Simple and Efficient Access to 3-Ethoxycarbonylpyrroles, Benzofurans, and Naphthofurans

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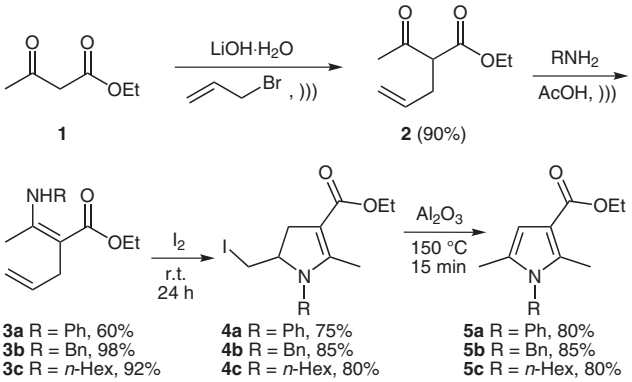
Abstract: An efficient method was developed for the synthesis of pyrrole and furan derivatives from enamines, phenols, and naphthols. The key steps involve iodocyclization and alumina-induced dehydroiodination reactions.

Key words: alumina, dehydroiodination, heterocycles, iodocyclization, ultrasound

Pyrrole and furan derivatives are important classes of five-membered heterocycles found in many natural products.[1,2] They have attracted much attention because of their broad spectrum of pharmacological activities, including antineoplastic,[3] antipsychotic,[4] antimicrobial, antioxidant, and anti-retroviral activities.[5] These compounds can be readily prepared by iodine-promoted cyclization of functionally substituted alkenes.[6,7] For example, we have previously reported a synthesis of pyrroles by iodocyclization of β-enamino esters followed by elimination.[6] However, the new demand for synthetic routes that are rapid, inexpensive, and environmentally safe must be considered. In seeking more-efficient methods, we decide to try adapting a method that we previously developed for the synthesis of pyrroles.[6] First, we found that ultrasound promotes the alkylation of ethyl acetoacetate enolates with allyl bromide. This procedure, which can be performed without a solvent, satisfies the demands of environmentally benign green chemistry. Because of the recent popularity of organic transformations under solvent-free conditions, we also examined the dehydroiodination of the intermediate products on alumina in the absence of a solvent. Benzofurans and naphthofurans were similarly prepared by the same protocol.

The required starting material, ethyl 2-acetylpent-4-enoate (**2**), was prepared by alkylation of ethyl acetoacetate (**1**) with allyl bromide in the presence of lithium hydroxide monohydrate and water in an ultrasound bath at room temperature. Our process resulted in an improvement in the concentration and yields of monoalkyl products in comparison with all other methods that use various organic solvents.[8] The condensation of amines with β-keto ester **2** by the method that we previously described,[9] followed by treatment with iodine, afforded the corresponding (iodomethyl)dihydropyrroles derivatives **4** in excellent yields. Finally, reaction of the iodo derivatives with neutral alumina at 150 °C gave the required pyrrole derivatives **5** in good yields (Scheme 1).



Scheme 1 Synthesis of 3-ethoxycarbonylpyrroles

The dehydroiodination reaction can be carried out without a solvent and the reaction mixture must be treated with an organic solvent that is capable of dissolving the organic materials; alumina is therefore suitable as both a support and a reactant because it can be easily removed by filtration at the end of the preparation and recycled.

The method was also used for the synthesis of benzofurans and naphthofuran derivatives. Thus, reaction of phenols **6**,[10] and **9**[11] and naphthols **12**[12] and **15**[10] with iodine, followed by treatment with alumina with heating gave the corresponding products **8**,[13] **11**,[14] **14**,[15] and **17**,[7] respectively (Scheme 2 and Scheme 3).

Finally, this method was extended to the synthesis of 2,4-dimethylfuro[3,2-*c*]quinoline (**20**).[16] 3-Allyl-2-methylquinolin-4-ol (**18**), which we recently prepared,[17] underwent iodocyclization to give dihydrofuran derivative **19**[11] in excellent yield. Dehydroiodination of **19**, as described above, gave the desired product, as shown in Scheme 4.

