DABCO: An Organo-Base Catalyzed One-Pot Multicomponent Strategy for the Synthesis of 1, 4-Polyhydroquinoline Derivatives.

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Abstract

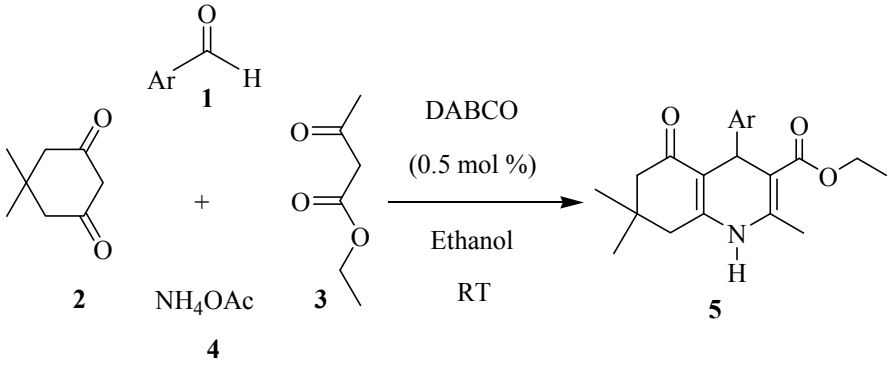
In the report we describes four component as aromatic aldehydes, dimedone, ethyl acetoacetate and ammonium acetate as nitrogen source for the synthesis of a series of 1, 4-polyhydroquinoline derivatives using 1,4-diazabicyclo[2.2.2]octane (DABCO) as catalyst at room temperature in ethanol. This method provides a green and convenient one-pot route for the synthesis of a series of 1,4-polyhydroquinoline derivatives consisting various aryl substituent's. The key advantages of this process are operational simplicity, inexpensive catalyst, short reaction time, simple workup and non-chromatographic purification, which make it an attractive route for the synthesis of a series of 1, 4-polyhydroquinoline derivatives. DABCO has received considerable attention as an inexpensive, eco-friendly, high reactive, easy to handle and non-toxic catalyst for organic transformations as a green organo-base catalyst, and affords good to excellent yields of 1, 4-polyhydroquinoline derivatives.

Keywords: 1, 4-Diazabicyclo[2.2.2]octane (DABCO), environmentally friendly, one pot, Multi-component reaction, polyhydroquinoline derivative.

Introduction

One pot multi-component reactions are important in combinatorial chemistry due to their ability to prepare targeted molecule with more efficiency and atom economy following the concept of green chemistry by the reaction of three or more reactants together in one pot and in a single step. Therefore, the design of novel MCRs has attracted great attention from research groups working in medicinal chemistry and drug chemistry [1]. 1, 4-polyhydroquinolines has diverse applications in medicinal chemistry, as calcium channel blockers, vascodilator, antiatherosclerotic, bronchodilator, antitumor, geroprotective, antidiabetic activity, antiasthmatic, antibacterial antihypertensive anti-inflammatory antimalarial, geroprotective, and tyrosine kinase inhibiting materials [2]. Numerous synthetic methods have been reported for the preparation of 1, 4-polyhydroquinoline derivatives under classical or modified conditions [3-24]. However, the low yields, occurrence of several side products, use of stoichiometric amount of reagents, expensive metal precursors, catalysts that are harmful to environment, use of expensive and toxic transition metallic reagents, complicated work-up methods and longer reaction times limit the use of these methods. Therefore, for the increasing environmental and economic concerns in recent years, it is now essential or chemists to search environmentally reactions under normal reaction conditions as many as possible. In this regard, we report, simple, mild and efficient method for the one-pot synthesis of 1, 4-polyhydroquinoline derivatives using DABCO as a catalyst. In recent years, 1, 4-diazabicyclo [2.2.2] octane (DABCO) has received considerable attention as an inexpensive, eco-friendly, high reactive, easy

to handle and non-toxic catalyst for various organic transformations [25] such as for Bayllis-Hillman reaction [26] the synthesis of naphtopyran [27], benzopyrans [28], 3-cyanopyrimidones [29], dihydropyranochromenes [30], and imidazoles [31], pyridazine derivatives [32]. In the present work we report DABCO as an efficient catalyst to promote rapid one-pot multicomponent reaction between aromatic aldehydes, dimedone, and ethyl acetoacetate and ammonium acetate in ethanol at room temperature to form 1, 4-polyhydroquinoline derivatives. As a part of our interest in DABCO [33], we report DABCO as environmentally friendly catalyst for the synthesis of 1, 4-polyhydroquinoline derivatives under normal reaction conditions. (Scheme 1)



