Scientific paper

Ultrasound-promoted One-pot Synthesis of 8-Aryl-7,8-dihydro-[1,3]-dioxolo[4,5-g]quinolin-6(5*H*)-one Derivatives under Catalyst-free and Solvent-free Conditions

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Abstract

An ultrasound-accelerated one-pot procedure has been explored for the synthesis of 8-aryl-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one derivatives using the reaction between 3,4-methylendioxyaniline **1**, aldehyde **2** and isopropylidene malonate **3** under catalyst-free and solvent-free conditions. High yields of the products, mild reaction condition, environmentally friendly procedure, catalyst- and solvent-free conditions are the main advantages of this protocol.

Keywords: Quinolin-6(5*H*)-ones, Ultrasound-irradiation, Catalyst-free, Solvent-free, One-pot reaction

1. Introduction

Substituted quinolines are one of the oldest known classes of pharmaceutical agents and their relevance in chemotherapy especially against malaria is widely known.[1] Beside antimalarials, a spectrum of other pharmacological activities[2] has been the major reason for the development of novel and efficient synthesis of this heterocycle. As a result, the recent past has witnessed the publication of several simple and elegant synthesis of substituted quinolines.[3]

Nevertheless, a new, solvent-free, one-pot method from readily accessible starting materials, which would permit delivery of this motif decorated with functional groups amenable to further diversification, should be of great synthetic relevance.

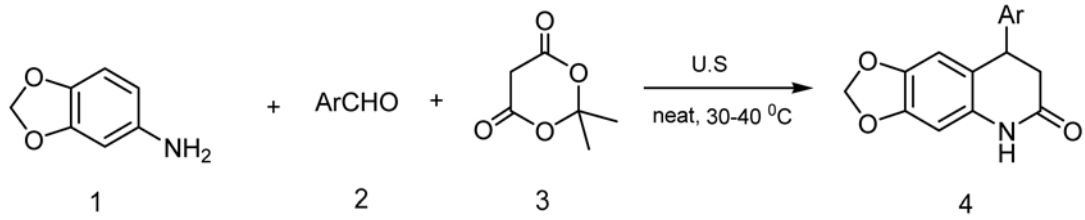
Multi-component reactions (MCRs) have proved to be notably successful in generating products in a single synthetic operation.[4–5] The development of new MCRs[6] and improvement of known multi-component reactions are the subjects of considerable current interest.

Recently, an area of intense synthetic endeavor has emphasized the use and design of reagents without the use of any solvent. Avoiding organic solvents during the reactions in organic synthesis leads to clean, efficient and cost-effective technology. In solid state reactions, work up is considerably simplified, cost is reduced, increased amounts of reactants can be used in the same equipment, reactivities and sometimes selectivities are enhanced without dilution.[7]

In recent years, the use of ultrasound in organic transformations is well known to enhance reaction rates, yields and selectivity of reaction. In several cases, it facilitates organic transformation at ambient conditions which otherwise require drastic conditions of temperature and pressure.[8–9]

Sonochemistry can be defined as the chemical effect caused by ultrasound in a broad sense, but it is generally understood as the chemical outcome of acoustic cavitation, evolution and collapse of micro bubbles as a result of ultrasonic irradiation. According to the most widely accepted hot spot theory, the gas phase of the cavity reaches high temperatures (5000 K) and pressures (170 MPa).[10] As the life time of this hot spot is very short (10^{-6} s), the rate of the temperature variation is as rapid as 10^{10} K s^{-1}.[11]

Because of their wide range of biological, industrial and synthetic applications, substituted quinolines have recently received a great deal of attention. In continuation of our work on solvent-free, ultrasound conditions,[12] we present here, for the first time, a simple, mild and efficient



1 2 3 4

Scheme 1. One-pot three-component reaction of 3,4-methylendioxyaniline **1**, aromatic aldehyde **2** and isopropylidene malonate **3** under ultrasound irradiation.

synthesis of 8-aryl-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-ones in high yields (Scheme 1).

