

SUPPORTING INFORMATION

Palladium-Catalyzed Carbonylative Annulation of Internal Alkynes: Synthesis of 3,4-

Disubstituted Coumarins

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General. All ¹H and ¹³C NMR spectra were recorded at 400 and 100.5 MHz respectively. Thin-layer chromatography (TLC) was performed using commercially prepared 60-mesh silica gel plates, and visualization was effected with short wavelength UV light (254 nm) or basic KMnO₄ solution [3 g KMnO₄ + 20 g K₂CO₃ + 5 ml NaOH (5 %) + 300 ml of H₂O]. All melting points are uncorrected.

Reagents and starting materials. Pyridine was purified by distillation from CaH₂ and stored over molecular sieves. 2-Iodophenol was purified by recrystallization from hexanes. All other commercially available alkynes and aryl iodides were used without further

purification. 1-Phenyl-3-methyl-1-butyne,¹ 1-phenyl-3,3-dimethyl-1-butyne,² 1-phenyl-3-methoxy-1-propyne³, 1-phenyl-2-butyne-1-one⁴, 4'-hydroxy-3'-iodoacetophenone (**33**),⁵ ethyl 4-hydroxy-3-iodobenzoate (**36**),⁶ 1-iodo-2-naphthol (**43**),⁶ 2,5-diiodo-1,4-hydroxyquinone (**44**),⁷ ethyl 1,6-dihydro-5-iodo-6-oxo-3-pyridinecarboxylate (**47**)^{8,9} and 2-(2-iodophenyl)-2-propanol (**52b**)¹⁰ were prepared following the published literature procedures. 2-Iodo-4-methoxyphenol (**40**) and 2-iodo-5-methoxyphenol (**42**) were obtained from Dr. George A. Kraus.¹¹

The following starting materials were prepared as indicated.

3-Benzyloxy-1-phenyl-1-propyne. A solution of 3-phenyl-2-propyn-1-ol (1.32 g, 10 mmol) in dry THF (5 ml) was added dropwise over 30 min to a suspension of 95 % dry NaH (0.265 g, 10.5 mmol) in dry THF (5 ml) cooled to 0 °C. The mixture was stirred at 0 °C for 5 min, and then allowed to warm to room temperature. A solution of benzyl chloride (1.65 g, 13 mmol) and 25 mg of KI in dry THF (5 ml) was added to the resulting mixture over 10 min. The reaction mixture was stirred at room temperature for 4.5 h, then 20 ml of water was added, and the mixture was extracted with hexanes. The organic extracts were combined, washed with water, dried over anhydrous MgSO₄, concentrated under reduced pressure, and dried *in vacuo*. Column chromatography on silica gel using 8:1 hexanes/EtOAc as eluent afforded 0.61 g (27 %) of the desired compound as a yellow liquid: ¹H NMR (CDCl₃) δ 7.45-7.47 (m, 2H), 7.31-7.40 (m, 8H), 4.68 (s, 2H), 4.40 (s, 2H); ¹³C NMR (CDCl₃) δ 137.7, 132.0, 128.7, 128.6, 128.5, 128.4, 128.1, 122.9, 86.7, 85.2, 71.9, 58.1.

1-Benzyloxy-2-butyne. This ether was prepared using the method above, but employing 2-butyne-1-ol (0.70 g, 10 mmol). Column chromatography on silica gel using 8:1 hexanes/EtOAc as eluent afforded 0.43 g (27 %) of the desired compound as a yellow liquid:

^1H NMR (CDCl_3) δ 7.28-7.36 (m, 6H), 4.57 (d, J = 0.4 Hz, 2H), 4.12 (m, 2H), 1.86 (t, J = 2.0 Hz, 3H); ^{13}C NMR (CDCl_3) δ 137.8, 128.6, 128.2, 128.1, 82.9, 75.2, 71.6, 57.9, 3.8.

