Heterocyclic Letters
Vol. 8| No.3|543-551|May-July|2018
ISSN : (print) 2231–3087 / (online) 2230-9632
CODEN: HLEEAI
http://heteroletters.org

RAPID AND EFFICIENT ONE-POT SYNTHESIS OF 2-AMINO-4-ARYL-4*H*-CHROMENES CATALYZED BY $H_3PMo_{12}O_{40}$ UNDER ULTRASOUND IRRADIATION

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Abstract: A simple and efficient synthesis of 2-amino-4-aryl-7-hydroxy-4*H*-chromene-3-carbonitriles and ethyl 2-amino-4-aryl-7-hydroxy-4*H*-chromene-3-carboxylates was achieved *via* a one-pot three-component reaction of resorcinol, aromatic aldehydes, and malononitrile or ethyl cyanoacetate in the presence of a catalytic amount of phosphomolybdic acid ($H_3PMo_{12}O_{40}$) as a reusable inorganic catalyst under ultrasonic irradiation. The catalyst is inexpensive and readily available and can be recovered conveniently and reused efficiently such that a considerable catalytic activity still could be achieved after the fifth run. Other key features of this methodology are operational simplicity, high yields, and short reaction times.

Keywords: $H_3PMo_{12}O_{40}$, 2-Amino-4-aryl-4*H*-chromenes, Ultrasonic irradiation

Introduction

The synthesis of compounds containing chromene moiety has attracted widespread attention due to the diverse applications of these compounds including antihypertensive[i], antiviral[ii], antimalarial[iii], antiproliferative[iv], antivascular[v], antibacterial[vi], anti-HIV[vii], anti-Alzheimer[viii], and antitumor[ix] effects. They have also been widely employed as cosmetics, pigments[x], and potent biodegradable agrochemicals[xi]. Certain chromenes are known as potential inhibitors of Src kinase[xii], NF-κB[xiii], TNF-α[xiv], butyrylcholinesterase[xv], aldose reductase[xvi], PTP1B[xvii], α-Glucosidase[xviii], and AChE[xix]. 2-Amino-4*H*-chroemnes, in particular, have been reported to demonstrate useful proapoptotic properties for the treatment of a wide range of cancer ailments[xx,xxi]. In cancer chemotherapy, 2-amino-4*H*-chromene **I** (Figure 1) was marked for drug development due to its high inhibition of tumor-associated Bcl-2 proteins[xxii]. Modified structures **II** and **III** were able to induce apoptosis (programmed cell death) in several cancer cell lines[xxiii]. 2-Acylamino-7-hydroxy-4*H*-chromene derivative **IV,** was also found to have potential ability in the enhancement of cognitive functions, thus it is used in the treatment of

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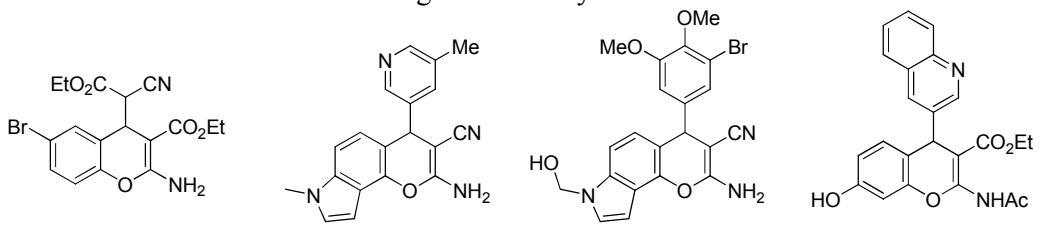
neurodegenerative diseases[xxiv]. Moreover, 2-amino-4*H*-chromenes have also been shown to exhibit antibacterial[xxv-xxvii] and antifungal[xxvii] activity.



