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One-pot multi-component process for the synthesis of 4-azaphenanthrene-3,10-dione, 1,8-dioxo-octahydroxanthene and tetrahydrobenzo[*b*]pyran derivatives catalyzed by the deep eutectic solvent choline chloride-oxalic acid

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Abstract: An effective method based on choline chloride (ChCl)-oxalic acid (Ox) deep eutectic solvent was proposed for the synthesis of 4-azaphenanthrene-3,10-dione, 1,8-dioxo-octahydroxanthene and tetrahydrobenzo[*b*]pyran derivatives. The eutectic mixture worked as both the solvent and acidic catalyst for conversion. The impacts of different variables, including the composition and volume of ChCl-Ox, and temperature, on reaction yield were studied for optimization. The crucial advantages of this process are simplicity of the experimental procedure, high yields, short reaction times, high recyclability, and the use of safe and inexpensive components.

Keywords: 4-azaphenanthrene-3,10-dione; deep eutectic solvent; dioxo-octahydroxanthene; multi-component reactions; solvent-free synthesis; tetrahydrobenzo[*b*]pyran.

1 Introduction

A multi-component reaction (MCR) is a convergent reaction with at least three components to form a single product, which incorporates most or even all of the starting materials. MCRs have an important role in the synthesis of different types of organic compounds [1–3]. In recent years, 4*H*-pyran and 2-pyridone derivatives have been considered as remarkable compounds because of their pharmaceutical activities [4–9]. They exhibit a wide range of biological properties, including antifungal, antiviral, antibiotic, anti-HIV and antitumor [10–14].

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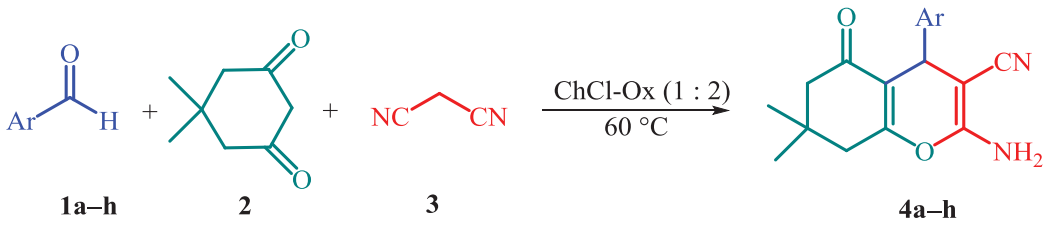
A number of methods are available in literature for the synthesis of these heterocycles. The acid-catalyzed condensation of dimedone with aromatic aldehydes to get 1,8-dioxo-octahydroxanthenes seems to be a highly efficient reaction [4, 15]. It is worthy to mention that, with the addition of malononitrile to the reaction mixture, the preparation of tetrahydrobenzo[*b*]pyran scaffolds would be possible [16, 17]. The conventional synthetic method for the preparation of 4-azaphenanthrene-3,10-diones involves a four-component condensation of Meldrum's acid, an aldehyde, primary amine or ammonium acetate and different 1,3-dicarbonyl compounds under different conditions [8, 9].

A deep eutectic solvent (DES) is a fluid generally composed of two or three cheap and safe components that are capable of associating with each other through hydrogen bond interactions. DESs typically have freezing points lower than those of starting individual components. One of the most widespread components used for the formation of DES is choline chloride (ChCl), an inexpensive, chemically and thermally stable, biodegradable, nontoxic and recyclable quaternary ammonium salt [18–20]. ChCl is capable of rapidly forming a DES in combination with hydrogen donors such as acids [21], alcohols [22], amines [23] or amides [24]. DES based on ChCl and organic Brønsted acid is a well-known system for synthesizing different heterocycles acting both as a solvent and a Brønsted-acidic catalyst [25].

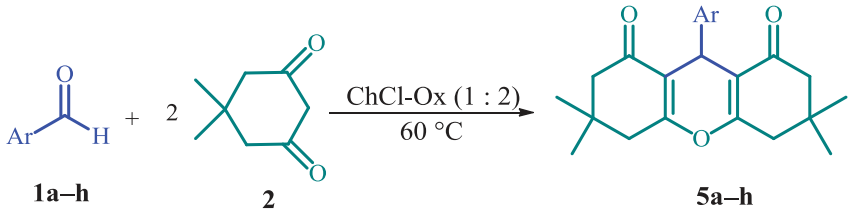
In continuation of our research work on the applications of green catalysts for organic reactions [26–29], we have carried out the synthesis of tetrahydrobenzo[*b*]pyrans (Scheme 1), dioxo-octahydroxanthenes (Scheme 2) and 4-azaphenanthrene-3,10-diones (Scheme 3) using the DESs ChCl and oxalic acid (ChCl-Ox), an ionic liquid, at room temperature.

