ONE-POT SYNTHESIS OF PHTHALAZIN-4-HETEROYL-4*H*-PYRAN DERIVATIVES IN [BMIM][OH]

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Keywords: Phthalic acid, ethyl cyanohydrazide, active methylene compounds, heteroaromatic aldehydes, [bmim][OH].

A [bmim][OH] mediated, green and efficient synthesis of phthalazines derivatives have been developed by condensing phthalic acid (**1**), ethyl cyanohydrazide (**2**), heteroaromatic aldehydes (**3a-3f**) and active methylene compounds (**4**) at 60-65 ºC for 60-90 min. The importance of this method includes shorter reaction time and high yield.

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INTRODUCTION

Nowadays ionic liquids (ILs) are used widely as reaction medium in organic chemistry. ILs have non-volatile nature at room temperature and have been used to develop eco-friendly methods for synthesis of organic compounds including heterocycles.[2-4] Multicomponent reactions (MCR) are one-pot reactions which contains three to more components in single reaction vessel to give a final desired product containing substantial components of all the reactants.[5] One of great challenges in modern medicinal chemistry is design and discovery of pharmaceutical active molecules.

Heterocyclic products which have nitrogen atom are well known and their use as pharmaceutical active compounds and agrochemicals are increasing.[6] Heterocyclic compounds having phthalazine moiety have received considerable attention because of their biological active properties and clinical applications.[7] Phthalazine compounds possess anti-convulsant,[8] cardiotonic,[9] and vasorelaxant,[10] properties. This has led to a large number of reports about the synthesis of phthalazine compounds.[11] Nevertheless the development of new synthetic methods for the efficient preparation of heterocycles containing phthalazine ring fragment is an interesting challenge.

Therefore, we decide to prepare phthalazine containg pyran moiety. Here, we report one-pot four component reaction for tiltle compounds in weakly basic 1-butyl-3-methylimidazolium hydroxide ([bmim][OH]) medium.

RESULTS AND DISCUSSION

First, we have studied a one pot, four-component reaction of phthalic acid (**1**) (1 mmol), ethyl cyanohydrazide[12] (**2**) (1 mmol), furfuraldehyde (**3a**) (1 mmol), and malononitrile (**4**) (1 mmol) in different ionic liquid medium at different temperatures to form 2-amino-6-(1,4-dioxo-3,4-dihydrophthalazin-2(1H)-yl)-4-(furan-2-yl)-4H-pyran-3,5-dicarbonitrile (**5a**) as a model reation. It was found that the reaction in the presence of [bmim][OH] as medium for 1 h at 60-65 ˚C gave the highest yield (88 %) and the clean product **5a** (Table 1, entry 1). Here, initially compound **1** was reacted with **2** in [bmim][OH] at 60-65 ˚C for 18 min to form 3-(1,4-dioxo-3,4-dihydrophthalazin-(1H)-yl)-3-oxo-propanenitrile as intermediate **6** (confirmed by TLC, showing the absence of starting materials). Then to this reaction mixture added **3a** and **4** and again heated at 60-65 ˚C for 1.5 h to form **5a** excellent yield (88 %) on simple work-up of the reaction mixture. The structure of the compound **5a** has been confirmed by ^{1}H NMR, IR and mass spectroscopy.

Table 1. Effect of ionic liquid and temperature on the synthesis of **5a**.

Entry	Ionic liquid	Temp., C ˚C	Time, h /h	Yield of, 5a, %
1	[bmim][OH]	60-65	1	88
6	[bmim][Br]	60-65	3	70
5	[DBUH][OAc]	60-65	2	83
4	[bmim][OH]	80-85	1	80
5	[[bmim][OH]	40-45	2	75
6	[bmim][OH]	30-35	6	70

