An efficient one pot three-component synthesis of dihydropyran[3,2-c] chromenes using ammonium metavanadate as catalyst

Balasaheb V. Shitole, Nana V. Shitole, Murlidhar S. Shingare and Gopal K. Kakde

Abstract

We report ammonium metavanadate catalyzed one-pot synthesis of 3,4-dihydropyran[3,2-c]chromenes, from aldehydes, active methylene compounds malononitrile and 4-hydroxycoumarin in water:ethanol(1:1) under reflux. The attractive features of this process are mild reaction conditions, short reaction times, easy isolation of products, and excellent yields.

Keywords:
Chromenes
Multi-component reaction
Ammonium metavanadate

1. Introduction

The development of multi-component reactions (MCRs) designed to produce elaborate biologically active compounds has become an important area of research in organic, combinatorial and medicinal chemistry. One-pot multi-component reaction strategies offer significant advantages over conventional linear-type syntheses by virtue of their convergence, productivity, facile execution and high yields. 2-amino-tetrahydro-4H-chromene derivatives represent an important class of bioactive molecules. They are often used in cosmetics, pigments and utilized as potential agrochemicals. Some derivatives of chromenes constitute a core skeleton of many natural products and bioactive molecules which seize various pharmacological actions, such as diuretic, anti-coagulant, anti-cancer, anti-HIV, antitumor, anti-malarial activities, anti-alzheimer, anti-lepukemic and antibacterial and anti-anaphylactic activities.

A number of methods have been reported for the synthesis of 3,4-dihydropyran[3,2-c]chromenes with the catalysts diammonium hydrogen phosphate, H₆P₂W₁₈O₆₂·18H₂O, tetrabutylammonium...
bromide, hexamethylenetetramine, 1,8-diazabicyclo[5.4.0]undec-7-ene, sodium dodecylsulfate, triethylenetetraminum trifluoroacetate, α-Fe$_2$O$_3$ nanoparticles, 4-(dimethylamino) pyridine, CuO nanoparticles, silica-bonded N-propylpiperazine sodium n-propionate, silica-grafted ionic liquid, potassium phthalimide in aqueous media, piperidine-functionalized poly(ethylene glycol) bridged dicationic liquid, polymer supported sulfanilic acid, basic ionic liquid, ammonium acetate, cellulose-SO$_3$H electrolysis in an undivided cell in the presence of sodium bromide as an electrolyte, piperidine/triethyl amine in aqueous media and many of these procedures have merit; however, most require refluxing for hours in organic solvents, complex steps, use of expensive catalysts and tedious work-up. We decided to investigate ammonium metavanadate for use as catalyst for the synthesis of dihydropyrano[3,2-c]chromene derivatives in aqueous ethanol. Hence the search continues for a better catalyst in the synthesis of dihydropyrano[3,2-c] chromenes in terms of operational simplicity and economic viability. Herein we report the use of ammonium metavanadate (NH$_4$VO$_3$) as a water soluble, inorganic acid that meets the demand for an economic catalyst. It is employed similar to vanadium pentoxide and as a catalyst in oxidation reactions with other cocatalysts. It is a reagent used in analytical chemistry, the photographic industry, and the textile industry. This is the first report of utilizing ammonium metavanadate as a catalyst for the synthesis of dihydropyrano[3,2-c] chromenes.

2. Results and Discussion

As a contribution of our research work devoted to the development of useful synthetic methodologie. We herein report an eco-friendly, facile and efficient methodology for the synthesis of dihydropyrano[3,2-c] chromene. This method involves the efficient synthesis of substituted dihydropyrano[3,2-c] chromenes by treatment of 4-chlorobenzaldehyde (1mmol), malononitrile (1mmol), 4-hydroxycoumarine (1mmol) and ammonium metavanadate (7.5mol%) as catalyst dissolved in 5 ml of ethanol:water(1:1) at reflux temperature for 8 - 14 min (Scheme 1).

Scheme 1. An eco-friendly, facile and efficient methodology for the synthesis of dihydropyrano[3,2-c] chromene

To evaluate the effect of solvent, various solvents such as water, ethanol:water (1:3,v:v), ethanol:water (1:2,v:v), ethanol:water (1:1,v:v) and ethanol were used for the model reaction. The desired product was obtained in 39, 47, 65, 94 and 94% yields respectively after 10 min at reflux condition. Water:ethanol (1:1) stand out as the solvent of choice among the solvents tested. Because of the rapid conversion and excellent yield (93%) of desired product obtained (Table 1, entry 4) as the product formed in lower yields (39-65%) by using other solvents (Table 1, entries 1-3).

