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Potassium phthalimide: an efficient and simple organocatalyst for the one-pot synthesis of dihydropyrano[3,2-c]chromenes in aqueous media

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Abstract A series of dihydropyrano[3,2-c]chromene derivatives were obtained via treating cyano methylene compounds (malononitrile or ethyl cyanoacetate), aromatic aldehydes, and 4-hydroxycoumarin in the presence of potassium phthalimide, as a green, mild, efficient, and commercially available organocatalyst in aqueous media. This procedure was easily performed, which provides a straightforward route for the synthesis of diverse dihydropyrano[3,2-c]chromene compounds. Safety, short reaction times, environmentally friendly, no use of hazardous organic solvents, and high yields are remarkable advantages of this process.

Keywords Three-component process · Dihydropyrano[3,2-c]chromenes · Potassium phthalimide · Cyano compounds

Introduction

Since the first multicomponent process (MCP) was described by Strecker in 1850 [1], MCPs have been demonstrated to be highly valuable tools for the expedient creation of numerous chemical compounds including natural products and biologically active compounds [2]. Recently, considerable attention has been focused on MCPs owing to their high efficacy, mild conditions, simplistic completing, environmental friendliness, and minimal reaction times [3–6]. In addition to this, the other features of MCPs are the following: (1) simple procedures for the formation of final products in a one-pot process from at least three starting materials, (2) green bond-forming, (3) atomic and structural economy, (4) minimization of waste produced, (5) easy construction of complex organic molecules, and (6) avoidance of complicated purification processes [4–10].

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Carrying out MCPs in water as the reaction medium is one of the most suitable methods, which will be a significant component of green chemistry [11–15].

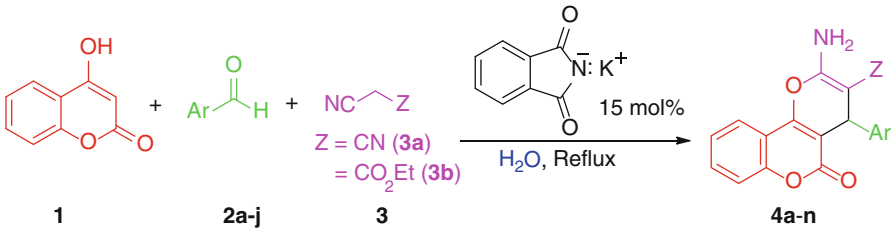
On the other hand, the heterocyclic scaffold comprising chromene moiety is present in compounds possessing a variety of biologically activities, such as spasmolytic, diuretic, anticoagulant, antianaphylactic [16], antimicrobial [17], antifungal [18], antibacterial [19], antioxidant [20], antileishmanial [21], antitumor [22], local anesthetic [23], antihistaminic [24], antiallergenic [25], and treatment of Alzheimer's disease [26] and schizophrenia [27]. Moreover, a number of chromenes are widely employed as pigments [28], cosmetics [29], and laser dyes [30].

Taking into consideration the above-mentioned characteristics of chromene derivatives, significant attention has been focused on the advance of ecologically benign methodologies towards synthesizing this heterocyclic (chromene) scaffold by cyclization of 4-hydroxycoumarin, aldehydes, and cyano compounds (i.e., malononitrile and ethyl cyanoacetate).

Several synthetic routes have been widely used for the preparation of dihydropyrano[3,2-c]chromenes. These compounds have been synthesized in the presence of a variety of homogeneous or heterogeneous catalysts such as hexamethylenetetramine (HMT) [31], heteropolyacid [32, 33], diammonium hydrogen phosphate [34], DBU [35], piperidine [36], tetrabutylammonium bromide (TBAB) [37], ionic liquid [38, 39], morpholine [40], 4-(dimethylamino)pyridine (DMAP) [41], potassium phthalimide-*N*-oxyl (POPINO) [42], ruthenium complexes [43], 3-hydroxypropanaminium acetate (HPAA) [44], silica-bonded *N*-propylpiperazine sodium *n*-propionate (SBPPSP) [45], silica gel [46], sulfonic acid functionalized silica [47], 2-hydroxyethanaminium acetate [48], α-Fe_2O_3 [49], cellulose-SO_3H [50], potassium sodium tartrate ($KNaC_4H_4O_6{\cdot}4H_2O$) [51], and meglumine [52].