2. Experimental

Chemicals used in this work were purchased from Aldrich and Merck chemical companies and used without purification. IR spectra were recorded on a Shimadzu 435-U-04 FT spectrometer as KBr pellets. ^{1}H and ^{13}C NMR spectra were measured in $CDCl_3$ with a Bruker DRX-400 Avance instrument at 400 and 100 MHz, respectively, using Me_4Si as internal standard. Mass spectra were recorded with a spectrometer Finnigan-MAT 8430 operating at an ionization potential of 70 eV. Melting points were measured on a SMPI apparatus. Elemental analyses for C, H and N were performed using a Perkin–Elmer 2400 series analyzer. Ultrasonication was performed in a Transsoni 660/H ultrasound cleaner with a frequency of 35 kHz and an output power of 70 W. The reactions were performed in open vessels.

2. 1. Ultrasound-promoted Condensation of 3,4-Methylendioxyaniline, Isopropylidene Malonate and Aromatic Aldehydes

General procedure. A mixture of 3,4-methylendioxyaniline **1** (0.132 g, 1 mmol), aldehyde **2** (1 mmol) and isopropylidene malonate **3** (0.15 g, 1 mmol) in a flask was placed in a water bath and sonicated at 30–40 °C for an appropriate time (Tables 1 and 2) until the reaction was completed as monitored by TLC (*n*-hexane/EtOAc; 2:1). The reaction mixture was then washed with water and crude product purified on 20 × 20 cm^2 TLC plates coated with silica gel 60 HF-254 using *n*-hexane/EtOAc (2:1) as the eluent. The separated products were first exposed to air for few minutes and then dried in an oven at 100 °C. For further purification, the products were crystallized from MeOH. The structures of these products were found to be the expected 8-aryl-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-ones **4a**–**i** confirmed by their spectral data (IR, ^{1}H and ^{13}C NMR, MS) and elemental analysis as given below.

8-Phenyl-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one (**4a**): Brown solid; m.p. 193–195 °C, IR (KBr) (ν_{max}, cm^{-1}): 3191 (NH), 1674 (C=O); MS *m/z* (%): 267 (M); ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 3.15–3.08 (2H, m, H_7), 4.30–4.27 (1H, t, H_8), 5.19 (2H, s, OCH_2), 6.33 (1H, s, Ar-H), 7.41–7.21 (5H, m, Ar-H), 8.49 (1H, s, NH); ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 39.7, 43.7, 97.7, 128.3, 128.5, 128.7, 128.8, 128.9, 129.0, 129.1, 129.2, 129.5, 129.6, 168.1; Anal. Calcd. for $C_{16}H_{13}NO_3$: C, 71.91, H, 4.86, N, 5.24; found C, 71.76, H, 4.74, N, 5.12.

8-(4-Methylphenyl)-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one (**4b**): Brown solid; m.p. 203–205 °C; IR (KBr) (ν_{max}, cm^{-1}): 3291 (NH), 1671 (C=O); MS *m/z* (%): 281 (M); ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 1.09 (3H, s, CH_3), 2.97–2.83 (2H, m, H_7), 4.69–4.66 (1H, t, H_8), 6.13 (2H, s, OCH_2), 7.26 (1H, s, Ar-H), 7.34–7.32 (2H, d, Ar-H), 7.48 (1H, s, Ar-H), 7.58 (1H, s, NH), 8.04–8.02 (2H, d, Ar-H); ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 21.3, 31.9, 43.0, 99.3, 101.6, 123.8, 127.1, 128.3, 129.5, 129.7, 129.8, 138.9, 143.6, 147.6, 172.5; Anal. Calcd. for $C_{17}H_{15}NO_3$: C, 72.59, H, 5.33, N, 4.98; found C, 72.50, H, 5.25, N, 5.05.

