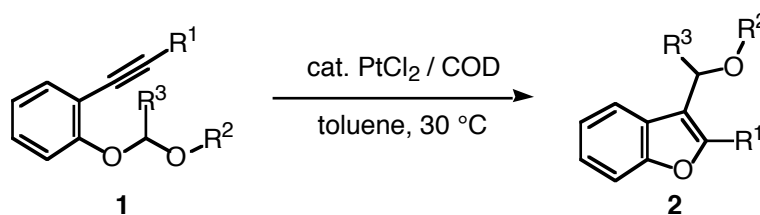


**Synthesis of 2,3-Disubstituted Benzofurans by
Platinum-Olefin-Catalyzed Carboalkoxylation of *ortho*-Alkynylphenyl
Acetals**

Itaru Nakamura, Yuya Mizushima, Yoshinori Yamamoto*

Department of Chemistry, Graduate School of Science, Tohoku University, Sendai 980-8578 Japan

(Supporting Information)

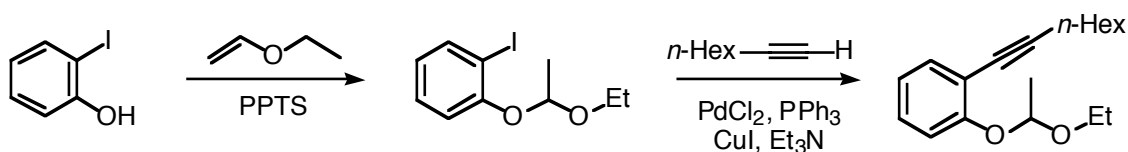


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General. Spectroscopic measurements were carried out with following instruments: JEOL JNM AL 400 (^1H and ^{13}C NMR), JASCO FT/IR-460*Plus* (FT-IR), HITACHI M-2500s (HRMS). PtCl_2 was purchased from WAKO Co. Ltd. The starting materials **1** were prepared from 2-iodophenol through acetalization and Sonogashira coupling. Chromatography was performed with Kanto Chemical silica gel 60N (spherical, neutral, 100-210 μm) or Fuji Silysia silica gel BW-300 (200-400 mesh).

Preparation of Starting Material 1a.



(a) To a mixture of *o*-iodophenol (2.2 g, 10 mmol) and pyridinium *p*-toluenesulfonate (251 mg, 1 mmol) in CH_2Cl_2 (20 ml) was added vinyl ethyl ether (2.9 ml, 30 mmol) at 0 $^\circ\text{C}$. The reaction mixture was stirred at room temperature for 16 hours. The solvent was removed in vacuo and the residue was purified by silica gel column chromatography using CH_2Cl_2 / Hexane (2/1) as eluent to obtain acetaldehyde *o*-iodophenyl ethyl acetal in 80% yield.

(b) To a mixture of PdCl_2 (35 mg, 0.2 mmol), CuI (55 mg, 0.288 mmol), PPh_3 (105 mg, 0.4 mmol), and Et_3N (3.3 ml, 24 mmol) in 10 ml of CH_3CN were added iodophenyl ethyl acetal (8 mmol) in 6 ml of CH_3CN and 1-octyne (12 mmol). The reaction mixture was stirred at room temperature for 1 hour. The reaction mixture was diluted with ether. The organic layer was washed by saturated ammonium chloride aqueous solution and brine and dried over Na_2SO_4 . After evaporation, the crude product was purified by silica gel column chromatography using EtOAc / Hexane (1/20) as eluent to obtain **1a** in 94% yield.

General Procedure of Carboalkoxylation of 1. To PtCl_2 (1.6 mg, 0.006 mmol) in 0.5 ml of toluene and 1,5-Cyclooctadiene (2.9 μL , 0.024 mmol) was added the acetal **1** (0.3 mmol) in 0.7 ml of toluene under Ar atmosphere in a pressure vial. After heating at 30 $^\circ\text{C}$ for 1-24 hours, the reaction mixture was filtered through a short silica gel column using ethyl acetate as an eluent. Separation by silica gel column chromatography using hexane/ EtOAc (30/1) as an eluent afforded the products **2**.

3-(1-Ethoxyethyl)-2-hexylbenzofuran (2a). IR (neat) 3055, 2975-2860, 1622, 1370, 1101, 746 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.16 (t, $J = 7.2$ Hz, 3H), 1.26-1.40 (m, 6H), 1.58 (d, $J = 6.8$ Hz, 3H), 1.72 (quint, $J = 7.3$ Hz, 2H), 2.74 (td, $J = 7.3, 3.1$ Hz, 2H), 3.30-3.41 (m, 2H), 4.66 (q, $J = 6.8$ Hz, 1H), 7.15-7.22 (m, 2H), 7.39 (dd, $J = 7.4, 1.4$ Hz, 1H), 7.71 (d, $J = 7.2$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.08, 15.43, 22.42, 22.60, 26.60, 28.45, 29.02, 31.60, 63.58, 70.17, 110.59, 116.03, 120.38, 122.06, 123.09, 127.47, 154.01, 155.01. Anal Calcd for $\text{C}_{18}\text{H}_{26}\text{O}_2$ (274.40): C, 78.79; H, 9.55. Found: C, 79.05; H, 9.73. HRMS (ESI) Calcd for $\text{C}_{18}\text{H}_{26}\text{O}_2$ ($\text{M}+\text{Na}$) $^+$: m/z 297.1825. Found: m/z 297.1825.

2-(4-Chlorobutyl)-3-(1-ethoxyethyl)benzofuran (2b). IR (neat) 3055, 2975-2867, 1622, 1455, 1370, 1089, 1012, 940, 849, 748 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 1.17 (t, $J = 7.0$ Hz, 3H), 1.58 (d, $J = 6.8$ Hz, 3H), 1.81-1.93 (m, 4H), 2.75-2.85 (m, 2H), 3.33-3.40 (m, 2H), 3.56 (t, $J = 6.2$ Hz, 2H), 4.66 (q, $J = 6.8$ Hz, 1H), 7.17-7.25 (m, 2H), 7.39 (dd, $J = 7.0, 1.0$ Hz, 1H), 7.71 (dd, $J = 7.0, 1.8$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 15.38, 22.40, 25.66, 25.76, 31.96, 44.51, 63.63, 70.06, 110.60, 116.52, 120.34, 122.13, 123.27, 127.28, 153.82, 153.94. Anal Calcd for $\text{C}_{16}\text{H}_{21}\text{ClO}_2$ (280.79): C, 68.44; H, 7.54; Cl, 12.63. Found: C, 68.47; H, 7.67; Cl, 12.52. HRMS (ESI) Calcd for $\text{C}_{16}\text{H}_{21}\text{ClO}_2$ ($\text{M}+\text{Na}$) $^+$: m/z 303.1122. Found: m/z 303.1123.

2-Cyclohexyl-3-(1-ethoxyethyl)benzofuran (2c). IR (neat) 3087-2857, 1963, 1786, 1621, 1446, 943, 845, 756 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 1.16 (t, $J = 7.2$ Hz, 3H), 1.29-1.44 (m, 3H), 1.58 (d, $J = 6.4$ Hz, 3H), 1.77-1.89 (m, 7H), 2.80-2.88 (m, 1H), 3.30-3.39 (m, 2H), 4.71 (q, $J = 6.4$ Hz, 1H), 7.14-7.23 (m, 2H), 7.39 (dd, $J = 7.2, 1.2$ Hz, 1H), 7.71 (d, $J = 6.9$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 15.38, 22.60, 25.81, 26.37, 26.38, 31.44, 31.66, 36.45, 63.51, 69.94, 110.58, 114.35, 120.41, 121.93, 122.93, 127.44, 153.78, 158.69. Anal Calcd for $\text{C}_{18}\text{H}_{24}\text{O}_2$ (272.38): C, 79.37; H, 8.88. Found: C, 79.11; H, 8.91. HRMS (ESI) Calcd for $\text{C}_{18}\text{H}_{24}\text{O}_2$ ($\text{M}+\text{Na}$) $^+$: m/z 295.1669. Found: m/z 295.1669.

3-(1-Ethoxyethyl)-2-(4-methoxyphenyl)benzofuran (2e). IR (neat) 3226, 3049-2843, 2042, 1604, 1508, 1449, 1257, 1176-1087, 1026, 937, 833, 749 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 1.12 (t, $J = 7.0$ Hz, 3H), 1.70 (d, $J = 6.4$ Hz, 3H), 3.27-3.39 (m, 2H), 3.87 (s, 3H), 4.97 (q, $J = 6.4$ Hz, 1H), 7.00-7.03 (m, 2H), 7.22 (ddd, $J = 7.4, 7.4, 1.1$ Hz, 1H), 7.28 (ddd, $J = 8.0, 7.2, 1.6$ Hz, 1H), 7.48 (d, $J = 8.0$ Hz, 1H), 7.64-7.68 (m, 2H), 7.87 (dd, $J = 7.8, 1.0$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 15.43, 22.01, 55.36, 63.70, 70.27, 110.86, 114.06, 116.74, 121.26, 122.39, 123.30, 123.94, 128.18, 129.05, 151.59, 153.94, 159.77. Anal Calcd for $\text{C}_{19}\text{H}_{20}\text{O}_3$ (296.36): C, 77.00; H, 6.80. Found: C, 76.83; H, 6.87. HRMS (ESI) Calcd for $\text{C}_{19}\text{H}_{20}\text{O}_3$ ($\text{M}+\text{Na}$) $^+$: m/z 319.1305. Found:

m/z 319.1305.

