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Synthesis of dibenzo[*e*,*g*]isoindol-1-ones *via* photoinduced intramolecular annulation of 3,4-diphenyl-1*H*-pyrrol-2(5*H*)-ones

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ABSTRACT

An efficient, oxidant and photocatalyst-free approach for the synthesis of polycyclic-fused isoindolinone derivatives is reported *via* annulation of 3,4-diphenyl-3-pyrrolin-2-ones along with release of the hydrogen gas under an argon atmosphere in EtOH by irradiation with a 500 W mercury lamp at room temperature. The described approach is atom-economic and environmentally friendly and tolerates various electron-donating and electron-withdrawing groups. In addition, selected annulation products, dibenzo[*e*,*g*]isoindole-1-ones, were successfully oxidized into dibenzo[*e*,*g*]isoindole-1,3(2*H*)- diones in DMSO in the presence of CH_3ONa at room temperature.

1. Introduction

Isoindolinones are an important class of nitrogen heterocycles and their derivatives are in some bioactive natural products and drug molecules [1]. Selected isoindolinone derivatives with certain biological activities are shown in Fig. 1. For instance, indoprofen serves as an anti-inflammatory agent [2]. Chlortalidone was used as a diuretic and antihypertensive prescription drug [3]. Indolocarbazole analogue Gö6976 has been employed in selective PKC inhibitor and HIV-1 antagonist [4]. Moreover, heterocyclic fused isoindolinones have also been reported and pyrrolocarbazole (X = NH; Y=S) was found to be a potent inhibitor of mixed-lineage kinases (MLK1/3) [5].

Although, plenty of approaches have been reported for the preparation of isoindolinones [6], there is only a small number of known reports discussing the synthesis of dibenzo[*e*,*g*]isoindol-1-one [7]. Dibenzo[*e*,*g*]isoindol-1-one derivatives were obtained by the Scholl-type oxidative cyclization reaction in the presence of $PhI(O_2CCF_3)_2$ and $BF_3{\bullet}Et_2O$ at −40 °C for 4 h. However, the annulation is limited to arenes contanining methoxy group, since at least two methoxy groups are required as the electron-donating group at each of the aromatic rings (Scheme 1a) [8]. Later, the same conditions were used to synthesize benzo[*a*]carbazoles and indolo[2,3-*a*] carbazole analogues by Pelkey's group, which eliminated the requirement of methoxy group, but indoles are still electron-rich and nucleophilic [9]. Regarding to the Pelkey's work for the synthesis of dibenzo[*e*,*g*]isoindol-1-ones, the reaction generally requires not only the electron-rich arenes, but also the position of methoxy group is critical.

Photochemical reactions have become an important tool for organic chemists. The photons have been recognized as an ideal clean reagent for organic synthesis comparing to toxic chemical activators [10]. Polycyclic aromatic hydrocarbons (PAHs) could be easily achieved with the photochemical reactions, whereas, it would be difficult to access with traditional chemical conditions [11–13]. Furthermore, milder reaction condition, broad functional group could be tolerated, and high atom efficiency are other advantages of photochemical reactions to thermochemical reactions [14,15].

Given our interest in the development of transition-metal and/or additive-free photoinduced intramolecular dehydrogenative cyclization of 2,3-diaryl-4*H*-chromen-4-ones and 3,4-diarylfuran-2(5*H*)-ones for the synthesis of oxygen heterocycles [16,17], we would like to describe the synthesis of dibenzo[*e*,*g*]isoindol-1-ones with good functional group tolerance *via* the annulation of 3,4-

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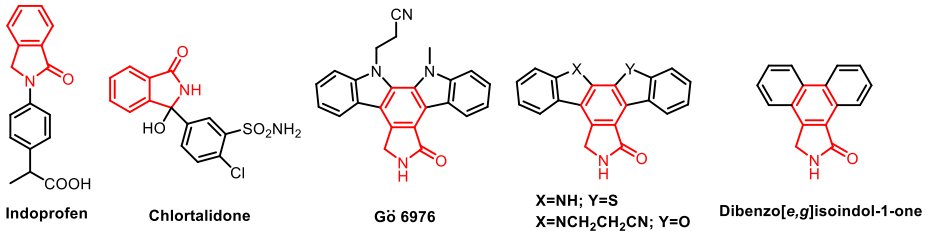
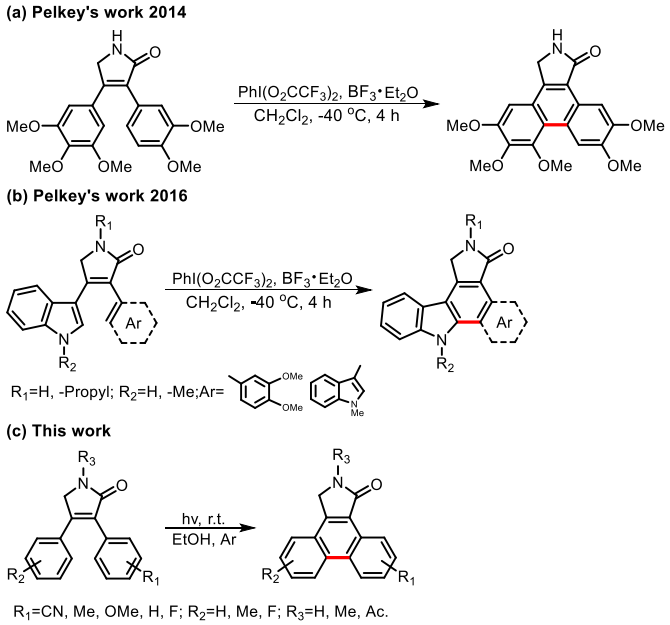
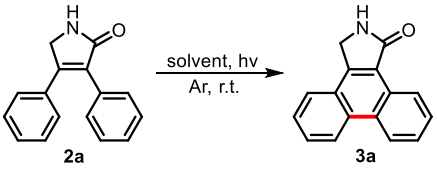


Fig. 1. Biologically active isoindolinone derivatives.



Scheme 1. Synthetic approaches to polycyclic-fused isoindolinones.

Table 1
Optimization of intramolecular cyclization of **2a**[a].



entry	conc. (mM)	solvent (v/v)	time (h)	yield (%)[b]
1	10	Me_2CO	6	21
2	10	DMF	6	N.D.
3	10	Toluene	6	N.D.
4	10	CH_2Cl_2	6	trace
5	10	MeOH	6	37
6	**10**	**EtOH**	**6**	**61**
7	5	EtOH	6	54
8	15	EtOH	6	49
9	10	EtOH	4	50
10	10	EtOH	8	58
11	10	EtOH	6	46[c]

[a] **2a** (35.3 mg, 0.150 mmol) was irradiated with a 500 W high-pressure mercury lamp in various solvents at room temperature under an Ar atmosphere. N.D.: not detected.
[b] Isolated yield.
[c] Open air.

diphenyl-3-pyrrolin-2-ones in EtOH at room temperature (Scheme 1c), which avoids usage of any oxidant and photocatalyst. Most importantly, the described method overcomes the shortcoming of Scholl-type oxidative cyclization reactions [8,9], since the presence of methoxy groups is not required.

