Supplementary Information for

Platinum-catalysed diborylation of arynes: synthesis and reaction of 1,2-diborylarenes

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General remarks.

All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique under a purified argon atmosphere. Nuclear magnetic resonance spectra were taken on a JEOL EX-270 (^{1}H, 270 MHz; ^{13}C, 67.8 MHz) spectrometer or a JEOL Lambda-400 (^{1}H, 400 MHz; ^{13}C, 99.5 MHz) spectrometer using residual chloroform (^{1}H, δ = 7.25), benzene (^{1}H, δ = 7.15), $CDCl_3$ (^{13}C, δ = 77.0) or C_6D_6 (^{13}C, δ = 128.0) as an internal standard. ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, br = broad, m = multiplet), coupling constants (Hz), integration. High-resolution mass spectra were obtained with a JEOL JMS-SX102A spectrometer. Melting points were measured with Yanaco Micro Melting Point apparatus and uncorrected. Preparative recycling gel permeation chromatography was performed with GL Science PU 614 equipped with Shodex GPC H-2001L and -2002L columns (chloroform or toluene as an eluent). Unless otherwise noted, commercially available reagents were used without purification. 18-Crown-6 was recrystallized from distilled MeCN. KF (spray-dried) was vacuum dried at 100 °C for 12 h. DME was distilled from sodium/benzophenone ketyl. MeCN was distilled from phosphorus pentoxide.

Aryne precursors.

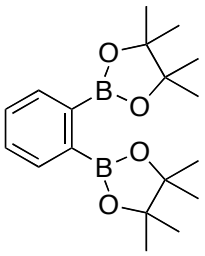
2-(Trimethylsilyl)phenyl triflate (**1a**),[1] 4-methoxy-2-(trimethylsilyl)phenyl triflate (**1b**),[2] 4-methyl-2-(trimethylsilyl)phenyl triflate (**1c**),[3] 4-fluoro-2-(trimethylsilyl)phenyl triflate (**1d**),[4] 4-phenyl-2-(trimethylsilyl)phenyl triflate (**1e**),[2] 6-methyl-2-(trimethylsilyl)phenyl triflate (**1f**),[2] 3,4-dimethoxy-2-(trimethylsilyl)phenyl triflate (**1g**),[2] 4,5-dimethyl-2-(trimethylsilyl)phenyl triflate (**1h**),[5] 3,6-dimethoxy-2-(trimethylsilyl)phenyl triflate (**1i**)[5] and 3,6-dimethyl-2-(trimethylsilyl)phenyl triflate (**1j**)[4] were prepared according to literature procedures.

Diborylation of arynes: a general procedure.

A DME solution (2 mL) of $Pt(dba)_2$ (2.0 mg, 3.0 μmol) and 1-adamantyl isocyanide (2.4 mg, 0.015 mmol) was stirred at room temperature for 0.5 h. To this solution were added 18-Crown-6 (0.032 g, 0.12 mmol), bis(pinacolato)diboron (0.021 g, 0.084 mmol), an aryne precursor (0.060 mmol) and KF (7.0 mg, 0.12 mmol), and the resulting mixture was stirred at 80 °C for the period as specified in Tables 1 and 2. The mixture was diluted with ethyl acetate and washed with cold brine. The organic layer was dried over $MgSO_4$ and concentrated in vacuo. Preparative recycling gel permeation chromatography (chloroform as an eluent) gave the corresponding product.

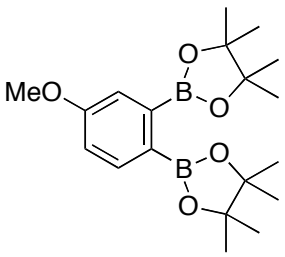
In ^{13}C NMR spectra, boron-bound carbons were not detected because of quadrupolar relaxation.

1,2-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzene (2a)



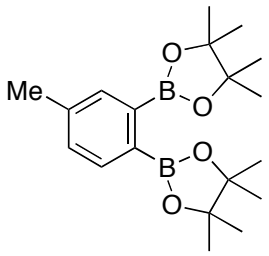
Isolated in 77% yield as a white solid: mp 108–111 °C; ^1H NMR ($CDCl_3$) δ 1.35 (s, 24 H), 7.32–7.40 (m, 2 H), 7.59–7.67 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 24.9, 83.9, 129.1, 133.4; HRMS Calcd for $C_{18}H_{28}B_2O_4$: M^+, 330.2174. Found: *m/z* 330.2176.

1,2-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-4-methoxybenzene (2b)



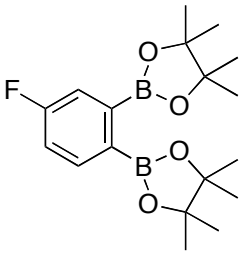
Isolated in 78% yield as a colorless oil: ^1H NMR ($CDCl_3$) δ 1.33 (s, 12 H), 1.36 (s, 12 H), 3.81 (s, 3 H), 6.88 (dd, *J* = 8.2, 2.6 Hz, 1 H), 7.09 (d, *J* = 2.6 Hz, 1 H), 7.65 (d, *J* = 8.2 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 24.8, 24.9, 55.0, 83.6, 83.9, 114.5, 118.1, 136.0, 160.5; HRMS Calcd for $C_{19}H_{30}B_2O_5$: M^+, 360.2279. Found: *m/z* 360.2290.

1,2-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-4-methylbenzene (2c)



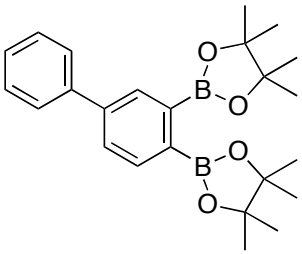
Isolated in 73% yield as a pale yellow solid: mp 69–73 °C; ^{1}H NMR ($CDCl_3$) δ 1.34 (s, 12 H), 1.36 (s, 12 H), 2.31 (s, 3 H), 7.17 (d, J = 7.3 Hz, 1 H), 7.43 (s, 1 H), 7.55 (d, J = 7.3 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 21.5, 24.9, 83.7, 83.8, 129.8, 133.8, 134.0, 139.0; HRMS Calcd for $C_{19}H_{30}B_2O_4$: M^+, 344.2330. Found: *m*/*z* 344.2330.

1,2-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-4-fluorobenzene (2d)



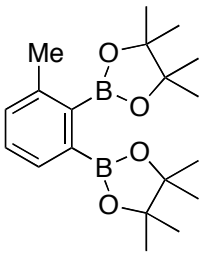
Isolated in 73% yield as a pale yellow oil: ^{1}H NMR ($CDCl_3$) δ 1.34 (s, 12 H), 1.36 (s, 12 H), 7.04 (td, J = 8.6, 2.6 Hz, 1 H), 7.28 (dd, J = 9.6, 2.6 Hz, 1 H), 7.66 (dd, J = 8.2, 5.9 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 24.9, 84.0, 84.1, 115.9 (d, $J_{C\text{-}F}$ = 19.7 Hz), 119.8 (d, $J_{C\text{-}F}$ = 18.8 Hz), 136.1 (d, $J_{C\text{-}F}$ = 7.4 Hz), 163.7 (d, $J_{C\text{-}F}$ = 250.0 Hz); HRMS Calcd for $C_{18}H_{27}B_2FO_4$: M^+, 348.2079. Found: *m*/*z* 348.2083.

1,2-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-4-phenylbenzene (2e)



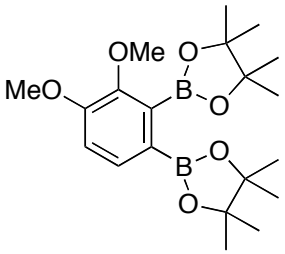
Isolated in 67% yield as a pale yellow solid: mp 122–125 °C; ^{1}H NMR ($CDCl_3$) δ 1.37 (s, 12 H), 1.38 (s, 12 H), 7.29–7.45 (m, 3 H), 7.58–7.63 (m, 3 H), 7.73 (d, J = 7.9 Hz, 1 H), 7.85 (d, J = 2.0 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 24.9, 83.8, 83.9, 127.21, 127.24, 127.7, 128.6, 132.1, 134.2, 141.1, 141.7; HRMS Calcd for $C_{24}H_{32}B_2O_4$: M^+, 406.2487. Found: *m*/*z* 406.2489.

1,2-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-3-methylbenzene (2f)



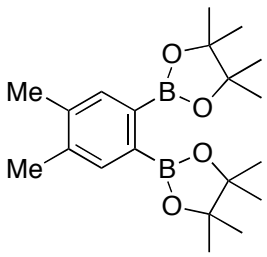
Isolated in 84% yield as a pale yellow solid: mp 57–58 °C; ^{1}H NMR ($CDCl_3$) δ 1.35 (s, 12 H), 1.45 (s, 12 H), 2.43 (s, 3 H), 7.18–7.29 (m, 2 H), 7.64 (dd, *J* = 6.3, 2.3 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 21.9, 24.8, 25.4, 83.8, 128.0, 131.6, 132.2, 140.3; HRMS Calcd for $C_{19}H_{30}B_2O_4$: M^+, 344.2330. Found: *m*/*z* 344.2334.

1,2-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-3,4-dimethoxybenzene (2g)



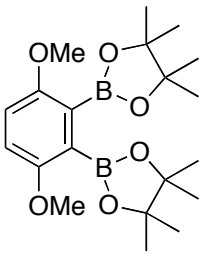
Isolated in 67% yield as a pale yellow solid: mp 111–112 °C; ^{1}H NMR ($CDCl_3$) δ 1.29 (s, 12 H), 1.41 (s, 12 H), 3.80 (s, 3 H), 3.84 (s, 3 H), 6.86 (d, *J* = 7.9 Hz, 1 H), 7.54 (d, *J* = 7.9 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 24.8, 25.1, 55.3, 61.1, 83.7, 83.9, 112.1, 132.8, 151.2, 154.2; HRMS Calcd for $C_{20}H_{32}B_2O_6$: M^+, 390.2385. Found: *m*/*z* 390.2388.

1,2-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-4,5-dimethylbenzene (2h)



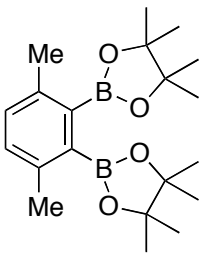
Isolated in 80% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 1.34 (s, 24 H), 2.23 (s, 6 H), 7.41 (s, 2 H); ^{13}C NMR ($CDCl_3$) δ 19.6, 24.8, 83.6, 134.9, 137.6; HRMS Calcd for $C_{20}H_{32}B_2O_4$: M^+, 358.2487. Found: *m*/*z* 358.2490.

1,2-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-3,6-dimethoxybenzene (2i)



Isolated in 50% yield as a brown solid: mp 128–131 °C; ^{1}H NMR ($CDCl_3$) δ 1.36 (s, 24 H), 3.72 (s, 6 H), 6.82 (s, 2 H); ^{13}C NMR ($CDCl_3$) δ 24.9, 57.0, 83.7, 113.9, 157.9; HRMS Calcd for $C_{20}H_{32}B_2O_6$: M^+, 390.2385. Found: *m*/*z* 390.2391.

1,2-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-3,6-dimethylbenzene (2j)

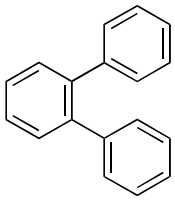


Isolated in 44% yield as a pale yellow solid: mp 136–140 °C; ^{1}H NMR ($CDCl_3$) δ 1.37 (s, 24 H), 2.39 (s, 6 H), 7.01 (s, 2 H); ^{13}C NMR ($CDCl_3$) δ 22.0, 25.2, 83.6, 130.8, 139.2; HRMS Calcd for $C_{20}H_{32}B_2O_4$: M^+, 358.2487. Found: *m*/*z* 358.2484.

Cross-coupling of 2a for synthesis of symmetrical *ortho*-terphenyls: a general procedure.

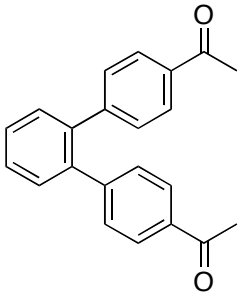
A DME solution (1.4 mL) of **2a** (0.013 g, 0.039 mmol), an aryl halide (0.086 mmol), Cs_2CO_3 (0.029 g, 0.090 mmol), $Pd[P(t\text{-}Bu)_3]_2$ (1.0 mg, 2.0 μmol) and H_2O (0.015 mL) was stirred at 80 °C for the period as specified in Table 3. The mixture was diluted with ethyl acetate and washed with brine. The organic layer was dried over $MgSO_4$ and concentrated in vacuo. Preparative recycling gel permeation chromatography (chloroform or toluene as an eluent) gave the corresponding product.

1,2-Diphenylbenzene (3a)[6]



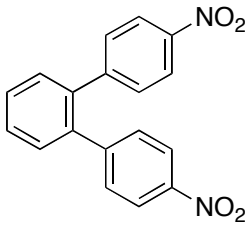
Isolated in 85% yield: ^{1}H NMR ($CDCl_3$) δ 7.12–7.25 (m, 10 H), 7.39–7.46 (m, 4 H); ^{13}C NMR ($CDCl_3$) δ 126.4, 127.5, 127.8, 129.9, 130.6, 140.6, 141.5.

1,2-Di(4-acetylphenyl)benzene (3b)[7]



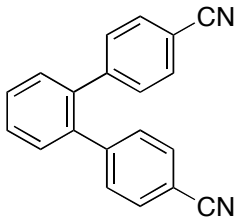
Isolated in 82% yield: ^{1}H NMR ($CDCl_3$) δ 2.57 (s, 6 H), 7.21 (d, J = 8.6 Hz, 4 H), 7.42–7.50 (m, 4 H), 7.81 (d, J = 8.6 Hz, 4 H); ^{13}C NMR ($CDCl_3$) δ 26.6, 128.2, 128.3, 130.0, 130.6, 135.4, 139.5, 146.0, 197.8.

1,2-Di(4-nitrophenyl)benzene (3c)[8]



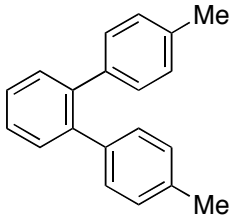
Isolated in 72% yield: ^{1}H NMR ($CDCl_3$) δ 7.28 (d, *J* = 8.9 Hz, 4 H), 7.43–7.57 (m, 4 H), 8.10 (d, *J* = 8.9 Hz, 4 H); ^{13}C NMR ($CDCl_3$) δ 123.5, 129.1, 130.59, 130.64, 138.3, 146.8, 147.4.

