5. 1 Introduction to Benzimidazolo Quinazolinone

Nitrogen containing heterocycles have always played a major role in the pharmaceutical and agrochemical industries because of their potent physiological properties, which have resulted in numerous applications [1]. Quinazolinone and their derivatives are building block for around more than 150 naturally occurring alkaloid isolated from plant kingdom, microorganisms and animals. There has been enormous increase in the interest among biologist and chemist in their synthesis and bioactivity of quinazolinone derivatives. Compound having 4(3H)-quinazolinone ring system is known to possess antitumor, antimicrobial and anticoagulant activities [2-6]. Quinazolinone have been frequently used in medicines [7-9].

Diseases of the arterial tree cause more premature deaths than all other diseases such as cancer. Among the major risk factors for arterial diseases, high blood pressure is the most important one [10]. Triazolo quinazolinone derivatives are an important class of natural products and exhibit a wide range of spectrum of biological activities, such as antihypertensive [11], antihistaminic [12], analgesic, anti-inflammatory [13], anticancer [14], and anti HIV activities [15].

5. 2 Recent Literature Survey

Ravinder Goud et al. [16] proposed the mechanism for formation of benzimidazolo quinazolinone as follow. Initially C-O bond of benzaldehyde is polarized by iodine to make carbonyl carbon more electron deficient. In the next step, nucleophilic attack by active methylene group of dimeredone to produce intermediate A. This upon dehydration produced compound B. It was then reacted with 2-aminobenzimidazole to produce compound C. In the last step the nucleophilic addition initiated by nitrogen led to intramolecular cyclization to form final product D. (Scheme 5.1).
Several different strategies for the synthesis of substituted triazolo/benzimidazolo quinazolinones are known. The most common protocol is the reaction of substituted aldehydes with 3-amino-1,2,4-triazole and 2-aminobenzimidazole as the amine sources and dimedone in the presence of acid or basic catalysts. There are literature reports about the synthesis of 6,6-dimethyl-9-phenyl-5,6,7,9-tetrahydro-[1,2,4]triazolo[5,1-b]quinazolin-8(4H)-one and 3,3-dimethyl-12-phenyl-1,2,3,4,5,12-hexatrahydro-[4,5]imidazolo[2,1-b]quinazolin-1-one derivatives by treatment of 3-amino-1,2,4-triazolo or 2-aminobenzimidazole with aldehydes and dimedone [17-21]. The cyclocondensation was realized by heating the starting materials in DMF under reflux conditions or in the presence of sulfamic acid using acetonitrile as solvent under reflux conditions [19] or in the presence of ionic liquids [20] or in the presence of heteropolyacids [21]. However, many of these methods are associated with various drawbacks such as use of metal catalysts, harsh reaction
conditions, tedious experimental procedures, unsatisfactory yields, long reaction times, and usage of expensive and moisture sensitive catalysts. Hence, there is a need to develop a rapid, efficient, and environmentally benign synthetic protocol for the synthesis of triazolo/benzimidazolo quinazolinone derivatives.

Majid et al. [19] reported three-component, one-pot synthesis of the [1,2,4]triazolo/benzimidazolo quinazolinones by condensation of 2-amino benzimidazole or 3-amino-1,2,4-triazole as amine sources with dimedone and different aldehydes in the presence of sulfamic acid as a reusable, green catalyst in acetonitrile and under heating conditions.

Ravinder Goud et al. [16] developed an efficient and facile method for the synthesis of 1,2,4-triazolo[5,1-b]quinazolin-8(4H)-one derivatives and 3,3-dimethyl-12-phenyl-3,4,5,12-tetrahydrobenzo [4,5] imidazo[2,1-b]quinazolin 1(2H)-one derivatives by treatment of the corresponding aldehydes with 3-amino-1,2,4-triazoles or 2-amino benzimidazole and dimedone by using iodine in the presence of acetonitrile at reflux conditions within 10–15 min. Lipson et al.[17] have reported synthesis of 9-aryl-6,6-dimethyl-5,6,7,9-tetrahydro-1,2,4-triazolo- [5,1 b] quinazolin-8(4H) ones by condensation reaction of 3-amino-1,2,4-triazole with p-substituted benzaldehydes and dimedone under a variety of conditions.

In the mainstream of current interest, multicomponent processes have recently gained considerable economic and ecological interest as they address fundamental principles of synthetic efficiency and reaction design. Multicomponent reactions (MCRs) have been proven to be a very graceful and rapid way to access complex structures in a single synthetic operation from simple building blocks and show high selectivity [22-24]. Multicomponent reactions (MCRs) are one-pot processes in which three or more components come together to form a product containing substantial elements of all the reactants [25, 26]. They lead to a reduction in the amount of waste generated by a synthetic route because of the minimization of by-products, reaction and extraction solvents, and silica gel for chromatographic purification of intermediate compounds. They provide an inherently more efficient approach to chemical synthesis than conventional bimolecular reactions, and considerable effort is focused on the development of new MCRs and the full exploitation of
already known MCRs [27]. In this context, it was planned to synthesize benzimidazolo quinazolinone derivatives using the prepared catalyst ENPFSA under conventional and microwave irradiation methods in a multicomponent reaction of thirteen different aromatic aldehydes, 2-aminobenzimidazole and dimedone/cyclohexane-1, 3-dione using dimethyl formamide (DMF) as the solvent.

5.3 Objectives

➢ To find out new synthetic pathway for well-known multicomponent condensation reactions of aromatic aldehydes, 2-aminobenzimidazole and dimedone/cyclohexane-1, 3-dione by employing polymer supported sulphanilic acid (ENPFSA) as the heterogeneous catalyst under different energy source such as conventional method and microwave irradiation.

➢ To optimize the conditions under conventional method and microwave irradiation to get maximum yield in shorter duration.

➢ To check the scope of the novel protocol for synthesizing a good library of benzimidazolo quinazolinone derivatives.

➢ To characterize the synthesized by $^1$H NMR, $^{13}$C NMR, APT, IR, MASS spectroscopic techniques.

5.4 Result and Discussion

5.4.1 Scheme

The synthesis of 4a (Scheme 5.2) was carried out by one pot multicomponent condensation reaction between aromatic aldehyde 1a-m, 2-aminobenzimidazole 2 and cyclohexane-1,3-diketone 3a/n by using 5% w/w of ENPFSA with respect to 2-aminobenzimidazole in DMF as the solvent by using two different energy source such as conventional and microwave irradiation.
5.4.2 Optimization

The cyclocondensation reaction between benzaldehyde 1a (0.01 mole), 2-aminobenzimidazole 2 (0.01) and dimerdone 3a (0.01 mole) in DMF (10 mL) in a under reflux to afford 3,3-dimethyl-12-phenyl-1,2,3,4,5,12-hexahydro [4,5] imidazo [2,1-d] quinazolin-1-one 6 (Scheme 5.2) was chosen as the model reaction for optimization.