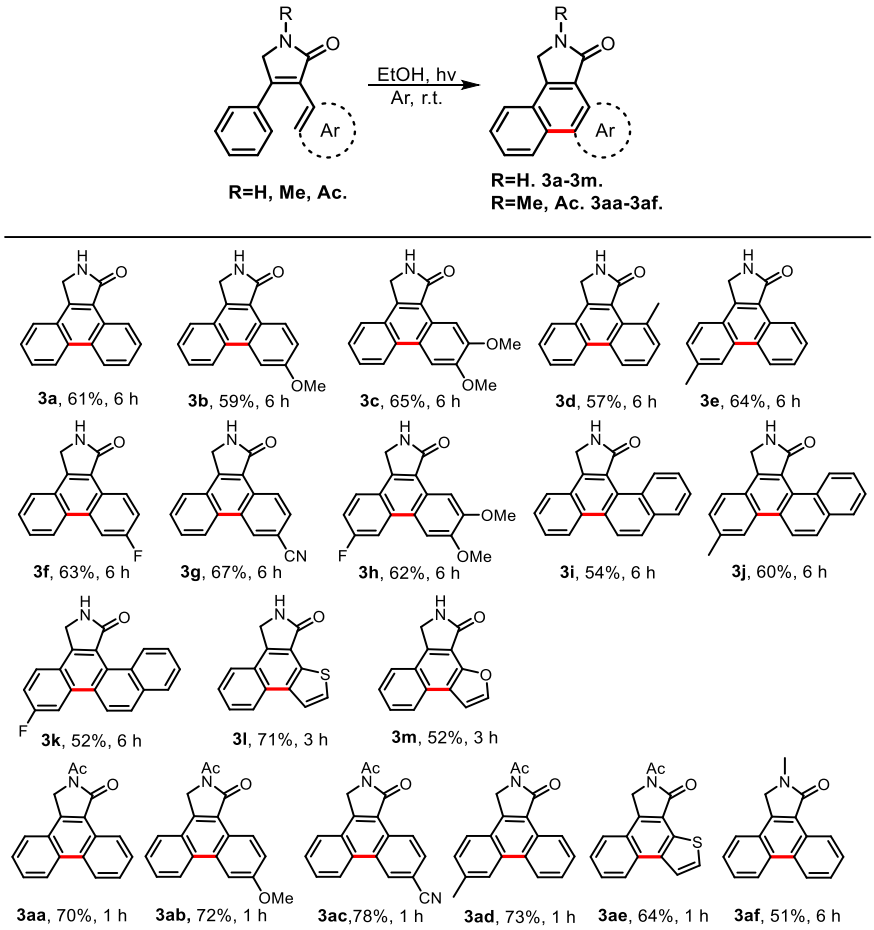
2. Results and discussion

Substrate 1-acetyl-3,4-diphenyl-1,5-dihydropyrrol-2-one (**1a**) and 3,4-diphenyl-3-pyrrolin- 2-one (**2a**) were synthesized according to the literature report [18]. Irradiation of 3,4-diphenyl-3-pyrrolin-2-one **2a** in various solvents under different conditions was performed and corresponding data are presented in Table 1. Initially, irradiation of **2a** in Me_2CO (15 mL) with a 500 W high-pressure mercury lamp at ambient temperature under an argon atmosphere for 6 h gave annulation product **3a** in 21% (Table 1, entry 1). Replacement of Me_2CO with other aprotic solvents, e.g. *N*,*N*-dimethylformamide (DMF), toluene, dichloromethane (DCM), resulted in inferior yields of **3a** (entries 2–4). To our delight, the yields of **3a** were improved to 37% and 61% with polar protic solvents (entries 5–6). Meanwhile, different concentrations were explored (entries 6–8) and it is noticed that **3a** was obtained in highest yield with 10 mM concentration (entry 6, 61%). Finally, irradiation time (4–8 h) was screened and the annulation product **3a** was obtained in 50%, 61% and 58% separately (entries 6, 9 and 10). It is important to note that the **3a** could be also obtained under open air, but with slightly decreased yield (entry 11, 46%). Thus, irradiation of **2a** in EtOH (10 mM) at room temperature for 6 h was determined to be the optimal condition. The results of detecting the hydrogen and emission spectrum of the 500 W high-pressure mercury lamp are shown in the Support Information (SI, Figures S3-5).

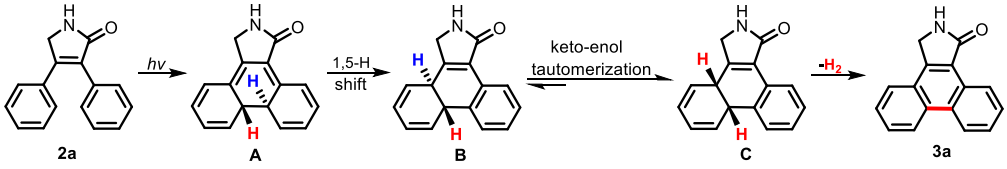
With the optimized conditions in hand, generality of the annulation was examined and the yields of 3 are summarized in Table 2. It was found that no significant substituent effect was observed for the substrates (3a-3h) bearing either electron-withdrawing groups (e.g., F, CN) or electron-donating groups (e.g., Me, OMe), since the yields of annulation products are close (57%–67%). Naphthalene and *N*-methyl substituted substrate were also subjected to the optimal condition and 3i-3k, 3af were also obtained in similar yields (51–60%). Moreover, thiophenyl and furanyl substituted substrate also gave desired products (3l-3m) in moderate yields (52%, 71%) with only half of the irradiation time (3 h), which is likely due to the higher activity of heteroaromatics. Finally, irradiation of the *N*-acetyl-substituted substrates (1a, 1b, 1g, 1e, 1l) also yielded the annulation products (3aa-3ae) but with slightly higher yields (64–78%) in significant shortened time (1h), which could be explained by the presence of an acetyl on the nitrogen atom. The structures of annulation products 3 were characterized by ^{1}H NMR, ^{13}C NMR, ^{19}F NMR, HRMS and IR.

Based on the mechanism studies [16,17,19,20] and experimental evidences, a plausible mechanism for the formation of **3a** is put forward and shown in Scheme 2. Irradiation of 3,4-diphenyl-3-

Table 2
substrate Scope[a].



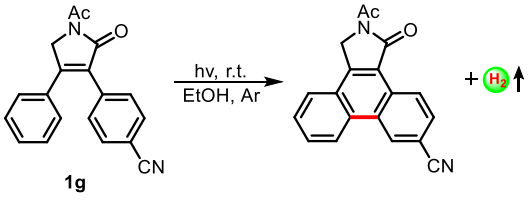
[a]Condition: **2** (35.3 mg, 0.150 mmol) in EtOH (15 mL, 10 mM) was irradiated with a 500 W high-pressure mercury at room temperature under Ar atmosphere until substrates were completely consumed indicated by TLC. Isolated yield.



Scheme 2. Plausible reaction mechanism.

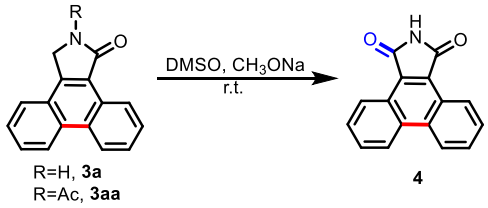
pyrrolin-2-one **2a** with 500 W Hg lamp at room temperature generated intermediate **A** *via* an intramolecular 6π-electron cyclization [19], followed by a thermal suprafacial [1,5]-H shift [20] to give intermediate **B**, which is driven by the rearomatization of the benzene ring. Subsequently, a more stable *syn*-isomer intermediate **C** is obtained *via* the keto-enol isomerization of **B**. In the end, the annulation product **3a** is obtained by hydrogen evolution *via syn*-elimination. Which shares similar transformations reported in literature [16,17]. It is important to note that the annulation product **3a** is also obtained in low yield under the open air (in presence of oxygen, Table 1, entry 11), which indicated the photochemical cyclization proceeded through the S1 state [11b,16].

Most importantly, the presence of H_2 byproduct was successfully detected *via* GC during annulation of **1g** (Scheme 3), which proved solid evidence for the proposed mechanism (Scheme 2).



Scheme 3. Detection of the only byproduct H_2 for irradiation **1g**.

It has been reported that treatment of 3,4-diphenyl-1*H*-pyrrol-2(5*H*)-one **2a** with NaH in DMF under an oxygen atmosphere at 0 °C



Scheme 4. Synthesis of dibenzo[*e,g*]isoindole-1,3(2*H*)-dione.

gave 3,4-diphenyl-1*H*-pyrrole-2,5-dione in moderate yields [21]. Since maleimide moiety has been extensively found in drugs, pesticides and dyes [22], these conditions were used to convert **3a** and **3aa** into dibenzo[*e,g*]isoindole-1,3(2*H*)-dione **4** under an oxygen atmosphere (Scheme 4). Sodium methoxide was added to the solution of **3a** or **3aa** in DMSO at room temperature to provide direct access to dibenzo[*e,g*]isoindole-1,3(2*H*)-dione **4** in 72% and 60%, which further confirmed the practical feasibility of the new developed method.

3. Conclusion

In summary, we have successfully demonstrated the synthesis of dibenzo[*e,g*]isoindol-1-one derivatives *via* annulation of 3,4-diphenyl-3-pyrrolin-2-one in EtOH with irradiation a 500 W mercury lamp under room temperature along with hydrogen evolution. Compared with reported methods, the described method offers several notable advantages: a) tolerates electron-donating and electron-withdrawing groups and heteroaromatics, such as thiophene and furan, b) eliminates the usage of any expensive transition-metal catalyst or photocatalyst, and c) atom-economic and environmental friendly with H_2 as the only byproduct. Additionally, annulation products **3a** and **3aa** were successfully oxidized into dibenzo[*e,g*]isoindole-1,3(2*H*)-dione analogue **4** in moderate yields that demonstrate the practical applications of this method.

4. Experimental section

4.1. General information

All reactions were determined by thin layer chromatography (TLC). TLC used 60 mesh silica gel. The crude products were performed by flash column chromatography (200–300 mesh) on silica gel. The starting materials were purchased from Energy Chemical. ^{1}H and ^{13}C NMR spectra were recorded on Bruker 400 or 600 MHz spectrometer in $CDCl_3$ and DMSO. All chemical shifts were given as δ value (ppm) with referenced to the residual solvent peaks [$CDCl_3$ (7.26 ppm) or DMSO-d_6 (2.50)] for ^{1}H NMR and the $CDCl_3$ (77.16 ppm) or DMSO-d_6 (39.52) for ^{13}C NMR spectra. High-resolution mass spectra (HRMS) were obtained using the electron-spray ionization (ESI) and (APCI) technique. Melting points (uncorrected) were measured with a X-5 micro-melting point apparatus. IR spectra were recorded with a Nicollet 170SX FT-IR spectrophotometer with KBr pellets. All the irradiation experiments were performed in a BL-GHX-V photo-chemical reactor equipped with a 500 W high-pressure mercury lamp at room temperature. The emission spectrum of the 500 W high-pressure mercury lamp is provided in Supporting Information (Figure S5).