Many of the procedures have their own merits. However, most of them suffer from one or more drawbacks, including complex steps, prolonged reaction times, low yields, tedious work-up procedures, and using expensive catalysts and organic solvents. Therefore, the development of an efficient, rapid, simple, and green procedure for the preparation of pyranochromenes is of considerable interest. Water is one of the best solvents in organic transformations and has received great attention due to its features such as being environmentally friendly, safe, non-toxic, non-flammable, clean, green, inexpensive, and readily available. Also, the use of water not only diminishes the risk of organic solvents but also improves the rate of many chemical reactions [53–55].

The development of solid basic catalytic systems utilizing inexpensive, clean, environmentally benign, and commercially available catalysts has been a challenge in organic synthesis [56, 57]. Potassium phthalimide (PPI) is a mild, green, inexpensive, efficient, basic recyclable catalyst and stable reagent. It has been utilized as a reagent in the synthesis of primary amines by the Gabriel method [58, 59], the synthesis of phthalimide derivatives [60–62], and as a catalyst for the preparation of cyanohydrin trimethylsilyl ethers [63, 64]. We have recently reported that PPI is an efficient and green organocatalyst for the synthesis of isoxazol-5(4*H*)-ones [65] and 4-aryl-7-(arylmethylene)-3,4,6,7-tetrahydro-1*H*-cyclopenta[*d*]pyrimidin-2(5*H*)-ones-/thiones [66]. Our literature survey revealed that there is no report



Scheme 1 Preparation of dihydropyrano[3,2-c]chromene derivatives (**4a**–**n**) via one-pot, three-component process of 4-hydroxycoumarin (**1**), aryl aldehydes (**2**), and cyano compounds (**3a**, **b**) catalyzed by PPI in water

on the use of PPI as a catalyst in the synthesis of dihydropyrano[3,2-c]chromene derivatives. For the first time, we report the applicability of PPI as a readily available, efficient, and solid basic organocatalyst for the synthesis of a wide variety of dihydropyrano[3,2-c]chromenes via the one-pot three-component process (Scheme 1).

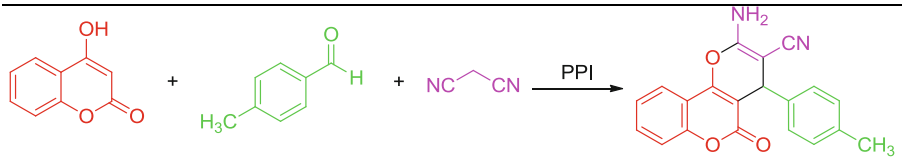
Experimental

All chemicals, unless otherwise specified, were purchased from commercial sources and were used without further purification, with the exception of furan-2-carbaldehyde and benzaldehyde, which were distilled before use. The products were characterized by a comparison of their physical data with those of known samples or by their spectral data. Melting points were measured on a Buchi 510 melting point apparatus and are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded at ambient temperature using a BRUKER AVANCE DRX-500 and 400 MHz using DMSO-d_6 as solvent. FT-IR spectra were recorded on a Perkin Elmer RXI spectrometer. The development of reactions was monitored by thin layer chromatography (TLC) analysis on Merck pre-coated silica gel 60 F_{254} aluminum sheets, visualized by UV light.

General procedure for the synthesis of dihydropyrano[3,2-c]chromene derivatives (**4a**–**n**)

A reaction mixture of aromatic aldehyde **2** (1 mmol), 4-hydroxycoumarin **1** (1 mmol), malononitrile or ethyl cyanoacetate **3** (1 mmol), and PPI (15 mol%) in distilled water (5 mL) was refluxed for 10–40 min. During the reflux, the progress of the reaction mixture was monitored by TLC analysis. After completion of the reaction, the reaction mixture was cooled to room temperature and the precipitated solid was filtered, washed with cold distilled water (4 mL), and air-dried to obtain the pure products. If necessary, the solid products can be recrystallized from hot ethanol. After removal of the water from the filtered solution, the catalyst is recovered and used for the subsequent reaction.