3-(1-Ethoxyethyl)-2-phenylbenzofuran (2f). IR (neat) 3057-2870, 1590, 1493, 1454, 1371, 1256, 1210, 1125-1062, 936, 746 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 1.12 (t, $J = 7.2$ Hz, 3H), 1.72 (d, $J = 6.4$ Hz, 3H), 3.28-3.39 (m, 2H), 5.01 (q, $J = 6.4$ Hz, 1H), 7.24 (ddd, $J = 7.5, 7.5, 1.1$ Hz, 1H), 7.30 (ddd, $J = 7.5, 7.5, 1.1$ Hz, 1H), 7.37-7.41 (m, 1H), 7.46-7.51 (m, 3H), 7.72 (dd, $J = 8.6, 1.4$ Hz, 1H), 7.91 (d, $J = 7.8$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 15.34, 21.99, 63.70, 70.20, 110.92, 118.07, 121.51, 122.41, 124.45, 127.55, 128.41, 128.49, 130.61, 151.32, 154.06. Anal Calcd for $\text{C}_{18}\text{H}_{18}\text{O}_2$ (266.33): C, 81.17; H, 6.81. Found: C, 81.25; H, 6.94. HRMS (ESI) Calcd for $\text{C}_{18}\text{H}_{18}\text{O}_2$ ($\text{M}+\text{Na}$) $^+$: m/z 289.1199. Found: m/z 289.1199

3-(1-Ethoxyethyl)-2-(4-trifluoromethylphenyl)benzofuran (2g). IR (neat) 3059-2883, 1625, 1448, 1408, 1322, 1164-1070, 936, 847, 755 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 1.13 (t, $J = 7.2$ Hz, 3H), 1.72 (d, $J = 6.6$ Hz, 3H), 3.30 (m, 2H), 5.02 (q, $J = 6.6$ Hz, 1H), 7.25-7.29 (m, 1H), 7.35 (ddd, $J = 8.0, 7.2, 1.2$ Hz, 1H), 7.52 (d, $J = 7.6$ Hz, 1H), 7.73-7.75 (m, 2H), 7.87-7.93 (m, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ Anal Calcd for $\text{C}_{19}\text{H}_{17}\text{F}_3\text{O}_2$ (334.33): C, 68.26; H, 5.13; F, 17.05. Found: C, 68.19; H, 5.17; F, 16.88. HRMS (ESI) Calcd for $\text{C}_{19}\text{H}_{17}\text{F}_3\text{O}_2$ ($\text{M}+\text{Na}$) $^+$: m/z 357.1073. Found: m/z 357.1074.

3-Methoxymethyl-2-propylbenzofuran (2h). IR (neat) 3056, 2962-2816, 1627, 1455, 1174, 1094, 910, 747 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 0.98 (t, $J = 7.4$ Hz, 3H), 1.77 (sext, $J = 7.4$ Hz, 2H), 3.36 (s, 3H), 4.56 (s, 2H), 7.18-7.24 (m, 2H), 7.37-7.41 (m, 1H), 7.57-7.59 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 13.83, 21.75, 28.46, 57.69, 64.59, 110.60, 111.56, 119.28, 122.38, 123.36, 128.83, 153.89, 157.01. Anal Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_2$ (204.26): C, 76.44; H, 7.90. Found: C, 76.16; H, 7.92. HRMS (ESI) Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_2$ ($\text{M}+\text{Na}$) $^+$: m/z 227.1043. Found: m/z 227.1044.

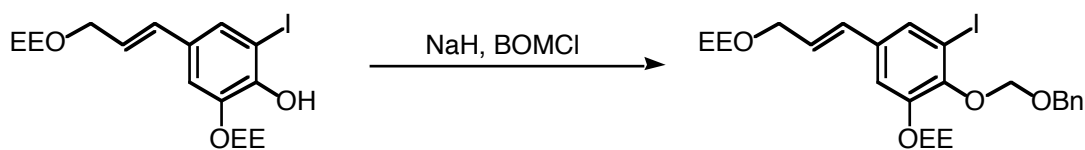
3-Methoxymethyl-2-phenylbenzofuran (2i). IR (neat) 3061-2817, 1592, 1455, 1255, 1190, 1088, 949, 745 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 3.47 (s, 3H), 4.74 (s, 2H), 7.25-7.33 (m, 2H), 7.41 (tt, $J = 7.2, 1.5$ Hz, 1H), 7.47-7.53 (m, 3H), 7.69-7.71 (m, 1H), 7.83-7.86 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 57.94, 64.60, 110.99, 112.43, 119.63, 122.74, 124.39, 127.31, 128.57, 128.64, 129.75, 130.21, 153.75, 153.94. Anal Calcd for $\text{C}_{16}\text{H}_{14}\text{O}_2$ (238.28): C, 80.65; H, 5.92. Found: C, 80.42; H, 6.18. HRMS (ESI) Calcd for $\text{C}_{16}\text{H}_{14}\text{O}_2$ ($\text{M}+\text{Na}$) $^+$: m/z 261.0886. Found: m/z 261.0885.

3-Benzyloxymethyl-2-propylbenzofuran (2j). IR (neat) 3087-2871, 1626, 1455, 1175, 1072, 1028, 746 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 0.96 (t, $J = 7.6$ Hz, 3H), 1.75 (sext, $J = 7.6$ Hz, 2H), 2.73 (t, $J = 7.6$ Hz, 2H), 4.55 (s, 2H), 4.64 (s, 2H), 7.21-7.24 (m, 2H), 7.28-7.33 (m, 1H), 7.34-7.36 (m, 4H), 7.41 (dd, $J = 6.4, 2.4$ Hz, 1H), 7.57-7.60 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 13.84, 21.75, 28.49, 62.13, 71.74, 110.63, 111.61, 119.38, 122.41, 123.39, 127.59, 127.79, 128.34, 128.86, 138.18, 153.94, 157.07. Anal Calcd for $\text{C}_{19}\text{H}_{20}\text{O}_2$ (280.36): C, 81.40; H, 7.19. Found: C, 81.60; H, 7.15. HRMS (ESI) Calcd for $\text{C}_{19}\text{H}_{20}\text{O}_2$ ($\text{M}+\text{Na}$) $^+$: m/z 303.1356. Found: m/z 303.1358.

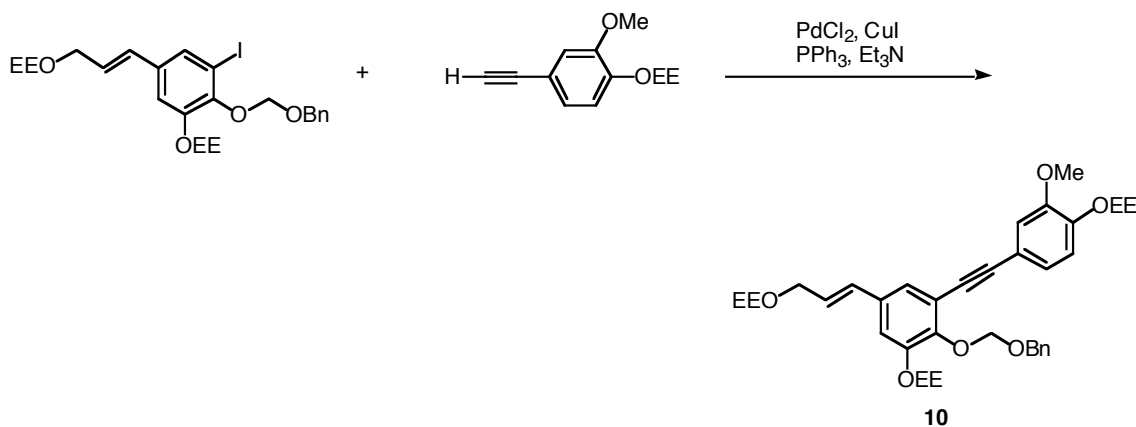
3-Benzyloxymethyl-2-phenylbenzofuran (2k). IR (neat) 3084-3033, 2925-2858, 1591, 1496, 1454, 1352, 1252, 1202, 1065, 744 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 4.64 (s, 2H), 4.82 (s, 2H), 7.23-7.41 (m, 8H), 7.43-7.47 (m, 2H), 7.51 (dd, $J = 8.2, 0.6$ Hz, 1H), 7.64 (d, $J = 8.0$ Hz, 1H), 7.82-7.84 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 62.19, 72.20, 111.06, 112.50, 119.77, 122.82, 124.48, 127.38, 127.70, 128.02, 128.36, 128.62, 128.69, 129.82, 130.26, 137.94, 153.86, 154.04. Anal Calcd for $\text{C}_{22}\text{H}_{18}\text{O}_2$ (314.38): C, 84.05; H, 5.77. Found: C, 84.19; H, 6.00. HRMS (ESI) Calcd for $\text{C}_{22}\text{H}_{18}\text{O}_2$ ($\text{M}+\text{Na}$) $^+$: m/z 337.1199. Found: m/z 337.1198.