4.2. General procedure for the synthesis of 1-Acetyl-3,4-diphenyl-1,5-dihydropyrrol-2-one ***(1)***

Triethylamine (2.3 mL, 16 mmol) was slowly added to the solution of *N*-(2-oxo-2-phenylethyl)-2-phenylacetamide (253 mg, 1 mmol) in acetic anhydride (3.3 mL) at 0 °C and was stirred at room temperature for 12 h. When the reaction was completed (detected by TLC), the reaction mixture was partitioned with water and ethyl acetate. Organic layer was combined, washed (saturated citric acid solution, saturated $NaHCO_3$ solution and brine), dried and concentrated *in vacuo*. The residue was purified by silica column chromatography (ethyl acetate/petroleum ether, 1:5) to give corresponding product **1a** (207.8 mg, 75%) [18]. Similarly, products **1b-1m** were obtained with the same method as described above.

4.2.1. 1-Acetyl-3,4-diphenyl-1,5-dihydropyrrol-2-one ***(1a)*** *[18]*

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.41–7.28 (m, 10H), 4.76 (s, 2H), 2.65 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.6, 169.8, 150.9, 132.1, 131.9, 131.0, 130.5, 130.2, 129.6, 129.0, 128.8, 128.1, 50.02, 24.8.

4.2.2. 1-Acetyl-3-(4-methoxyphenyl)-4-phenyl-1,5-dihydropyrrol-2-one ***(1b)***

Yellow solid. Yield: 69%, 211.8 mg. mp: 160.1–161.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.37–7.30 (m, 7H), 6.91 (d, J = 8.7 Hz, 2H), 4.72 (s, 2H), 3.83 (s, 3H), 2.65 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.6, 170.1, 156.0, 149.9, 132.4, 131.4, 130.9, 130.3, 129.0, 128.0, 123.1, 114.3, 55.4, 50.0, 24.8; IR (KBr), ν (cm^{-1}) 3059, 2928, 1705, 1605, 1340, 1300, 1159, 899, 766, 633; HRMS (ESI) *m/z*: calcd for $C_{19}H_{17}NO_3Na$ $[M + Na]^+$ 330.1101; found 330.1110.

4.2.3. 1-Acetyl-3-(3′,4′-dimethoxyphenyl)-4-phenyl-1,5-dihydropyrrol-2-one ***(1c)***

White solid. Yield: 71%, 267.7 mg. mp: 138.2–139.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.38–7.36 (m, 3H), 7.35–7.31 (m, 2H), 6.99 (dd, J = 8.3, 1.9 Hz, 1H), 6.88 (d, J = 8.3 Hz, 1H), 6.85 (d, J = 1.8 Hz, 1H), 4.73 (s, 2H), 3.90 (s, 3H), 3.73 (s, 3H), 2.65 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.6, 169.9, 150.1, 149.6, 149.1, 132.3, 131.5, 130.4, 129.0, 128.1, 123.3, 122.5, 112.6, 111.4, 56.0, 55.9, 50.0, 24.8; IR (KBr), ν (cm^{-1}) 2933, 1716, 1691, 1514, 1377, 1257, 1139, 1022, 769, 536; HRMS (ESI) *m/z*: calcd for $C_{20}H_{19}NO_4Na$ $[M + Na]^+$ 360.1206; found 360.1212.

4.2.4. 1-Acetyl-4-phenyl-3-(o-tolyl)-1,5-dihydropyrrol-2-one ***(1d)***

White solid. Yield: 66%, 192.1 mg. mp: 156.1–157.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.37–7.26 (m, 8H), 7.17 (d, J = 7.5 Hz, 1H), 4.89 (d, J = 19.1, 1H), 4.84 (d, J = 19.1, 1H), 2.67 (s, 3H), 2.14 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.5, 169.8, 150.7, 136.9, 132.4, 131.9, 131.1, 130.7, 129.8, 129.0, 129.0, 127.5, 126.5, 49.7, 24.7, 19.9; IR (KBr), ν (cm^{-1}) 3057, 2923, 1716, 1688, 1340, 1298, 1197, 1037, 902, 761, 541; HRMS (ESI) *m/z*: calcd for $C_{19}H_{17}NO_2Na$ $[M + Na]^+$ 314.1151; found 314.1160.

4.2.5. 1-Acetyl-3-phenyl-4-(p-tolyl)-1,5-dihydropyrrol-2-one ***(1e)***

White solid. Yield: 69%, 200.28 mg. mp: 147.1–148.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.39–7.35 (m, 5H), 7.24 (d, J = 8.2 Hz, 2H), 7.11 (d, J = 8.1 Hz, 2H), 4.74 (s, 2H), 2.64 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.6, 169.9, 150.9, 141.1, 131.2, 131.1, 129.7, 129.6, 129.1, 128.8, 128.7, 128.0, 49.9, 24.8, 21.6; IR (KBr), ν (cm^{-1}) 2923, 1720, 1650, 1604, 1346, 1292, 1118, 819, 540; HRMS (ESI) *m/z*: calcd for $C_{19}H_{17}NO_2Na$ $[M + Na]^+$ 314.1151; found 314.1158.

4.2.6. 1-Acetyl-3-(4′-fluorophenyl)-4-phenyl-1,5-dihydropyrrol-2-one ***(1f)***

White solid. Yield: 62%, 182.9 mg. mp: 184.5–185.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.39–7.32 (m, 7H), 7.07 (t, J = 8.7 Hz, 2H), 4.74 (s, 2H), 2.64 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.5, 169.7, 163.0 (d, ^1J = 247.21 Hz), 151.1, 131.9, 131.5 (d, ^3J = 8.31 Hz), 130.8, 130.6, 129.1, 128.0, 126.8 (d, ^4J = 3.18 Hz), 115.9 (d,

^{2}J = 21.33 Hz), 50.0, 24.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm) −112.17; IR (KBr), ν (cm^{-1}) 3064, 2927, 1710, 1510, 1336, 1294, 1155, 1097, 975, 850, 769, 686, 526; HRMS (ESI) *m/z*: calcd for $C_{18}H_{14}FNO_2Na$ $[M + Na]^+$ 318.0901; found 318.0902.

4.2.7. 4-(1-Acetyl-2-oxo-4-phenyl-2,5-dihydropyrrol-3-yl) benzonitrile **(1g)**

Gray solid. Yield: 64%, 193.3 mg. mp: 191.1–192.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.66 (d, J = 8.3 Hz, 2H), 7.50 (d, J = 8.3 Hz, 2H), 7.42 (t, J = 7.4 Hz, 1H), 7.35 (t, J = 7.7 Hz, 2H), 7.28 (d, J = 7.4 Hz, 2H), 4.77 (s, 2H), 2.64 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.3, 168.9, 153.3, 135.7, 132.5, 131.3, 131.1, 130.5, 130.1, 129.3, 128.0, 118.6, 112.5, 50.3, 24.8; IR (KBr), ν (cm^{-1}) 2927, 2227, 1697, 1344, 1298, 1112, 975, 854, 769, 547; HRMS (ESI) *m/z*: calcd for $C_{19}H_{14}N_2O_2Na$ $[M + Na]^+$ 325.0947; found 325.0954.

4.2.8. 1-Acetyl-3-(3′,4′-dimethoxyphenyl)-4-(4′-fluorophenyl)-1,5-dihydropyrrol-2-one **(1h)**

Yellow solid. Yield: 60%, 213.0 mg. mp: 154.6–155.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.39–7.35 (m, 2H), 7.04–7.00 (m, 2H), 6.94 (dd, J = 8.3, 1.9 Hz, 1H), 6.88 (d, J = 8.3 Hz, 1H), 6.85 (d, J = 1.8 Hz, 1H), 4.70 (s, 2H), 3.90 (s, 3H), 3.76 (s, 3H), 2.64 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.5, 169.8, 163.7 (d, ^{1}J = 251.17 Hz), 149.6, 149.2, 148.8, 131.4, 130.1 (d, ^{3}J = 8.35 Hz), 128.3 (d, ^{4}J = 3.18 Hz), 123.1, 122.4, 116.2 (d, ^{2}J = 21.76 Hz), 112.4, 111.5, 56.0, 56.0, 49.9, 24.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm) −108.74; IR (KBr), ν (cm^{-1}) 3062, 2937, 1726, 1681, 1595, 1512, 1309, 1245, 1137, 1016, 813, 756, 655; HRMS (ESI) *m/z*: calcd for $C_{20}H_{18}FNO_4Na$ $[M + Na]^+$ 378.1112; found 378.1116.