Scheme 1: One pot four component synthesis of, 1, 4-polyhydroquinoline derivatives.

Material and Methods

All the melting points were determined by open capillary method. The purity of compounds was checked by Blaker-Flex silica gel 1B-F (1.55 cm) TLC plates, and the spots were detected by UV light absorption. ^{1}H NMR spectral data was registered on Brucker avance II-400 NMR spectrometer (400 MHz) in $CDCl_3$ using tetramethylsilane as an internal standard. The IR spectra were recorded using KBr disc on Shimadzu FTIR spectrophotometer. The chemicals used are of AR grade from Sd fine and Loba chemicals.

Typical procedure

The mixture of 4-N, N-dimethylbenzaldehyde (5 mmol), dimedone (5 mmol), ethylacetoacetate (5 mmol), ammonium acetate (7.5 mmol) and 1,4-diazabicyclo[2.2.2]octane (DABCO) (0.5 mol%) in ethanol was stirred at room temperature. After complete conversion (monitored by TLC) a solid product obtained was filtered and washed with water. The crude product obtained was recrystallized from ethanol. The MPs are took by open capillary method and matched with authentic samples characterized by IR and ^{1}H NMR spectroscopy.

Ethyl 1, 4, 5, 6, 7, 8-hexahydro-2, 7, 7-trimethyl-5-oxo-4-phenylquinoline-3-carboxylate: **(5a)** Pale Yellow Solid, mp: 204-205 ^{0}C, IR (KBr) $\bar{\upsilon}$ =: 3293, 3058, 2958, 1676, 1610 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 0.93 (s, 3H, CH_3), 1.04 (s, 3H, CH_3), 1.23 (t, *J*=7.1 Hz, 3H, CH_3), 2.22-2.10 (m, 4H, $2CH_2$), 2.30 (s, 3H, CH_3), 4.10 (q, *J*=7.1 Hz, 2H, CH_2), .07 (s, 1H, CH), 7.34-7.08 (m, 5H, CHAr), 7.75 (s, 1H, NH), 7.34-7.08 (m, 5H, CHAr),

Ethyl 1, 4, 5, 6, 7, 8-hexahydro-2, 7, 7-trimethyl-4-(4-chlorophenyl)-5-oxoquinoline-3-carboxylate: **(5b)** Pale yellow solid, mp: 242-244 ^{0}C, IR (KBr) ῡ: 3273, 3201, 3076, 1706, 1648, 1279,1214; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.93 (s, 3H),1.07 (s, 3H), 1.19 (t, J = 7.2 Hz, 3H), 2.12-2.35 (m, 4H),2.37 (m, 3H), 4.06 (q, J = 7.2 Hz, 2H), 5.02 (s, 1H), 6.13 (s,1H), 7.15 (d, J = 8 Hz, 2H), 7.31 (d, J = 8 Hz, 2H).

Ethyl 1, 4, 5, 6, 7, 8-hexahydro-2, 7, 7-trimethyl-4-(4-(dimethylamino)phenyl)-5-oxoquinoline-3-carboxylate: **(5c)** Pale yellow solid, mp: 231–233^{0}C. IR (KBr) ῡ = 3280, 3200, 3076, 2955, 2800, 1686, 1606, 1219 cm^{-1}. ^{1}HNMR (400 MHz, $CDCl_3$): δ = 0.94 (s, 3H, C-CH_3), 1.04 (s, 3H, C-CH_3), 1.33 (t, J = 7.1 Hz, 3H, CH_3CH_2O), 2.18–2.16 (m, 4H, 2 x CH_2), 2.33 (s, 3H, =C-CH_3), 2.85 (s, 6H, $N(CH_3)_2$), 4.07 (q, J = 7.1 Hz, 2H, OCH_2CH_3), 4.94 (s, 1H, Ar-CH), 6.57 (d, J = 8.8 Hz, 2H, Ar-H), 7.15 (d, J = 8.8 Hz, 2H, Ar-H).