***tert*-Butyldimethyl-(2-phenylbenzofuran-3-ylmethoxy)silane (2l).** IR (neat) 3062-2854, 1876, 1757, 1594, 1455, 1362, 850, 764 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 0.12 (s, 6H), 0.93 (s, 9H), 4.97 (s, 2H), 7.24-7.32 (m, 2H), 7.38-7.42 (m, 1H), 7.46-7.52 (m, 3H), 7.69 (d, $J = 7.9$ Hz, 1H), 7.82 (d, $J = 7.2$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ -5.08, 18.45, 25.99, 56.10, 111.05, 115.21, 119.95, 122.58, 124.29, 127.38, 128.54, 129.56, 130.51, 152.91, 153.89. Anal Calcd for $\text{C}_{21}\text{H}_{26}\text{O}_2\text{Si}$ (338.52): C, 74.51; H, 7.74. Found: C, 74.59; H, 7.80. HRMS (ESI) Calcd for $\text{C}_{21}\text{H}_{26}\text{O}_2\text{Si}$ ($\text{M}+\text{Na}$) $^+$: m/z 361.1594. Found: m/z 361.1596.

The *ortho*-alkynylphenyl benzyl acetal (**10**).

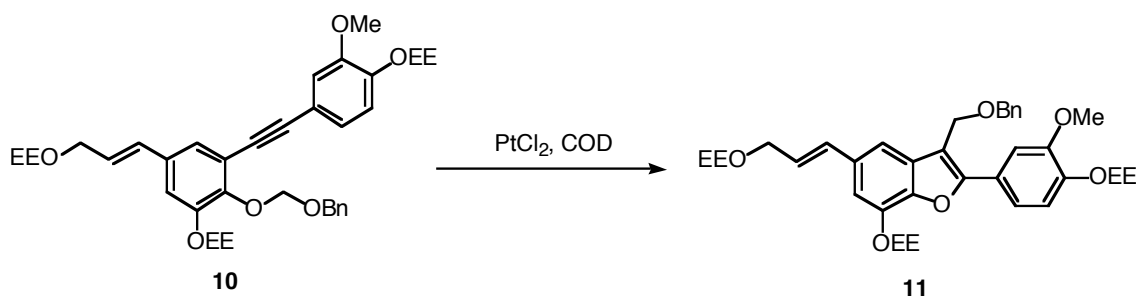


(a) To a THF solution (2.5 ml) of NaH was added **9** (1.0 mmol) in THF (0.5 ml) at 0 °C. After 1 hour, BOMCl (1.5 ml) was added to the reaction mixture at 0 °C. After stirring at room temperature for 5 hours, the reaction was quenched with Et₃N (0.2 ml). Water was added to the reaction mixture and extracted with ether. The organic phase was washed with brine and dried over Na₂SO₄. Purification by silica gel column chromatography using hexane/ethyl acetate (2/1) gave the corresponding BOM ether quantitatively. IR (neat) 2977-2934, 1737, 1572, 1509 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 1.13-1.24 (m, 6 H), 1.36 (d, *J* = 5.6 Hz, 3H), 1.48 (d, *J* = 5.2 Hz, 3H), 3.50-3.54 (m, 2H), 3.65-3.75 (m, 2H), 4.15 (dd, *J* = 4.8, 6.0 Hz, 1H), 4.24 (m, 1H), 4.79 (q, *J* = 5.2 Hz, 1H), 4.96 (s, 2H), 5.40 (s, 2H), 5.36 (q, *J* = 5.6 Hz), 6.18 (m, 1H), 6.46 (d, *J* = 15.6 Hz), 7.11 (s, 1H), 7.28-7.39 (m, 5H), 7.48 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 15.3, 15.4, 19.9, 20.3, 60.7, 61.8, 71.9, 77.2, 92.7, 96.7, 99.2, 100.8, 116.5, 126.9, 127.7, 127.9, 128.3, 129.6, 130.5, 135.0, 146.8, 149.1. HRMS (ESI) Calcd. C₂₅H₃₃IO₆ (M+Na)⁺: *m/z* 579.1214. Found: *m/z* 579.1216.

