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Facile one-pot multicomponent synthesis of 2-amino-6-(2-oxo-2*H*-chromen-3-yl)-4-arylpyridine-3-carbonitriles using Brønsted acidic ionic liquid as catalyst under solvent-free conditions

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ABSTRACT

A series of 2-amino pyridine-3-carbinitrile derivatives incorporated coumarin moiety has developed *via* multicomponent condensation of 3-acetyl-2*H*-chromen-2-one, arylaldehydes, malononitrile and ammonium acetate utilizing Brønsted acidic ionic liquid, (4-sulfobutyl)tris(4-sulfophenyl)phosphonium hydrogen sulfate as catalyst under solvent-free conditions. Good yields, short reaction times, straight forward workup, reusability of the ionic liquid and green conditions are the most obvious advantages of this methodology.

Keywords*: 2-Amino pyridine-3-carbinitrile; Multicomponent condensation; 3-Acetyl-2H-chromen-2-ones; (4-Sulfobutyl)tris-(4-sulfophenyl)phosphonium hydrogen sulfate.*

1. Introduction

The development of simple synthetic routes for complex organic molecules from readily available starting materials and reagents *via* one-pot reaction is an important task in organic synthesis [1]. Multi-component reactions (MCRs) have emerged as a powerful tools for the rapid and efficient synthesis of a wide variety of organic molecules [2]. MCRs under solvent-free conditions have attracted much interest from chemists particularly from the view point of green chemistry; these reactions offer greater possibilities for molecular diversity per step with a minimum of synthetic time, labour, cost, and waste production. Moreover, nowadays ionic liquids (ILs) playing an important role in synthetic chemistry due to their interesting properties like high thermal stability, non volatility, eco friendly and reusability [3]. (4-Sulfobutyl) tris(4-sulfophenyl)phosphonium hydrogen sulfate is an important ionic liquid used for the various chemical transformations such as synthesis of benzoxanthene [4], chromeno pyrimidines [5, 6], fused 3,4-dihydropyrimidin-2(1*H*)-ones and thiones [7], coumarin derivatives [8] and oxathioacetalization of carbonyl compounds [9].

Many naturally occurring and synthetic compounds containing the cyanopyridine scaffold possess interesting biological properties. Among them, 3-cyanopyridine derivatives have played an important role in the pharmaceutical industries due to their wide range of applications such as antitumor [10], antimicrobial [11], analgesic, anti-inflammatory, antipyretic [12], anticancer [13], anticoagulant [14] and cardiotonic [15] activities. They are also found to be selective IKK-β serine-threonine protein kinase inhibitors [16]. On the other hand coumarin derivatives were also found to possess antimicrobial [17], analgesic, anti-pyretic [18], antiamoebic [19], antioxidant [20] and anticancer [21] activities. These are widely used as additives to food, perfumes, cosmetics, optical brightener, dispersed fluorescence and laser dyes [22,23].

From the past few decades many methods have been developed for the preparation of 2-amino-3-cyano pyridine derivatives under microwave irradiation [24] and using various catalytic systems such as ytterbium

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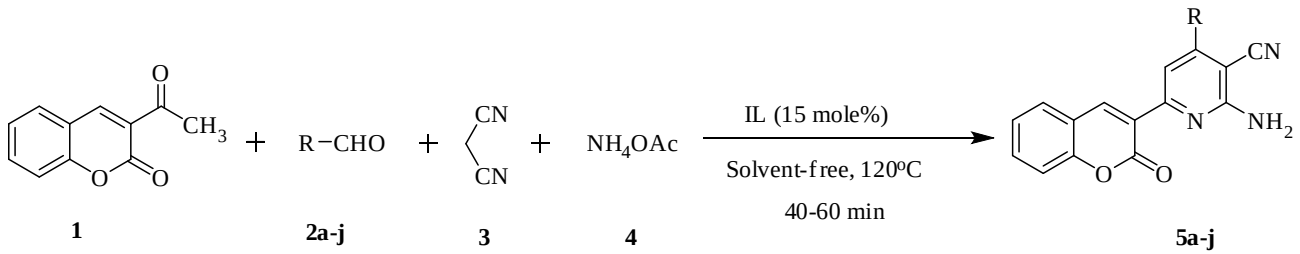