Methyl 3-hydroxy-4-iodobenzoate (38). A solution of NaNO_2 (0.45 g, 6.6 mmol) in 2.5 ml of water was added over 15 min to an ice-cold solution of methyl 4-amino-3-hydroxybenzoate (1.0 g, 6.0 mmol) in 3 ml of conc. HCl and 4 g of ice. The resulting solution was stirred at 0 °C for 20 min, and then added over 25 min to a stirred and cooled (0 °C) solution of KI (9.96 g, 16 mmol) in 15 ml of water. The resulting mixture was stirred at room temperature for 18 h. The mixture was extracted with CH_2Cl_2 . The organic extracts were combined, washed with 10% aq NaHCO_3 and water, dried over anhydrous MgSO_4 , and concentrated under reduced pressure. Column chromatography on silica gel using 2:1 hexanes/ EtOAc as eluent afforded 0.61 g (37%) of the desired compound as a red solid, mp 164-166 °C (lit.¹² 145-148 °C): ^1H NMR (d_6 -acetone) δ 9.46 (s, 1H), 7.87 (d, J = 8.0 Hz, 1H), 7.54 (d, J = 2.0 Hz, 1H), 7.25 (dd, J = 2.0, 8.0 Hz, 1H), 3.85 (s, 3H); ^{13}C NMR (d_6 -acetone) δ 166.8, 157.7, 140.5, 132.7, 122.8, 116.0, 91.0, 52.6; MS m/z (rel intensity) 278 (100, M^+), 247 (77), 218 (16); HRMS calcd for $\text{C}_8\text{H}_7\text{IO}_3$: 277.9440, found: 277.9445.

General procedure for the palladium-catalyzed synthesis of coumarins. The 2-iodophenol (0.5 mmol), the alkyne (2.5 mmol), pyridine (79 mg, 1.0 mmol), $n\text{-Bu}_4\text{NCl}$ (139 mg, 0.5 mmol), $\text{Pd}(\text{OAc})_2$ (5.6 mg, 5 mol %, 0.025 mmol), and DMF (5 ml) were placed in a 4 dram vial. The vial was purged with CO for 2 min, and then connected to a balloon of CO . The reaction mixture was stirred at 120 °C for 24 h, then allowed to cool to room temperature, diluted with EtOAc , washed with water, dried over anhydrous MgSO_4 , and

concentrated under reduced pressure. The product was isolated by flash chromatography on silica gel.

Full characterization and spectral data for compounds **7-11**, **14**, **19**, **20**, **23-25**, **37**, **39**, **41** and **48** can be found in ref. 23.

3,4-Diethyl-2*H*-1-benzopyran-2-one (6). Yellow viscous oil; ^1H NMR (CDCl_3) δ 7.61 (dd, $J = 1.6, 8.0$ Hz, 1H), 7.44 (ddd, $J = 1.2, 7.6, 8.4$ Hz, 1H), 7.26-7.32 (m, 2H), 2.85 (q, $J = 7.6$ Hz, 2H), 2.67 (q, $J = 7.6$ Hz, 2H), 1.28 (t, $J = 7.6$ Hz, 3H), 1.19 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (CDCl_3) δ 161.9, 152.8, 151.1, 130.5, 127.5, 124.5, 124.2, 119.6, 117.1, 21.7, 21.0, 14.0, 13.6; IR (CHCl_3 , cm^{-1}) 3063, 2973, 2938, 2878, 1711, 1606; MS m/z (rel intensity) 202 (100, M^+), 187 (37), 159 (35); HRMS calcd for $\text{C}_{13}\text{H}_{14}\text{O}_2$: 202.0994, found: 202.0997.

4-Isopropyl-3-phenyl-2*H*-1-benzopyran-2-one (12). White solid, mp 205-206 $^\circ\text{C}$; ^1H NMR (CDCl_3) δ 7.97 (dd, $J = 1.2, 8.0$ Hz, 1H), 7.39-7.54 (m, 5H), 7.25-7.31 (m, 3H), 3.24 (septet, $J = 7.2$ Hz, 1H), 1.38 (d, $J = 7.2$ Hz, 6H); ^{13}C NMR (CDCl_3) δ 161.6, 156.6, 153.6, 135.5, 131.0, 129.6, 128.8, 128.3, 127.2, 126.8, 123.8, 118.7, 117.8, 31.7, 21.5; IR (CHCl_3 , cm^{-1}) 2983, 1714, 1699; MS m/z (rel intensity) 264 (100, M^+), 221 (50). Anal. Calcd for $\text{C}_{18}\text{H}_{16}\text{O}_2$: C, 81.79; H, 6.10. Found: C, 81.68; H, 6.16.