4.2.9. 1-Acetyl-3-(naphthalen-1-yl)-4-phenyl-1,5-dihydropyrrol-2-one **(1i)**

White solid. Yield: 70%, 228.9 mg. mp: 183.5–184.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.90 (dd, J = 15.6, 8.2 Hz, 2H), 7.63 (d, J = 8.4 Hz, 1H), 7.53–7.49 (m, 1H), 7.46 (t, J = 7.4 Hz, 1H), 7.40–7.35 (m, 2H), 7.25–7.22 (m, 1H), 7.20 (d, J = 7.5 Hz, 2H), 7.14 (t, J = 7.7 Hz, 2H), 4.96 (s, 2H), 2.63 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.6, 170.1, 152.1, 134.0, 131.5, 131.3, 130.8, 129.5, 129.3, 129.0, 128.8, 128.0, 127.9, 126.8, 126.4, 125.8, 125.0, 50.0, 24.7; IR (KBr), ν (cm^{-1}) 3053, 2923, 1720, 1692, 1338, 1296, 1201, 1099, 904, 800, 545; HRMS (ESI) *m/z*: calcd for $C_{22}H_{17}NO_2Na$ $[M + Na]^+$ 350.1151; found 350.1152.

4.2.10. 1-Acetyl-3-(naphthalen-1-yl)-4-(p-tolyl)-1,5-dihydropyrrol-2-one **(1j)**

White solid. Yield: 69%, 235.3 mg. mp: 162.1–163.6 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.93 (dd, J = 13.7, 8.2 Hz, 2H), 7.66 (d, J = 8.4 Hz, 1H), 7.54 (t, J = 7.3 Hz, 1H), 7.49 (t, J = 7.4 Hz, 1H), 7.41 (t, J = 6.5 Hz, 2H), 7.12 (d, J = 8.2 Hz, 2H), 6.97 (d, J = 8.2 Hz, 2H), 4.97 (s, 2H), 2.66 (s, 3H), 2.25 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.6, 170.2, 152.1, 141.4, 134.0, 131.4, 130.3, 129.7, 129.6, 129.4, 128.8, 128.7, 128.0, 127.8, 126.7, 126.3, 125.8, 125.1, 49.9, 24.7, 21.5; IR (KBr), ν (cm^{-1}) 3043, 2923, 1905, 1708, 1632, 1294, 1195, 1095, 966, 794, 547; HRMS (ESI) *m/z*: calcd for $C_{23}H_{19}NO_2Na$ $[M + Na]^+$ 364.1308; found 364.1318.

4.2.11. 1-Acetyl-4-(4′-fluorophenyl)-3-(naphthalen-1-yl)-1,5-dihydropyrrol-2-one **(1k)**

Yellow solid. Yield: 65%, 224.2 mg. mp: 190.5–192.1 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.94 (dd, J = 17.5, 8.2 Hz, 2H), 7.62 (d, J = 8.4 Hz, 1H), 7.55 (t, J = 7.6 Hz, 1H), 7.50 (t, J = 7.5 Hz, 1H), 7.40 (t, J = 8.1 Hz, 2H), 7.21 (dd, J = 8.7, 5.3 Hz, 2H), 6.86 (t, J = 8.6 Hz, 2H), 4.97 (d, J = 19.1 Hz, 1H), 4.93 (d, J = 19.1 Hz, 1H), 2.66 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.6, 169.9, 163.9 (d, ^{1}J = 251.74 Hz), 150.8, 134.1, 131.2, 131.0, 130.0 (d, ^{2}J = 8.62 Hz), 129.6, 129.0, 128.9, 128.0, 127.8 (d, ^{4}J = 3.25 Hz), 126.9, 126.5, 125.9, 124.9, 116.2 (d, ^{2}J = 21.78 Hz), 49.9, 24.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm) −107.96; IR (KBr), ν (cm^{-1}) 3062, 1720, 1510, 1303, 1201, 966, 840, 800, 624; HRMS (ESI) *m/z*: calcd for $C_{22}H_{16}FNO_2Na$ $[M + Na]^+$ 368.1057; found 368.1058.

4.2.12. 1-Acetyl-4-phenyl-3-(thiophen-2-yl)-1,5-dihydropyrrol-2-one **(1l)**

White solid. Yield: 72%, 203.8 mg. mp: 90.6–91.8 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.47–7.42 (m, 5H), 7.37 (d, J = 5.1 Hz, 1H), 7.34 (d, J = 3.6 Hz, 1H), 7.01 (dd, J = 4.9, 3.8 Hz, 1H), 4.67 (s, 2H), 2.67 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.4, 168.9, 150.1, 132.6, 131.2, 130.5, 129.2, 128.4, 127.9, 127.5, 127.0, 125.6, 51.0, 24.8; IR (KBr), ν (cm^{-1}) 3082, 1717, 1294, 1151, 970, 845, 700; HRMS (ESI) *m/z*: calcd for $C_{16}H_{13}SNO_2Na$ $[M + Na]^+$ 306.0559; found 306.0567.

4.2.13. 1-Acetyl-3-(furan-2-yl)-4-phenyl-1,5-dihydropyrrol-2-one **(1m)**

Yellow solid. Yield: 67%, 178.9 mg. mp: 114.5–115.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.47–7.42 (m, 5H), 7.35 (d, J = 1.2 Hz, 1H), 7.13 (d, J = 3.4 Hz, 1H), 6.49 (dd, J = 3.4, 1.8 Hz, 1H), 4.67 (s, 2H), 2.64 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.3, 168.0, 148.5, 145.6, 142.8, 132.6, 130.4, 128.6, 128.2, 121.2, 112.5, 111.5, 50.7, 24.8; IR (KBr), ν (cm^{-1}) 2931, 1722, 1684, 1346, 1139, 815, 736, 536; HRMS (ESI) *m/z*: calcd for $C_{16}H_{13}NO_3Na$ $[M + Na]^+$ 290.0788; found 290.0794.

4.3. General procedure for the synthesis of 3,4-diphenyl-1H-pyrrol-2(5H)-one **(2)**

A solution of sodium methoxide in MeOH (500 μL, 0.5 mmol, 30%) was added to the solution of 1-acetyl-3,4-diphenyl-1,5-dihydropyrrol-2-one **1a** (138.5 mg, 0.500 mmol) in dry MeOH (25 mL) at 0 °C under an Ar atmosphere. When the reaction was completed (detected by TLC), acetic acid (61 μL, 1 mmol) was slowly added and the reaction mixture was concentrated *in vacuo*. After partition with water and CH_2Cl_2, the organic layer was combined, washed (brine), and concentrated *in vacuo*. The residue was purified by silica column chromatography (ethyl acetate/dichloromethane, 1:3) to afford the corresponding product **2a** (70.5 mg, 60%) [18]. Similarly, compounds **2b-2m** were obtained with the same method as described above.

4.3.1. 3,4-Diphenyl-1H-pyrrol-2(5H)-one **(2a)** [18]

^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.48 (s, 1H), 7.32–7.23 (m, 10H), 4.33 (s, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 172.4, 150.4, 133.3, 132.4, 131.8, 129.3, 129.0, 128.6, 128.2, 127.7, 127.5, 47.5.

4.3.2. 3-(4′-Methoxyphenyl)-4-phenyl-1H-pyrrol-2(5H)-one **(2b)** [23]

^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.47 (s, 1H), 7.33 (s, 5H), 7.22 (d, J = 8.7 Hz, 2H), 6.90 (d, J = 8.7 Hz, 2H), 4.32 (s, 2H), 3.76 (s, 3H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 172.6, 158.8, 149.2, 133.6, 131.1, 130.5, 128.8, 128.6, 127.4, 124.3, 113.6, 55.0, 47.4.

4.3.3. 3-(3′,4′-Dimethoxyphenyl)-4-phenyl-1H-pyrrol-2(5H)-one **(2c)** [8]

^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.48 (s, 1H), 7.37–7.33 (m, 5H), 6.93–6.88 (m, 2H), 6.84 (d, J = 1.7 Hz, 1H), 4.32 (s, 2H), 3.75 (s, 3H), 3.56 (s, 3H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 172.5, 149.5, 148.5, 148.2, 133.7, 131.2, 128.8, 128.6, 127.6, 124.5, 121.9, 113.0, 111.5, 55.4, 55.2, 47.5.

4.3.4. 4-Phenyl-3-(o-tolyl)-1H-pyrrol-2(5H)-one **(2d)**

White solid. Yield: 68%, 84.7 mg. mp: 174.1–175.5 °C. ^{1}H NMR

(600 MHz, DMSO) δ (ppm) 8.47 (s, 1H), 7.31–7.25 (m, 5H), 7.23–7.18 (m, 3H), 7.04 (d, J = 7.4 Hz, 1H), 4.46 (dd, J = 19.2, 23.2 Hz, 2H), 2.04 (s, 3H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 172.5, 149.9, 136.4, 133.1, 133.0, 132.9, 130.0, 129.7, 129.2, 128.6, 128.0, 126.9, 125.9, 47.2, 19.4; IR (KBr), ν (cm^{-1}) 3435, 3277, 3055, 2918, 1693, 1448, 1360, 1217, 1084, 754, 638; HRMS (ESI) *m/z*: calcd for $C_{17}H_{15}NONa$ [M + Na]$^+$ 272.1046; found 272.1050.

