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ONE-POT FOUR-COMPONENT SYNTHESIS OF 1,4-DIHYDROPYRANO[2,3-*C*]PYRAZOLE-5-CARBONITRILES CATALYZED BY POTASSIUM PHTHALIMIDE

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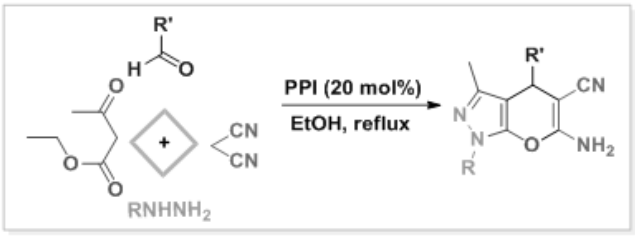
The potassium phthalimide (PPI) promoted a domino Knoevenagel-Michael-cyclocondensation of a wide variety of aldehydes, ethyl acetoacetate, malononitrile, and hydrazine derivatives to afford the 1,4-dihydropyrano[2,3-*c*]pyrazole-5-carbonitriles in high yields. The reactions were performed in refluxing ethanol at shorter reaction times. The significant advantages of this reaction include one-pot process, commercially accessible organocatalyst, simple work-up procedure, reusability of the catalyst, low cost, the use of ethanol as a green solvent, and environmentally friendly solid basic catalyst.



INTRODUCTION

The multicomponent reactions (MCRs) play a pivotal role in the achievement of many useful organic compounds such as 1,4-dihydropyrano[2,3-c]pyrazoles without the isolation of intermediates with economic viability. Compared with the conventional organic transformations, MCRs are usually associated with a number of benefits such as shorter reaction times, operational simplicity, avoidance of time-consuming, energy saving, green bond-forming efficiency, atomic and structural economy, access to a library of organic compounds, and avoidance of the complicated separation and purification processes.[1,2] So far, a wide range of biological effects, including antimicrobial, analgesic, vasodilator, anticancer, anti-inflammatory, human Chk1 kinase inhibitory, molluscicidal, and anti-fungicidal have been described for the 1,4-dihydropyrano[2,3-c]pyrazole compounds.

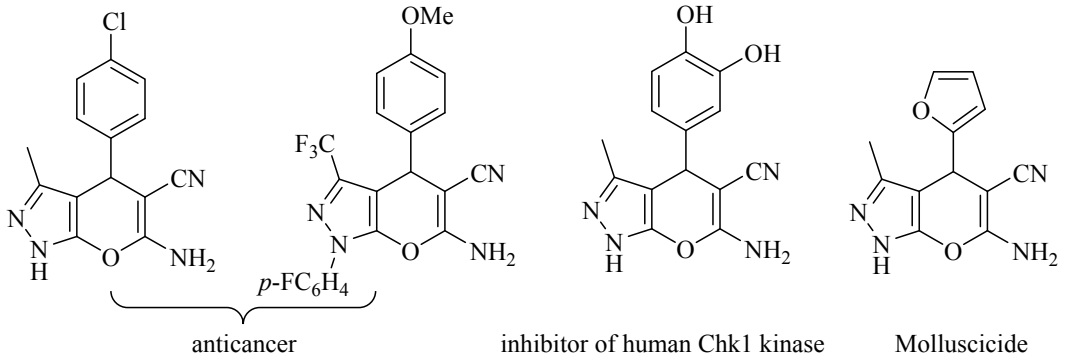


Figure 1

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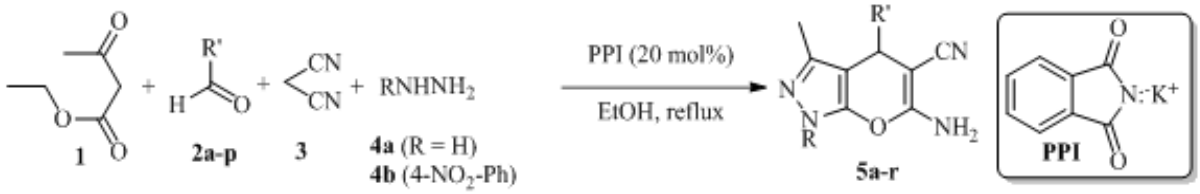

Scheme 1

Examples of biologically active 1,4-dihydropyrano[2,3-*c*]pyrazoles have been shown in Fig. 1.[2-4]

