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Studies in microwave mediated solvent-free synthesis of 5-aryl-2,3-diphenylpyrroles from 4-aryl-1,2-diphenyl-2-butene-1,4-diones

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4-Aryl-1,2-diphenyl-2-butene-1,4-diones and ammonium formate when irradiated with microwaves furnish 5-aryl-2,3-diphenylpyrroles in good yield under solvent-free conditions.

Keywords: Triarylpyrroles, microwave, solvent-free synthesis

The pyrrole ring is a fundamental heterocycle found as a core structure in many natural and non-natural products[1]. In view of its importance, there is a continuous effort to develop expedient synthesis of pyrroles[2]. It is of interest to synthesize 5-aryl-2,3-triphenylpyrroles because triarylpyrroles, in general, have been evaluated as estrogenase blockers[3]. A convenient synthesis of some 5-aryl-2,3-diphenyl-pyrroles starting from readily available 4-aryl-1,2-diphenyl-2-butene-1,4-diones and ammonium formate under solvent-free microwave irradiation conditions[4] is reported here.

A sample of 1,2,4-triphenyl-2-butene-1,4-dione (dibenzoyl styrene) **1a**, a condensation product of acetophenone and benzil, loaded on ammonium formate, was subjected to microwave irradiation at 270W for 2 min. by which time reaction was complete (TLC). From the crude product 2,3,5-triphenyl-1*H*-pyrrole **2a** was isolated in 65% yield (**Scheme I**). A small amount (28%) of 3,3,5-triphenyl-2,3-dihydro-1*H*-2-pyrrolone was also formed in this reaction. It is already shown that 2,3,5-triphenylpyrrole could be prepared conveniently from 1,2,4-triphenyl-2-butene-1,4-dione using bench top reagents, ammonium formate and palladium on carbon[5]. The present finding shows that the transformation can be achieved conveniently under solvent-free microwave irradiation, as a load on ammonium formate, without expensive palladium on carbon. It was shown earlier[6] that 3,3,5-triphenyl-2,3-dihydro-1*H*-2-pyrrolone was the only product formed when the microwave irradiation **1a** and ammonium formate was carried in polyethylene glycol (PEG-200). Thus, the present study shows that under solvent-free conditions pyrrole **2a** is the major product, whereas under solution phase conditions pyrrolone was the major product.

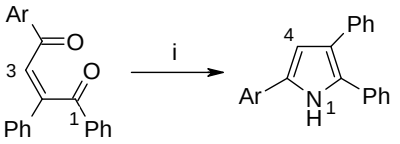
The transformation of **1a** to **2a** was taken as a test case to optimize conditions required for the formation of pyrrole. The conversion of dione **1a** to pyrrole **2a** did not take place when aqueous ammonia was used instead of ammonium formate. This result indicated that in addition to ammonia, which is released by disintegration of ammonium formate, formic acid is also required to promote transformation of **1a** to **2a**. By varying power input of the microwave oven it was found that the transformation of **1a** to **2a** worked well at 270W. Generality of the transformation of 2-butene-1,4-dione **1a** to triphenylpyrrole **2a** was proved by subjecting butenediones **1b-g** having electron-withdrawing Cl (**1b**) or electron-donating CH_3 (**1c**), 4-OCH_3 (**1d**), 3,4-$(OCH_3)_2$ (**1e**) groups in the aryl ring to the reaction with ammonium formate under solvent-free conditions. In all the cases pyrroles **2b-e** were formed in good yield (59-66%). There was no correlation between electron-withdrawing or electron-donating nature of aryl substituent to the yields of pyrrole **2**, or time taken for completion of the reaction. The triarylpyrroles **2b-e** are new compounds and they were characterized on the basis of analytical and spectra (IR, ^{1}H NMR, ^{13}C NMR and DEPT) which compared well with the spectra of the parent compound **2a**. In continuation, 2-butene-1,4-diones **1f** and **1g** having biphenyl and 2-naphthyl groups on C-4 respectively were subjected to microwave irradiation as a load on ammonium formate. In both the cases triarylpyrroles, *viz*, 5-(biphen-4yl)-2,3-diphenyl-1*H*-pyrrole **2f** and 5-(2-

naphthyl)-2,3-diphenyl-1*H*-pyrrole **2g** were formed in good yield (46-66%). Among the pyrroles **2b-g** prepared, **2f** and **2g** are interesting compounds because they have four aryl rings in conjugation.

