Supporting Information for

Zinc(II)-Catalyzed Grignard Addition to Ketones with RMgBr and RMgI

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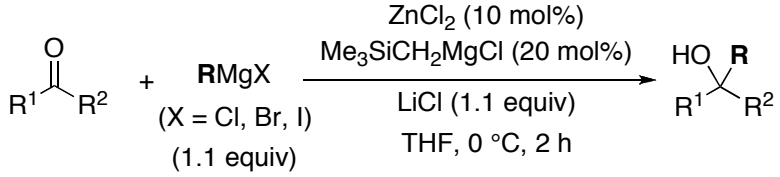
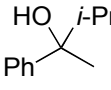
1. General Methods. ^{1}H NMR spectra were measured on a Varian Mercury-300 (300 MHz) spectrometer at ambient temperature. Data were recorded as follows: chemical shift in ppm from internal tetramethylsilane on the δ scale, multiplicity (s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet), coupling constant (Hz), integration, and assignment. ^{13}C NMR spectra were measured on Varian Mercury-300 (75 MHz) spectrometer. Chemical shifts were recorded in ppm from the solvent resonance employed as the internal standard (deuterochloroform at 77.1 ppm). Low and high resolution mass spectral analyses (LRMS and HRMS, FAB) were performed at Chemical Instrument Center, Nagoya University (JEOL JMS-700). For thin-layer chromatography (TLC) analysis throughout this work, Merck precoated TLC plates (silica gel 60GF254 0.25 mm) were used. The products were purified by neutral column chromatography on silica gel (Kanto Chemical Co., Inc. 37560). Visualization was accomplished by UV light (254 nm), anisaldehyde, $KMnO_4$ and phosphomolybdic acid. In experiments that required dry solvents, such as tetrahydrofuran and diethylether, were distilled in prior to use. Zinc chloride (Wako), Lithium chloride (Aldrich), (Trimethylsilyl)methylmagnesium chloride (1.0 *M* in Et_2O, Aldrich), Methylmagnesium iodide (3.0 *M* in Et_2O, Aldrich), Ethylmagnesium bromide (1.0 *M* in THF, Aldrich), Isopropylmagnesium chloride (2.0 *M* in THF, Aldrich), Isopropylmagnesium bromide (0.7 *M* in THF, Kanto Chemical Co., Inc.), 3-Butenylmagnesium bromide (0.5 *M* in THF, Aldrich), *n*-Hexylmagnesium bromide (2.0 *M* in Et_2O, Aldrich), Cyclohexylmagnesium bromide (1.0 *M* in THF, TCI), 4-Fluorophenylmagnesium bromide (1.0 *M* in THF, Aldrich), *n*-Octylmagnesium bromide (2.0 *M* in Et_2O, Aldrich), were used. 1-Naphthylmagnesium bromide (1.0 *M* in THF) was prepared from 1-bromonaphthalene and magnesium turnings (Wako). All the Grignard reagents were titrated prior to use against a solution of 1,10-phenanthoroline/*n*-BuLi/*s*-BuOH in benzene.

2. General procedure for $ZnCl_2$–$TMSCH_2MgCl$–LiCl-catalyzed Grignard reaction of ketones (Tables 1 and 2).



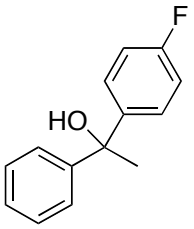
To a pyrex Schlenk tube, $ZnCl_2$ (40.8 mg, 0.30 mmol) was added and melt-dried by a heat gun under reduced pressure (<5 Torr) within 5 min. LiCl (139.9 mg, 3.3 mmol) was added to the pyrex Schlenk tube containing $ZnCl_2$, and again the mixture was heated by a heat gun under reduced pressure (<5 Torr) briefly (within 3–5 min). To the mixture, (trimethylsilyl)methylmagnesium chloride (1.0 *M* in Et_2O, 0.60 mL, 0.60 mmol) was added at room temperature. This mixture was stirred at that temperature for 15 min. RMgX (0.5–2.0 *M* in THF or Et_2O, 3.3 mmol) was added, and the solution* was stirred at that temperature for 45 min (* If a Grignard reagent is >1 *M*, the solution of the Grignard reagent is diluted with THF to 1 *M* in situ. If a Grignard reagent is <1 *M*, the solution is used as it is.). Then, the solution was cooled at 0 °C, and ketone (**1**) (3.0 mmol) was added over 1 h by a syringe pump (If a ketone is solid state, a THF solution (ca. 2 mL) is prepared in advance.). The mixture was stirred at 0 °C for 2 h, and the reaction was monitored by TLC. The resulting mixture was quenched by saturated aqueous NH_4Cl (10 mL), extracted with AcOEt (10 mL × 3), and washed by brine (10 mL). The combined extracts were dried over $MgSO_4$. The organic phase was concentrated under reduced pressure and the resultant residue was purified by neutral silica gel column chromatography (eluent: hexane/EtOAc), to give the desired product.

