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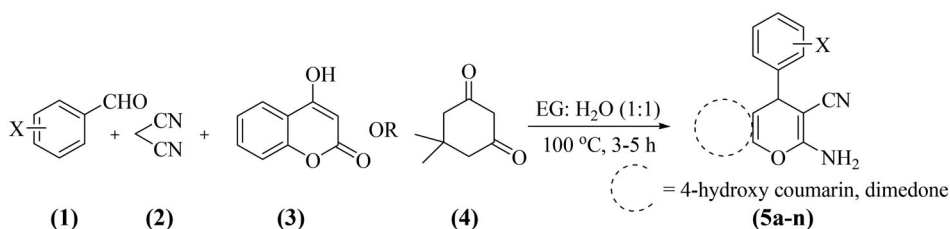
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Recently, development of environmentally benign and clean synthetic protocols has become a goal of modern organic synthesis. Consequently design and development of catalyst-free reactions have both received tremendous attention in the area of green organic synthesis. Efforts are being made to reduce toxicity levels of catalysts by multi-directional modifications leading to the development of organic reactions under catalyst-free conditions.¹ Multi-component reactions (MCRs) in aqueous media without catalysts are highly desirable tools of green chemistry.^{2–3} As a commonly-available chemical, ethylene glycol (EG) has been used for protection of carbonyl compounds in organic synthesis,⁴ and in a large number of industrial processes.⁵ EG is emerging as an efficient and promising solvent for such organic transformations as the synthesis of pyrans,⁶ 4H-pyrimidobenzothiazoles,⁷ pyrimidines,⁸ 2-arylbenzothiazoles and azomethines.⁹

Considerable attention has been focused on the synthesis, reactions and biological activities of dihydropyrano[c]chromene derivatives.¹⁰ Coumarins and dihydropyrano[c]-chromenes constitute the skeleton of such commercial drugs as warfarin, hymecromone and coumatetralyl.^{11,12} Pyran derivatives are useful owing to their promising biological activities.^{13,14} They are as well used as photoactive materials.¹⁵ Pyran derivatives were reported as inhibitors of insulin-regulated amino peptidase related to enhancement of memory and learning functions.¹⁶ Besides this, pyran derivatives are also well documented for anticancer and antibacterial activity.¹⁷ Consequently, numerous catalysts have been developed for the synthesis of these compounds including the use of piperazine,¹⁸ copper nanoparticles,¹⁹ morpholine,²⁰ phenylboronic acid,²¹ poly(4-vinylpyridine),²² silica-bonded *N*-propylpiperazine sodium *n*-propionate,²³ 4-(dimethylamino)pyridine,²⁴ amino functionalized silica gel,²⁵ Ce₁Mg_{0.6}Zr_{0.4}O₂,²⁶ neat silica gel,²⁷ urea,²⁸ and 1,4-diazabicyclo[2.2.2]octane under microwave irradiation.²⁹ Each of these methods has its own value, but in many cases there are also such drawbacks as long reaction times, use of volatile solvents, low yields or harsh reaction conditions. In continuation of our interest in the development of efficient and convenient



Scheme 1. EG:H₂O promoted catalyst-free one-pot multicomponent synthesis of pyran annulated heterocyclic compounds.

methodologies for the synthesis of biologically active heterocyclic compounds,³⁰ we now report on the preparation of pyran annulated derivatives (**5a-n**), namely 3,4-dihydropyrano[c]chromenes and tetrahydrobenzo[b]pyrans. This has been achieved by the one-pot three component condensation of aldehydes (**1**), malononitrile (**2**) and 4-hydroxycoumarin (**3**) or dimedone (**4**) under catalyst free conditions in EG:water (1:1) within 3-5 h (Scheme 1, Table 1). The procedure is noteworthy for its simplicity and convenience.

To determine the optimum conditions, the reaction of 4-chlorobenzaldehyde (1 mmol), 4-hydroxycoumarin (1 mmol) and malononitrile (1 mmol) was chosen. We investigated solvents, temperatures and times as reaction variables, and the results are summarized in Table 2. The work-up procedure consisted of simply pouring the reaction mixture into ice cold water to precipitate the solid, isolated by filtration. As can be seen (Entry 14), the best outcome was obtained in EG:H₂O (1:1) at reflux (100 °C) within 3.5 hours. As seen from Table 1, under the optimized conditions, diversely substituted aromatic aldehydes smoothly afforded the corresponding products in very good to excellent yields.

Aromatic aldehydes having electron-withdrawing groups (Table 1, Entries 9, 10, 13) on the aromatic ring were observed to react faster than those with electron-donating groups. *ortho*-Substituted aromatic aldehydes resulted in lower yields and required comparatively longer times, most probably due to steric effects (Table 1, Entries 5, 6, 7).