Table 1. Screening of solvents

<table>
<thead>
<tr>
<th>Entry</th>
<th>Solvent</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>water</td>
<td>39</td>
</tr>
<tr>
<td>2</td>
<td>ethanol, water (1:3)</td>
<td>47</td>
</tr>
<tr>
<td>3</td>
<td>ethanol, water (1:2)</td>
<td>65</td>
</tr>
<tr>
<td>4</td>
<td>ethanol, water (1:1)</td>
<td>93</td>
</tr>
<tr>
<td>5</td>
<td>ethanol</td>
<td>93</td>
</tr>
</tbody>
</table>
To determine the appropriate concentration of the catalyst ammonium metavanadate, it has been investigated the model reaction first without catalyst and very less product is obtained (i.e. trace) at different concentrations of catalyst like 2.5, 5, 7.5 and 10 mol% the product formed in 57, 72, 93 and 93% yields, respectively (Table 2). This indicates that 7.5 mol% of ammonium metavanadate is sufficient for the best result by considering the reaction time and yield of product. A role of ammonium metavanadate has been proposed to activate the carbonyl compound by binding of ammonium metavanadate with the carbonyl oxygen which ultimately enhances the electrophilicity of the carbonyl carbon leads to increase in the reaction rate.

Table 2. Optimization of the amount of Ammonium metavanadate

<table>
<thead>
<tr>
<th>Entry</th>
<th>Ammonium metavanadate (mol %)</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.5</td>
<td>57</td>
</tr>
<tr>
<td>2</td>
<td>5</td>
<td>72</td>
</tr>
<tr>
<td>3</td>
<td>7.5</td>
<td>93</td>
</tr>
<tr>
<td>4</td>
<td>10</td>
<td>93</td>
</tr>
</tbody>
</table>

*aReaction conditions: 1 (1 mmol), 2 (1 mmol), 3 (1 mmol) ammonium metavanadate in water ethanol (1:1) at reflux temperature.;
bIsolated yields

In order to show the merit of NH₄VO₃ in comparison with the other catalyst used for the similar reaction, a side by side comparison was run with some of the more common catalysts used for this chemistry. The results are presented in Table -3. It is evident from the results that NH₄VO₃ was an effective catalyst for the synthesis of dihydropyrano[3,2-c] chromenes.

Table 3. Effect of different catalysts for the synthesis of 3,4-dihydropyrano[c]chromenes from the condensation of on the reaction of benzaldehyde, 4-hydroxycoumarin and malononitrile

<table>
<thead>
<tr>
<th>Entry</th>
<th>Catalyst</th>
<th>Catalyst Conc.</th>
<th>Solvent/Medium</th>
<th>Temp (°C)</th>
<th>Time (min)</th>
<th>Yield (%)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DAHP</td>
<td>(10 mol%)</td>
<td>Ethanol–water</td>
<td>25</td>
<td>240</td>
<td>85</td>
<td>16</td>
</tr>
<tr>
<td>2</td>
<td>H₃P₂W₁₈O₆₂·18H₂O</td>
<td>(10 mol%)</td>
<td>Ethanol</td>
<td>Reflux</td>
<td>30-85</td>
<td>80</td>
<td>17</td>
</tr>
<tr>
<td>3</td>
<td>TBAB</td>
<td>(10 mol%)</td>
<td>Water</td>
<td>Reflux</td>
<td>45-60</td>
<td>93</td>
<td>18</td>
</tr>
<tr>
<td>4</td>
<td>(CH₂)₆N₄</td>
<td>(10 mol%)</td>
<td>Ethanol</td>
<td>Reflux</td>
<td>40</td>
<td>95</td>
<td>19</td>
</tr>
<tr>
<td>5</td>
<td>SDS</td>
<td>(20 mol%)</td>
<td>Water</td>
<td>60</td>
<td>150</td>
<td>88</td>
<td>21</td>
</tr>
<tr>
<td>6</td>
<td>[TETA]TFA</td>
<td>(10 mol%)</td>
<td>Ethanol–water</td>
<td>Reflux</td>
<td>30</td>
<td>95</td>
<td>22</td>
</tr>
<tr>
<td>7</td>
<td>α-Fe₂O₃</td>
<td>(10 wt%)</td>
<td>Ethanol</td>
<td>Reflux</td>
<td>30</td>
<td>93</td>
<td>23</td>
</tr>
<tr>
<td>8</td>
<td>DMAP</td>
<td>(20 mol%)</td>
<td>Ethanol</td>
<td>Reflux</td>
<td>4</td>
<td>94</td>
<td>24</td>
</tr>
<tr>
<td>9</td>
<td>CuO nanoparticles</td>
<td>(15 mol%)</td>
<td>Water</td>
<td>100</td>
<td>6</td>
<td>93</td>
<td>25</td>
</tr>
<tr>
<td>10</td>
<td>ammonium metavanadate</td>
<td>(7.5 mol%)</td>
<td>Ethanol–water</td>
<td>Reflux</td>
<td>9</td>
<td>94</td>
<td>Present method</td>
</tr>
</tbody>
</table>