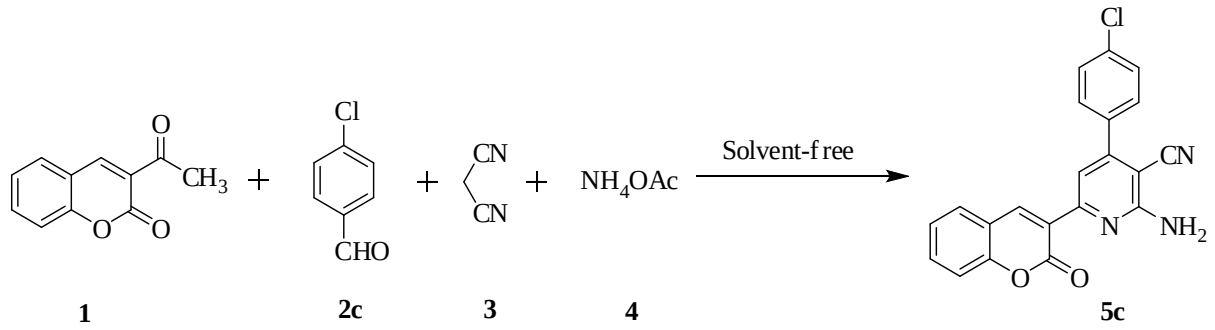
2a/5a: R = C_6H_5 **2e/5e:** R = 4-$NO_2C_6H_4$ **2i/5i:** R = 4-OHC_6H_4

2b/5b: R = 2-ClC_6H_4 **2f/5f:** R = 4-$CH_3C_6H_4$ **2j/5j:** R = 4-OH-3-$OCH_3C_6H_3$

2c/5c: R = 4-ClC_6H_4 **2g/5g:** R = 4-$OCH_3C_6H_4$

2d/5d: R = 3-$NO_2C_6H_4$ **2h/5h:** R = 3,4-$(OCH_3)_2C_6H_3$

Scheme 1. Synthesis of 2-amino-6-(2-oxo-2*H*-chromen-3-yl)-4-arylpyridine-3-carbonitriles catalyzed by IL under neat conditions.

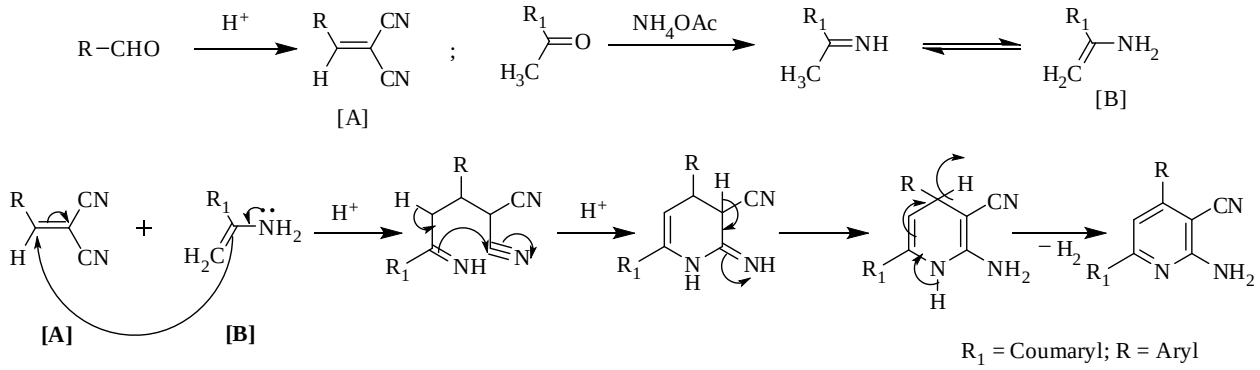


Scheme 2. Synthesis of 2-amino-4-(4-chlorophenyl)-6-(2-oxo-2*H*-chromen-3-yl) pyridine-3-carbonitrile.

perfluorooctanoate [25], silica-bound *N*-propyl triethylenetetramine sulfamic acid [26] and ionic liquids such as ethylammonium nitrate [27] and 3-methyl-1-(4-sulfonylbutyl) imidazolium hydrogen sulfate [28]. Most of these methods suffer from one or severe drawbacks such as low yield, long reaction time, tedious workup, use of toxic solvents, expensive reagents and involving multistep process. Recently, Zhou and co-workers [29] have reported the synthesis of 2-amino-3-cyano pyridine derivatives incorporated coumarin moiety but the yield of the products are moderate and the protocol has not environmentally benign. Therefore we have developed an eco-friendly one-pot method using inexpensive Brønsted acidic ionic liquid, (4-sulfobutyl) tris-(4-sulfophenyl) phosphonium hydrogen sulfate (IL) as catalyst under solvent-free conditions with good yields.

