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Iodine-Catalyzed Synthesis of 1,2-Diaryldiketones by Oxidative Cleavage of 1,3-Diaryldiketones with DMSO

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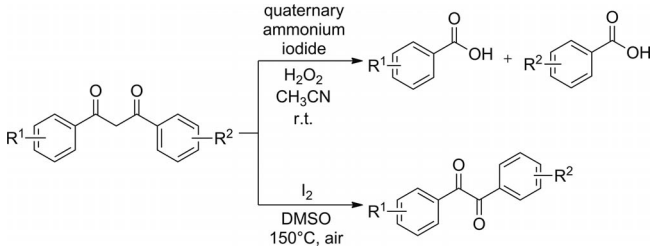
Keywords: Iodine / Cleavage reactions / Ketones / Oxidation

A metal-free, efficient, practical, and convenient process based on an iodine-catalyzed oxidative cleavage reaction has been developed to form 1,2-diaryldiketons in high yields from 1,3-diaryldiketones. The reaction is performed in DMSO and in air, and a mechanism was proposed according to the reaction evidence.

Introduction

1,2-Diketone derivatives are valuable compounds, which are versatile building blocks capable of undergoing a variety of chemical transformations,[1] especially for the synthesis of biologically active heterocyclic compounds.[2] Several synthesis methods have been reported to obtain this structure, including the oxidation of benzyl derivatives or 1,2-diols,[3] oxidation of alkynes or alkenes[4] and epoxides,[5] and the Wacker reaction.[6] Also, a few examples synthesizing 1,2-diketones from 1,3-diketones have been developed that proceed in moderate yields.[7] However, these methods have several drawbacks, such as low chemoselectivity, high toxicity, expensive metal catalysts and raw materials, and the requirement of special equipment. Recently, we developed the conversion of 1,3-diketones into carboxylic acids by oxidative cleavage catalyzed by quaternary ammonium iodides.[8] Herein, we report a method for the preparation of 1,2-diaryldiketones by oxidative cleavage of 1,3-diaryldiketones with DMSO catalyzed by iodine (Scheme 1).



Scheme 1. Oxidative cleavage of 1,3-diketones.

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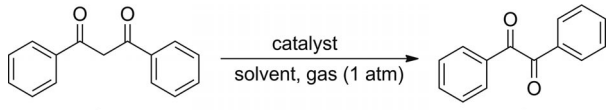
Results and Discussion

Initially dibenzoylmethane (**1a**) was treated with iodine (1.0 equiv.) at 150 °C in DMSO under an oxygen atmosphere. The corresponding product, benzil (**2a**), was obtained in 76% yield (Table 1, Entry 1).

Table 1. Optimization of reaction conditions.[a]



1a **2a**

Entry	Catalyst (mol-%)	Solvent	*T* [°C]	Yield [%][d]
1	I_2 (100)[b]	DMSO	150	76
2	I_2 (100)[c]	DMSO	150	96
3	I_2 (100)	DMSO	150	97
4	I_2 (100)	DMSO	120	10
5	I_2 (100)	DMF	150	<10
6	I_2 (100)	NMP[e]	150	<5
7	I_2 (100)	mesitylene	150	trace
8	I_2 (40)	DMSO	150	97
9	Br_2 (40)	DMSO	150	68
10	I_2 (10)	DMSO	150	97
11	I_2 (5)	DMSO	150	97
12	I_2 (2.5)	DMSO	150	97
13	I_2 (1.0)	DMSO	150	90

[a] Reaction conditions: **1a** (0.5 mmol) and catalyst in solvent (1 mL) for 27 h in air. [b] Under an O_2 atmosphere. [c] Under an argon atmosphere. [d] Yield after chromatography. [e] *N*-Methyl-2-pyrrolidone.