Because of the presence of the 1,4-dihydropyrano[2,3-c]pyrazole scaffold in several biologically active compounds, new approaches in the direction of the synthesis of highly functionalized pyrano[2,3-c]pyrazoles are of great attention. Typically, processes involved in the synthesis of these fused heterocyclic compounds are catalyzed by diverse conditions and catalysts. Some of the newer catalysts include β-cyclodextrin (β-CD),[5] cocamidopropyl betaine (CAPB),[6] lipase,[7] urea,[8] ZrO_2 nanoparticles,[9] cetyltrimethylammonium chloride (CTACl),[10] NaOH,[11] poly(4-vinylpyridine),[12, 13] CuO-CeO_2,[14] meglumine,[15] tetraethylammonium bromide (TEABr),[16] silica-bonded *N*-propylpiperazine sodium *n*-propionate (SBPPSP),[17] nickel nanoparticles,[18] *β*-cyclodextrin-SO_3H,[19] silica sodium carbonate (SSC)[20] NaOAc,[21] silica-supported tetramethylguanidine (SiO_2TMG),[22] SnO_2 quantum dots,[23] (S)-proline,[24] methyltriphenylphosphonium bromide,[25] iodine,[26] tungstate sulfuric acid,[27] molecular sieves,[28] isonicotinic acid,[29] DABCO,[30] phase transfer catalyst,[31] thiourea dioxide (TUD),[32] *p*-TsOH,[33] phenylboronic acid,[34] and starch-sulfuric acid (SSA).[35] We have also recently published an article regarding synthesis of pyrano[2,3-c]pyrazoles using sodium benzoate as the catalyst.[36]

Potassium phthalimide (PPI), on the other hand, is a stable, mild, green, inexpensive, commercially available, and efficient basic recyclable organocatalyst. Literature survey shows that there are no reports about the synthesis of pyrano[2,3-c]pyrazole heterocycles using PPI as the catalyst. This report describes the one-pot, four-component synthesis of several derivatives of 1,4-dihydropyrano[2,3-c]pyrazoles via the reaction of ethyl acetoacetate (**1**), aldehydes (**2a-p**), malononitrile (**3**), and hydrazine derivatives (**4a-b**) in the presence of PPI (Scheme 1). This is the first attempt toward the synthesis of 6-amino-3-methyl-4-substituted-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile derivatives (**5a-r**) using PPI as the catalyst.

RESULTS AND DISCUSSION

To find out the optimization of the reaction conditions, we studied the effect of different solvents and amounts of the catalyst PPI for the four-component reaction (4CR) of ethyl acetoacetate (**1**), vanillin (**2k**), malononitrile (**3**), and hydrazine monohydrate (**4a**) as the model reaction (Table 1).

Table 1

Optimization of the reaction conditions for the synthesis 6-amino-4-(4-hydroxy-3-methoxyphenyl)-3-methyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile (**5k**) (model reaction)[a]

Entry	Solvent	Catalyst (mol%)	Time (min)	Isolated Yields (%)[b]
1[c]	EtOH	-	60	0
2[d]	H_2O	-	60	Trace
3[e]	H_2O	PPI (5)	15	65
4	H_2O	PPI (10)	10	82
5	H_2O	PPI (15)	8	89
6	H_2O	PPI (20)	6	90
7	EtOH	PPI (10)	6	70
8	EtOH	PPI (15)	5	85
9	**EtOH**	**PPI (20)**	**5**	**96**
10	EtOH	PPI (25)	5	94
11	CH_2Cl_2	PPI (20)	12	72
12	EtOAc	PPI (20)	30	41
13	-[f]	PPI (20)	22	58
14[g]	None	PPI (20)	40	65
15	EtOH	Sodium sulfide (20)	40	70

Table 1 (continued)

16	EtOH	Sodium tetraborate (15)	40	65
17	EtOH	KHP[h] (20)	40	72
18	EtOH	Sodium azide (20)	40	70
19	EtOH	TBAP[i] (20)	40	62
20	EtOH	Sulfanilic acid (15)	40	74

[a] All Reaction conditions: Ethyl acetoacetate **1** (1 mmol), vanillin **2k** (1 mmol), malononitrile **3** (1 mmol), hydrazine monohydrate **4** (1.2 mmol), and the solvent (5 mL) at reflux.
[b] All yields refer to isolated yields.
[c] The reaction was checked at r.t., 50, 80, 100, and 120 °C and the product not formed.
[d] The reaction was implemented at reflux.
[e] Potassium phthalimide.
[f] H_2O-EtOH (1:1, v/v).
[g] The reaction was implemented at 100 °C.
[h] Potassium hydrogen phthalate.
[i] Tetrabutylammonium perchlorate.
The optimal conditions are specified in bold.