2. Experimental

All the reagents were purchased from Aldrich/Merck and used without further purification. Melting points were determined in open capillaries using Stuart SMP30 apparatus and are uncorrected. The progress of the reactions as well as purity of compounds was monitored by thin layer chromatography with F254 silica-gel precoated sheets using hexane/ethyl acetate 8/2 as eluent; UV light and iodine vapours were used for detection. Products were characterized by comparing with authentic samples and by spectroscopy data (IR and ^{1}H NMR). IR spectra were recorded on Perkin-Elmer 100S spectrometer using KBr pellet and values are expressed in cm^{-1}. ^{1}H NMR spectra were recorded on Bruker 400 MHz spectrometer using appropriate solvent and TMS as



Scheme 3. Proposed mechanism for the formation of 2-amino-3-cyano pyridine nucleus catalyzed by Brønsted acidic ionic liquid (IL).

internal standard, chemical shifts are expressed in ppm. Elemental analysis was performed on a Carlo Erba modal EA1108 and the values are ± 0.4% of the theoretical ones. Mass spectra were recorded on a Jeol JMSD-300 spectrometer.

2.1. Synthesis of (4-sulfobutyl)tris(4-sulfophenyl) phosphonium hydrogen sulfate (IL)

Preparation of zwitterions

A mixture of triphenylphosphine (20 mmol) and 1,4-butane sultone (20 mmol) were taken in 100 mL of toluene and magnetically stirred for 12 h at its refluxing temperature. The solid zwitterion separated out was filtered under vacuum, washed with ether three times and dried at 110˚C for 2 h. The product zwitterions were formed in good yield (90%).

Spectral data

^{1}H NMR (D_2O, 400 MHz, ppm) δ: 1.21 (s, 2H), 2.34 (s, 2H), 3.82 (s, 2H), 4.11 (s, 2H), 7.43-8.49 (m, 15H); ^{13}C NMR (D_2O, 100 MHz, ppm) δ: 143.91, 134.77, 131.43, 127.45, 37.98, 32.82, 23.74, 19.89.

Preparation of IL

Quadruple molar sulfuric acid (40 mmol) was added to the zwitterions (10 mmol) present in a three necked round bottomed flask equipped with a magnetic stirrer and a thermometer. The mixture was stirred for 10 h at temperature from 80-150 ˚C to form the ionic liquid. Then, the IL phase was washed repeatedly with toluene and ether to remove non-ionic residues, and dried in vacuum at 110 ˚C. A black viscous liquid was formed quantitatively in high purity (Scheme 4).

Spectral data

^{1}H NMR (D_2O, 400 MHz, ppm) δ: 1.87 (s, 2H), 1.29-1.39 (m, 4H), 1.81-1.93 (m, 2H), 3.54 (t, *J* = 7.6 Hz, 3H), 7.95 (d, *J* = 6.4 Hz, 6H), 7.52 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (D_2O, 100 MHz, ppm) δ: 149.84, 135.42, 128.12, 122.43, 52.03, 26.64, 22.64, 20.61.

2.2. General Procedure for the synthesis of 2-amino-6-(2-oxo-2H-chromen-3-yl)-4-arylpyridine-3-carbonitriles (5a-j):

To a mixture of 3-acetyl-2*H*-chromen-2-one (1 mmol), arylaldehyde (1 mmol), malononitrile (1.1 mmol) and ammonium acetate (3 mmol); IL (15 mole%) was added and heated at 120˚C for an appropriate time as shown in Table 2. After completion of the reaction (monitored by TLC), 5 mL of water was added and stirred at r.t. for additional 5 min. The solid separated out was filtered, washed with water, dried and recystallized from acetic acid. The aqueous layer containing IL was recovered under reduced pressure, washed with acetone, dried and reused for subsequent reactions.