As shown in Scheme 5, we propose that an intermediate **A** may be involved in the reaction; because of its basic character, alumina may abstract a proton to give an exometh-

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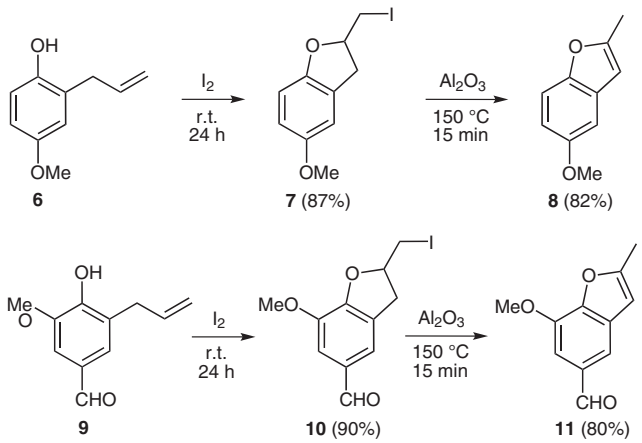
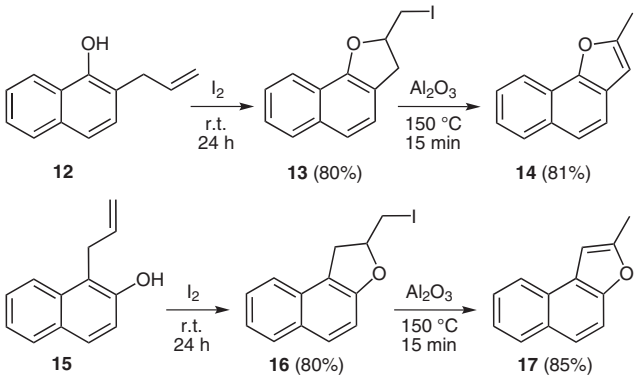
ylene intermediate **B**, which aromatizes to give the product **C**.

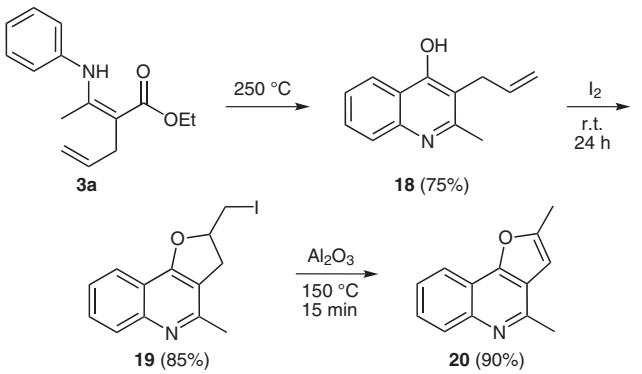
The simplicity of the whole process, the ready availability of starting materials, and the high overall yield make this strategy very attractive for the synthesis of this very important group of heterocyclic compounds, which can serve key intermediates for the synthesis of several biologically active products.



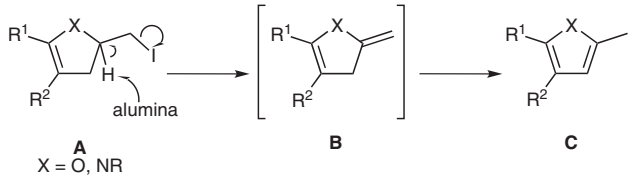
Scheme 2 Synthesis of benzofurans **8** and **11**



Scheme 3 Synthesis of naphthofurans **14** and **17**



Scheme 4 Synthesis of 2,4-dimethylfuro[3,2-*c*]quinoline (**20**)



Scheme 5 Proposed reaction mechanism

^{1}H NMR and ^{13}C NMR spectra were recorded with a Bruker DPX300 spectrometer operating at 300 MHz and 75 MHz, respectively, in $CDCl_3$ as a solvent with TMS as an internal standard. Chemical shifts are reported in parts per million (ppm, δ units). Coupling constants are reported in units of hertz (Hz), if applicable. IR spectra were recorded with a Bomem MB100-FTIR spectrometer, measured in percentage of transmittance (% T) on samples prepared as films or KBr pellets. All GC/MS studies were performed on a Shimadzu 14B/QP5050A with a DB1 column (30 m). For liquid chromatography, 70–230 mesh silica gel (Merck) was used as the stationary phase.