As shown in Table 1, in the absence of any catalyst and solvent at different temperatures, the formation of product (**5k**) was not observed (Table 1, entry 1). When the model reaction was conducted in water only a trace amount of **5k** was detected after 1 h (Table 1, entry 2). By adding PPI catalyst (5 mol %) to the reaction mixture, the product was formed in 15 min with 65 % isolated yield (Table 1, entry 3). Prolonging the reaction time had no effect on the improvement of the yield. The yield of product **5k** in water was improved as the amount of the catalyst increased from 5 to 10, 15, and 20 mol% (Table 1, entries 4-6). These results indicate that the catalyst has a significant effect on the reaction time and yield. The better yields in shorter reaction times was obtained in comparison with water when the same reaction was performed using various amounts of PPI (10, 15, and 20 mol%) in refluxing ethanol (Table 1, entries 7-9). An increase in the amount of PPI did not result in an improvement in the yield (Table 1, entry 10). Moreover, when this four-component reaction (4CR) was implemented in CH_2Cl_2, EtOAc, and aqueous ethanol (1:1, v/v), the corresponding product (**5k**) was achieved in the yields of 72%, 41%, and 58%, respectively (Table 1, entries 11-13). The reaction was also carried out in the solvent-free conditions in the presence of 20 mol% of the catalyst; and the **5k** was obtained with the 65% yield at longer reaction time (Table 1, entry 14). Then, the model reaction was tested in the presence of catalytic amounts of several available compounds such as sodium sulfide, sodium tetraborate, potassium hydrogen phthalate (KHP), sodium azide, tetrabutylammonium perchlorate (TBAP), and sulfanilic acid in refluxing ethanol (Table 1, entries 15-20) but these compounds could not promote the reaction efficiently even at prolonged reaction times and yields were not satisfactory compared with the PPI. Based on the above results, 20 mol% of PPI and refluxing ethanol was chosen as the best reaction conditions for this 4CR (Table 1, entry 9). To search the scope of the PPI towards the synthesis a wide variety of 6-amino-3-methyl-4-substituted-1,4-dihydropyrano[2,3-*c*]pyrazole-5-carbonitriles, available aryl aldehydes were tested in this 4CR. It was found that the reaction proceeded efficiently and afforded the targeted products (**5a-r**) in good to high yields. The results were listed in Table 2. All the products synthesized in this 4CR are precipitated in the reaction vessel after cooling. The targeted compounds were then obtained in high yields without any chromatographic purification methods.

Table 2

Synthesis of 1,4-dihydropyrano[2,3-c]pyrazoles (**5a-r**)[a]

Entry	R'/R	Product	Time (min)	Isolated yields (%)	Mp (°C)	
					Found	Reported [Ref.]
1	C_6H_5 (**2a**)/H (**4a**)	**5a**	22	90	242-244	244-246[6]
2	4-Me-C_6H_4 (**2b**)/H (**4a**)	**5b**	20	94	204-206	205-208[6]
3	4-MeO-C_6H_4 (**2c**)/H (**4a**)	**5c**	20	87	206-207	208-211[31]

Table 2 (continued)

4	4-HO-C_6H_4 (**2d**)/H (**4a**)	**5d**	20	86	225-227	225-226[31]
5	4- Me_2N-C_6H_4 (**2e**)/H (**4a**)	**5e**	25	85	216-218	217-219[31]
6	4-NO_2-C_6H_4 (**2f**)/H (**4a**)	**5f**	15	95	249-250	248-250[6]
7	3-NO_2-C_6H_4 (**2g**)/H (**4a**)	**5g**	18	90	235-236	235-236[31]
8	2-NO_2-C_6H_4 (**2h**) /H (**4a**)	**5h**	18	86	223-225	221-225[6]
9	4-Cl-C_6H_4 (**2i**) /H (**4a**)	**5i**	15	88	230-232	234-236[6]
10	2-Cl-C_6H_4 (**2j**) /H (**4a**)	**5j**	20	84	247-248	246-248[31]
11	4-HO-3-MeO-C_6H_3 (**2k**)/H (**4a**)	**5k**	5	96	238-239	235-238[28]
12	3,4-di-MeO-C_6H_3 (**2l**)/H (**4a**)	**5l**	25	88	189-191	188-191[28]
13	2,4-di-Cl-C_6H_4 (**2m**) /H (**4a**)	**5m**	15	82	236-238	237-238[6]
14	2-Thienyl (**2n**) /H (**4a**)	**5n**	25	90	192-194	192-194[22]
15	Butyraldehyde (**2o**)/H (**4a**)	**5o**	40	80	144-145	143-144[22]
16	Isobutyraldehyde (**2p**)/H (**4a**)	**5p**	35	82	181-183	182-183[18]
17	C_6H_5 (**2a**)/*p*-NO_2-Ph (**4b**)	**5q**	15	89	214-216	215-216[29]
18	4-Cl-C_6H_4 (**2i**)/*p*-NO_2-Ph (**4b**)	**5r**	18	91	237-239	244-246[6]

[a] Reaction conditions: substrates; ethanol (5 mL); PPI (20 mol%); stirred at reflux.