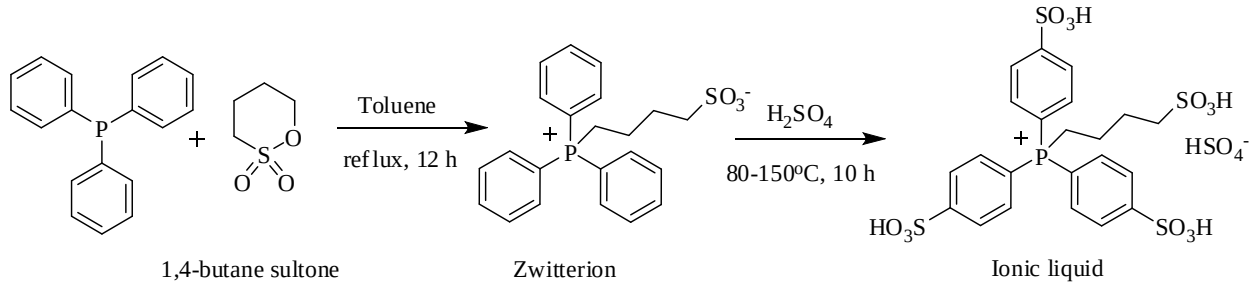
The selected spectral data

2-amino-6-(2-oxo-2H-chromen-3-yl)-4-phenylpyridine -3-carbonitrile (5a)

IR (KBr, υ_{max}, cm^{-1}): 3449, 3352 (NH_2), 2221 (C≡N), 1723 (O-C=O), 1612 (C=N), 1584 (C=C); ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 6.96 (d, *J* = 9.2 Hz, 3H), 7.42-7.57 (m, 5H), 7.61-7.74 (m, 3H), 7.80 (d, *J* = 7.6 Hz, 1H), 8.40 (s, 1H); MS (ESI) m/z: 362 (M+Na); Anal. Calcd. for $C_{21}H_{13}N_3O_2$: C, 74.33; H, 3.86; N, 12.38; Found: C, 74.52; H, 3.69; N, 12.57.

2-amino-4-(2-chlorophenyl)-6-(2-oxo-2H-chromen-3-yl) pyridine-3-carbonitrile (5b)

IR (KBr, υ_{max}, cm^{-1}): 3450, 3382 (NH_2), 2219 (C≡N), 1721 (O-C=O), 1603 (C=N), 1589 (C=C), 669 (C-Cl); ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 7.04 (d, *J* = 9.2 Hz, 3H), 7.46-7.55 (m, 5H), 7.71-7.75 (m, 1H), 8.04 (d, *J* = 7.6 Hz, 1H), 8.28 (d, *J* = 7.2 Hz, 1H), 8.61 (s, 1H); MS (ESI) m/z: 374 (M+H); Anal. Calcd. for $C_{21}H_{12}ClN_3O_2$: C, 67.48; H, 3.24; N, 11.24; Found: C, 67.68; H, 3.15; N, 11.51.



Scheme 4. Synthesis of (4-sulfobutyl)tris(4-sulfophenyl)phosphonium hydrogen sulphate.

2-amino-4-(4-chlorophenyl)-6-(2-oxo-2H-chromen-3-yl)pyridine-3-carbonitrile (5c)

IR (KBr, υ_{max}, cm^{-1}): 3457, 3368 (NH_2), 2222 (C≡N), 1726 (O-C=O), 1612 (C=N), 1594 (C=C), 678 (C-Cl); ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 6.97 (d, *J* = 9.2 Hz, 3H), 7.44-7.53 (m, 2H), 7.61-7.81 (m, 6H), 8.40 (s, 1H); MS (ESI) m/z: 374 (M+H); Anal. Calcd. for $C_{21}H_{12}ClN_3O_2$: C, 67.48; H, 3.24; N, 11.24; Found: C, 67.63; H, 3.14; N, 11.47.

2-amino-4-(3-nitrophenyl)-6-(2-oxo-2H-chromen-3-yl) pyridine-3-carbonitrile (5d)

IR (KBr, υ_{max}, cm^{-1}): 3452, 3394 (NH_2), 2217 (C≡N), 1725 (O-C=O), 1606 (C=N), 1587 (C=C), 1575 (NO_2); ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 7.08 (d, *J* = 9.2 Hz, 3H), 7.43-7.53 (m, 2H), 7.71-7.88 (m, 3H), 8.11-8.14 (m, 1H), 8.37-8.40 (m, 2H), 8.41 (s 1H); MS (ESI) m/z: 385 (M+H); Anal. Calcd. for $C_{21}H_{12}N_4O_4$: C, 65.62; H, 3.15; N, 14.58; Found: C, 65.88; H, 3.32; N, 14.26.