4.3.5. 3-Phenyl-4-(p-tolyl)-1H-pyrrol-2(5H)-one **(2e)**

Yellow solid. Yield: 72%, 89.6 mg. mp: 209.1–210.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.48 (s, 1H), 7.37–7.30 (m, 3H), 7.27 (d, J = 6.8 Hz, 2H), 7.20 (d, J = 8.1 Hz, 2H), 7.12 (d, J = 8.0 Hz, 2H), 4.33 (s, 2H), 2.27 (s, 3H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 172.5, 150.4, 138.7, 132.5, 131.1, 130.4, 129.3, 129.2, 128.2, 127.6, 127.4, 47.5, 20.8; IR (KBr), ν (cm^{-1}) 3184, 3063, 2868, 1686, 1447, 1366, 1225, 702; HRMS (ESI) *m/z*: calcd for $C_{17}H_{15}NONa$ [M + Na]$^+$ 272.1046; found 272.1048.

4.3.6. 3-(4′-Fluorophenyl)-4-phenyl-1H-pyrrol-2(5H)-one **(2f)**

White solid. Yield: 70%, 88.5 mg. Mp: 189.5–190.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) δ 8.55 (s, 1H), 7.34–7.30 (m, 7H), 7.18 (t, J = 8.9 Hz, 2H), 4.36 (s, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 172.3, 161.7 (d, ^1J = 243.19 Hz), 150.7, 133.3, 131.4 (d, ^3J = 8.32 Hz), 130.6, 129.1, 128.7, 128.6 (d, ^4J = 3.03 Hz), 127.5, 115.2 (d, ^2J = 21.40 Hz), 47.6; ^{19}F NMR (376 MHz, DMSO) δ (ppm) −109.15; IR (KBr), ν (cm^{-1}) 3192, 3049, 1678, 1506, 1371, 1227, 1090, 974, 770, 590; HRMS (ESI) *m/z*: calcd for $C_{16}H_{12}FNO$ Na [M + Na]$^+$ 276.0795; found 276.0802.

4.3.7. 4-(2-Oxo-4-phenyl-1H-pyrrol-3-yl)benzonitrile **(2g)**

White solid. Yield: 64%, 83.2 mg. Mp: 203.3–204.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.66 (s, 1H), 7.82 (d, J = 8.3 Hz, 2H), 7.48 (d, J = 8.3 Hz, 2H), 7.39–7.34 (m, 3H), 7.31–7.30 (m, 2H), 4.41 (s, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 171.6, 153.0, 137.4, 132.8, 132.1, 130.3, 130.1, 129.5, 128.8, 127.6, 118.8, 110.3, 47.9; IR (KBr), ν (cm^{-1}) 3290, 3194, 3045, 2230, 1680, 1447, 1367, 1086, 980, 770, 546; HRMS (ESI) *m/z*: calcd for $C_{17}H_{12}N_2ONa$ [M + Na]$^+$ 283.0842; found 283.0845.

4.3.8. 3-(3′,4′-Dimethoxyphenyl)-4-(4″-fluorophenyl)-1H-pyrrol-2(5H)-one **(2h)**

White solid. Yield: 68%, 106.4 mg. Mp: 190.1–191.4 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.48 (s, 1H), 7.40 (dd, J = 8.7, 5.6 Hz, 2H), 7.20 (t, J = 8.8 Hz, 2H), 6.94 (d, J = 8.3 Hz, 1H), 6.87 (dd, J = 8.3, 1.8 Hz, 1H), 6.84 (d, J = 1.7 Hz, 1H), 4.31 (s, 2H), 3.76 (s, 3H), 3.59 (s, 3H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 172.46, 162.17 (d, ^1J = 245.29 Hz), 148.55, 148.31, 148.28, 131.28, 130.12 (d, ^4J = 2.92 Hz), 129.83 (d, ^4J = 7.33 Hz), 124.33, 121.89, 115.58 (d, ^2J = 21.66 Hz), 112.89, 111.59, 55.40, 55.29, 47.45; ^{19}F NMR (376 MHz, DMSO) δ (ppm) −112.10; IR (KBr), ν (cm^{-1}) 3169, 3057, 2841, 1680, 1514, 1366, 1254, 1138, 1024, 837, 750, 579; HRMS (ESI) *m/z*: calcd for $C_{18}H_{16}FNO_3Na$ [M + Na]$^+$ 336.1006; found 336.1007.

4.3.9. 3-(Naphthalen-1-yl)-4-phenyl-1H-pyrrol-2(5H)-one **(2i)**

White solid. Yield: 65%, 92.6 mg. Mp: 212.1–213.4 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 8.50 (s, 1H), 7.91 (t, J = 7.3 Hz, 2H), 7.76 (d, J = 8.4 Hz, 1H), 7.56–7.51 (m, 1H), 7.48 (t, J = 7.4 Hz, 1H), 7.43–7.39 (m, 2H), 7.21–7.18 (m, 1H), 7.14–7.09 (m, 4H), 4.53 (s, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 175.3, 152.0, 134.1, 132.7, 132.3, 131.7, 130.5, 129.5, 128.9, 128.7, 128.6, 127.9, 127.3, 126.5, 126.2, 125.9, 125.6, 48.5; IR (KBr), ν (cm^{-1}) 3437, 3184, 3057, 2851, 1684, 1447, 1352, 1221, 1103, 779, 640, 534; HRMS (ESI) *m/z*: calcd for $C_{20}H_{15}NONa$ [M + Na]$^+$ 308.1046; found 308.1050.

4.3.10. 3-(Naphthalen-1-yl)-4-(p-tolyl)-1H-pyrrol-2(5H)-one **(2j)**

Yellow solid. Yield: 69%, 103.1 mg. Mp: 222.4–223.8 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.54 (s, 1H), 7.97 (t, J = 8.1 Hz, 2H), 7.63 (d, J = 8.4 Hz, 1H), 7.55 (dd, J = 8.1, 7.1 Hz, 1H), 7.52–7.48 (m, 1H), 7.41–7.38 (m, 1H), 7.32 (dd, J = 7.0, 1.0 Hz, 1H), 7.05 (d, J = 8.3 Hz, 2H), 6.96 (d, J = 8.1 Hz, 2H), 4.59 (d, J = 19.0, 1H), 4.53 (d, J = 19.0, 1H), 2.16 (s, 3H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 173.0, 151.4, 138.9, 133.3, 131.4, 131.2, 130.8, 130.0, 129.1, 128.3, 128.1, 127.5, 127.0, 126.2, 126.0, 125.7, 125.3, 47.4, 20.8; IR (KBr), ν (cm^{-1}) 3269, 1688, 1443, 1352, 1225, 1016, 889, 777, 656; HRMS (ESI) *m/z*: calcd for $C_{21}H_{17}NONa$ [M + Na]$^+$ 322.1202; found 322.1210.

4.3.11. 4-(4′-Fluorophenyl)-3-(naphthalen-1-yl)-1H-pyrrol-2(5H)-one **(2k)**

Yellow solid. Yield: 63%, 95.4 mg. Mp: 209.1–210.8 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.61 (s, 1H), 7.98 (dd, J = 8.1, 4.5 Hz, 2H), 7.63 (d, J = 8.4 Hz, 1H), 7.56 (dd, J = 8.1, 7.1 Hz, 1H), 7.53–7.49 (m, 1H), 7.43–7.39 (m, 1H), 7.34 (d, J = 7.0 Hz, 1H), 7.23–7.18 (m, 2H), 7.04 (t, J = 8.9 Hz, 2H), 4.62 (d, J = 19.2, 1H), 4.55 (d, J = 19.2, 1H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 172.8, 162.3 (d, ^1J = 246.28 Hz), 150.3, 133.3, 131.5, 131.1, 131.0, 129.4 (d, ^4J = 3.13 Hz), 129.3 (d, ^3J = 8.50 Hz), 128.4, 128.3, 127.6, 126.2, 126.1, 125.8, 125.2, 115.6 (d, ^2J = 21.69 Hz), 47.5; ^{19}F NMR (376 MHz, DMSO) δ −111.36; IR (KBr), ν (cm^{-1}) 3182, 3063, 2849, 1684, 1510, 1352, 1232, 1016, 891, 779; HRMS (ESI) *m/z*: calcd for $C_{20}H_{14}FNONa$ [M + Na]$^+$ 326.0952; found 326.0960.

4.3.12. 4-Phenyl-3-(thiophen-2-yl)-1H-pyrrol-2(5H)-one **(2l)**

Yellow solid. Yield: 70%, 84.3 mg. Mp: 177.7–178.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.66 (s, 1H), 7.51–7.41 (m, 6H), 7.29 (d, J = 3.0 Hz, 1H), 7.00 (dd, J = 5.0, 3.7 Hz, 1H), 4.28 (s, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 171.4, 149.8, 134.0, 132.5, 129.1, 128.8, 127.7, 127.0, 126.8, 126.6, 124.9, 48.7; IR (KBr), ν (cm^{-1}) 3186, 3065, 2839, 1688, 1362, 845, 700; HRMS (ESI) *m/z*: calcd for $C_{14}H_{11}SNONa$ [M + Na]$^+$ 264.0454; found 264.0459.