The amount of ENPFSA was used in the ratio of w/w with respect to the 2-aminobenzimidazole. The optimization was carried out with respect to the amount of catalyst, duration of reaction and the corresponding yield. The progress of the reaction was continuously monitored by TLC using aluminum sheets precoated with silica gel 60 F\textsubscript{254} (Merck) under ethyl acetate: n-hexane in the ratio 50:50. The characteristic data are shown in Table 5.1. First reaction was carried out reaction in the absence of catalyst (Table 5.1, entry 1), it was found that the reaction did not proceed. Next, the amount of catalyst was varied with respect to 2-amino benzimidazole (Table 5.1, entries 2-8). It was found that only 75% and 80% yield were obtained by taking 1% and 7% amount of catalyst ENPFSA (Table 5.1, entry 2 & 8) respectively with reaction time 60 minutes. The best result (88% yield of product 6) was obtained in reaction time 40 minutes by using 5% amount of catalyst with respect to 2-amino benzimidazole (Table 5.1, entry 5).

<table>
<thead>
<tr>
<th>Entry</th>
<th>Amount of catalyst (%)</th>
<th>Reaction time (min)\textsuperscript{b}</th>
<th>Yield (%)\textsuperscript{c}</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>60</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>50</td>
<td>70</td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td>45</td>
<td>75</td>
</tr>
<tr>
<td>4</td>
<td>3</td>
<td>45</td>
<td>80</td>
</tr>
<tr>
<td>5</td>
<td>4</td>
<td>40</td>
<td>85</td>
</tr>
<tr>
<td>6</td>
<td>5</td>
<td>40</td>
<td>88</td>
</tr>
<tr>
<td>7</td>
<td>6</td>
<td>40</td>
<td>85</td>
</tr>
<tr>
<td>8</td>
<td>7</td>
<td>40</td>
<td>80</td>
</tr>
</tbody>
</table>

\textsuperscript{a} DMF is used as solvent; \textsuperscript{b} Reaction was monitored by TLC; \textsuperscript{c} Isolated yields.

The results revealed that only 5% of ENPFSA on the weight basis of 2-amino benzimidazole is enough to lead the reaction to produce benzimidazolo
quinazolinones in as high as 88% yield. Thus 5% ENPFSA was selected as the amount of catalyst in the preparation of series of substituted benzimidazolo quinazolinones by changing the aldehydes and cyclic 1, 3- diketone.

Our next target of the present work was to synthesize all the benzimidazolo quinazolinones by taking the assistance of microwave irradiation (MWI) technique using DMF as the solvent. These series of experiments were also optimized with respect to power levels. The amount of ENPFSA used was 5% w/w with respect to 2-amino benzimidazole. The reactions as presented in Scheme 5.2 were carried out under microwave irradiation. The MWI reactions were carried out in the Scientific Microwave system CATA-R CATALYST SYSTEM. The optimization of the power levels was checked with reference to duration of reaction, yield improvement. The characteristic data are given in Table 5.2.

Table 5.2 Data representing the optimization for synthesis of benzimidazolo quinazolinones by the assistance of MWI technique.\(^{a,b}\)

<table>
<thead>
<tr>
<th>Power Levels in Watt</th>
<th>Reaction Time (min)(^{c})</th>
<th>% Isolated Yield(^{d})</th>
</tr>
</thead>
<tbody>
<tr>
<td>140</td>
<td>4</td>
<td>75</td>
</tr>
<tr>
<td>210</td>
<td>3.5</td>
<td>85</td>
</tr>
<tr>
<td><strong>240</strong></td>
<td><strong>3</strong></td>
<td><strong>90</strong></td>
</tr>
<tr>
<td>280</td>
<td>3</td>
<td>80</td>
</tr>
<tr>
<td>350</td>
<td>3</td>
<td>80</td>
</tr>
</tbody>
</table>

\(^{a}\)DMF is used as solvent; \(^{b}\)5% w/w amount of catalyst with respect to 2-amino benzimidazole.; \(^{c}\)Reaction was monitored by TLC.; \(^{d}\)Isolated yields.

From the above experimental data, it becomes clear that more efficient results were obtained at 240 W power level of the MW instrument. At this power level benzimidazolo quinazolinone were obtained in 90% yield with very good purity in 3 min.

5.4.3 Characteristics Data Showing the Synthesis of Benzimidazolo Quinazolinones

By using these optimized conditions, various benzimidazolo quinazolinone derivatives were synthesized in shorter time as well as in high yields. It was observed that the aldehydes bearing electron withdrawing group undergoes conversion in shorter reaction time with high yield (compounds 5d, 5e, 5f, 5m, 5p, 5q, & 5r) as compared to aldehydes bearing electron donating group (compounds 5h, 5i, 5j, 5k, 5l, 5t, 5u, & 5v). Negligible change is observed in
reaction time as well as yield of product upon replacing dimedone by cyclohexane-1, 3- dione.

Table 5.3 The characteristic data showing the synthesis of benzimidazolo quinazolinones\textsuperscript{a,b}.

<table>
<thead>
<tr>
<th>Code</th>
<th>R</th>
<th>R\textsubscript{1}</th>
<th>Microwave Irradiation 240 W</th>
<th>Thermal Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Reaction Time\textsuperscript{c} (m)</td>
<td>Yield\textsuperscript{d} (%)</td>
</tr>
<tr>
<td>5a</td>
<td>C\textsubscript{6}H\textsubscript{5} -</td>
<td>CH\textsubscript{3}</td>
<td>3</td>
<td>90</td>
</tr>
<tr>
<td>5b</td>
<td>4-CH\textsubscript{3}-C\textsubscript{6}H\textsubscript{4} -</td>
<td>CH\textsubscript{3}</td>
<td>3.5</td>
<td>85</td>
</tr>
<tr>
<td>5c</td>
<td>2-C\textsubscript{4}H\textsubscript{3}O -</td>
<td>CH\textsubscript{3}</td>
<td>3</td>
<td>90</td>
</tr>
<tr>
<td>5d</td>
<td>4-NO\textsubscript{2}-C\textsubscript{6}H\textsubscript{4} -</td>
<td>CH\textsubscript{3}</td>
<td>3</td>
<td>88</td>
</tr>
<tr>
<td>5e</td>
<td>3-NO\textsubscript{2}-C\textsubscript{6}H\textsubscript{4} -</td>
<td>CH\textsubscript{3}</td>
<td>3</td>
<td>90</td>
</tr>
<tr>
<td>5f</td>
<td>2-NO\textsubscript{2}-C\textsubscript{6}H\textsubscript{4} -</td>
<td>CH\textsubscript{3}</td>
<td>3</td>
<td>85</td>
</tr>
<tr>
<td>5g</td>
<td>4-Cl-C\textsubscript{6}H\textsubscript{4} -</td>
<td>CH\textsubscript{3}</td>
<td>3</td>
<td>90</td>
</tr>
<tr>
<td>5h</td>
<td>4-OH-3-OCH\textsubscript{3}-C\textsubscript{6}H\textsubscript{3} -</td>
<td>CH\textsubscript{3}</td>
<td>3.5</td>
<td>80</td>
</tr>
<tr>
<td>5i</td>
<td>4-OH-C\textsubscript{6}H\textsubscript{4} -</td>
<td>CH\textsubscript{3}</td>
<td>3.5</td>
<td>80</td>
</tr>
<tr>
<td>5j</td>
<td>4-OCH\textsubscript{3}-C\textsubscript{6}H\textsubscript{4} -</td>
<td>CH\textsubscript{3}</td>
<td>3.5</td>
<td>80</td>
</tr>
<tr>
<td>5k</td>
<td>2-OH-C\textsubscript{6}H\textsubscript{4} -</td>
<td>CH\textsubscript{3}</td>
<td>3.5</td>
<td>80</td>
</tr>
<tr>
<td>5l</td>
<td>3-OH-C\textsubscript{6}H\textsubscript{4} -</td>
<td>CH\textsubscript{3}</td>
<td>3.5</td>
<td>80</td>
</tr>
<tr>
<td>5m</td>
<td>2-COOH- C\textsubscript{6}H\textsubscript{4} -</td>
<td>CH\textsubscript{3}</td>
<td>3</td>
<td>85</td>
</tr>
<tr>
<td>5n</td>
<td>C\textsubscript{6}H\textsubscript{5} -</td>
<td>H</td>
<td>3</td>
<td>90</td>
</tr>
<tr>
<td>5o</td>
<td>4-CH\textsubscript{3}-C\textsubscript{6}H\textsubscript{4} -</td>
<td>H</td>
<td>3.5</td>
<td>80</td>
</tr>
<tr>
<td>5p</td>
<td>4-NO\textsubscript{2}-C\textsubscript{6}H\textsubscript{4} -</td>
<td>H</td>
<td>3</td>
<td>85</td>
</tr>
<tr>
<td>5q</td>
<td>3-NO\textsubscript{2}-C\textsubscript{6}H\textsubscript{4} -</td>
<td>H</td>
<td>3</td>
<td>90</td>
</tr>
<tr>
<td>5r</td>
<td>2-NO\textsubscript{2}-C\textsubscript{6}H\textsubscript{4} -</td>
<td>H</td>
<td>3</td>
<td>88</td>
</tr>
<tr>
<td>5s</td>
<td>4-Cl-C\textsubscript{6}H\textsubscript{4} -</td>
<td>H</td>
<td>3</td>
<td>85</td>
</tr>
<tr>
<td>5t</td>
<td>4-OH-C\textsubscript{6}H\textsubscript{4} -</td>
<td>H</td>
<td>3.5</td>
<td>80</td>
</tr>
<tr>
<td>5u</td>
<td>3-OH-C\textsubscript{6}H\textsubscript{4} -</td>
<td>H</td>
<td>3.5</td>
<td>80</td>
</tr>
<tr>
<td>5v</td>
<td>2-OH-C\textsubscript{6}H\textsubscript{4} -</td>
<td>H</td>
<td>3.5</td>
<td>80</td>
</tr>
</tbody>
</table>