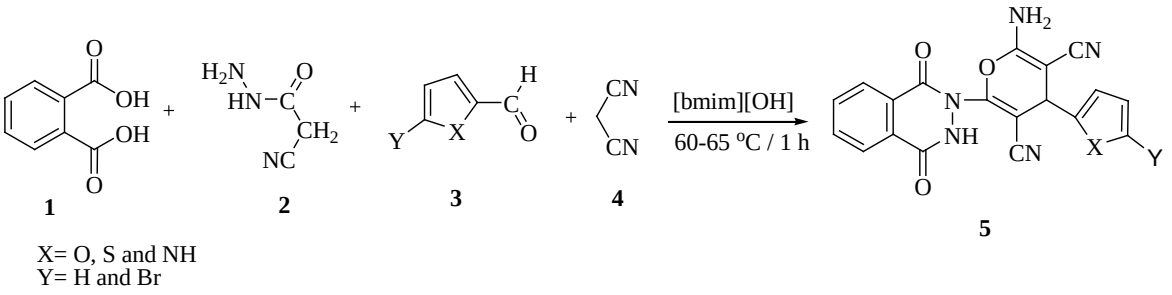
Table 2. The effect of amount of [bmim][OH] on the yield of **5a**.

Entry	mmol of [bmim][OH]	Time, h	Yield of 5a (%)
1	0.5	4	80
2	1	2	88
3	2	2	82

After finalizing the above optimization conditions, the one-pot reaction has been carried out at different temperature (room temperature, 40, 60 and 80 ˚C) in the presence of [bmim][OH] to get desired compound **5a**. It was found that the one-pot reaction of **1** (1 mmol), **2** (1 mmol), **3a** (1 mmol) and **4a** (1 mmol)] in the presence of [bmim][OH] as medium (1 mmol) for 120 min at 60-65 ˚C gave the highest yield (88 %) and the clean product **5a** (Table 1, entry 1). In order to examine effect of different quantity of [bmim][OH], the one-pot reaction has been carried out at different quantity (0.5, 1 and 2 mmol) of

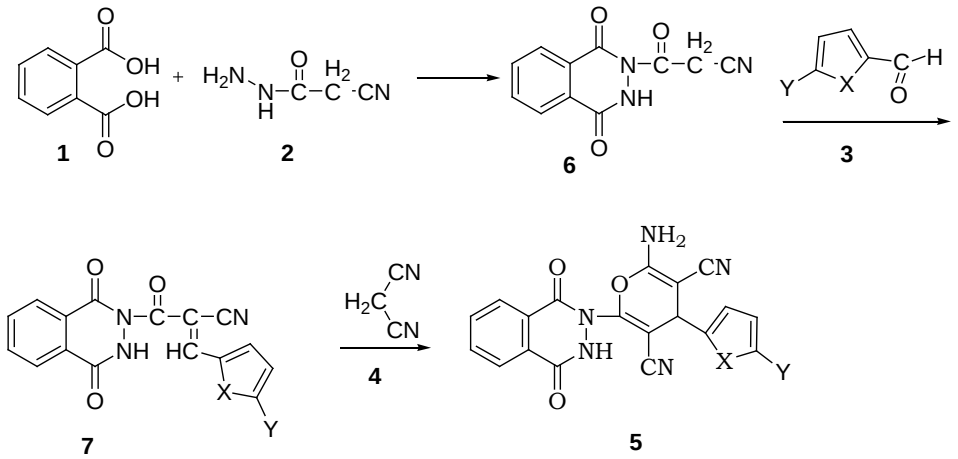
[bmim][OH] with respect of phthalic acid **1**. However, it was found that the one-pot reaction of **1** (1 mmol), **2** (1 mmol), **3a** (1 mmol) and 4a (1 mmol)] in the presence of [bmim][OH] (1 mmol) as medium for 2 h at 60-65 ˚C gave the highest yield (88 %) (Table 2, entry 2).

After having optimized the reaction conditions, the generality of the reaction was investigated by carrying out the condensation of several others heteroaromatic aldehydes (**3b-3f**) respectively in [bmim][OH] medium at 60-65 ˚C for 2 h giving **5b-5f** in very good yields and no side product formation was detected. It was found that this method works with a wide variety of substrates. It is worthy to mention that the reaction of **1, 2, 3a-3f** and **4** could get high yield and require short reaction time for formation of **5a-5f**. (Scheme 1).



1 **2** **3** **4** **5**

Scheme 1. One-pot sysnthesis of **5 a-f**.