3. Products.

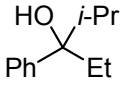


3-Methyl-2-phenylbutan-2-ol (2a, Table 1):[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.80 (d, *J* = 6.9 Hz, 3H), 0.89 (d, *J* = 6.9 Hz, 3H), 1.53 (s, 3H), 1.56 (s, 1H), 2.02 (septet, *J* = 6.9 Hz, 1H), 7.20-7.45 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 17.2, 17.4, 26.7, 38.6, 77.8, 125.2, 126.4, 127.8, 147.8. HRMS (FAB+) calcd for $C_{11}H_{15}$ $[M–OH]^+$ 147.1174, found 147.1170.

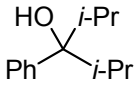
2-Phenylpropan-2-ol (Entry 1 in Table 2):[2] ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.58 (s, 3H), 1.76 (s, 1H), 7.24 (t, *J* = 7.3 Hz, 1H), 7.34 (t, *J* = 7.3 Hz, 2H), 7.49 (d, *J* = 7.3 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 31.7, 72.5, 124.4, 126.7, 128.2, 149.1. HRMS (FAB+) calcd for $C_9H_{12}NaO$ $[M+Na]^+$ 159.0786, found 159.0789.



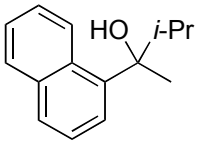
1-(4-Fluorophenyl)-1-phenylethanol (Entry 2 in Table 2):[3] ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.93 (s, 3H), 2.17 (s, 1H), 6.98 (t, J = 8.7 Hz, 2H), 7.20-7.43 (m, 7H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 31.0, 76.7, 114.9 (d, *J* = 20.9 Hz), 125.8, 127.1, 127.6 (d, *J* = 7.6 Hz), 128.3, 143.8, 147.7, 161.7 (d, *J* = 244 Hz). HRMS (FAB+) calcd for $C_{14}H_{12}F$ $[M–OH]^+$ 199.0923, found 199.0925.



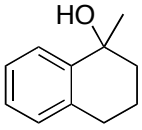
2-Methyl-3-phenyl-3-pentanol (Entry 3 in Table 2):[1,2] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.68 (t, *J* = 6.9 Hz, 3H), 0.72 (d, *J* = 6.9 Hz, 3H), 0.94 (d, *J* = 6.9 Hz, 3H), 1.58 (s, 1H), 1.89 (q, *J* = 6.9 Hz, 2H), 2.05 (septet, *J* = 6.9 Hz, 1H), 7.18-7.40 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 7.9, 16.6, 17.5, 32.0, 37.5, 79.3, 125.9, 126.1, 127.7, 145.0. HRMS (FAB+) calcd for $C_{12}H_{17}$ $[M–OH]^+$ 161.1330, found 161.1326.



2,4-Dimethyl-3-phenyl-3-pentanol (Entry 4 in Table 2):[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.76 (d, *J* = 6.6 Hz, 6H), 0.84 (d, *J* = 6.6 Hz, 6H), 1.50 (s, 1H), 2.31 (m, 2H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.37 (t, *J* = 7.5 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.5, 17.4, 33.7, 80.9, 126.1, 126.6, 127.2, 142.8. HRMS (FAB+) calcd for $C_{13}H_{19}$ $[M–OH]^+$ 175.1487, found 175.1487.

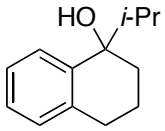


3-Methyl-2-(naphthalen-1-yl)butan-2-ol (Entry 5 in Table 2):[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.78 (d, *J* = 6.9 Hz, 3H), 0.99 (d, *J* = 6.9 Hz, 3H), 1.74 (s, 3H), 1.93 (bs, 1H), 2.81 (m, 1H), 7.00-8.05 (m, 6H), 8.76 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 17.0, 18.2, 25.1, 36.1, 78.9, 124.2, 124.5, 124.9, 127.1, 128.3, 129.1, 130.8, 135.0, 143.2. HRMS (FAB+) calcd for $C_{15}H_{17}$ $[M–OH]^+$ 197.1330, found 197.1333.