\textsuperscript{a} Solvent DMF as a medium of reaction; \textsuperscript{b} 5\% w/w amount of catalyst with respect to 2-amino benzimidazole; \textsuperscript{c} Reaction was monitored by TLC; \textsuperscript{d} Isolated yields.

All the compounds were crystallized by using DMF- distilled water system and percentage yield was calculated after crystalization step. All the synthesized compounds are characterized by \textsuperscript{1}H NMR, \textsuperscript{13}C NMR, APT, IR spectroscopy and MASS spectrometry. All the data were in agreement with cited literature.
5.4.4 Mechanism

The formation of benzimidazolo quinazolinone derivatives is outlined in the following mechanism (Scheme 5.3). Aldehyde stabilized in the interlayer of ENPFSA via interaction with H⁺ by partial polarization of carbonyl group reacts readily with 1, 3- cyclic diketone to produced intermediate I which upon loss of water molecule produced II. Amino group of 2- amino benzimidazole make nucleophilic attack on carbonyl carbon of II followed by dehydration produced III. Cyclization of III produced target product IV.

5.5 Recyclability of Catalyst

Recyclability of the catalyst was studied by using the ENPFSA recovered from the previous batch. Reaction between benzaldehyde (0.01 mole), 2-aminobenzimidazole (0.01) and dimedone (0.01 mole) in DMF (10 mL) in a 250 round bottom flask was taken as the model reaction. The reaction proceeded smoothly yielding 88–80% of product at reflux temperature for five successive
turns (Table 5.4). This result indicates that the activity of catalyst was not getting much affected upon recycling at least for five times.

### Table 5.4 Recyclability of catalyst

<table>
<thead>
<tr>
<th>No. of cycle</th>
<th>Reaction time (min)</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>40</td>
<td>88</td>
</tr>
<tr>
<td>2</td>
<td>40</td>
<td>85</td>
</tr>
<tr>
<td>3</td>
<td>40</td>
<td>80</td>
</tr>
<tr>
<td>4</td>
<td>40</td>
<td>80</td>
</tr>
<tr>
<td>5</td>
<td>40</td>
<td>80</td>
</tr>
</tbody>
</table>

*aReaction was monitored by TLC; bIsolated yields.

#### 5.6 Conclusions

High yielding, one pot multicomponent reaction of aromatic aldehydes, 2-amino benzimidazole, dimedone/cyclohexane-1, 3-dione to afford benzimidazolo quinazolinones under microwave irradiation and thermal condition has been developed. The conditions are mild, and a wide range of functional groups can be tolerated. ENPFSA as catalyst offers advantages including simplicity of operation, easy workup procedure, product obtained in high yields with excellent purity, less time consuming and the recyclability of the catalyst.

#### 5.7 Experimental

Melting points were determined using μThermoCal10 (Analab scientific Pvt. Ltd.) melting point apparatus and are uncorrected. TLC was carried out using aluminum sheets precoated with silica gel 60 F$_{254}$.

#### 5.7.1 Chemicals and Reagents

All chemicals used were of laboratory reagent grade and used without further purification. Various aromatic aldehydes, 2-amino benzimidazole, dimedone and cyclohexane-1,3-dione were used as received from Merck, Mumbai, India. All the solvents were supplied by Sisco Chem. Pvt. Ltd., Mumbai, India.

#### 5.7.2 General Procedure for Synthesis of Quinoxalines

A 100 mL round-bottomed flask equipped with a mechanical stirrer and reflux condenser was charged with 2-amino benzimidazole (0.1 mmol), dimedone (0.1 mmol), aromatic aldehyde (0.1 mmol), DMF (5mL) and 5% w/w ENPFSA with respect to 2-amino benzimidazole. The reaction mixture was
stirred for the indicated time as required to complete. Upon completion of the reaction, monitored by thin-layer chromatography (TLC), the mixture was cooled to room temperature. The catalyst was separated by filtration. Recrystallization of product is done in DMF- distilled water solution.

The synthesized compounds had been characterized by FT-IR, $^1$H NMR, $^{13}$C NMR and MASS spectroscopy. A variety of substituted aromatic aldehydes were condensed with 2-amino benzimidazole and dimedone/cyclohexane-1, 3-dione. The recovered catalyst was washed with ethanol, chloroform, diethyl ether and subsequently dried at 80 °C to recycle in the subsequent reaction. Compounds 5a-v was synthesized by taking properly substituted aromatic aldehydes in the reaction mixture.

5.8 Characterization

Melting points were determined using µ ThermoCal10 (Analab scientific Pvt. Ltd.) melting point apparatus and are uncorrected. $^1$H NMR and $^{13}$C NMR spectra were recorded on a Bruker Avance 400 spectrometer operating at 400 MHz for $^1$H NMR, and 100 MHz for $^{13}$C NMR, as solutions in DMSO-$d_6$. Chemical shifts (δ) are in ppm and referenced to the residual protic solvent. FT-IR spectra were recorded on Shimadzu FT-IR 8401 spectrometer using KBr disc and are expressed in wave numbers (cm$^{-1}$). The mass spectra (LCMS) were recorded on Shimadzu LCMS-2010 spectrometer.