2 Results and discussion

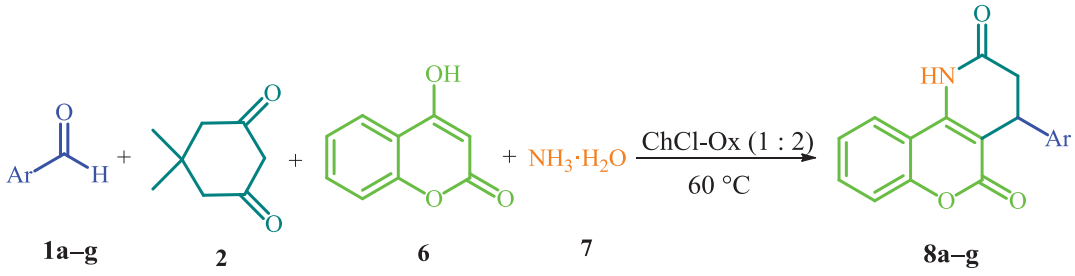
As stated before, DESs are cheap ionic liquid-like green solvents. In this work the DES ChCl-Ox was selected where Ox works not only as the hydrogen bond donor but also as the catalyst for the MCR. In order to study the



Scheme 1: Synthesis of tetrahydrobenzopyran derivatives **4a–h** from aromatic aldehydes **1a–h**, dimedone (**2**) and malononitrile (**3**) using ChCl-Ox.



Scheme 2: Synthesis of 1,8-dioxo-octahydroxanthene derivatives **5a–h** from aromatic aldehydes **1a–h** and dimedone (**2**) using ChCl-Ox.



Scheme 3: Synthesis of 4-azaphenanthrene-3,10-dione derivatives **8a–g** from aromatic aldehydes **1a–g** with 4-hydroxycoumarin (**6**), ammonia solution and dimedone (**2**) using ChCl-Ox.

catalytic efficiency of ChCl-Ox, we used 1 mmol of 4-chlorobenzaldehyde (**1**, Ar = 4-Cl–C_6H_4), 1 mmol of dimedone (**2**) and 1 mmol of malononitrile (**3**) as a model reaction for the synthesis of 2-amino-3-cyano-4-(4-chlorophenyl)-7,7-dimethyl-5-oxo-4*H*-5,6,7,8-tetrahydrobenzo[*b*]pyran (**4c**; Scheme 1, see Table 2 for formula). The reaction was carried out in different sets of conditions, and the effects of the ratio of ChCl to Ox, amount of catalyst, time and temperature on the yield were studied. To investigate the effect of molar ratio, the ammonium salt ChCl was mixed with Ox at different molar ratios (Table 1, entries 1–4). As is shown in Table 1, by increasing the molar ratio of ChCl to Ox to 1:2, the yield of the desired product increased to 92%. Therefore, this molar ratio of ChCl to Ox was selected as the best DES composition and was used for the following optimization experiments. To optimize the required catalyst amount, the model reaction was carried out in the presence of various amounts of the catalyst, and according to the obtained results, 0.5 mL was chosen as the best catalyst amount (Table 1, entries 5–7). A further increase of the amount of catalyst had no pronounced effect on the yield, indicating that the acidity requirement is well fulfilled by this amount of catalyst (Table 1, entry 8). The effect of the reaction temperature was also studied by performing the model reaction at different temperatures varying from room temperature to 80°C, again using 0.5 mL of the DES. It was found that the condensation reaction proceeded efficiently when the temperature was increased to 60°C (Table 1, entries 9–13). Other ChCl-based DESs such as ChCl-malonic acid, ChCl-glycine, ChCl-citric acid and ChCl-urea were also examined. The results clearly show that ChCl-Ox is the optimal solvent and catalyst for the reaction (Table 1, entries 13–17). It is worth to note that in a blank reaction without a catalyst (entry 18) only trace amounts of the corresponding product were obtained even after prolonged reaction times (2 h).

With these results in hand, we extended our studies to the reaction of dimedone and malononitrile with a variety

Table 1: Investigation of the DES in the synthesis of 2-amino-3-cyano-4-(4-chlorophenyl)-7,7-dimethyl-5-oxo-4*H*-5,6,7,8-tetrahydrobenzo[*b*]pyran **4c** under different conditions.