Table 1 Optimization reaction conditions for the one-pot, three-component synthesis of 2-amino-5-oxo-4-(*p*-tolyl)-4,5-dihydropyrano[3,2-c]chromene-3-carbon-itri-l-e (**4f**)



1 **2f** **3a** **4f**

Entry	Solvent	Amounts of catalyst (mol%)	Time (min)	Temp. (°C)	Yield (%)[a]
1	H_2O	–	30	r.t.	Trace
2	H_2O	–	30	Reflux	20
3	H_2O	5	30	r.t.	25
4	H_2O	10	30	r.t.	30
5	H_2O	15	30	r.t.	35
6	H_2O	20	30	r.t.	37
7	H_2O	5	25	Reflux	45
8	H_2O	10	20	Reflux	80
9	**H_2O**	**15**	**20**	**Reflux**	**94**
10	H_2O	20	20	Reflux	92
11	EtOH	15	20	Reflux	53
12	MeCN	15	20	Reflux	Trace
13	1,4-dioxane	15	20	Reflux	Trace
14	CH_2Cl_2	15	20	Reflux	Trace
15	EtOAc	15	20	Reflux	Trace
16	$CHCl_3$	15	20	Reflux	Trace
17	H_2O/EtOH[b]	15	20	Reflux	67

Reaction conditions: 4-hydroxycoumarin (1 mmol), 4-methylbenzaldehyde (1 mmol), malononitrile (1 mmol), solvent (5 mL)

Optimized conditions shown in bold

[a] Isolated yields

[b] Ratio solvent is 1:1 (v/v)

Results and discussion

In the beginning, we first performed the search for the optimization reaction conditions using the reaction between equimolar quantities (1 mmol) of 4-hydroxycoumarin (**1**), 4-methylbenzaldehyde (**2f**), and malononitrile (**3a**) as the model. Various reaction conditions were investigated, including solvents, amounts of catalyst, and temperatures. The results are summarized in Table 1.

As can be seen in Table 1, in free-catalyst conditions, the reaction did not proceed at room temperature. Only a trace amount of 2-amino-5-oxo-4-(*p*-tolyl)-4,5-dihydropyrano[3,2-c]chromene-3-carbon-itri-l-e (**4f**) was obtained in this case (Table 1, entry 1). Also, it was revealed that the reaction was rather slow and resulted in poor yield (20 %) in the absence of the catalyst when the reaction was

carried out in refluxing water for 30 min (Table 1, entry 2), which indicated that the catalyst was necessary for this transformation, albeit, increasing reaction time showed no significant change in the yield of the reaction. The yield also increased slightly by adding an amount of PPI to the reaction mixture at room temperature (Table 1, entry 3). The effect of catalyst loading on the completion of the reaction at room temperature was also studied. As can be seen in Table 1, when the amount of catalyst was increased to 20 mol% at room temperature, a slight improvement in the yield was achieved (Table 1, entries 4–6). In the next step, the reaction was carried out with various amounts of catalyst in water under reflux conditions. It was found that, by increasing the amount of catalyst from 5 to 15 mol% under reflux conditions, the yield of 2-amino-5-oxo-4-(*p*-tolyl)-4,5-dihydropyrano[3,2-c]chromene-3-carbonitrile (**4f**) was improved (Table 1, entries 7–9). A larger loading amount of the catalyst (20 mol%) neither increased the yield nor shortened the conversion time (Table 1, entry 10). Furthermore, the model reaction was implemented using the other bases such as Et_3N, NaOH, K_2CO_3, and Na_2CO_3. In the presence of these bases after 2 h, only a trace amount of product **4f** was observed. On the other hand, the EtOH, and a mixture of EtOH:H_2O (1:1, v/v) afforded a lower yield of the desired product **4f** under similar conditions (Table 1, entries 11, 17). Also, performing the reaction in the other organic solvents such as MeCN, 1,4-dioxane, CH_2Cl_2, EtOAc, and $CHCl_3$ le d to a trace amount of product **4f** (Table 1, entries 12–16). Hence, the 15 mol% catalyst loading and refluxing in water was considered to be the best conditions for this type of reaction (Table 1, entry 9, the optimum conditions shown in bold).