To study the generality of this process, variety of examples were illustrated for the synthesis of dihydropyrano[3,2-c] chromenes and the results are summarized in Table 4. The reaction is compatible for various substituents such as -CH₃, -OCH₃, -OH, -N(CH₃)₂, and –Cl. The formation of desired product has been confirmed by ¹H NMR and IR spectroscopic analysis techniques and compared with the corresponding literature data.
Table 4. Synthesis of dihydropyrano[3,2-c] chromenes using Ammonium metavanadate

<table>
<thead>
<tr>
<th>Sr.No.</th>
<th>Ar-CHO</th>
<th>Product</th>
<th>Time (min)</th>
<th>Yield(%)</th>
<th>M. P°C</th>
<th>Found</th>
<th>Reported</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>C6H5</td>
<td>4a</td>
<td>10</td>
<td>94</td>
<td></td>
<td>257-259</td>
<td>256-258[16]</td>
</tr>
<tr>
<td>2</td>
<td>4-ClC6H4</td>
<td>4b</td>
<td>09</td>
<td>93</td>
<td></td>
<td>260-262</td>
<td>263-265[16]</td>
</tr>
<tr>
<td>3</td>
<td>4-OHC6H4</td>
<td>4c</td>
<td>14</td>
<td>92</td>
<td></td>
<td>262-264</td>
<td>266-268[27]</td>
</tr>
<tr>
<td>4</td>
<td>4-CH3C6H4</td>
<td>4d</td>
<td>12</td>
<td>93</td>
<td></td>
<td>259-261</td>
<td>253-255[27]</td>
</tr>
<tr>
<td>5</td>
<td>2-ClC6H4</td>
<td>4e</td>
<td>11</td>
<td>90</td>
<td></td>
<td>243-245</td>
<td>245-246[31]</td>
</tr>
<tr>
<td>6</td>
<td>3-ClC6H4</td>
<td>4f</td>
<td>10</td>
<td>91</td>
<td></td>
<td>244-246</td>
<td>241-243[27]</td>
</tr>
<tr>
<td>7</td>
<td>4-NO2C6H4</td>
<td>4g</td>
<td>08</td>
<td>95</td>
<td></td>
<td>255-257</td>
<td>258-260[16]</td>
</tr>
<tr>
<td>8</td>
<td>4-OCH3C6H4</td>
<td>4h</td>
<td>12</td>
<td>90</td>
<td></td>
<td>242-244</td>
<td>240-242[16]</td>
</tr>
<tr>
<td>9</td>
<td>3-NO2C6H4</td>
<td>4i</td>
<td>09</td>
<td>92</td>
<td></td>
<td>260-262</td>
<td>262-264[16]</td>
</tr>
<tr>
<td>10</td>
<td>2-NO2C6H4</td>
<td>4j</td>
<td>10</td>
<td>89</td>
<td></td>
<td>261-263</td>
<td>258-260[27]</td>
</tr>
<tr>
<td>11</td>
<td>2,4 - Cl2-C6H3</td>
<td>4k</td>
<td>12</td>
<td>91</td>
<td></td>
<td>260-262</td>
<td>257-259[16]</td>
</tr>
<tr>
<td>12</td>
<td>3,4,5-(OCH3)3C6H2</td>
<td>4l</td>
<td>13</td>
<td>90</td>
<td></td>
<td>238-240</td>
<td>236-238[27]</td>
</tr>
<tr>
<td>13</td>
<td>4-F C6H4</td>
<td>4m</td>
<td>09</td>
<td>94</td>
<td></td>
<td>256-258</td>
<td>258-259[31]</td>
</tr>
<tr>
<td>14</td>
<td>4-(CH3)2NC6H4</td>
<td>4n</td>
<td>14</td>
<td>92</td>
<td></td>
<td>262-263</td>
<td>265-267[31]</td>
</tr>
</tbody>
</table>