1,2-Di(4-cyanophenyl)benzene (3d)



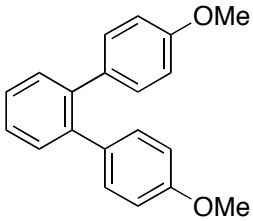
Isolated in 64% yield as a pale yellow solid: mp 184–188 °C; ^{1}H NMR ($CDCl_3$) δ 7.20 (d, *J* = 7.9 Hz, 4 H), 7.40–7.55 (m, 8 H); ^{13}C NMR ($CDCl_3$) δ 110.9, 118.6, 128.9, 130.4, 130.6, 132.0, 138.6, 145.5; HRMS Calcd for $C_{20}H_{12}N_2$: M^+, 280.1000. Found: *m*/*z* 280.1007.

1,2-Di(4-methylphenyl)benzene (3e)[9]



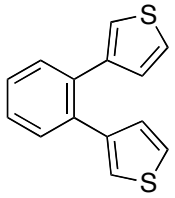
Isolated in 64% yield: ^{1}H NMR ($CDCl_3$) δ 2.32 (s, 6 H), 7.02–7.08 (m, 8 H), 7.37–7.43 (m, 4 H); ^{13}C NMR ($CDCl_3$) δ 21.1, 127.2, 128.6, 129.7, 130.6, 135.9, 138.7, 140.4.

1,2-Di(4-methoxyphenyl)benzene (3f)[9]



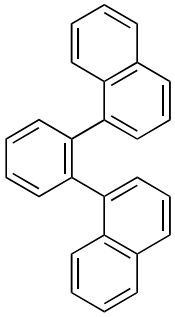
Isolated in 55% yield: ^{1}H NMR ($CDCl_3$) δ 3.78 (s, 6 H), 6.76 (d, *J* = 8.6 Hz, 4 H), 7.06 (d, *J* = 8.6 Hz, 4 H), 7.34–7.40 (m, 4 H); ^{13}C NMR ($CDCl_3$) δ 55.1, 113.3, 127.1, 130.5, 130.9, 134.1, 140.0, 158.2.

1,2-Di(3-thienyl)benzene (3g)



Isolated in 74% yield as a brown oil: ^{1}H NMR ($CDCl_3$) δ 6.79 (dd, J = 5.0, 1.0 Hz, 2 H), 7.06 (dd, J = 3.0, 1.0 Hz, 2 H), 7.17 (dd, J = 5.0, 3.0 Hz, 2 H), 7.32–7.49 (m, 4 H); ^{13}C NMR ($CDCl_3$) δ 122.8, 124.6, 127.5, 128.9, 130.2, 135.4, 142.1; HRMS Calcd for $C_{14}H_{10}S_2$: M^+, 242.0224. Found: m/z 242.0222.

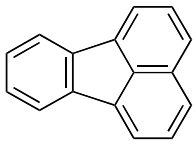
1,2-Di(1-naphthyl)benzene (3h)
(A mixture of two conformers)



Isolated in 74% yield as a white solid: mp 164–167 °C; ^{1}H NMR ($CDCl_3$) δ 6.87–7.02 (m), 7.05–7.13 (m), 7.18–7.30 (m), 7.40–7.65 (m), 7.72–7.90 (m); ^{13}C NMR ($CDCl_3$) δ 124.4, 124.8, 125.19, 125.21, 125.4, 125.7, 126.2, 126.4, 126.8, 126.95, 126.98, 127.1, 127.2, 127.8, 128.1, 128.2, 131.58, 131.64, 131.7, 132.5, 133.2, 133.3, 138.8, 138.9, 140.2, 140.3; HRMS Calcd for $C_{26}H_{18}$: M^+, 330.1409. Found: m/z 330.1407.

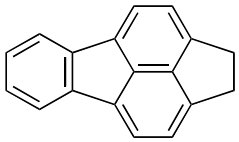
Cross-coupling of 2a for synthesis of fluoranthenes: a general procedure.
A DME solution (1.4 mL) of **2a** (0.013 g, 0.039 mmol), an 1,8-dihalonaphthalene (0.043 mmol), Cs_2CO_3 (0.029 g, 0.090 mmol), Pd[P(*t*-Bu)$_3$]$_2$ (1.0 mg, 2.0 μmol) and H_2O (0.015 mL) was stirred at 80 °C for the period as specified in Scheme 2. The mixture was diluted with ethyl acetate and washed with brine. The organic layer was dried over $MgSO_4$ and concentrated in vacuo. Preparative recycling gel permeation chromatography (chloroform or toluene as an eluent) gave the corresponding product.

Fluoranthene (3i)[6]



Isolated in 56% yield: ^{1}H NMR ($CDCl_3$) δ 7.29–7.38 (m, 2 H), 7.51–7.63 (m, 2 H), 7.72–7.92 (m, 6 H); ^{13}C NMR ($CDCl_3$) δ 120.0, 121.5, 126.6, 127.5, 127.9, 130.0, 132.5, 137.0, 139.4.

1,2-Dihydrocyclopenta[*cd*]fluoranthene (3j)[10]



Isolated in 71% yield: ^{1}H NMR (C_6D_6) δ 3.02 (s, 4 H), 7.18–7.24 (m, 2 H), 7.25–7.30 (m, 2 H), 7.74–7.77 (m, 2 H), 7.84–7.88 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 32.4, 120.8, 122.05, 122.07, 126.7, 132.6, 136.7, 140.2, 145.9.

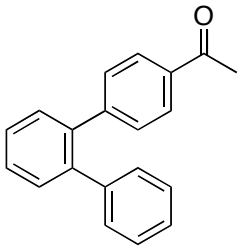
Synthesis of 2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)biphenyl.

A DME solution (1.2 mL) of **2a** (0.14 g, 0.42 mmol), iodobenzene (0.086 g, 0.42 mmol), KOH (0.35 g, 0.63 mmol), $Pd(PPh_3)_4$ (0.024 g, 0.021 mmol) and H_2O (0.21 mL) was stirred at 40 °C for 23 h. The mixture was diluted with ethyl acetate and washed with brine. The organic layer was dried over $MgSO_4$ and concentrated in vacuo. Preparative recycling gel permeation chromatography (chloroform as an eluent) gave 2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)biphenyl (0.066 g, 56% yield).

Cross-coupling for synthesis of unsymmetrical *ortho*-terphenyls: a general procedure.

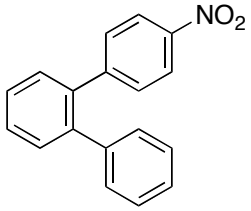
A DME solution (1.4 mL) of 2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)biphenyl (0.011 g, 0.039 mmol), an aryl halide (0.043 mmol), Cs_2CO_3 (0.015 g, 0.045 mmol), $Pd[P(t\text{-}Bu)_3]_2$ (1.0 mg, 2.0 μmol) and H_2O (7.5 μL) was stirred at 80 °C for the period as specified in Table 4. The mixture was diluted with ethyl acetate and washed with brine. The organic layer was dried over $MgSO_4$ and concentrated in vacuo. Preparative recycling gel permeation chromatography (chloroform or toluene as an eluent) gave the corresponding product.

1-(4-Acetylphenyl)-2-phenylbenzene (4a)[11]



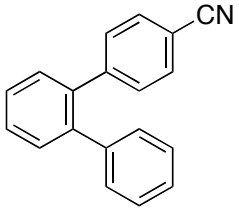
Isolated in 93% yield: ^{1}H NMR ($CDCl_3$) δ 2.57 (s, 3 H), 7.10–7.16 (m, 2 H), 7.19–7.27 (m, 4 H), 7.41–7.48 (m. 5 H), 7.81 (d, J = 8.6 Hz, 2 H); ^{13}C NMR ($CDCl_3$) δ 26.6, 126.7, 127.6, 127.97, 128.04, 128.2, 129.8, 130.1, 130.4, 130.8, 135.1, 139.4, 140.7, 141.0, 146.6, 197.9.

1-(4-Nitrophenyl)-2-phenylbenzene (4b)



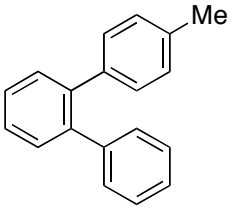
Isolated in 86% yield as a brown solid: mp 81–83 °C; ^{1}H NMR ($CDCl_3$) δ 7.07–7.13 (m, 2 H), 7.20–7.33 (m, 5 H), 7.40–7.50 (m, 4 H), 8.06 (d, J = 8.9 Hz, 2 H); ^{13}C NMR ($CDCl_3$) δ 123.2, 127.0, 127.8, 128.2, 128.6, 128.8, 129.8, 130.3, 130.6, 130.9, 138.2, 140.5, 140.7, 148.5; HRMS Calcd for $C_{18}H_{13}NO_2$: M^+, 275.0946. Found: m/z 275.0943.

1-(4-Cyanophenyl)-2-phenylbenzene (4c)



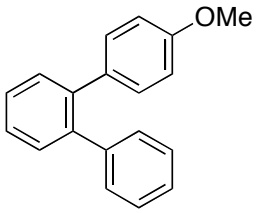
Isolated in 72% yield as a white solid: mp 105–109 °C; ^{1}H NMR ($CDCl_3$) δ 7.04–7.13 (m, 2 H), 7.16–7.31 (m, 5 H), 7.35–7.54 (m, 6 H); ^{13}C NMR ($CDCl_3$) δ 110.2, 119.0, 126.9, 127.8, 128.2, 128.6, 129.8, 130.2, 130.5, 130.9, 131.7, 138.6, 140.59, 140.64, 146.4; HRMS Calcd for $C_{19}H_{13}N$: M^+, 255.1048. Found: m/z 255.1040.

1-(4-Methylphenyl)-2-phenylbenzene (4d)[12]



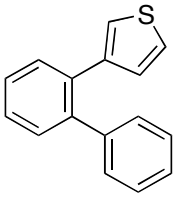
Isolated in 85% yield: ^{1}H NMR ($CDCl_3$) δ 2.31 (s, 3 H), 7.03 (brs, 4 H), 7.13–7.24 (m, 5 H), 7.41 (brs, 4H); ^{13}C NMR ($CDCl_3$) δ 21.1, 126.3, 127.2, 127.4, 127.8, 128.6, 129.7, 129.9, 130.6, 136.0, 138.5, 140.5, 141.7.

1-(4-Methoxyphenyl)-2-phenylbenzene (4e)[11]



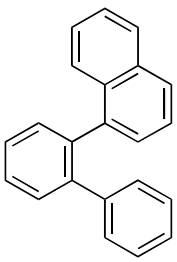
Isolated in 68% yield: ^{1}H NMR ($CDCl_3$) δ 3.77 (s, 3 H), 6.75 (d, *J* = 8.9 Hz, 2 H), 7.05 (d, *J* = 8.9 Hz, 2 H), 7.12–7.25 (m, 5 H), 7.40 (brs, 4 H); ^{13}C NMR ($CDCl_3$) δ 55.1, 113.3, 126.3, 127.1, 127.5, 127.9, 129.8, 130.5, 130.6, 130.9, 133.8, 140.1, 140.4, 141.6, 158.2.

1-Phenyl-2-(3-thienyl)benzene (4f)



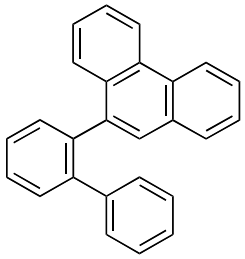
Isolated in 87% yield as a pale yellow solid: mp 62–63 °C; ^{1}H NMR ($CDCl_3$) δ 6.71 (dd, *J* = 4.9, 1.3 Hz, 1 H), 6.99 (dd, *J* = 3.0, 1.3 Hz, 1 H), 7.11 (dd, *J* = 4.9, 3.0 Hz, 1 H), 7.16–7.30 (m, 5 H), 7.36–7.42 (m, 3 H), 7.45–7.52 (m, 1 H); ^{13}C NMR ($CDCl_3$) δ 123.0, 124.5, 126.7, 127.4, 127.5, 127.9, 129.1, 129.5, 130.1, 130.6, 135.2, 140.5, 141.6, 141.9; HRMS Calcd for $C_{16}H_{12}S$: M^+, 236.0660. Found: *m/z* 236.0662.

1-(1-Naphthyl)-2-phenylbenzene (4g)



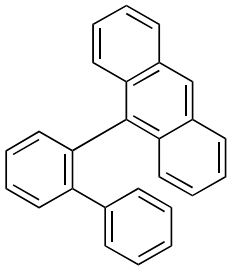
Isolated in 87% yield as a white solid: mp 111–113 °C; ^{1}H NMR ($CDCl_3$) δ 6.95–7.10 (m, 5 H), 7.13–7.57 (m, 8 H), 7.62–7.85 (m, 3 H); ^{13}C NMR ($CDCl_3$) δ 125.0, 125.5, 125.7, 126.25, 126.34, 126.9, 127.2, 127.6, 127.8, 128.05, 128.14, 129.1, 130.3, 131.7, 132.2, 133.4, 138.9, 139.3, 141.3, 141.9; HRMS Calcd for $C_{22}H_{16}$: M^+, 280.1252. Found: *m/z* 280.1248.

1-(9-Phenanthryl)-2-phenylbenzene (4h)



Isolated in 81% yield as a white solid: mp 105–107 °C; ^{1}H NMR ($CDCl_3$) δ 6.95–7.05 (m, 3 H), 7.12–7.20 (m, 2 H), 7.38–7.80 (m, 11 H), 8.67 (d, *J* = 8.2 Hz, 2 H); ^{13}C NMR ($CDCl_3$) δ 122.4, 122.6, 126.2, 126.3, 126.35, 126.41, 126.6, 127.1, 127.2, 127.7, 127.9, 128.6, 128.7, 129.0, 129.8, 130.2, 130.3, 131.4, 131.5, 131.7, 138.1, 138.9, 141.3, 142.0; HRMS Calcd for $C_{26}H_{18}$: M^+, 330.1409. Found: *m/z* 330.1401.