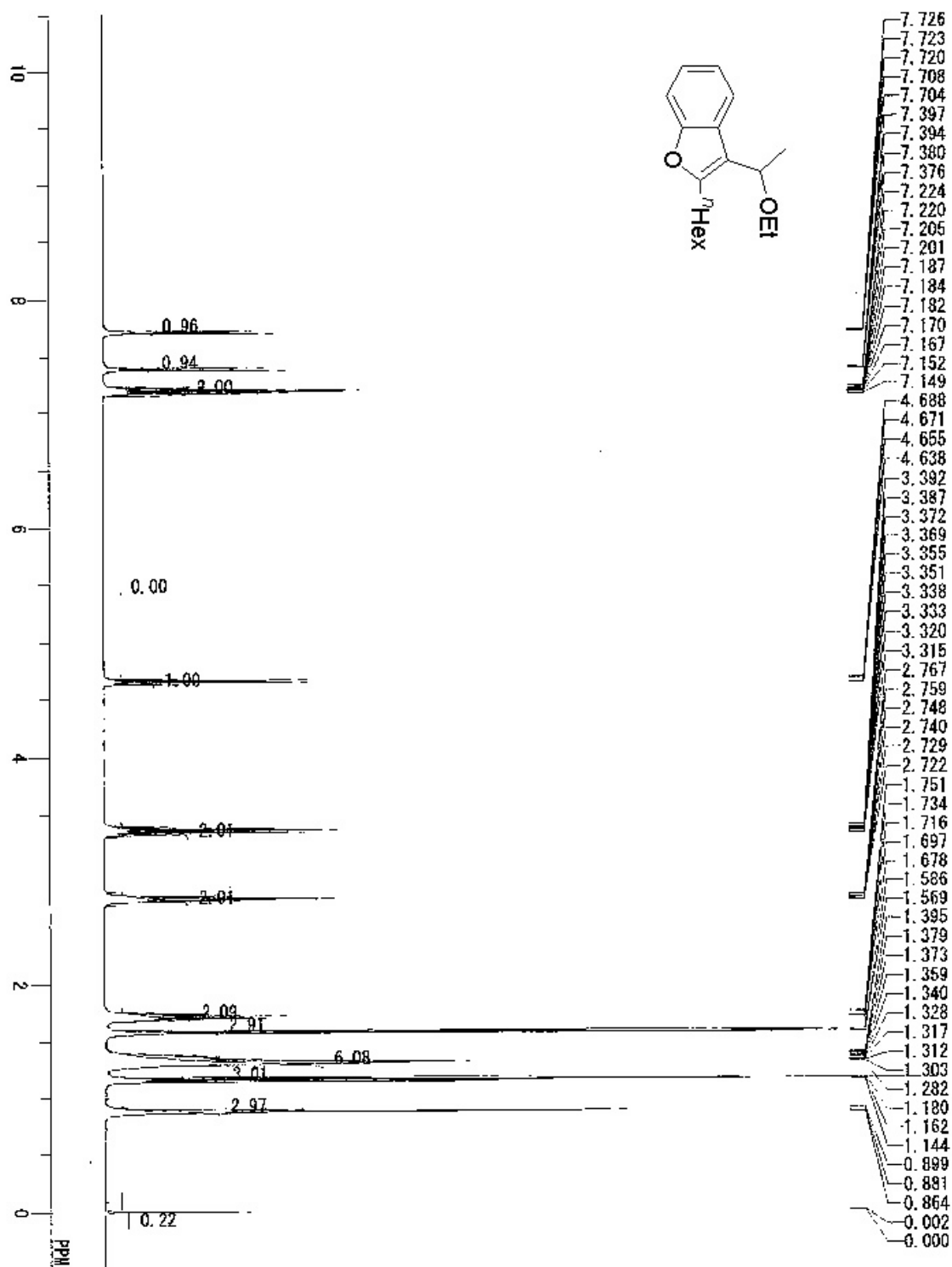


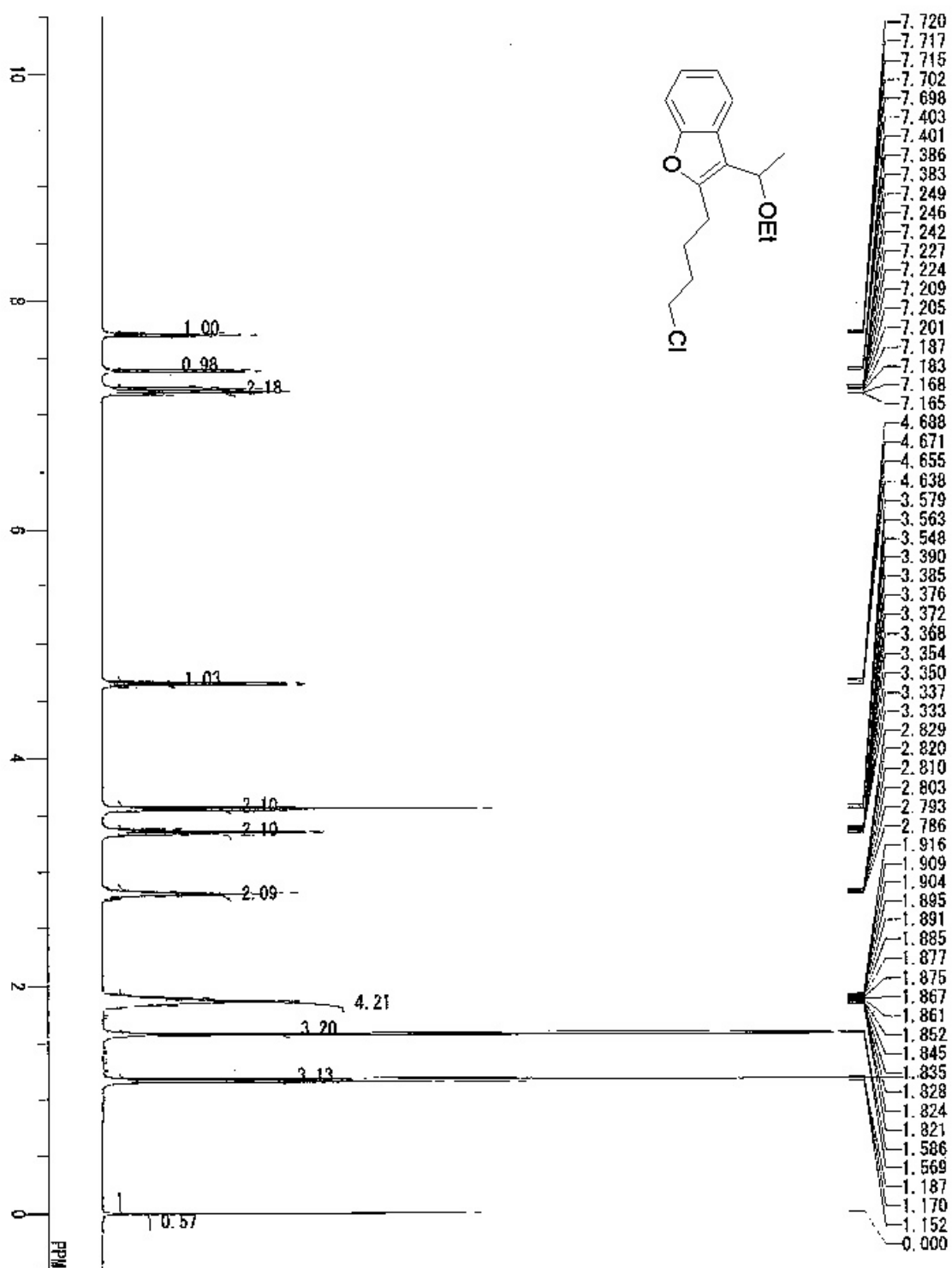
(b) To a THF solution (2 ml) of Pd(PPh₃)₄ (0.1 mmol), CuI (0.2 mmol), and Et₃N (3 mmol) were added the BOM ether (1 mmol) and 4-(1-ethoxyethyl)-3-methoxyphenylacetylene (1.3 mmol) at room temperature. After stirring 2 hours at room temperature, ether was added to the reaction mixture. The ethereal solution was washed with a saturated aqueous solution of ammonium chloride and water and dried over Na₂SO₄. After concentration of the solution under reduced pressure, the crude product was purified by silica gel column chromatography using hexane/ethyl acetate (5/1) as an eluent to give **10** in 64% yield. IR (neat) 2977-2934, 2210, 1737, 1572, 1509 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 1.07-1.15 (m, 9H), 1.28 (d, *J* = 5.2 Hz, 3H), 1.42-1.43 (m, 6H), 3.43-3.49 (m, 4H), 3.50-3.57 (m, 2H), 3.59-3.74 (m, 2H), 3.59 (s, 3H), 4.72 (q, *J* = 5.2 Hz, 1H),

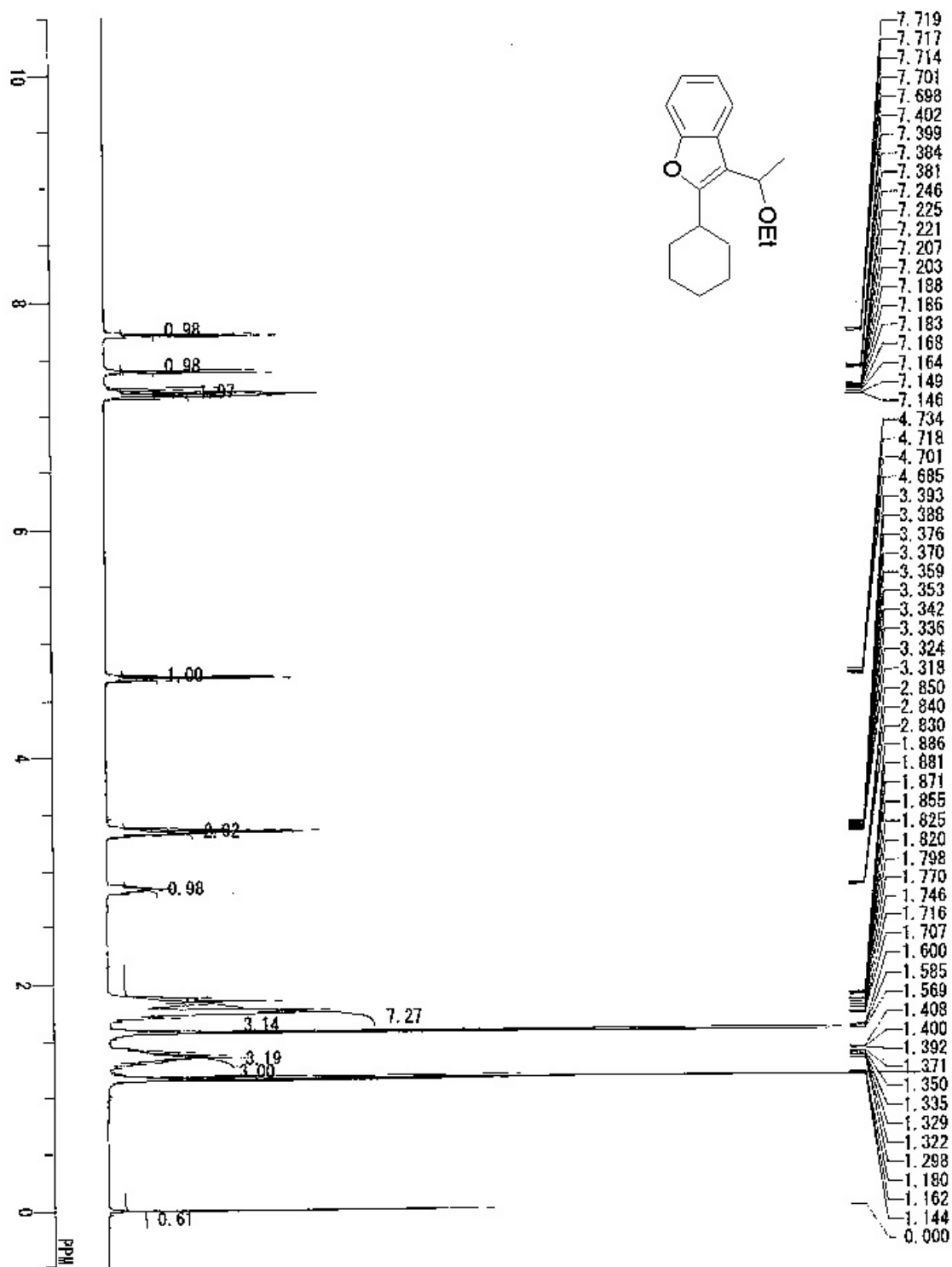
4.91 (s, 2H), 5.29-5.34 (m, 2H), 5.33 (s, 2H), 6.17 (m, 1H), 6.44 (d, $J = 16.0$ Hz, 1H), 6.91-6.96 (m, 3H), 7.06 (s, 1H), 7.14-7.25 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 15.15, 15.24, 15.4, 19.9, 20.3, 20.4, 55.8, 60.6, 61.7, 62.0, 65.5, 71.0, 84.8, 93.4, 96.4, 99.1, 100.8, 101.0, 114.9, 116.7, 117.1, 118.6, 118.7, 124.6, 124.8, 126.4, 127.5, 127.9, 128.2, 130.5, 133.0, 137.5, 146.4, 147.6, 149.6, 150.3. HRMS (ESI) Calcd for $\text{C}_{38}\text{H}_{48}\text{O}_9$ ($\text{M}+\text{Na}$) $^+$: m/z 671.3191. Found: m/z 671.3188.