No.	Catalyst (molar ratio)	Catalyst amount (mL)	Temp. (°C)	Time (min)	Yield[a] (%)
1	ChCl:oxalic acid (2:1)	0.5	60	15	60
2	ChCl:oxalic acid (1:1)	0.5	60	15	55
3	ChCl:oxalic acid (1:1.5)	0.5	60	15	80
4	ChCl:oxalic acid (1:2)	0.5	60	15	92
5	ChCl:oxalic acid (1:2)	0.1	60	15	35
6	ChCl:oxalic acid (1:2)	0.3	60	15	68
7	ChCl:oxalic acid (1:2)	0.4	60	15	81
8	ChCl:oxalic acid (1:2)	0.6	60	15	93
9	ChCl:oxalic acid (1:2)	0.5	Rt	120	Trace
10	ChCl:oxalic acid (1:2)	0.5	40	15	65
11	ChCl:oxalic acid (1:2)	0.5	50	15	81
12	ChCl:oxalic acid (1:2)	0.5	60	15	92
13	ChCl:oxalic acid (1:2)	0.5	70	15	92
14	ChCl:citric acid (1:2)	0.5	60	15	65
15	ChCl:glycine (1:2)	0.5	60	15	50
16	ChCl:malonic acid (1:2)	0.5	60	15	82
17	ChCl:urea (1:2)	0.5	60	15	45
18	None	–	60	120	Trace

[a]Isolated yields.

of electron-donating and electron-withdrawing aldehydes to evaluate the scope of this methodology (Scheme 1). The results are presented in Table 2. It is found that substituents in the aromatic ring of the aldehydes have a minor effect on the reaction times. Aromatic aldehydes with electron-withdrawing groups reacted faster than those with electron-donating groups.

The scope and generality of this catalytic system was shown in the reaction of various types of aromatic aldehydes with dimedone (Scheme 2). When the reaction was carried out using one equivalent of aromatic aldehyde and two equivalents of dimedone in the presence of 0.5 mL of ChCl-Ox under the same reaction conditions used in the synthesis of the tetrahydrobenzopyran derivatives **4a–h**, 1,8-dioxo-octahydroxanthene derivatives **5a–h** were obtained in good to excellent yields (Table 3). The progress of the reaction was monitored by thin-layer chromatography (TLC) (EtOAc–hexane, 4:1, v/v). Work-up of the reaction was very easy. After completion of the reaction, simple filtration of the reaction mixture provided the crude product. Evaporation of the solvent under reduced pressure and recrystallization gave the final pure product.

To show the further applicability of this catalytic system, the synthesis of 4-azaphenanthrene-3,10-dione derivatives **8a–g** was investigated (Scheme 3, Table 4). The reaction of aromatic aldehydes with 4-hydroxycoumarin, ammonia solution and dimedone in the presence of catalytic amounts of ChCl-Ox (0.5 mL) produced the corresponding 1-phenyl-1,4-dihydro-2*H*-9-oxa-4-aza-phenanthrene-3,10-diones in good to excellent yields in short reaction times.

In general, the mechanism of such synthetic procedures involves the Knoevenagel reaction, Michael addition and intra-molecular cyclization. A proposed mechanism to demonstrate the role of catalyst for the synthesis of tetrahydrobenzo[*b*]pyran is shown in Scheme 4. The arylidenemalononitrile intermediate (I) was formed initially by the Knoevenagel condensation between activated aldehyde and malononitrile in the presence of ChCl-Ox. Subsequently, the Michael addition of the enolizable dimedone to the arylidenemalononitrile intermediate (I), followed by intramolecular cyclization and final tautomerization of intermediates, afforded the desired product (Scheme 4). A similar mechanism may occur for the formation of 4-azaphenanthrene-3,10-dione and 1,8-dioxo-octahydroxanthene derivatives.

In environmentally friendly methodologies, the recovery of the prepared catalyst is highly preferable. For this purpose, the model reaction was selected again to investigate the reusability of the DES catalyst under the optimal conditions. After completion of the reaction, the crude product was separated by simple filtration. The DES catalyst was recovered by removing the aqueous layer under vacuum from the filtrate. As is shown in Fig. 1, the DES

Table 2: Three-component reaction of aromatic aldehydes **1a–h**, dimedone (**2**) and malononitrile (**3**) to give tetrahydrobenzopyran derivatives **4a–h**.