For compounds 5e & 5i of the series are taken as representative compounds and their spectra are included at the end of the section for perusal. $^1$H NMR spectrum for 5e & 5i are given in Figure 5.1 & Figure 5.6 respectively, $^{13}$C NMR spectrum for the same compounds are given in Figure 5.2 & Figure 5.7 respectively. APT spectrum of same compounds are given in Figure 5.3 & 5.8. The infrared spectrum is shown in Figure 5.4 for 5e and Figure 5.9 for 5i. The mass spectrum obtained for the same compounds are given in Figure 5.5 & Figure 5.10 respectively. The molecular structures and characterization of all the synthesized quinazolinones are given below.
<table>
<thead>
<tr>
<th>5a 3,3-Dimethyl-12-phenyl-1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular Formula</td>
</tr>
<tr>
<td>Molecular Weight (g·mol⁻¹)</td>
</tr>
<tr>
<td>Melting Point (°C)</td>
</tr>
</tbody>
</table>

**¹H NMR (400 MHz, DMSO, δ ppm):** 11.30 (s, 1H, NH), 6.62 (s, 1H, CH), 7.90-7.10 (m, 9H, Ar-H), 2.30-1.80 (m, 4H, CH₂), 1.06-0.96 (m, 6H, CH₃)  

**¹³C NMR (100 MHz, DMSO, δ ppm):** 193.4, 150.3, 140.3, 138.9, 136.7, 135.5, 128.5, 127.5, 125.9, 122.0, 118.9, 110.4, 110.2, 55.4, 50.2, 42.8, 32.2, 27.2  

**DEPT-135:** Up peaks: 193.4, 150.3, 140.3, 138.9, 136.7, 135.5, 110.2, 50.2, 42.8, 32.2; Down peaks: 128.5, 127.5, 125.9, 122.0, 118.9, 110.4, 55.4, 27.2  

**IR (KBr):** 3445, 2885, 1640, 1618, 1565  

**LC-MS:** 344.4  

% C, H, N Analysis: Calculated: C, 76.94; H, 6.16; N, 12.24  
Observed: C, 76.98; H, 6.19; N, 12.29

<table>
<thead>
<tr>
<th>5b 3,3-Dimethyl-12-(4-methyl phenyl)-1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular Formula</td>
</tr>
<tr>
<td>Molecular Weight (g·mol⁻¹)</td>
</tr>
<tr>
<td>Melting Point (°C)</td>
</tr>
</tbody>
</table>

**¹H NMR (400 MHz, DMSO, δ ppm):** 11.30 (s, 1H, NH), 6.62 (s, 1H, CH), 7.90-6.70 (m, 8H, Ar-H), 2.88-2.03 (m, 4H, CH₂), 2.34 (s, 3H, CH₃) 1.06-0.96 (m, 6H, CH₃)  

**¹³C NMR (100 MHz, DMSO, δ ppm):** 193.8, 152.6, 141.2, 138.9, 135.6, 134.8, 134.3, 128.5, 123.1, 118.4, 110.3, 110.2, 56.8, 50.2, 42.8, 32.6, 27.3, 21.2  

**DEPT-135:** Up peaks: 193.8, 152.6, 141.2, 138.9, 135.6, 134.8, 134.3, 110.3, 50.2, 42.8; Down peaks: 128.5, 123.1, 118.4, 110.2, 56.8, 32.6, 27.3, 21.2  

**IR (KBr):** 3435, 2898, 1646, 1615, 1590, 1567  

**LC-MS:** 358.5  

% C, H, N Analysis: Calculated: C, 77.28; H, 6.49; N, 11.76  
Observed: C, 77.31; H, 6.52; N, 11.79
5c 3,3-Dimethyl-12- (4-furyl)- 1,2,3,4,5,12-
hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

Molecular Formula  \( \text{C}_{20}\text{H}_{19}\text{N}_{3}\text{O}_{2} \)
Molecular Weight (g·mol\(^{-1}\)) 333.38
Melting Point (°C) > 300

\( ^{1}\text{H NMR} (400\text{ MHz, DMSO, } \delta \text{ ppm}): \) 11.30 (s, 1H, NH), 7.60 - 6.10 (m, 7H, Ar- H), 5.80 (s, 1H, CH), 2.88-2.03 (m, 4H, CH\(_{2}\)), 1.06-0.96 (m, 6H, CH\(_{3}\))

\( ^{13}\text{C NMR} (100\text{ MHz, DMSO, } \delta \text{ ppm}): \) 193.5, 152.6, 152.4, 142.3, 141.2, 134.1, 138.6, 123.2, 118.6, 110.4, 109.2, 106.8, 57.6, 50.2, 42.4, 32.6, 27.2

**DEPT-135:** Up peaks: 193.5, 152.6, 152.4, 141.2, 134.1, 138.6, 110.4, 50.2, 42.4, 32.6

**Down peaks:** 142.3, 123.2, 118.6, 110.1, 109.2, 106.8, 57.6, 27.2

IR (KBr): 3436, 2894, 1643, 1618, 1590, 1566

LC-MS: 334.4

% C, H, N Analysis: Calculated: C, 72.05; H, 5.74; N, 12.60
Observed: C, 72.09; H, 5.79; N, 12.69

5d 3,3-Dimethyl-12- (4-nitro phenyl)- 1,2,3,4,5,12-
hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

Molecular Formula  \( \text{C}_{22}\text{H}_{20}\text{N}_{4}\text{O}_{3} \)
Molecular Weight (g·mol\(^{-1}\)) 388.42
Melting Point (°C) > 300

\( ^{1}\text{H NMR} (400\text{ MHz, DMSO, } \delta \text{ ppm}): \) 11.32 (s, 1H, NH), 8.20 - 7.20 (m, 8H, Ar- H), 6.62 (s, 1H, CH), 2.88-2.03 (m, 4H, CH\(_{2}\)), 1.06-0.96 (m, 6H, CH\(_{3}\))

\( ^{13}\text{C NMR} (100\text{ MHz, DMSO, } \delta \text{ ppm}): \) 193.4, 152.6, 144.5, 143.6, 141.4, 138.5, 134.4, 129.2, 123.4, 123.1, 118.4, 110.4, 110.2, 56.4, 50.4, 42.4, 32.6, 27.2

**DEPT-135:** Up peaks: 193.4, 152.6, 144.5, 143.6, 141.4, 138.5, 134.4, 50.4, 42.4, 32.6

**Down peaks:** 129.2, 123.4, 123.1, 118.4, 110.4, 110.2, 56.4, 27.2

IR (KBr): 3540, 3045, 1644, 1612, 1592, 1566

LC-MS: 389.4

% C, H, N Analysis: Calculated: C, 68.03; H, 5.19; N, 14.42
Observed: C, 68.08; H, 5.29; N, 14.48
5e 3,3-Dimethyl-12-(3-nitro phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

<table>
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<tr>
<th>Molecular Formula</th>
<th>C_{22}H_{20}N_{4}O_{3}</th>
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<tr>
<td>Molecular Weight (g·mol⁻¹)</td>
<td>388.42</td>
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<tr>
<td>Melting Point (°C)</td>
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</tr>
</tbody>
</table>

¹H NMR (400 MHz, DMSO, δ ppm): 11.30 (s, 1H, NH), 8.28-6.97 (m, 8H, Ar- H), 6.66 (s, 1H, CH), 2.73-2.05 (m, 4H, CH₂), 1.07-0.92 (m, 6H, CH₃)