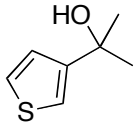


1-Methyl-1,2,3,4-tetrahydronaphthalen-1-ol (Entry 6 in Table 2):[4] ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.56 (s, 3H), 1.74 (s, 1H), 1.77-2.24 (m, 4H), 2.70-2.88 (m, 2H), 7.07 (d, *J* = 7.5 Hz, 1H), 7.17 (t, *J* = 7.5 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.59 (d, *J* = 7.5 Hz, 1H). ^{13}C NMR (75

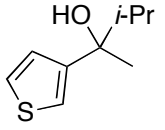
MHz, $CDCl_3$) δ 20.5, 30.0, 30.8, 39.9, 70.6, 126.3, 126.4, 127.1, 128.9, 136.3, 142.9. HRMS (FAB+) calcd for $C_{11}H_{13}$ $[M–OH]^+$ 145.1017, found 145.1020.



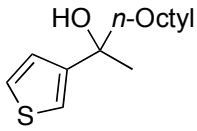
1-Isopropyl-1,2,3,4-tetrahydro-1-naphthol (Entry 7 in Table 2):[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.65 (d, *J* = 6.9 Hz, 3H), 1.09 (d, *J* = 6.9 Hz, 3H), 1.68-1.94 (m, 4H), 2.40 (septet, *J* = 6.9 Hz, 1H), 2.60-2.84 (m, 2H), 7.09 (d, *J* = 7.2 Hz, 1H), 7.12-7.26 (m, 2H), 7.52 (d, *J* = 7.2 Hz, 1H). HRMS (FAB+) calcd for $C_{13}H_{17}$ $[M–OH]^+$ 173.1330, found 173.1335.



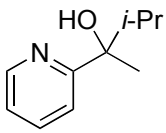
2-(Thiophen-3-yl)propan-2-ol (Entry 8 in Table 2): ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.59 (s, 6H), 1.82 (s, 1H), 7.13 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.18 (dd, *J* = 3.0, 1.2 Hz, 1H), 7.28 (dd, *J* = 5.0, 3.0 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 31.3, 71.0, 118.8, 125.7, 125.8, 151.0. HRMS (FAB+) calcd for C_7H_9S $[M–OH]^+$ 125.0425, found 125.0424.



3-Methyl-2-(3-thienyl)-2-butanol (Entry 9 in Table 2):[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.86 (d, *J* = 6.9 Hz, 3H), 0.87 (d, *J* = 6.9 Hz, 3H), 1.51 (s, 3H), 1.71 (s, 1H), 2.00 (septet, *J* = 6.9 Hz, 1H), 7.04 (dd, *J* = 5.1, 1.5 Hz, 1H), 7.13 (d, *J* = 3.3, 1.5 Hz, 1H), 7.26 (d, *J* = 5.1, 3.3 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 17.3, 17.5, 26.3, 38.6, 76.1, 119.8, 125.2, 126.1, 149.6. HRMS (FAB+) calcd for $C_9H_{13}S$ $[M–OH]^+$ 153.0738, found 153.0734

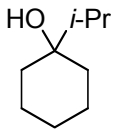


2-(Thiophen-3-yl)decan-2-ol (Entry 10 in Table 2): ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.86 (t, *J* = 6.9 Hz, 3H), 1.23 (m, 12H), 1.54 (s, 3H), 1.77 (m, 2H), 1.79 (s, 1H), 7.04 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.14 (dd, *J* = 3.3, 1.2 Hz, 1H), 7.04 (dd, *J* = 5.1, 3.3 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.1, 22.7, 24.1, 29.3, 29.5, 29.6, 29.9, 31.9, 43.9, 73.7, 119.3, 125.7, 125.8, 150.1. HRMS (FAB+) calcd for $C_{14}H_{23}S$ $[M–OH]^+$ 223.1520, found 223.1518.