3-Isopropyl-4-phenyl-2*H*-1-benzopyran-2-one (13). White solid, mp 113-115 $^\circ\text{C}$; ^1H NMR (CDCl_3) δ 7.49-7.55 (m, 3H), 7.43 (ddd, $J = 1.2, 8.0, 8.4$ Hz, 1H), 7.33 (d, $J = 8.0$ Hz, 1H), 7.21-7.23 (m, 2H), 7.09 (ddd, $J = 0.8, 7.6, 8.4$ Hz, 1H), 6.87 (dd, $J = 1.2, 8.0$ Hz, 1H), 2.74 (septet, $J = 7.1$ Hz, 1H), 1.26 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (CDCl_3) δ 160.1, 152.8, 150.2, 135.6, 131.7, 130.6, 129.1, 128.6, 128.2, 127.7, 123.9, 121.2, 116.4, 30.6, 20.1; IR (neat, cm^{-1}) 2956, 1716; MS m/z (rel intensity) 264 (53, M^+), 263 (100), 249 (21); HRMS calcd for $\text{C}_{18}\text{H}_{16}\text{O}_2$: 264.1150, found: 264.1156.

3-Cyclohexyl-4-methyl-2H-1-benzopyran-2-one (15). White solid, mp 126-128 °C; ^1H NMR (CDCl_3) δ 7.63 (dd, $J = 1.2, 8.8$ Hz, 1H), 7.43 (ddd, $J = 1.2, 7.2, 8.0$ Hz, 1H), 7.24-7.29 (m, 2H), 2.89-2.95 (m, 1H), 2.46 (s, 3H), 2.16-2.24 (m, 2H), 1.84-1.86 (m, 2H), 1.70-1.72 (m, 1H), 1.54-1.58 (m, 2H), 1.32-1.36 (m, 3H); ^{13}C NMR (CDCl_3) δ 160.2, 152.5, 145.5, 130.7, 130.4, 124.8, 124.0, 121.1, 116.7, 40.2, 29.4, 27.2, 25.9, 15.0; IR (CHCl_3 , cm^{-1}) 2919, 2864, 1709, 1602; MS m/z (rel intensity) 242 (100, M^+), 227 (97), 227 (99), 225 (66), 186 (47), 173 (49); HRMS calcd for $\text{C}_{16}\text{H}_{18}\text{O}_2$: 242.1307, found: 242.1312. Anal. Calcd for $\text{C}_{16}\text{H}_{18}\text{O}_2$: C, 79.31; H, 7.49. Found: C, 79.52; H, 7.78.

4-Cyclohexyl-3-methyl-2H-1-benzopyran-2-one (16). Colorless oil; ^1H NMR (CDCl_3) δ 7.42 (dd, $J = 7.6, 8.0$ Hz, 1H), 7.30 (dd, $J = 1.2, 8.0$ Hz, 1H), 7.20-7.29 (m, 2H), 3.13-3.19 (m, 1H), 2.28 (s, 3H), 1.99-2.10 (m, 2H), 1.81-1.94 (m, 5H), 1.40-1.47 (m, 3H); only a small amount has been isolated, so no other spectral data were obtained; MS m/z (rel intensity) 242 (28, M^+), 84 (100); HRMS calcd for $\text{C}_{16}\text{H}_{18}\text{O}_2$: 242.1307, found: 242.1312.

4-Methoxymethyl-3-phenyl-2H-1-benzopyran-2-one (17) and 3-methoxymethyl-4-phenyl-2H-1-benzopyran-2-one (18). These compounds were obtained after column chromatography as a 3:1 inseparable mixture. Recrystallization from hexanes/ethyl acetate afforded pure **4-methoxymethyl-3-phenyl-2H-1-benzopyran-2-one (17)** (major isomer): off-white solid, mp 151-154 °C; ^1H NMR (CDCl_3) δ 7.86 (dd, $J = 1.2, 8.0$ Hz, 1H), 7.53 (ddd, $J = 0.4, 7.6, 8.0$ Hz, 1H), 7.43-7.49 (m, 3H), 7.31-7.38 (m, 4H), 4.42 (s, 2H), 3.34 (s, 3H); ^{13}C NMR (CDCl_3) δ 161.3, 153.3, 145.4, 133.5, 131.6, 130.3, 129.5, 128.9, 128.6, 126.4, 124.6, 119.4, 117.0, 68.7, 58.8; IR (CHCl_3 , cm^{-1}) 3063, 2908, 1721, 1606; MS m/z (rel intensity) 266 (100, M^+), 251 (40); HRMS calcd for $\text{C}_{17}\text{H}_{14}\text{O}_3$: 266.0943, found: 266.0948. Spectral data for **3-methoxymethyl-4-phenyl-2H-1-benzopyran-2-one (18)** (minor isomer):