2-amino-4-(3,4-dimethoxyphenyl)-6-(2-oxo-2H-chromen-3-yl)pyridine-3-carbonitrile (5h)

IR (KBr, υ_{max}, cm^{-1}): 3445, 3356 (NH_2), 2219 (C≡N), 1723 (O-C=O), 1609 (C=N), 1592 (C=C), 1165 (C-O-C); ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 3.82 (s, 3H), 3.83 (s, 3H), 6.86 (s, 2H), 7.00 (s, 1H), 7.11-7.22 (m, 3H), 7.43-7.46 (m, 1H), 7.52 (d, *J* = 8.4 Hz, 1H), 7.70-7.82 (m, 2H), 8.39 (s, 1H); MS (ESI) m/z: 400 (M+H); Anal. Calcd. for $C_{23}H_{17}N_3O_4$: C, 69.17; H, 4.29; N, 10.52; Found: C, 69.40; H, 4.57; N, 10.29.

2-amino-4-(4-hydroxy-3-methoxyphenyl)-6-(2-oxo-2H-chromen-3-yl) pyridine-3-carbonitrile (5j)

IR (KBr, υ_{max}, cm^{-1}): 3462 (OH), 3438, 3367 (NH_2), 2220 (C≡N), 1724 (O-C=O), 1606 (C=N), 1590 (C=C), 1167 (C-O-C); ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 3.84 (s, 3H), 6.94-7.09 (m, 4H), 7.34-7.50 (m, 5H), 7.63 (s, 1H), 8.81 (s, 1H), 9.59 (s, 1H); MS (ESI) m/z: 386 (M+H); Anal. Calcd. for $C_{22}H_{15}N_3O_4$: C, 68.57; H, 3.92; N, 10.90; Found: C, 68.76; H, 4.12; N, 10.64.

3. Results and discussion

In continuation of our interest on applications of Brønsted acidic ionic liquid for the synthesis of biologically important molecules [6-8], herein we report a simple, mild and efficient procedure for the synthesis of 2-amino-6-(2-oxo-2*H*-chromen-3-yl)-4-arylpyridine-3-carbonitriles (5a-j) *via* multicomponent condensation of 3-acetyl-2*H*-chromen-2-one (1), arylaldehydes (2a-j), malononitrile (3) and ammonium acetate (4) using inexpensive, environmental friendly and reusable Brønsted acidic ionic liquid (IL) as catalyst with good yields under solvent-free conditions at 120˚C (Scheme 1).

To illustrate the need of the IL for this reaction, an experiment was conducted with 3-acetyl-2*H*-chromen-2-one, *p*-chlorobenzaldehyde, malononitrile and ammonium acetate (Scheme 2) in ethanol under solvent-free conditions with and without IL at different temperatures and the observations are as follows; without IL, in ethanol we observed only 62% of product (5c) yield even after prolonged reaction time (7 h) at its refluxing temperature, under solvent-free conditions the yields obtained were very poor, side products were formed and reactants were not involved completely in the reaction, but with 5 mole% of the IL surprisingly, we observed 68% product yield at 120˚C within 2 h. We also observed the increase in temperature has not altered the product (5c) yield. To improve the yield of the product (5c), the above same reaction was carried out by varying the amount of IL and obtained the maximum yield (92%) with 15 mole%. Also observed lowering the reaction temperature results in low yield of the product, increase in temperature and amount of catalyst has not altered the product yield and reaction times (Table 1).

At these optimistic conditions (15 mol% of IL, heating under solvent-free at 120˚C), various 2-amino-6-(2-oxo-2*H*-chromen-3-yl)-4-arylpyridine-3-carbonitrile derivatives were prepared and the results were postulated in Table 2. After completion of the reaction shown by TLC, the IL was recovered by evaporating

Table 1. Optimizing the reaction conditions.