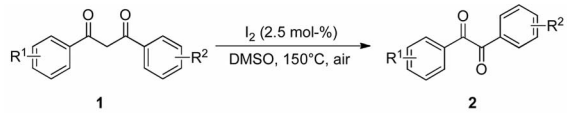
Interestingly, whether the reaction was carried out in air or under an argon atmosphere, **1a** was almost completely converted into benzil after 27 h (Table 1, Entries 2 & 3). Only a small amount of benzil was observed when the temperature was decrease to 120 °C because of the formation of 2,2-dihydroxy-1,3-diphenylpropane-1,3-dione (Table 1,

Entry 4). Moreover, benzil was not formed in great quantities when the reaction was performed in air in other solvents such as DMF, NMP (*N*-methyl-2-pyrrolidone), and mesitylene (Table 1, Entries 5–7). It was believed that DMSO was acting as the oxidant rather than oxygen for the oxidative cleavage of **1a** as a result of the oxidation potential of DMSO.[9,4d–4j] A decrease in the amount of the catalyst to 2.5 mol-% did not effect the formation of benzil (Table 1, Entry 12). However, when the amount of iodine was reduced to 1.0 mol-% the yield of the product dropped to 90% (Table 1, Entry 13). When the catalyst was changed to bromine, the reaction became inefficient (Table 1, Entry 9). This result was in accord with our former research in oxidative cleavage reactions catalyzed by quaternary ammonium salts.[8]

With the optimized conditions in hand, the oxidative cleavage of various substituted 1,3-diaryldiketons was catalyzed by iodine (2.5 mol-%) in DMSO at 150 °C in air. *meta*/*para*-Methyl- and methoxy-substituted 1,3-diaryldiketones were transformed into the corresponding benzil derivatives in excellent yields (Table 2, Entries 2–4). However, when the methoxy group was at the *ortho* position of the aromatic ring of the substrate the reaction only afforded the product in moderate yields presumably due to the steric hindrance near the carbonyl group (Table 2, Entries 5 & 8). The introduction of a chlorine atom on the aromatic ring induced a decrease in the yield, whereas a fluorine atom on the ring promoted the reaction well (Table 2, Entries 6 & 7). Furthermore, 1,3-diaryldiketones bearing both electron-donating and electron-withdrawing groups on each aromatic ring could be converted into the corresponding 1,2-diaryldiketones in good yields under the conditions (Table 2, Entries 9–12). However, electron-withdrawing groups on both aromatics rings led to a reduced yield (Table 2, Entry 13).

On the basis of some related literature,[7f,9] the mechanism of this oxidative cleavage was studied. It was found that this reaction could be divided into two stages connected by key intermediate 2,2-dihydroxy-1,3-diphenylpropane-1,3-dione (**3**). Compound **1a** was treated with iodine (100 mol-%) in DMSO at 120 °C in air, and intermediate **3** was obtained in 85% yield (Table 1, Entry 4). The structure of **3** was determined by X-ray analysis (Figure 1). In the second stage, the benzil product was formed completely by using intermediate **3** as the substrate in the presence of a catalytic amount of hydroiodic acid in DMSO at 150 °C in air (Scheme 2). Thus, the mechanism of 1,2-diaryldiketone formation from 1,3-diaryldiketone was proposed (Scheme 3). Substrate **1a** reacted with iodine to form the 2-iodo derivative in DMSO, which was oxidized by DMSO to obtain the triketone via the sulfonium salt.[9a,9b] At the same time, hydrogen iodide was generated in the system. The triketone was easily converted into key intermediate **3** by the effects of a small amount of water.[10] Intermediate **6** was formed by a reverse benzilic acid rearrangement in the presence of hydrogen iodide.[11,7e] On the other hand, hydrogen iodide could be oxidized by DMSO to regenerate iodine, which was used in the first stage.[9a,9b] Then, final

Table 2. Oxidative cleavage of 1,3-diaryldiketones to 1,2-diaryldiketones.[a]



Entry	Substrate	Product	Yield [%][c]
1	**1a**	**2a**	97
2	**1b**	**2b**	85
3	**1c**	**2c**	92
4	**1d**	**2d**	95
5	**1e**	**2e**	73
6	**1f**	**2f**	80
7	**1g**	**2g**	94
8	**1h**	**2h**	72
9	**1i**	**2i**	90
10	**1j**	**2j**	84
11	**1k**	**2k**	86
12	**1l**	**2l**	93
13	**1m**	**2m**	57