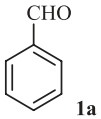
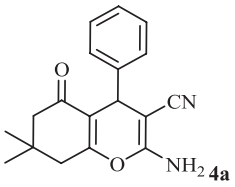
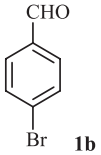
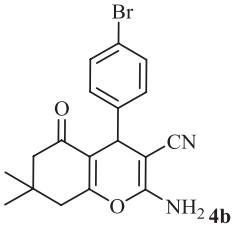
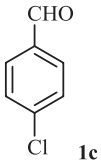
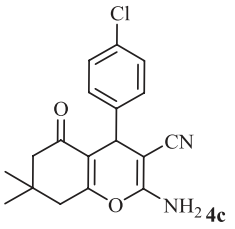
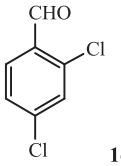
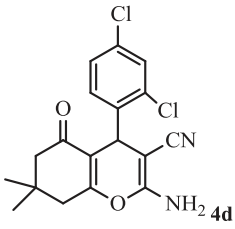
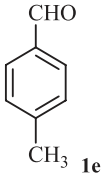
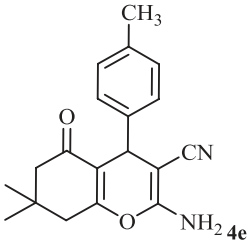
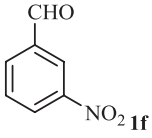
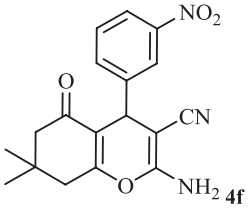
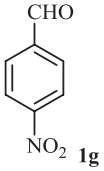
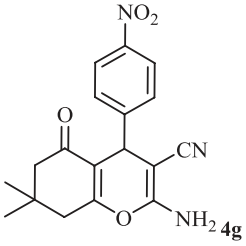
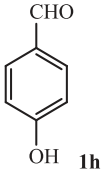
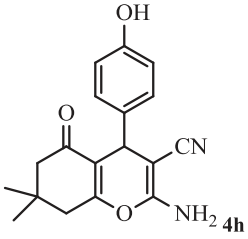
No.	Substrate	Product	Time (min)	Yield[a] (%)	M.p. (°C) [ref.]
1	 **1a**	 **4a**	20	87	229–231 [30]
2	 **1b**	 **4b**	15	92	201–203 [31]
3	 **1c**	 **4c**	15	92	211–213 [30]
4	 **1d**	 **4d**	15	88	118–120 [30]
5	 **1e**	 **4e**	25	90	218–220 [30]
6	 **1f**	 **4f**	20	85	210–212 [30]

Table 2 (continued)

No.	Substrate	Product	Time (min)	Yield[a] (%)	M.p. (°C) [ref.]
7	 **1g**	 **4g**	15	87	178–180 [30]
8	 **1h**	 **4h**	30	85	205–207 [31]

[a]Isolated yields.

could be recycled and reused up to four times with only a slight decrease in catalytic activity.

3 Conclusion

In summary, we have developed a novel multi-component approach for the one-pot synthesis of 4-azaphenanthrene-3,10-dione, 1,8-dioxo-octahydroxanthene and tetrahydrobenzo[*b*]pyran derivatives using a deep eutectic mixture of ChCl and Ox in a molar ratio of 1:2 as a catalyst. High yields, an environmentally benign solvent, the good reusability of the catalyst and simple reaction conditions are the noteworthy aspects of the protocol.

4 Experimental section

4.1 Materials and methods

All chemicals were commercial and were used as received. The reactions were monitored by TLC on silica gel polygram SILG/UV 254 plates. The yields of products refer to isolated compounds. Melting points were determined using Stuart scientific apparatus. FT-IR spectra were obtained as potassium bromide pellets in the range of 400–4000 cm^{-1} using a BOMEM MB-Series 1998 FT-IR spectrophotometer. The ^{1}H and ^{13}C NMR spectra of samples were recorded with a Bruker Advanced DPX 400-MHz.

4.2 Preparation of a deep eutectic solvent

In this study, the eutectic mixtures of ChCl and Ox were synthesized at molar ratios of 2:1, 1:1 and 1:2 and utilized as solvents and catalyst for the one-pot synthesis of tetrahydrobenzo[*b*]pyran (**4a–h**), 1,8-dioxo-octahydroxanthene (**5a–h**) and 4-azaphenanthrene-3,10-dione derivatives (**8a–h**). To prepare these mixtures, ChCl and Ox were mixed at $T = 60°C$ for approximately 15 min until a homogeneous, colorless liquid formed. The obtained DES was used without any further purification.