It was intended to prepare 5-(9-anthryl)-2,3-diphenyl-1*H*-pyrrole **6** having pyrrole unit attached to C-9 of anthracene unit (**Scheme II**). The precursor, 4-(9-anthryl)-1,2-diphenyl-2-butene-1,4-dione **4** was prepared from 9-acetylanthracene[7] and benzil using KOH as a base. Microwave mediated irradiation of the 2-butene-1,4-dione **4** as thin film on ammonium formate furnished double bond reduced product 4-(9-anthryl)-1,2-diphenyl-1,4-butanedione **5** only, and there was no trace of desired pyrrole **6**. Entropic and steric factors to ring cyclization leading to intermediates for pyrrole **6** may be acting as a deterrent for the progress of the reaction.

Possible mechanism for the formation of pyrroles **1a-g** is given in **Scheme III**. Under the microwave irradiation, ammonium formate decomposes to ammonia and formic acid[8]. Liberated formic acid reduces the enone double bond in **1** through transfer hydrogenation to furnish diones **7**. The Paal-Knorr transformation of dione **7** with ammonia furnishes 5-aryl-2,3-diphenylpyrroles **2a-g** through a reaction cascade as shown in **Scheme III**. Final dehydration assisted by formic acid delivers the pyrrole **2**.

In conclusion it was shown that triaryl pyrroles **2a-g** can be prepared conveniently in good yield by the reaction of 2-butene-1,4-diones **1a-g** with ammonium formate under solvent-free conditions and microwave irradiation.

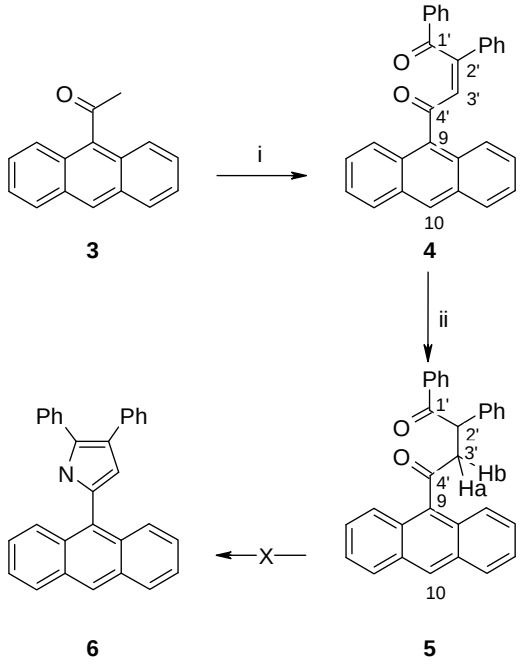


1a-g **2a-g**

1a: Ar = C_6H_5 **2a**: Ar = C_6H_5
1b: Ar = 4-Cl-C_6H_4 **2b**: Ar = 4-Cl-C_6H_4
1c: Ar = 4-CH_3-C_6H_4 **2c**: Ar = 4-CH_3-C_6H_4
1d: Ar = 4-OCH_3-C_6H_4 **2d**: Ar = 4-OCH_3-C_6H_4
1e: Ar = 3,4-$(OCH_3)_2$-C_6H_3 **2e**: Ar = 3,4-$(OCH_3)_2$-C_6H_3
1f: Ar = 4-C_6H_5-C_6H_4 **2f**: Ar = 4-C_6H_5-C_6H_4
1g: Ar = 2-$C_{10}H_7$ **2g**: Ar = 2-$C_{10}H_7$

Reagents and conditions: (i) $NH_4^+HCOO^-$, MW 270 W, 2 min, 46-66%.