8-(4-Chlorophenyl)-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one (**4c**): Pale brown solid; m.p. 209–211 °C; IR (KBr) (ν_{max}, cm^{-1}): 3205 (NH), 1676 (C=O); MS *m/z* (%): 300 (M), 302 (M+2); ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 2.95–2.84 (2H, m, H_7), 4.25–4.18 (1H, t, H_8), 5.89 (2H, s, OCH_2), 6.38 (1H, s, Ar-H), 6.44 (1H, s, Ar-H), 7.45–7.20 (4H, m, Ar-H), 8.83 (1H, s, NH); ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 36.1, 40.1, 98.3, 104.6, 115.2, 122.1, 126.5, 128.3, 130.4, 133.2, 140.5, 145.1, 148.2, 169.4; Anal. Calcd. for $C_{16}H_{12}ClNO_3$: C, 63.68, H, 3.98, N, 4.64; found C, 63.52, H, 4.06, N, 4.74.

8-(4-Methoxyphenyl)-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one (**4d**): Brown solid; m.p. 235–237 °C; IR (KBr) (ν_{max}, cm^{-1}): 3350 (NH), 1665 (C=O); MS *m/z* (%): 297 (M); ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 2.88–2.81 (2H, m, H_7), 3.65 (3H, s, OCH_3), 4.64–4.53 (1H, t, H_8), 5.90 (2H, s, OCH_2), 6.38 (1H, s, Ar-H), 6.43 (1H, s, Ar-H), 7.40–6.77 (4H, m, Ar-H), 8.87 (1H, s, NH); ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 34.7, 38.4, 57.1, 97.9, 103.2, 112.4, 122.6, 128.6, 130.6, 134.7, 140.4, 145.4,

153.7, 156.3, 170.8; Anal. Calcd. for $C_{17}H_{15}NO_4$: C, 68.68, H, 5.05, N, 4.71; found C, 68.78, H, 4.89, N, 4.64.

8-(2,4-Dichlorophenyl)-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one (**4e**): Pale cream solid; m.p. 254–256 °C; IR (KBr) (ν_{max}, cm^{-1}): 3206 (NH), 1675 (C=O); MS *m/z* (%): 335 (M), 337 (M+2), 339 (M+4); ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 2.97–2.83 (2H, m, H_7), 4.69–4.66 (1H, t, H_8), 5.96 (2H, s, OCH_2), 6.43 (1H, s, Ar-H), 6.48 (1H, s, Ar-H), 7.47–6.87 (3H, m, Ar-H), 8.72 (1H, s, NH); ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 36.8, 38.1, 97.9, 101.5, 108.3, 116.9, 127.7, 129.8, 129.9, 131.7, 133.7, 134.2, 137.5, 144.0, 147.7, 169.8; Anal. Calcd. for $C_{16}H_{11}Cl_2NO_3$: C, 57.14, H, 3.27, N, 4.16; found C, 57.06, H, 3.39, N, 4.24.

8-(2-Chlorophenyl)-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one (**4f**): Brown solid; m.p. 200–202 °C; IR (KBr) (ν_{max}, cm^{-1}): 3198 (NH), 1679 (C=O); MS *m/z* (%): 301 (M), 303 (M+2); ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 2.94–2.92 (2H, m, H_7), 4.76–4.73 (1H, t, H_8), 5.95 (2H, s, OCH_2), 7.46–6.44 (6H, m, Ar-H), 8.29 (1H, s, NH); ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 34.4, 38.5, 97.7, 108.4, 112.2, 115.7, 120.9, 127.4, 128.6, 128.9, 130.1, 138.8, 143.9, 147.5, 170.6; Anal. Calcd. for $C_{16}H_{12}ClNO_3$: C, 63.68, H, 3.98, N, 4.64; found C, 63.56, H, 4.06, N, 4.52.

8-(2-Nitrophenyl)-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one (**4g**): Brown solid; m.p. 203–205 °C; IR (KBr) (ν_{max}, cm^{-1}): 3193 (NH), 1674 (C=O); MS *m/z* (%): 312 (M); ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 2.74–2.72 (2H, m, H_7), 4.26–4.23 (1H, t, H_8), 6.15 (2H, s, OCH_2), 7.95–7.41 (7H, m, NH, Ar-H); ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 29.7, 31.9, 99.3, 103.2, 108.4, 117.8, 127.2, 128.4, 129.4, 129.6, 129.7, 138.8, 143.8, 157.1, 160.3, 176.5; Anal. Calcd. for $C_{16}H_{12}N_2O_5$: C, 61.53, H, 3.84, N, 8.97; found C, 61.70, H, 3.93, N, 8.94.