Ethyl 1, 4, 5, 6, 7, 8-hexahydro--2, 7, 7-trimethyl-4-(4-methoxyphenyl)-5-oxoquinoline-3-carboxylate: **(5d)** Pale yellow solid, mp: 264-266^{0}C. IR (KBr) ῡ: 3302, 3076, 2959, 1696, 1645, 1610, 1483 cm^{-1}. ^{1}HNMR (400 MHz, $CDCl_3$): δ = 0.95 (s, 3H, CH_3), 1.07 (s, 3H, CH_3), 1.2 (t, 3H, J =7 Hz, OCH_2CH_3), 2.12-2.28 (m, 4H, 2 x CH_2), 2.39 (s, 3H, CH_3), 3.75 (s, 3H, OCH_3), 4.08 (q, 2H, J =7 Hz, OCH_2CH_3), 5.04 (s, 1H, CH), 5.93 (s, 1H, NH), 6.62-7.2 (m, 5H, Ar H).

Ethyl 1, 4, 5, 6, 7, 8-hexahydro-2, 7, 7-trimethyl-4-(4-nitrophenyl)-5-oxoquinoline-3-carboxylate: **(5e)** Pale yellow solid, mp: 240–241^{0}C. IR (KBr) ῡ: 3506, 3282, 3200, 2964, 1676, 1604, 1489, 1306, 1223, 1166, 870,755 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 0.91 (s, 3H), 1.08 (s, 3H), 1.18 (t, J = 7.2 Hz, 3H), 2.19 (d, J = 8.8 Hz, 2H), 2.31 (d, J = 13.6 Hz, 2H), 2.39 (s, 3H), 4.06 (q, J = 7.0 Hz, 2H), 5.17 (s, 1H), 6.56 (br s, 1H, NH), 7.50 (d, J = 8.8 Hz, 2H), 8.09 (d, J = 8.8 Hz,2H),

Ethyl 1, 4, 5, 6, 7, 8-hexahydro-2, 7, 7-trimethyl-4-(2-chlorophenyl)-5-oxoquinoline-3-carboxylate: **(5f)** Pale Yellow Solid, mp: 208-210^{0}C. IR (KBr) ῡ: 3296, 3207, 3092, 1707, 1610, 1156, 830 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 0.91 (s, 3H, C-CH_3), 1.02(s, 3H, C-CH_3), 1.20 - 1.26 (t, *J* = 7.00 Hz, 3H, CH_3-CH_2O), 2.09 - 2.30 (m, 4H, 2 x CH_2), 2.33 (s, 3H, =C-CH_3), 4.01 (q, *J* = 6.4 Hz, 2H, OCH_2CH_3), 4.88 (s, 1H, Ar-CH), 6.35 (s, 1H, NH), 7.01 - 7.20 (m, 4H, ArH).

Ethyl 1, 4, 5, 6, 7, 8-hexahydro-2,7,7-Trimethyl-4-(furan-2-yl)-5-oxoquinoline-3-carboxylate: **(5g)** Pale Yellow Solid, mp:144-146°C, IR (KBr) ῡ : 3344, 1701, 1649, 1485, 1371, 1207, 1120, 727 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.27 (t, 6H, *J*=7.1 Hz, OCH_2CH_3), 2.19 - 2.30 (m, 4H, 2 x CH_2), 2.32 (s, 6H, $2CH_3$), 4.11 (q, *J*=7.1 Hz, 4H, CH_3CH_2O), 5.20 (s, 1H, CH), 6.22-5.94 (m, 3H, CHAr), 7.22 (s, 1H, NH);