4.3.13. 3-(Furan-2-yl)-4-phenyl-1H-pyrrol-2(5H)-one **(2m)**

White solid. Yield: 58%, 65.2 mg. Mp: 138.1–139.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.62 (s, 1H), 7.60 (d, J = 0.9 Hz, 1H), 7.48 (dd, J = 7.9, 1.6 Hz, 2H), 7.43–7.39 (m, 3H), 7.07 (d, J = 3.3 Hz, 1H), 6.55 (dd, J = 3.3, 1.8 Hz, 1H), 4.32 (s, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 170.7, 148.4, 146.9, 142.8, 133.9, 129.0, 128.2, 128.0, 120.9, 111.3, 110.9, 48.2; IR (KBr), ν (cm^{-1}) 3192, 3067, 2881, 1697, 1447, 1352, 1227, 1013, 878, 743; HRMS (ESI) *m/z*: calcd for $C_{14}H_{11}NO_2Na$ [M + H]$^+$ 248.0682; found 248.0688.

4.3.14. 1-Methyl-3,4-diphenyl-1H-pyrrol-2(5H)-one **(2n)** [24]

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) δ 7.41–7.40 (m, 2H), 7.34–7.27 (m, 8H), 4.29 (s, 2H), 3.18 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 171.00, 147.19, 133.43, 133.01, 132.23, 129.65, 129.20, 128.83, 128.51, 128.18, 127.71, 54.58, 29.54.

4.4. General procedure for the synthesis of 2,3-dihydro-1H-dibenzo[e,g]isoindol-1-one (**3a-3m, 3af**)

The solution of 3,4-diphenyl-1*H*-pyrrol-2(5*H*)-one **2a** (35.3 mg, 0.150 mmol) in EtOH (15 mL) in a quartz tube irradiated with a high-pressure mercury lamp (500 W) at 25 °C for 6 h under an argon atmosphere. Volatiles were removed under reduced pressure and the residue was purified by column chromatography on silica (ethyl acetate/dichloromethane, 1:3) to give **3a** (21.3 mg, 61%). Similarly, compounds **3b-3m, 3af** were obtained with the same method as described above.

*4.4.1. 2,3-Dihydro-1H-dibenzo[e,g]isoindol-1-one **(3a)***

White solid. Yield: 61%, 21.3 mg. mp: 258.2–259.7 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 9.28–9.23 (m, 1H), 8.94–8.88 (m, 2H), 8.75 (s, 1H), 8.10 (d, J = 7.8 Hz, 1H), 7.83 (t, J = 7.2 Hz, 1H), 7.78–7.73 (m, 3H), 4.79 (s, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 171.8, 144.5, 131.2, 129.8, 128.8, 127.5, 127.4, 127.3, 127.0, 126.6, 124.5, 124.3, 123.8, 123.5, 123.4, 43.9; IR (KBr), ν (cm^{-1}) 3069, 2893, 1699, 1423, 1337, 1092, 988, 760, 540; HRMS (APCI) *m/z*: calcd for $C_{16}H_{12}NO$ $[M + H]^+$ 234.0913; found 234.0913.

*4.4.2. 2,3-Dihydro-9-methoxy-1H-dibenzo[e,g]isoindol-1-one **(3b)***

White solid. Yield: 59%, 23.3 mg. mp: 251.3–252.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 9.16 (d, J = 8.9 Hz, 1H), 8.93 (d, J = 8.3 Hz, 1H), 8.69 (s, 1H), 8.27 (s, 1H), 8.05 (d, J = 7.8 Hz, 1H), 7.80 (t, J = 7.5 Hz, 1H), 7.74 (t, J = 7.3 Hz, 1H), 7.38 (d, J = 8.6 Hz, 1H), 4.73 (s, 2H), 4.01 (s, 3H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 171.9, 158.3, 141.8, 131.7, 130.7, 128.3, 127.5, 127.0, 125.0, 124.4, 124.4, 124.2, 121.7, 117.3, 105.0, 55.5, 43.8; IR (KBr), ν (cm^{-1}) 1697, 1337, 1220, 1153, 1098, 858, 733; HRMS (APCI) *m/z*: calcd for $C_{17}H_{14}NO_2$ $[M + H]^+$ 264.1019; found 264.1027.

*4.4.3. 2,3-Dihydro-9,10-dimethoxy-1H-dibenzo[e,g]isoindol-1-one **(3c)** [8]*

^{1}H NMR (400 MHz, DMSO) δ (ppm) 8.85 (d, J = 8.4 Hz, 1H), 8.72 (s, 2H), 8.20 (s, 1H), 8.01 (d, J = 7.8 Hz, 1H), 7.76 (t, J = 7.2 Hz, 1H), 7.66 (t, J = 7.4 Hz, 1H), 4.72 (s, 2H), 4.03 (s, 3H), 3.93 (s, 3H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 172.1, 149.6, 149.1, 142.1, 130.8, 128.1, 126.2, 125.8, 124.7, 124.3, 123.8, 123.7, 122.3, 104.4, 103.7, 55.7, 55.4, 43.7.

*4.4.4. 2,3-Dihydro-11-methyl-1H-dibenzo[e,g]isoindol-1-one **(3d)***

Yellow solid. Yield: 57%, 21.1 mg. mp: 212.2–214.1 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.88 (d, J = 8.4 Hz, 1H), 8.76 (d, J = 8.2 Hz, 1H), 8.57 (s, 1H), 8.05 (d, J = 7.8 Hz, 1H), 7.80 (t, J = 7.3 Hz, 1H), 7.73 (t, J = 7.4 Hz, 1H), 7.61 (t, J = 7.7 Hz, 1H), 7.52 (d, J = 7.1 Hz, 1H), 4.78 (s, 2H), 3.03 (s, 3H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 171.6, 146.7, 135.8, 131.8, 131.4, 130.6, 129.1, 127.5, 127.0, 126.6, 126.5, 126.4, 124.1, 123.9, 121.2, 43.9, 25.1; IR (KBr), ν (cm^{-1}) 3072, 2924, 2855, 1695, 1456, 1261, 1097, 804, 527; HRMS (APCI) *m/z*: calcd for $C_{17}H_{14}NO$ $[M + H]^+$ 248.1070; found 248.1079.

*4.4.5. 2,3-Dihydro-6-methyl-1H-dibenzo[e,g]isoindol-1-one **(3e)***

Yellow solid. Yield: 64%, 23.7 mg. mp: 250.5–251.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 9.26–9.19 (m, 1H), 8.90–8.85 (m, 1H), 8.73 (s, 1H), 8.69 (s, 1H), 7.98 (d, J = 8.1 Hz, 1H), 7.71 (dd, J = 9.0, 4.8 Hz, 2H), 7.59 (d, J = 8.1 Hz, 1H), 4.75 (s, 2H), 2.62 (s, 3H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 171.9, 144.6, 138.6, 131.3, 129.6, 129.0, 127.5, 127.3, 126.8, 124.5, 124.3, 123.5, 123.5, 123.5, 123.4, 43.9, 21.8; IR (KBr), ν (cm^{-1}) 3179, 3065, 2885, 1691, 1454, 1339, 1223, 1036, 779; HRMS (APCI) *m/z*: calcd for $C_{17}H_{14}NO$ $[M + H]^+$ 248.1070; found 248.1082.

*4.4.6. 2,3-Dihydro-9-fluoro-1H-dibenzo[e,g]isoindol-1-one **(3f)***

Yellow solid. Yield: 63%, 23.7 mg. mp: 264.8–265.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 9.27 (dd, J = 8.9, 6.4 Hz, 1H), 8.87 (d, J = 8.1 Hz, 1H), 8.78 (s, 1H), 8.66 (dd, J = 11.7, 2.4 Hz, 1H), 8.07 (d, J = 7.6 Hz, 1H), 7.83–7.76 (m, 2H), 7.61 (m, 1H), 4.75 (s, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 171.6, 161.1 (d, ^1J = 242.02 Hz), 143.8, 132.0 (d, ^3J = 8.38 Hz), 130.6 (d, ^4J = 4.20 Hz), 128.8, 128.1, 127.0, 126.0 (d, ^3J = 8.76 Hz), 124.5, 124.3, 124.1, 124.1, 116.1 (d, ^2J = 23.43 Hz), 108.8 (d, ^2J = 22.40 Hz), 43.9; ^{19}F NMR (376 MHz, DMSO) δ (ppm) −112.19; IR (KBr), ν (cm^{-1}) 3069, 2883, 1691, 1522, 1441, 1340, 1178, 1140, 829, 762, 594; HRMS (APCI) *m/z*: calcd for $C_{16}H_{11}FNO$ $[M + H]^+$ 252.0819; found 252.0828.

*4.4.7. 2,3-Dihydro-3-oxo-1H-dibenzo[e,g]isoindole-6-carbonitrile **(3g)***

Yellow solid. Yield: 67%, 25.9 mg. mp: 289.2–290.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 9.42 (s, 1H), 9.33 (d, J = 8.3 Hz, 1H), 9.02 (d, J = 8.2 Hz, 1H), 8.87 (s, 1H), 8.12 (d, J = 7.7 Hz, 1H), 8.05 (d, J = 8.3 Hz, 1H), 7.88 (t, J = 7.4 Hz, 1H), 7.83 (t, J = 7.3 Hz, 1H), 4.81 (s, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 170.6, 147.2, 130.0, 129.3, 129.3, 128.9, 128.4, 128.2, 128.0, 126.6, 124.2, 124.0, 123.7, 123.5, 118.6, 109.1, 43.7; IR (KBr), ν (cm^{-1}) 3074, 2874, 2228, 1690, 1450, 1070, 760, 554; HRMS (APCI) *m/z*: calcd for $C_{17}H_{11}N_2O$ $[M + H]^+$ 259.0866; found 259.0875.

