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단 신

알파-(메틸술피닐)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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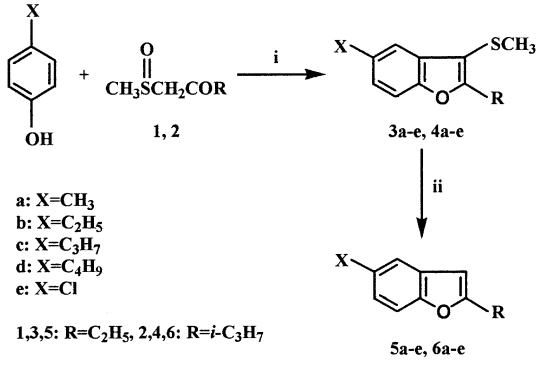
As a part of our continuing study concerning the carbon-carbon bond forming reactions utilizing 1-acyl-1-thiocarbocations, we have reported the Friedel-Crafts reaction of substituted phenols with 1-acyl-1-chlorosulfides leading to 2-methylbenzofuran derivatives,[1] 2-arylbenzofuran derivatives,[2] and 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 reported[5] in 68% yield.

The previous study on Pummerer reaction with α-(methylsulfinyl) acetone[4] 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, $ClCH_2CH_2Cl$, reflux, 1 h; (ii) Raney-Ni (W-2), EtOH, 60-65 °C, 1 h.

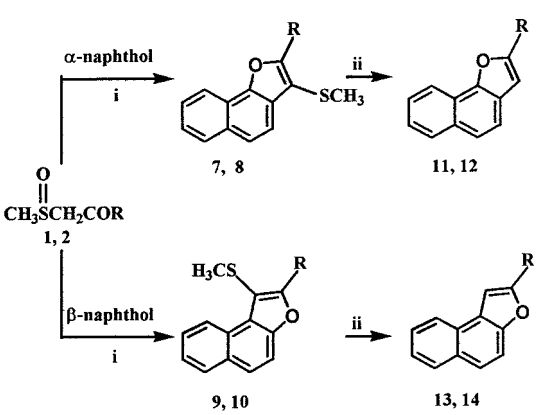
The reactions of other arenes generally took place smoothly in the presence of *p*-toluenesulfonic acid and gave 2-ethyl-3-(methylthio) benzofuran derivatives **3b-e** in satisfactory yields.

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 derivaives have so far been reported in the literature, the following methods being representative: 1) the reaction of 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 ditritlates with phenoxide anion.[9]



Scheme 2. Reagents and conditions: (i) *p*-TsOH, $ClCH_2CH_2Cl$, reflux, 1 h; (ii) Raney-Ni (W-2), EtOH, 60-65 °C, 1 h.

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. ^{1}H 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 $MgSO_4$, 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}; ^{1}H NMR ($CDCl_3$) δ1.10 (t, 3H, J=7.02Hz), 2.65 (q, 2H,