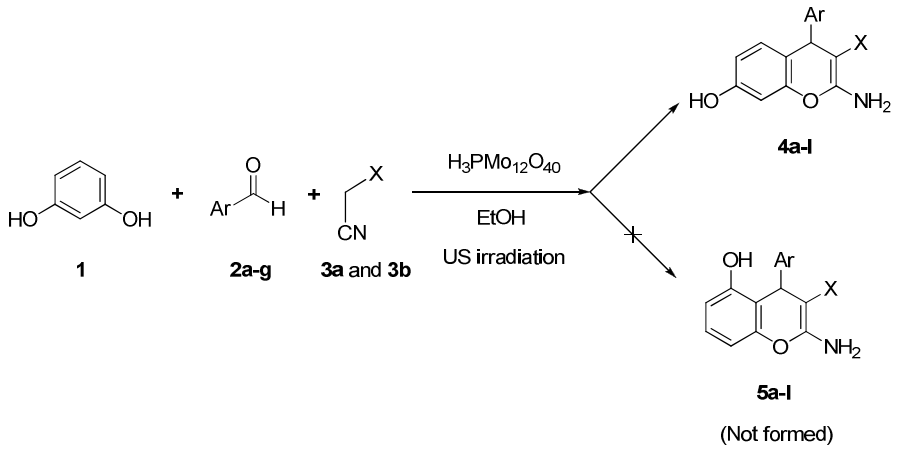
I **II** **III** **IV**

Figure 1. Structures of some biologically active 2-amino-4*H*-chromenes

In view of these useful properties, it is not surprising that the development of synthetic approaches to chromene ring system has attracted considerable interest over the years. A perusal of the literature reveals that there are a number of methods for the synthesis of 2-amino-4*H*-chromenes including one-pot reaction of salicylaldehyde, malononitrile, and nitroalkanes catalyzed by DBU[xxviii], reaction of malononitrile with in situ generated *ortho*-quinone methides from 2-(arylsulfonyl)alkyl phenols[xxix], potassium carbonate catalyzed conjugate addition-cyclization reaction of malononitrile with Knoevenagel adducts[xxx], and condensation of salicylaldehydes with 3 equiv of malononitrile[xxxi]. The synthesis using ultrasound irradiation in the presence of Fe_3O_4-chitosan nanoparticles[xxxii], or by electrolysis in the presence of NaBr as an electrolyte[xxxiii] have also been reported for these compounds. In addition, they can be accessed using stepwise reaction of an aromatic aldehyde and ethyl cyanoacetate followed by cyclization with activated phenols or resorcinol in the presence of piperidine as catalyst[xxxiv]. An improved method has also been reported for the synthesis of these compounds *via* a one-pot three-component reaction of resorcinol, an aromatic aldehyde and malononitrile or ethyl cyanoacetate initiated by various catalysts[xxxv-xlii]. Nevertheless, the discovery of new methodologies using new efficient reusable catalysts for the synthesis of 2-amino-4*H*-chromenes is of certain demand.

The application of ultrasonic (US) irradiation as very significant nonconventional technique in organic synthesis has attracted much research interest because of the simplicity in operation and enhanced reaction rates. Compared with traditional methods, the salient features and benefits of US irradiation technique includes reduced reaction times, improved yields, enhanced selectivity, and reduced energy consumption[xliii-xlv].

Based on these precedents and also in extension of our previous works on the development of new environmentally friendly methods for the synthesis of organic compounds using reusable catalysts[xlvi-xlxii], we report here the application of phosphomolybdic acid, $H_3PMo_{12}O_{40}$, a Keggin-type heteropolyacid, as catalyst in the synthesis of 2-amino-4-aryl-7-hydroxy-4*H*-chromenes **4a-l** by one-pot three-component reaction of resorcinol **1**, aromatic aldehydes **2a-g**, and malononitrile **3a** or ethyl cyanoacetate **3b** under US irradiation (Scheme 1).



Scheme 1. $H_3PMo_{12}O_{40}$ catalyzed synthesis of 2-amino-4-aryl-7-hydroxy-4*H*-chromenes under US irradiation

Experimental

All chemicals were purchased from Merck and Aldrich and used without purification. Melting points were measured on a Stuart SMP3 melting point apparatus. The ^{1}H spectra were measured on a Bruker 300 FT spectrometer using TMS as the internal standard. IR spectra were recorded on a Tensor 27 Bruker spectrophotometer in KBr disks. Ultrasonication was performed by Soltec sonicator (Italy, 2200ETH S3) at a frequency of 40 kHz and a nominal power of 260 W.

General procedure for the synthesis of 2-amino-4-aryl-7-hydroxy-4*H*-chromenes 4a-l catalyzed by $H_3PMo_{12}O_{40}$

Method A (US irradiation). A mixture of resorcinol **1** (1 mmol), an aromatic aldehyde **2a-g** (1 mmol), malononitrile **3a** or ethyl cyanoacetate **3b** (1 mmol) and $H_3PMo_{12}O_{40}$ (15 mol%) in ethanol (5 mL) was sonicated at 60 °C for 4-10 min. The reaction was monitored by TLC. Upon completion of the transformation, the reaction mixture was cooled to room temperature. This resulted in the precipitation of the product, which was collected by filtration, washed repeatedly with cold water and recrystallized from ethanol to give products **4a-l** in high yields.