¹³C NMR (100 MHz, DMSO, δ ppm): 193.4, 152.6, 147.2, 141.4, 132.5, 132.4, 128.7, 125.2, 121.7, 118.3, 116.4, 110.5, 108.2, 56.1, 50.4, 32.7, 29.4, 27.6

DEPT-135: Up peaks: 193.4, 152.6, 147.2, 141.4, 132.5, 132.4, 128.7, 50.4, 32.7, 29.4

Down peaks: 125.2, 121.7, 118.3, 116.4, 110.5, 108.2, 56.4, 27.2

IR (KBr): 3542, 3049, 1644, 1618, 1592, 1572

LC-MS: 389.4

% C, H, N Analysis: Calculated: C, 68.03; H, 5.19; N, 14.42
Observed: C, 68.08; H, 5.22; N, 14.46

5f 3,3-Dimethyl-12-(2-nitro phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

<table>
<thead>
<tr>
<th>Molecular Formula</th>
<th>C_{22}H_{20}N_{4}O_{3}</th>
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<tbody>
<tr>
<td>Molecular Weight (g·mol⁻¹)</td>
<td>388.42</td>
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<tr>
<td>Melting Point (°C)</td>
<td>&gt; 300</td>
</tr>
</tbody>
</table>

¹H NMR (400 MHz, DMSO, δ ppm): 11.30 (s, 1H, NH), 8.10-7.20 (m, 8H, Ar- H), 6.64 (s, 1H, CH), 2.85-2.04 (m, 4H, CH₂), 1.06-0.96 (m, 6H, CH₃)

¹³C NMR (100 MHz, DMSO, δ ppm): 193.4, 152.6, 149.5, 132.6, 141.4, 138.5, 134.4, 134.2, 129.7, 126.4, 124.6, 123.1, 118.4, 110.4, 110.2, 52.2, 50.4, 42.4, 32.7, 27.2

DEPT-135: Up peaks: 193.4, 152.6, 149.5, 132.6, 141.4, 138.5, 134.4, 50.4, 42.4, 32.6

Down peaks: 134.2, 129.7, 126.4, 124.6, 123.1, 118.4, 110.4, 110.2, 52.2, 27.2

IR (KBr): 3545, 3049, 1644, 1614, 1592, 1569

LC-MS: 389.4

% C, H, N Analysis: Calculated: C, 68.03; H, 5.19; N, 14.42
Observed: C, 68.06; H, 5.23; N, 14.48


Synthesis of benzimidazolo quinazolinones

Chapter-5

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5g 12- (4-chloro phenyl)- 3,3-Dimethyl-1,2,3,4,5,12-
hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

Molecular Formula: C_{22}H_{20}ClN_{3}O

Molecular Weight (g·mol^{-1}): 377.87

Melting Point (°C): > 300

{H NMR (400 MHz, DMSO, δ ppm): 11.33 (s, 1H, NH), 8.21- 7.20 (m, 8H, Ar- H), 6.65 (s, 1H, CH), 2.87-2.33 (m, 4H, CH_{2}), 1.16-0.97 (m, 6H, CH_{3})

{C NMR (100 MHz, DMSO, δ ppm): 193.5, 153.7, 144.6, 144.7, 141.5, 138.6, 135.4, 50.6, 42.4, 32.6, 130.2, 125.5, 124.1, 116.5, 110.6, 110.4, 56.4, 27.3

DEPT-135: Up peaks: 193.5, 153.7, 144.6, 144.7, 141.5, 138.6, 135.4, 50.6, 42.4, 32.6

Down peaks: 130.2, 125.5, 124.1, 116.5, 110.6, 110.4, 56.4, 27.3

IR (KBr): 3485, 3045, 1645, 1613, 1590, 1567

LC-MS: 378.8

% C, H, N Analysis: Calculated: C, 69.93; H, 5.33; N, 11.12

Observed: C, 69.99; H, 5.38; N, 11.18

5h 3,3-Dimethyl-12- (4-hydroxy-3-methoxy phenyl)-1,2,3,4,5,12-
hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

Molecular Formula: C_{23}H_{23}N_{3}O_{3}

Molecular Weight (g·mol^{-1}): 389.45

Melting Point (°C): > 300

{H NMR (400 MHz, DMSO, δ ppm): 11.30 (s, 1H, NH), 9.34 (s, 1H, OH), 6.62 (s, 1H, CH), 7.60- 6.50 (m, 7H, Ar- H), 3.6 (s, 3H, OCH_{3}), 2.88-2.03 (m, 4H, CH_{2}), 1.06-0.96 (m, 6H, CH_{3})

{C NMR (100 MHz, DMSO, δ ppm): 193.2, 152.6, 147.6, 145.6, 141.4, 138.4, 134.4, 131.4, 123.1, 122.8, 118.4, 115.6, 114.8, 110.4, 110.2, 56.4, 50.4, 42.8, 32.6, 27.2

DEPT-135: Up peaks: 193.2, 152.6, 147.6, 145.6, 141.4, 138.4, 134.4, 131.4, 110, 50.4, 42.8, 32.6

Down peaks: 123.1, 122.8, 118.4, 115.6, 114.8, 110.2, 56.4, 27.2

IR (KBr): 3488, 3049, 1667, 1613, 1595, 1569

LC-MS: 390.5

% C, H, N Analysis: Calculated: C, 70.93; H, 5.95; N, 10.89

Observed: C, 70.98; H, 5.99; N, 10.94
Synthesis of benzimidazolo quinazolinones

5i 3,3-Dimethyl-12- (4-hydroxy phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

Molecular Formula \( \text{C}_{22}\text{H}_{21}\text{N}_{3}\text{O}_{2} \)

Molecular Weight (g· mol\(^{-1}\)) 359.42

Melting Point (°C) > 300

\(^{1}\text{H} \) NMR (400 MHz, DMSO, \( \delta \) ppm): 11.05 (s, 1H, NH), 9.35 (s, 1H, OH), 6.30 (s, 1H, CH), 7.38- 6.60 (m, 8H, Ar- H), 2.89-2.04 (m, 4H, CH\(_2\)), 1.06-0.96 (m, 6H, CH\(_3\))

\(^{13}\text{C} \) NMR (100 MHz, DMSO, \( \delta \) ppm): 193.1, 157.2, 150.3, 145.8, 142.4, 132.5, 132.4, 128.6, 122.1, 120.7, 117.3, 115.3, 110.5, 107.1, 54.1, 50.4, 40.1, 32.7, 29.2, 27.1

DEPT-135: Up peaks: 193.1, 157.2, 150.3, 145.8, 142.4, 132.5, 132.4, 50.4, 40.1, 32.7

Down peaks: 128.6, 122.1, 120.7, 117.3, 115.3, 110.5, 107.1, 54.1, 29.2, 27.1

IR (KBr): 3448, 2890, 1641, 1612, 1588, 1568

LC-MS: 360.5

% C, H, N Analysis: Calculated: C, 73.52; H, 5.89; N, 11.69
Observed: C, 73.58; H, 5.96; N, 11.73

5j 3,3-Dimethyl-12- (4-methoxy phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

Molecular Formula \( \text{C}_{23}\text{H}_{23}\text{N}_{3}\text{O}_{2} \)