Based on the results presented in Table 2, it can be seen that all reactions underwent clearly furnishing 6-amino-3-methyl-4-substituted-1,4-dihydropyrano[2,3-*c*]pyrazole-5-carbonitriles (**5a-r**) in high yields with relatively shorter reaction times. A wide range of substituted benzaldehydes bearing electron-rich substituents such as *p*-methyl, *p*-methoxy, *p*-hydroxy, *p*-*N,N*-dimethylamino, and 3,4-dimethoxy (Table 2, entries 2-5 and 12) and electron-deficient groups (NO_2 and Cl) at ortho, para, and meta positions (Table 2, entries 6-10 and 13) underwent successful 4CR with ethyl acetoacetate (**1**), malononitrile (**3**), and hydrazine monohydrate (**4a**) in refluxing ethanol. It seems that the electronic effects and the nature of the substituents on the benzaldehyde ring have slight effect on the reaction. This reaction was also affected by steric effect. For example, 2-nitrobenzaldehyd (Table 2, entry 8) and 2-chlorobenzaldehyde (Table 2, entry 10) required longer reaction times compared to 4-nitrobenzaldeyde (Table 2, entry 6) and 4-chlorobenzaldehyde (Table 2, entry 9), which is probably related to sterically hindered effects of *ortho* position.

The reaction of ethyl acetoacetate (**1**), malononitrile (**3**), hydrazine monohydrate (**4a**), and various cyclic ketones such as cyclohexanone and cyclopentanone was also performed under the same optimal reaction conditions, however, the corresponding 1,4-dihydropyrano[2,3-c]pyrazole products were formed in low yields. In addition, the reaction using substituted hydrazines like phenyl hydrazine and methyl hydrazine was implemented and lead to product formation in 20-30 % yield after 30 h. When ethyl benzoylacetate was used instead of ethyl acetoacetate, the product was formed in trace yields.

On the other hand, the reaction using hetero-aromatic aldehydes such as thiophene-2-carbaldehyde leads to the corresponding product in high yield (Table 2, entry 14). Using aliphatic aldehydes such as butyraldehyde (**2o**) and isobutyraldehyde (**2p**) afforded pyranopyrazole products in 80% and 82% yield, respectively (Table 2, entries 15 and 16). In another investigation, the condensation of ethyl acetoacetate (**1**), malononitrile (**3**), 4-nitrophenyl hydrazine (**4b**), and arylaldehydes **2a** and **2i** using PPI was investigated. In this case, corresponding 1,4-dihydropyrano[2,3-*c*]pyrazole-5-carbonitriles (**5q-r**) were obtained in reasonable yields and reaction times (Table 2, entries 17 and 18).

In another attempt, the reusability of the catalyst was also examined on the model reaction. After the completion of the reaction, the solid product was treated with water and filtered off. The catalyst was recovered after the removal of solvent from the filtrate and reused for four subsequent cycles (Table 3). It showed nearly the same activity as a fresh catalyst along but with a slight decrease of yield.

Table 3

The Reusability PPI in refluxing ethanol for the synthesis of **5k**[a]

No. of recycles	Fresh	Run 1	Run 2	Run 3	Run 4
Time (min.)	5	5	6	8	10
Isolated yields (%)	96	94	91	87	80

[a] The reaction conditions are similar to the optimized conditions described for the model reaction.

Table 4

Comparison of the results of PPI catalyst with various reported catalysts for the synthesis of **5b**

Entry	Catalyst (mol%) [mg]/conditions	Time (min.)	Yield (%)[Ref.]
1	*β*-Cyclodextrin (10)/H_2O-EtOH, 80 °C	30	86[5]
2	Lipase [20]/EtOH, 30 °C	60	97[7]
3	CTACl (20)/EtOH, 90 °C	250	82[10]
4	NaOH (5)/H_2O, 100 °C	60	93[11]
5	Meglumine (10)/H_2O-EtOH, r.t.	17	90[14]
6	TEABr (10)/H_2O, reflux	10	88[15]
7	Molecular sieves [100]/US, EtOH, reflux	35	88[28]
8	PPI (20)/EtOH, reflux	20	94[Current work]

CTACl: cetyltrimethylammonium chloride; US: ultrasound, TEABr: tetraethylammonium bromide.