Ethyl 1, 4, 5, 6, 7, 8-hexahydro-2, 7, 7-trimethyl-4-(3-chlorophenyl)-5-oxoquinoline-3-carboxylate : (**5h**) Pale yellow solid, mp 210-212^{0}C, IR (KBr) ῡ = 3289, 2948, 1677, 1610, 1150, 755 cm^{-1}, ^{1}H NMR (400 MHz, $CDCl_3$) : δ = 1.1 (s, 3H, C-CH_3), 1.8 (s, 3H, C-CH_3), 1.3 (t, *J* = 7.2 Hz, 3H, CH_3 CH_2O), 1.8-2.8 (m, 4H, 2 x CH_2), 1.7 (s, 3H, =C-CH_3), 4.1 (q, *J* = 6.8 Hz, 2H, OCH_2CH_3), 4.4 (s, 1H, Ar-CH), 5.9 (s, 1H, NH), 6.9 (d, *J* = 8.4Hz, 1H, Ar-H), 7.08 - 7.15 (m, 2H, 2 x Ar-H), 7.28 (d, *J* = 8.4Hz, 1H, Ar-H);

Ethyl 1, 4, 5, 6, 7, 8-hexahydro-4-(4-hydroxy-3-methoxyphenyl)-2, 7, 7-trimethyl-5-oxoquinoline-3-carboxylate: **(5i)** Pale yellow solid, mp: 212–214^{0}C, IR (KBr) ῡ: 3395, 3290, 3072, 2951, 1697, 1639, 1481 cm^{-1}.^{1}H-NMR (400 MHz, $CDCl_3$) : δ = 0.93 (s, 3H, C-CH3). 1.06 (s,3H, C-CH_3), 1.21 (t, J = 7.0 Hz, 3H, OCH_2CH_3), 2.16 (d, J = 5.6 Hz, 2H, CH_2), 2.29 (d, J = 6.0 Hz, 2H, CH_2), 3.82 (s, 3H, OCH_3), 2.35 (s, 3H, C-CH_3), 4.05 (q, J = 7.0 Hz, 2H, OCH_2CH_3), 4.94 (s, 1H), 6.59 (s, 1H), 6.68 (d, J = 1.2 Hz, 2H), 6.89 (br s,1H, NH), 7.82 (br s, 1H, OH),

Ethyl 1, 4, 5, 6, 7, 8-hexahydro-2, 7, 7-trimethyl-4-(2-nitrophenyl)-5-oxoquinoline-3-carboxylate: **(5j)** Pale yellow solid, mp: 208-210^{0}C, IR (KBr) ῡ: 3296, 3207, 3092, 1707, 1610, 1300, 1156, 830 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.94 (s, 3H, C-CH_3), 1.11(s, 3H, C-CH_3), 1.20 - 1.26 (t, *J* = 7.00 Hz, 3H, CH_3-CH_2O), 2.09 - 2.30 (m, 4H, 2 x CH_2), 2.33 (s, 3H, =C-CH_3), 4.01 (q, *J* = 6.4 Hz, 2H, OCH_2CH_3), 4.88 (s, 1H, Ar-CH), 6.35 (s, 1H, NH), 7.01 - 7.20 (m, 4H, ArH)

Ethyl 1, 4, 5, 6, 7, 8-hexahyro-2, 7, 7-trimethyl-4-styryl -5-oxoquinoline-3-carboxylate: **(5k)** Pale yellow solid, mp: 204-206^{0}C, IR (KBr) ῡ: 3200, 3076, 2955, 2800, 1686, 1606, 1219 cm^{-1}; ^{1}HNMR (400 MHz, $CDCl_3$): δ = 0.95 (s, 3H, C-CH_3), 1.1 (s 3H, C-CH_3), 1.3 (t, 3H, CH_2-CH_3); 1. 71 (s, 3H, C=C-CH_3), 1.88 – 2.86 (m, 4H, 2 x CH_2), 3.9 (s, 1H Ar-CH), 4.19 (q, J= 7.1 Hz, 2H, OCH_2CH_3), 5.8 (s, 1H, NH), 6.1 (d, J=13.7 Hz, 1H), 6.33 (d, J= 13.7 Hz, 1H), 7.1-7.3 (m, 5H, Ar-H)