1 **2** **6** **3** **7** **4** **5**

Scheme 2. Step-wise synthesis of **5**.

The synthesis of **5** could also be achieved in step-wise syntheses. Thus, a mixture of **1** and **2** was heated at 60-65 ºC for 0.5 h in [bmim][OH] medium to form intermediate **6**.[13] Then, **6** was reacted with **3** at 60-65 ˚C for 0.5 h in [bmim][OH] medium to form intermediate 7[13] then **7** was reacted with **4** at 60-65 ˚C for 0.5 h in [bmim][OH] medium to form **5**. The reaction was monitored by TLC.

The structures of these products have been established earlier on the basis of their spectral data (Scheme 2).

Furthermore, the compound **5** was assigned E-configuration on the presumption that bulky groups in a trans position would confer thermal stability on the molecule. This has been found to be case by a careful examination of the Frame-work molecular models of both E and Z-configurations of **5** wherein it was observed that there was minimum number of steric interactions in the E-configuration.

EXPERIMENTAL

Melting points are uncorrected and were determined in open capillary tubes in sulphuric acid bath. TLC was run on silica gel-G and visualization was done using iodine vapour or UV light. IR spectra were recorded using Perkin – Elmer 1000 instrument on KBr pellets. ^{1}H NMR spectra were recorded in DMSO-d_6 using TMS as internal standard at 400 MHz operating frequency. Mass spectra were recorded on Agilent-LCMS instrument. All reagents were purchased from Merck or Aldrich and used without further purification. [bmim][OH] was prepared as reported elsewhere.[13]

Preparation of 5a-5f from 1, 2, 3a-3f and 4

A mixture of **1** (1 mmol) and **2** (1 mmol) was heated at 60-65 ºC for 0.5 h in [bmim][OH] (1 mmol) for 0.5 h, when

no starting materials was detected by TLC. To this reaction mass added compounds **3** and **4** and again heated at 60-65 °C for 1 h, when no starting materials was detected by TLC. After the reaction was complete, cold water was added to the reaction mixture and solid part was separated by filtration. The product was recrystallized from ethanol to obtain **5**.

2-Amino-6-(1,4-dioxo-3,4-dihydrophthalazin-2(1H)-yl)-4-(furan-2-yl)-4H-pyran-3,5-dicarbonitrile (5a)

Yield 88 %, m.p. 206–208 °C. IR (KBr): 3302-3406 (br, medium, -NH-), 2211 (s, strong, -CN-), 1715 (s, strong, -CO- of amide group), 1655 (s, strong, -CO- of amide group) $cm^{-1.}$ ^{1}H NMR (DMSO-d_6, 400 MHz) δ = 6.2 (s, 1H, -CH), 7.0-8.2 (m, 7H, Ar-H), 9.6 (s, 2H, $–NH_2$), 11.4 (s, 1H, -NH, D_2O exchangeable). ^{13}C NMR (DMSO-d_6, 100 MHz) δ = 44.7, 74.6, 85.4, 113.7, 114.3, 123.6, 126.7, 127.8, 129.6, 131.8, 133.2, 137.1, 155.4, 157.5, 163.1, 163.8. $M^{+\cdot}$+1 = 374.

2-Amino-6-(1,4-dioxo-3,4-dihydrophthalazin-2(1H)-yl)-4-(thiophen-2-yl)-4H-pyran-3,5-dicarbonitrile (5b)

Yield 86 %, m.p. 182–190 °C; IR (KBr) : 3306-3401 cm^{-1} (br, medium, -NH-), 2218 cm^{-1} (s, strong, -CN-), 1706 cm^{-1} (s, strong, -CO- of amide group), 1659 cm^{-1} (s, strong, -CO- of amide group); ^{1}H NMR (DMSO-d_6, 400 MHz): δ 6.5 (s, 1H, -CH), 7.4-8.1 (m, 7H, Ar-H), 9.8 (s, 2H, $–NH_2$), 12.1 (s, 1H, -NH, D_2O exchangeable); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 49.5, 74.5, 87.3, 113.9, 115.1, 124.2, 127.8, 128.6, 129.1, 130.7, 134.3, 137.0, 155.8, 157.3, 163.0, 163.5; $M^{+\cdot}$+1 =390.