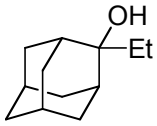


3-Methyl-2-(pyridin-2-yl)butan-2-ol (Entry 11 in Table 2):[5] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.65 (d, *J* = 6.9 Hz, 3H), 0.99 (d, *J* = 6.9 Hz, 3H), 1.49 (s, 3H), 1.97 (septet, *J* = 6.9 Hz, 1H), 5.19 (s, 1H), 7.19 (dd, *J* = 6.0, 4.6 Hz, 1H), 7.29 (d, *J* = 7.8 Hz, 1H), 7.69 (dd, *J* = 7.8, 6.0 Hz,

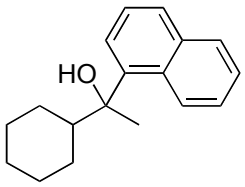
1H), 7.19 (d, J = 4.6 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.9, 17.1, 26.2, 38.5, 75.3, 119.5, 121.6, 136.6, 146.9, 165.0. HRMS (FAB+) calcd for $C_{10}H_{14}N$ $[M–OH]^+$ 148.1126, found 148.1125.



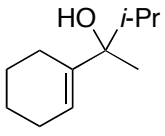
1-Isopropyl-1-cyclohexanol (Entry 12 in Table 2):[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.90 (d, J = 6.9 Hz, 6H), 1.07 (s, 1H), 1.20-1.65 (m, 11H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.7, 22.0, 26.0, 34.2, 37.6, 73.1. HRMS (FAB+) calcd for C_9H_{17} $[M–OH]^+$ 125.1330, found 125.1327.



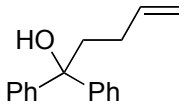
2-Ethtyl-2-adamantanol (Entry 13 in Table 2):[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.89 (t, J = 7.8 Hz, 3H), 1.32 (s, 1H), 1.69 (d, J = 7.5 Hz, 2H), 1.50-2.25 (m, 14H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 6.3, 27.5, 30.6, 33.2, 34.7, 36.8, 38.5, 39.4, 74.9. HRMS (FAB+) calcd for $C_{12}H_{19}$ $[M–OH]^+$ 163.1487, found 163.1485.



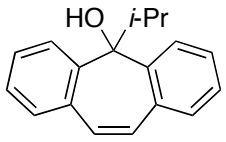
1-Cyclohexyl-1-(naphthalen-1-yl)ethanol (Entry 14 in Table 2):[6] ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.00-1.90 (m, 10H), 1.75 (s, 3H), 1.94 (bs, 1H), 2.37 (m, 1H), 7.39 (t, J = 7.8 Hz, 1H), 7.42-7.50 (m, 2H), 7.53 (d, J = 6.3 Hz, 1H), 7.75 (d, J = 8.1 Hz, 1H), 7.85 (m, 1H), 8.77 (d, J = 7.2 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 26.2, 26.5, 26.7, 26.8, 27.2, 28.3, 46.8, 79.0, 124.5, 124.6, 125.0, 125.1, 127.2, 128.3, 129.2, 130.9, 134.9, 143.1. HRMS (FAB+) calcd for $C_{18}H_{21}$ $[M–OH]^+$ 237.1643, found 237.1641.



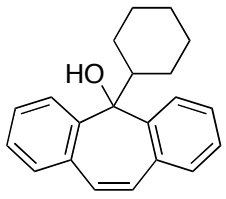
2-Cyclohexenyl-3-methylbutan-2-ol (Entry 15 in Table 2): ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.82 (d, J = 6.9 Hz, 3H), 0.87 (d, J = 6.9 Hz, 3 H), 1.21 (s, 3H), 1.49-1.66 (m, 4H), 1.83 (septet, J = 6.9 Hz, 1H), 1.95 (m, 2H), 2.03-2.10 (m, 3H), 5.69 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.8, 22.4, 23.1, 23.8, 24.7, 25.1, 34.0, 77.0, 119.7, 142.7. HRMS (FAB+) calcd for $C_{11}H_{19}$ $[M–OH]^+$ 151.1487, found 151.1486.



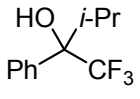
1,1-Diphenylpent-4-en-1-ol (Entry 16 in Table 2):[7] ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.02-2.10 (m, 2H), 2.17 (s, 1H), 2.35-2.43 (m, 2H), 4.95 (d, J = 9.9 Hz, 1H), 5.00 (d, J = 17.4 Hz, 1H), 5.85 (ddt, J = 17.4, 9.9, 6.9 Hz, 1H), 7.22 (t, J = 7.2 Hz, 2H), 7.31 (t, J = 7.2 Hz, 4H), 7.41 (t, J = 7.2 Hz, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 28.5, 41.2, 78.5, 114.9, 126.2, 127.1, 128.4, 138.9, 147.1. HRMS (FAB+) calcd for $C_{17}H_{17}$ $[M–OH]^+$ 221.1330, found 221.1329.