[a] Reaction conditions: **1a** (0.5 mmol) and I_2 (2.5 mol-%) in DMSO (1 mL) at 150 °C for 27 h in air. [b] Yield after chromatography.

benzil product **2a** can be generated when intermediate **6** undergoes oxidative decarboxylation in the presence of iodine.[12]

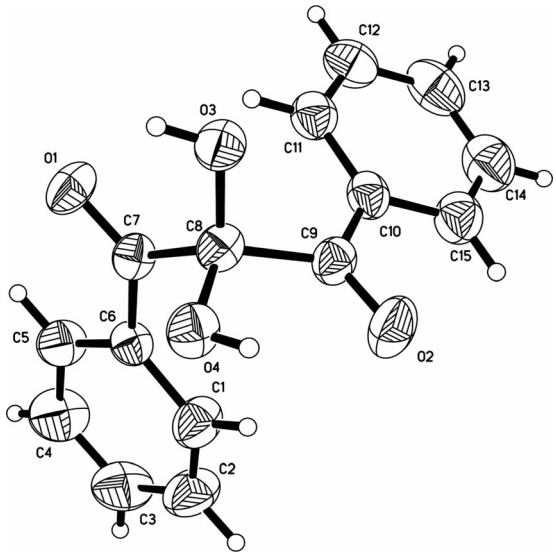
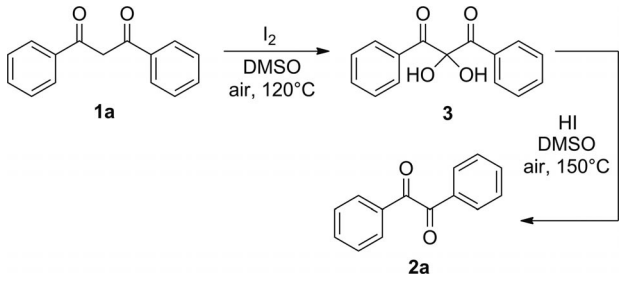


Figure 1. The structure of intermediate **3**.



Scheme 2. Study of the reaction mechanism.

Conclusions

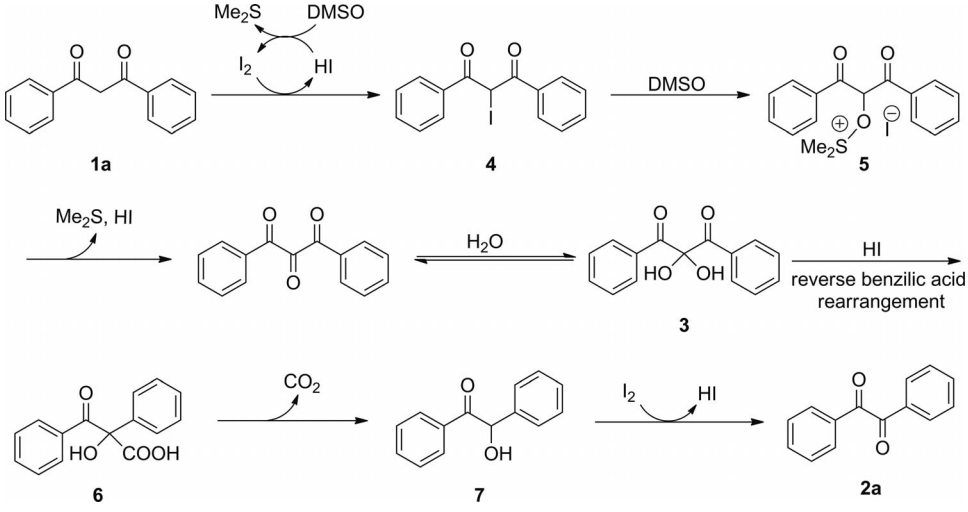
In conclusion, a practical and convenient oxidative cleavage for the conversion of 1,3-diaryldiketones into 1,2-diaryldiketones was developed; the process is catalyzed by iodine with DMSO as the terminal oxidant and solvent. All the substrates and reagents are cheap and available in comparison to alkenes, alkynes, and noble metals. A mechanism was proposed for which two stages, oxidation and cleavage, existed in the system.

