CHAPTER-2

SYNTHESIS OF NOVEL QUINAZOLINE COMPOUNDS
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2. [A] INTRODUCTION:

The quinazoline chemistry with its diverse biological properties like anticonvulsant\(^1\), antibacterial\(^2\), antifungal\(^3\), antitubercular\(^4\), antiviral\(^5\), and anti-HIV\(^6\) has received importance in recent years. Quinazolines have also been reported to oxidase inhibitor, tribulin\(^7\), Hydrazides of several nitrogen containing heterocyclic derivatives were reported as potential antitubercular agent\(^8\).

2. [B] PHARMACOLOGICAL APPLICATION OF QUINAZOLINE DERIVATIVES:

Gilberto et al.\(^9\) claimed that the methyl 1-(2, 6-dichloro phenyl)-2-oxo-m-tolyl-1, 2, 3, 4-tetrahydroquinazoline-7-carboxylate and methyl 1-(2, 6-dichloro phenyl)-5-(2-fluorophenyl) -2-oxo- 1, 2, 3, 4-tetrahydroquinazoline-7-carboxylate were found to have MAPK inhibitors to treat chronic inflammatory states. Similarly Stelmach et. al\(^10\) reported 7-(1-tert-butylpiperidin-4-yl)-1-(2,6-dichlorophenyl)-5-(4-fluorophenyl)-3,4-dihydroquinazolin-2(1H)-one as potent, orally bioavailable inhibitor.

Where R=3-C\(_6\)H\(_4\)CH\(_3\), 2.R=2-F- C\(_6\)H\(_4\)
Ozaki et al.\textsuperscript{11} and Sadanandam et al.\textsuperscript{12} synthesized several 1, 2-disubstituted dihydro quinazolines.

A promising compound, such as 1-(phenethyl)-2-(2-ethoxyphenyl)-2, 3-dihydro-4(1H) quinazolone is considered as an anti-inflammatory agent.

Schlapbach et al.\textsuperscript{13} presented dihydro Quinazolinone with P38 kinase (MAPK) inhibitors as anti-inflammatory agents.

Piaz and Giovannoni\textsuperscript{14}, Lowe III et al.\textsuperscript{15} and Cottam et al.\textsuperscript{16} claimed some of the most interesting nitraquazone derived phosphodiesterase-4 (PDE 4) inhibitors as promising agents for the treatment of asthma.
Chapter 2

Quinazolinones as Antifungal agents

Ghorab et al (2000) synthesized key intermediate octahydroquinazoline obtained in one pot synthesis by a modification of the Biginelli reaction with phenacyl bromide and bromo malononitrile to furnish thiazolo [2, 3-b] quinazoline and they found the interaction of compound with formamide, formic acid and phenyl isothiocyanate yielded the corresponding pyrimidino thiazolo [2, 3-b] quinazolines and evaluated for their antifungal activity against Candida albicans. 17

\[ \text{Quinazolinones as Antimutagenic agents} \]

Kohli et al (2009) synthesized quinazolinone derivatives by treating 2-Chloro-N-(4-oxo-2-phenylquinazolin-3(4H)-yl) acetamide with the different substituted phenols in presence of anhydrous potassium carbonate & catalytic amount of potassium iodide in dry acetone and chemical structures of the synthesized compounds were confirmed by means of their m.p., TLC, IR and 1HNMR data and they reported that the synthesized compounds were evaluated for antibacterial activity by cup plate method by measuring inhibition zone 18.