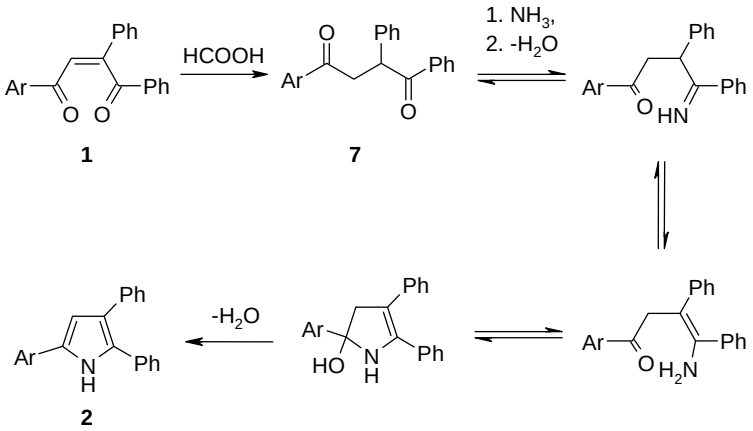
Scheme I



3 **4** **6** **5**

Reagents and conditions: (i) Benzil, 10% KOH in EtOH, 20°C, 2 hr, 89%; (ii) $NH_4^+HCOO^-$, MW 270W, 2 min, 36%.

Scheme II



1 **7** **2**

Scheme III

Experimental Section

General solvents: ethanol, methanol, ethyl acetate, acetone, dichloromethane, diethyl ether and hexanes were distilled before use. The progression of all the reactions was monitored by TLC plates (silica gel, 0.25 mm thickness) using hexane/ethyl acetate mixture as eluent and the plates were visualized with iodine vapours or UV light. Column chromatography was accomplished on silica gel (100-200 mesh, SD fine, India) using hexane-ethyl acetate as eluent. Melting points were recorded by a Gallenkamp melting point apparatus and were uncorrected. The UV spectra were recorded as dilute solution in methanol using Hitachi ratio beam U-1800 spectrometer. The IR spectra were recorded as KBr pellets using ABB BOMEM MB 104 FT-IR spectrometer. The ^{1}H NMR spectra, the ^{13}C NMR spectra and DEPT were recorded as solution in $CDCl_3$ using JEOL 60 MHz or Bruker 300 MHz or JEOL 400 MHz spectrometers with TMS (0 ppm) or $CDCl_3$ (77 ppm) as the internal standard. The ene-diones **1a-g** were prepared *via* dehydrative condensation of the corresponding acetophenone and benzil following literature procedures[9]. Details of experimental conditions and spectroscopic data for hitherto unknown ene-diones (**1e**, **1g** and **4**) are given below. Stereochemistry of double bond in **1** was taken as *Z* in analogy with the findings from UV spectra, which exhibited λ_{max} at 302 nm and 235 nm[10].

Representative procedure for the synthesis of 2-butene-1,4-diones 1e, 1g and 4 from corresponding aryl methyl ketones: **Synthesis of 4-(3,4-dimethoxyphenyl)-1,2-diphenyl-2-butene-1,4-dione 1e**. The solution of 3,4-dimethoxyacetophenone (3.56 g, 23.7 mmoles) and benzil (5 g, 23.7 mmoles) in 10% ethanolic KOH (75 mL) was stirred for 2 hr and left at 5°C overnight. Resulting solid was filtered and recrystallized using hexane and dichloromethane (95:5) to furnish 4-(3,4-dimethoxyphenyl)-1,5-diphenylbut-2-ene-1,4-dione as pale yellow solid in 74% yield; m.p. 182-84°C; R_f (90:10 hexane/EtOAc) 0.36; UV (methanol): 344 nm (logε = 3.54), 306 nm (logε = 3.58), 236 nm (logε = 3.64); IR (KBr): 3063, 1671, 1646, 1575, 1512, 1258, 776 and 690 cm^{-1}; ^{1}H NMR (60 MHz; CCl_4:$CDCl_3$, 1:1): δ 4.06 (s, 3H, -OCH_3), 4.14 (s, 3H, -OCH_3), 7.1 (d, *J* = 7.2 Hz, 1H, Ar-H), 7.4-8.2 (m, 13H, Ar-H and =CH). Anal. Calcd for $C_{23}H_{20}O_4$: C, 77.41; H, 5.41. Found: C, 77.37; H, 5.45%.