1-(9-Anthryl)-2-phenylbenzene (4i)



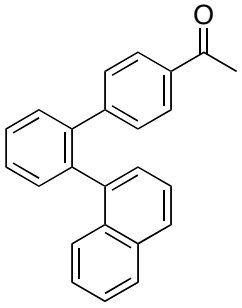
Isolated in 64% yield as a pale yellow solid: mp 120–123 °C; ^{1}H NMR ($CDCl_3$) δ 6.71–7.07 (m, 5 H), 7.22–7.84 (m, 10 H), 7.95 (d, *J* = 7.8 Hz, 2 H), 8.38 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ 124.9, 125.3, 126.4, 126.5, 126.8, 127.2, 127.3, 128.1, 128.3, 128.4, 130.2, 130.4, 131.1, 132.4, 136.1, 136.9, 141.1, 143.1; HRMS Calcd for $C_{26}H_{18}$: M^+, 330.1409. Found: *m/z* 330.1402.

One-batch cross-coupling.

A DME solution (4 mL) of **2a** (0.026 g, 0.078 mmol), 1-iodonaphthalene (0.021 g, 0.082 mmol), Cs_2CO_3 (0.032 g, 0.099 mmol), Pd[P(*t*-Bu)$_3$]$_2$ (2.0 mg, 3.9 μmol) and H_2O (0.020 mL) was stirred at 60 °C for 7 h before addition of 4-iodoacetophenone (0.039 g, 0.16

mmol), Cs_2CO_3 (0.032 g, 0.099 mmol) and H_2O (0.020 mL). The resulting mixture was stirred at 80 °C for 24 h, diluted with ethyl acetate and washed with brine. The organic layer was dried over $MgSO_4$ and concentrated in vacuo. Preparative thin layer chromatography (dichloromethane/hexane as an eluent) followed by preparative recycling gel permeation chromatography (toluene as an eluent) gave **4j**.

1-(4-Acetylphenyl)-2-(1-naphthyl)-benzene (4j)

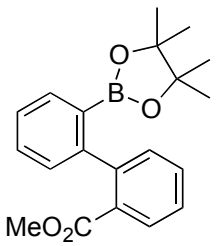


Isolated in 43% yield as a waxy colorless solid: ^{1}H NMR ($CDCl_3$) δ 2.45 (s, 3 H), 7.02–7.21 (m, 3 H), 7.22–7.67 (m, 10 H), 7.68–7.86 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 26.5, 125.0, 125.6, 126.0, 127.5, 127.65, 127.73, 127.9, 128.1, 128.2, 129.2, 130.1, 131.8, 132.0, 133.4, 134.9, 138.8, 138.9, 140.7, 146.4, 197.8; HRMS Calcd for $C_{24}H_{18}O$: M^+, 322.1358. Found: *m/z* 322.1363.

Cross-coupling of 2a for synthesis of monoborylbiaryls: a general procedure.

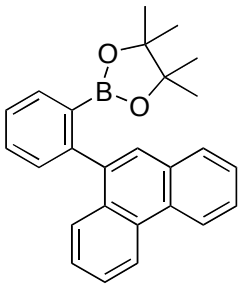
A DME solution (4 mL) of **2a** (0.026 g, 0.078 mmol), an aryl halide (0.082 mmol), Cs_2CO_3 (0.032 g, 0.098 mmol), $Pd[P(t\text{-}Bu)_3]_2$ (2.0 mg, 3.9 μmol) and H_2O (0.020 mL) was stirred at 60 °C for the period as specified in Scheme 4. The mixture was diluted with ethyl acetate and washed with brine. The organic layer was dried over $MgSO_4$ and concentrated in vacuo. Preparative thin layer chromatography (hexane/ethyl acetate as an eluent) gave the corresponding product.

Methyl 2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)biphenyl-2'-carboxylate (5a)



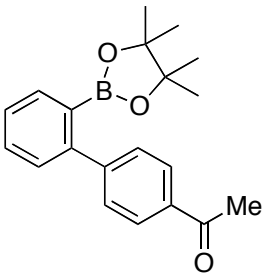
Isolated in 82% yield as a pale yellow waxy solid: ^{1}H NMR ($CDCl_3$) δ 1.07 (s, 12 H), 3.57 (s, 3 H), 7.12–7.56 (m, 6 H), 7.75 (d, *J* = 7.3 Hz, 1 H), 7.93 (d, *J* = 6.9 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 24.5, 51.5, 83.2, 126.1, 126.6, 128.3, 129.4, 129.8, 130.3, 130.7, 131.2, 134.0, 144.8, 147.8, 167.9; HRMS Calcd for $C_{20}H_{23}BO_4$: M^+, 338.1689. Found: *m*/*z* 338.1688.

9-[2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]phenanthrene (5b)



Isolated in 55% yield as an ocher solid: mp 104–106 °C; ^{1}H NMR ($CDCl_3$) δ 0.72 (s, 6 H), 0.87 (s, 6 H), 7.35–7.75 (m, 9 H), 7.80–7.95 (m, 2 H), 8.65–8.85 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 24.1, 24.3, 83.3, 122.38, 122.43, 122.55, 122.60, 126.0, 126.1, 126.3, 126.5, 126.7, 127.1, 128.6, 129.9, 130.1, 130.2, 130.4, 131.6, 132.6, 134.3, 140.1, 145.9; HRMS Calcd for $C_{26}H_{25}BO_2$: M^+, 380.1948. Found: *m*/*z* 380.1951.

4'-Acetyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)biphenyl (5c)



Isolated in 31% yield as a white solid: mp 104–107 °C; ^{1}H NMR ($CDCl_3$) δ 1.20 (s, 12 H), 2.65 (s, 3 H), 7.33–7.42 (m, 2 H), 7.44–7.53 (m, 3 H), 7.77 (d, *J* = 7.6 Hz, 1 H), 7.98 (d, *J* = 8.6 Hz, 2 H); ^{13}C NMR ($CDCl_3$) δ 24.6, 26.7, 83.9, 127.0, 127.9, 128.9, 129.4, 130.3, 134.9, 135.5, 146.4, 148.1, 198.1; HRMS Calcd for $C_{20}H_{23}BO_3$: M^+, 322.1740. Found: *m*/*z* 322.1737.

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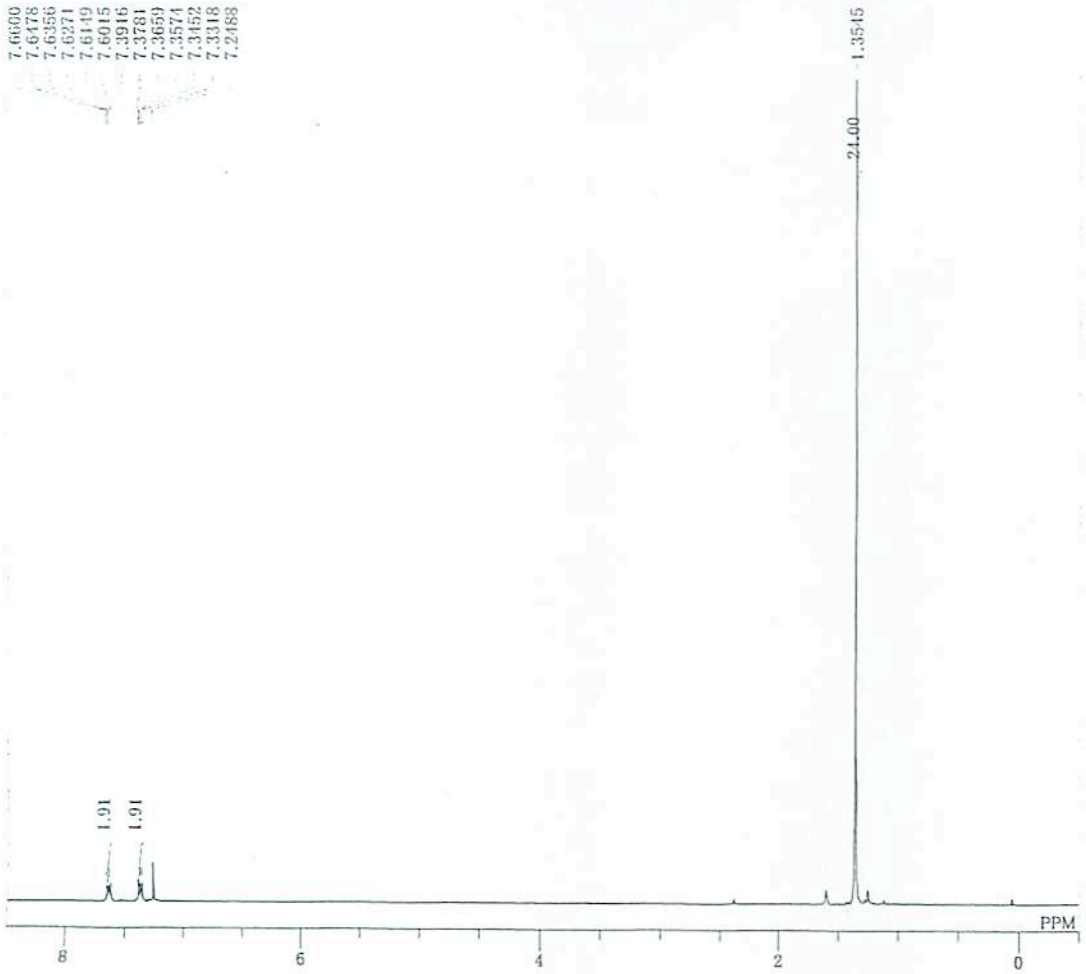

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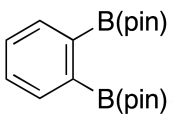
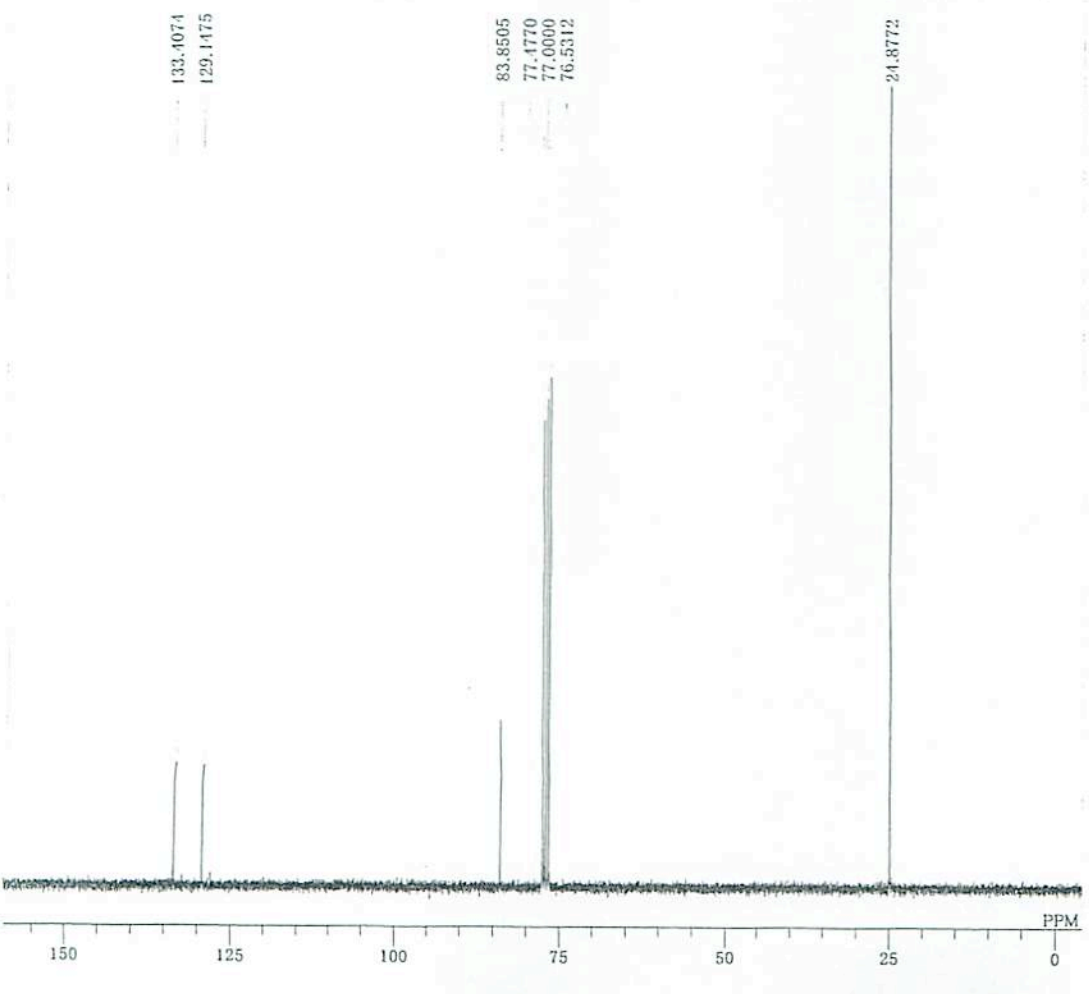


2a



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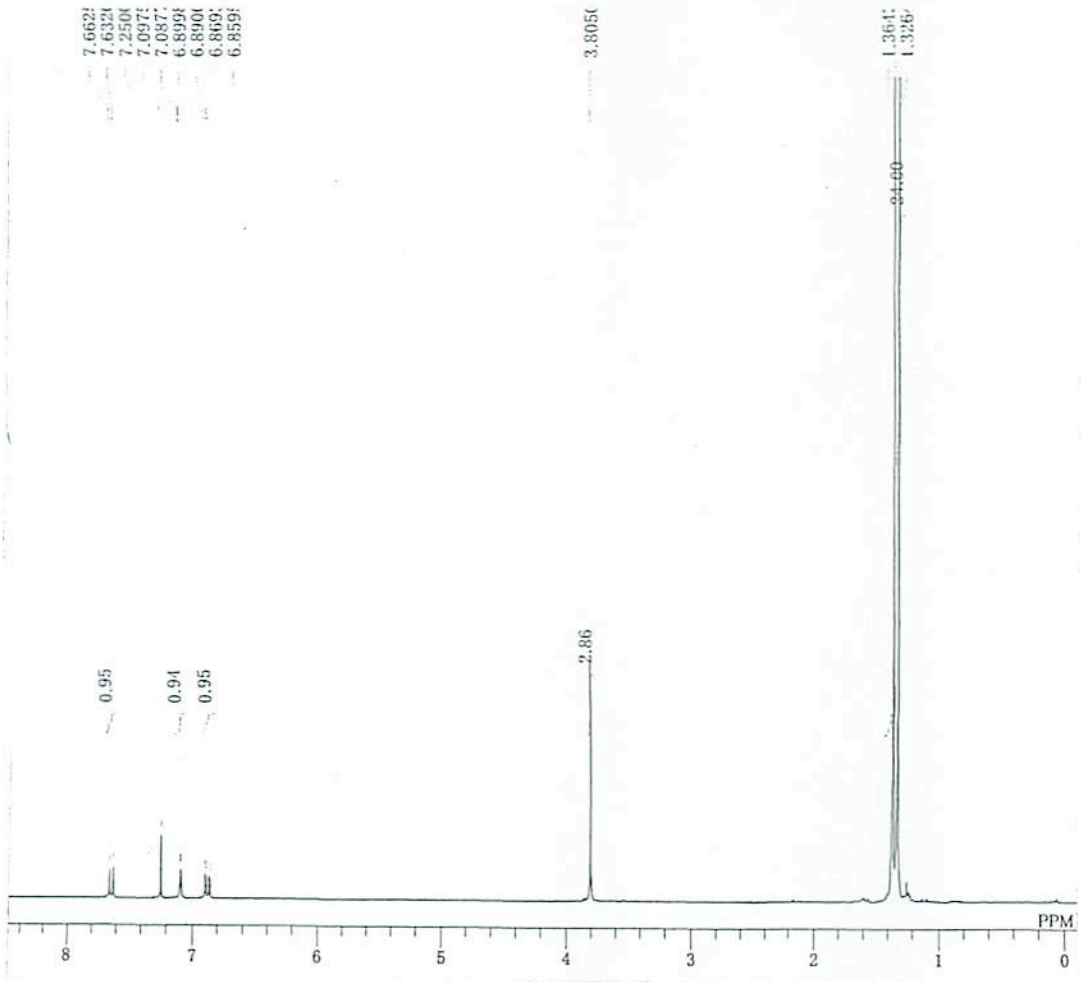
RGAIN	28