Entry[a]	Amount of IL (mole%)	Solvent	Temperature (^oC)	Time (min)/(h)	Yield[b] (%)
1	-	Ethanol	Rt	[12]	-
2	-	Ethanol	Reflux	[7]	62
3	-	Solvent-free	Rt	120	-
4	-	Solvent-free	80	120	18
5	-	Solvent-free	120	120	34
6	-	Solvent-free	150	120	34
7	5	Ethanol	Rt	[4]	Trace
8	5	Ethanol	Reflux	[4]	67
9	5	Solvent-free	Rt	120	Trace
10	5	Solvent-free	80	120	54
11	6	Solvent-free	120	120	68
12	5	Solvent-free	150	120	68
13	10	Ethanol	Reflux	[4]	73
14	10	Solvent-free	120	60	79
15	15	Ethanol	Reflux	[4]	81
16	15	Solvent-free	60	120	85
17	15	Solvent-free	90	120	88
18	15	Solvent-free	120	60	92
19	20	Ethanol	Reflux	[4]	85
20	20	Solvent-free	120	60	91

[a]Reaction conditions: 3-acetyl-2*H*-chromen-2-one (1 mmol), *p*-chlorobenzaldehyde (1 mmol), malononitrile (1.1 mmol) and ammonium acetate (3 mmol).
[b]Isolated yields of **5c**.

the aqueous layer, washed with acetone, dried and reused for subsequent reactions for additional five times without significant loss in the product (5c) yield (Table 3). All the synthesized compounds were characterised by analytical and spectral (IR, ^{1}H NMR and Mass) studies and compared with the literature values.

A plausible mechanism for the formation of 2-amino-3-cyano pyridine nucleus in the presence of IL has shown in Scheme 3. Here, the intermediate arylidenemalononitrile **[A]** which was obtained by the reaction of arylaldehydes and malononitrile reacts with coumarylethenamine **[B]** followed by the cyclization, rearrangement and dehydrogenation afford the final compound in shorter reaction times with good yields.

4. Conclusion

In conclusion, a facile and efficient procedure for the synthesis of biologically important 3-cyano pyridine derivatives incorporated coumarin moiety at 6th position has been prepared using environmental friendly Brønsted acidic ionic liquid, (4-sulfobutyl) tris(4-sulfophenyl)phosphonium hydrogen sulfate as catalyst under solvent-free conditions at 120˚C. The prominent advantages of this methodology are reusability of the IL, operational simplicity, easy workup procedure, avoiding hazardous organic solvents and good yields of product.

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Table 2. Synthesis of 2-amino-6-(2-oxo-2*H*-chromen-3-yl)-4-arylpyridine-3-carbonitriles using IL as catalyst.

Entry[a]	Aldehyde	Time (min)	Yield[b] (%)	Melting Point (^oC)	
				Found	Lit. [Ref.]
5a	Benzaldehyde	60	89	258-259	234-236 [29]
5b	2-Chlorobenzaldehyde	60	90	287-289	-
5c	4-Chlorobenzaldehyde	60	92	303-305	245-247 [29]
5d	3-Nitrobenzaldehyde	60	88	262-264	286-288 [29]
5e	4-Nitrobenzaldehyde	60	89	256-258	252-254 [29]
5f	4-Methylbenzaldehyde	40	92	271-273	271-273 [29]
5g	4-Methoxybenzaldehyde	50	92	266-268	268-270 [29]
5h	3,4-Dimethoxybenzaldehyde	40	94	278-279	-
5i	4-Hydroxybenzaldehyde	50	91	260-262	263-264 [29]
5j	4- Hydroxy-3-methoxybenzaldehyde	50	92	270-272	-

[a]Reaction conditions: 3-acetyl-2*H*-chromen-2-one (1 mmol), arylaldehyde (1 mmol), malononitrile (1.1 mmol), ammonium acetate (3 mmol) and IL (15 mole%) heat at 120^{o}C.
[b]Isolated yields after purification.

Table 3. Effect of reusability of IL on product **5c** yield.[a]

Run	Cycle	Time (min)	Yield[a] (%)
1	0	60	92
2	1	60	92
3	2	60	90
4	3	60	90
5	4	60	88
6	5	60	87

[a]Reaction conditions: 3-acetyl-2*H*-chromen-2-one (1 mmol), *p*-chlorobenzaldehyde (1 mmol), malononitrile (1.1 mmol), ammonium acetate (3 mmol) and IL (15 mole%) heat at 120^{o}C.
[b]Yield refers to pure isolated product 5c.

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