4-(2-Naphthyl)-1, 2-diphenyl-2-butene-1,4-dione 1g. Yield 74% (pale yellow solid); m.p. 156-58°C; R_f (85:15 hexane/EtOAc) 0.52; UV (methanol): 311 nm (logε = 3.57), 262 nm (logε = 3.56); IR (KBr): 3055, 1650, 1569, 1218, 763 and 696 cm^{-1}; ^{1}H NMR (60 MHz; CCl_4: $CDCl_3$, 1:1): δ 7.2-8.0 (m, 17H, Ar-H and =CH), 8.5 (s, 1H, C-1H of naphthalene moiety. Anal. Calcd for $C_{26}H_{18}O_2$: C, 86.17; H, 5.01. Found: C, 86.09; H, 5.07%.

4-(9-Anthryl)-1,2-diphenyl-2-butene-1,4-dione 4. Yield 89% (bright yellow solid); m.p. 160-62°C; R_f (85:15 hexanes/EtOAc) 0.43; UV (methanol): 380 nm (logε = 3.30), 361 nm (logε = 3.34), 341 nm (logε = 3.39), 305 nm (logε = 3.36); IR (KBr): 3053, 1671, 1554, 1111, 743 and 689 cm^{-1}; ^{1}H NMR (60 MHz; CCl_4: $CDCl_3$, 1:1): δ 7.1-8.1 (m, 19H, Ar-H and =CH), 8.4 (s, 1H, C-10H of anthracene moiety). Anal. Calcd for $C_{30}H_{20}O_2$: C, 87.36; H, 4.89. Found: C, 87.29; H, 4.73%.

Representative procedure for the synthesis of pyrroles 2a-g from 2-butene-1,4-diones 1a-g: Synthesis of 2,3,5-triphenyl-1*H*-pyrrole 2a. To the solution of 2-butene-1,4-dione **1a** (100 mg, 0.32 mmoles) in 5 mL dichloromethane ammonium formate (404 mg, 6.4 mmoles) was added and swirled for 2 min, then the solvent was evaporated *in vacuo*, to result in a free flowing solid. This solid was irradiated in a domestic microwave oven at 200 W for 2 min. After completion of the reaction (TLC), the reaction mixture, which by now became a viscous mass, was cooled (rt) and transferred on to a short column of silica gel (5 × 1 cm) and eluted with hexane/EtOAc (90:10 to 50:50). Removal of solvent from pooled fractions yielded 2,3,5-triphenyl-1*H*-2-pyrrole **2a** as a white solid (62 mg, 65%); m.p. 138-40°C, lit. (ref. 11) 140°C; R_f (90:10 hexanes/EtOAc) 0.38; UV (methanol): 237 nm (logε = 4.54), 255 nm (logε = 4.30), 317 nm (logε = 4.51); IR (KBr): 3426, 3071, 1603, 1484, 760 and 697 cm^{-1}; ^{1}H NMR (300 MHz; $CDCl_3$): δ 6.7 (s, 1H, C-4H of pyrrole ring), 7.2-7.6 (m, 15H, Ar-H), 8.4 (brs, 1H, -NH); ^{13}C NMR (100 MHz, $CDCl_3$) 108.5 (*C*-4H of pyrrole ring), 123.7 (Ar-*C*H × 2), 123.8 (*C*-3 of pyrrole ring), 125.9 (Ar-*C*H), 126.5 (Ar-*C*H), 126.9 (Ar-*C*H), 127.4 (Ar-*C*H × 2), 128.3 (Ar-*C*H × 2), 128.4 (Ar-*C*H × 2), 128.7 (Ar-*C*H ×2), 128.9 (Ar-*C*H × 2), 129.3 (Ar-*C*), 132.2 (*C*-2 of pyrrole ring and Ar-*C*), 133.0 (Ar-*C*), 136.3 (*C*-5 of pyrrole ring).