Quinazolinones as Antimutagenic agents

Cakici et al (2010) synthesized (S)-4-aminoquinazoline alcohols a simple synthetic method for the preparation of enantiomerically pure from (S)-quinazolinone alcohols by key steps including chlorination, nucleophilic ipso substitution, and deacetylation is presented. Mutagenic and antimutagenic properties of the (S)-4-
Aminoquinazoline alcohols were investigated by using Salmonella typhimurium, and Escherichia coli tester strains at 0.01, 0.1, and 1 µg/plate concentrations. (S)-4-aminoquinazoline alcohols were found to be genotoxically safe at the tested concentrations. Among the tested (S)-4-aminoquinazoline alcohols, the best antimutagenic activity was obtained with a methyl derivative at 0.1 µg/plate dose.10

Quinazolinones as Anticoccidial agents

Changwen et al (2010) synthesized a series of 3-(2-(2-methoxyphenyl)-2-oxoethyl) quinazolinone derivatives as anticoccidial agents by modifying the quinazoline ring of febrifugine against Eimeria tenella. 3-(2-(2-methoxyphenyl) 2-oxoethyl) quinazolinone derivatives possesses high anticoccidial activity and may serve as a lead compound for the development of anticoccidial drugs in the future.20

Quinazolinones as Anticonvulsant agents

Aly et al (2010) synthesized novel 3-aryl-4(3H)-quinazolinone-2-carboxaldehydes, their corresponding Schiff’s base and thio- semicarbazones derivatives and reported Compounds below as anticonvulsants.21
Quinazolinones as Anti-inflammatory agents

Kumar et al (2003) synthesized various 2-(substituted phenyl methylene imino) amino acetyl methylene-3-(2'-substitutedindol-3'-yl)-halosubstituted-4(3H) quinazolinone and 2-(substituted phenyl amino methylene acetyl-4'-oxo-1'-thiazolidinyl-3-(2''-substituted indol-3''-yl)-4-(3H)-quinazolinones and reported that compound exhibited good anti-inflammatory activity.\(^{22}\)

Balakumar et al\(^{23}\) (2010) synthesized a series of novel 8 or 10-trifluoromethyl-substituted-imidazo [1, 2-c] quinazolines and evaluated for their anti-inflammatory activity.
2.[C]. REACTIVITY OF QUINAZOLINE:

Quinazolines is stable in cold dilute acid and alkaline solutions, but it is destroyed when these solutions are boiled. 2- Aminobenzaldehyde, ammonia and formic acid are formed when quinazoline is boiled with hydrochloric acid.

2.[C]. 1. Aromatic substitution of quinazoline:

The two known nucleophilic substitution reactions of quinazoline namely with sodamide and hydrazine, presumably proceed via the intermediate addition products and gave 4-amino and 4-hydrazine quinazoline. Nitration is the only known electrophilic substitution reaction of quinazoline. Theoretical considerations show that the expected order of reactivity is at positions 8 > 6 > 5 > 7 > 4 > 2. Quinazoline gives 6-nitroquinazoline with fuming nitric acid in concentrated H₂SO₄. No oxidation of the heterocyclic ring can occur under these conditions because the hydrated cation is not present.

2.[C]. 2. N-Alkylation of quinazoline:

Alkylation of quinazoline takes place on N- 3-methyl-3-ethyl-3-alkyl and 3-benzyl quinazolinium salts readily take up a molecule of alcohol to form the corresponding 4-alkoxy-3-alkyl-3,4-dihydro quinazolinium salts. These salts yield the pseudo bases, 3-alkyl-3, 4-dihydro-4-hydroxy quinazolines on treatment with strong alkali.
2.[C]. 3. Addition reactions of quinazoline:

Quinazoline is very reactive towards anionid reagents which attack position 4. Sodium bisulphate, hydrogen cyanide, acetophene, acetone, 2- butanone and cyclohexanone add across the 3,4-double bond of quinazoline. Methyl, ethyl, isopropyl, benzyl, t-buty1 and phenyl magnesium halides and phenyl lithium also add across the 3, 4-double bond to give the corresponding 4-substituted 3, 4-dihydroquinazolines.