To compare the effectiveness of PPI with other catalysts in the synthesis of 6-amino-3-methyl-4-(*p*-tolyl)-1,4-dihydropyrano[2,3-*c*]pyrazole-5-carbonitrile (**5b**), results of the 4CR of ethyl acetoacetate (**1**), 4-methylbenzaldehyde (**2b**), malononitrile (**3**), and hydrazine monohydrate (**4a**) have presented in Table 4. The results clearly show that the present procedure is better than the others in terms of reaction time and product yield.

EXPERIMENTAL

All the reagents were obtained from commercial sources and used without further purification. Melting points were measured on a Büchi 510 melting point apparatus and are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded at ambient temperature on a BRUKER AVANCE DRX-400 MHz spectrophotometer using DMSO-d_6 as the solvent. The purity of synthesized compounds as well as the progress of the reactions was monitored by thin layer chromatography (TLC) analysis on Merck pre-coated silica gel 60 F_{254} aluminum sheets, visualized by UV light. Elemental microanalyses were performed on an Elementar Vario EL III analyzer. All of the targeted products are reported in the literature and are characterized by comparison of their spectral and physical data on the basis of literature descriptions.

General procedure for the synthesis of 6-amino-3-methyl-4-substituted-1,4-dihydropyrano[2,3-*c*]pyrazole-5-carbonitrile derivatives (5a-r)

To a mixture of ethyl acetoacetate (**1**, 1 mmol), aryl aldehyde (**2**, 1 mmol), malononitrile (**3**, 1 mmol), hydrazine monohydrate derivatives (**4**, 1.2 mmol), and 20 mol% of PPI was stirred in refluxing ethanol (5 mL). The progress of the reaction was monitored by TLC analysis. After the completion of the reaction, the system was cooled to room temperature. Water (10 mL) was added to the reaction mixture, and the crude product was obtained by filtration followed by washing with aqueous ethanol. The pure targeted products thus obtained after recrystallization from ethanol. The catalyst was recovered by evaporation of the solvent from the filtrate, dried and reused for subsequent reactions. Data for representative compounds **5c** and **5f** were as follows:

6-Amino-4-(4-methoxyphenyl)-3-methyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile (**5c**).
IR (KBr, cm^{-1}): ν = 3445, 3331, 3070, 2195, 1648, 1608, 1580, 1515, 1425, 1135, 1144, 1058, 1030, 755; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 1.77 (s, 3H, CH_3), 3.68 (s, 3H, OCH_3), 4.54 (s, 1H, CH), 6.78 (s, br, 2H, NH_2), 6.84 (d, 2H, *J* = 8.4 Hz, ArH), 7.05 (d, 2H, *J* = 8.5 Hz, ArH), 12.03 (s, 1H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 11.2, 25.4, 56.4, 62.0, 114.3, 114.8, 127.4, 128.5, 141.0, 144.3, 151.9, 158.6, 165.2. Anal. Calcd for $C_{15}H_{14}N_4O_2$ (%): C, 63.82; H, 5.00; N, 19.85. Found: C, 63.79; H, 4.95; N, 19.81.

6-Amino-3-methyl-4-(4-nitrophenyl)-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile (**5f**).
IR (KBr, cm^{-1}): ν = 3405, 3370, 3314, 3080, 2195, 1660, 1610, 1585, 1520, 1440, 1395, 1335, 1230, 1170, 1140, 1060, 1025, 750; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 1.80 (s, 3H, CH_3), 4.81 (s, 1H, CH), 7.04 (s, br, 2H, NH_2), 7.47 (d, 2H, *J* = 8.4 Hz, ArH), 8.20 (d, 2H, *J* = 8.4 Hz, ArH), 12.16 (s, 1H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 10.1, 34.1, 61.0, 98.6, 120.7, 124.1, 129.2, 135.9, 146.6, 149.2, 151.7, 161.4. Anal. Calcd for $C_{14}H_{11}N_5O_3$ (%): C, 56.57; H, 3.73; N, 23.56. Found: C, 56.53; H, 3.75; N, 23.43.

CONCLUSIONS

In conclusion, this 4CR giving high yield of 6-amino-4-substituted-3-methyl-1,4-dihydropyrano[2,3-*c*]pyrazole-5-carbonitriles in relatively shorter reaction times. The one-pot four-component procedure is relatively quick, easy,

efficient, and offers an alternative method for the synthesis of 1,4-dihydropyrano[2,3-*c*]pyrazole heterocycles. Other obvious features of this method include atom-economy, eco-friendliness, cost-effectiveness, and no chromatographic purification.

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