Ethyl 1, 4, 5, 6, 7, 8-hexahydro-2, 7, 7-trimethyl-4-(4-(dimethylamino)styryl)-5-oxoquinoline-3-carboxylate: **(5l)** Radish yellow solid, mp: 234–236^{0}C, IR (KBr) ῡ = 3284, 3210, 3056, 2950, 2800, 1676, 1610, 1210 cm^{-1}, ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.94 (s, 3H, C-CH_3), 1.04 (s, 3H, C-CH_3), 1.33 (t, J = 7.1 Hz, 3H, OCH_2-CH_3), 2.18–2.16 (m, 4H, 2 x CH_2), 2.33 (s, 3H, C-CH_3), 2.85 (s, 6H, $N(CH_3)_2$), 4.07 (q, J = 7.1 Hz, 2H, OCH_2CH_3), 4.94 (s, 1H Ar-CH), 6.07 (d, J = 13.4 1H, C=CH), 6.3 (d, J = 13.4, 1H, C=CH), 6.57 (d, J = 8.8 Hz, 2H, Ar-H), 7.15 (d, J = 8.8 Hz, 2H, Ar-H).

Ethyl 1, 4, 5, 6, 7, 8-hexahydro-2, 7, 7-trimethyl-4-(3-nitrophenyl)-5-oxoquinoline-3-carboxylate: **(5m)** Pale yellow solid, mp: 164-166^{0}C, IR (KBr) ῡ: 3299, 2958, 1687, 1610, 1164, 757 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) : δ = 0.92 (s, 3H, C-CH_3), 1.05 (s, 3H, C-CH_3), 1.21 (t, *J* = 7.2 Hz, 3H, CH_3CH_2O), 2.10 - 2.31 (m, 4H, 2 x CH_2), 2.36 (s, 3H, =C-CH_3), 4.06 (q, *J* = 6.8 Hz, 2H, OCH_2CH_3), 4.95 (s, 1H, Ar-CH), 6.36 (s, 1H, NH), 6.70 (d, *J* = 8.4Hz, 1H, Ar-H), 7.02 - 7.15 (m, 2H, 2 x Ar-H), 7.38 (d, *J* = 8.4Hz, 1H, Ar-H).

Results and Discussion

In a solvent less condition experiment was carried out with *p*-N, N-dimethylbenzaldehyde **1** (5c, Entry 3), ethylacetoacetate **3**, dimedone **2** and ammonium acetate **4** as a nitrogen precursor in the absence of DABCO and the required product was not found (monitored by TLC) even after grinding for 4h at room temperature (Table 2) (Entry 3). Later on, it was decided that the suitable conditions can apply for condensation is in a solvent we follow several solvents like methanol, ethanol, acetonitrile, tetrahydrofuran, dichloroethane (Table 3) and the catalytic amount of catalyst out of which we observed that ethanol was the suitable solvent for the preparation of 1,4-polyhydroquinoline derivatives in presence of DABCO (0.5mol %) (Table 3, Entry 3). Our optimization studies revealed that the yield increased smoothly with catalyst load up to 0.5 mol % and after for 0.6 mol % and so on that there was sharp drop

in the yield (Table 2). This drop in yield may be attributed to the coagulation of 1,4-diazabicyclo[2,2,2]octane (DABCO) which decreases the effective surface area of the catalyst.

The results summarized (Table 1), aromatic aldehydes, hetero-aromatic aldehydes (entry 1), and α-β unsaturated aldehydes (Entry 11, 12), were reacted very well to afford the corresponding products of 1,4-polyhydroquinoline derivatives in very good to excellent yields. In general, it is to be observed that the aromatic aldehydes having electron-donating groups (Entry 3, 4, 9) and hetero-aromatic (Entry 7) aldehydes are reacting a little faster when compared with other aldehydes. In a similar manner, aromatic aldehydes containing electron withdrawing groups (Entry 2, 5, 6, 8, 10, and 13) are reacting comparatively a little slower in term of conversion as well as yields in the presence of the catalyst DABCO. In general, all the reactions were completed within 1 to 3h, and the obtained yields were 73% to 92% (Table 1).