2.[D]. DIFFERENT METHODS FOR THE SYNTHESIS OF QUINAZOLINE:

2.[D]. 1. Niementowski’s synthesis:

Nientowskis’s found that substituted anthranilic acid when reacted with formamide at 125 - 130°C for 4 hours gave 86% yield of oxoquinazoline

\[
\begin{array}{c}
\text{COOH} \\
\text{NH}_2 \\
\text{R} \\
\end{array}
\quad \xrightarrow{R'\text{CONH}_2} 
\begin{array}{c}
\text{O} \\
\text{NH} \\
\text{R} \\
\end{array}
\]

Where, R= H, OH, CH3

2.[D]. 2. Grimmel, Guinther and Morgan’s synthesis:

3 moles of 2- substituted acetyl amino benzoic acid, when heated with 3 moles of an amine together with one mole of phosphorous trichloride in toluene for two hours, gave high yields of 2,3-disubstituted 3,4-dihydro-4-oxoquinazolines

\[
\begin{array}{c}
\text{COOH} \\
\text{NH} \text{COR} \\
\end{array}
\quad + \quad 3\text{R'NH}_2 \quad + \quad \text{PCl}_3 
\quad \rightarrow 
\begin{array}{c}
\text{O} \\
\text{R'} \\
\text{R''} \\
\end{array}
\]

Where R, R’= -CH₃, -C₂H₅

2.[D]. 3. Sen and Ray’s synthesis:

Boiling a solution of normal (or) isobutyrylanilides with urethane and phosphorous pentoxide in xylene gave 2-propyl and 2-isopropyl-3,4-dihydro-4-
oxoquinazolines\textsuperscript{26}.

\begin{align*}
\text{R} & \quad \xrightarrow{\text{H}_2\text{NCOOC}_2\text{H}_5\text{P}_2\text{O}_5, \text{Xylene, 3 - 5 hrs}} \\
\text{NHCOR'} & \quad \text{O} \\
\text{N} & \quad \text{NH} \\
\text{R} & \quad \text{O}
\end{align*}

(R = Me, OMe, OEt; R' = Me, Et, Pr, Iso-Pro, Ph)

\textbf{2.[D]. 4. From anthranilic acid and urea:}

The fusion of anthranilic acid with urea to give 1, 2, 3, 4-tetrahydro-2, 4-dioxyquinazoline was first described by Griess\textsuperscript{26}.

\begin{align*}
\text{R} & \quad \xrightarrow{\text{Urea}} \\
\text{NH}_2 & \quad \text{O} \\
\text{N} & \quad \text{NH} \\
\text{R} & \quad \text{O}
\end{align*}

\textbf{2.[D]. 5. From 2-ethoxycarbonylamino benzoic esters (or) amides:}

When 2-ethoxycarbonylamino benzamide and its 4-methyl derivatives are heated above their melting points, they lose water and from 1, 2, 3, 4-tetrahydro-2,4-Dioxyquinazoline\textsuperscript{26}.
This chapter described the experimental method of synthesis of 2-Chloromethyl-4-methyl-quinazoline. Reduction of 2-Nitro acetophenone by using tin(II)chloride dihydrate in presence of concentrated hydrochloric acid in Methanol to give 2-Amino acetophenone. Then 2-Amino acetophenone react with HCl gas in Dioxane to give hydrochloride salt of 2-Amino acetophenone. It reacted with chloro acetonitrile to give finally 2-chloromethyl-4-methyl-quinazoline.

![2-Chloromethyl-4-methyl-quinazoline](image)

Various quinazoline derivatives are reported to possess promising biological activities including antibacterial, antifungal, antiviral and more over some hydrazides, hyrazones\(^2\) and Mannich bases are well known as antibacterial, antifungal and antiviral agents. In view of these findings, it was considered worthwhile to synthesize some derivatives quinazoline.

2. [E]. MATERIALS:

All the chemicals used in the present study were of A.R.grade methanol, tin (II)chloride dihydrate, Concentrated HCl, Sodium hydroxide, Toluene, Cyclohexane, 2-Methyl Cyclohexane, Dioxane, Sodium sulphate, Chloro acetonitrile [SD’s fine chemical Ltd., Mumbai] were used without further purification.