Molecular Weight (g· mol\(^{-1}\)) 373.45

Melting Point (°C) > 300

\(^{1}\text{H} \) NMR (400 MHz, DMSO, \( \delta \) ppm): 11.30 (s, 1H, NH), 7.60- 6.70 (m, 8H, Ar- H), 6.62 (s, 1H, CH), 3.6 (s, 3H, OCH\(_3\)), 2.88-2.03 (m, 4H, CH\(_2\)), 1.06-0.96 (m, 6H, CH\(_3\))

\(^{13}\text{C} \) NMR (100 MHz, DMSO, \( \delta \) ppm): 193.4, 157.4, 152.4, 141.2, 138.6, 134.3, 130.3, 130.2, 130.0, 123.1, 118.4, 114.4, 114.3, 110.4, 110.1, 56.4, 55.6, 50.6, 42.4, 32.6, 27.1

DEPT-135: Up peaks: 193.4, 157.4, 152.4, 141.2, 138.6, 134.3, 130.2, 110.4, 50.6, 42.4, 32.6

Down peaks: 130.3, 130.0, 123.1, 118.4, 114.4, 114.3, 110.1, 56.4, 55.6, 27.1

IR (KBr): 3435, 2890, 1641, 1612, 1590, 1566

LC-MS: 374.5

% C, H, N Analysis: Calculated: C, 73.97; H, 6.21; N, 11.25
Observed: C, 74.01; H, 6.25; N, 11.29
5k 3,3-Dimethyl-12- (2-hydroxy phenyl)-1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

<table>
<thead>
<tr>
<th>Molecular Formula</th>
<th>C_{22}H_{21}N_{3}O_{2}</th>
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<tbody>
<tr>
<td>Molecular Weight (g·mol⁻¹)</td>
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</tr>
<tr>
<td>Melting Point (°C)</td>
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</tr>
</tbody>
</table>

¹H NMR (400 MHz, DMSO, δ ppm): 11.32 (s, 1H, NH), 9.34 (s, 1H, OH), 7.60- 6.80 (m, 8H, Ar- H), 6.62 (s, 1H, CH), 2.88-2.03 (m, 4H, CH₂), 1.06-0.96 (m, 6H, CH₃)

¹³C NMR (100 MHz, DMSO, δ ppm): 193.1, 157.4, 150.6, 145.6, 142.4, 132.8, 132.6, 128.7, 122.3, 120.7, 117.3, 115.4, 110.5, 107.1, 54.1, 50.4, 40.1, 32.7, 29.2, 27.1

DEPT-135: Up peaks: 193.1, 157.4, 150.6, 145.6, 142.4, 132.8, 132.6, 50.4, 40.1, 32.7

Down peaks: 128.7, 122.3, 120.7, 117.3, 115.4, 110.5, 107.1, 54.1, 29.2, 27.1

IR (KBr): 3435, 2894, 1645, 1615, 1590, 1568

LC-MS: 360.4

% C, H, N Analysis: Calculated: C, 73.52; H, 5.89; N, 11.69

Observed: C, 73.58; H, 5.96; N, 11.73

5l 3,3-Dimethyl-12- (3-hydroxy phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

<table>
<thead>
<tr>
<th>Molecular Formula</th>
<th>C_{22}H_{21}N_{3}O_{2}</th>
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<tr>
<td>Molecular Weight (g·mol⁻¹)</td>
<td>359.42</td>
</tr>
<tr>
<td>Melting Point (°C)</td>
<td>&gt; 300</td>
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</tbody>
</table>

¹H NMR (400 MHz, DMSO, δ ppm): 11.30 (s, 1H, NH), 9.32 (s, 1H, OH), 7.60- 6.75 (m, 8H, Ar- H), 6.62 (s, 1H, CH), 7.60-7.65 (m, 8H, Ar- H), 2.88-2.03 (m, 4H, CH₂), 1.06-0.96 (m, 6H, CH₃)

¹³C NMR (100 MHz, DMSO, δ ppm): 193.2, 157.4, 150.5, 145.6, 142.6, 132.4, 132.2, 128.7, 122.3, 120.8, 117.4, 115.3, 110.5, 107.2, 54.1, 50.4, 40.1, 32.7, 29.2, 27.1

DEPT-135: Up peaks: 193.2, 157.4, 150.5, 145.6, 142.6, 132.4, 132.2, 50.4, 40.1, 32.7

Down peaks: 128.7, 122.3, 120.8, 117.4, 115.3, 110.5, 107.2, 54.1, 29.2, 27.1

IR (KBr): 3438, 2892, 1645, 1615, 1590, 1568

LC-MS: 360.4

% C, H, N Analysis: Calculated: C, 73.52; H, 5.89; N, 11.69

Observed: C, 73.58; H, 5.94; N, 11.74
### 5m 3,3-Dimethyl-12- (2-carboxyl phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

<table>
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<th>Property</th>
<th>Value</th>
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<td>Melting Point (°C)</td>
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**1H NMR (400 MHz, DMSO, δ ppm):**

- 11.30 (s, 1H, NH), 11.2 (s, 1H, COOH), 7.90-7.20 (m, 8H, Ar-H), 6.62 (s, 1H, OH), 2.88-2.03 (m, 4H, CH2), 1.06-0.96 (m, 6H, CH3)

**13C NMR (100 MHz, DMSO, δ ppm):**

- 193.4, 172.8, 152.6, 141.2, 139.2, 138.6, 134.4, 133.6, 131.4, 130.4, 128.7, 125.4, 123.2, 118.6, 110.4, 110.2, 53.6, 50.6, 42.4, 32.6, 27.2

**DEPT-135:**

- Up peaks: 193.4, 172.8, 152.6, 141.2, 139.2, 138.6, 134.4, 131.4, 110.4, 50.6, 42.4, 32.6

- Down peaks: 133.6, 130.4, 128.7, 125.4, 123.2, 118.6, 110.2, 53.6, 27.2

**IR (KBr):**

- 3413, 2891, 1645, 1615, 1592, 1564

**LC-MS:**

- 388.4

% C, H, N Analysis:

- Calculated: C, 71.30; H, 5.46; N, 10.85
- Observed: C, 71.34; H, 5.49; N, 10.89

---

### 5n 12- phenyl- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

<table>
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<th>Value</th>
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<td>Melting Point (°C)</td>
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**1H NMR (400 MHz, DMSO, δ ppm):**

- 11.40 (s, 1H, NH), 7.60-7.20 (m, 9H, Ar-H), 6.62 (s, 1H, CH), 3.0-1.6 (m, 6H, CH2)

**13C NMR (100 MHz, DMSO, δ ppm):**

- 193.4, 152.4, 141.2, 138.6, 137.6, 134.4, 129.1, 128.4, 123.4, 118.4, 110.4, 110.2, 56.2, 36.4, 27.2, 22.3

**DEPT-135:**

- Up peaks: 193.4, 152.4, 141.2, 138.6, 137.6, 134.4, 110.4, 36.4, 27.2, 22.3

- Down peaks: 129.1, 128.4, 125.6, 123.4, 118.4, 110.2, 56.2

**IR (KBr):**

- 3438, 2890, 1645, 1615, 1592, 1564

**LC-MS:**

- 316.4

% C, H, N Analysis:

- Calculated: C, 76.17; H, 5.43; N, 13.32
- Observed: C, 76.21; H, 5.48; N, 13.39
5o 12- (4- methyl phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