Experimental Section

General Methods: ^{1}H NMR and ^{13}C NMR spectra were obtained with a Bruker Avance 600 spectrometer in $CDCl_3$ with TMS as an internal standard. Infrared spectra were recorded with a Bruker Tensor 27 FTIR spectrophotometer by using KBr pellets. GC–MS was performed with a Finnigan Trace DSQ chromatograph. High-resolution mass spectra were obtained with a TOF-MS instrument. Elemental analyses were recorded with a Series II 2400. A single crystal was detected with a Bruker Smart 2 X-ray diffraction instrument. 1,3-Diaryldiketones were synthesized according to the literature.[13] All other reagents were obtained from commercial sources.

Typical Procedure for the Oxidative Cleavage of 1,3-Diaryldiketone to 1,2-Diaryldiketones: A mixture of the β-dicarbonyl compound (0.5 mmol), iodine (2.5 mol-%), and DMSO (1 mL) in a Schlenk tube was stirred in air at 150 °C. The process of the reaction was monitored by TLC [ethyl acetate (EA)/petroleum ether (PE) = 1:20]. When the reaction was complete, the reaction mixture was treated with saturated $Na_2S_2O_3$ solution (8 mL) and ethyl acetate (8 mL). The organic and aqueous layers were separated, and the aqueous layer was extracted with ethyl acetate (5 × 3 mL). The combined organic extracts were dried with anhydrous sodium sulfate, filtered, and concentrated under vacuum. The residue was purified by flash column chromatography (PE/EA) to afford the corresponding 1,2-diketone.

Benzil (2a):[4b] Yellow solid, m.p. 93–94 °C. IR (KBr): $\tilde{\nu}$ = 3063, 2963, 1661, 1593, 1449, 1324, 1262, 1211, 1097, 796, 719, 681 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.52 (t, J = 7.2 Hz, 4 H), 7.65



Scheme 3. Possible reaction pathway.

(t, J = 7.1 Hz, 2 H), 7.98 (d, J = 7.3 Hz, 4 H) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 129.0, 129.9, 132.9, 134.8, 194.5 ppm. MS (EI): m/z = 210 $[M]^+$.

1-(3-Methoxyphenyl)-2-phenylethane-1,2-dione (2b):[4g] Yellow solid, m.p. 90–91 °C. IR (KBr): $\tilde{\nu}$ = 3073, 3008, 1662, 1593, 1484, 1453, 1337, 1299, 1204, 1175, 932, 833, 778, 721, 689 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 3.88 (s, 3 H), 7.22 (d, J = 7.9 Hz, 1 H), 7.42 (t, J = 7.4 Hz, 1 H), 7.56–7.49 (m, 4 H), 7.67 (t, J = 6.7 Hz, 1 H), 7.98 (d, J = 6.8 Hz, 2 H) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 55.5, 112.8, 121.9, 123.2, 129.0, 129.9, 130.1, 133.0, 134.3, 134.9, 160.1, 194.4, 194.5 ppm. MS (EI): m/z = 240 $[M]^+$.

1-(4-Methoxyphenyl)-2-phenylethane-1,2-dione (2c):[4b] Yellow solid, m.p. 61–62 °C. IR (KBr): $\tilde{\nu}$ = 2981, 2943, 1674, 1600, 1572, 1509, 1454, 1300, 1218, 106, 1020, 847, 762, 720 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 3.88 (s, 3 H), 6.97 (d, J = 8.8 Hz, 2 H), 7.51 (t, J = 7.7 Hz, 2 H), 7.65 (t, J = 7.4 Hz, 1 H), 7.98–7.94 (m, 4 H) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 55.6, 114.4, 126.1, 128.9, 129.9, 132.4, 133.2, 134.7, 165.0, 193.2, 194.9 ppm. MS (EI): m/z = 240 $[M]^+$.