8-(2-Methoxyphenyl)-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one (**4h**): Pale yellow solid; m.p. 223–225 °C; IR (KBr) (ν_{max}, cm^{-1}): 3229 (NH), 1683 (C=O); MS *m/z* (%): 297 (M); ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 2.97–2.83 (2H, m, H_7), 3.88 (3H, s, OCH_3), 4.63–4.60 (1H, t, H_8), 5.93 (2H, s, OCH_2), 6.41 (1H, s, Ar-H), 6.47 (1H, s, Ar-H), 6.89–6.88 (2H, m, Ar-H), 6.94–6.92 (1H, d, Ar-H), 7.26–7.24 (1H, dd, Ar-H), 8.04 (1H, s, NH); ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 35.7, 39.1, 55.3, 97.5, 101.3, 108.5, 110.6, 118.8, 120.8, 128.2, 128.3, 131.6, 138.3, 143.6, 151.2, 155.0, 170.4; Anal. Calcd. for $C_{17}H_{15}NO_4$: C, 68.68, H, 5.05, N 4.71; found C, 68.84, H, 5.12, N, 4.83.

8-(3-Bromophenyl)-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one (**4i**): Pale yellow solid; m.p. 207–209 °C; IR (KBr) (ν_{max}, cm^{-1}): 3194 (NH), 1672 (C=O); MS *m/z* (%): 345 (M), 347 (M+2); ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 2.93–2.85 (2H, m, H_7), 4.19–4.16 (1H, t, H_8), 5.98 (2H, s, OCH_2), 6.42 (1H, s, Ar-H), 6.46 (1H, s, Ar-H), 7.25–7.12 (2H, m, Ar-H), 7.44–7.42 (2H, d, Ar-H), 8.42 (1H, s, NH); ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 39.3, 41.8, 101.5, 101.6, 101.7, 108.4, 108.7, 118.1, 123.0, 126.4, 128.8, 130.5, 130.6, 130.8, 130.9, 169.9; Anal. Calcd. for $C_{16}H_{12}BrNO_3$: C, 55.50, H, 3.46, N, 4.04; found C, 55.64, H, 3.54, N 3.95.

3. Results and Discussion

In order to establish the reaction conditions, the reaction of 3,4-methylendioxyaniline, benzaldehyde and isopropylidene malonate was chosen as a model reaction (Table 2, Entry a). The effects of solvent and conditions on the rate and yield of the reaction were studied using different solvents such as MeOH, MeCN, DMF, EtOAc, Et_2O, $CHCl_3$, CCl_4, *n*-hexane as well as solvent-free condition both under ultrasonication and conventional heating at various temperatures (Table 1). The results summarized in Table 1 indicated that the best result in terms of yield and reaction rate was obtained under solvent-free and ultrasound irradiation conditions at 30–40 °C (Entry 1). However, as shown in this Table, the yields of the reaction obtained using the solvents MeCN (75%), EtOAc (76%) and DMF (78%) are comparable with that obtained under solvent-free condition.

Table 1. Screening of the solvents and conditions for the synthesis of 8-aryl-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one[a]