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CTEMP 11.2 c
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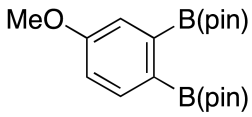
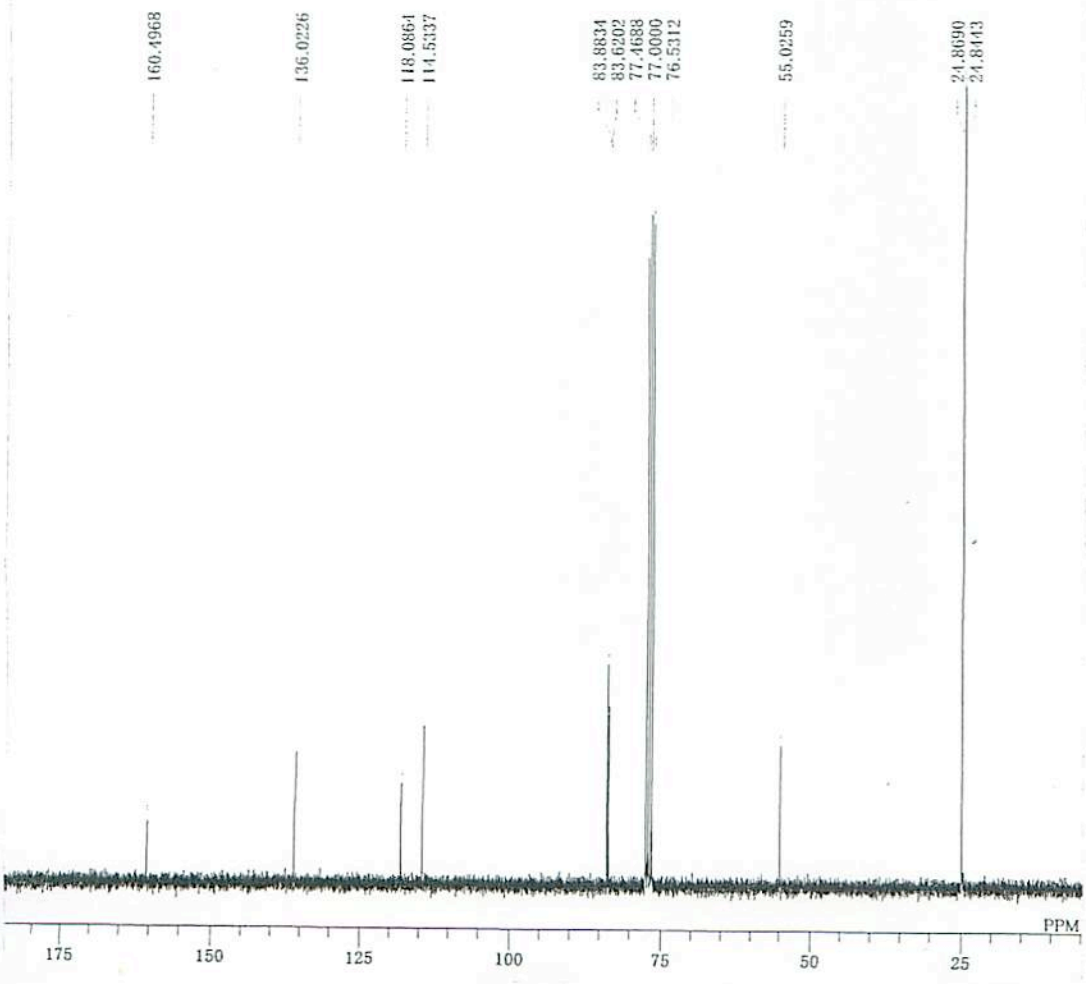
EXREF 7.25 ppm
BF 0.12 Hz
RGAIN 18



2b



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FREQU 18306.64 Hz
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CTEMP 11.6 c
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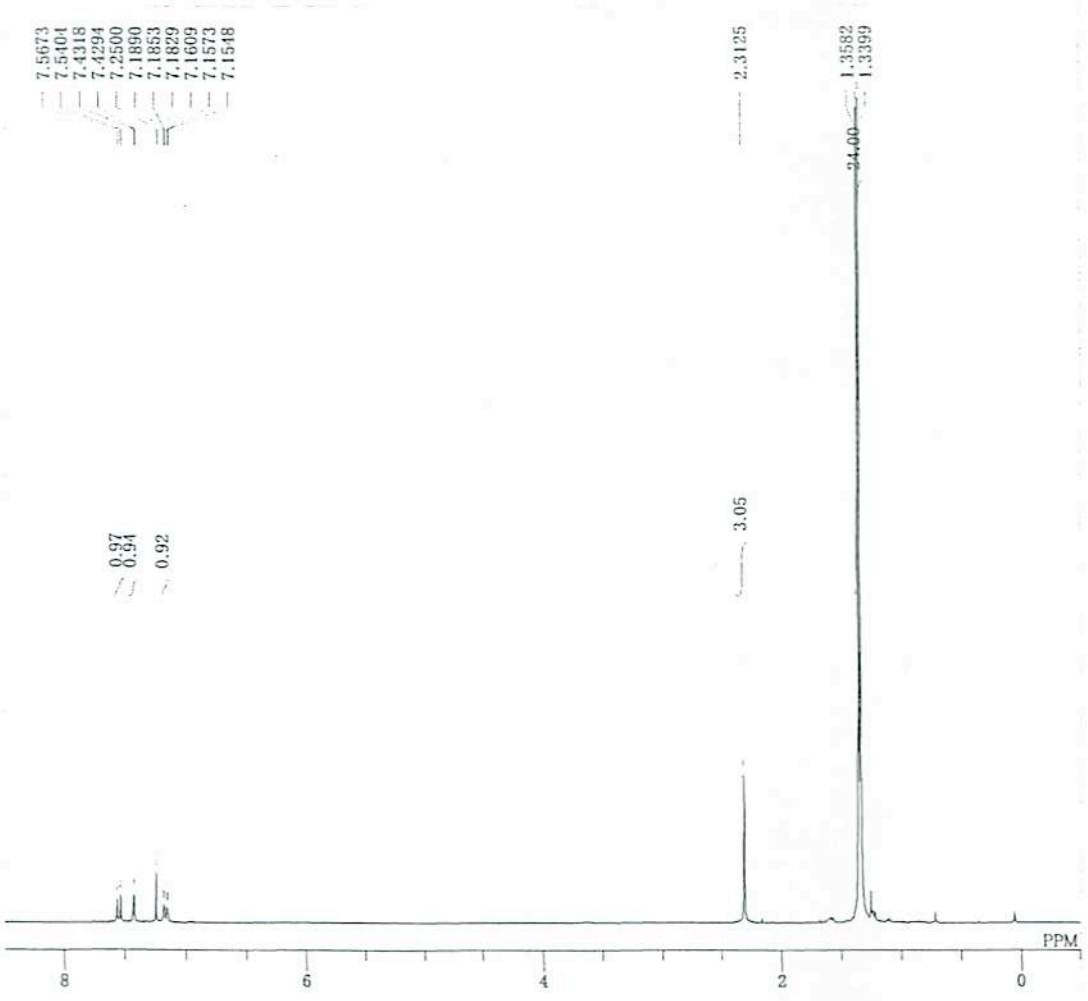
RGAIN 27



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ACQTM	3.0331 sec
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IRNUC	1H
CTEMP	13.3 c
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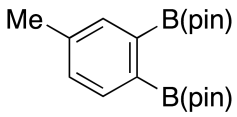
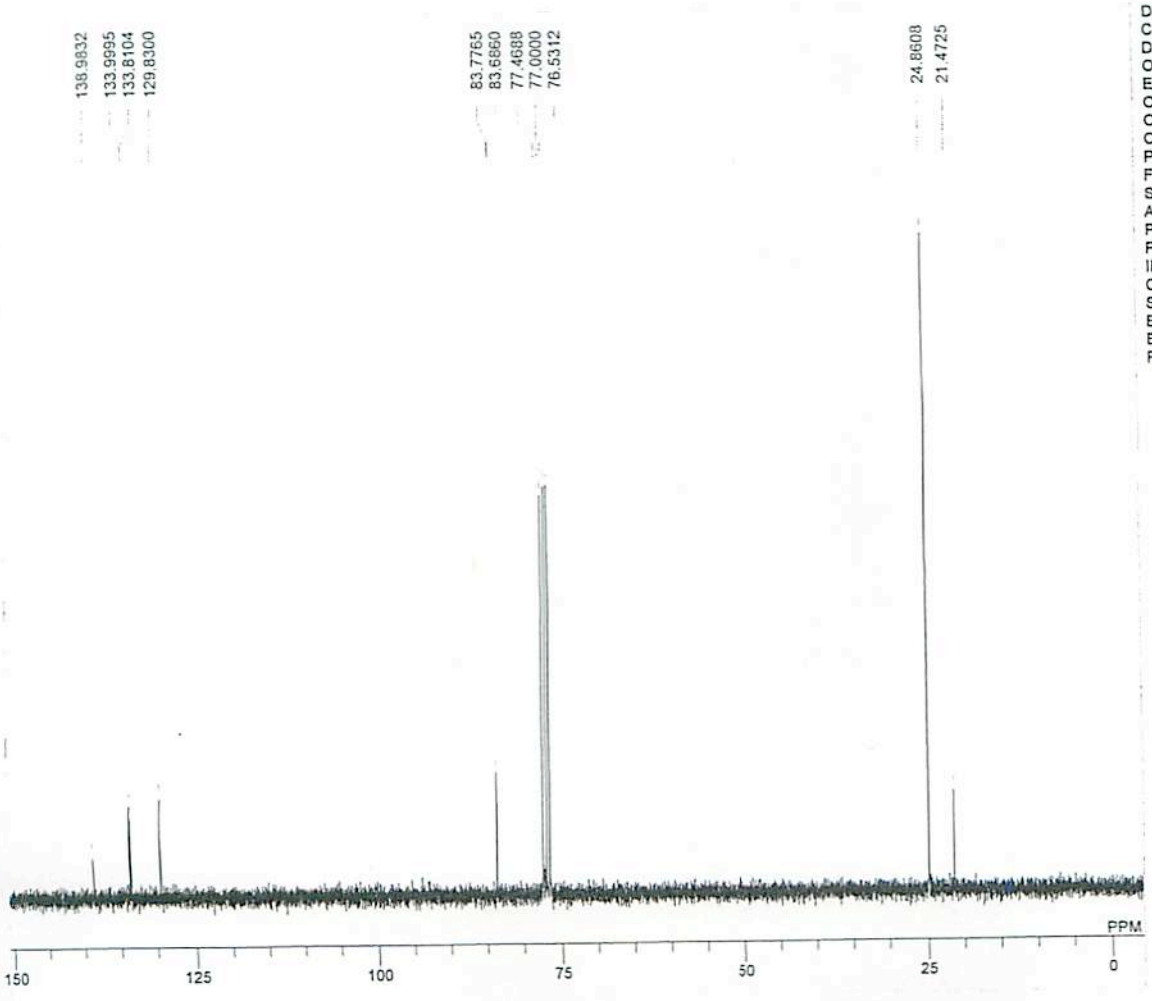
RGAIN	17



2c



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PD	1.2100 sec
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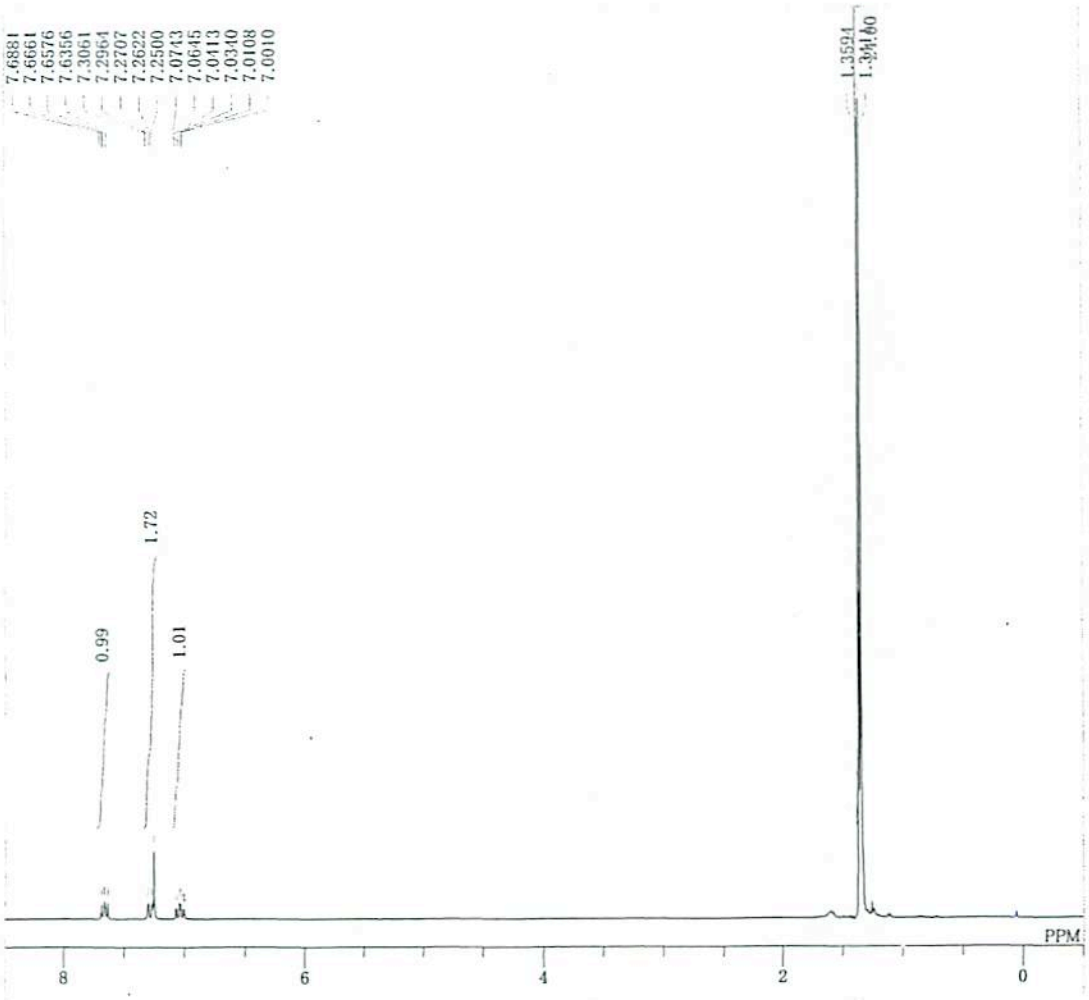
RGAIN	27