1-Phenyl-2-(*p*-tolyl)ethane-1,2-dione (2d):[7e] Yellow oil. IR (KBr): $\tilde{\nu}$ = 3062, 2924, 1679, 1601, 1450, 1322, 1213, 1120, 935, 880, 831, 784, 747 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.44 (s, 3 H), 7.31 (d, J = 8.0 Hz, 2 H), 7.51 (t, J = 7.7 Hz, 2 H), 7.65 (t, J = 7.4 Hz, 1 H), 7.87 (d, J = 8.1 Hz, 2 H), 7.97 (d, J = 7.1 Hz, 2 H) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 21.9, 129.0, 129.7, 129.9, 130.0, 130.6, 133.1, 134.8, 146.2, 194.3, 194.8 ppm. MS (EI): m/z = 224 $[M]^+$.

1-(2-Methoxyphenyl)-2-phenylethane-1,2-dione (2e):[4d] Yellow solid, m.p. 70–71 °C. IR (KBr): $\tilde{\nu}$ = 2942, 1680, 1599, 1487, 1313, 1273, 1208, 1018, 878, 756, 722 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 3.56 (s, 3 H), 6.93 (d, J = 8.3 Hz, 1 H), 7.14 (t, J = 7.4 Hz, 1 H), 7.50 (t, J = 7.8 Hz, 2 H), 7.62–7.58 (m, 2 H), 7.92 (d, J = 7.2 Hz, 2 H), 8.03 (dd, J = 1.6, 7.7 Hz, 1 H) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 54.6, 111.3, 120.5, 122.8, 127.7, 128.3, 129.5, 131.9, 132.7, 135.5, 159.4, 192.5, 193.6 ppm. MS (EI): m/z = 240 $[M]^+$.

1-(4-Chlorophenyl)-2-phenylethane-1,2-dione (2f):[4e] Yellow solid, m.p. 75–76 °C. IR (KBr): $\tilde{\nu}$ = 2924, 1688, 1587, 1450, 1322, 1211, 1095, 876, 835, 798, 757, 682 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.48–7.53 (m, 4 H), 7.67 (t, J = 7.0 Hz, 1 H), 7.92 (d, J = 8.4 Hz, 2 H), 7.96 (d, J = 7.9 Hz, 2 H) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 129.1, 129.4, 129.9, 131.2, 131.4, 132.8, 135.0, 141.6, 193.0, 193.9 ppm. MS (EI): m/z = 244 $[M]^+$.

1-(3-Fluorophenyl)-2-phenylethane-1,2-dione (2g):[4c] Yellow solid, m.p. 65–66 °C. IR (KBr): $\tilde{\nu}$ = 3065, 293, 1670, 1588, 1482, 1446, 1323, 1241, 1152, 837, 781, 750, 717 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.37 (t, J = 8.2 Hz, 1 H), 7.48–7.54 (m, 3 H), 7.66–7.71 (m, 2 H), 7.74 (d, J = 7.6 Hz, 1 H), 7.97 (d, J = 8.0 Hz, 2 H) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 116.0, 116.2, 121.9, 122.1, 125.9, 126.0, 129.1, 130.7, 130.8, 132.7, 135.1, 162.1, 163.7, 193.0, 193.1, 193.7 ppm. MS (EI): m/z = 228 $[M]^+$.

1-(2-Methoxyphenyl)-2-(*p*-tolyl)ethane-1,2-dione (2h):[4f] Yellow solid, m.p. 98–100 °C. IR (KBr): $\tilde{\nu}$ = 2946, 1668, 1684, 1462, 1273, 1206, 1112, 1018, 881, 831, 754, 611 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.43 (s, 3 H), 3.57 (s, 3 H), 6.93 (d, J = 8.4 Hz, 1 H), 7.13 (t, J = 7.3 Hz, 1 H), 7.29 (d, J = 8.0 Hz, 2 H), 7.60–7.57 (m, 1 H), 7.82 (d, J = 8.1 Hz, 2 H), 8.02 (dd, J = 1.7, 7.8 Hz, 1 H) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 20.8, 54.7, 111.4, 120.5, 123.0, 128.4, 128.5, 129.4, 129.5, 135.3, 143.8, 159.4, 192.3, 193.7 ppm. MS (EI): m/z = 254 $[M]^+$.