Using the optimized conditions (water, 15 mol% PPI, reflux), a series of products **4a**–**n** were synthesized with this simple reaction procedure. The reaction time and percentage yield for each of the products are presented in Table 2.

The results indicated that this PPI-catalyzed three-component reaction worked well for a wide range of aryl aldehydes possessing various functional groups including electron-donating and electron-withdrawing substituents. In all cases, the reaction proceeded smoothly. The electronic nature of the functional groups on the phenyl ring at C-4 position has no significant effect on the yield of products; however, the reaction times are affected. Substrates with electron-withdrawing groups (Table 2, entries 2–4, 12–13) reacted rapidly, whereas electron-donating groups (Table 2, entries 6–9 and 14) decreased the reactivity, requiring a longer reaction time. In addition to the aryl aldehydes, the reaction with electron-rich aromatic heterocyclic aldehydes such as furan-2-carbaldehyde was also smoothly developed and a high yield achieved (Table 2, entry 10). It should be noted that the purification of the title compounds is very easy. After cooling the reaction mixture to room temperature, the solid products were formed, which can be easily separated by simple filtering. The desired products are sufficiently pure. If further purification is required, they can be recrystallized from ethanol. It was found that this reaction involving less reactive ethyl cyanoacetate (Table 2, entries 12–14) gave corresponding products in lower yields and required longer reaction times than malononitrile, which may be ascribed to the competency of the cyanide group in stabilizing the reaction intermediates compared to the ester group [42, 67, 68].

Table 2 Synthesis of dihydropyrano[3,2-c]chromenes (**4**) using 4-hydroxycoumarin (**1**), aromatic aldehydes (**2**), and malononitrile (**3a**) or ethyl cyanoacetate (**3b**) catalyzed by PPI[a]

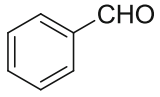
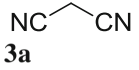
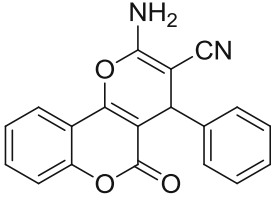
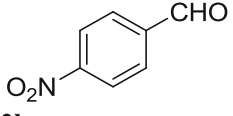
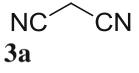
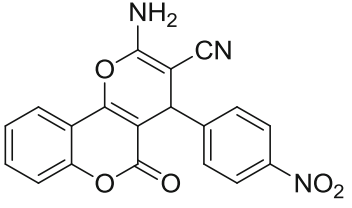
Entry	Aldehyde	Cyano compound	Product	Time (min)	Yield (%)[a]	Mp (°C)	
						Found	Lit. [References]
1	 **2a**	 **3a**	 **4a**	15	95	260–262	257–258 [38, 39] 261–263 [42]
2	 **2b**	 **3a**	 **4b**	10	95	250–251	259–260 [38, 39] 250–252 [42]

Table 2 continued

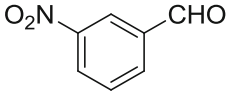
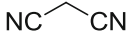
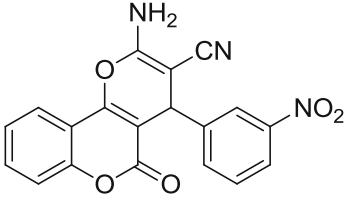
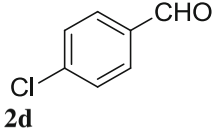
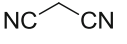
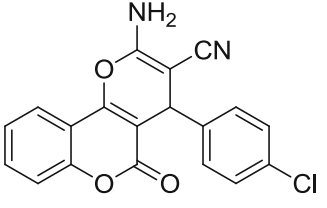
Entry	Aldehyde	Cyano compound	Product	Time (min)	Yield (%)[a]	Mp (°C)	
						Found	Lit. [References]
3	 **2c**	 **3a**	 **4c**	10	93	259–260	258–260 [38, 39]
4	 **2d**	 **3a**	 **4d**	10	96	265–266	264–266 [38, 39]