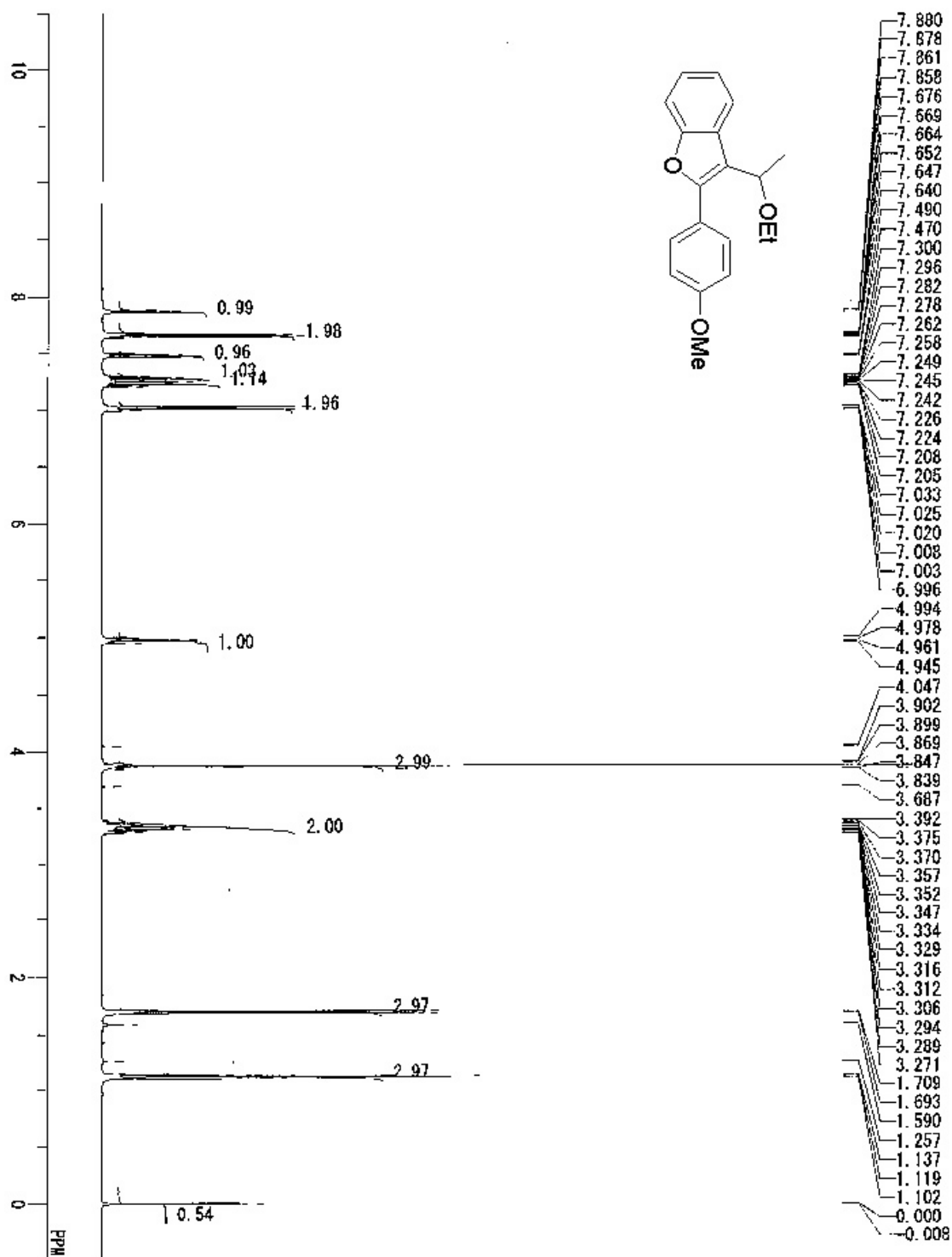


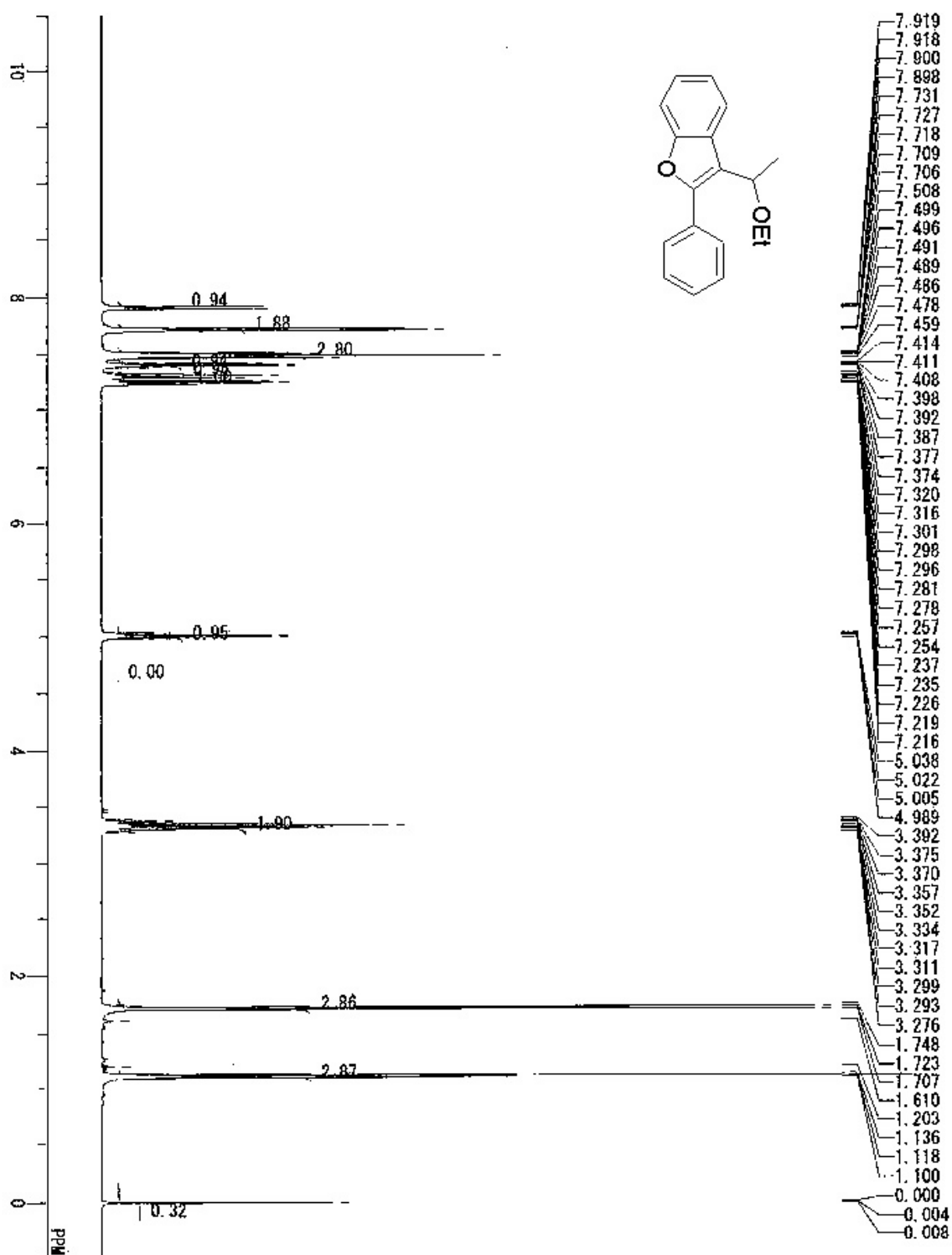
Vibsanol presursor 11. To a mixture of PtCl_2 (20 mol%) and 1,5-cycloocatadiene (80 mol%) in toluene (0.5 ml) was added **10** in toluene (0.3 ml) at room temperature. After stirring at 30 °C for 1 hour, the reaction mixture was filtered through a short silica gel column using ethyl acetate as an eluent. Separation by silica gel column chromatography using hexane/EtOAc (2/1) as an eluent afforded **11** in 76% yield. IR (neat) 2977-2934, 2247, 1737, 1596, 1509, 1466 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 1.20-1.27 (m, 9H), 1.39 (d, $J = 5.2$ Hz, 3H), 1.55 (d, $J = 5.2$ Hz, 3H), 1.61 (d, $J = 4.8$ Hz, 3H), 3.55-3.71 (m, 4H), 3.86 (s, 3H), 3.87-4.01 (m, 2H), 4.65 (s, 2H), 4.77 (s, 2H), 4.83 (q, $J = 5.6$ Hz, 1H), 5.44 (q, $J = 5.2$ Hz, 1H), 5.64 (q, $J = 5.6$ Hz, 1H), 6.28 (m, 1H), 6.67 (d, $J = 16.0$ Hz, 1H), 7.09 (d, $J = 1.6$ Hz, 1H), 7.14-7.45 (m, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 15.2, 15.3, 15.4, 20.0, 20.3, 20.9, 55.9, 60.6, 62.0, 62.1, 62.8, 65.9, 72.1, 99.1, 101.0, 101.7, 111.2, 111.7, 112.0, 112.3, 118.8, 120.3, 124.6, 125.2, 127.7, 127.9, 128.3, 132.2, 133.0, 137.9, 141.6, 144.1, 146.6, 150.7, 154.9. HRMS (ESI) Calcd for $\text{C}_{38}\text{H}_{48}\text{O}_9$ ($\text{M}+\text{Na}$) $^+$: m/z 671.3191. Found: m/z 671.3187.