Method B (conventional heating). A mixture of resorcinol **1** (1 mmol), an aromatic aldehyde **2a-g** (1 mmol), malononitrile **3a** or ethyl cyanoacetate **3b** (1 mmol) and $H_3PMo_{12}O_{40}$ (15 mol%) in ethanol (5 mL) was heated at 60 °C for 25-50 min. The reaction was monitored by TLC. Upon completion of the transformation, the reaction mixture was cooled to room temperature. The precipitate was collected by filtration, washed repeatedly with cold water and recrystallized from ethanol to give products **4a-l** in high yields.

Results and discussion

Our interest in the use of nonconventional energy sources[xlxiii-xlxvi], prompted us to study the synthesis of 2-amino-4-aryl-7-hydroxy-4*H*-chromenes **4a-l** under US irradiation. At first, the synthesis of compound **4a** was selected as a model reaction to optimize the reaction conditions. The reaction was carried out by heating a mixture of resorcinol **1** (1 mmol), benzaldehyde **2a** (1 mmol), and malononitrile **3a** (1 mmol) in the absence or presence of $H_3PMo_{12}O_{40}$ as catalyst in different solvents, including H_2O, MeOH, EtOH, and CH_3CN under US irradiation. The results are summarized in Table 1. A blank reaction without catalyst in H_2O or EtOH at 60 °C gave only low yield of the product (entries 1 and 2). The

reaction was efficiently catalyzed by $H_3PMo_{12}O_{40}$ and ethanol proved to be a much better solvent in terms of yield as well as reaction time than all the others. The excellent yield of the product was obtained when the reaction was conducted in EtOH at 60 °C in the presence of 15 mol% of the $H_3PMo_{12}O_{40}$ catalyst (entry 20). No significant improvement in yield or reaction time was observed using higher amount of the catalyst. All subsequent reactions were carried out in these optimized conditions.

Using these optimized reaction conditions, the scope and efficiency of this approach was explored for the synthesis of a wide variety of 2-amino-4-aryl-7-hydroxy-4*H*-chromenes by reaction of resorcinol with aromatic aldehydes, and malononitrile or ethyl cyanoacetate, and the obtained results are summarized in Table 2. All the reactions delivered excellent product yields over short reaction times and accommodated a wide range of aromatic aldehydes bearing both, electron-donating and electron-withdrawing substituents.

Table 1. Screening of reaction parameters for the formation of compound **4a** catalyzed by $H_3PMo_{12}O_{40}$[a]

Entry	Catalyst (mol%)	Solvent	T (°C)	Time (min)	Isolated Yield (%)
1	-----	H_2O	60	90	23
2	-----	EtOH	60	90	25
3	10	H_2O	r.t.	45	54
4	10	H_2O	60	30	59
5	10	MeOH	r.t.	35	58
6	10	MeOH	60	20	63
7	10	EtOH	r.t.	30	61
8	10	EtOH	60	15	72
9	12	H_2O	r.t.	35	62
10	12	H_2O	60	20	67
11	12	MeOH	r.t.	30	66
12	12	MeOH	60	15	72
13	12	EtOH	r.t.	25	70
14	12	EtOH	60	10	81
15	15	H_2O	r.t.	30	68
16	15	H_2O	60	15	75
17	15	MeOH	r.t.	25	75
18	15	MeOH	60	10	81
19	15	EtOH	r.t.	20	78
20	15	EtOH	60	7	90
21	17	EtOH	60	7	90
22	17	H_2O	60	15	72
23	17	MeOH	60	10	80
24	15	CH_3CN	60	15	74
25	17	CH_3CN	60	20	75

[a]Reaction conditions: resorcinol **1** (1 mmol), benzaldehyde **2a** (1 mmol), and malononitrile **3a** (1 mmol) under ultrasonic irradiation.