^1H NMR (CDCl_3) δ 7.31-7.55 (m, 7H), 7.15 (ddd, $J = 1.2, 6.8, 8.0$ Hz, 1H), 7.10 (dd, $J = 1.2, 8.0$ Hz, 1H), 4.13 (s, 2H), 3.31 (s, 3H); ^{13}C NMR (CDCl_3) δ 161.7, 154.9, 153.5, 133.7, 132.0, 129.2, 128.8, 128.5, 128.2, 124.3, 122.6, 120.4, 67.3, 59.0 (one sp^2 carbon missing due to overlap).

4-Benzyloxymethyl-3-methyl-2H-1-benzopyran-2-one (21). Colorless oil; ^1H NMR (CDCl_3) δ 7.74 (dd, $J = 1.6, 8.0$ Hz, 1H), 7.46 (ddd, $J = 1.2, 8.0, 8.4$ Hz, 1H), 7.24-7.39 (m, 7H), 4.71 (s, 2H), 4.63 (s, 2H), 2.22 (s, 3H); ^{13}C NMR (CDCl_3) δ 162.3, 152.5, 144.1, 137.4, 130.8, 128.8, 128.4, 128.2, 125.4, 125.3, 124.4, 119.4, 116.9, 73.2, 64.7, 13.4; IR (neat, cm^{-1}) 2929, 2848, 1726, 904; MS m/z (rel intensity) 280 (16, M^+), 174 (100), 146 (51), 115 (24), 91 (45); HRMS calcd for $\text{C}_{18}\text{H}_{16}\text{O}_3$: 280.1099, found: 280.1104.

3-Benzyloxymethyl-4-methyl-2H-1-benzopyran-2-one (22). Colorless oil; ^1H NMR (CDCl_3) δ 7.65 (dd, $J = 1.6, 8.0$ Hz, 1H), 7.52 (ddd, $J = 1.6, 7.6, 8.4$ Hz, 1H), 7.24-7.39 (m, 7H), 4.65 (s, 2H), 4.62 (s, 2H), 2.50 (s, 3H); ^{13}C NMR (CDCl_3) δ 161.7, 153.0, 151.8, 138.2, 131.9, 128.6, 128.2, 128.0, 125.1, 124.4, 122.5, 120.6, 117.1, 73.2, 64.2, 15.3; IR (neat, cm^{-1}) 3063, 2926, 2858, 1717, 1700, 1606, 1085; MS m/z (rel intensity) 281 (100), 280 (42, M^+), 174 (31), 91 (23); HRMS calcd for $\text{C}_{18}\text{H}_{16}\text{O}_3$: 280.1099, found: 280.1104.

2-Benzoylmethyl-2-methyl-1-benzofuran-3(2H)-one (26). Yellow oil; ^1H NMR (CDCl_3) δ 7.86-7.88 (m, 2H), 7.76 (dd, $J = 0.8, 8.4$ Hz, 1H), 7.57-7.62 (m, 2H), 7.39-7.43 (m, 2H), 7.09-7.13 (m, 1H), 7.04 (d, $J = 8.4$ Hz, 1H), 3.83 (d, $J = 18.0$ Hz, 1H), 3.62 (d, $J = 17.6$ Hz, 1H), 1.53 (s, 3H); ^{13}C NMR (CDCl_3) δ 203.5, 194.9, 170.9, 137.6, 136.3, 133.7, 128.8, 128.4, 124.6, 122.0, 121.2, 86.5, 45.8, 23.0; IR (CHCl_3 , cm^{-1}) 3019, 1718, 1695, 1616, 1216; MS m/z (rel intensity) 266 (69, M^+), 161 (23), 105 (100); HRMS calcd for $\text{C}_{17}\text{H}_{14}\text{O}_3$: 266.0943, found: 266.0947.