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EXMOD NON
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OBSET 112.00 KHz
OBFIN 5800.00 Hz
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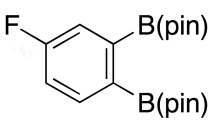
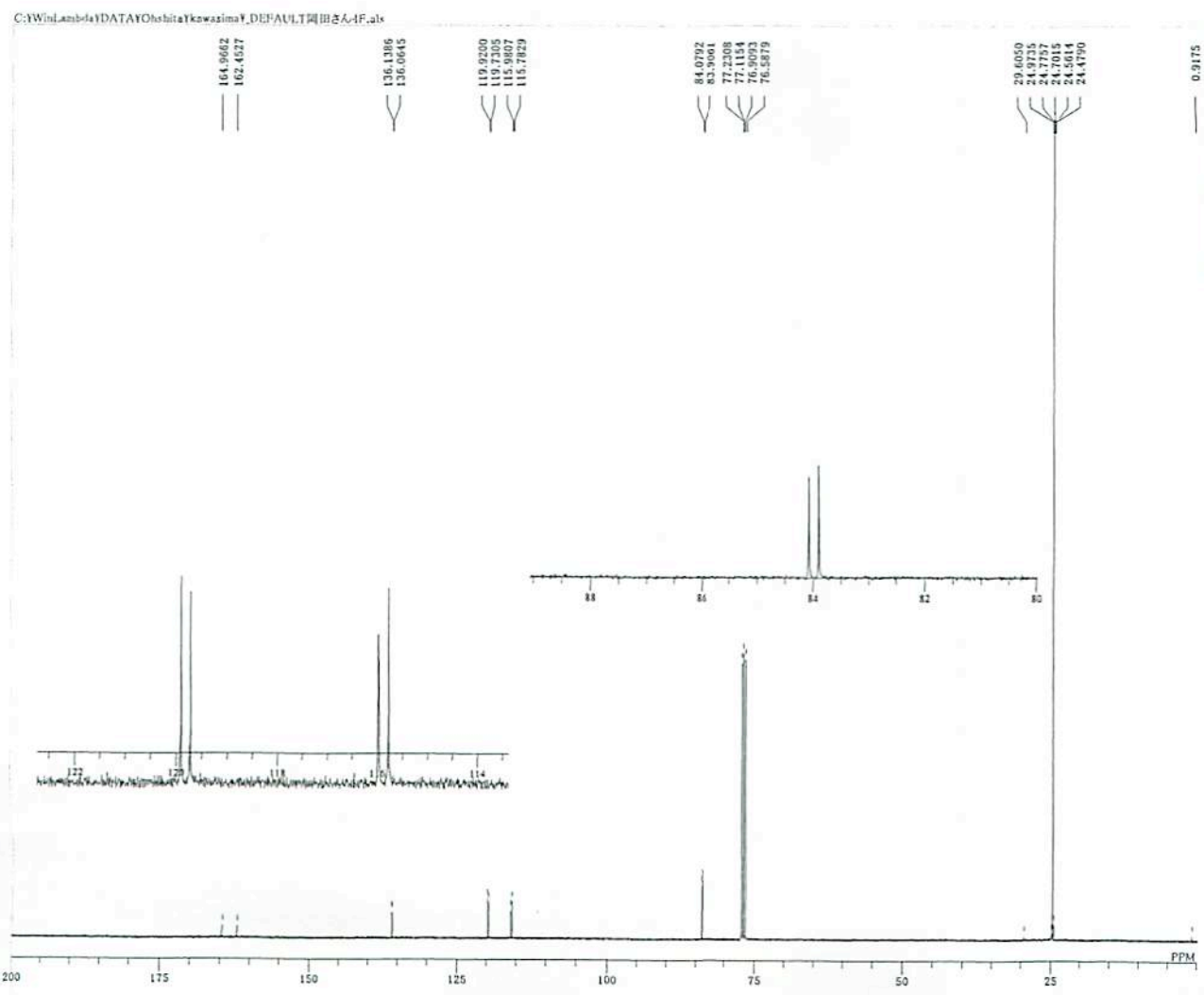
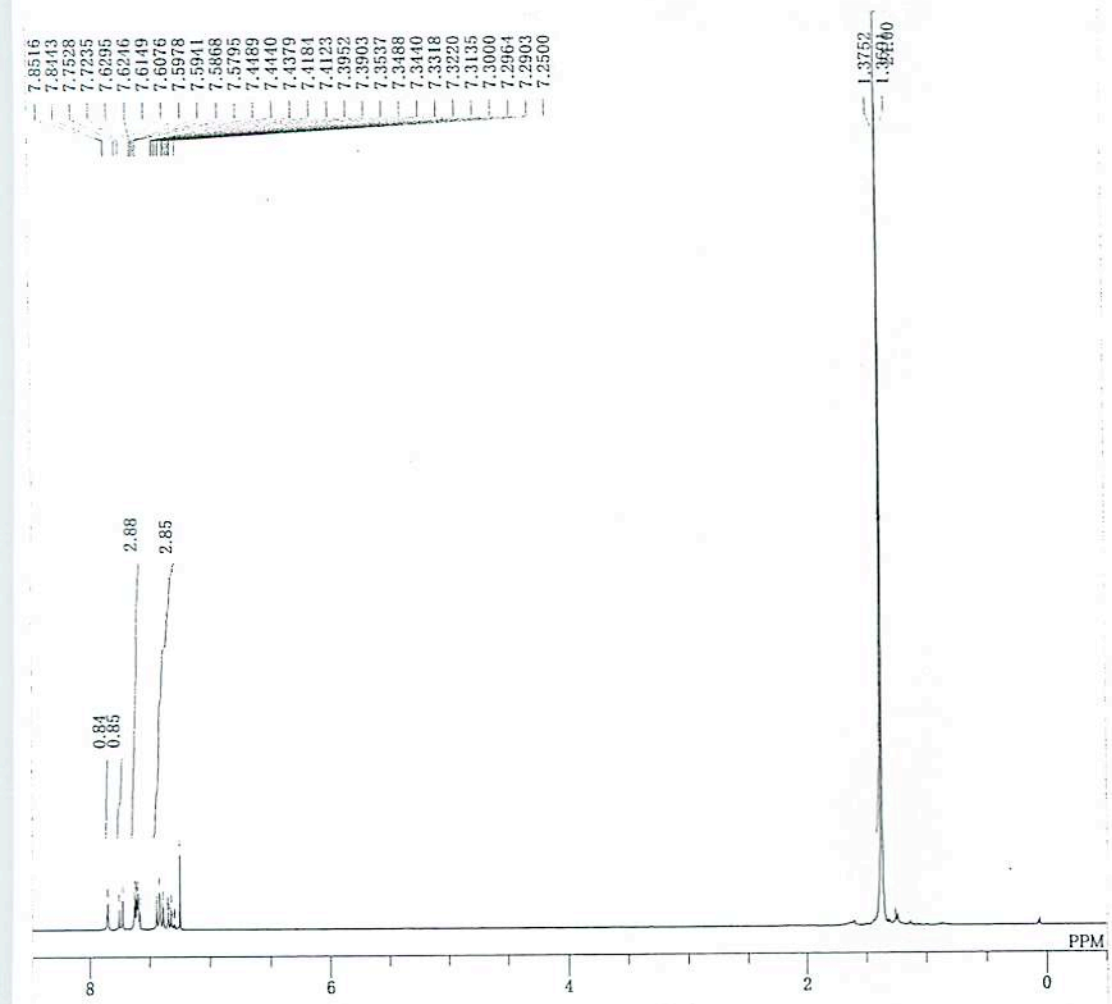
FREQU 5401.76 Hz
SCANS 8
ACQTM 3.0331 sec
PD 3.9670 sec
PW1 5.40 usec
IRNUC 1H
CTEMP 11.1 c
SLVNT CDCL3
EXREF 7.25 ppm
BF 0.12 Hz
RGAIN 18



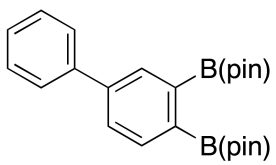
2d



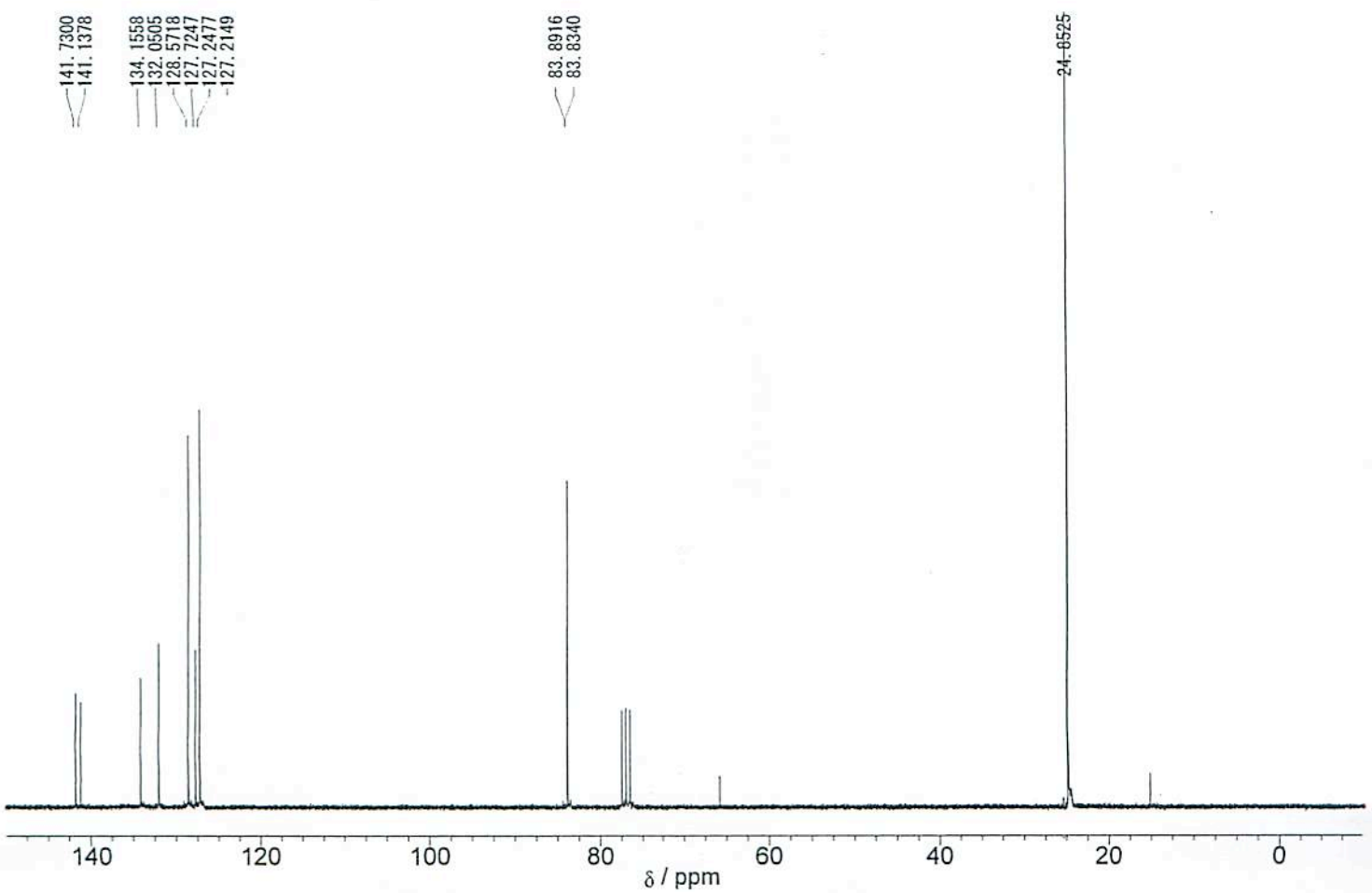
DFILE _DEFAULT岡田さん4F.als
COMNT
DATIM
OBNUC 13C
EXMOD bcm
OBFRQ 99.45 MHz
OBSET 0.00 KHz
OBFIN 104750.00 Hz
POINT 32768
FREQU 26881.72 Hz
SCANS 12342
ACQTM 1.2190 sec
PD 1.7810 sec
PW1 4.25 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT C6D6
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 27