3. Conclusions

In conclusion, this paper has described a simple and proficient approach for the synthesis of dihydropyrano[3,2-c] chromenes catalyzed by ammonium metavanadate in aqueous alcoholic media. Present methodology offers very attractive features such as simple experimental procedure, higher yields and economic viability, when compared with other method as well as with other catalysts, and will have wide scope in organic synthesis.

Acknowledgements

We are thankful to the University Grants Commission, New Delhi, for financial support which is gratefully acknowledged and the Sophisticated Analytical Instrument Facility, Punjab University, Chandigarh for providing spectroscopic data.

4. Experimental

4.1. Materials and Methods

Chemicals were purchased from Merck, Fluka and Aldrich chemical companies. All yields refer to isolated products unless otherwise stated. Melting points were determined in an open capillary. 1H nuclear magnetic resonance (NMR) (500 MHz) with tetramethylsilane as internal standard and dimethylsulfoxide DMSO-d6 as solvent. Fourier transform infrared (IR) spectra were obtained as KBr discs on a Shimadzu spectrometer. Mass spectra (MS) were determined on a Varion-Saturn 2000 GC/MS instrument.

4.2. General procedure for the synthesis of substituted of 3,4-dihydropyrano[c]chromenes.

A mixture of substituted aromatic aldehyde (1mmol), malononitrile (1mmol) and 4-hydroxycoumarine (1mmol) in the presence of ammonium metavanadate (7.5mol %) as a catalyst was stirred at reflux temperature in ethanol:water (1:1) (7 ml) for 8-14 minutes. After the appropriate time, the mixture was cool than poor on ice cold water solidified the product filtered its. The crude solid material was purified by recrystallization from ethanol.

4.3 Spectral data for selected compounds

2-amino-4,5-dihydro-5-oxo-4-phenylpyrano[3,2-c]chromene-3-carbonitrile (4a)

IR (KBr) : 3376 (NH2), 2195 (CN), 1703 (C=O) cm⁻¹; 1H NMR (d6-DMSO, 400 MHz) δ : 4.46 (s, 1H, CH), 7.23–7.91 (m, 11H, Ar, NH2) ppm; 13C NMR (d6-DMSO, 100 MHz), δ : 37.4, 58.5, 104.5, 113.4, 117.0, 119.6, 122.9, 125.1, 127.6, 128.1, 128.9, 133.4, 143.8, 152.6, 153.9, 158.4, 159.9 ppm.
2-amino-4-(4-chlorophenyl)-4,5-dihydro-5-oxopyrano[3,2-c]chromene-3-carbonitrile(4b)
IR (KBr): 3281 (NH$_2$), 2185 (CN), 1701 (C=O) cm$^{-1}$; $^1$H NMR (d$_6$-DMSO 400 MHz) $\delta$: 4.68 (s, 1H, CH), 7.47–8.19 (m, 10H, Ar, NH$_2$) ppm. $^{13}$C NMR (d$_6$-DMSO, 100 MHz), $\delta$: 57.2, 103.2, 113.3, 117.0, 119.3, 123.0, 124.1, 124.7, 125.1, 129.6, 129.7, 133.6, 147.0, 151.2, 152.7, 154.4, 158.5, 160.0 ppm.

2-amino-4,5-dihydro-4-(4-hydroxyphenyl)-5-oxopyrano[3,2-c]chromene-3-carbonitrile(4c)
IR (KBr): 3353 (NH$_2$), 2157 (CN), 1712 (C=O) cm$^{-1}$; $^1$H NMR (d$_6$-DMSO 400 MHz) $\delta$: 4.51 (s, 1H, CH), 7.47–8.05 (m, 10H, Ar, NH$_2$) ppm. 9.03 (s, OH) ppm. $^{13}$C NMR (d$_6$-DMSO, 100 MHz), $\delta$: 58.8, 104.5, 112.8, 115.6, 115.9, 119.8, 122.5, 125.0, 128.9, 133.2, 133.8, 152.4, 154.1, 156.8, 158.3, 160.2 ppm.