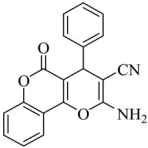
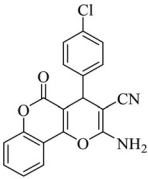
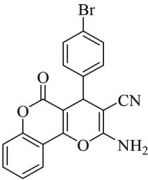
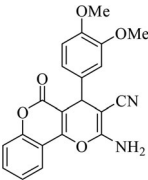
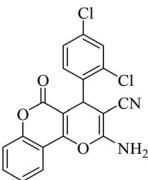
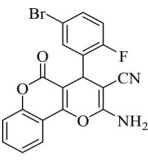
We explored the analogous reactions with dimedone as the dicarbonyl partner. These reactions were rapid and high-yielding (Table 1, Entries 11, 12, 13 and 14). The reactions were remarkably clean and no column chromatographic purification was required. Authenticity of all of the products was established by comparing their melting points with those reported in literature cited in the table and by analysis of spectral data, viz., ¹H NMR, ¹³C NMR, IR and mass spectra. For novel compounds, satisfactory elemental analyses were obtained.

In summary, we have found a simple and convenient procedure for the one-pot MCR synthesis of pyran annulated heterocyclic compounds under catalyst free conditions using aqueous ethylene glycol. In the preparation of these useful compounds, the method offers ease of operation and high yields, as well as adhering to the green principle of eliminating the need for a catalyst.

Experimental section

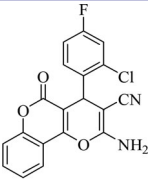
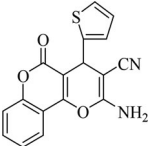
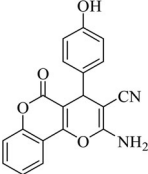
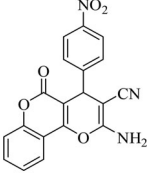
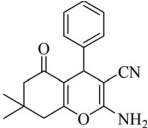
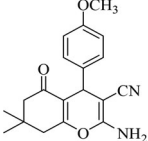
Chemicals were purchased from SD Fine or Sigma Aldrich and used without further purification. Reactions were monitored on silica-gel coated aluminium TLC plates

Table 1. Yields of aqueous ethylene glycol promoted synthesis of pyran annulated heterocyclic compounds.

Entry	X	1,3-Dicarbonyl	Product	Time (h)	Yield (%) ¹	mp (°C) Obs. [Lit.] [Ref.]
1	H	4-Hydroxycoumarin		3	89	255-256 [255-256] [19]
2	4-Cl	4-Hydroxycoumarin		3.5	90	258-260 [260-262] [18]
3	4-Br	4-Hydroxycoumarin		3.5	86	252-254 [250-252] [31]
4	3,4-(OMe) ₂	4-Hydroxycoumarin		4	89	229-230 [230-233] [18]
5	2,4-Cl ₂	4-Hydroxycoumarin		4	87	258-259 [258-260] [31]
6	5-Br, 2-F	4-Hydroxycoumarin		5	84	231-233 [P] ²

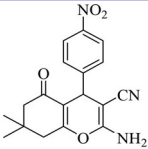
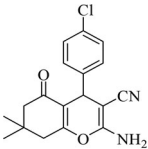
(continued)

Table 1. Continued.

Entry	X	1,3-Dicarbonyl	Product	Time (h)	Yield (%) ¹	mp (°C) Obs. [Lit.] [Ref.]
7	2-Cl,4-F	4-Hydroxycoumarin	 5g	5	88	256-257 [P]
8	2-Thiophene	4-Hydroxycoumarin	 5h	4	82	219-220 [220-222] [32]
9	4-OH	4-Hydroxycoumarin	 5i	4.5	80	265-266 [265-267] [21]
10	4-NO ₂	4-Hydroxycoumarin	 5j	3	90	257-259 [258-260] [23]
11	H	Dimedone	 5k	3	90	230-232 [232-233] [18]
12	4-OMe	Dimedone	 5l	3.5	88	201-202 [198-200] [32]

(continued)

Table 1. Continued.

Entry	X	1,3-Dicarbonyl	Product	Time (h)	Yield (%) ¹	mp (°C) Obs. [Lit.] [Ref.]
13	4-NO ₂	Dimedone	 5m	3	92	178-180 [176-177] [32]
14	4-Cl	Dimedone	 5n	3.5	87	215-216 [217-218] [18]

¹Isolated yields from reactions of aldehyde (2 mmol), malononitrile (2 mmol) and dimedone or 4-hydroxycoumarin (2 mmol) in EG:H₂O (1:1) (2 mL) at 100 °C.