Table 2. $H_3PMo_{12}O_{40}$ catalyzed synthesis of 2-amino-4-aryl-7-hydroxy-4*H*-chromenes **4a-l**[a]

Entry	Ar	X	Product	Method A (US irradiation)		Method B (Conventional heating)	
				Time (min)	Yield (%)	Time (min)	Yield (%)
1	C_6H_5	CN	**4a**	7	90	30	79
2	4-ClC_6H_4	CN	**4b**	5	94	25	82
3	4-FC_6H_4	CN	**4c**	4	95	25	84
4	4-BrC_6H_4	CN	**4d**	5	92	25	80
5	4-$MeOC_6H_4$	CN	**4e**	10	90	45	77
6	4-MeC_6H_4	CN	**4f**	10	88	40	78
7	3-$O_2NC_6H_4$	CN	**4g**	5	92	30	81

8	C_6H_5	CO_2Et	**4h**	10	90	35	80
9	4-ClC_6H_4	CO_2Et	**4i**	8	91	30	85
10	4-FC_6H_4	CO_2Et	**4j**	6	90	25	84
11	4-BrC_6H_4	CO_2Et	**4k**	7	91	35	79
12	4-$MeOC_6H_4$	CO_2Et	**4l**	10	89	50	75

[a]Reaction conditions: resorcinol **1** (1 mmol), aromatic aldehydes **2a-g** (1 mmol), and malononitrile **3a** or ethyl cyanoacetate **3b** (1 mmol), $H_3PMo_{12}O_{40}$ (15 mol%) under US irradiation in EtOH (5 mL) at 60 °C (Method A), or in EtOH (5 mL) at 60 °C (Method B).

All the products were characterized by comparison of their melting points with those of authentic samples and for some cases using ^{1}H NMR spectral data. According to the aromatic region of chromene ring in ^{1}H NMR spectral data, it is proved that due to steric hindrance between two hydroxyl groups or crowding of the aryl group with the remaining OH group in the product, resorcinol **1** reacts at position C-4 instead of position C-2, and therefore 2-amino-4-aryl-7-hydroxy-4*H*-chromenes **4a-l** are formed not the corresponding 5-hydroxy isomers **5a-l** (Scheme 1). For example, as shown in Figure 2, the ^{1}H NMR spectrum of the compound isolated from the reaction of resorcinol with benzaldehyde, and malononitrile in DMSO-d_6 showed a doublet at δ = 6.43 for H-8 with a *metha* coupling (*J* =2.4 Hz), a doublet of doublet at δ = 6.50 for H-6 with *ortho* and *metha* couplings (J_1 =8.4, J_2 = 2.1 Hz), and a doublet at δ = 6.82 for H-5 with an *ortho* coupling (*J* =8.4 Hz) in aromatic region for chromene ring which is in accord with structure **4a** and not **5a**.

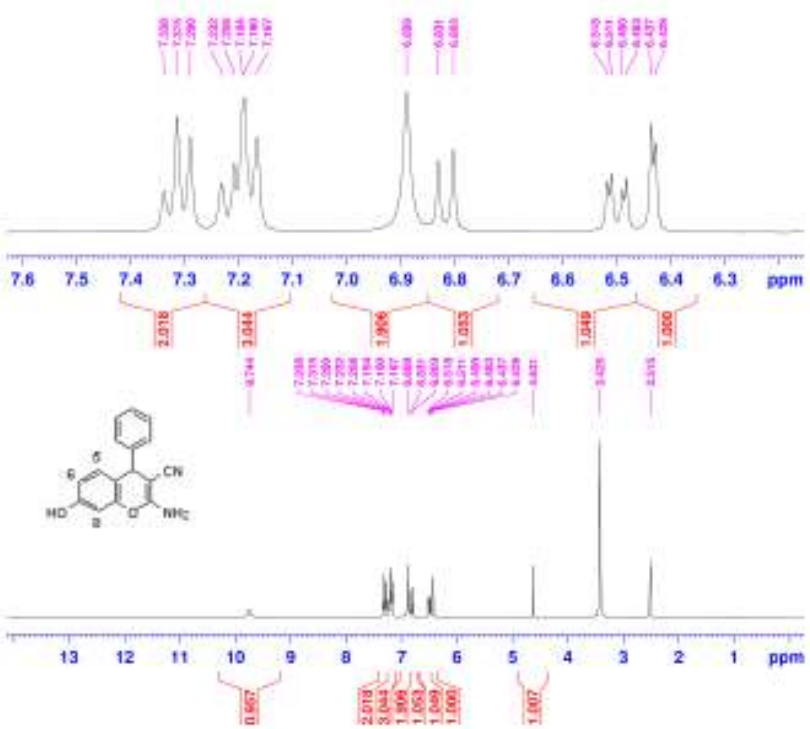


Figure 2. The ^{1}H NMR spectrum of compound **4a** in DMSO-d_6 and its expanded view

For comparison, a classical method using conventional heating for the synthesis of 2-amino-4-aryl-7-hydroxy-4*H*-chromenes **4a-l** was also investigated by heating a mixture of resorcinol **1**, an aromatic aldehyde **2a-g**, and malononitrile **3a** or ethyl cyanoacetate **3b** in ethanol for

the indicated time (Table 2). It was very obvious that the classical approach is a tedious method affording a relatively lower yield of 4**a-l** with much longer reaction time.