Ethyl-2-acetylpent-4-enoate (2)

A mixture of ethyl acetoacetate (**1**; 10.5 mmol), LiOH (10.5 mmol), H_2O (2 mL), and allyl bromide (10.0 mmol) was placed in an ultrasound bath (USC 700, 40 KHz) for 15 min. The mixture was then extracted with EtOAc (20 mL), dried (anhyd Na_2SO_4), and concentrated. For technical reasons, the products from three independent reactions were combined and purified by fractional distillation at 115 °C/16 mm Hg; yield: 4.60 g (90%).

Iodocyclization; General Procedure

A soln of the substituted alkene derivative (5 mmol) in CH_2Cl_2 (25 mL) was treated with $NaHCO_3$ (5.5 mmol) and I_2 (5.5 mmol). The mixture was stirred at r.t. for 24 h then extracted with EtOAc. The combined organic layers were washed sequentially with aq $Na_2S_2O_3$ and sat. aq NaCl, dried ($MgSO_4$), and concentrated under reduced pressure.

Ethyl 5-(Iodomethyl)-2-methyl-1-phenyl-4,5-dihydro-1*H*-pyrrole-3-carboxylate (4a)

Yield: 75%.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.30 (t, *J* = 7.3 Hz, 3 H), 2.08 (s, 3 H), 2.65 (dd, *J* = 8.0, 15.1 Hz, 1 H), 3.05 (dd, *J* = 10.0, 15.1 Hz, 1 H), 3.15 (dd, *J* = 3.0, 10.5 Hz, 1 H), 3.25 (dd, *J* = 7.3, 10.5 Hz, 1 H), 4.15 (q, *J* = 7.3 Hz, 2 H), 4.05–4.20 (m, 1 H), 7.10–7.45 (m, 5 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 11.3, 13.9, 14.5, 35.1, 58.5, 64.2, 98.5, 126.8, 127.5, 129.5, 140.2, 157.5, 166.7.

Ethyl 1-Benzyl-5-(iodomethyl)-2-methyl-4,5-dihydro-1*H*-pyrrole-3-carboxylate (4b)

Yield: 85%.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.27 (t, *J* = 7.1 Hz, 3 H), 2.29 (s, 3 H), 2.53 (dd, *J* = 7.2, 15.1 Hz, 1 H), 3.04 (dd, *J* = 11.2, 15.1 Hz, 1 H), 3.10–3.20 (m, 2 H), 3.54–3.67 (m, 1 H), 4.15 (q, 7.1 Hz, 2 H), 4.25 (d, *J* = 16.8 Hz, 1 H), 4.52 (d, *J* = 16.8 Hz, 1 H), 7.14–7.35 (m, 5 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 10.9, 12.3, 14.6, 35.2, 47.9, 58.6, 60.7, 95.7, 126.7, 127.5, 128.8, 137.1, 159.6, 166.9.

Ethyl 1-Hexyl-5-(iodomethyl)-2-methyl-4,5-dihydro-1*H*-pyrrole-3-carboxylate (4c)

Yield: 80%.