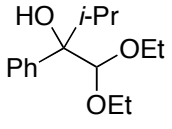
5-Isopropyl-5H-dibenzo[a,d]cyclohepten-5-ol (Entry 17 in Table 2):[8] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.47 (d, J = 6.9 Hz, 6H), 2.31 (s, 1H), 2.88 (septet, J = 6.9 Hz, 1H), 6.92 (s, 2H), 7.25 (t, J = 7.5 Hz, 2H), 7.32 (d, J = 7.5 Hz, 2H), 7.40 (t, J = 7.5 Hz, 2H), 7.91 (t, J = 7.5 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.3, 28.0, 79.2, 124.7, 126.3, 128.4, 129.4, 131.4, 132.3, 142.5. HRMS (FAB+) calcd for $C_{18}H_{17}$ $[M–OH]^+$ 233.1330, found 233.1329.



5-Cyclohexyl-5H-dibenzo[a,d]cyclohepten-5-ol (Entry 18 and 23 in Table 2):[9] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.76-1.06 (m, 10H), 2.38 (s, 1H), 2.52 (m, 1H), 6.95 (s, 2H), 7.26 (td, J = 7.5, 1.2 Hz, 2H), 7.32 (dd, J = 7.5, 1.5 Hz, 2H), 7.41 (td, J = 8.1, 1.5 Hz, 2H), 7.92 (dd, J = 8.1, 1.2 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 26.3, 26.5, 26.8, 38.3, 79.2, 124.8, 126.2, 128.3, 129.4, 131.4, 132.3, 142.2. HRMS (FAB+) calcd for $C_{21}H_{21}$ $[M–OH]^+$ 273.1643, found 273.1642.

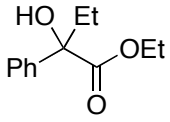


1,1,1-Trifluoro-3-methyl-2-phenylbutan-2-ol (Entry 19 in Table 2):[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.71 (d, J = 6.9 Hz, 3H), 1.11 (d, J = 6.9 Hz, 3H), 2.39 (bs, 1H), 2.51 (septet, J = 6.9 Hz, 1H), 7.30-7.45 (m, 3H), 7.50-7.58 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.7, 17.2, 33.7, 79.6 (q, J = 26.9 Hz), 125.6, 126.0 (q, J = 285.5), 128.1, 128.2, 137.8. ^{19}F NMR (282 MHz, $CDCl_3$) δ –74.2. HRMS (FAB+) calcd for $C_{11}H_{13}F_3O$ $[M]^+$ 218.0918, found 218.0914.



1,1-Diethoxy-3-methyl-2-phenylbutan-2-ol (Entry 20 in Table 2): ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.70 (d, J = 6.6 Hz, 3H), 0.94 (d, J = 6.6 Hz, 3H), 1.10 (t, J = 6.9 Hz, 3H), 1.25 (t,

J = 6.9 Hz, 3H), 2.36 (septet, *J* = 6.9 Hz, 1H), 2.69 (s, 1H), 3.38-3.60 (m, 2H), 3.60-3.90 (m, 2H), 4.68 (s, 1H), 7.18-7.34 (m, 3H), 7.47-7.53 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 15.3, 15.4, 16.9, 17.5, 33.7, 65.3, 66.0, 79.6, 106.6, 126.2, 126.3, 127.3, 142.7 HRMS (FAB+) calcd for $C_{15}H_{23}O_2$ $[M–OH]^+$ 235.1698, found 235.1702.



Ethyl 2-hydroxy-2-phenylbutanoate (Entry 21 in Table 2):[1] ^{1}H NMR (300 M127.8Hz, $CDCl_3$) δ 0.92 (t, *J* = 7.2 Hz, 3H), 1.28 (t, *J* = 7.2 Hz, 3H), 2.01 (dq, *J* = 14.7, 7.2 Hz, 1H), 2.24 (dq, *J* = 14.7, 7.2 Hz, 1H), 3.77 (s, 1H), 4.19 (dq, *J* = 10.8, 7.2 Hz, 1H), 4.29 (dq, *J* = 10.8, 7.2 Hz, 1H), 7.24-7.40 (m, 3H), 7.60 (d, *J* = 7.8 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 8.0, 14.1, 32.7, 62.4, 78.6, 125.6, 127.6, 128.2, 141.9, 175.4. HRMS (FAB+) calcd for $C_{12}H_{15}O_2$ $[M–OH]^+$ 191.1072, found 191.1072.

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