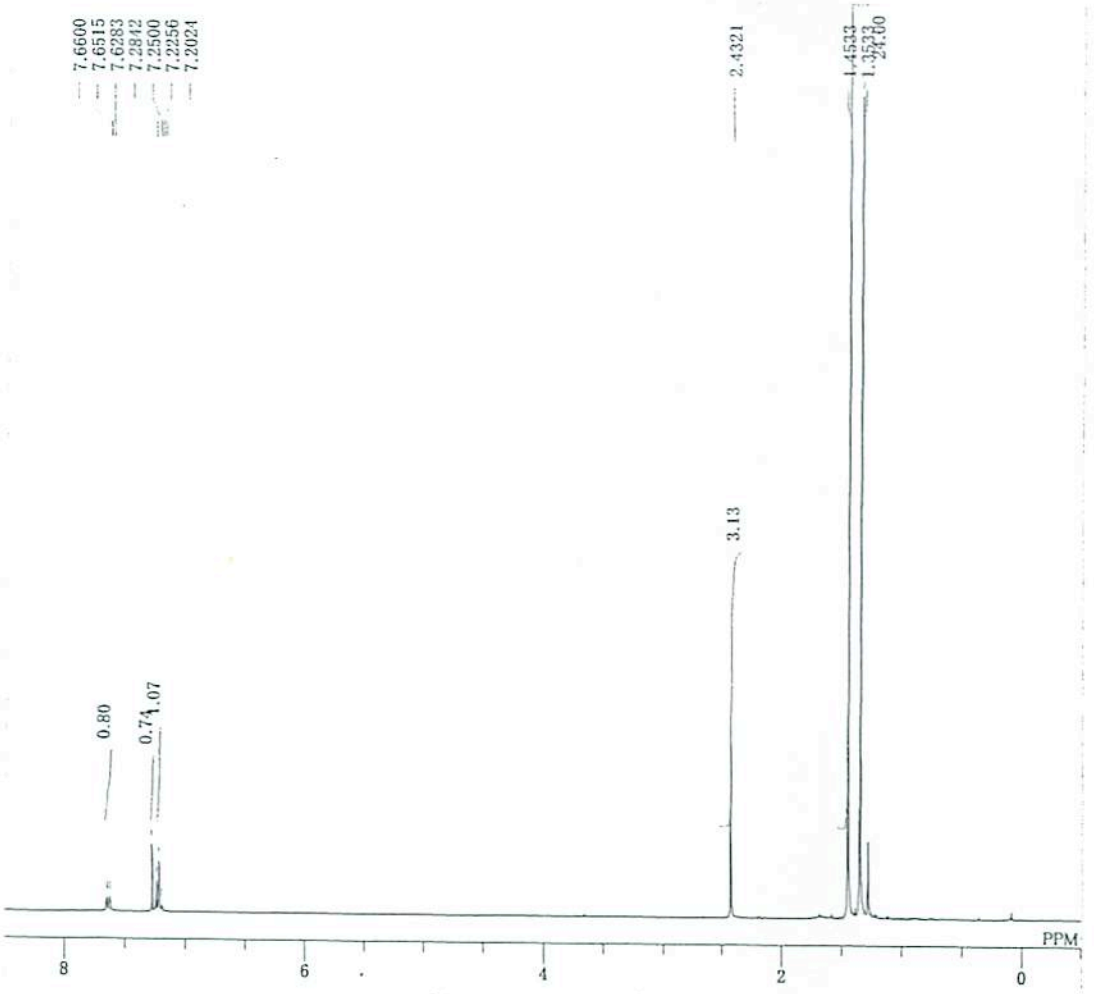


DFILE _DEFAULT.ALS
COMNT
DATIM
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 16384
FREQU 5401.76 Hz
SCANS 8
ACQTM 3.0331 sec
PD 3.9670 sec
PW1 5.40 usec
IRNUC 1H
CTEMP 10.3 c
SLVNT CDCL3
EXREF 7.25 ppm
BF 0.12 Hz
RGAIN 18

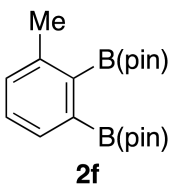


2e

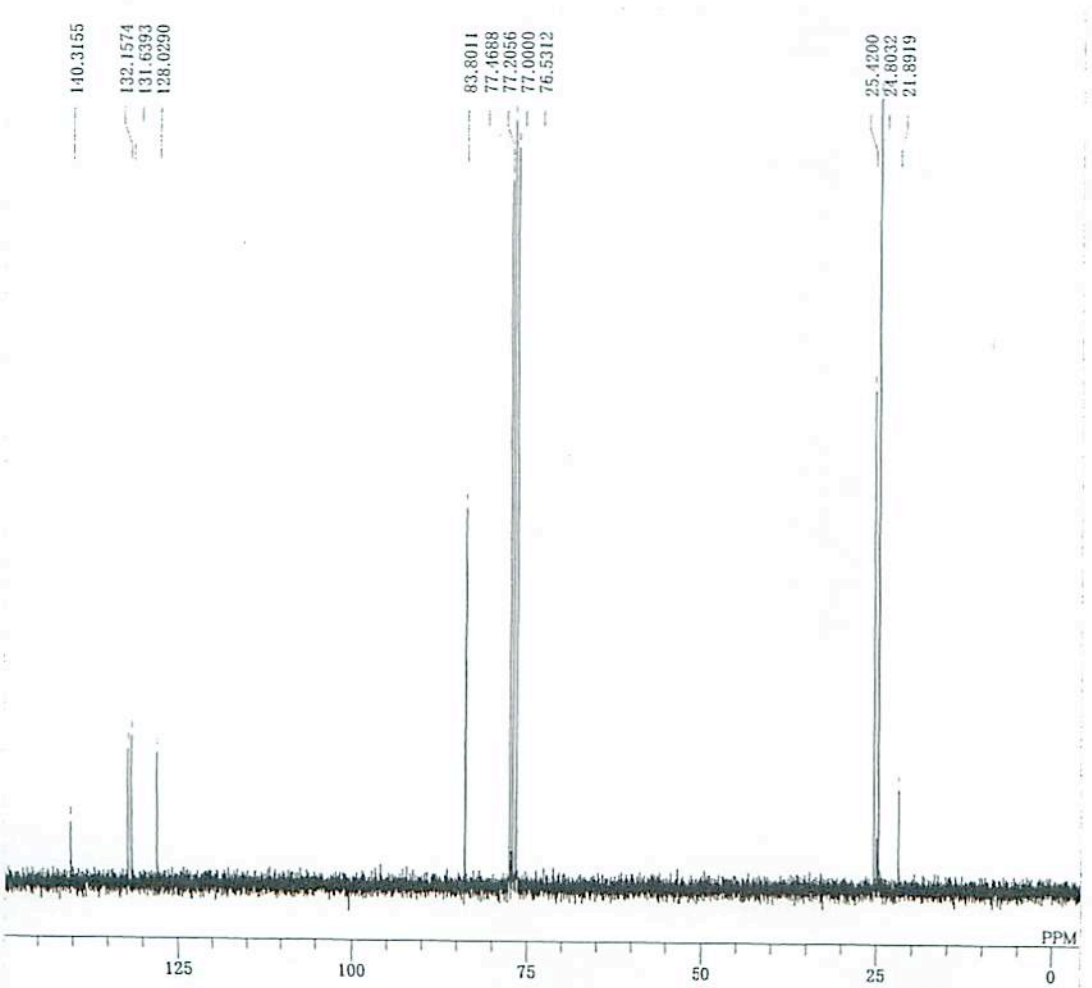




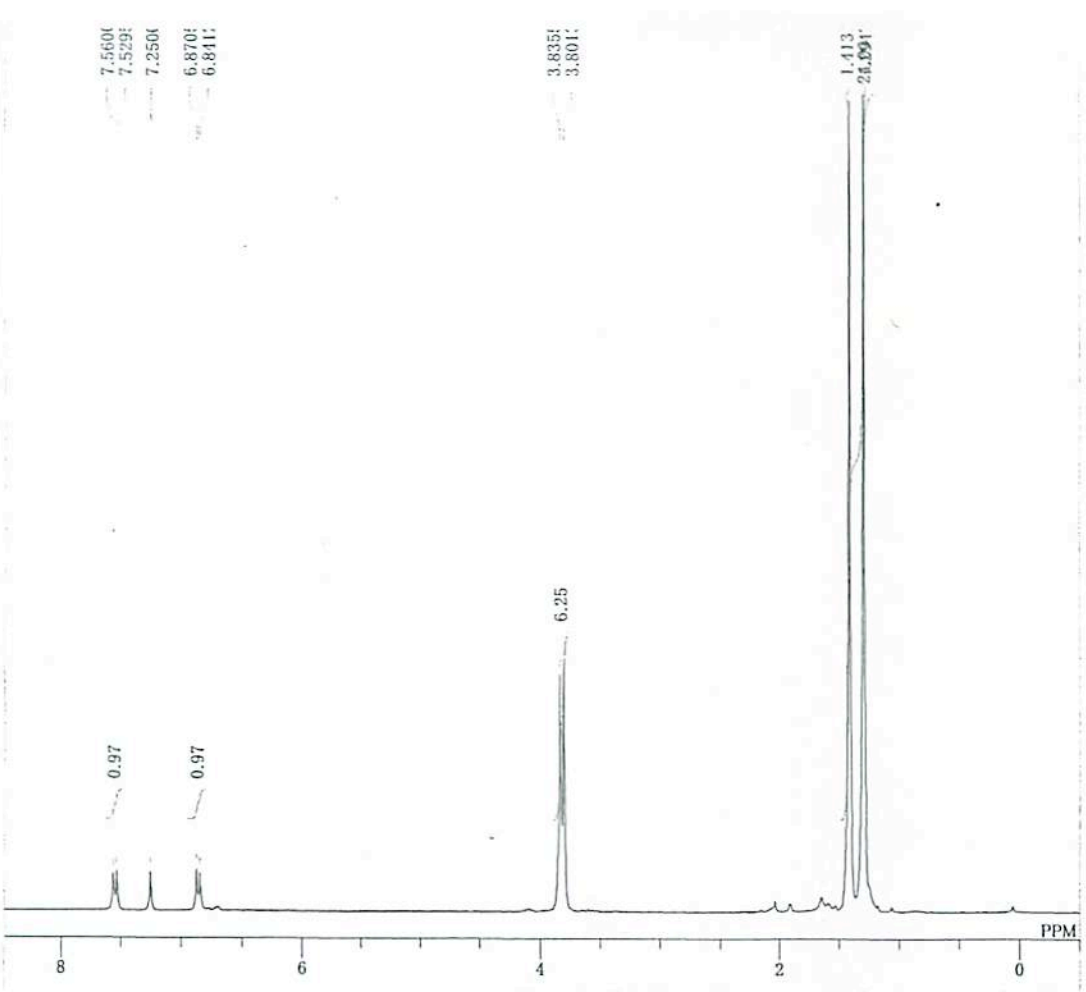
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	8
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	13.2 c
SLVNT	CDCL3
EXREF	7.25 ppm
BF	0.12 Hz
RGAIN	18



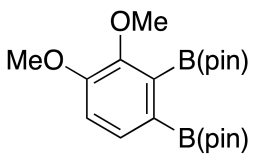
2f



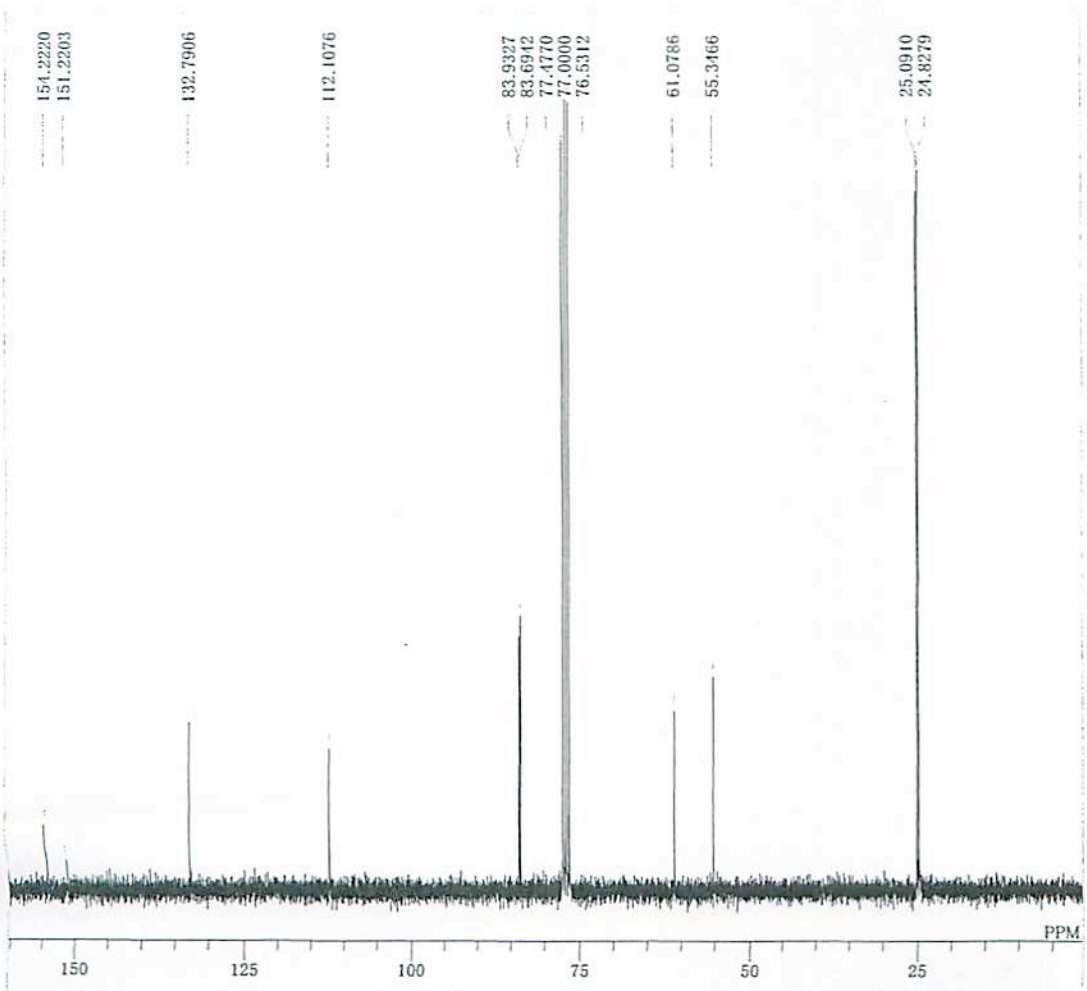
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	1027
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	14.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	27