Table 1. DABCO Catalyzed synthesis of 1,4-polyhydroquinoline derivatives.[a]

Entry	Aldehyde 1	Product 5	Time (Min.)	Yield[b] (%)	M.P. ^{0}C	
					Found	Literature
1	C_6H_5	5a	60	84	204-205	202-204 [10]
2	4-Cl C_6H_4	5b	90	85	242-244	245-246 [19]
3	4-Me_2N C_6H_4	5c	60	92	231-233	229-231 [9]
4	4-MeO C_6H_4	5d	60	89	264-266	257-259 [10]
5	4-NO_2 C_6H_4	5e	180	86	240-241	242-244 [9]
6	2-Cl C_6H_4	5f	120	82	208-210	208-210 [19]
7	2-Furyl	5g	30	90	244-246	248-249 [10]
8	3-Cl C_6H_4	5h	180	73	210-212	c
9	3-CH_3O, 4OH C_6H_3	5i	60	90	212-214	209-211 [19]
10	2-NO_2 C_6H_4	5j	180	79	208-210	205-207 [9]
11	C_6H_5CH=CH	5k	180	86	204-206	204-206 [10]
12	4-$(CH_3)_2N$ C_6H_4CH=CH	5l	120	89	234-236	c
13	3-NO_2 C_6H_4	5m	180	79	164-166	176-178 [9]

[a] Reaction conditions: (5c) aldehyde (5 mmol), dimedone (5 mmol), ethyl acetoacetate (5 mmol) and ammonium acetate (7.5 mmol) in presence of DABCO (0.5 mol%) in ethanol (5 ml) at room temperature.
[b] Isolated yields [c] Newly synthesized and characterized by FTIR and ^{1}H NMR.

Table 2. Optimization of catalyst for the synthesis of 1,4-polyhydroquinoline derivatives.[a]

Entry	Catalyst (mol %)	Time (min.)	Yield (%) [b]
1	00	240	No reaction
2	0.1	180	46
3	0.2	180	62
4	0.3	180	80
5	0.4	120	88
6	0.5	60	92
7	0.6	30	80
8	0.7	30	68

[a] Reaction conditions: (5c) aldehyde (5 mmol), dimedone (5 mmol), ethyl acetoacetate (5 mmol) and ammonium acetate (7.5 mmol) in presence of DABCO (0.5 mol%) in ethanol (5 ml) at room temperature.
[b] Isolated yields.

Table 3: Optimization of solvent effect for the synthesis of 1,4-polyhydroquinoline derivatives.[a]

Entry	Solvent	Amount of catalyst	Time (min)	Yield (%) [b]
1	No solvent	0.5	240	No reaction
2	Methanol	0.5	90	68
3	Acetonitrile	0.5	210	82
4	Tetrahydrofuran	0.5	120	60
5	Ethanol	0.5	60	92
6	Dichloroethane	0.5	180	58

[a] Reaction conditions: (5c) aldehyde (5 mmol), dimedone (5 mmol), ethyl acetoacetate (5 mmol) and ammonium acetate (7.5 mmol) in presence of DABCO (0.5 mol%) in ethanol (5 ml) at room temperature.
[b] Isolated yields

Conclusion

A simple workup procedure, mild reaction condition, selectivity, low toxicity, moderate Lewis basicity and good to excellent yields make this methodology a valid alternative to other methods found in the literature in the field of 1,4-polyhydroquinoline derivatives. The one pot multi component synthesis of 1,4-polyhydroquinoline derivatives by the polycondensation of aromatic aldehydes, ethyl acetoacetate, dimedone and ammonium acetate as a nitrogen precursor in presence of catalytic amount of diazabicyclo[2.2.2]octane (DABCO) (0.5 mol%) as a green base-organo-catalyst in ethanol at room temperature is achieved.

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