2. [F]. EXPERIMENTAL:

2-Chloromethyl-4-methyl-quinazoline prepared and confirmed by NMR, FT-IR, Mass spectral studies.

All novel compounds synthesized in the present study are characterized by Colour, MP, Elemental analysis, FT-IR spectral, Mass spectra, \(^1\)H-NMR and \(^13\)C-NMR spectral studies.
• Melting points were taken in one side open capillaries on a Melting point apparatus having model number VMP-D of a make VEEGO.
• The Mass spectra of all 48 compounds were recorded on the instrument named LCMS-2010A of make Shimadzu (Oxygen Healthcare Ltd, Ahmadabad, India).
• Carbon, hydrogen and nitrogen were estimated on a Thermo fisher (Thermo electron corporation Limited), Flash Elemental Analyzer-1112.
• The $^1$H NMR and $^{13}$C NMR spectra in deuterated Chloroform-DMSO-MeOD of all novel compounds were recorded on a AVANCE II 300 of make BRUKER spectrophotometer using TMS [(CH$_3$)$_4$Si] as internal standard.
• This series compounds $^1$H NMR, $^{13}$C NMR was recorded on AVANCE II 300 of make BRUKER, IR, Mass are recorded at (Zydus Research Centre, Ahmedabad, India).
• The infrared spectra of the twenty four studied in the present work were recorded on the model FT-IR-PRESTIGE of Shimadzu in KBr (Zydus Research Center, Ahmedabad, India).
2. PREPARATION OF 2-CHLOROMETHYL-4-METHYL-QUINAZOLINE:

The 2-Chloromethyl-4-methyl-quinazoline used in the present study was prepared as follows:

Reaction Scheme

![Reaction Scheme Image]

2-Chloromethyl-4-methyl-quinazoline

Fig. 1

2-Chloromethyl-4-methyl-quinazoline
(I) Preparation of 1-(2-Amino-phenyl)-ethanone:

Process:

1-(2-Nitro-phenyl)-ethanone (0.1 mol) were take in Methanol (50ml) and add tin (II) chloride dihydarte (4 mol) and stir for 30 min at 25-35°C temperature. Then drop wise addition of Con.Hcl (10ml) in reaction mass within 20-30min at 25 to 35°C temperature. And heat the reaction mass up to 55-65°C temperature for 6-7 hrs. Stirring continue up to clear and then cool it to 25-35°C and stir for overnight on mechanical stirrer. Then check TLC, it complies. Poured the reaction mass into crushed ice and stirred it for 30 min, then add 40% sodium hydroxide (30ml) at 25-35°C temperature. Check pH, it’s around 11-12. Then add Dichloromethane (50ml) and stir for 15 min. and separate lower organic layer and charge aqueous layer and given another dichloromethane (25ml) and then separate lower organic layer, combine both organic layer and give a water wash (25ml) then separate organic layer. Dried organic layer on sodium sulphate (5gm). Then distill out solvent completely then get oily residue. Add cyclohexana (10ml) and stir for 2-3 hrs at 20-25°C temperature. Product precipitate, then filter the product and was with cyclohexana (5ml). Dry the product at 50-60°C.Recristalised in Cyclohexane and Isopropyl alcohol to get compound (I).

(II) Preparation of 1-(2-Amino-phenyl)-ethanone Hydrochloride:

Process:

Take 1-(2-Amino-phenyl)-ethanone (0.3 mol) in Dioxane (50 ml) and cool down the reaction mass up to 5-10°C. Then HCl gas purging in reaction mass for 3-4 hrs below 10°C temperature (0.5 mol). It was used for further step without any isolation and purification.