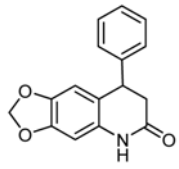
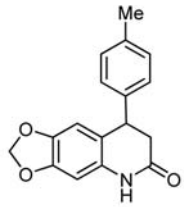
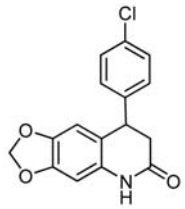
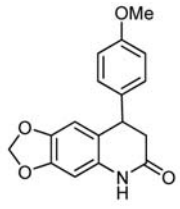
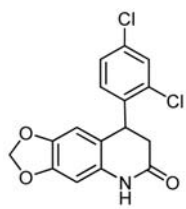
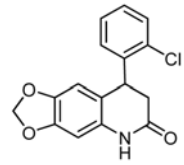
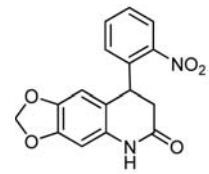
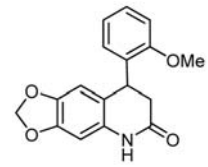
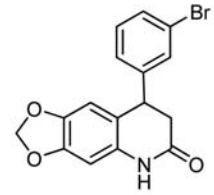
Entry	Conditions	Method	Time (min)	Yield[b] (%)
1	Solvent-free / 30–40 °C	ultrasound	50	83
2	MeOH / 30–40 °C	ultrasound	60	70
3	MeCN / 30–40 °C	ultrasound	60	75
4	EtOAc / 30–40 °C	ultrasound	60	76
5	DMF / 30–40 °C	ultrasound	60	78
6	CCl_4 / 30–40 °C	ultrasound	60	69
7	Et_2O / 30–40 °C	ultrasound	60	73
8	*n*-hexane / 30–40 °C	ultrasound	60	60
9	Solvent-free / rt	thermal	60	74
10	Solvent-free / 60 °C	thermal	60	72
11	Solvent-free / 100 °C	thermal	60	71
12	Solvent-free / reflux	thermal	60	71
13	MeOH / rt	thermal	60	65
14	MeCN / rt	thermal	60	69
15	EtOAc / rt	thermal	60	71
16	DMF / rt	thermal	60	73
17	$CHCl_3$ / rt	thermal	60	67
18	Et_2O / rt	thermal	60	66
19	CCl_4 / rt	thermal	60	65
20	*n*-hexane /rt	thermal	60	55

[a] **Conditions**: 3,4-methylendioxyaniline (1 mmol), benzaldehyde (1 mmol), isopropylidene malonate (1 mmol), solvent (2 mL).
[b] Isolated yields.

The scope of the reaction was extended to a variety of structurally diverse aldehydes using the optimized conditions. The results obtained are summarized in Table 2. As seen in this table, the aromatic aldehydes having electron-donating as well as electron-withdrawing groups were uniformly transformed into the corresponding 8-aryl-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-ones in high yields (76–88%) within 60–75 min. All of the products **4** exhibited a multiplet in the region 3.15–2.74 ppm for H-7 and a triplet in the region 4.76–4.16 ppm for H-8 in their ^{1}H NMR spectra, and three distinguishing peaks in the regions 43.7–31.9 (C-8), 41.8–29.7 (C-7) and 176.5–168.1 (C=O) ppm in their ^{13}C NMR spectra.

A possible mechanism to explain the formation of the products **4a–i** is depicted in Scheme 2. The formation of products **4a–i** can be rationalized by initial formation of heterocyclic system **5** through the standard Knoevenagel condensation of **3** with the aromatic aldehyde **2**. Subsequent Michael-type addition of 3,4-methylenedioxyanilin **1** to **5** followed successively by cyclization, dehydration and air oxidation affords the corresponding products **4a–i**.

Table 2. Ultrasound-promoted synthesis of 8-aryl-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one derivatives **4a–i** [a].

Entry	Ar	Product 4	Time (min)	Yield [b] (%)
a	C_6H_5		65	83
b	4-MeC_6H_4		70	77
c	4-ClC_6H_4		60	85
d	4-$MeOC_6H_4$		75	76
e	2,4-$Cl_2C_6H_3$		70	88
f	2-ClC_6H_4		70	85
g	2-$NO_2C_6H_4$ [13]		70	82
h	2-$MeOC_6H_4$		75	77
i	3-BrC_6H_4		70	86

[a] **Conditions**: 3,4-methylendioxyaniline (1 mmol), aromatic aldehyde (1 mmol), isopropylidene malonate (1 mmol), sonication at 30–40 °C. [b] Isolated yield.

