

단 신

알파-(메틸sul피닐)케톤류를 이용한 치환페놀류로부터 2-알킬벤조푸란 유도체의 합성

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Synthesis of 2-Alkylbenzofuran Derivatives from Substituted Phenols Using α -(Methylsulfinyl)ketones

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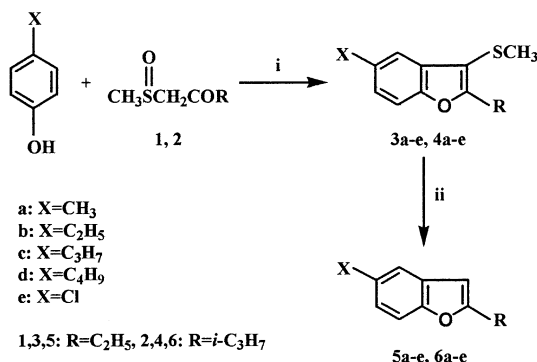
As a part of our continuing study concerning the carbon-carbon bond forming reactions utilizing 1-acyl-1-thiocarbocations, we have reported the Friedel-Crafts reaction of substituted phenols with 1-acyl-1-chlorosulfides leading to 2-methylbenzofuran derivatives,¹ 2-arylbenzofuran derivatives,² and a precursor of demethoxyegonol.³

In the preceding paper,⁴ we showed that the one-pot reaction of substituted phenols with 1-acyl-1-thiocarbocationic intermediate generated from α -(methylsulfinyl)acetone in the presence of *p*-toluenesulfonic acid provided a convenient method for synthesizing 2-methylbenzofuran derivatives through reductive desulfurization of the resulting products. In the present paper the method is applied to syntheses of 2-ethylbenzofurans **3** and 2-isopropylbenzofurans **4**, in which methylsulfinylmethyl alkyl ketone (**1** and **2**) are employed as electrophiles in place of α -(methylsulfinyl)acetone.

For the preparation of the starting materials, oxidation of methylthiomethyl ethyl ketone with sodium metaperiodate in aqueous methanol afforded α -(methylsulfinyl)ethyl ketone (**1**) in 82% yield, and α -(methylsulfinyl)isobutyl ketone (**2**) was obtained from the reaction of ethyl isobutyrate with methylsulfinyl carbanion according to the procedure reported⁵ in 68% yield.

The previous study on Pummerer reaction with α -(methylsulfinyl) acetone⁴ revealed that the reaction requires three equivalents of anhydrous *p*-toluenesulfonic acid without a Dean-Stark water separator. On the basis of this information, the Pummerer reaction of *para*-substituted phenols with **1** and **2** was established as shown in Scheme 1.

The treatment of equimolar amounts of *p*-cresol and the sulfoxide **1** in 1,2-dichloroethane with three equivalents of *p*-toluenesulfonic acid under reflux gave 2-ethyl-5-methyl-3-(methylthio)benzofuran (**3a**) in 68% yield.



Scheme 1. Reagents and conditions: (i) *p*-TsOH, ClCH₂CH₂Cl, reflux, 1 h; (ii) Raney-Ni (W-2), EtOH, 60-65 °C, 1 h.

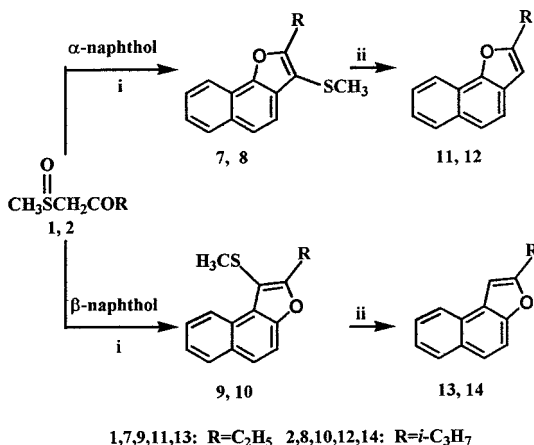
The reactions of other arenes generally took place smoothly in the presence of *p*-toluenesulfonic acid and gave 2-ethyl-3-(methylthio) benzofuran derivatives **3b-e** in satisfactory yields.