Ethyl 2-oxo-4-phenyl-2H-1-benzopyran-3-carboxylate (27). Yellow oil; compound **27** was detected by GCMS (however, it was not isolated in a pure form). Every fraction obtained by column chromatography on silica gel using 4:1 ethyl acetate/hexanes as the eluent contained an unidentified by-product: ^1H NMR (CDCl_3) δ 7.59 (ddd, $J = 2.0, 6.6, 8.6$ Hz, 1H), 7.16-7.51 (m, 8H), 4.08 (q, $J = 7.2$ Hz, 2H), 0.97 (t, $J = 7.2$ Hz, 3H); GCMS m/z (rel intensity) 294 (35, M^+), 265 (15), 250 (100), 221 (30), 163 (35).

Ethyl 2-oxo-3-phenyl-2H-1-benzopyran-4-carboxylate (28). Yellow solid, mp 131-134 °C; ^1H NMR (CDCl_3) δ 7.58 (ddd, $J = 1.2, 7.2, 8.0$ Hz, 1H), 7.52 (dd, $J = 1.2, 7.8$ Hz, 1H), 7.39-7.45 (m, 5H), 7.33 (ddd, $J = 0.8, 7.2, 8.0$ Hz, 1H), 4.16 (q, $J = 7.2$ Hz, 2H), 0.98 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (CDCl_3) δ 165.2, 160.7, 153.6, 143.4, 133.2, 132.3, 129.5, 129.3, 128.5, 126.7, 126.2, 125.0, 117.3, 116.7, 62.4, 13.8; IR (CHCl_3 , cm^{-1}) 1733, 1607; MS m/z (rel intensity) 294 (100, M^+), 221 (100); HRMS calcd for $\text{C}_{18}\text{H}_{14}\text{O}_4$: 294.0892, found: 294.0897.

4-Methyl-2H-1-benzopyran-2-one (30). White solid; ^1H NMR (CDCl_3) δ 7.62 (dd, $J = 1.2, 8.0$ Hz, 1H), 7.54 (ddd, $J = 1.2, 7.6, 8.8$ Hz, 1H), 7.29-7.36 (m, 2H), 6.31 (d, $J = 1.2$ Hz, 1H), 2.45 (d, $J = 1.2$ Hz, 3H); ^{13}C NMR (CDCl_3) δ 161.0, 153.7, 152.6, 132.0, 132.0, 124.8, 124.4, 120.2, 117.3, 115.3, 18.9. The spectral data are identical to those reported in the literature.¹⁴

Ethyl 4-methyl-2-oxo-2H-1-benzopyran-3-carboxylate (31) was identified by comparison of its ^1H NMR spectral data with previously reported data.¹⁵ Only a small amount of **31** was isolated, therefore no other spectral data have been obtained.

Ethyl 3-methyl-2-oxo-2H-1-benzopyran-4-carboxylate (32). White solid, mp 89-90 °C; ^1H NMR (CDCl_3) δ 7.69 (dd, $J = 1.2, 8.0$ Hz, 1H), 7.59 (ddd, $J = 1.6, 7.6, 8.4$ Hz, 1H), 7.32-

7.37 (m, 2H), 4.44 (q, $J = 7.2$ Hz, 1H), 2.49 (s, 3H), 1.41 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (CDCl_3) δ 165.1, 158.0, 153.2, 150.2, 133.0, 125.5, 124.9, 121.7, 119.4, 117.4, 62.4, 16.3, 14.4; IR (neat, cm^{-1}) 2914, 1724; MS m/z (rel intensity) 232 (61, M^+), 186 (100), 160 (34); HRMS calcd for $\text{C}_{13}\text{H}_{12}\text{O}_4$: 232.0736, found: 232.0738.

6-Acetyl-3,4-dipropyl-2H-1-benzopyran-2-one (34). White solid, mp 114-116 °C; ^1H NMR (CDCl_3) δ 8.25 (d, $J = 2.0$ Hz, 1H), 8.04 (dd, $J = 2.0, 8.4$ Hz, 1H), 7.36 (d, $J = 8.8$ Hz, 1H), 2.83-2.87 (m, 2H), 2.61-2.66 (m, 5H), 1.57-1.71 (m, 4H), 1.13 (t, $J = 7.4$ Hz, 3H), 1.04 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (CDCl_3) δ 196.6, 161.2, 155.8, 150.0, 133.3, 130.6, 127.7, 125.5, 120.0, 117.4, 30.6, 30.0, 26.8, 23.1, 22.5, 14.7, 14.5; IR (neat, cm^{-1}) 2963, 2933, 2873, 1716, 1676, 1606. Anal. Calcd for $\text{C}_{17}\text{H}_{20}\text{O}_3$: C, 74.97; H, 7.40. Found: C, 74.95; H, 7.40.