2-amino-4,5-dihydro-5-oxo-4-p-tolylpyrano[3,2-c]chromene-3-carbonitrile(4d)
IR (KBr): 3333 (NH$_2$), 2878 (CH$_3$), 2166 (CN), 1708 (C=O) cm$^{-1}$; $^1$H NMR (d$_6$-DMSO 400 MHz) $\delta$: 2.26 (s, 3H), 4.42 (s, 1H, CH), 7.32–8.61 (m, 10H, Ar, NH$_2$) ppm. $^{13}$C NMR (d$_6$-DMSO, 100 MHz), $\delta$: 21.4, 58.3, 103.9, 113.2, 116.8, 119.2, 123.2, 125.2, 127.9, 128.7, 133.7, 135.9, 139.8, 152.9, 152.9, 159.1, 160.2 ppm.

2-amino-4-(2-chlorophenyl)-4,5-dihydro-5-oxopyrano[3,2-c]chromene-3-carbonitrile (4e)
IR (KBr) : 3342 (NH$_2$), 2159 (CN), 1707 (C=O) cm$^{-1}$; $^1$H NMR (d$_6$-DMSO 400 MHz) $\delta$: 4.46 (s, 1H, CH), 7.52–8.91 (m, 10H, Ar, NH$_2$) ppm. $^{13}$C NMR (d$_6$-DMSO, 100 MHz), $\delta$: 57.7, 103.9, 112.8, 115.6, 116.1, 119.4, 122.7, 125.3, 128.5, 132.9, 134.4, 152.4, 154.3, 157.9, 158.1, 159.9 ppm.

2-amino-4-(3-chlorophenyl)-4,5-dihydro-5-oxopyrano[3,2-c]chromene-3-carbonitrile (4f)
IR (KBr) : 3376 (NH$_2$), 2195 (CN), 1708(C=O) cm$^{-1}$; $^1$H NMR (d$_6$-DMSO, 400 MHz) $\delta$: 4.42 (s, 1H, CH), 7.21–8.71 (m, 10H, Ar, NH$_2$) ppm; $^{13}$C NMR (d$_6$-DMSO, 100 MHz), $\delta$: 57.81, 104.3, 114.3, 116.2, 119.2, 122.9, 125.1, 127.0, 127.6, 127.8, 130.3, 133.8, 132.9, 146.2, 152.6, 155.4, 158.3, 158.4, 160.3 ppm.

2-amino-4,5-dihydro-4-(4-nitrophenyl)-5-oxopyrano[3,2-c]chromene-3-carbonitrile (4g)
IR (KBr) : 3367 (NH$_2$), 2887 (CH$_3$), 2162 (CN), 1708(C=O) cm$^{-1}$; $^1$H NMR (d$_6$-DMSO, 400 MHz) $\delta$: 3.75 (s CH$_3$) 4.42 (s, 1H, CH), 7.33–8.22 (m, 10H, Ar, NH$_2$) ppm; $^{13}$C NMR (d$_6$-DMSO, 100 MHz), $\delta$: 52.9, 57.6, 104.1, 113.1, 115.7, 116.9, 119.2, 122.9, 124.2, 124.2, 125.2, 126.7, 134.1, 138.1, 152.1, 152.5, 152.8, 159.5 ppm.

2-amino-4,5-dihydro-4-(4-methoxyphenyl)-5-oxopyrano[3,2-c]chromene-3-carbonitrile (4i)
IR (KBr) : 3342 (NH$_2$), 2159 (CN), 1707 (C=O) cm$^{-1}$; $^1$H NMR (d$_6$-DMSO, 400 MHz) $\delta$: 4.43 (s, 1H, CH), 7.21–8.71 (m, 10H, Ar, NH$_2$) ppm; $^{13}$C NMR (d$_6$-DMSO, 100 MHz), $\delta$: 57.81, 104.3, 114.3, 116.2, 119.2, 122.9, 125.1, 127.0, 127.6, 127.8, 130.3, 133.8, 132.9, 146.2, 152.6, 155.4, 158.3, 158.4, 160.3 ppm.