²[P] present work.

(Merck) in ethyl acetate: n-hexane as the mobile phase. Melting points of the products were determined on a digital melting point apparatus (Optics Technology) using capillaries open at one end and were uncorrected. ¹H (400 MHz) and ¹³C (100 MHz) NMR spectra were recorded on a Bruker ACF 200 spectrometer and chemical shifts were reported on δ ppm scale in the specified solvents. First order coupling constants were in the range of 5-7 Hz and are not individually reported. IR spectra were recorded on Shimadzu FTIR spectrophotometer (Prestige-IR21) using KBr pellets. Mass spectra were recorded on Thermo Finnigan Surveyor MS-Q spectrometer.

General procedure for the aqueous ethylene glycol promoted synthesis of 3,4-dihydropyrano[c]chromenes and tetrahydrobenzo[b]pyrans

A mixture of aldehyde (2 mmol) and malononitrile (2 mmol) in (1:1) ethylene glycol: H₂O (2 mL) was stirred at room temperature for 10 minutes. An appropriate amount of 4-hydroxycoumarin or dimedone (2 mmol) was then added and the reaction mixture was refluxed at 100 °C for the specified time (Table 1). The progress of the reaction was monitored by TLC in 50% ethyl acetate:n-hexane in the case of dihydropyrano[c]chromenes and 30% ethyl acetate:n-hexane in the case of tetrahydrobenzo[b]pyrans. After completion of the reaction as monitored by TLC, the reaction mixture was poured into ice cold water. The resulting precipitated solid was collected by filtration, washed with cold water (3 x 5 mL) to remove any traces of ethylene glycol and further purified by recrystallization from ethanol. Many of the products were known compounds, identified by comparison of their melting points with those in the literature cited in Table 1. Complete characterization of novel compounds (5f and 5g) and examples of representative spectroscopic data for known compounds are provided below.

Table 2. Optimization of reaction conditions for the model reaction of 4-chlorobenzaldehyde, malononitrile and 4-hydroxycoumarin.

Entry	Reaction conditions	Yield (%) ^a
1	H ₂ O, 100 °C, reflux, 5h	37
2	Dioxane : H ₂ O (1:1), 100 °C, reflux, 5 h	Trace
3	EtOH: H ₂ O (1:1), 80 °C, reflux, 5h	64
4	MeOH: H ₂ O (1:1), 70 °C, reflux, 5h	70
5	IPA: H ₂ O (1:1), 80 °C, reflux, 5h	40
6	Acetone : H ₂ O (1:1), 60 °C, reflux, 5	58
7	ACN : H ₂ O (1:1), 85 °C, reflux, 5h	25
8	DMSO : H ₂ O (1:1), 100 °C, reflux, 5h	30
9	THF: H ₂ O (1:1), 70 °C, reflux, 5 h	40
10	Triethanol amine, 80 °C, reflux, 5h	80
11	EG, 100 °C, reflux, 5h	61
12	Glycerol : H ₂ O (1:1), 100 °C, reflux, 3.5 h	78
13	PEG : H ₂ O (1:1), reflux, 4 h	87-91 [ref 33]
14	EG : H ₂ O (1:1) , reflux, 3.5 h	90 [Present work]

^aYields for the model reaction of 4-chlorobenzaldehyde (1 mmol), malononitrile (1 mmol) and 4-hydroxycoumarin (1 mmol).

2-Amino-4-(4-bromophenyl)-4, 5-dihydro-5-oxopyrano [3, 2-c]-chromene-3-carbonitrile (5c)

M.P. = 252-254 °C; ¹H NMR (400 MHz, DMSO-d₆): δ = 7.6-7.9 (brs, 2H, -NH₂), 7.45 (m, 6H), 7.25 (s, 2H), 4.51 (s, 1H) ppm; ¹³C NMR (100.6 MHz, DMSO-d₆) δ = 159.53, 157.95, 153.54, 152.16, 142.71, 132.99, 131.33, 129.97, 124.66, 122.51, 120.20, 116.58, 112.93, 103.43, 57.40 ppm; IR (KBr) ν = 3400, 3305, 2205, 1710, 1650, 1502, 1460, 1211, 1070, 696 cm⁻¹; MS (70 eV): *m/z* = 395.2 (M + 1)⁺.