4.3 General procedure for the synthesis of tetrahydrobenzo[*b*]pyrans 4a–h

Aldehyde (1 mol), dimedone (1 mol), malononitrile (1 mol) and DES (0.5 mL) as catalysts were mixed and heated at 60°C for 15–30 min. After completion of the reaction monitored by TLC, water (5 mL) was added and the solid product was filtered and washed with H_2O. Then hot EtOH was poured on the precipitate until the product was solved. Finally, products were recrystallized for more purification (Table 2).

Table 3: Three-component reaction of aromatic aldehydes and dimedone to give 1,8-dioxo-octahydroxanthene derivatives **5a–h**.

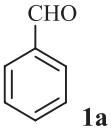
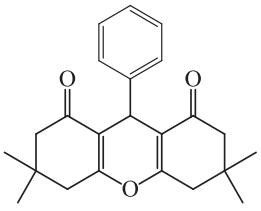
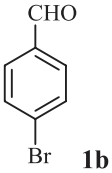
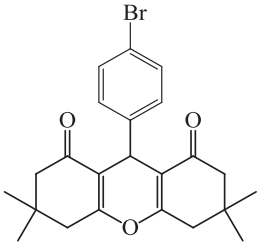
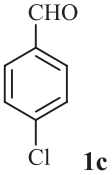
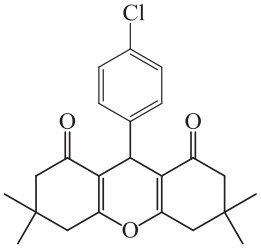
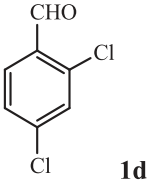
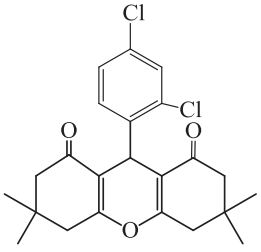
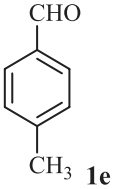
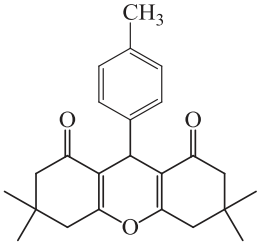
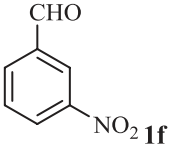
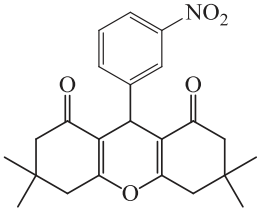
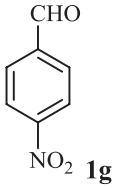
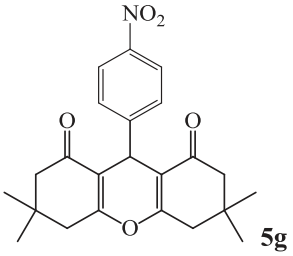
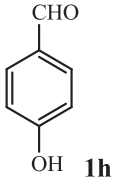
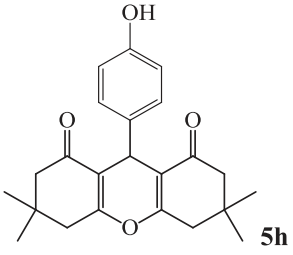
No.	Substrate	Product	Time (min)	Yield[a] (%)	M.p. (°C) [ref.]
1	 **1a**	 **5a**	15	90	200–202 [32]
2	 **1b**	 **5b**	20	92	232–234 [31]
3	 **1c**	 **5c**	15	93	225–227 [31]
4	 **1d**	 **5d**	**10**	88	240–243 [31]
5	 **1e**	 **5e**	20	90	210–212 [31]
6	 **1f**	 **5f**	20	85	219–222 [31]

Table 3 (continued)

No.	Substrate	Product	Time (min)	Yield[a] (%)	M.p. (°C) [ref.]
7	 **1g**	 **5g**	10	90	220–222 [31]
8	 **1h**	 **5h**	25	85	242–244 [31]

[a]Isolated yields.

4.4 General procedure for the synthesis of 1,8-dioxo-octahydroxanthene 5a–h

Aromatic aldehydes (1 mmol), dimedone (2 mol) and ChCl-Ox (0.5 mL) were added into a 5 mL round-bottom flask and were stirred at 60°C for appropriate time according to Table 3. The progress of the reaction was monitored by TLC. After completion of the reaction, water (5 mL) was added and the solid was separated by filtration. The crude products were obtained in high purity after purification by recrystallization from ethanol. All the products were known compounds and were characterized by comparing spectroscopic data and their melting points to the literature values.