According to the above procedure, *para*-substituted phenols were allowed to react with the sulfoxide **2** to give the 2-isopropyl-3-(methylthio)benzofuran derivatives **4a-e** in significant yields.

The adducts **3** obtained through the above Pummerer reaction could easily be desulfurized into the corresponding 2-ethylbenzofurans **5** upon heating with Raney nickel in ethanol. Thus, the adducts **3a-e** were converted into **5a-e**, respectively, in good yields. Also the desulfurization of **4** with Raney nickel in ethanol furnished the corresponding 2-isopropylbenzofurans **6**.

To this end, we have examined the reactions of **1** and **2** with naphthol isomers. Under the reaction conditions such as described for the Pummerer reaction with *para*-substituted phenols, this reaction gave 2-alkyl-3-(methylthio)naphtho[1,2-*b*]furans (**7** and **8**) and 2-alkyl-1-(methylthio)naphtho[2,1-*b*]furans (**9** and **10**) in moderate yields, respectively. (Scheme 2) The desulfurization of adducts (**7,8,9**, and **10**) with Raney nickel in ethanol afforded the corresponding naphthofurans (**11,12,13**, and **14**, respectively).

Many synthetic methods for 2-alkylbenzofuran derivatives have so far been reported in the literature, the following methods being representative: 1) the reaction of



Scheme 2. Reagents and conditions: (i) *p*-TsOH, CICH₂CH₂Cl, reflux, 1 h; (ii) Raney-Ni (W-2), EtOH, 60-65 °C, 1 h.

acid chloride or acid anhydride with *o*-hydroxybenzyl triphenylphosphonium bromide in the presence of triethylamine,⁶ 2) the reaction of (2-methoxyphenyl)ethynes with lithium iodide in 2,4,6-trimethylpyridine,⁷ 3) intramolecular [2+2] cycloaddition reaction of ketene and carbonyl groups,⁸ and 4) the cyclization of alkynyl(*p*-phenylene) bisiodonium ditriflates with phenoxide anion.⁹

In summary, we developed a general route for the formation of 2-alkylbenzofuran derivatives (**5** and **6**). Our method consists of two steps: i) the one-pot synthesis of 2-alkyl-3-(methylthio)benzofurans (**3** and **4**) by the reaction of *para*-substituted phenols with 1-acyl-1-thiocarbocationic intermediates generated from the sulfoxides (**1** and **2**) in the presence of anhydrous *p*-toluenesulfonic acid, and ii) the reductive desulfurization of the resulting products (**3** and **4**).

This method will provide a promising route for the preparation of 2-substituted benzofuran skeleton. As a preliminary research towards the synthesis of products bearing 2-arylbenzofuran moiety, the above Pummerer reaction with various α -(methylsulfinyl) ketones is proceeding.

EXPERIMENTAL

IR spectra were obtained from JASCO FT/IR-300E spectrometer. ¹H NMR spectra were recorded from Hitachi R-1500 FT NMR (60 MHz) spectrometer using tetramethylsilane as an internal standard. Mass data were obtained from Hewlett Packard 5970 GC/MS system (EI, 70 eV). Merck silica gel 60 (70-230 mesh) was used for column chromatography.

Methylsulfinylmethyl ethyl ketone (1). A solution of sodium metaperiodate (6 g, 28 mmol) in water (30 mL) was added in small portions to a stirred solution of methylthiomethyl ethyl ketone (3 g, 25.4 mmol) in methanol (60 mL) at 0 °C and the mixture was further stirred at room temperature for 12 h. Inorganic materials were filtered off and the filtrate was extracted with chloroform (3×20 mL). The combined extracts were dried over MgSO₄, and concentrated under reduced pressure. The residue was purified by column chromatography (acetone) to give **1** (2.79 g, 82%) as an oil. IR (neat) 2979, 2899, 1709 (C=O), 1411, 1378, 1122, 1025 (S=O) cm⁻¹; ¹H NMR (CDCl₃) δ 1.10 (t, 3H, J=7.02Hz), 2.65 (q, 2H,

$J=7.02\text{Hz}$), 2.69 (s, 3H), 3.76 (d, 2H, $J=1.20\text{Hz}$); MS m/z 134 (M^+).