6-Acetyl-3,4-diphenyl-2H-1-benzopyran-2-one (35). White solid, mp 155-158 °C; ^1H NMR (CDCl_3) δ 8.13 (dd, $J = 2.0, 8.6$ Hz, 1H), 7.86 (d, $J = 2.0$ Hz, 1H), 7.49 (d, $J = 8.4$ Hz, 1H), 7.32-7.36 (m, 3H), 7.19-7.21 (m, 3H), 7.12-7.15 (m, 4H), 2.49 (s, 3H); ^{13}C NMR (CDCl_3) δ 196.3, 160.7, 156.2, 151.5, 133.9, 133.6, 133.4, 131.4, 130.6, 129.5, 129.0, 128.8, 128.7, 128.1, 128.0, 127.9, 120.6, 117.4, 26.6; IR (neat, cm^{-1}) 3063, 1726, 1681, 1601; MS m/z (rel intensity) 340 (100, M^+), 325 (64), 297 (93), 239 (59); HRMS calcd for $\text{C}_{23}\text{H}_{16}\text{O}_3$: 340.1099, found: 340.1107.

3,4,8,9-Tetrapropylbenzo[1,2-*b*:4,5-*b'*]dipyran-2,7-dione (45). Yellow solid, mp 188-190 °C; ^1H NMR (CDCl_3) δ 7.49 (s, 1H), 2.75-2.80 (m, 2H), 2.62-2.66 (m, 2H), 1.58-1.68 (m, 4H), 1.12 (t, $J = 7.4$ Hz, 3H), 1.04 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (CDCl_3) δ 161.4, 148.9, 148.6, 128.7, 121.6, 111.9, 30.9, 30.1, 22.9, 22.5, 14.7, 14.5; IR (CHCl_3 , cm^{-1}) 2965, 2874, 1699, 1601; MS m/z (rel intensity) 382 (100, M^+), 353 (41), 339 (52). Anal. Calcd for $\text{C}_{24}\text{H}_{30}\text{O}_4$: C, 75.36; H, 7.91. Found: C, 75.48; H, 7.93.

Carbonylative annulation of internal alkynes with *o*-iodobenzyl alcohols. The *o*-iodobenzyl alcohol (0.5 mmol), the alkyne (2.5 mmol), pyridine (79 mg, 1.0 mmol), *n*-Bu₄NCl (139 mg, 0.5 mmol), Pd(OAc)₂ (5.6 mg, 5 mol %, 0.025 mmol), and DMF (5 ml) were placed in a 4 dram vial. The vial was purged with CO for 2 min, then connected to a balloon of CO. Upon completion of the reaction (for the reaction temperatures and times see Table 3), the reaction mixture was cooled to room temperature, diluted with EtOAc, washed with water, dried over anhydrous MgSO₄, and concentrated under reduced pressure. The products were isolated by flash chromatography on silica gel.

1(3*H*)-Isobenzofuranone (53a). White solid, the spectral properties are identical to those reported in the literature.¹⁶

3,3-Dimethyl-1(3*H*)-isobenzofuranone (53b). White solid, spectral properties are identical to those reported in the literature.¹⁷

4,5-Diphenyl-1,3-dihydrobenzo[*c*]oxepin-3-one (54b). White crystals; ¹H NMR (CDCl₃) δ 7.44-7.50 (m, 3H), 7.39 (ddd, *J* = 1.2, 7.2, 8.0 Hz, 1H), 7.31 (ddd, *J* = 1.2, 7.6, 8.4 Hz, 1H), 7.12-7.19 (m, 6H), 6.96-6.98 (m, 3H), 5.51 (br s, 1H), 5.05 (br s, 1H); ¹³C NMR (CDCl₃) δ 169.3, 145.3, 140.4, 140.2, 136.7, 136.1, 133.0, 131.3, 131.2, 130.7, 129.5, 129.4, 128.4, 128.1, 128.0, 127.9, 127.7, 68.6; IR (CHCl₃, cm⁻¹) 3057, 1718; MS *m/z* (rel intensity) 312 (29, M⁺), 235 (42), 233 (100), 260 (51); HRMS Calcd for C₂₂H₁₆O₂: 312.1150. Found: 312.1155.

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