2-Amino-6-(1,4-dioxo-3,4-dihydrophthalazin-2(1H)-yl)-4-(1H-pyrrol-2-yl)-4H-pyran-3,5-dicarbonitrile (5c)

Yield 84 %, m.p. 193–195 °C. IR (KBr): 3303-3405 (br, medium, -NH-), 2217 (s, strong, -CN-), 1704 (s, strong, -CO- of amide group), 1658 (s, strong, -CO- of amide group) cm^{-1}. ^{1}HNMR (DMSO-d_6, 400 MHz) δ = 6.0 (s, 1H, -CH), 7.2-8.4 (m, 7H, Ar-H), 9.3 (s, 2H, $–NH_2$), 11.4 (s, 1H, -NH, D_2O exchangeable), 11.9 (s, 1H, -NH, D_2O exchangeable). ^{13}CNMR (DMSO-d_6, 100 MHz) δ = 47.5, 74.6, 86.3, 115.7, 116.2, 123.4, 126.7, 127.4, 128.8, 132.4, 133.2, 134.1, 154.5, 156.3, 163.1, 163.4. $M^{+\cdot}$+1 = 373.

2-Amino-4-(5-bromofuran-2-yl)-6-(1,4-dioxo-3,4-dihydrophthalazin-2(1H)-yl)-4H-pyran-3,5-dicarbonitrile (5d)

Yield 83 %, m.p. 211–212 °C. IR (KBr): 3305-3405 (br, medium, -NH-), 2215 (s, strong, -CN-), 1716 (s, strong, -CO- of amide group), 1658 (s, strong, -CO- of amide group) cm^{-1}. ^{1}H NMR (DMSO-d_6, 400 MHz) δ = 6.1 (s, 1H, -CH), 7.0-8.2 (m, 6H, Ar-H), 9.5 (s, 2H, $–NH_2$), 11.2 (s, 1H, -NH, D_2O exchangeable). ^{13}C NMR (DMSO-d_6, 100 MHz) δ = 44.9, 74.9, 85.8, 113.9, 114.9, 123.1, 126.2, 127.1, 129.2, 131.4, 133.5, 137.2, 155.2, 157.6, 163.2, 163.1. M^{+}+1 =451.

2-Amino-4-(5-bromothiophen-2-yl)-6-(1,4-dioxo-3,4-dihydrophthalazin-2(1H)-yl)-4H-pyran-3,5-dicarbonitrile (5e)

Yield 82 %, m.p. 192–194 °C. IR (KBr): 3302-3401 (br, medium, -NH-), 2213 (s, strong, -CN-), 1702 (s, strong, -CO- of amide group), 1655 (s, strong, -CO- of amide group) cm^{-1}. ^{1}H NMR (DMSO-d_6, 400 MHz) δ = 6.1 (s, 1H, -CH), 7.4-8.1 (m, 6H, Ar-H), 9.5 (s, 2H, $–NH_2$), 12.2 (s, 1H, -NH, D_2O exchangeable). ^{13}C NMR (DMSO-d_6, 100 MHz) δ = 49.1, 74.3, 87.2, 113.6, 115.2, 124.1, 127.4, 128.8, 129.2, 130.5, 134.8, 137.2, 155.6, 157.4, 163.1, 163.3. M^{+}+1 =469.

2-Amino-4-(5-bromo-1H-pyrrol-2-yl)-6-(1,4-dioxo-3,4-dihydrophthalazin-2(1H)-yl)-4H-pyran-3,5-dicarbonitrile (5f)

Yield 88 %, m.p. 186-188 °C. IR (KBr): 3301-3402 (br, medium, -NH-), 2214 (s, strong, -CN-), 1701 (s, strong, -CO- of amide group), 1659 (s, strong, -CO- of amide group) cm^{-1}. ^{1}H NMR (DMSO-d_6, 400 MHz) δ = 6.3 (s, 1H, -CH), 7.2-8.4 (m, 6H, Ar-H), 9.5 (s, 2H, $–NH_2$), 11.3 (s, 1H, -NH, D_2O exchangeable), 11.8 (s, 1H, -NH, D_2O exchangeable). ^{13}C NMR (DMSO-d_6, 100 MHz) δ = 47.3, 74.8, 86.2, 115.1, 116.1, 123.8, 126.8, 127.8, 128.9, 132.1, 133.3, 134.2, 154.3, 156.1, 163.2, 163.1. M^{+}+1 =452.