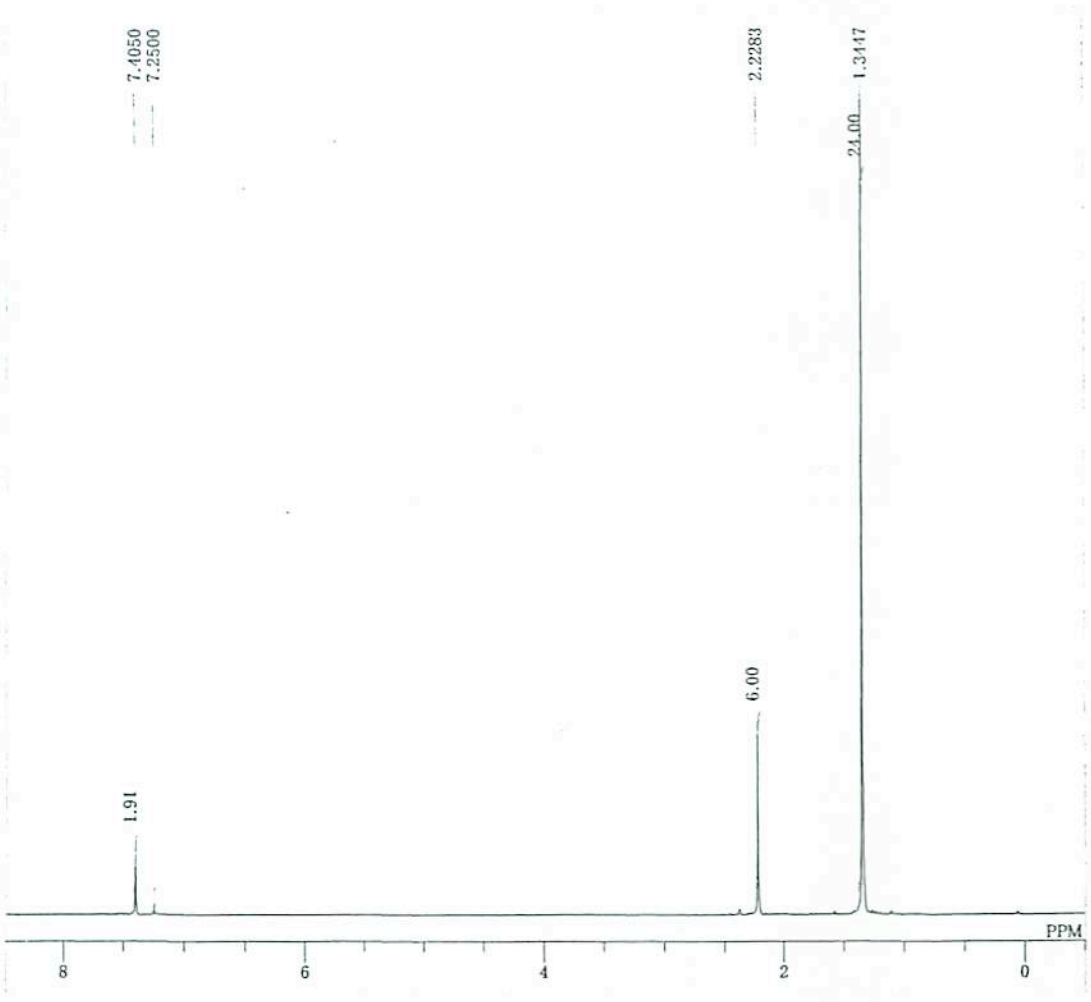
COMNT
DATIM
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 16384
FREQU 5401.76 Hz
SCANS 8
ACQTM 3.0331 sec
PD 3.9670 sec
PW1 5.40 usec
IRNUC 1H
CTEMP 12.4 c
SLVNT CDCL3
EXREF 7.25 ppm
BF 0.12 Hz
RGAIN 17



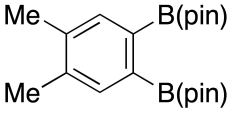
2g



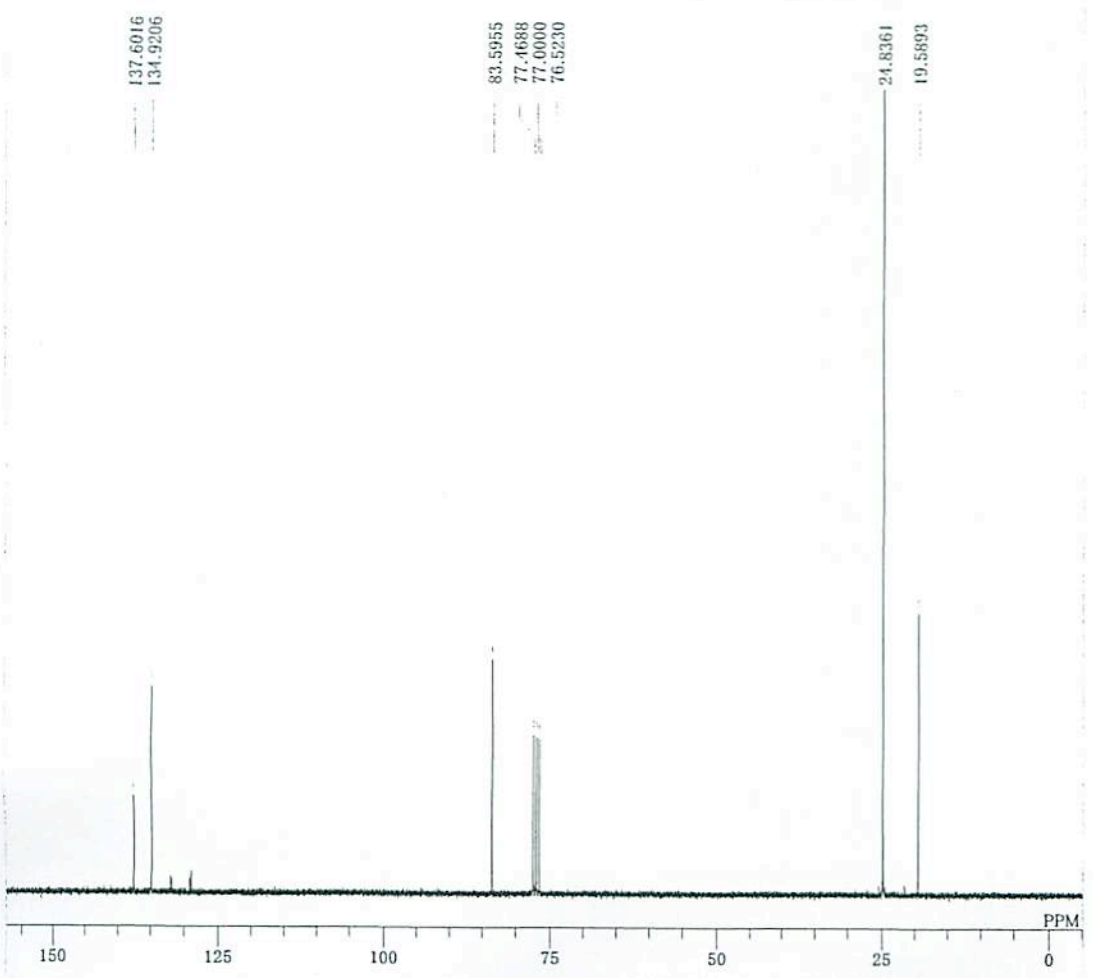
DFILE _DEFAULT.ALS
COMNT
DATIM
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.00 Hz
POINT 32768
FREQU 18306.64 Hz
SCANS 1265
ACQTM 1.7900 sec
PD 1.2100 sec
PW1 3.50 usec
IRNUC 1H
CTEMP 14.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 25



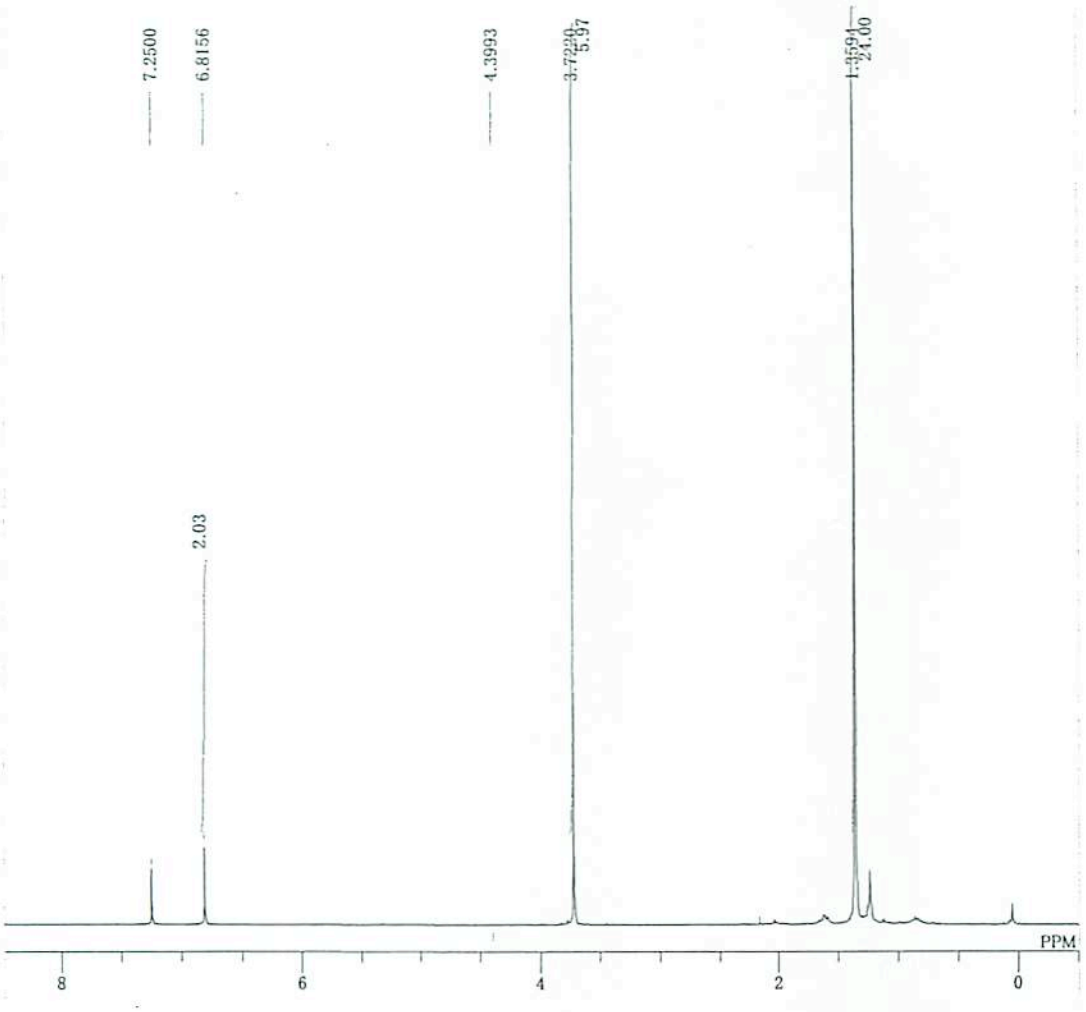
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	8
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	10.8 c
SLVNT	CDCL3
EXREF	7.25 ppm
BF	0.12 Hz
RGAIN	13



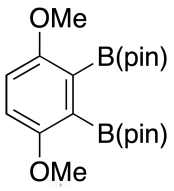
2h



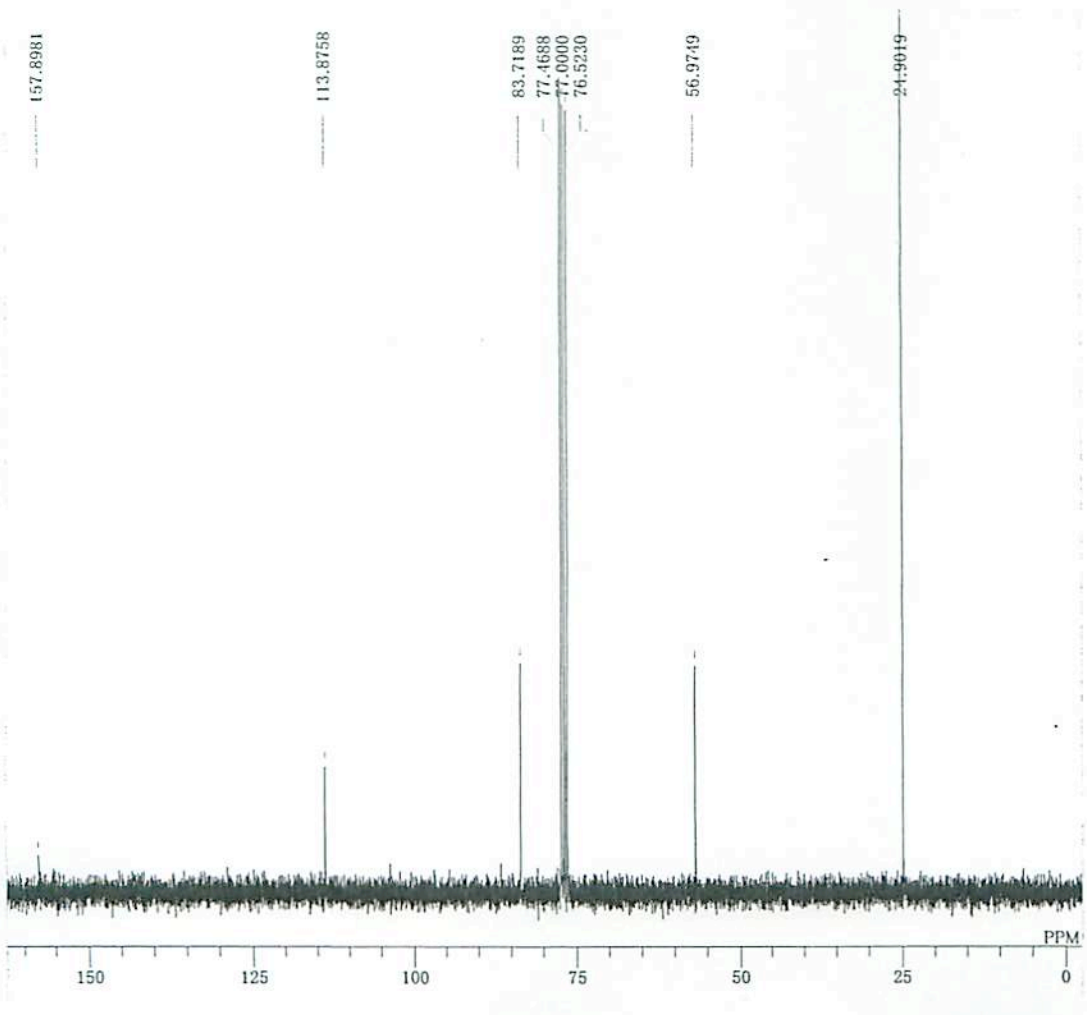
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	863
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	12.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	27