5-(4-Chlorophenyl)-2,3-diphenyl-1*H*-pyrrole 2b. Yield 64% (white solid); m.p. 81-83°C; R_f (90:10 hexanes/EtOAc) 0.71; UV (methanol): 250 nm (logε = 4.75), 324 nm (logε = 4.56); IR (KBr): 3417, 3065,

1606, 1503, 856, 815, 759 and 696 cm^{-1}; ^1H NMR (60 MHz; $CDCl_3$): δ 6.5 (s, 1H, C-4H of pyrrole ring), 7.1-7.6 (m, 14H, Ar-H), 8.3 (brs, 1H, -NH) ppm; ^{13}C NMR (100 MHz): 109.1 (*C*-4H of pyrrole ring), 124.1 (*C*-3 of pyrrole ring), 125.0 (Ar-*C*H × 2), 126.2 (Ar-*C*H), 127.3 (Ar-*C*H), 127.6 (Ar-*C*H × 2), 128.5 (Ar-*C*H × 4), 128.9 (Ar-*C*H × 2), 129.2 (Ar-*C*H × 2), 129.9 (Ar-*C*), 130.9 (Ar-*C*), 131.2 (Ar-*C*-Cl), 132.1 (*C*-2 of pyrrole ring), 133.0 (Ar-*C*), 136.2 (*C*-5 of pyrrole ring) . Anal. Calcd for $C_{22}H_{16}ClN$: C, 80.11; H, 4.89; N, 4.27. Found: C, 79.46; H, 4.84; N, 4.17%.

5-(4-Methylphenyl)-2,3-diphenyl-1*H*-pyrrole 2c. Yield 66% (white solid); m.p. 120-22°C; R_f (90:10 hexanes/EtOAc) 0.71; UV (methanol): 237 nm (logε = 4.34), 255 nm (logε = 4.30); IR (KBr): 3426, 3059, 1603, 1483, 1261, 760 and 692 cm^{-1}; ^1H NMR (60 MHz; CCl_4) δ 2.4 (s, 3H, CH_3), 6.60 (s, 1H, C-4H of pyrrole ring), 7.0-7.6 (m, 14H, Ar-H), 8.3 (brs, 1H, –NH); ^{13}C NMR (100 MHz): 21.3 (-*C*H_3), 108.3 (*C*-4H of pyrrole ring), 123.9 (Ar-*C*H × 2), 124.1 (*C*-3 of pyrrole ring), 126.0 (Ar-*C*H), 127.0 (Ar-*C*H), 127.5 (Ar-*C*H × 2), 128.4 (Ar-*C*H × 2), 128.5 (Ar-*C*H × 2), 128.8 (Ar-*C*H × 2), 129.8 (Ar-*C*H × 2), 132.6 (*C*-2 of pyrrole ring), 133.3 (Ar-*C*) , 136.4 (Ar-*C* × 2), 136.6 (C-5 of pyrrole ring and Ar-*C*) ppm. Anal. Calcd for $C_{23}H_{19}N$: C, 89.28; H, 6.19; N, 4.53. Found: C, 89.66; H, 6.13; N, 4.61%.

5-(4-Methoxyphenyl)-2,3-diphenyl-1*H*-pyrrole 2d. Yield 62% (white solid); m.p. 172-74 °C; R_f (90:10 hexanes/EtOAc) 0.55; UV (methanol): 257 nm (logε = 4.60), 308 nm (logε = 4.71); IR (KBr): 3359, 2927, 3060, 1606, 1494, 1237, 1187, 1005, 767 and 692 cm^{-1}; ^1H NMR (60 MHz; CCl_4): δ 3.8 (s, 3H, *OCH*$_3$), 6.5 (s, 1H, C-4H of pyrrole ring), 6.8-7.4 (m, 14H, Ar-H), 8.2 (s, 1H, -NH); ^{13}C NMR (100 MHz) 55.4 (–O*C*H_3), 107.7 (*C*-4H of pyrrole ring), 114.6 (Ar-*C*H × 2), 123.8 (*C*-3 of pyrrole ring), 125.3 (Ar-*C*H × 2), 125.9 (Ar-*C*H), 126.8 (Ar-*C*H), 127.5 (Ar-*C*H × 2), 128.4 (Ar-*C*H × 2), 128.5 (Ar-*C*H × 2), 128.7 (Ar-*C*H × 2), 129.3 (Ar-*C*), 130.7 (Ar-*C*), 132.4 (*C*-2 of pyrrole ring), 133.3 (Ar-*C*), 136.6 (*C*-2 of pyrrole ring), 158.7 (Ar-*C*-O-CH_3) ppm. Anal. Calcd for $C_{23}H_{19}NO$: C, 84.89; H, 5.89; N, 4.30. Found: C, 83.78; H, 5.93; N, 4.37%.