(III) Preparation of 2-chloromethyl-4-methyl-quinazoline:

Process:

Take 1-(2-Amino-phenyl)-ethanone Hydrochloride (0.3 mol) and cool down the reaction mass up to 0-5°C temperature. Then slowly drop wise addition of Chloro acetonitrile (0.35 mol) then stir for 3-4 hrs. Then check TLC. It complies. Then added chill water (50 ml) in reaction mass and added sodium hydroxide solution to bring pH 11-12. And stir for 30 min. then added Con HCl (15 ml) in reaction mass to bring pH 4-5 and stir for 30 min. then added Toluene (25 ml) and stir for 15 min and separate
the toluene and dried over sodium sulphate. Then distil out completely get oily residue add Cyclohexane and heat the reaction mass up to 55-60°C temperature. then remove heating and cool down the reaction mass up to 10-15°C and stir for 30min and filter the solid and wash with chill Cyclohexane (5 ml). Dry the product at 50-60°C. Purifying in 2-Methyl Cyclohexane to obtain pure crystalline 2-chloromethyl-4-methyl-quinazoline compound (III).

2.[H].INTERPRETATION OF 2-CHLOROMETHYL-4-METHYL-QUINAZOLINE BY SPECTROSCOPY:

(1) 2-chloromethyl-4-methyl-quinazoline:

- **Structural Formula**

```
N
N
CH3
Cl
```

<table>
<thead>
<tr>
<th>Observed</th>
<th>Reported</th>
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<tbody>
<tr>
<td>Color</td>
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<tr>
<td>Molecular Formula</td>
<td>C10H9ClN2</td>
</tr>
<tr>
<td>Molecular Weight</td>
<td>192.64</td>
</tr>
<tr>
<td>Melting Point</td>
<td>62-65°C</td>
</tr>
<tr>
<td>Yield</td>
<td>52%</td>
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<td>Elemental Analysis</td>
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<table>
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<th>Element</th>
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<th>%N</th>
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<tr>
<td>Calculated</td>
<td>62.35</td>
<td>4.71</td>
<td>14.54</td>
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<tr>
<td>Found</td>
<td>61.05</td>
<td>4.01</td>
<td>14.27</td>
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• **IR Spectra** : Fig. 1.1

Interpretation from the recorded IR spectrum, the wave numbers of corresponding groups are shown in table given below.

<table>
<thead>
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<th>Wave number (cm(^{-1}))</th>
<th>Assignment</th>
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<tbody>
<tr>
<td>3021.96</td>
<td>Aromatic –CH stretching</td>
</tr>
<tr>
<td>2916.98</td>
<td>Aliphatic –CH stretching</td>
</tr>
<tr>
<td>1575.86</td>
<td>Aromatic –C=C– stretching</td>
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<tr>
<td>1560.52</td>
<td>-C=C- ring skeleton</td>
</tr>
<tr>
<td>1396.65</td>
<td>–C=C– ring skeleton vibration</td>
</tr>
<tr>
<td>757.95</td>
<td>C-Cl stretching</td>
</tr>
</tbody>
</table>

• **Mass Spectral data** : Fig. 1.2

The Positive ion mass spectral analysis of A1 observes at 192.9 m/z (M\(^+\)), Confirms the theoretical molecular weight i.e. 192.64

• **\(^1\)H NMR Spectra** : Fig. 1.3

**Interpretation:** From the recorded \(^1\)H-NMR spectrum, chemical shifts and the multiplicity of the corresponding protons are shown in table given below.

<table>
<thead>
<tr>
<th>Chemical Shift  (δ value in ppm)</th>
<th>Multiplicity</th>
<th>Assigned Proton</th>
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<tbody>
<tr>
<td>2.984</td>
<td>Singlet</td>
<td>(5)</td>
</tr>
<tr>
<td>4.859</td>
<td>Singlet</td>
<td>(6)</td>
</tr>
<tr>
<td>7.636-7.687</td>
<td>Multiplet</td>
<td>(3)</td>
</tr>
<tr>
<td>7.881-7.932</td>
<td>Multiplet</td>
<td>(2)</td>
</tr>
<tr>
<td>8.019-8.047</td>
<td>Doublet</td>
<td>(4)</td>
</tr>
<tr>
<td>8.098-8.125</td>
<td>Doublet</td>
<td>(1)</td>
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• **$^{13}$C NMR Spectra**: Fig. 1.4

**Interpretation**: From the recorded $^{13}$C-NMR spectrum, chemical shifts and the multiplicity of the corresponding protons are shown in table given below.