4.5 General procedure for the preparation of 4-azaphenanthrene-3,10-diones 8a–g

To a mixture of 4-hydroxycoumarin (1 mmol), 28–30% ammonia solution (5 mmol), dimedone (1 mmol) and aromatic aldehyde (1 mmol), ChCl-Ox (0.5 mL) was added. The mixture was stirred at 60°C for appropriate time according to Table 4. After completion of the reaction, as indicated by TLC, H_2O (5 mL) was added to the reaction mixture and then the precipitate was filtered off and washed with EtOH and water. Finally, the crude product was purified by recrystallization with EtOH to afford the corresponding products in high yields.

4.6 Characterization data for compounds 8c, 8d, 8g and 4e

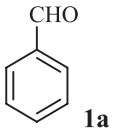
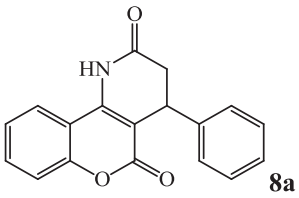
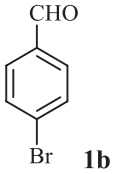
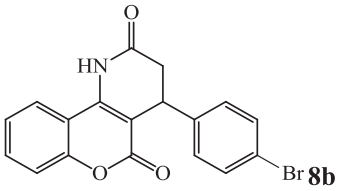
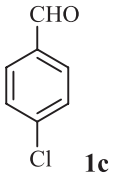
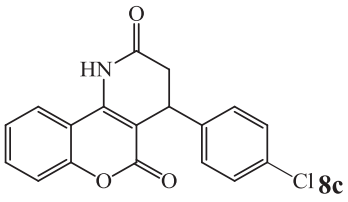
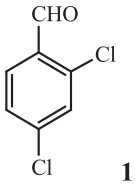
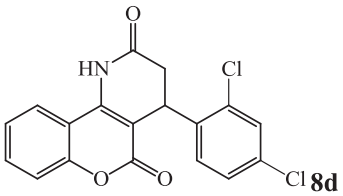
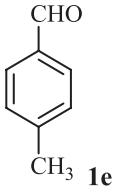
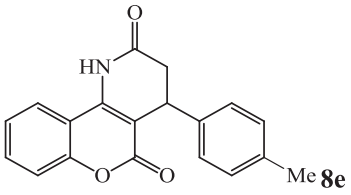
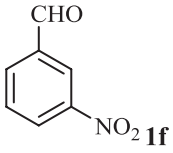
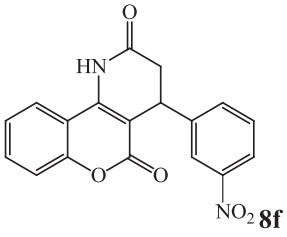
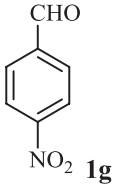
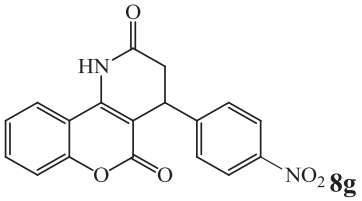
4.6.1 1-(4-Chloro-phenyl)-1,4-dihydro-2*H*-9-oxa-4-aza-phenanthrene-3,10-dione (8c)

White powder; m.p. 218–220°C. – FT-IR (KBr, cm^{-1}): ν = 3185, 3139, 2923, 1690, 1637, 1605, 1571, 1515, 1464, 1407, 1368, 1318, 1240, 1195, 1012, 999, 829, 769, 739, 627, 547. – ^{1}H NMR (DMSO-d_6, 500 MHz, ppm): δ = 2.58 (dd, 1H), 3.32 (d, 1H), 4.37 (d, 1H), 7.20–8.24 (m, ArH, 8H), 11.01 (s, NH, 1H). – ^{13}C NMR (DMSO-d_6, 125 MHz, ppm): δ = 171.6, 160.1, 152.7, 147.2, 141.7, 134.0, 133.1, 130.2, 130.0, 125.7, 124.6, 118.4, 114.5, 104.4, 37.05, 36.4.