2-amino-4,5-dihydro-4-(3-nitrophenyl)-5-oxopyrano[3,2-c]chromene-3-carbonitrile (4j)
IR (KBr) : 3352 (NH$_2$), 2171 (CN), 1709 (C=O) cm$^{-1}$; $^1$H NMR (d$_6$-DMSO, 400 MHz) $\delta$: 4.41 (s, 1H, CH), 7.11–8.52 (m, 10H, Ar, NH$_2$) ppm; $^{13}$C NMR (d$_6$-DMSO, 100 MHz), $\delta$: 58.6, 104.3, 112.9, 118.0, 119.2, 122.3, 122.3, 122.9, 125.1, 129.8, 133.6, 135.2, 145.6, 148.3, 152.3, 153.9, 158.5, 158.7, 161.1 ppm.

2-amino-4,5-dihydro-4-(2-nitrophenyl)-5-oxopyrano[3,2-c]chromene-3-carbonitrile (4j)
IR (KBr) : 3352 (NH$_2$), 2171 (CN), 1709 (C=O) cm$^{-1}$; $^1$H NMR (d$_6$-DMSO, 400 MHz) $\delta$: 4.41 (s, 1H, CH), 7.21–8.52 (m, 10H, Ar, NH$_2$) ppm; $^{13}$C NMR (d$_6$-DMSO, 100 MHz), $\delta$: 57.3, 103.24, 119.3, 117.5, 118.9, 123.8, 124.6, 125.8, 129.7, 133.3, 147.3, 151.4, 152.4, 154.4, 158.5, 158.7, 161.2 ppm.
2-amino-4-(2,4-dichlorophenyl)-4,5-dihydro-5-oxopyrano[3,2-c]chromene-3-carbonitrile (4k)
IR (KBr): 3321 (NH₂), 2157 (CN), 1702 (C=O) cm⁻¹; ¹H NMR (d₆-DMSO, 400 MHz) δ : 4.44 (s, 1H, CH), 7.29–8.87 (m, 09H, Ar, NH₂) ppm; ¹³C NMR (d₆-DMSO, 100 MHz), δ : 34.4, 57.7, 102.9, 113.9, 117.2, 119.4, 123.2, 124.9, 128.6, 129.4, 133.1, 132.2, 133.6, 134.1, 139.1, 153.7, 154.6, 158.5, 160.4 ppm.

2-amino-4,5-dihydro-4-(3,4,5-trimethoxyphenyl)-5-oxopyrano[3,2-c]chromene-3-carbonitrile (4l)
IR (KBr): 3331 (NH₂), 2177 (CN), 1701 (C=O) cm⁻¹; ¹H NMR (d₆-DMSO, 400 MHz) δ : 3.64 (s, 3 H), 3.72 (s, 6 H), 4.43 (s, 1H, CH), 7.29–8.87 (m, 08H, Ar, NH₂) ppm; ¹³C NMR (d₆-DMSO, 100 MHz), δ : 56.36, 58.32, 60.39, 104.11, 105.38, 113.54, 117.05, 119.71, 123.04, 125.11, 133.39, 137.03, 139.46, 152.64, 153.30, 153.98, 158.38, 160.14.

2-amino-4-(4-fluorophenyl)-4,5-dihydro-5-oxopyrano[3,2-c]chromene-3-carbonitrile (4m)
IR (KBr): 3366 (NH₂), 2887 (CH₃), 2151 (CN), 1705 (C=O) cm⁻¹; ¹H NMR (d₆-DMSO, 400 MHz) δ : 4.42 (s, 1H, CH), 7.41–8.22 (m, 10H, Ar, NH₂) ppm; ¹³C NMR (d₆-DMSO, 100 MHz), δ : 52.9, 57.6, 104.1, 113.1, 115.7, 116.9, 119.2, 122.9, 124.2, 124.2, 125.2, 126.7, 134.1, 138.1, 152.1, 152.5, 158.2, 160.5 ppm.

2-amino-4-(4-(dimethylamino)phenyl)-4,5-dihydro-5-oxopyrano[3,2-c]chromene-3-carbonitrile (4n)
IR (KBr): 3234 (NH₂), 2187 (CN), 1702 (C=O) cm⁻¹; ¹HNMR (d₆-DMSO 400 MHz) δ : 231 (s CH₃) 4.68 (s, 1H, CH), 7.37–8.11 (m, 10H, Ar, NH₂) ppm. ¹³C NMR (d₆-DMSO, 100 MHz), δ : 34.6, 56.2, 103.2, 113.4 117.3, 119.3, 122.9, 124.2, 124.9, 125.2, 125.8, 129.2, 128.9, 132.3, 146.7, 151.2, 152.7, 154.4, 158.5, 160.1 ppm.

References


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