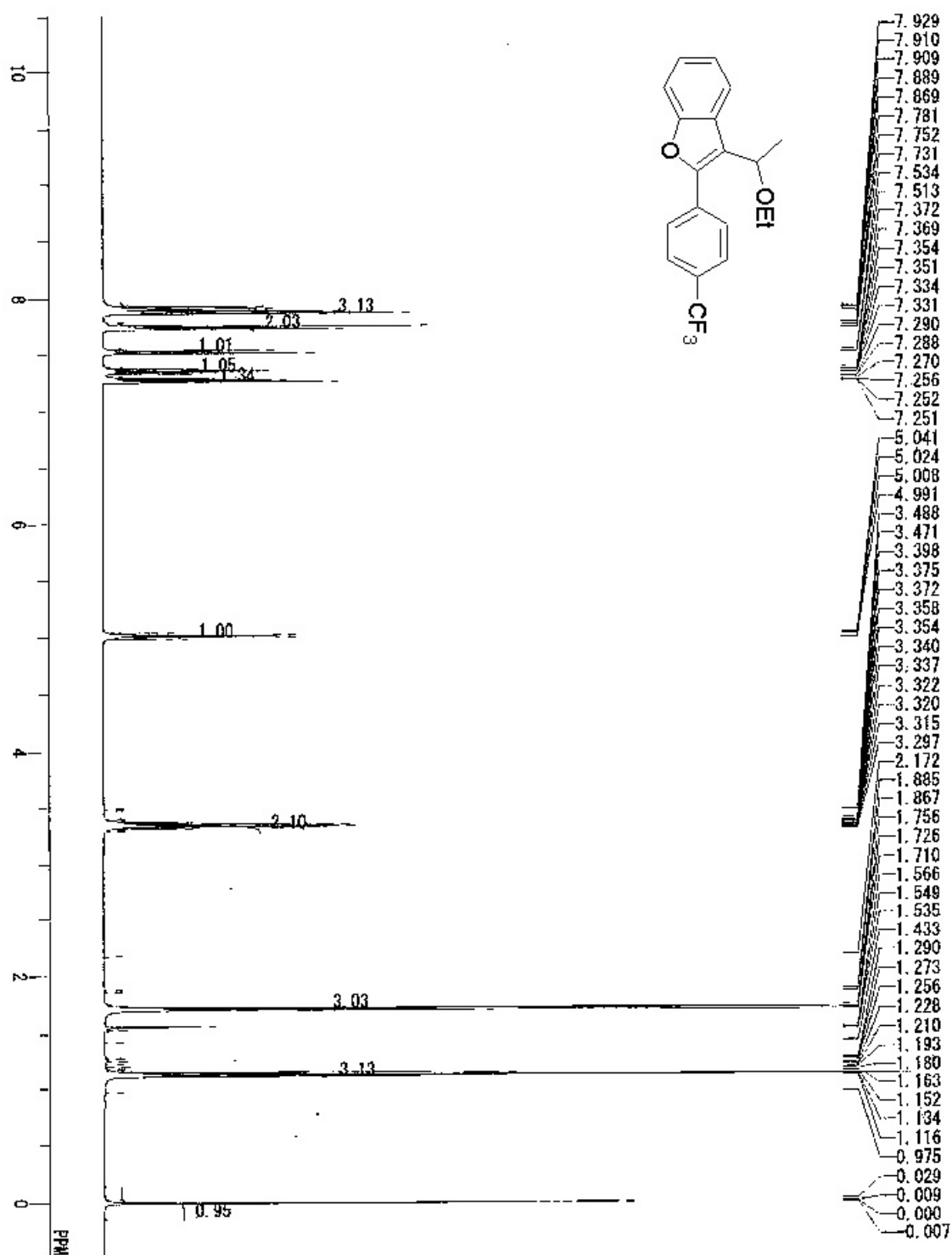
¹H NMR of 2a

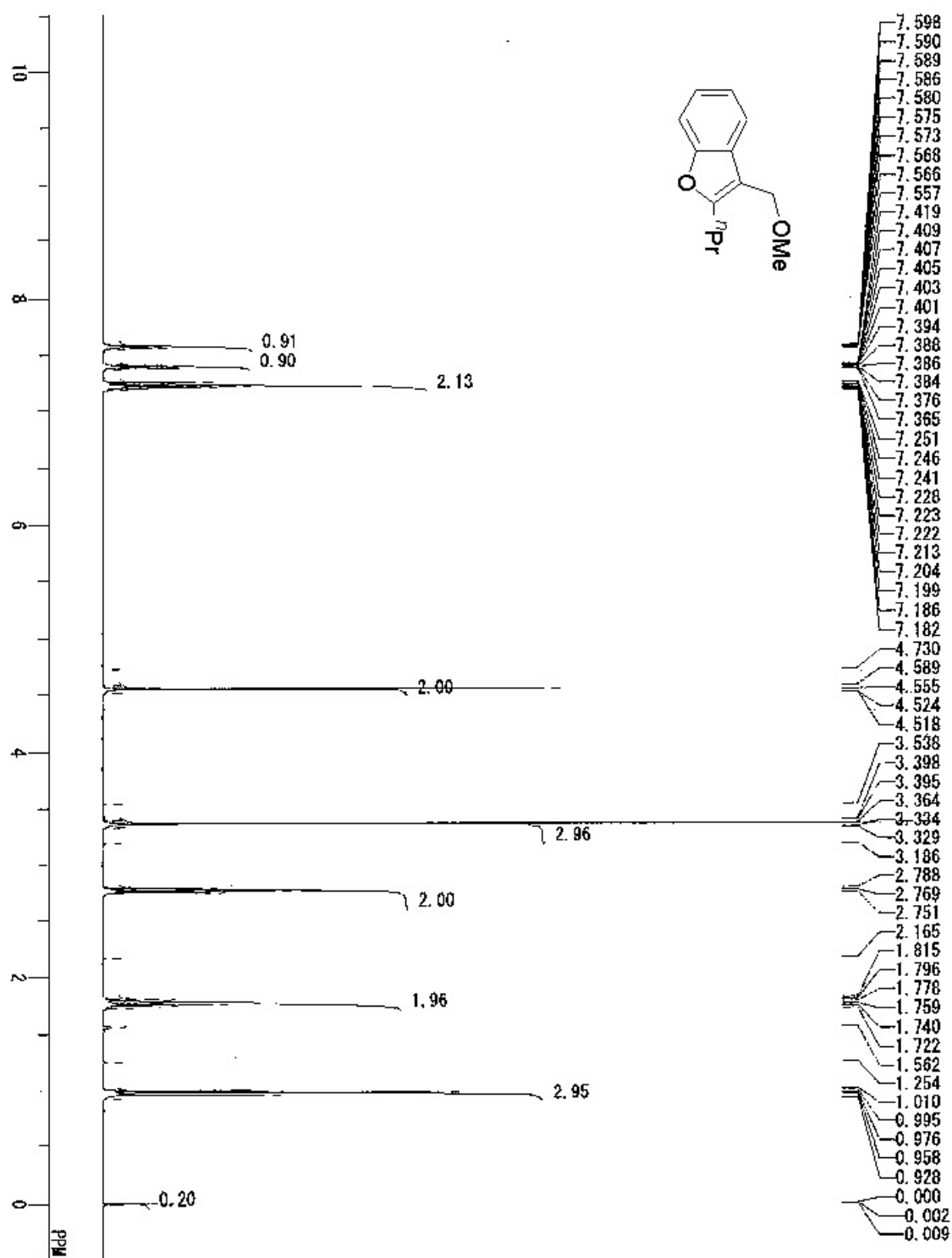
¹H NMR of **2b**

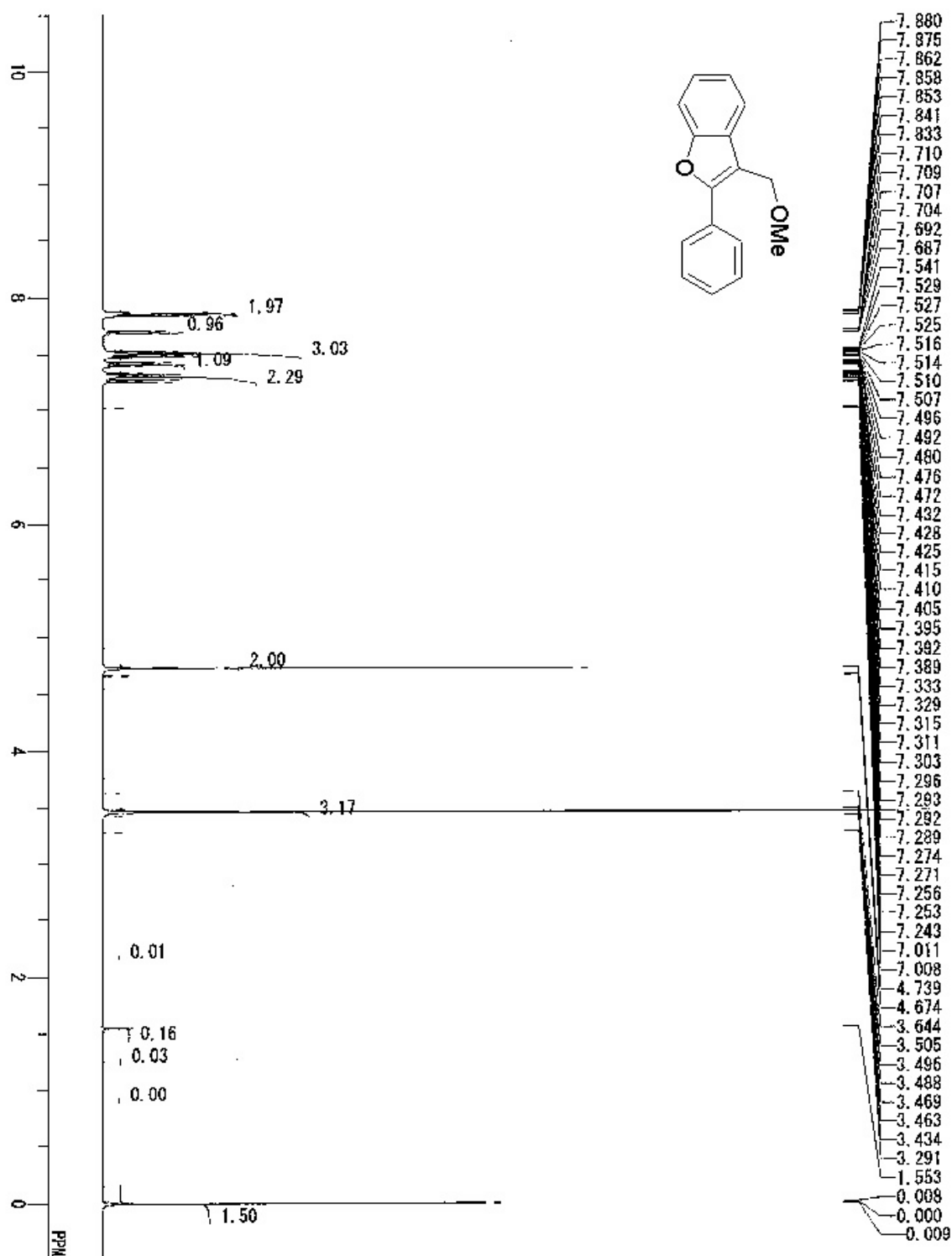
¹H NMR of 2c