Methylsulfinylmethyl isopropyl ketone (2). A suspension of sodium hydride (60% mineral oil dispersion; 3 g, 75 mmol) in dimethyl sulfoxide (50 mL) was heated with stirring at 70–75 °C for 40 min under nitrogen. After cooling to room temperature, tetrahydrofuran (20 mL) was added. To the reaction mixture was added ethyl isobutyrate (3.48 g, 30 mmol) at 0 °C, and the stirring was continued for 90 min at the room temperature. The mixture was poured into water (200 mL), acidified with aqueous HCl to a pH of 3–4, and thoroughly extracted with chloroform (3×50 mL). The combined extracts were washed with water (3×30 mL), dried over MgSO_4 , and evaporated off. The residue was purified by column chromatography (acetone) to give **2** (3 g, 68%) as an oil. IR (neat) 2981, 2894, 1710 ($\text{C}=\text{O}$), 1416, 1377, 1123, 1038 ($\text{S}=\text{O}$) cm^{-1} ; ^1H NMR (CDCl_3) δ 1.16 (d, 6H, $J=7.02\text{Hz}$), 2.54–2.87 (m, 1H), 2.71 (s, 3H), 3.86 (s, 2H); MS m/z 148 (M^+).

General procedure for synthesis of 2-ethyl-3-(methylthio)benzofurans (3). A solution of a *para*-substituted phenol (1.49 mmol), **1** (200 mg, 1.49 mmol) and anhydrous *p*-toluenesulfonic acid (769 mg, 4.47 mmol) in 1,2-dichloroethane (15 mL) was refluxed for 1 h. The reaction mixture was washed with water, and dried over MgSO_4 . The solvent was removed *in vacuo* and the residue was purified by column chromatography (hexane/ethyl acetate=15/1) to give **3**. **3a**: Yield 68%, oil, IR (neat) 2978, 2921, 1578, 1473, 1265, 1190, 1153, 1023 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.30 (t, 3H, $J=7.50\text{Hz}$), 2.30 (s, 3H), 2.46 (s, 3H), 2.93 (q, 2H, $J=7.50\text{Hz}$), 6.97–7.42 (m, 3H); MS m/z 206 (M^+). **3b**: Yield 65%, oil, IR (neat) 2969, 2867, 1588, 1465, 1278, 1189, 1155, 1022 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.30 (t, 6H, $J=7.62\text{Hz}$), 2.31 (s, 3H), 2.57–3.14 (m, 4H), 7.02–7.43 (m, 3H); MS m/z 220 (M^+). **3c**: Yield 67%, oil, IR (neat) 2922, 2867, 1588, 1464, 1266, 1189, 1155, 1033 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.95 (t, 3H, $J=7.02\text{Hz}$), 1.30 (t, 3H, $J=7.62\text{Hz}$), 1.47–1.88 (m, 2H), 2.31 (s, 3H), 2.54–3.13 (m, 4H), 6.98–7.42 (m, 3H); MS m/z 234 (M^+). **3d**: Yield 70%, oil, IR (neat) 2962, 1577, 1469, 1378, 1278, 1189 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.30 (t, 3H, $J=7.62\text{Hz}$), 1.39 (s, 9H), 2.31 (s, 3H), 2.93 (q, 2H, $J=7.62\text{Hz}$), 7.24–7.62 (m, 3H); MS m/z 248 (M^+). **3e**: Yield 40%, oil, IR (neat) 2933, 1588, 1448, 1266,

1177, 1077 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.31 (t, 3H, $J=7.62\text{Hz}$), 2.30 (s, 3H), 2.94 (q, 2H, $J=7.62\text{Hz}$), 7.27–7.68 (m, 3H); MS m/z 226 (M^+).