We also used our optimized reaction conditions to evaluate the reusability of the catalyst $H_3PMo_{12}O_{40}$ in the model reaction. After completion of the reaction, the reaction mixture was cooled to room temperature, the product was collected by filtration, and washed repeatedly with cold water. The combined filtrate was evaporated to dryness under reduced pressure. The solid catalyst was collected, dried at 60 °C under vacuum for 1 h and reused for the same experiment. We found that the catalyst could be used at least five times with only a slight reduction in activity (Figure 3).

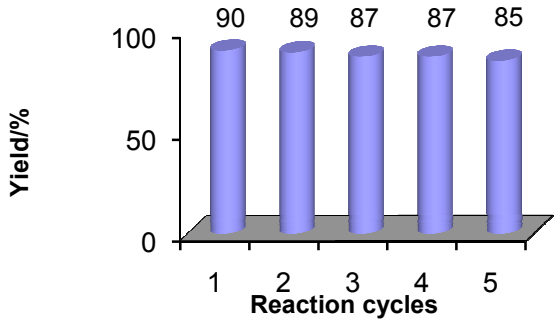
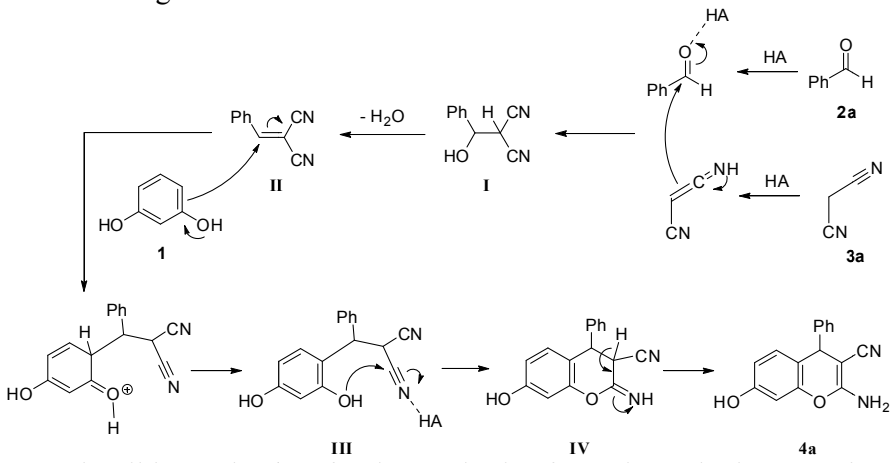


Figure 3. Reusability of $H_3PMo_{12}O_{40}$ in the synthesis of **4a** in model reaction.

Considering the Brønsted acidic nature of $H_3PMo_{12}O_{40}$ ≡ HA, a possible mechanism is proposed as depicted in Scheme 2. The catalyst plays a significant role in increasing the electrophilic character of the electrophiles in the reaction. The reaction occurs *via* initial formation of the intermediate **I** which on dehydration affords intermediate **II.** This intermediate reacts subsequently with resorcinol **1** to give the intermediate **III**. Cyclization of the later intermediate followed by tautomerization gave the final product **4a** *via* the intermediate **IV**. Under these conditions, attempts to isolate the intermediates failed even after careful monitoring of the reactions.



Scheme 2. Plausible mechanism for the synthesis of 2-amino-7-hydroxy-4-phenyl-4*H*-chromene-3-carbonitril **4a** catalyzed by $H_3PMo_{12}O_{40}$ ≡ HA

Conclusion

In summary, we successfully developed a simple, efficient and ecofriendly method for the synthesis of 2-amino-4-aryl-7-hydroxy-4*H*-chromenes **4a-l** by one-pot three-component

reaction of resorcinol, aromatic aldehydes, and malononitrile or ethyl cyanoacetate using $H_3PMo_{12}O_{40}$ as a readily available, cheap, and reusable inorganic catalyst under ultrasonic irradiation and also by thermal heating. In comparison, the reactions carried out with the assistance of US technique are faster and the yields are higher than conventional method. Some attractive features of this protocol are excellent yields, simple procedure, short reaction times, easy work-up, high catalytic activity and recyclability and reusability of the catalyst. The catalyst can be used at least five times without substantial reduction in its catalytic activity.

Acknowledgement
This work was supported by Islamic Azad University, Mashhad Branch, Iran.

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Received on April 3, 2018.