Table 2 continued

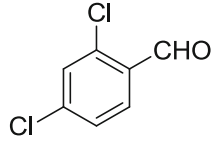
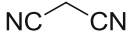
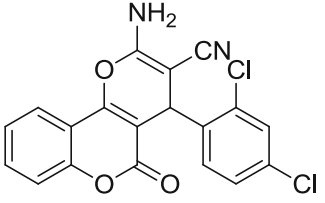
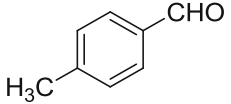
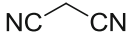
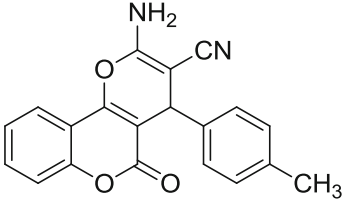
Entry	Aldehyde	Cyano compound	Product	Time (min)	Yield (%)[a]	Mp (°C)	
						Found	Lit. [References]
5	 **2e**	 **3a**	 **4e**	15	90	252–255	253–255 [38, 39]
6	 **2f**	 **3a**	 **4f**	20	94	257–259	258–260 [38, 39]

Table 2 continued

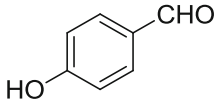
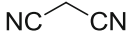
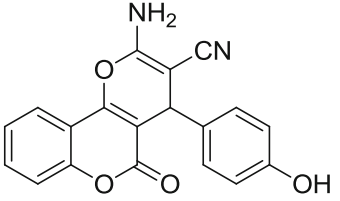
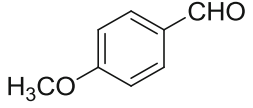
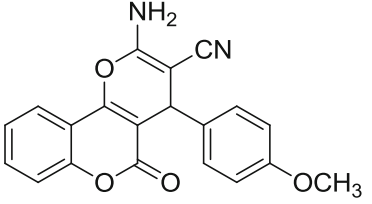
Entry	Aldehyde	Cyano compound	Product	Time (min)	Yield (%)[a]	Mp (°C)	
						Found	Lit. [References]
7	 **2g**	 **3a**	 **4g**	20	92	259–261	260–261 [38, 39]
8	 **2h**	 **3a**	 **4h**	20	93	235–236	235–237 [38, 39]

Table 2 continued

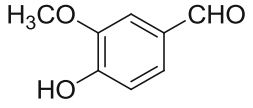
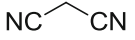
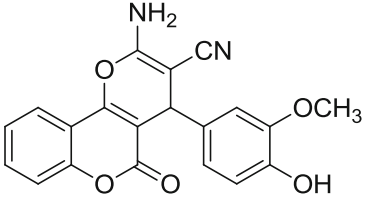
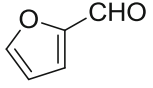
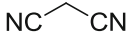
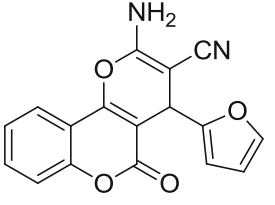
Entry	Aldehyde	Cyano compound	Product	Time (min)	Yield (%)[a]	Mp (°C)	
						Found	Lit. [References]
9	 **2i**	 **3a**	 **4i**	25	94	252–254	253–254 [31]
10	 **2j**	 **3a**	 **4j**	20	93	252–254	252–253 [35]