5-(3, 4-Dimethoxylphenyl)-2, 3-diphenyl-1*H*-pyrrole 2e. Yield 59% (white solid); m.p 200°C; R_f (90:10 hexanes/EtOAc) 0.30; UV (methanol: 239 nm (logε = 4.47), 316 nm (logε = 4.60); IR (KBr): 3363, 1594, 1500, 1438, 1250, 1031, 764 and 692 cm^{-1}; ^1H NMR (60 MHz; CCl_4): δ 4.2 (s, 6H, –OCH_3), 6.90 (s, 1H, C-4H pyrrole ring), 7.3-7.6 (m, 13H, Ar-H), 11.9 (brs, 1H, -NH); ^{13}C NMR (100 MHz) 56.01 (–O*C*H_3 × 2), 107.9 (Ar-*C*H × 2), 108.2 (*C*-4H of pyrrole ring), 111.9 (Ar-*C*H), 116.4 (Ar-*C*H), 123.7 (*C*-3 of pyrrole ring and Ar-*C*), 125.9 (Ar-*C*H), 126.9 (Ar-*C*H), 127.5 (Ar-*C*H), 128.4 (Ar-*C*H × 2), 128.5 (Ar-*C*H × 2), 128.7 (Ar-*C*H × 2), 132.5 (*C*-2 of pyrrole ring), 133.3 (Ar-*C*), 136.6 (*C*-5 of pyrrole ring and Ar-*C*), 148.2 (Ar-*C*-O-CH_3), 149.5 (Ar-*C*-O-CH_3) . Anal. Calcd for $C_{24}H_{19}NO_2$: C, 81.10; H, 5.96; N, 3.94. Found: C, 80.93; H, 5.70; N, 3.97%.

5-(Biphen-4-yl)-2,3-diphenyl-1*H*-pyrrole 2f. Yield 46% (yellow solid); m.p. 208-10°C; R_f (90:10 hexanes/EtOAc) 0.61; UV (methanol): 207 nm (logε = 4.57), 240 nm (logε = 4.20), 258 nm (logε = 4.18), 343 nm (logε = 4.45); IR (KBr): 3417, 3066, 1600, 763 and 694 cm^{-1}; ^1H NMR (60 MHz; $CDCl_3$: MeOH D_4, 1:1): δ 6.6 (s, 1H, C-4H pyrrole ring), 7.2-7.5 (m, 19H, Ar-H), 8.4 (brs, 1H, -NH); ^{13}C NMR (100 MHz): 108.9 (*C*-4H of pyrrole ring), 124.0 (*C*-3 of pyrrole ring), 124.1 (Ar-*C*H × 2), 126.0 (Ar-*C*H), 126.8 (Ar-*C*H × 2), 127.2 (Ar-*C*H), 127.4 (Ar-*C*H) 127.5 (Ar-*C*H × 2), 127.6 (Ar-*C*H ×2), 128.3 (Ar-*C*H × 2), 128.4 (Ar-*C*H × 2), 128.7 (Ar-*C*H × 2), 128.8 (Ar-*C*H × 2), 129.4 (Ar-*C*), 131.2 (Ar-*C*), 131.9 (*C*-2 of pyrrole ring), 133.1 (Ar-*C*), 136.3 (*C*-5 of pyrrole ring), 139.2 (Ar-*C*), 140.6 (Ar-*C*). Anal. Calcd for $C_{28}H_{21}N$: C, 90.53; H, 5.70; N, 3.77. Found: C, 89.83; H, 5.87; N, 3.80%.