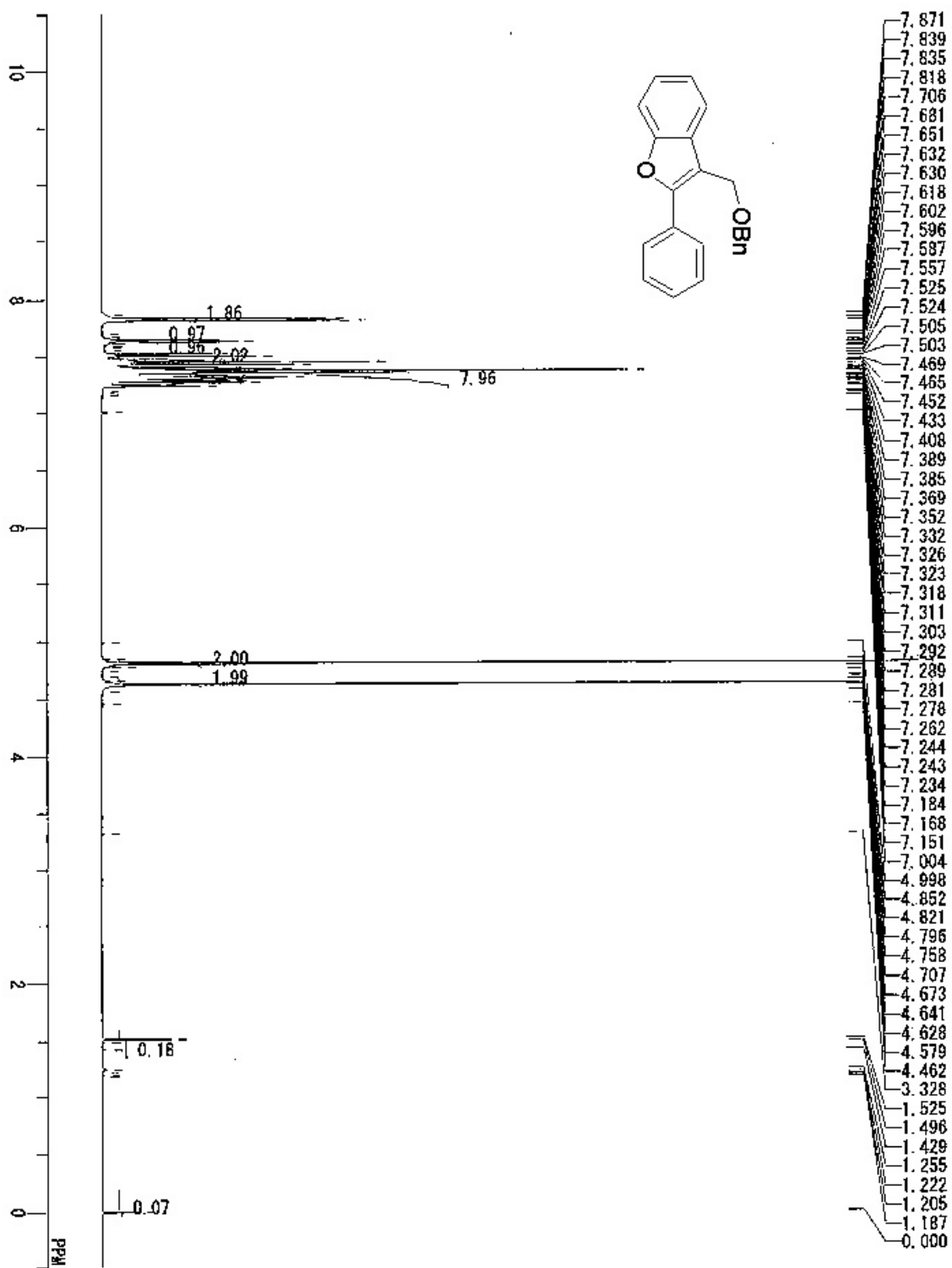
¹H NMR of 2e

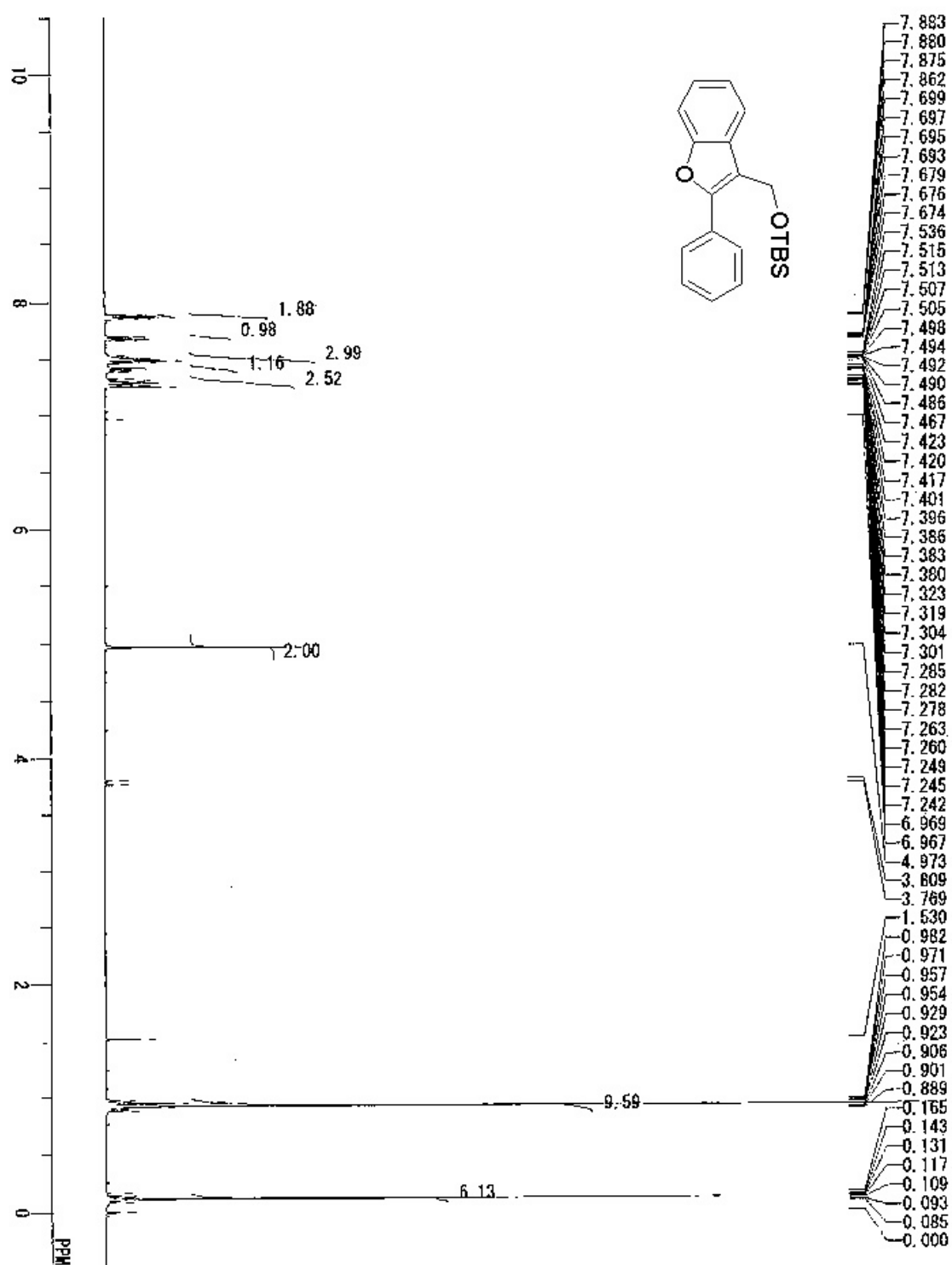
¹H NMR of 2f

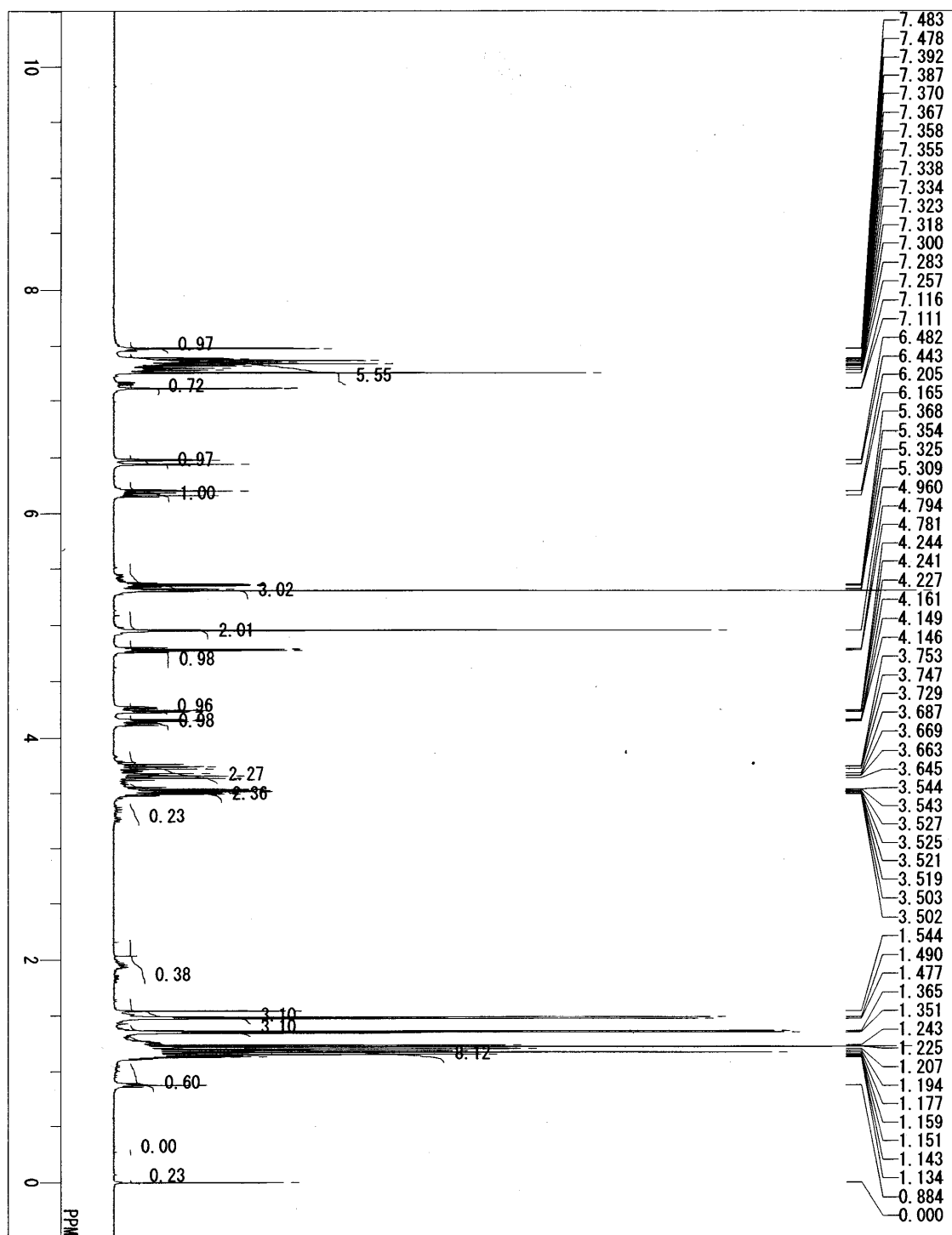
¹H NMR of 2g