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<th>Chemical Shift (δ value in ppm)</th>
<th>Nature of Carbon</th>
<th>Assigned Carbon</th>
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<tr>
<td>21.98</td>
<td>Secondary</td>
<td>(8)</td>
</tr>
<tr>
<td>48.23</td>
<td>Secondary</td>
<td>(10)</td>
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<td>122.81</td>
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<td>128.55</td>
<td>Tertiary</td>
<td>(3)</td>
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<tr>
<td>128.63</td>
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<td>(1)</td>
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<td>134.92</td>
<td>Tertiary</td>
<td>(2)</td>
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<td>(6)</td>
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<td>161.09</td>
<td>Quaternary</td>
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<tr>
<td>170.11</td>
<td>Quaternary</td>
<td>(9)</td>
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</tbody>
</table>
Fig. 1.2
Fig. 1.3
Fig. 1.4
2. Preparation of substituted 4-Methyl quinazoline derivatives (General Reaction Scheme)

\[
\begin{align*}
\text{2-Chloromethyl-4-methyl-quinazoline} & \quad \text{K}_2\text{CO}_3 \\
\text{C}_{10}\text{H}_{9}\text{ClN}_2 & \quad \text{DMF} \\
\text{Mol. Wt.: 192.64} & \\
\end{align*}
\]

R= substituted Piperazine, Piperadine, Morpholine, substituted Morpholine, Phenyl amine, Pyrrolidine, substituted pyrrolidine, aliphatic amine, aromatic amine, substituted pyrrole

(1) Preparation of 4-(4-Methyl-quinazolin-2-ylmethyl)-piperazine-1-carboxylic acid tert-butyl ester (DJP/D102):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline(0.01 mol) and Piperazine-1-carboxilic acid tert-butyl ester (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D102) and dry the material at 50-55°C.

Colour: Pale Yellow, M. F: C_{19}H_{26}N_{4}O_{2}, M.W: 342.44, MP: 184-190°C, Yield: 82%

(2) Preparation of 1-(4-Methyl-quinazolin-2-ylmethyl)-piperidine-4-carboxylic acid ethyl ester (DJP/D103).

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline(0.01 mol) and Piperidine-4-carboxylic acid ethyl ester (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction
mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D103) and dry the material at 50-55°C.

Colour: Yellow, M. F: C\textsubscript{18}H\textsubscript{23}N\textsubscript{3}O\textsubscript{2}, M.W: 313.39, MP: 150-154°C, Yield: 78%

(3) Preparation of 2-(4-Ethyl-piperazin-1-ylmethyl)-4-methyl-quinazoline (DJP/D104).

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and N-Ethyl Piperazine (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D104) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C\textsubscript{16}H\textsubscript{22}N\textsubscript{4}, M.W: 270.37, MP: 125-128°C, Yield: 75%

(4) Preparation of Methyl-quinazolin-2-ylmethyl)-piperidine-4-carboxylic acid hydrazide (DJP/D105).

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and Piperadine-4-carboxylic acid hydrazide (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D105) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C\textsubscript{16}H\textsubscript{21}N\textsubscript{5}O, M.W: 299.37, MP: 175-178°C, Yield: 80%
(5) Preparation of 4-Methyl-2-morpholin-4-ylmethyl-quinazoline (DJP/D106).

Process:
A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and Morpholine (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5 hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40 ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. Then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D106) and dry the material at 50-55°C.