4. Conclusion

We have described an ultrasound-promoted three-component one-pot procedure for the synthesis of 8-aryl-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-one derivatives under catalyst-free and solvent-free conditions. High yields, environmentally friendly nature, simplicity and mildness are the main merits of this method.

5. Acknowledgements

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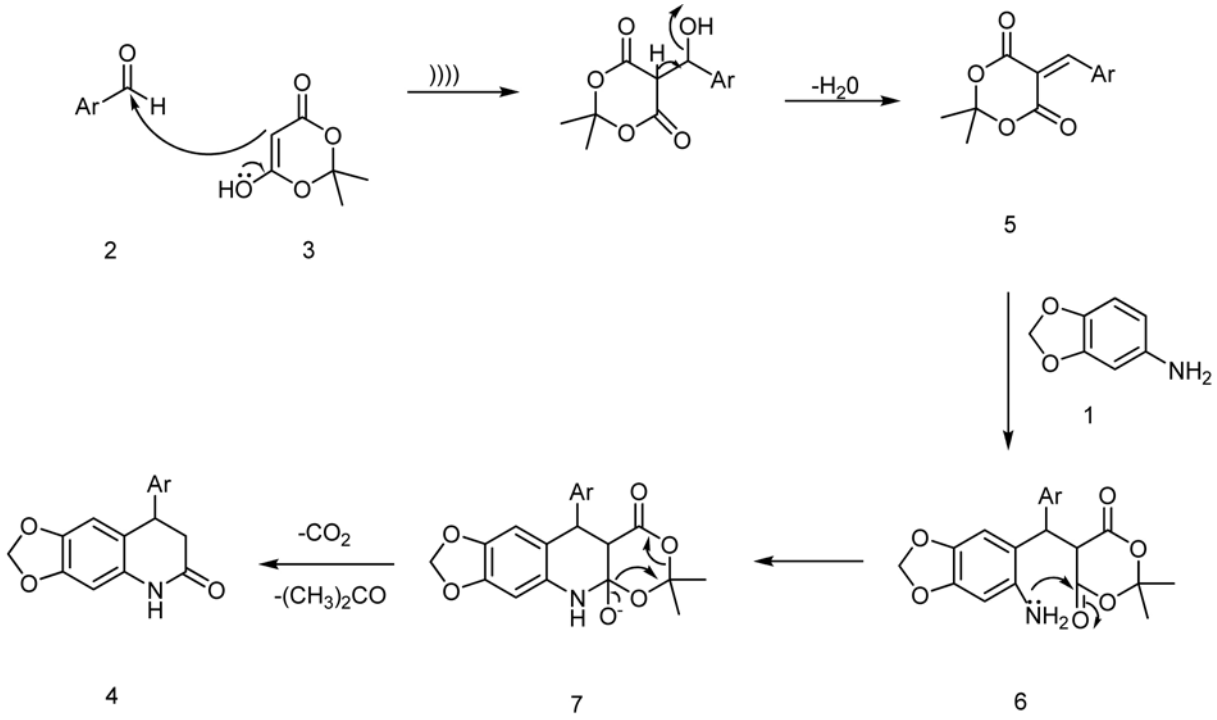

Scheme 2. Mechanism for synthesis 8-aryl-7,8-dihydro-[1,3]-dioxolo[4,5-*g*]quinolin-6(5*H*)-ones.

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Povzetek

Raziskali smo sintezo 8-aril-7,8-dihidro-[1,3]-dioksolo[4,5-*g*]kinolin-6(5*H*)-onskih derivatov, ki nastanejo pri reakciji med 3,4-metilendioksianilinom **1**, aldehidom **2** and izopropiliden malonatom **3** brez uporabe katalizatorjev in topila. Reakcija je pospešena z uporabo ultrazvočnega obsevanja in poteka v eni sami posodi ("one-pot" postopek). Visoki izkoristki produktov, nežni reakcijski pogoji, okolju prijazni postopki ter pogoji brez uporabe katalizatorjev in topil so glavne odlike tega sinteznega pristopa.