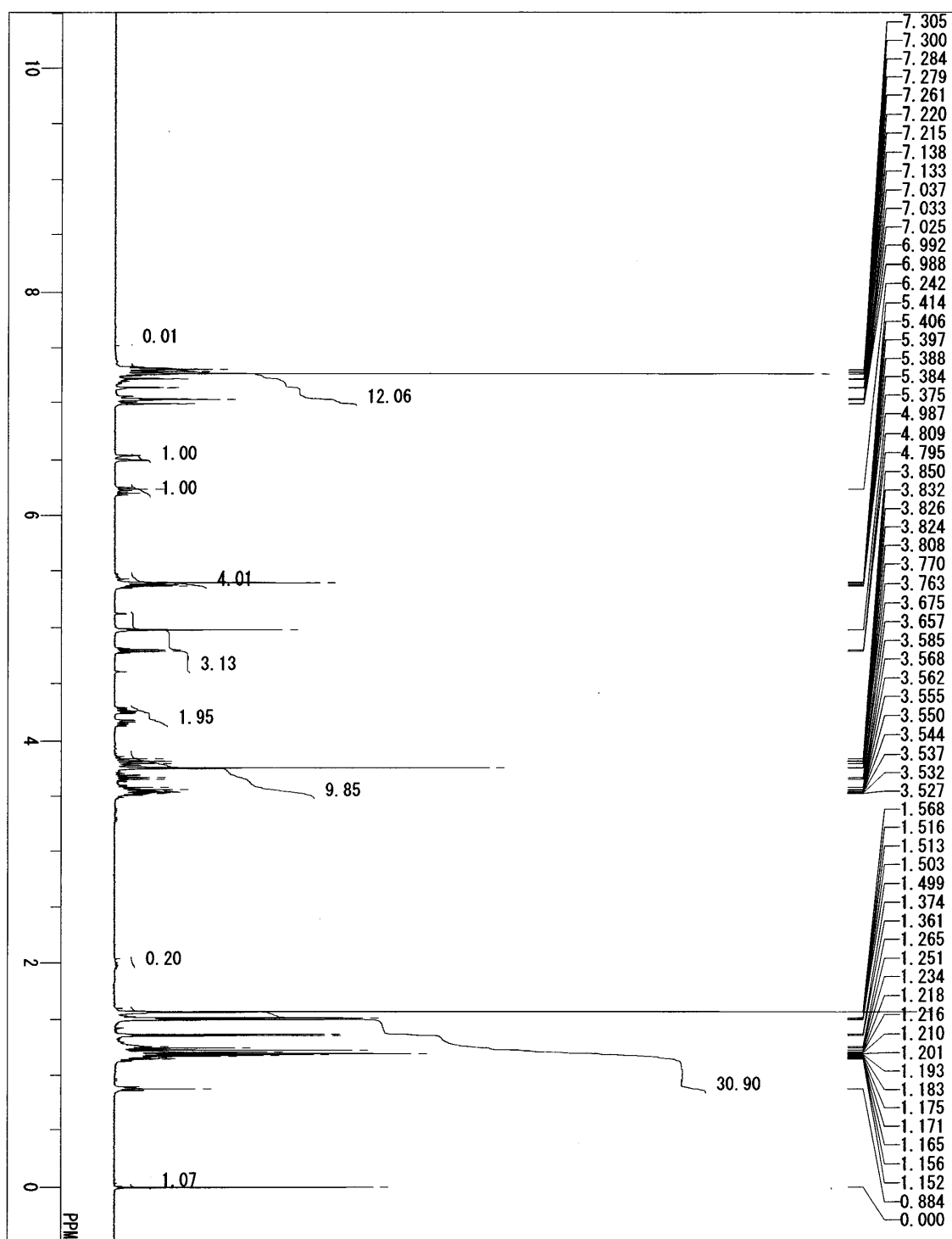
¹H NMR of **2h**

¹H NMR of 2i

¹H NMR of **2k**

¹H NMR of 21

¹H NMR of the BOM ether

^1H NMR of 10.

¹H NMR of 11