General procedure for synthesis of 2-isopropyl-3-(methylthio)benzofurans (4). According to the same procedure for the preparation of **3**, compounds **4** were obtained from a *para*-substituted phenol (2.0 mmol), **2** (300 mg, 2.0 mmol) and anhydrous *p*-toluenesulfonic acid (1 g, 6.0 mmol). **4a**: Yield 63%, oil, IR (neat) 2966, 2922, 2867, 1566, 1477, 1266, 1200, 1055, 1033 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.34 (d, 6H, $J=7.02\text{Hz}$), 2.31 (s, 3H), 2.45 (s, 3H), 3.32–3.76 (m, 1H), 6.96–7.43 (m, 3H); MS m/z 220 (M^+). **4b**: Yield 68%, oil, IR (neat) 2966, 2922, 2867, 1656, 1577, 1477, 1266, 1200, 1064, 1033 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.22 (t, 3H, $J=7.62\text{Hz}$), 1.34 (d, 6H, $J=7.02\text{Hz}$), 2.31 (s, 3H), 2.76 (q, 2H, $J=7.62\text{Hz}$), 3.31–3.76 (m, 1H), 7.01–7.48 (m, 3H); MS m/z 234 (M^+). **4c**: Yield 62%, oil, IR (neat) 2965, 2921, 2867, 1577, 1477, 1266, 1200, 1155, 1066 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.95 (t, 3H, $J=7.02\text{Hz}$), 1.33 (d, 6H, $J=7.02\text{Hz}$), 1.49–2.04 (m, 2H), 2.31 (s, 3H), 2.71 (t, 2H, $J=6.48\text{Hz}$), 3.21–3.74 (m, 1H), 6.97–7.42 (m, 3H); MS m/z 248 (M^+). **4d**: Yield 70%, oil, IR (neat) 2965, 2867, 1577, 1477, 1366, 1200, 1132, 1066 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.33 (d, 6H, $J=7.02\text{Hz}$), 1.39 (s, 9H), 2.31 (s, 3H), 3.32–3.86 (m, 1H), 7.35 (br s, 2H), 7.61 (br s, 1H); MS m/z 262 (M^+). **4e**: Yield 40%, oil, IR (neat) 2955, 2911, 2867, 1577, 1455, 1366, 1310, 1255, 1189, cm^{-1} ; ^1H NMR (CDCl_3) δ 1.34 (d, 6H, $J=7.02\text{Hz}$), 2.30 (s, 3H), 3.22–3.78 (m, 1H), 7.27 (br s, 2H), 7.56 (br s, 1H); MS m/z 240 (M^+).

General procedure for synthesis of 2-ethylbenzofurans (5). The compound **3** (150–200 mg) was heated at 60–65 °C in ethanol (20 mL) containing Raney nickel (W-2, 1–1.5 g) for 1 h. The Raney nickel was removed by filtration and the solvent was evaporated off. The residue was chromatographed with hexane/ethyl acetate (15/1) as an eluent to give **5**. **5a**: Yield 86%, oil, IR (neat) 2974, 1598, 1469, 1266, 1198, 1150, 1055 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.32 (t, 3H, $J=7.62\text{Hz}$), 2.41 (s, 3H), 2.79 (q, 2H, $J=7.62\text{Hz}$), 6.29 (s, 1H), 6.98–7.38 (m, 3H); MS m/z 160 (M^+). **5b**: Yield 84%, oil, IR (neat) 2966, 2931, 1600, 1473, 1320, 1265, 1235, 1195, 1148, 1121, 1055, cm^{-1} ; ^1H NMR (CDCl_3) δ 1.02–1.44 (m, 6H), 2.59–2.98 (m, 4H), 6.31 (s, 1H), 7.10–7.41 (m, 3H); MS m/z 174 (M^+). **5c**: Yield 91%, oil, IR (neat) 2960, 2930, 2871,

1599, 1473, 1447, 1263, 1195, 1148, 1054 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.94 (t, 3H, $J=7.08\text{Hz}$), 1.32 (t, 3H, $J=7.08\text{Hz}$), 1.42-1.97 (m, 2H), 2.54-2.98 (m, 4H), 6.31 (s, 1H), 6.94-7.40 (m, 3H); MS m/z 188 (M^+). **5d**: Yield 88%, oil, IR (neat) 2962, 1598, 1471, 1365, 1272, 1176, 1132 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.31 (t, 3H, $J=7.62\text{Hz}$), 1.36 (s, 9H), 2.78 (q, 2H, $J=7.62\text{Hz}$), 6.33 (s, 1H), 7.29 (br s, 2H), 7.48 (br s, 1H); MS m/z 202 (M^+). **5e**: Yield 75%, oil, IR (neat) 2976, 2939, 1598, 1447, 1259, 1234, 1171, 1140, 1063 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.32 (t, 3H, $J=7.62\text{Hz}$), 2.80 (q, 2H, $J=7.62\text{Hz}$), 6.33 (s, 1H), 7.05-7.44 (m, 3H); MS m/z 180 (M^+).

