties \(^{(7,8,9)}\).
There are several synthetic analogs of these compounds such as cromokalim, demiflin and flavoxate have been developed into useful medicines\textsuperscript{(10-11)}. 2,3,Dialkyl and 2,3,diaryl chroman-4-ones exhibit antiestrogen activity with strong affinity for the estrogen receptor site in human breast cancer cell cytosol\textsuperscript{(12)}.

Centchroman and 2,2-dimethyl, 3,4-diaryl chroman with trade name saheli is used as a birth control pill in India\textsuperscript{(13)}. There are few reports in literature for the synthesis of 2,3-disubstituted chromanones & chroman-4-ols\textsuperscript{(14-18)} having potential bioactivity\textsuperscript{(17-18)}.

Venkati & Krupadanam\textsuperscript{(19)} has reported synthesis of chroman-4-one from chalcone by using ethanol & HCl.

M. Giasuddin Ahmed & et al reported synthesis of chromene from cyclic $\beta$-diketone and chalcone\textsuperscript{(20)}.

The flavonoids are considered as potential for human health as well as constitute an important part of human diet. They are also considered as active principles in various medicinal plants\textsuperscript{(21)} and have been reported to possess anticancer, antioxidant, antimicrobial, antihypertensive, antiulcer, antipyretic, and, antidiabetic properties etc. Bahar Ahmed et al reported antihypertensive properties of some flavonoids\textsuperscript{(22)}.

N.S. Joshi & C.H.Gill\textsuperscript{(23)} reported synthesis of pyrazole-substituted chromones & their biological activities.
In the present work 2-(1,3-di-phenyl-1H-pyrazol-4-yl) chromones 2 are prepared by the reaction of 1-(2-hydroxy phenyl)-3-(1,3-di-phenyl-1H-pyrazol-4-yl) propan-1,3-diones 1 with succinic anhydride.
2.3.3. EXPERIMENTAL:-

General procedure for synthesis of 2(1,3-diphenyl -1H-pyrazol-4-yl) chromones

Mixture of compound 1 (0.001 mole) & 1 gm of succinic anhydride was taken in a round bottomed flask. It was dissolved in 10 ml pyridine. The reaction mixture was then heated under reflux for 2 hrs. After completion of heating contents were cooled and poured over crushed ice and acidified with acetic acid. The resulting product was separated by filtration and recrystalized from ethanol to afford pure compounds (2a-2h). The compounds synthesized by above procedure are listed in table.7 with their physical constant and percentage yield. Their structures are confirmed by I.R. & NMR spectral studies.

Table. 7

<table>
<thead>
<tr>
<th>Compound no.</th>
<th>R1</th>
<th>R2</th>
<th>R3</th>
<th>M.P. °C</th>
<th>% Yield</th>
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</table>
2.3.4. DISCUSSION OF SPECTRA :

IR spectra:

IR spectra of representative compounds of this series were scanned by using Perkin Elmer FT I.R. spectrophotometer.

IR spectrum (KBr disc) of (2b) showed following characteristic absorption bands.

- $1631 \text{ cm}^{-1}$ due to $\text{C=O}$ stretching
- $1539 \text{ cm}^{-1}$ due to $\text{C=N}$ stretching
- $1448 \text{ cm}^{-1}$ due to $\text{Ar-N}$ stretching

NMR spectra:

The NMR spectra of representative compounds in this series were scanned on Varian 300 MHz spectrophotometer using DMSO-$d_6$ and CDCl$_3$ as a solvent and TMS as an internal standard.

Compound 2b exhibited following characteristic signals in $\delta$ ppm.

- $8.75$ singlet $1\text{H}$ Pyrazole-H
- $7.9-7.24$ multiplet $12\text{H}$ Ar-H
- $6.39$ singlet $1\text{H}$ Pyran-H
- $2.29$ singlet $3\text{H}$ CH$_3$
- $2.80$ singlet $3\text{H}$ CH$_3$
2.3.5. REFERENCES :