Colour: Yellow. F.W: C\textsubscript{14}H\textsubscript{17}N\textsubscript{3}O, M.W: 243.30, MP: 110-114°C, Yield: 90%

(6) Preparation of Methyl-(4-methyl-quinazolin-2-ylmethyl)-phenyl-amine (DJP/D107)

Process:
A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and Methyl-phenyl amine (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5 hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40 ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. Then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D107) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C\textsubscript{17}H\textsubscript{17}N\textsubscript{3}, M.W: 263.34, MP: 124-128°C, Yield: 68%

(7) Preparation of 1-(4-Methyl-quinazolin-2-ylmethyl)-pyrrolidine-2-carboxylic acid amide (DJP/D108)

Process:
A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and Pyrrolidine-2-carboxylic amide (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5 hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40 ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. Then separate the lower organic layer. This
organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D108) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{15}H_{18}F_{2}N_{4}O, M.W: 270.33, MP: 136-139°C, Yield: 68%

(8) Preparation of 4-Methyl-2-(4-pyrimidin-2-yl-piperazin-1-ylmethyl) quinazoline (DJP/D109):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and 2-Piperazine-1-yl-pyrimidine (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D109) and dry the material at 50-55°C.


(9) Preparation of (2-Methyl-4-nitro-phenyl)-(4-methyl-quinazolin-2-ylmethyl)amine (DJP/D110):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and 2-Methyl-4-nitro-phenylamine (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D110) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{17}H_{16}N_{4}O_{2}, M.W: 308.33, MP: 113-136°C, Yield: 47%

(10) Preparation of [(4-Methyl-quinazolin-2-ylmethyl)-amino]-acetic acid ethyl ester (DJP/D111):

Process:
A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and Amino-acetic acid ethyl ester (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D111) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{14}H_{17}N_{3}O_{2}, M.W: 259.3, MP: 89-92°C, Yield: 77%

(11) Preparation of 4-[(4-Methyl-quinazolin-2-ylmethyl)-amino]-benzoic acid ethyl ester (DJP/D112):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and 4-Amino-benzoic acid ethyl eater (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D111) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{19}H_{19}N_{3}O_{2}, M.W: 321.37, MP: 122-125°C, Yield: 53%

(12) Preparation of 4-Methyl-2-[1,2,4]triazol-1-ylmethyl-quinazoline (DJP/D113):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and 1H-[1,2,4]Triazole (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D111) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{21}H_{11}N_{3}, M.W: 225.25, MP: 111-114°C, Yield: 39%
(13) Preparation of 4-Methyl-2-pyrrolidin-1-ylmethyl-quinazoline (DJP/D114):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and Pyrrolidine (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5 hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40 ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D114) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{14}H_{17}N_3, M.W: 227.3, MP: 68-71°C, Yield: 74%

(14) Preparation of 4-Methyl-2-(3-methyl-piperidin-1-ylmethyl)-quinazoline (DJP/D115):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and 3-Methyl-Piperidine (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5 hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40 ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D115) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{16}H_{21}N_3, M.W: 255.36, MP: 112-115°C, Yield: 87%

(15) Preparation of 1-(4-Methyl-quinazolin-2-ylmethyl)-1H-pyrrole-2-carbaldehyde (DJP/D116):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and Pyrrolidine-2-carboxylic acid amide (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5 hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40 ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min then separate the lower organic layer. This
organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D116) and dry the material at 50-55°C.

Colour: Yellow. F.W: C_{15}H_{13}N_{3}O, M.W: 251.28, MP: 156-160°C, Yield: 71%

(16) Preparation of 4-Methyl-2-piperidin-1-ylmethyl-quinazoline (DJP/D117):
Process:
A mixture of 2-Chloromethyl-4-methyl-quinazoline(0.01 mol) and Piperidine (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D117) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{15}H_{19}N_{3}, M.W: 241.33, MP: 148-152°C, Yield: 89%