IR (film): 1687, 1585, 1239, 1081 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.89 (t, *J* = 6.9 Hz, 3 H), 1.25 (t, *J* = 7.1 Hz, 3 H), 1.25–1.38 (m, 6 H), 1.50–1.60 (m, 2 H), 2.05 (s, 3 H), 2.50 (dd, *J* = 7.5, 15.0 Hz, 1 H), 3.00 (dd, *J* = 11.0, 15.0 Hz, 1 H), 3.08–3.17 (m, 2 H), 3.20 (t, *J* = 7.0 Hz, 2 H), 4.00–4.13 (m, 1 H), 4.15 (q, 7.1 Hz, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 11.3, 12.3, 13.8, 14.1, 22.4, 26.5, 27.4, 31.0, 35.9, 43.6, 59.2, 61.4, 110.1, 157.9, 166.9.

Anal. Calcd for $C_{15}H_{26}NO_2I$: C, 47.50; H, 6.91; N, 3.69. Found: C, 47.66; H, 6.88; N, 3.74.

2-(Iodomethyl)-5-methoxy-2,3-dihydro-1-benzofuran (7)
Yield: 87%.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.15 (dd, *J* = 6.9, 15.9 Hz, 1 H), 3.22–3.45 (m, 3 H), 3.74 (s, 3 H), 4.80–4.92 (m, 1 H), 6.79 (dd, *J* = 2.7, 8.9 Hz, 1 H), 6.94 (d, *J* = 2.7 Hz, 1 H), 7.27 (d, *J* = 8.9 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 9.1, 36.5, 56.0, 81.8, 109.5, 111.4, 113.1, 126.7, 153.3, 154.4.

2-(Iodomethyl)-2,3-dihydronaphtho[1,2-*b*]furan (13)
Yield: 80%.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.22 (dd, *J* = 6.5, 15.9 Hz, 1 H), 3.41 (dd, *J* = 7.8, 10.0 Hz, 1 H), 3.55 (dd, *J* = 4.7, 10.0 Hz, 1 H), 3.58 (dd, *J* = 9.3, 15.9 Hz, 1 H), 5.05–5.15 (m, 1 H), 7.30 (d, *J* = 8.2 Hz, 1 H), 7.39 (d, *J* = 8.2 Hz, 1 H), 7.40–7.46 (m, 2 H), 7.80 (d, *J* = 8.0 Hz, 1 H), 7.93 (d, *J* = 8.0 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 9.2, 36.9, 82.3, 118.7, 120.4, 120.7, 121.3, 122.7, 125.4, 125.8, 127.8, 133.5, 154.4.

2-(Iodomethyl)-1,2-dihydronaphtho[2,1-*b*]furan (16)
Yield: 80%.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.31 (dd, *J* = 8.7, 15.7 Hz, 1 H), 3.39–3.55 (m, 2 H), 3.67 (dd, *J* = 9.5, 15.7 Hz, 1 H), 5.05–5.14 (m, 1 H), 7.10 (d, *J* = 8.8 Hz, 1 H), 7.32 (t, *J* = 8.2 Hz, 1 H), 7.48 (t, *J* = 8.2 Hz, 1 H), 7.59 (d, *J* = 8.8 Hz, 1 H), 7.70 (d, *J* = 8.2 Hz, 1 H), 7.81 (d, *J* = 8.2 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 9.2, 35.1, 82.4, 112.0, 122.7, 123.1, 126.8, 128.7, 128.9, 129.3, 129.4, 130.7, 156.7.

2-(Iodomethyl)-4-methyl-2,3-dihydrofuro[3,2-*c*]quinoline (19)
Yield: 85%.

IR (KBr): 1644, 1592, 1550, 1505, 1331, 1158 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.02 (s, 3 H), 3.26 (dd, *J* = 6.5, 15.9 Hz, 1 H), 3.57–3.68 (m, 2 H), 3.66 (dd, *J* = 10.0, 15.9 Hz, 1 H), 5.38–5.48 (m, 1 H), 7.70 (t, *J* = 8.6, 1 H), 7.92 (t, *J* = 8.6 Hz, 1 H), 8.03 (d, *J* = 8.6 Hz, 1 H), 8.84 (d, *J* = 8.6 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 12.1, 23.1, 35.2, 84.4, 115.7, 117.2, 121.9, 126.1, 128.3, 130.1, 148.4, 156.3, 162.5.