<table>
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<th>Molecular Formula</th>
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\textsuperscript{1}H NMR (400 MHz, DMSO, δ ppm): 11.30 (s, 1H, NH), 7.70 - 7.10 (m, 8H, Ar-H)
6.62 (s, 1H, CH), 2.34 (s, 3H, CH\textsubscript{3}), 3.00-1.60 (m, 6H, CH\textsubscript{2})

\textsuperscript{13}C NMR (100 MHz, DMSO, δ ppm): 193.4, 152.6, 141.4, 138.6, 135.6, 134.6, 134.4,
128.7, 123.2, 118.5, 110.4, 110.2, 56.4, 36.2, 27.4, 22.1, 21.4

**DEPT-135**: Up peaks: 193.4, 152.6, 141.4, 138.6, 135.6, 134.6, 134.4, 110.4, 36.2, 27.4, 22.1
Down peaks: 128.7, 123.2, 118.5, 110.2, 56.4, 21.4

IR (KBr): 3433, 2892, 1641, 1595, 1568

%
C, H, N Analysis: Calculated: C, 76.57; H, 5.81; N, 12.76
Observed: C, 76.62; H, 5.86; N, 12.79

5p 12- (4- nitro phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

<table>
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<th>Molecular Formula</th>
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</table>

\textsuperscript{1}H NMR (400 MHz, DMSO, δ ppm): 11.30 (s, 1H, NH), 8.30 - 7.20 (m, 8H, Ar-H)
6.62 (s, 1H, CH), 3.00-1.60 (m, 6H, CH\textsubscript{2})

\textsuperscript{13}C NMR (100 MHz, DMSO, δ ppm): 193.4, 152.6, 144.7, 143.6, 141.4, 138.7, 134.6,
123.7, 123.2, 121.7, 118.5, 110.4, 110.2, 56.4, 36.2, 27.4, 22.4

**DEPT-135**: Up peaks: 193.4, 152.6, 144.7, 143.6, 141.4, 138.7, 134.6, 110.4, 36.2, 27.4, 22.4
Down peaks: 123.7, 123.2, 121.7, 118.5, 110.2, 56.4

IR (KBr): 3436, 2893, 1645, 1615, 1590, 1568

%
C, H, N Analysis: Calculated: C, 66.66; H, 4.48; N, 15.55
Observed: C, 66.68; H, 4.54; N, 15.61
### 5q 12- (3- nitro phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

<table>
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<th>Value</th>
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<tbody>
<tr>
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$^1\text{H} \text{NMR (400 MHz, DMSO, } \delta \text{ ppm)}: 11.30 \text{ (s, 1H, NH), 8.15 - 7.20 (m, 8H, Ar-H)}$

$^13\text{C} \text{NMR (100 MHz, DMSO, } \delta \text{ ppm)}: 193.4, 152.6, 147.2, 141.4, 138.7, 138.4, 135.2, 134.6, 129.4, 125.6, 123.2, 120.4, 118.5, 110.4, 110.2, 55.4, 36.2, 27.4, 22.4$

**DEPT-135:**
- **Up peaks:** 193.4, 152.6, 147.2, 141.4, 138.7, 138.4, 110.4, 36.2, 27.4, 22.4
- **Down peaks:** 135.2, 134.6, 129.4, 125.6, 123.2, 120.4, 118.5, 110.2, 55.4

**IR (KBr):** 3437, 2895, 1645, 1615, 1590, 1566

**LC-MS:** 361.4

% C, H, N Analysis:
- **Calculated:** C, 66.66; H, 4.48; N, 15.55
- **Observed:** C, 66.69; H, 4.52; N, 15.59

### 5r 12- (2- nitro phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
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</thead>
<tbody>
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<td>Molecular Formula</td>
<td>$\text{C}<em>{20}\text{H}</em>{16}\text{N}<em>{4}\text{O}</em>{3}$</td>
</tr>
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<td>Molecular Weight (g·mol⁻¹)</td>
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</tr>
<tr>
<td>Melting Point (°C)</td>
<td>&gt; 300</td>
</tr>
</tbody>
</table>

$^1\text{H} \text{NMR (400 MHz, DMSO, } \delta \text{ ppm)}: 11.30 \text{ (s, 1H, NH), 8.00 - 7.20 (m, 8H, Ar-H)}$

$^13\text{C} \text{NMR (100 MHz, DMSO, } \delta \text{ ppm)}: 193.4, 152.6, 149.2, 141.4, 138.7, 134.6, 132.6, 134.2, 129.7, 126.4, 124.6, 123.2, 118.5, 110.4, 110.2, 52.4, 36.2, 27.4, 22.4$

**DEPT-135:**
- **Up peaks:** 193.4, 152.6, 149.2, 141.4, 138.7, 134.6, 110.4, 36.2, 27.4, 22.4
- **Down peaks:** 134.2, 132.6, 129.7, 126.4, 124.6, 123.2, 118.5, 110.2, 52.4

**IR (KBr):** 3438, 2895, 1644, 1615, 1590, 1566

**LC-MS:** 361.4

% C, H, N Analysis:
- **Calculated:** C, 66.66; H, 4.48; N, 15.55
- **Observed:** C, 66.72; H, 4.56; N, 15.61
5s \(12\)- (4- chloro phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

<table>
<thead>
<tr>
<th>Molecular Formula</th>
<th>(C_{20}H_{16}ClN_3O)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular Weight (g· mol(^{-1}))</td>
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<tr>
<td>Melting Point ((^{\circ})C)</td>
<td>&gt; 300</td>
</tr>
</tbody>
</table>

\(^1\)H NMR (400 MHz, DMSO, \(\delta\) ppm): 11.30 (s, 1H, NH), 7.60-7.15 (m, 8H, Ar- H), 6.62 (s, 1H, OH), 3.0-1.6 (m, 6H, CH\(_2\) )

\(^13\)C NMR (100 MHz, DMSO, \(\delta\) ppm): 193.4, 152.4, 141.4, 138.6, 135.4, 134.4, 131.5, 130.2, 128.6, 123.1, 118.4, 110.4, 110.2, 56.4, 36.2, 27.4, 22.2

DEPT-135: Up peaks: 193.4, 152.4, 141.4, 138.6, 135.4, 134.4, 131.5, 110.4, 36.2, 27.4, 22.2

Down peaks: 130.2, 128.6, 123.1, 118.4, 110.2, 56.4

IR (KBr): 3438, 2890, 1644, 1615, 1590, 1564

LC-MS: 350.8

% C, H, N Analysis: Calculated: C, 68.67; H, 4.61; N, 12.01
Observed: C, 68.71; H, 4.68; N, 12.07

---

5t \(12\)- (4- hydroxy phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

<table>
<thead>
<tr>
<th>Molecular Formula</th>
<th>(C_{20}H_{17}N_3O_2)</th>
</tr>
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<tbody>
<tr>
<td>Molecular Weight (g· mol(^{-1}))</td>
<td>331.37</td>
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<tr>
<td>Melting Point ((^{\circ})C)</td>
<td>&gt; 300</td>
</tr>
</tbody>
</table>

\(^1\)H NMR (400 MHz, DMSO, \(\delta\) ppm): 11.30 (s, 1H, NH), 9.32 (s, 1H, OH), 7.60-6.63 (m, 8H, Ar- H), 6.62 (s, 1H, CH), 3.0-1.6 (m, 6H, CH\(_2\) )