(17) Preparation of 2-(2,6-Dimethylmorpholin-4-ylmethyl)-4-methyl-quinazoline (DJP/D118):
Process:
A mixture of 2-Chloromethyl-4-methyl-quinazoline(0.01 mol) and 3,5-dimethyl Morpholine (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D118) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{16}H_{21}N_{3}O, M.W: 271.36, MP: 112-115°C, Yield: 72%

(18) Preparation of 4-[(4-Methyl-quinazolin-2-ylmethyl)-amino]-2-trifluoro methyl-benzonitrile (DJP/D119):
Process:
A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and 4-Amino-2-trifluoromethyl-benzonitrile (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D119) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{18}H_{13}N_{4}F_{3}, M.W: 342.32, MP: 122-125°C, Yield: 57%

(19) Preparation of 2-(4-Methyl-quinazolin-2-ylmethyl)-2-aza-bicyclo[3.1.0]hexane-3-carboxylic acid amide (DJP/D120):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and 2-Aza-bicyclo[3.1.0]hexane-3-carboxylic acid (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D120) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{16}H_{18}N_{4}O, M.W: 282.34, MP: 155-158°C, Yield: 62%

(20) Preparation of Cyclopropyl-(4-methyl-quinazolin-2-ylmethyl)-amine (DJP/D121):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and Cyclopropyl amine (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over
sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D121) and dry the material at 50-55°C.


(21) Preparation of 3-[(4-Methyl-quinazolin-2-ylmethyl)-amino]-adamantan-1-ol (DJP/D122):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and Cyclopropyl amine (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D122) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{20}H_{25}N_{3}O, M.W: 323.43, MP: 178-181°C, Yield: 46%

(22) Preparation of 4-Methyl-2-(4-methyl-piperidin-1-ylmethyl)-quinazoline (DJP/D123):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and 4-Methyl Piperadine (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D123) and dry the material at 50-55°C.

Colour: Pale Yellow. F.W: C_{16}H_{21}N_{3}, M.W: 255.36, MP: 102-1.4°C, Yield: 63%

(23) Preparation of 1-(4-Methyl-quinazolin-2-ylmethyl)pyrrolidine-2-carbonitrile (DJP/D124):

Process:
Chapter 2

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and Pyrrolidine-2-Carbonitrile (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D124) and dry the material at 50-550C.


(24) Preparation of 1-(4-Methyl-quinazolin-2-ylmethyl)-piperadine-4-carboxylic acid methyl ester (DJP/D125):

Process:

A mixture of 2-Chloromethyl-4-methyl-quinazoline (0.01 mol) and Piperidine-4-carboxylic acid methyl ester (0.01 mol) in 30 ml of N,N-Dimethyl formamide and add potassium carbonate (0.02 mol) was heated 90-95°C temperature and stir the reaction mass for 4-5hrs. Then cool the reaction mass up to 25-30°C temperature. Then add water (40ml) and stir the reaction mass for 20 min then add Dichloromethane and stir for 15 min. then separate the lower organic layer. This organic layer dried over sodium sulphate. Then distil out solvent completely. Get oily residue. This residue crystallized in ether to get (DJP/D125) and dry the material at 50-550C.

2.[J]. Table: 1 Physical property of synthesized 4-Methyl quinazoline derivatives (DJP/D102 to DJP/D125):

![Chemical structure diagram]

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<th>R</th>
<th>Colour</th>
<th>M.F</th>
<th>M.W</th>
<th>M.P</th>
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<td>C&lt;sub&gt;15&lt;/sub&gt;H&lt;sub&gt;26&lt;/sub&gt;N&lt;sub&gt;4&lt;/sub&gt;O&lt;sub&gt;2&lt;/sub&gt;</td>
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REFERENCES:


27. Hong-Ze Li, Hai-Yun He 1, Yuan-Yuan Han, Xin Gu , Lin He , Qing-Rong Qi General Synthetic Procedure for 2-chloromethyl-4(3H)-quinazolinone Derivatives, Molecules 2010, 15, 9473-9485.