*4.4.8. 2,3-Dihydro-6-fluoro-9,10-dimethoxy-1H-dibenzo[e,g] isoindol-1-one **(3h)***

Yellow solid. Yield: 62%, 28.9 mg. mp: 276.3–278.1 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.68 (dd, J = 17.3, 5.5 Hz, 3H), 8.14 (s, 1H), 8.07 (dd, J = 8.8, 6.1 Hz, 1H), 7.55–7.52 (m, 1H), 4.70 (s, 2H), 4.03 (s, 3H), 3.93 (s, 3H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 171.9, 162.0 (d, ^1J = 243.07 Hz), 150.0, 149.2, 141.8, 132.8 (d, ^3J = 8.89 Hz), 127.0 (d, ^3J = 9.19 Hz), 124.3 (d, ^4J = 3.54 Hz), 123.1, 122.8, 122.7, 115.2 (d, ^2J = 24.39 Hz), 108.9 (d, ^2J = 22.15 Hz), 104.9, 103.6, 55.9, 55.4, 43.7; ^{19}F NMR (376 MHz, DMSO) δ (ppm) −110.94; IR (KBr), ν (cm^{-1}) 3071, 1686, 1522, 1431, 1261, 1157, 1026, 829, 781, 608; HRMS (APCI) *m/z*: calcd for $C_{18}H_{15}FNO_3$ $[M + H]^+$ 312.1030; found 312.1038.

*4.4.9. 12,13-Dihydro-11H-benzo[e]naphtho[2,1-g]isoindol-11-one **(3i)***

White solid. Yield: 54%, 22.9 mg. mp: 238.1–239.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 9.95 (d, J = 8.3 Hz, 1H), 8.95 (d, J = 8.4 Hz, 1H), 8.88 (d, J = 9.5 Hz, 2H), 8.13 (dd, J = 15.0, 8.5 Hz, 2H), 8.05 (d, J = 7.6 Hz, 1H), 7.85 (t, J = 7.5 Hz, 1H), 7.77 (t, J = 7.3 Hz, 1H), 7.65 (m, 2H), 4.89 (s, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 171.8, 146.8, 132.6, 131.3, 129.3, 129.1, 129.1, 128.0, 127.5, 127.3, 126.7, 126.5, 126.3, 126.0, 124.9, 124.2, 124.2, 121.4, 43.9; IR (KBr), ν (cm^{-1}) 3094, 2901, 1680, 1406, 1119, 797, 758, 538; HRMS (APCI) *m/z*: calcd for $C_{20}H_{14}NO$ $[M + H]^+$ 284.1070; found 284.1079.

*4.4.10. 3-Methyl-12,13-dihydro-11H-benzo[e]naphtho[2,1-g] isoindol-11-one **(3j)***

Yellow solid. Yield: 60%, 26.7 mg. mp: 256.4–257.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 9.94 (d, J = 8.4 Hz, 1H), 8.90 (d, J = 9.1 Hz, 1H), 8.80 (d, J = 19.2 Hz, 2H), 8.12 (d, J = 9.0 Hz, 1H), 8.05 (dd, J = 7.5, 4.9 Hz, 2H), 7.65 (dd, J = 10.7, 3.9 Hz, 1H), 7.61 (dd, J = 10.2, 4.8 Hz, 2H), 4.86 (s, 2H), 2.65 (s, 3H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 171.9, 146.8, 139.0, 132.6, 131.5, 131.3, 129.2, 129.2, 129.0, 127.8, 127.3, 126.7, 126.2, 125.4, 124.9, 124.6, 124.1, 123.7, 121.5, 43.9, 21.9; IR (KBr), ν (cm^{-1}) 3072, 2854, 1670, 1450, 1337, 1215, 1028, 760, 644; HRMS (APCI) *m/z*: calcd for $C_{21}H_{16}NO$ $[M + H]^+$ 298.1226; found 298.1225.

*4.4.11. 3-Fluoro-12,13-dihydro-11H-benzo[e]naphtho[2,1-g] isoindol-11-one **(3k)***

Yellow solid. Yield: 52%, 23.5 mg. mp: 260.1–261.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 9.94 (d, J = 8.4 Hz, 1H), 8.87 (s, 1H), 8.80 (d, J = 9.1 Hz, 1H), 8.72 (dd, J = 12.0, 2.2 Hz, 1H), 8.19 (dd, J = 8.8, 6.0 Hz, 1H), 8.10 (d, J = 9.0 Hz, 1H), 8.04 (d, J = 7.4 Hz, 1H), 7.68–7.61 (m, 3H), 4.84 (s, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 171.6, 162.5 (d, ^1J = 244.45 Hz), 146.6, 133.3 (d, ^3J = 9.00 Hz), 132.9, 131.5, 128.9, 128.9 (d, ^4J = 4.38 Hz), 128.0, 127.3, 127.2 (d, ^3J = 9.27 Hz), 127.0, 126.7, 125.8, 125.0, 123.5, 121.7, 116.7 (d, ^2J = 24.18 Hz), 109.2 (d, ^2J = 22.86 Hz), 43.9; ^{19}F NMR (376 MHz, DMSO) δ (ppm) −109.25; IR (KBr), ν (cm^{-1}) 3072, 1666, 1423, 1263, 1219, 1016, 870, 760, 596; HRMS (APCI) *m/z*: calcd for $C_{20}H_{13}FNO$ $[M + H]^+$ 302.0976; found 302.0982.

*4.4.12. 8,9-Dihydro-10H-benzo[e]thieno[3,2-g]isoindol-10-one **(3l)***

Yellow solid. Yield: 71%, 25.4 mg. mp: 261.2–262.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.85 (s, 1H), 8.62 (d, J = 8.2 Hz, 1H), 8.29 (d, J = 5.3 Hz, 1H), 8.10 (d, J = 8.0 Hz, 1H), 7.99 (d, J = 5.2 Hz, 1H), 7.77 (t, J = 7.4 Hz, 1H), 7.68 (t, J = 7.4 Hz, 1H), 4.85 (s, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 170.1, 140.9, 137.1, 130.1, 129.7, 128.4, 128.1, 126.3, 126.0, 124.9, 124.9, 124.5, 122.1, 45.1; IR (KBr), ν (cm^{-1}) 3180, 3072, 2876, 1693, 1346, 1167, 966, 868, 766; HRMS (APCI) *m/z*: calcd for $C_{14}H_{10}NOS$ $[M + H]^+$ 240.0478; found 240.0485.

*4.4.13. 8,9-Dihydro-10H-benzo[e]furo[3,2-g]isoindol-10-one **(3m)***

White solid. Yield: 52%, 17.4 mg. mp: 248.4–249.9 °C. ^{1}H NMR (600 MHz, DMSO) δ (ppm) 8.73 (s, 1H), 8.42 (d, J = 8.2 Hz, 1H), 8.20 (d, J = 2.0 Hz, 1H), 8.11 (d, J = 8.2 Hz, 1H), 7.78–7.75 (m, 1H), 7.70 (d, J = 2.0 Hz, 1H), 7.66–7.64 (m, 1H), 4.85 (s, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 168.5, 146.1, 145.6, 141.9, 128.8, 128.3, 125.6, 125.0, 124.5, 124.5, 123.6, 117.7, 106.1, 44.9; IR (KBr), ν (cm^{-1}) 3179, 3069, 2876, 1701, 1393, 1346, 1175, 1015, 885, 771, 646; HRMS (APCI) *m/z*: calcd for $C_{14}H_{10}NO_2$ $[M + H]^+$ 224.0706; found 224.0711.

*4.5. General procedure for the synthesis of 2-Acetyl-2,3-dihydro-1H-dibenzo [e,g]isoindol-1-one **(3aa-3ae)***

The solution of 1-acetyl-3,4-diphenyl-1,5-dihydropyrrol-2-one **1a** (41.6 mg, 0.150 mmol) in EtOH (15 mL) in a quartz tube irradiated with a high-pressure mercury lamp (500 W) at 25 °C for 1 h under an argon atmosphere. Volatiles were removed under reduced pressure and the residue was purified by column chromatography on silica (ethyl acetate/petroleum ether, 1:5) to give **3aa** (28.9 mg, 70%). Similarly, compounds **3ab-3ae** were obtained with the same method as described above.

*4.5.1. 2-Acetyl-2,3-dihydro-1H-dibenzo[e,g]isoindol-1-one **(3aa)***

White solid. Yield: 70%, 28.9 mg. mp: 226.2–227.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 9.14–9.12 (m, 1H), 8.67 (d, J = 8.3 Hz, 1H), 8.65–8.63 (m, 1H), 7.84 (d, J = 7.9 Hz, 1H), 7.80 (t, J = 7.6 Hz, 1H), 7.73–7.70 (m, 2H), 7.67 (t, J = 7.4 Hz, 1H), 4.94 (s, 2H), 2.74 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 171.0, 168.8, 143.0, 133.0, 130.6, 130.1, 128.3, 127.8, 127.8, 127.2, 126.0, 124.4, 124.3, 123.8, 123.6, 123.0, 46.8, 25.0; IR (KBr), ν (cm^{-1}) 1726, 1684, 1379, 1302, 1126, 895, 754, 617; HRMS (ESI) *m/z*: calcd for $C_{18}H_{13}NO_2Na$ $[M + Na]^+$ 298.0838; found 298.0830.