5-(2-Naphthyl)-2,3-diphenyl-1*H*-pyrrole 2g. Yield 66% (white solid); m.p. 192-194°C; R_f (90:10 hexanes/EtOAc) 0.48; UV (methanol): 215 nm (logε = 4.45), 262 nm (logε = 4.28), 330 nm (logε = 4.31); IR (KBr): 3418, 3053, 1606, 1265, 1068, 856, 759 and 694 cm^{-1}; ^1H NMR (300 MHz; $CDCl_3$): δ 6.3 (s, 1H, C-4H of the pyrrole ring), 6.4-7.9 (m, 17H, Ar-H), 8.6 (brs, 1H, -NH); ^{13}C NMR (100 MHz) 109.2 (*C*-4H of pyrrole ring), 121.0 (Ar-*C*H), 123.0 (Ar-*C*H), 124.0 (*C*-3 of pyrrole ring), 125.5 (Ar-*C*H), 126.0 (Ar-*C*H), 126.6 (Ar-*C*H), 127.0 (Ar-*C*H), 127.5 (Ar-*C*H × 2), 127.7 (Ar-*C*H), 127.8 (Ar-*C*H), 128.3 (Ar-*C*H × 2), 128.4 (Ar-*C*H × 2), 128.7 (Ar-*C*H × 2), 129.5 (Ar-*C*), 129.7 (Ar-*C*), 132.2 (*C*-2 of pyrrole ring and Ar-*C*), 133.0 (Ar-*C*), 133.7 (Ar-*C*), 136.3 (*C*-5 of pyrrole ring) . Anal. Calcd for $C_{26}H_{19}N$: C, 90.40; H, 5.54; N, 4.05. Found: C, 90.31; H, 5.27; N, 4.12%.

4-(9-Anthryl)-1,2-diphenyl-1,4-butanedione 5. Yield = 36% (white solid); mp 166-68 °C; R_f (85:15 hexanes/EtOAc) 0.60; UV (methanol): 301 nm (logε =

4.43), 237 nm (logε = 4.62); IR (KBr): 3052, 1668, 1594, 1447, 1381, 1312, 1168, 1105, 942, 743 and 697 cm^{-1}; ^1H NMR (400 MHz; $CDCl_3$): δ 3.40 (dd, *J* = 19.5, 3.9 Hz, 1H, C-3′H_a), 4.27 (dd, *J* = 19.5, 10.0 Hz, 1H, C-3′H_b), 5.5 (dd, *J* = 10.0, 3.9 Hz, 1H, C-2′*H*), 7.0-7.6 (m, 12H, Ar-H), 7.9 (d, *J* = 8.3 Hz, 2H, Ar-H), 8.0 (d, *J* = 8.3 Hz, 2H, Ar-H), 8.12 (d, *J* = 7.3 Hz, 2H, Ar-H), 8.5 (s, 1H, C-10*H* of anthracene moiety); ^{13}C NMR (100 MHz): 48.5 (*C*-2′H), 50.9 (*C*-3′H_aH_b), 124.4 (Ar-*C*H × 2), 125.4 (Ar-*C*H × 2), 126.7 (Ar-*C*H × 2), 127.1 (Ar-*C*H), 127.4 (Ar-*C*H), 128.2 (Ar-*C*H × 2), 128.5 (Ar-*C*H × 2), 128.6 (Ar-*C*H × 2), 129.0 (Ar-*C*H × 2), 129.2 (Ar-*C*H × 2), 130.9 (Ar-*C* × 2), 132.9 (Ar-*C*H), 135.5 (Ar-*C*), 136.5 (*C*-8a and *C*-9a of anthracene), 138.1 (*C*-4a and *C*-10a of anthracene), 198.9 (*C*-2′), 203.1 (*C*-1′) . Anal. Calcd for $C_{30}H_{22}O_2$: C, 86.93; H, 5.35. Found: C, 86.77; H, 5.33%.

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