J=7.02Hz), 2.69 (s, 3H), 3.76 (d, 2H, J=1.20Hz); 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 throughly 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 (C=O), 1416, 1377, 1123, 1038 (S=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ1.16 (d, 6H, J=7.02Hz), 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 ($CDCl_3$) δ1.30 (t, 3H, J=7.50Hz), 2.30 (s, 3H), 2.46 (s, 3H), 2.93 (q, 2H, J=7.50Hz), 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 ($CDCl_3$) δ1.30 (t, 6H, J=7.62Hz), 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 ($CDCl_3$) δ0.95 (t, 3H, J=7.02Hz), 1.30 (t, 3H, J=7.62Hz), 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 ($CDCl_3$) δ1.30 (t, 3H, J=7.62Hz), 1.39 (s, 9H), 2.31 (s, 3H), 2.93 (q, 2H, J=7.62Hz), 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 ($ClCCl_3$) δ1.31 (t, 3H, J=7.62Hz), 2.30 (s, 3H), 2.94 (q, 2H, J=7.62Hz), 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 ($ClCCl_3$) δ1.34 (d, 6H, J=7.02Hz), 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 ($ClCCl_3$) δ1.22 (t, 3H, J=7.62Hz), 1.34 (d, 6H, J=7.02Hz), 2.31 (s, 3H), 2.76 (q, 2H, J=7.62Hz), 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 ($CDCl_3$) δ0.95 (t, 3H, J=7.02Hz), 1.33 (d, 6H, J=7.02Hz), 1.49-2.04 (m, 2H), 2.31 (s, 3H), 2.71 (t, 2H, J=6.48Hz), 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 ($ClCCl_3$) δ1.33 (d, 6H, J=7.02Hz), 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 ($ClCCl_3$) δ1.34 (d, 6H, J=7.02 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 1h. 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 ($ClCCl_3$) δ1.32 (t, 3H, J=7.62Hz), 2.41 (s, 3H), 2.79 (q, 2H, J=7.62Hz), 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 ($CDCl_3$) δ1.02-1.44 (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}; ^{1}H NMR ($CDCl_3$) δ0.94 (t, 3H, J=7.08Hz), 1.32 (t, 3H, J=7.08Hz), 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}; ^{1}H NMR ($CDCl_3$) δ1.31 (t, 3H, J=7.62Hz), 1.36 (s, 9H), 2.78 (q, 2H, J=7.62Hz), 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}; ^{1}H NMR ($CDCl_3$) δ1.32 (t, 3H, J=7.62Hz), 2.80 (q, 2H, J=7.62Hz), 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}; ^{1}H NMR ($CDCl_3$) δ1.33 (d, 6H, J=7.02Hz), 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}; ^{1}H NMR ($CDCl_3$) δ1.19 (t, 3H, J=7.02Hz), 1.33 (d, 6H, J=7.02Hz), 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}; ^{1}H NMR ($CDCl_3$) δ0.94 (t, 3H, J=7.08Hz), 1.33 (d, 6H, J=6.48Hz), 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}; ^{1}H NMR ($CDCl_3$) δ1.33 (d, 6H, J=7.02Hz), 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}; ^{1}H NMR ($CDCl_3$) δ1.33 (d, 6H, J=7.02Hz), 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}; ^{1}H NMR ($CDCl_3$) δ1.40 (t, 3H, J=7.62Hz), 2.35 (s, 3H), 3.06 (q, 2H, J=7.62Hz), 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}; ^{1}H NMR $CDCl_3$) 1.44 (d, 6H, J=7.02Hz), 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}; ^{1}H NMR ($CDCl_3$) 1.37 (t, 3H, J=7.62Hz), 2.37 (s, 3H), 3.07 (q, 2H, J=7.62Hz), 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}; ^{1}H NMR ($CDCl_3$) δ1.40 (d, 6H, J=7.02Hz), 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-Ethylnaphtho[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}; ^{1}H NMR ($CDCl_3$) δ1.39 (t, 3H, J=7.62Hz), 2.92 (q, 2H, J=7.62Hz), 6.50 (s, 1H), 7.24-8.36 (m, 6H); MS m/z 196 (M^+).

2-Isopropylnaphtho[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}; ^{1}H NMR ($CDCl_3$) δ1.42 (d, 6H, J=7.02Hz), 3.02-3.64 (m, 1H), 6.50 (s, 1H), 7.25-8.37 (m, 6H); MS

m/z 210 (M^+).

2-Ethylnaphtho[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 ($CDCl_3$) δ1.40 (t, 3H, J=7.62Hz), 2.91 (q, 2H, J=7.62Hz), 6.88 (s, 1H), 7.25-8.04 (m, 6H); MS m/z 196 (M^+).

2-Isopropylnaphtho[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 ($CDCl_3$) δ1.40 (d, 6H, J=7.02Hz), 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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