Anal. Calcd for $C_{13}H_{13}INO$: C, 48.02; H, 3.72; N, 4.31. Found: C, 47.92; H, 3.83; N, 4.25.

Iodocyclization of 3-Allyl-4-hydroxy-5-methoxybenzaldehyde (9)
The procedure was the same as the general procedure, except that a 1:1 mixture of H_2O and MeOH was used as the solvent.

2-(Iodomethyl)-7-methoxy-2,3-dihydro-1-benzofuran-5-carbaldehyde (10)
Yield: 90%.

IR (film): 2715, 1705, 1655, 1580, 1105 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.16 (dd, *J* = 7.0, 16.1 Hz, 1 H), 3.41 (dd, *J* = 7.9, 10.3 Hz, 1 H), 3.50 (dd, *J* = 9.1, 16.1 Hz, 1 H), 3.53 (dd, *J* = 4.1, 10.3 Hz, 1 H), 3.94 (s, 3 H), 5.00–5.06 (m, 1 H), 7.33 (s, 1 H), 7.35 (s, 1 H), 9.81 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 9.1, 36.2, 56.0, 82.1, 110.4, 126.5, 126.8, 129.2, 149.1, 153.8, 190.7.

Anal. Calcd for $C_{11}H_{11}IO_3$: C, 41.53; H, 3.49. Found: C, 41.68; H, 3.54.

Dehydroiodination; General Procedure
Neutral activated Al_2O_3 (Merck 70–230 mesh; 30 g) was added to a stirred solution of the cyclic iodine derivative (1 mmol) in CH_2Cl_2 (20 mL). The solvent was removed under vacuum, and the mixture was heated at 160 °C for 15 min then cooled to r.t. CH_2Cl_2 (10 mL) was added, the Al_2O_3 was removed by Büchner filtration, and the organic phase was evaporated under vacuum.

Ethyl 2,5-Dimethyl-1-phenyl-1*H*-pyrrole-3-carboxylate (5a)
Yield: 80%.

IR (KBr): 1685, 1582, 1420, 1220, 1090 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.31 (t, *J* = 7.3 Hz, 3 H), 1.97 (s, 3 H), 2.28 (s, 3 H), 4.28 (q, *J* = 7.3 Hz, 2 H), 6.38 (s, 1 H), 7.10–7.55 (m, 5 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 12.1, 12.6, 14.3, 58.9, 107.5, 111.5, 128.0, 128.4, 128.7, 129.5, 136.0, 137.7, 165.6.

Anal. Calcd for $C_{15}H_{17}NO_2$: C, 74.05; H, 7.04; N, 5.76. Found: C, 73.89; H, 6.92; N, 5.69.

Ethyl 1-Benzyl-2,5-dimethyl-1*H*-pyrrole-3-carboxylate (5b)
Yield: 85%.

IR (KBr): 1701, 1692, 1606, 1581 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.29 (t, *J* = 7.1 Hz, 3 H), 2.09 (s, 3 H), 2.45 (s, 3 H), 4.26 (q, *J* = 7.1 Hz, 2 H), 5.01 (s, 2 H), 6.35 (s, 1 H), 6.85–7.32 (m, 5 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 11.1, 11.9, 14.4, 46.6, 59.0, 107.6, 111.0, 125.4, 127.2, 127.8, 128.7, 135.3, 136.9, 165.5.

Ethyl 1-Hexyl-2,5-dimethyl-1*H*-pyrrole-3-carboxylate (5c)
Yield: 80%.

IR (KBr): 1697, 1580, 1230, 1085 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.89 (t, *J* = 6.9 Hz, 3 H), 1.29–1.34 (m, 6 H), 1.31 (t, *J* = 7.1 Hz, 3 H), 1.50–1.65 (m, 2 H), 2.19 (s, 3 H), 2.51 (s, 3 H), 3.73 (t, *J* = 6.9 Hz, 2 H), 4.23 (q, *J* = 7.1 Hz, 2 H), 6.24 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 11.4, 14.0, 14.6, 22.6, 26.5, 26.9, 30.6, 31.5, 43.7, 59.1, 107.4, 119.2, 127.3, 132.2, 165.8.