*4.5.2. 2-Acetyl-9-methoxy-2,3-dihydro-1H-dibenzo[e,g]isoindol-1-one **(3ab)***

Yellow solid. Yield: 72%, 32.9 mg. mp: 277.2–278.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$, TFA-d_1) δ (ppm) 8.77 (d, J = 8.9 Hz, 1H), 8.48 (d, J = 8.4 Hz, 1H), 7.89 (d, J = 1.8 Hz, 1H), 7.74 (t, J = 7.5 Hz, 1H), 7.64–7.59 (m, 2H), 7.25 (dd, J = 6.7, 2.0 Hz, 1H), 4.67 (s, 2H), 4.01 (s, 3H), 2.71 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$, TFA-d_1) δ (ppm) 174.3, 168.8, 159.0, 141.0, 132.4, 132.3, 130.3, 128.0, 125.8, 125.5, 124.5, 123.8, 122.5, 121.0, 117.5, 105.4, 55.7, 46.9, 24.3; IR (KBr), ν (cm^{-1}) 2937, 2837, 1726, 1679, 1522, 1342, 1124, 1042, 895, 762, 627, 544; HRMS (APCI) *m/z*: calcd for $C_{19}H_{16}NO_3$ $[M + H]^+$ 306.1125; found 306.1134.

*4.5.3. 2-Acetyl-3-oxo-2,3-dihydro-1H-dibenzo[e,g]isoindole-6-carbonitrile **(3ac)***

Yellow solid. Yield: 78%, 35.1 mg. mp: 293.5–294.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$, TFA-d_1) δ (ppm) 9.31 (d, J = 8.4 Hz, 1H), 9.10 (s, 1H), 8.76 (d, J = 8.4 Hz, 1H), 8.09 (d, J = 7.9 Hz, 1H), 8.02–7.98 (m, 2H), 7.89 (t, J = 7.5 Hz, 1H), 5.27 (s, 2H), 2.87 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$, TFA-d_1) δ (ppm) 174.9, 168.6, 147.3, 132.3, 132.1, 130.8, 130.1, 129.9, 129.7, 129.1, 126.3, 125.6, 125.1, 124.1, 122.5, 110.3, 47.6, 24.5; IR (KBr), ν (cm^{-1}) 3099, 2930, 2226, 1726, 1686, 1306, 1126, 976, 841, 756, 538; HRMS (APCI) *m/z*: calcd for $C_{19}H_{13}N_2O_2$ $[M + H]^+$ 301.0972; found 301.0976.

*4.5.4. 2-Acetyl-6-methyl-2,3-dihydro-1H-dibenzo[e,g]isoindol-1-one **(3ad)***

White solid. Yield: 73%, 31.6 mg. mp: 293.5–294.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 9.15–9.13 (m, 1H), 8.65–8.63 (m, 1H), 8.46 (s, 1H), 7.75 (d, J = 8.1 Hz, 1H), 7.70 (dd, J = 6.2, 3.3 Hz, 2H), 7.50 (d, J = 8.1 Hz, 1H), 4.95 (s, 2H), 2.74 (s, 3H), 2.66 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 171.0, 168.9, 143.1, 140.6, 133.2, 130.3, 129.4, 128.1, 127.5, 127.4, 124.3, 124.3, 123.9, 123.7, 123.0, 122.7, 46.8, 24.9, 22.6; IR (KBr), ν (cm^{-1}) 3072, 1728, 1684, 1290, 1192, 972, 891, 777, 735, 584; HRMS (APCI) *m/z*: calcd for $C_{19}H_{16}NO_2$ $[M + H]^+$ 290.1176; found 290.1186.

*4.5.5. 9-Acetyl-8,9-dihydro-10H-benzo[e]furo[3,2-g]isoindol-10-one **(3ae)***

Yellow solid. Yield: 64%, 27.0 mg. mp: 243.2–244.9 °C. ^{1}H NMR (600 MHz, $CDCl_3$, TFA-d_1) δ (ppm) 8.40 (d, J = 8.2 Hz, 1H), 8.00 (d, J = 5.2 Hz, 1H), 7.90 (d, J = 8.1 Hz, 1H), 7.81 (t, J = 7.5 Hz, 1H), 7.76 (d, J = 5.2 Hz, 1H), 7.67 (t, J = 7.5 Hz, 1H), 5.09 (s, 2H), 2.81 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$, TFA-d_1) δ (ppm) 174.1, 168.0, 139.7, 138.4, 132.2, 130.5, 130.1, 128.7, 127.1, 125.4, 125.1, 124.6, 122.9, 121.7, 48.4, 24.4; IR (KBr), ν (cm^{-1}) 3098, 2924, 1722, 1688, 1344, 1134, 968, 872, 727, 623, 534; HRMS (APCI) *m/z*: calcd for $C_{16}H_{12}NO_2S$ $[M + H]^+$ 282.0583; found 282.0589.

*4.5.6. 2,3-Dihydro-2-methyl-1H-dibenzo[e,g]isoindol-1-one **(3af)** [7b]*

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 9.34–9.33 (m, 1H), 8.67 (d, J = 8.3 Hz, 1H), 8.64 (d, J = 8.9 Hz, 1H), 7.76 (d, J = 7.9 Hz, 1H), 7.72–7.67 (m, 3H), 7.61 (t, J = 7.4 Hz, 1H), 4.57 (s, 2H), 3.28 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 170.24, 140.83, 131.85, 130.41, 128.42, 127.70, 127.65, 127.22, 127.14, 126.62, 125.62, 124.68, 123.78, 123.60, 122.76, 50.84, 29.53.

*4.6. General procedure for the synthesis of 1H-dibenzo[e,g] isoindole-1,3(2H)-dione **(4)***

Sodium methoxide in MeOH (100 μL, 0.1 mmol, 30%) was added to the solution of **3a** or **3aa** (0.1 mmol) in DMSO (5 mL) at room temperature under an oxygen atmosphere. When the reaction was completed (detected by TLC), water was added and extracted with CH_2Cl_2. The organic layer was combined, washed (brine), and concentrated *in vacuo*. The residue was purified by silica column chromatography (ethyl acetate/petroleum ether, 1:2) to afford the product **4** in 72% and 60%.

*4.6.1. 1H-dibenzo[e,g]isoindole-1,3(2H)-dione **(4)** [17b]*

^{1}H NMR (600 MHz, DMSO) δ (ppm) 11.30 (s, 1H), 8.99–8.94 (m, 4H), 7.89–7.86 (m, 2H), 7.84–7.82 (m, 2H); ^{13}C NMR (151 MHz, DMSO) δ (ppm) 170.6, 132.7, 129.3, 128.4, 127.5, 125.1, 124.8, 123.7.

*4.7. Synthesis of 1 mmol Scale **3aa***

The solution of 1-acetyl-3,4-diphenyl-1,5-dihydropyrrol-2-one **1a** (277.0 mg, 1 mmol) in EtOH (100 mL) in a quartz tube irradiated with a high-pressure mercury lamp (500 W) at 25 °C for 4.5 h under an argon atmosphere. Volatiles were removed under reduced pressure and the residue was purified by column chromatography on silica (ethyl acetate/petroleum ether, 1:5) to afford the corresponding products **3aa** (176.0 mg, 64%).

4.8. Detection of H_2 generated by Annulation of **1g**

Using Ar as carrier gas, thermal conductivity detector (TCD temperature: 120 °C) and stainless steel column (column length: 2 m, column temperature: 40 °C, Tam TDS-01: 60–80 mesh) were used for gas chromatography analysis. Under the condition of gas velocity of 0.06 Mpa and the flow rate of 50 mL/min, gas was analyzed at room temperature. The ethanol (125 mL) was degassed for an hour by ultrasonic at room temperature. Then, sodium sulfite (14 g) and hydroquinone (1.1 g) was added and the mixture was refluxed for 3 h to remove the oxygen in the solvent. The compound of **1g** (151 mg, 0.500 mmol) was dissolved in 50 mL deoxidization chromatographic ethanol at room temperature in a quartz tube **I**. In the same conditions, quartz tube **II** without compound **1g** was prepared. All operations discussed above were performed under argon atmosphere. With strict sealing, quartz tube **I** and **II** were irradiated with a high-pressure mercury lamp (500 W) at room temperature for 1.5 h. According to the experiment data, the reference substance H_2 retention time t_{R1} was 1.413 min (Figure S3); the retention time t_{R2} in quartz tube **I** was 1.381 min (Figure S4), indicating the presence of H_2 in the tube **I**. However, H_2 was not observed in quartz tube **II**, further confirming the presence of H_2 was generated during the annulation of **1g.**

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tet.2021.131981.

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