1-(4-Methoxyphenyl)-2-(*p*-tolyl)ethane-1,2-dione (2i):[4e] Yellow solid, m.p. 80–82 °C. IR (KBr): $\tilde{\nu}$ = 2938, 1670, 1600, 1570, 1510, 1426, 1309, 1266, 1169, 1027, 885, 853, 758, 741 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.43 (s, 3 H), 3.88 (s, 3 H), 6.97 (d, J = 8.8 Hz, 2 H), 7.23 (d, J = 8.0 Hz, 2 H), 7.86 (d, J = 8.1 Hz, 2 H), 7.94 (d, J = 8.7 Hz, 2 H) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 21.9, 55.6, 114.3, 126.2, 129.7, 130.0, 130.8, 132.4, 146.0, 164.9, 193.4, 194.6 ppm. MS (EI): m/z = 254 $[M]^+$.

1,2-Di-*p*-tolylethane-1,2-dione (2j):[3g] Yellow solid, m.p. 103–104 °C. IR (KBr): $\tilde{\nu}$ = 2919, 1663, 1603, 1447, 1329, 1221, 1172, 885, 830, 782, 742 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.43 (s, 3 H), 7.30 (d, J = 7.9 Hz, 4 H), 7.86 (d, J = 8.0 Hz, 4 H) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 20.9, 126.7, 129.0, 129.7, 145.0, 193.5 ppm. MS (EI): m/z = 238 $[M]^+$.

1-(3-Fluorophenyl)-2-(4-methoxyphenyl)ethane-1,2-dione (2k): Yellow solid, m.p. 66–67 °C. IR (KBr): $\tilde{\nu}$ = 3075, 2929, 2842, 1671, 1600, 1511, 1443, 1312, 1149, 1026, 929, 884, 850, 783, 744 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 3.89 (s, 3 H), 6.99 (d, J = 8.9 Hz, 2 H), 7.36–7.33 (m, 1 H), 7.51–7.46 (m, 1 H), 7.69 (d, J = 9.0 Hz, 1 H), 7.73 (d, J = 7.7 Hz, 1 H), 7.94 (d, J = 8.9 Hz, 2 H) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 55.7, 114.5, 116.0, 116.2, 121.8, 121.9, 125.8, 125.9, 130.7, 132.5, 135.2, 162.0, 163.7, 165.2, 192.2, 193.3 ppm. MS (EI): m/z = 258 $[M]^+$. HRMS (EI): calcd. for $C_{15}H_{11}FO_3$ $[M]^+$ 258.0692; found 258.0691. $C_{15}H_{11}FO_3$ (258.07): calcd. C 69.76, H 4.29; found C 69.77, H 4.31.

1-(3-Fluorophenyl)-2-(*p*-tolyl)ethane-1,2-dione (2l): Yellow oil. IR (KBr): $\tilde{\nu}$ = 3072, 2925, 1683, 1602, 1484, 1445, 1306, 1238, 1151, 893, 806, 782, 742, 692 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.44 (s, 3 H), 7.31–7.36 (m, 3 H), 7.46–7.50 (m, 1 H), 7.69 (d, J = 8.8 Hz, 1 H), 7.73 (d, J = 7.7 Hz, 1 H), 7.86 (d, J = 7.7 Hz, 2 H) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 22.0, 116.0, 116.2, 121.8, 121.9, 125.9, 126.0, 129.8, 130.1, 130.3, 130.7, 130.8, 135.1, 135.2, 146.5, 162.0, 163.7, 193.2, 193.4 ppm. MS (EI): m/z = 242 $[M]^+$. HRMS (EI): calcd. for $C_{15}H_{11}FO_2$ $[M]^+$ 242.0743; found 242.0745. $C_{15}H_{11}FO_2$ (242.07): calcd. C 74.37, H 4.58; found C 74.36, H 4.57.

CCDC-844592 (for **3**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Supporting Information (see footnote on the first page of this article): ^{1}H and ^{13}C NMR spectra of the prepared compounds.

Acknowledgments

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