Preparation of 6 from 1 and 2 via stepwise reactions

A mixture of **1** and **2** was heated at 60-65 °C in [DBU][OAc] (1 mmol) for 0.5 h. After the completion of the reaction, as monitored by TLC, the reaction mixture was poured into ice-cold water. The precipitated product was filtered, washed with water, dried and recrystallized from ethanol. Yield 75 %, m.p. 152-154°C [lit.[13] m.p. 150-152 °C].

Preparation of compounds 7 from 6 and compounds 3

A mixture of **6** (1 mmol), **3a-3f** (1 mmol) and [DBU][OAc] (1 mmol) were heated at 60-65 °C for 0.5 h. After the completion of the reaction, as monitored by TLC, the reaction mixture was poured into ice-cold water. The precipitated product was filtered, washed with water, dried and recrystallized from ethanol. Yield ~ 86 %.

(E)-2-(1,4-Dioxo-1,2,3,4-tetrahydrophthalazine-2-carbonyl)-3-(furan-2-yl)acrylonitrile (7a)

M.p. 169-171 °C. IR (KBr): 3140-3438 (broad, medium, -NH-), 2258 (sharp, strong, -CN-), 1743 (sharp, strong, -CO-group), 1730 (sharp, strong, -CO- group), 1683 (sharp, strong, -CO- of amide group) cm^{-1}. ^{1}H NMR (DMSO-d_6, 400 MHz) δ = 6.8 (s, 1H, -CH), 7.5-8.2 (m, 7H, –ArH), 11.3 (s, 1H, -NH, D_2O exchangeable). ^{13}C NMR (DMSO-d_6, 100 MHz): δ 81.2, 81.2, 117.2, 122.4, 124.2, 127.4, 128.2, 128.6, 129.1, 129.1, 129.3, 133.1, 136.2, 164.3, 164.4, 164.8. $M^{+\cdot}$+1= 308.

(E)-2-(1,4-Dioxo-1,2,3,4-tetrahydrophthalazine-2-carbonyl)-3-(thiophen-2-yl)acrylonitrile (7b)

M.p. 160–162 °C. IR (KBr): 3294-3519 (broad, medium, -NH-), 2258 (sharp, strong, -CN-), 1794 (sharp, strong, -CO-group), 1748 (sharp, strong, -CO- of amide group),1682 (sharp, strong, -CO- of amide group) cm^{-1}. ^1H NMR (DMSO-d_6, 400 MHz) δ = 7.3 (s, 1H, -CH), 7.9-8.6 (m, 7H, –ArH), 11.3 (s, 1H, -NH, D_2O exchangeable). ^{13}C NMR (DMSO-d_6, 100 MHz): δ 81.3, 81.3, 117.2, 122.8, 124.0, 127.2, 128.2, 128.5, 129.0, 129.1, 129.5, 133.1, 136.0,164.4, 164.4, 164.7. $M^{+\cdot}$+1= 324.

(E)-2-(1,4-Dioxo-1,2,3,4-tetrahydrophthalazine-2-carbonyl)-3-(1H-pyrrol-2-yl)acrylonitrile (7c)

M.p. 169–171 °C. IR (KBr): 3046-3444 (broad, medium, -NH-), 2258 (sharp, strong, -CN-), 1773 (sharp, strong, -CO-group), 1730 (sharp, strong, -CO- group), 1673 (sharp, strong, -CO- of amide group) cm^{-1}. ^1H NMR (DMSO-d_6, 400 MHz) δ = 6.5 (s, 1H, -CH), 7.4-8.1 (m, 7H, –ArH), 11.1 (s, 1H, -NH, D_2O exchangeable), 12.1 (s, 1H, -NH, D_2O exchangeable). ^{13}C NMR (DMSO-d_6, 100 MHz): δ 81.0, 81.2, 117.1, 122.6, 124.5, 127.2, 128.2, 128.7, 129.1, 129.2, 129.3, 133.1, 136.2, 164.3, 164.6, 164.8. $M^{+\cdot}$+1= 307.