General procedure for synthesis of 2-isopropylbenzofurans (6). According to the same procedure for the preparation of **5**, compounds **6** were obtained from **4** (150-200 mg) and Raney nickel (W-2, 1-1.5 g) in ethanol (20 mL). The residue was chromatographed with hexane/ethyl acetate (20/1) as an eluent to give **6**. **6a**: Yield 85%, oil, IR (neat) 2966, 2922, 2867, 1588, 1477, 1266, 1200, 1133, 1066, 1044 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.33 (d, 6H, $J=7.02\text{Hz}$), 2.41 (s, 1H), 2.83-3.30 (m, 1H), 6.30 (s, 1H), 6.92-7.38 (m, 3H); MS m/z 174 (M^+). **6b**: Yield 83%, oil, IR (neat) 2966, 2921, 2867, 1588, 1477, 1266, 1200, 1122, 1077 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.19 (t, 3H, $J=7.02\text{Hz}$), 1.33 (d, 6H, $J=7.02\text{Hz}$), 2.52-3.28 (m, 3H), 6.30 (s, 1H), 6.96-7.40 (m, 3H); MS m/z 188 (M^+). **6c**: Yield 87%, oil, IR (neat) 2955, 2911, 2867, 1600, 1477, 1266, 1200, 1133, 1066 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.94 (t, 3H, $J=7.08\text{Hz}$), 1.33 (d, 6H, $J=6.48\text{Hz}$), 1.41-1.98 (m, 2H), 2.53-3.22 (m, 3H), 6.30 (s, 1H), 6.94-7.42 (m, 3H); MS m/z 202 (M^+). **6d**: Yield 85%, oil, IR (neat) 2955, 2867, 1600, 1477, 1366, 1266, 1167, 1122, 1077 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.33 (d, 6H, $J=7.02\text{Hz}$), 1.35 (s, 9H), 2.74-3.25 (m, 1H), 6.32 (s, 1H), 7.31 (br s, 2H), 7.49 (br s, 1H); MS m/z 216 (M^+). **6e**: Yield 78%, oil, IR (neat) 2966, 2922, 2867, 1588, 1455, 1322, 1266, 1167, 1066 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.33 (d, 6H, $J=7.02\text{Hz}$), 2.73-3.30 (m, 1H), 6.30 (s, 1H), 6.80-7.56 (m, 3H); MS m/z 194 (M^+).

2-Ethyl-3-(methylthio)naphtho[1,2-b]furan (7). According to the same procedure for the preparation of **3**, compound **7** was obtained from α -naphthol (430 mg, 3.0 mmol), **1** (400 mg, 3.0 mmol) and anhydrous *p*-toluenesulfonic acid (1.54 g, 9.0 mmol) in 36% yield (261 mg) as an oil. IR (neat) 2978, 2922, 1577, 1444, 1389, 1266,

1177, 1011 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.40 (t, 3H, $J=7.62\text{Hz}$), 2.35 (s, 3H), 3.06 (q, 2H, $J=7.62\text{Hz}$), 7.25-8.42 (m, 6H); MS m/z 242 (M^+).

2-Isopropyl-3-(methylthio)naphtho[1,2-b]furan (8). According to the same procedure for the preparation of **3**, compound **8** was obtained from α -naphthol (430 mg, 3.0 mmol), **2** (445 mg, 3.0 mmol) and anhydrous *p*-toluenesulfonic acid (1.54 g, 9.0 mmol) in 34% yield (261 mg) as an oil. IR (neat) 2966, 2911, 2867, 1577, 1522, 1467, 1378, 1310, 1077, cm^{-1} ; ^1H NMR (CDCl_3) δ 1.44 (d, 6H, $J=7.02\text{Hz}$), 2.35 (s, 3H), 3.31-7.85 (m, 1H), 7.23-8.38 (m, 6H); MS m/z 256 (M^+).