Table 2 continued

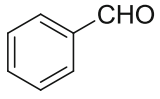
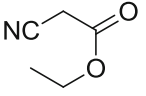
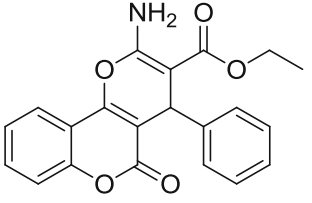
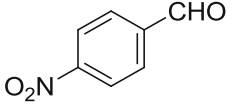
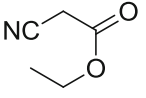
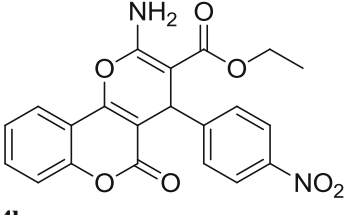
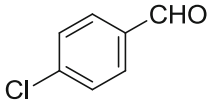
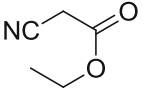
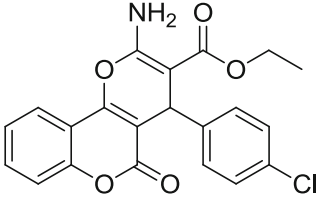
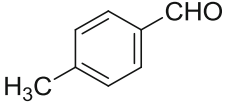
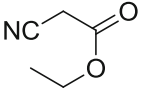
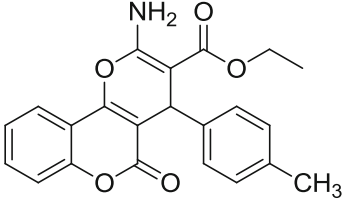
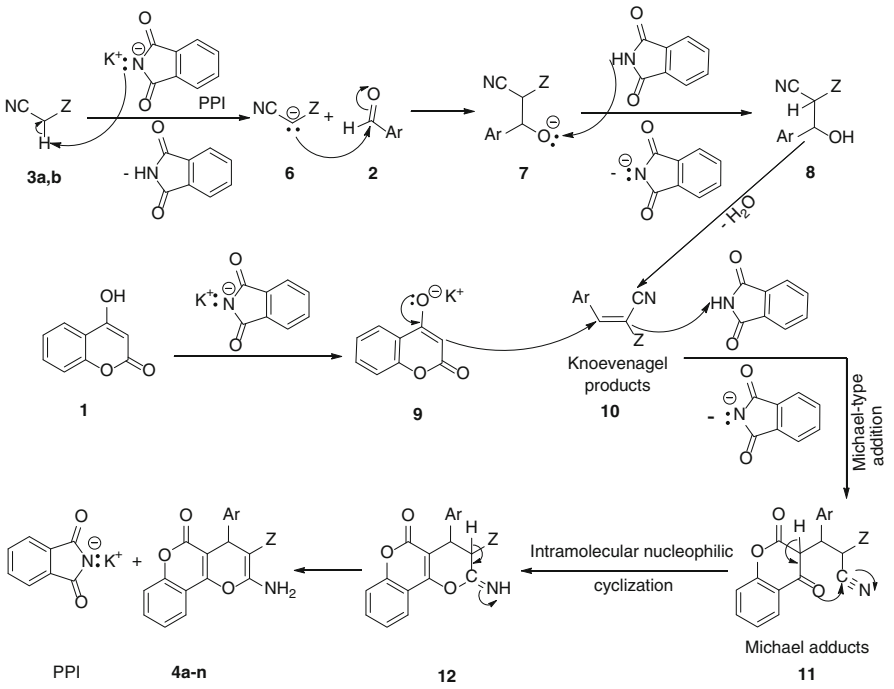
Entry	Aldehyde	Cyano compound	Product	Time (min)	Yield (%)[a]	Mp (°C)	
						Found	Lit. [References]
11	 **2a**	 **3b**	 **4k**	30	86	209–210	187–189 [41], 209–201 [42]
12	 **2b**	 **3b**	 **4l**	20	92	231–232	242–244 [41], 232–233 [42]

Table 2 continued

Entry	Aldehyde	Cyano compound	Product	Time (min)	Yield (%)[a]	Mp (°C)	
						Found	Lit. [References]
13	 **2d**	 **3b**	 **4m**	20	89	192–193	193–194 [41]
14	 **2f**	 **3b**	 **4n**	40	83	195–197	193–195 [42]

All of the reactions were carried out using 4-hydroxycoumarin (1 mmol), aromatic aldehydes (1 mmol), malononitrile (1 mmol) or ethyl cyanoacetate (1 mmol), water (5 mL), and PPI (15 mol%) under reflux

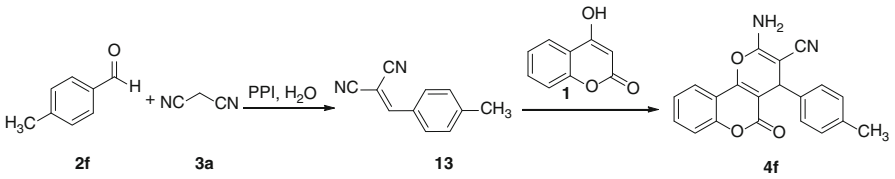
[a] Yields refer to those of pure isolated products