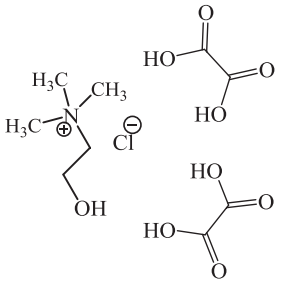
4.6.2 1-(2,4-Dichloro-phenyl)-1,4-dihydro-2*H*-9-oxa-4-aza-phenanthrene-3,10-dione (8d)

White powder; m.p. 185–187°C. – FT-IR (KBr, cm^{-1}): ν = 3239, 3038, 2923, 1722, 1692, 1671, 1633, 1609, 1574, 1519, 1463, 1363, 1324, 1200, 1165, 1049, 821, 766, 748, 630, 598, 480. – ^{1}H NMR (DMSO-d_6, 500 MHz, ppm): δ = 2.53 (dd,

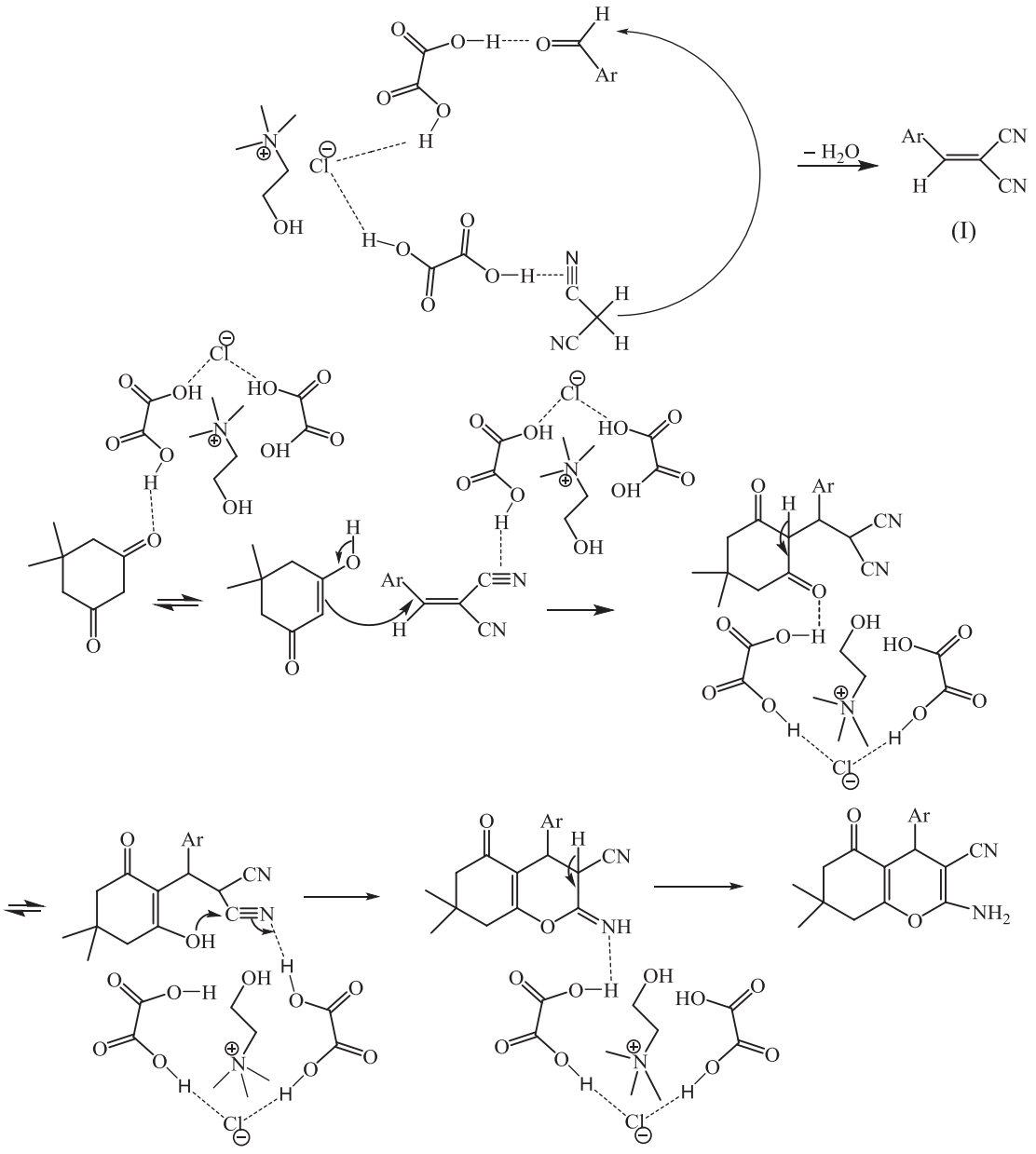
Table 4: Four-component reaction of 4-hydroxycoumarin (**6**), ammonia solution, dimedone (**2**) and aromatic aldehydes **1a–g** to give 1,8-dioxo-octahydroxanthene derivatives **8a–g**.