Anal. Calcd for $C_{15}H_{25}NO_2$: C, 71.67; H, 10.03; N, 5.57. Found: C, 71.82; H, 9.83; N, 5.45.

5-Methoxy-2-methyl-1-benzofuran (8)
Yield: 82%.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.42 (d, *J* = 1.0 Hz, 3 H), 3.82 (s, 3 H), 6.30 (q, *J* = 1.0 Hz, 1 H), 6.79 (dd, *J* = 2.6, 8.9 Hz, 1 H), 6.94 (d, J = 2.6 Hz, 1 H), 7.28 (d, *J* = 8.9 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.2, 58.5, 102.8, 103.1, 110.9, 111.3, 129.8, 149.7, 155.7, 156.3.

7-Methoxy-2-methyl-1-benzofuran-5-carbaldehyde (11)
Yield: 80%.

IR (KBr): 2715, 1715, 1660, 1573, 1110 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.39 (d, *J* = 0.8 Hz, 3 H), 3.98 (s, 3 H), 6.75 (q, *J* = 0.8 Hz, 1 H), 7.28 (s, 1 H), 7.35 (s, 1 H), 9.92 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 56.2, 101.9, 118.2, 123.5, 128.6, 129.5, 148.5, 154.8, 156.6, 191.5.

Anal. Calcd for $C_{11}H_{10}O_3$: C, 69.46; H, 5.30. Found: C, 69.33; H, 5.38.

2-Methylnaphtho[1,2-*b*]furan (14)
Yield: 81%.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.52 (d, *J* = 1.0 Hz, 3 H), 6.45 (q, *J* = 1.0 Hz, 1 H), 7.40 (t, *J* = 8.1 Hz, 1 H), 7.52 (t, *J* = 8.1 Hz, 1 H), 7.53 (d, *J* = 8.5 Hz, 1 H), 7.58 (d, *J* = 8.5 Hz, 1 H), 7.87 (d, *J* = 8.1 Hz, 1 H), 8.24 (d, *J* = 8.1 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.1, 103.6, 119.2, 119.6, 121.2, 122.9, 124.4, 124.5, 126.1, 128.3, 130.8, 149.8, 154.8.

2-Methylnaphtho[2,1-*b*]furan (17)
Yield: 85%.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.52 (d, *J* = 1.1 Hz, 3 H), 6.45 (q, *J* = 1.1 Hz, 1 H), 7.41 (t, *J* = 8.2 Hz, 1 H), 7.53 (t, *J* = 8.2 Hz, 1 H), 7.54 (d, *J* = 8.5 Hz, 1 H), 7.59 (d, *J* = 8.5 Hz, 1 H), 7.88 (d, *J* = 8.2 Hz, 1 H), 8.25 (d, *J* = 8.2 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 103.2, 118.1, 118.5, 121.8, 122.3, 125.2, 125.7, 128.5, 129.0, 130.1, 150.2, 154.4.

2,4-Dimethylfuro[3,2-*c*]quinoline (20)
Yield: 90%.

IR (KBr): 1658, 1589, 1520, 1250 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.53 (d, *J* = 1.1 Hz, 3 H), 2.80 (s, 3 H), 6.48 (q, *J* = 1.1 Hz, 1 H), 7.51 (t, *J* = 8.0 Hz, 1 H), 7.61 (t, *J* = 8.0 Hz, 1 H), 8.09 (d, *J* = 8.0 Hz, 1 H), 8.14 (d, *J* = 8.0 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.2, 23.2, 106.5, 116.2, 120.1, 121.9, 126.9, 128.5, 129.5, 145.3, 154.4, 154.1, 156.3.

Anal. Calcd for $C_{13}H_{11}NO$: C, 79.16; H, 5.62; N, 7.10. Found: C, 79.12; H, 5.69; N, 7.15.

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