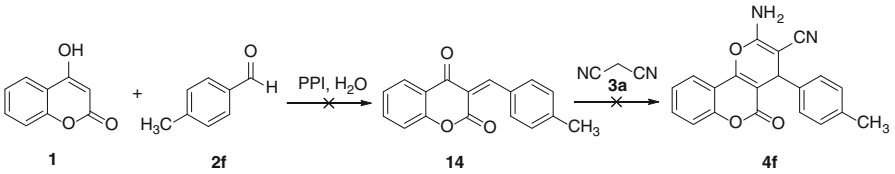
Scheme 2 Proposed mechanism for the formation of dihydropyrano[3,2-c]chromenes (**4a**–**n**)

A possible mechanism for the formation of final products (**4**) is proposed in Scheme 2. The removal of acidic hydrogen from active methylene nitriles (**3a**, **3b**) by PPI, leads to nitrile anion **6**. The arylidene nitrile intermediates (Knoevenagel products, **10**) are formed via the Knoevenagel condensation reaction of aldehydes (**2**) with the intermediate nitrile anion **6**. Then, the Michael-type addition of the enolizable compound to the intermediate **10** results in the in situ formation of intermediate **11** (Michael adducts), which subsequently undergoes intramolecular nucleophilic cyclization (Thorpe–Ziegler type reaction) and tautomerization to afford the desired compounds.

In order to validate the suggested mechanism, we conducted the synthesis of **4f** in two steps under similar conditions (Scheme 3). In the first step, the Knoevenagel condensation component **13** was prepared. Measuring the melting point of the solid product **13**, and comparison with known compounds (prepared according to Ref. [69]), confirmed the formation of arylidene malononitrile intermediate **13** in this step. Also, the FT-IR spectrum of intermediate **13** showed a nitrile peak at 2,222 cm^{-1}, indicating the presence of a malononitrile moiety. Then, the reaction between intermediate **13** and 4-hydroxycoumarin (**1**), can give product **4f** in similar to the one-pot version. Additional evidence was obtained by TLC analysis of the model reaction. TLC analysis 7 min after initiation of the reaction showed that spots of the 3-methylbenzaldehyde and malononitrile disappeared and a new spot was



Scheme 3 The synthesis of **4f** from **2f** and **3a** in two steps under an optimal conditions reaction



Scheme 4 The synthesis of **4f** from **1** and **2f** in two steps under an optimal conditions reaction

established. The new spot is related to condensation product **13**. This fact offers evidence in support of the suggested pathway.

Another pathway could be proposed for the reaction, as seen in Scheme 4. Under optimal reaction conditions, the reaction between 4-hydroxycoumarin (**1**) and 4-methylbenzaldehyde (**2f**) was investigated. Increasing the reaction time to 45 min did not lead exclusively to the formation of **14**. It can be concluded that the reaction proceeds via the suggested mechanism in Scheme 2.

In this reaction, the catalyst is recoverable. The catalyst was recovered by evaporation of the solvent from the filtrate solution after each run. The catalyst recycled was applied to consecutive runs in four series of the same model reaction under the optimized conditions for up to four runs (1st use: 94 %, isolated yield; 2nd use: 91 % isolated yield; 3rd use: 87 % isolated yield; and 4th use: 82 % isolated yield). Decreasing the yield is probably related to a slight reduction in the catalytic activity of the catalyst or a decrease in the amount of the catalyst recycled, which is attributed to the handling.

Conclusion

In summary, we have developed an efficient PPI-catalyzed one-pot, three-component methodology for the synthesis of a variety of dihydropyrano[3,2-c]chromene derivatives in high to excellent yields. This approach is very simple from the experimental point of view and would permit easy access to large families of dihydropyrano[3,2-c]chromenes. Clean, avoiding the use of hazardous organic solvents, minimizing the amount of waste for each organic transformation, reasonable reaction times, easy purification, aqueous conditions, efficiency, green, inexpensive, mild, and economic availability of the organocatalyst are the other noticeable features of this method.

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