(E)-3-(5-Bromofuran-2-yl)-2-(1,4-dioxo-1,2,3,4-tetrahydro-phthalazine-2-carbonyl)acrylonitrile (7d)

M.p. 156–159 °C. IR (KBr): 3145-3432 (br, medium, -NH-), 2259 (s, strong, -CN-), 1748 (s, strong, -CO- group), 1735 (s, strong, -CO- group), 1685 (s, strong, -CO- of amide group) cm^{-1}. ^1H NMR (DMSO-d_6, 400 MHz) δ = 6.9 (s, 1H, -CH), 7.5-8.2 (m, 6H, –ArH), 11.8 (s, 1H, -NH, D_2O exchangeable) ^{13}C NMR (DMSO-d_6, 100 MHz): δ 81.1, 81.6, 117.1, 122.3, 124.8, 127.6, 128.3, 128.9, 129.3, 129.6, 129.9, 133.2, 136.4, 164.5, 164.8, 164.9. $M^{+\cdot}$+1= 387.

(E)-3-(5-Bromothiophen-2-yl)-2-(1,4-dioxo-1,2,3,4-tetrahydro-phthalazine-2-carbonyl)acrylonitrile (7e)

M.p. 165–167 °C. IR (KBr): 3295-3512 (br, medium, -NH-), 2245 (s, strong, -CN-), 1793 (s, strong, -CO- group), 1749 (s, strong, -CO- of amide group), 1680 (s, strong, -CO- of amide group) cm^{-1}. ^1H NMR (DMSO-d_6, 400 MHz) δ = 7.1 (s, 1H, -CH), 7.9-8.6 (m, 6H, –ArH), 11.2 (s, 1H, -NH, D_2O exchangeable). ^{13}C NMR (DMSO-d_6, 100 MHz): δ 81.1, 81.5, 117.1, 122.4, 124.2, 127.3, 128.1, 128.3, 129.1, 129.5, 129.6, 133.2, 136.1, 164.2, 164.6, 164.8. $M^{+\cdot}$+1= 403.

(E)-3-(5-Bromo-1H-pyrrol-2-yl)-2-(1,4-dioxo-1,2,3,4-tetrahydrophthalazine-2-carbonyl) acrylonitrile (7f)

M.p. 171–173 °C. IR (KBr): 3048-3440 (br, medium, -NH-), 2252 (s, strong, -CN-), 1775 (s, strong, -CO- group), 1731 (s, strong, -CO- group), 1675 (s, strong, -CO- of amide group) cm^{-1}. ^1H NMR (DMSO-d_6, 400 MHz): δ 6.3 (s, 1H, -CH), 7.4-8.1 (m, 6H, –ArH), 11.4 (s, 1H, -NH, D_2O exchangeable), 12.2 (s, 1H, -NH, D_2O exchangeable). ^{13}C NMR (DMSO-d_6, 100 MHz): δ 81.3, 81.5, 117.4, 122.8, 124.8, 127.1, 128.5, 128.8, 129.2, 129.4, 129.8, 133.2, 136.3, 164.5, 164.6, 164.8. $M^{+\cdot}$+1= 386.

Preparation of compounds 5 from 7 and 4

A mixture of **7** (1 mmol), **4** (1 mmol) and [DBU] [OAc] (1 mmol) were heated at 60-65 °C for 1.0 h. After the completion of the reaction, as monitored by TLC, the reaction mixture was poured into ice-cold water. The precipitated product was filtered, washed with water, dried and recrystallized from ethanol to form **5**. Yield ~ 80%.

CONCLUSION

In summary, we have successfully devised a simple and green one pot as well as step-wise and tandem process for synthesis of novel 2-amino–6(1,4-dioxo-3,4-dihydrphthalazin-2(1*H*)-yl-4-heteroyl-4*H*-pyran-3,5-dicar-bonitriles with simple work up procedures.

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