2-Ethyl-1-(methylthio)naphtho[2,1-b]furan (9). According to the same procedure for the preparation of **3**, compound **9** was obtained from β -naphthol (430 mg, 3.0 mmol), **1** (400 mg, 3.0 mmol) and anhydrous *p*-toluenesulfonic acid (1.54 g, 9.0 mmol) in 52% yield (378 mg) as an oil. IR (neat) 2978, 2911, 1566, 1444, 1380, 1255, 1177, 1011 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.37 (t, 3H, $J=7.62\text{Hz}$), 2.37 (s, 3H), 3.07 (q, 2H, $J=7.62\text{Hz}$), 7.25-8.36 (m, 6H); MS m/z 242 (M^+).

2-Isopropyl-1-(methylthio)naphtho[2,1-b]furan (10). According to the same procedure for the preparation of **3**, compound **10** was obtained from β -naphthol (430 mg, 3.0 mmol), **2** (445 mg, 3.0 mmol) and anhydrous *p*-toluenesulfonic acid (1.54 g, 9.0 mmol) in 58% yield (445 mg) as an oil. IR (neat) 2966, 2911, 2867, 1566, 1389, 1255, 1155, 1066, 1011, cm^{-1} ; ^1H NMR (CDCl_3) δ 1.40 (d, 6H, $J=7.02\text{Hz}$), 2.37 (s, 3H), 3.50-3.88 (m, 1H), 7.24-8.05 (m, 5H), 9.17-9.34 (m, 1H); MS m/z 256 (M^+).

2-Ethyl-naphtho[1,2-b]furan (11). According to the same procedure for the preparation of **5**, compound **11** was obtained from **7** (101 mg, 0.42 mmol) and Raney nickel (W-2, 1.1 g) in 75% yield (62 mg) as an oil. IR (neat) 3060, 2973, 1637, 1572, 1523, 1458, 1387, 1313, 1267, 1171, 1082, 1003 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.39 (t, 3H, $J=7.62\text{Hz}$), 2.92 (q, 2H, $J=7.62\text{Hz}$), 6.50 (s, 1H), 7.24-8.36 (m, 6H); MS m/z 196 (M^+).

2-Isopropyl-naphtho[1,2-b]furan (12). According to the same procedure for the preparation of **5**, compound **12** was obtained from **8** (188 mg, 0.73 mmol) and Raney nickel (W-2, 1.9 g) in 73% yield (112 mg) as an oil. IR (neat) 3056, 2966, 2867, 1577, 1522, 1467, 1389, 1322, 1177, 1089 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.42 (d, 6H, $J=7.02\text{Hz}$), 3.02-3.64 (m, 1H), 6.50 (s, 1H), 7.25-8.37 (m, 6H); MS

m/z 210 (M^+).

2-Ethynaphtho[2,1-b]furan (13). According to the same procedure for the preparation of **5**, compound **13** was obtained from **9** (136 mg, 0.56 mmol) and Raney nickel (W-2, 1.4 g) in ethanol (20 mL) in 72% yield (79 mg) as an oil. IR (neat) 3056, 2973, 2936, 1655, 1629, 1577, 1523, 1459, 1448, 1385, 1256, 1164, 1055 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.40 (t, 3H, $J=7.62\text{Hz}$), 2.91 (q, 2H, $J=7.62\text{Hz}$), 6.88 (s, 1H), 7.25-8.04 (m, 6H); MS m/z 196 (M^+).

2-Isopropynaphtho[2,1-b]furan (14). According to the same procedure for the preparation of **5**, compound **14** was obtained from **10** (250 mg, 0.98 mmol) Raney nickel (W-2, 2.2 g) in ethanol (20 mL) in 78% yield (161 mg) as an oil. IR (neat) 3056, 2955, 2867, 1633, 1577, 1467, 1389, 1266, 1167, 1077 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.40 (d, 6H, $J=7.02\text{Hz}$), 2.82-3.38 (m, 1H), 6.84 (s, 1H), 7.24-8.16 (m, 6H); MS m/z 210 (M^+).

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