No.	Substrate	Product	Time (min)	Yield[a] (%)	M.p. (°C) [ref.]
1	 **1a**	 **8a**	20	90	184–186 [33]
2	 **1b**	 **8b**	15	92	201–203 [33]
3	 **1c**	 **8c**	15	90	218–220
4	 **1d**	 **8d**	15	88	185–187
5	 **1e**	 **8e**	30	90	184–186 [33]
6	 **1f**	 **8f**	25	85	192–194 [33]
7	 **1g**	 **8g**	15	90	201–203

[a]Isolated yields.



Deep eutectic solvent of choline chloride and oxalic acid in the molar ratio of 1 : 2



Scheme 4: Possible reaction pathway for the ChCl-Ox-catalyzed synthesis of tetrahydrobenzo[*b*]pyran derivatives **4a–h**.

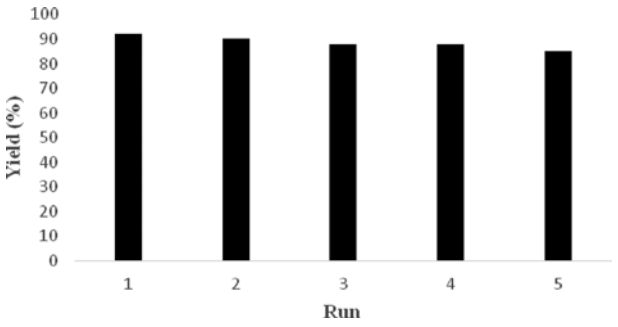


Fig. 1: The reusability of the solvent/catalyst ChCl-Ox.

1H), 3.33 (d, 1H), 4.65 (d, 1H), 7.20–8.24 (m, ArH, 7H), 11.13 (s, NH, 1H). – ^{13}C NMR (DMSO-d_6, 125 MHz, ppm): δ = 170.9, 161.4, 154.3, 148.4, 138.3, 134.9, 134.2, 131.0, 130.2, 129.3, 125.8, 124.8, 118.4, 114.5, 103, 37.6, 34.4.

4.6.3 1-(4-Nitro-phenyl)-1,4-dihydro-2*H*-9-oxa-4-aza-phenanthrene-3,10-dione (8g)

White powder; m.p. 201–203°C. – FT-IR (KBr, cm^{-1}): ν = 3265, 3078, 1723, 1690, 1684, 1625, 1511, 1459, 1411, 1348, 1277, 1231, 1196, 1155, 1112, 1070, 1004, 946, 906, 858, 758, 730, 632, 518. – ^{1}H NMR (DMSO-d_6, 500 MHz, ppm): δ = 2.89 (dd, 1H), 3.33 (d, 1H), 4.52 (d, 1H), 7.20–8.27 (m, ArH, 8H), 11.10 (s, NH, 1H). – ^{13}C NMR (DMSO-d_6, 125 MHz, ppm): δ = 171.3, 161.6, 154.3, 150.6, 148.1, 147.6, 134.1, 129.6, 125.7, 125.4, 124.7, 118.4, 114.5, 103.6, 38.9, 36.9.

4.6.4 2-Amino-3-cyano-4-(4-methylphenyl)-7,7-dimethyl-5-oxo-4*H*-5,6,7,8-tetrahydrobenzo[*b*]pyran (4e)

White powder; m.p. 218–220°C. – FT-IR (KBr, cm^{-1}): ν = 3425, 3321, 2995, 2187, 1672, 1638, 1598, 1484, 1365. – ^{1}H NMR (DMSO-d_6, 500 MHz, ppm): δ = 0.89 (s, 3H), 0.96 (s, 3H), 1.89 (d, 1H), 2.03 (d, 1H), 2.12 (s, 3H), 2.33 (s, 2H), 4.13 (s, 1H), 5.8 (s, NH, 2H), 6.90–9.96 (m, ArH, 4H). – ^{13}C NMR (DMSO-d_6, 125 MHz, ppm): δ = 196.0, 162.7, 158.8, 142.2, 136.0, 129.3, 127.5, 120.1, 113.2, 58.8, 50.4, 39.8, 35.5, 32.2, 28.8, 27.1, 21.0.

5 Supplementary information

Characterization of the products **8c**, **8d**, **8g** and **4e** is given as Supplementary information available online (http://dx.doi.org/10.1515/znb-2019-0155).

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Supplementary Material: The online version of this article offers supplementary material (https://doi.org/10.1515/znb-2019-0155).