2-Amino-4-(2, 4-dichlorophenyl)-4, 5-dihydro-5-oxopyrano[3,2-c]-chromene-3-carbonitrile (5e)

M.P. = 256-257 °C; ¹H NMR (400 MHz, DMSO-d₆): δ = 7.9 (d, 1H), 7.71 (t, 1H), 7.59 (s, 1H), 7.49 (m, 4H), 7.35 (m, 2H), 4.95 (s, 1H) ppm; ¹³C NMR (100.6 MHz, DMSO-d₆) δ = 159.38, 158.10, 154.13, 152.21, 139.39, 133.33, 133.10, 132.66, 132.35, 128.83, 127.84, 124.72, 122.51, 118.64, 116.61, 112.79, 102.45, 55.98 ppm; IR (KBr) ν = 3464, 3298, 3066, 2200, 1712, 1660, 1060, 750 cm⁻¹; MS (70 eV): *m/z* = 384.7 (M + 1)⁺.

2-Amino-4-(5-bromo-2-fluorophenyl)-4,5-dihydro-5-oxopyrano[3,2-c]chromene-3-carbonitrile (5f)

M.P. = 231-233 °C; ¹H NMR (400 MHz, DMSO-d₆): δ = 7.6-7.95 (brs, 2H, -NH₂), 7.15 (s, 1H), 5.91 (m, 6H), 4.75 (s, 1H) ppm; ¹³C NMR (100.6 MHz, DMSO-d₆) δ = 159.51, 158.26, 154.16, 152.21, 133.05, 132.66, 132.43, 132.29, 132.05, 131.97, 124.67, 122.51, 118.87, 117.79, 116.32, 112.92, 101.84, 55.75 ppm; IR (KBr) ν = 3458, 3225, 2904, 2198, 1695, 1604, 1560, 1053, 761 cm⁻¹; MS (70 eV): *m/z* = 412.5 (M + 1)⁺.

Anal. Calcd for C₁₉H₁₀BrFN₂O₃: C, 55.23; H, 2.44; N, 6.78. Found: C, 55.38; H, 2.56; N, 6.83.

2-Amino-4-(2-chloro-4-fluorophenyl)-4,5-dihydro-5-oxopyrano[3,2-c]chromene-3-carbonitrile (5g)

M.P. = 256-257 °C; ¹H NMR (400 MHz, DMSO-d₆): δ = 7.6-7.9 (brs, 2H, NH₂), 7.45 (m, 6H), 7.12 (s, 1H), 4.99 (s, 1H) ppm; ¹³C NMR (100.6 MHz, DMSO-d₆): δ = 161.93,

159.47, 158.07, 154.04, 152.19, 136.66, 133.13, 132.18, 124.69, 122.50, 118.70, 116.59, 116.38, 114.98, 114.77, 112.82, 102.64, 56.27 ppm; IR (KBr) ν = 3453, 3208, 2910, 2186, 1684, 1603, 1553, 1052, 708 cm^{-1} ; MS (70 eV): m/z = 370.08 ($M + 1$)⁺.

Anal. Calcd for: C, 61.89; H, 2.73; N, 7.60. Found: C, 61.62; H, 2.85; N, 7.42.

2-Amino-5,6,7,8-tetrahydro-7,7-dimethyl-5-oxo-4-phenyl-4H-chromene-3-carbonitrile (5k)

M.P. = 230-232 °C; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 7.32 (t, 2H), 7.16 (m, 3H), 7.10 (s, 2H, -NH₂), 4.19 (s, 1H), 2.52 (s, 2H), 2.24 (dd 1H), 2.12 (dd, 1H), 1.04 (s, 3H, -CH₃), 0.96 (s, 3H, -CH₃) ppm; ¹³C NMR (100.6 MHz, DMSO-*d*₆): δ = 195.61, 162.46, 158.47, 144.72, 128.30, 127.12, 126.54, 119.69, 112.73, 58.30, 35.56, 31.77, 28.38, 26.78 ppm; IR (KBr) ν = 3390, 3250, 3195, 2200, 1685 cm^{-1} ; MS (70 eV): m/z = 295 ($M + 1$)⁺.

2-Amino-5,6,7,8-tetrahydro-4-(4-methoxyphenyl)-7,7-dimethyl-5-oxo-4H-chromene-3-carbonitrile (5l)

M.P. = 201-202 °C; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 7.07 (dd, 2H), 6.97 (s, 2H, -NH₂), 6.83 (dd, 2H), 4.71 (s, 1H), 3.76 (s, 3H, -OCH₃), 2.5 (s, 2H), 2.23 (d, 1H), 2.1 (d, 1H), 1.03 (s, 3H, -CH₃), 0.98 (s, 3H, -CH₃) ppm; ¹³C NMR (100.6 MHz, DMSO-*d*₆): δ = 195.59, 162.08, 158.41, 157.90, 136.82, 128.19, 119.75, 113.65, 113.01, 58.62, 34.75, 31.73, 28.39, 26.76 ppm; IR (KBr) ν = 3320, 2970, 2199, 1688, 1641, 1605, 1508, 1370, 1213, 889 cm^{-1} .

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