\(^13\)C NMR (100 MHz, DMSO, \(\delta\) ppm): 193.4, 155.6, 152.6, 141.4, 138.7, 134.4, 130.6, 130.4, 123.2, 118.6, 115.6, 110.4, 110.2, 56.4, 36.2, 27.4, 22.2

DEPT-135: Up peaks: 193.4, 155.6, 152.6, 141.4, 138.7, 134.4, 130.4, 110.4, 36.2, 27.4, 22.2

Down peaks: 130.6, 123.2, 118.6, 115.6, 110.2, 56.4

IR (KBr): 3438, 2895, 1645, 1618, 1590, 1569

LC-MS: 332.4

% C, H, N Analysis: Calculated: C, 72.49; H, 5.17; N, 12.68
Observed: C, 72.52; H, 5.19; N, 12.74
5u 12- (3-hydroxy phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

**Molecular Formula**  
\( C_{20}H_{17}N_{3}O_{2} \)

**Molecular Weight (g·mol\(^{-1}\))**  
331.37

**Melting Point (°C)**  
> 300

**\(^1\)H NMR (400 MHz, DMSO, \( \delta \) ppm):**  
11.30 (s, 1H, NH), 9.32 (s, 1H, OH), 7.20-6.65 (m, 8H, Ar- H), 6.62 (s, 1H, CH), 3.0-1.6 (m, 6H, CH₂)

**\(^13\)C NMR (100 MHz, DMSO, \( \delta \) ppm):**  
193.4, 155.6, 152.6, 141.4, 139.2, 138.7, 134.4, 130.2, 123.4, 123.2, 121.4, 112.6, 56.4, 36.2, 27.4, 22.2

**DEPT-135:**  
Up peaks: 193.4, 155.6, 152.6, 141.4, 139.2, 138.7, 134.4, 36.2, 27.4, 22.2

Down peaks: 130.2, 123.4, 123.2, 121.4, 112.6, 56.4

**IR (KBr):**  
3438, 2893, 1646, 1617, 1590, 1569

**LC-MS:**  
332.3

**% C, H, N Analysis:**  
Calculated: C, 72.49; H, 5.17; N, 12.68  
Observed: C, 72.54; H, 5.20; N, 12.76

---

5v 12- (2-hydroxy phenyl)- 1,2,3,4,5,12-hexahydro [4, 5] imidazo [2,1-d] quinazolin-1-one

**Molecular Formula**  
\( C_{20}H_{17}N_{3}O_{2} \)

**Molecular Weight (g·mol\(^{-1}\))**  
331.37

**Melting Point (°C)**  
> 300

**\(^1\)H NMR (400 MHz, DMSO, \( \delta \) ppm):**  
11.30 (s, 1H, NH), 9.32 (s, 1H, OH), 7.60-6.63 (m, 8H, Ar- H), 6.62 (s, 1H, CH), 3.0-1.6 (m, 6H, CH₂)

**\(^13\)C NMR (100 MHz, DMSO, \( \delta \) ppm):**  
193.4, 155.6, 152.6, 141.4, 138.7, 134.4, 130.6, 127.2, 124.7, 123.2, 121.4, 118.6, 115.6, 110.4, 110.2, 50.4, 36.2, 27.4, 22.2

**DEPT-135:**  
Up peaks: 193.4, 155.6, 152.6, 141.4, 138.7, 134.4, 124.7, 118.6, 36.2, 27.4, 22.2

Down peaks: 130.6, 127.2, 123.2, 121.4, 118.6, 115.6, 110.4, 50.4

**IR (KBr):**  
3433, 2894, 1646, 1617, 1590, 1569

**LC-MS:**  
332.3

**% C, H, N Analysis:**  
Calculated: C, 72.49; H, 5.17; N, 12.68  
Observed: C, 72.59; H, 5.22; N, 12.78
Figure 5.1 $^1$H NMR spectrum of compound 5e

Figure 5.2 $^{13}$C NMR spectrum of compound 5e
**Figure 5.3** APT spectrum of compound 5e

**Figure 5.4** IR spectrum of compound 5e
Figure 5.5 Mass spectrum of compound 5e

Figure 5.6 $^1$HNMR spectrum of compound 5i
Figure 5.7 $^{13}$C NMR spectrum of compound 5i

Figure 5.8 APT spectrum of compound 5i
Figure 5.9 IR spectrum of compound 5i

Figure 5.10 Mass spectrum of compound 5i
References:


Synthesis of benzimidazolo quinazolinones


Lipson, V. V.; Desenko, S. M.; Shirobokova, M. G.; Borodina, V. V., Synthesis of 9-Aryl-6,6-dimethyl-5,6,7,9-tetrahydro-1,2,4-triazolo[5,1-b]quinazolin-8(4H)ones. *Chemistry of Heterocyclic Compounds* 2003, 39, 1213-1217.


Heravi, M. M.; Ranjbar, L.; Derikvand, F.; Alimadadi, B.; Oskooie, H. A.; Bamoharram, F. F., A three component one-pot procedure for the synthesis of [1,2,4]triazolo/benzimidazolo-quinazolinone derivatives in the presence of H$_6$P$_2$W$_{18}$O$_{44}$H$_2$O as a green and reusable catalyst. *Molecular Diversity* 2008, 12, 181-185.


6.1 Introduction

An antioxidant is an agent capable of slowing or preventing the oxidation of other molecules. Oxidation reactions can produce free radicals, which start chain reactions that damage cells. Antioxidants terminate these chain reactions by trapping free radical intermediates, and inhibit other oxidation reactions by being oxidized themselves. As a result, antioxidants are often reducing agents such as thiols, ascorbic acid or polyphenols. In addition, natural antioxidants in medicine have many industrial uses, such as preservatives in food, cosmetics and preventing the degradation of rubber and gasoline.

An antioxidant is “any substance that, when present at low concentrations as compared to oxidisable substrate, significantly delays or prevents oxidation of that substrate” [1]. The term antioxidant originally was used to refer specifically to a chemical that prevents the oxygen consumption. In the late 19th and early 20th century, an extensive study was devoted to uses of these agents in important industrial processes such as prevention of metal corrosion, vulcanization of rubber, and polymerization of fuels in fouling of internal combustion engines. Early research on antioxidants in biology focused on preventing the oxidation of unsaturated fats, which cause rancidity. Antioxidant activity could be measured simply by placing the fat in a closed container with oxygen and measuring the rate of oxygen consumption. However, it was the identification test of vitamins A, C and E as antioxidants that revolutionized the field and led to the realization of the importance of antioxidants in the biochemistry of living organisms. The possible mechanism of their action was first explored when it was recognized that a substance with anti-oxidative activity is likely to be one that itself is readily oxidized. Research into how vitamin-E prevents the process of lipid peroxidation led to the identification of antioxidants as reducing agents that prevent oxidative reactions, often by scavenging reactive oxygen species before they can damage cells.

Jayanna et al. [2] synthesized benzoazoles and screened them for biological activities such as in vitro antioxidant (total antioxidant capacity, total reductive capability and DPPH radical scavenging activity. Temiz-Arpaci et al. [3] reported in vitro antioxidant properties of some new benzoazoles, benzimidazoles, and benzothiazoles by their effects on the rat liver microsomal