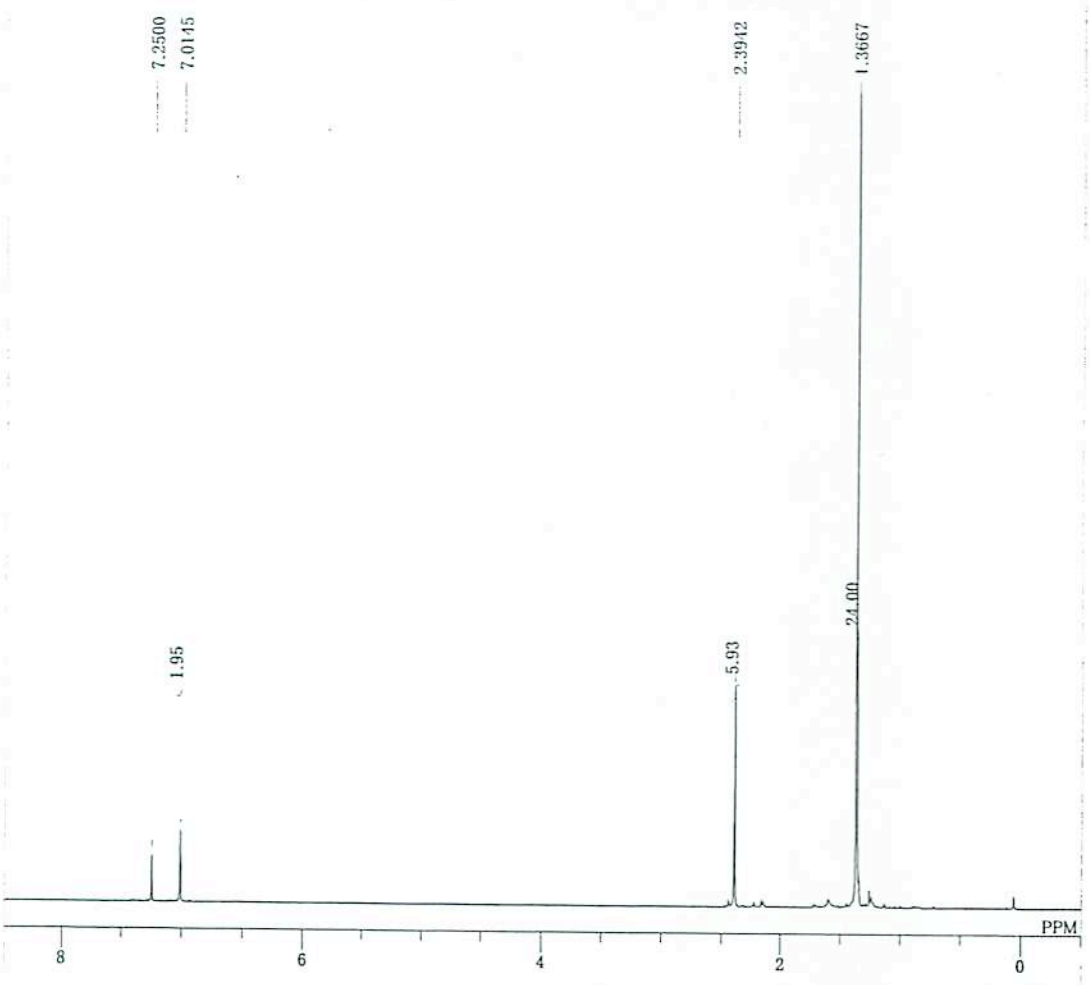
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	8
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	10.3 c
SLVNT	CDCL3
EXREF	7.25 ppm
BF	0.12 Hz
RGAIN	19



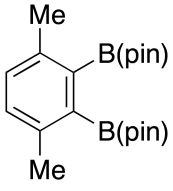
2i



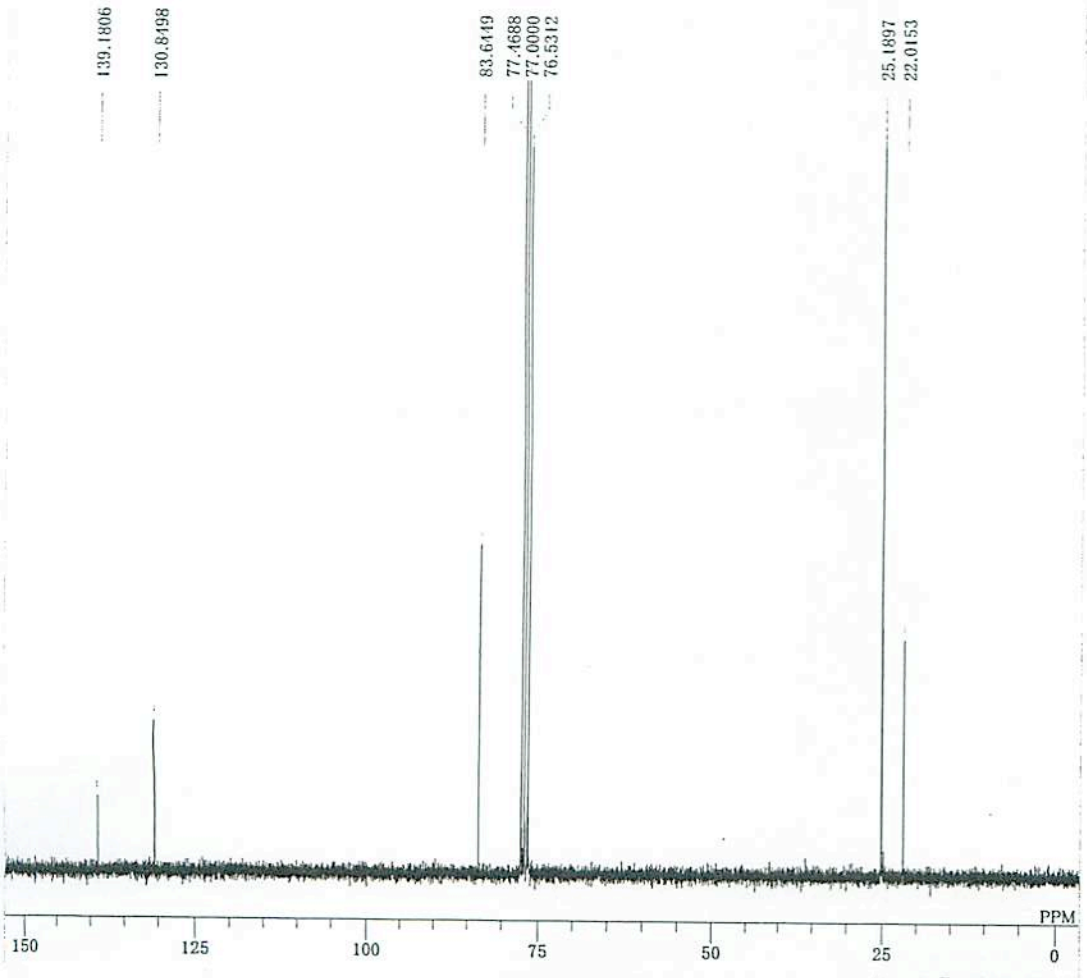
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	743
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	13.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	27



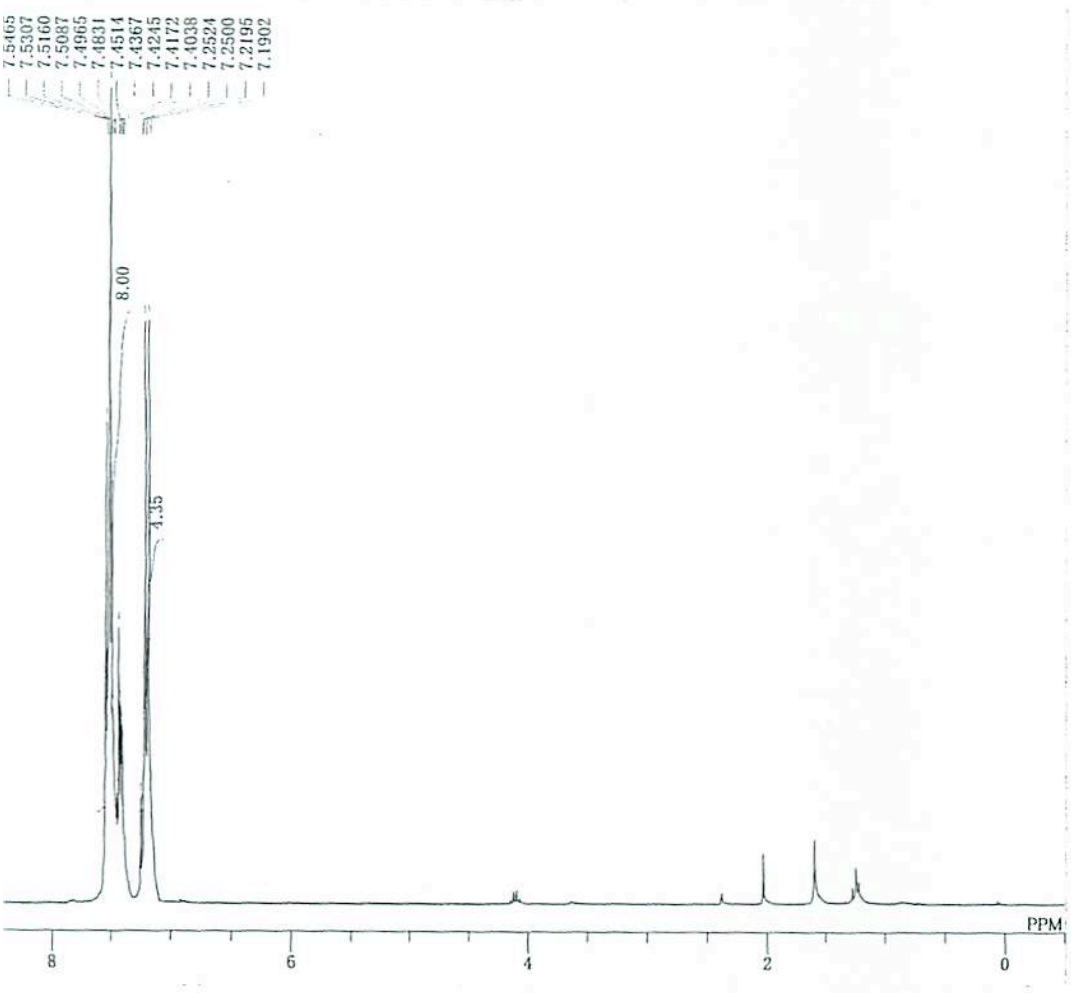
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	8
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	13.1 c
SLVNT	CDCL3
EXREF	7.25 ppm
BF	0.12 Hz
RGAIN	19



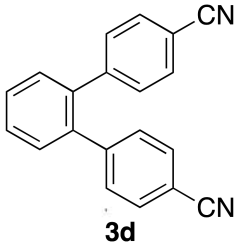
2j



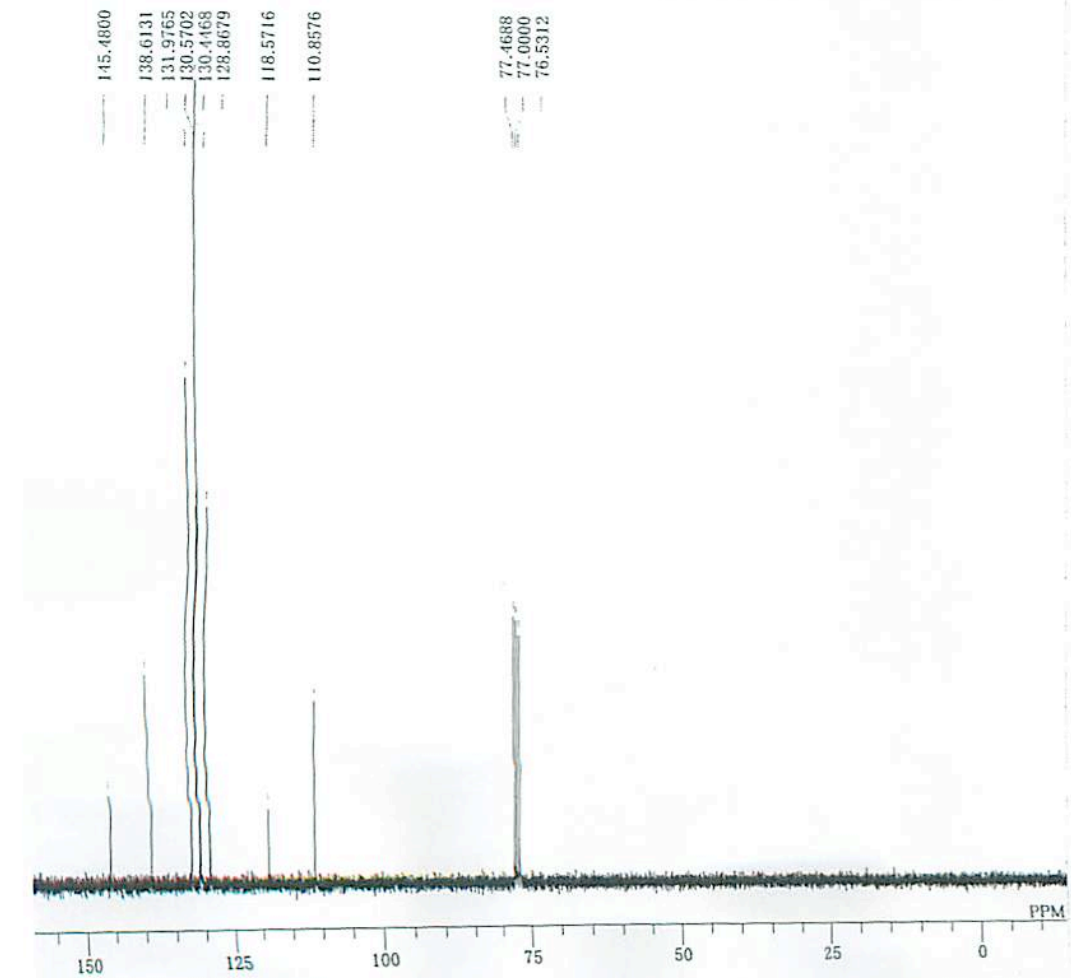
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	1778
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	14.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	27



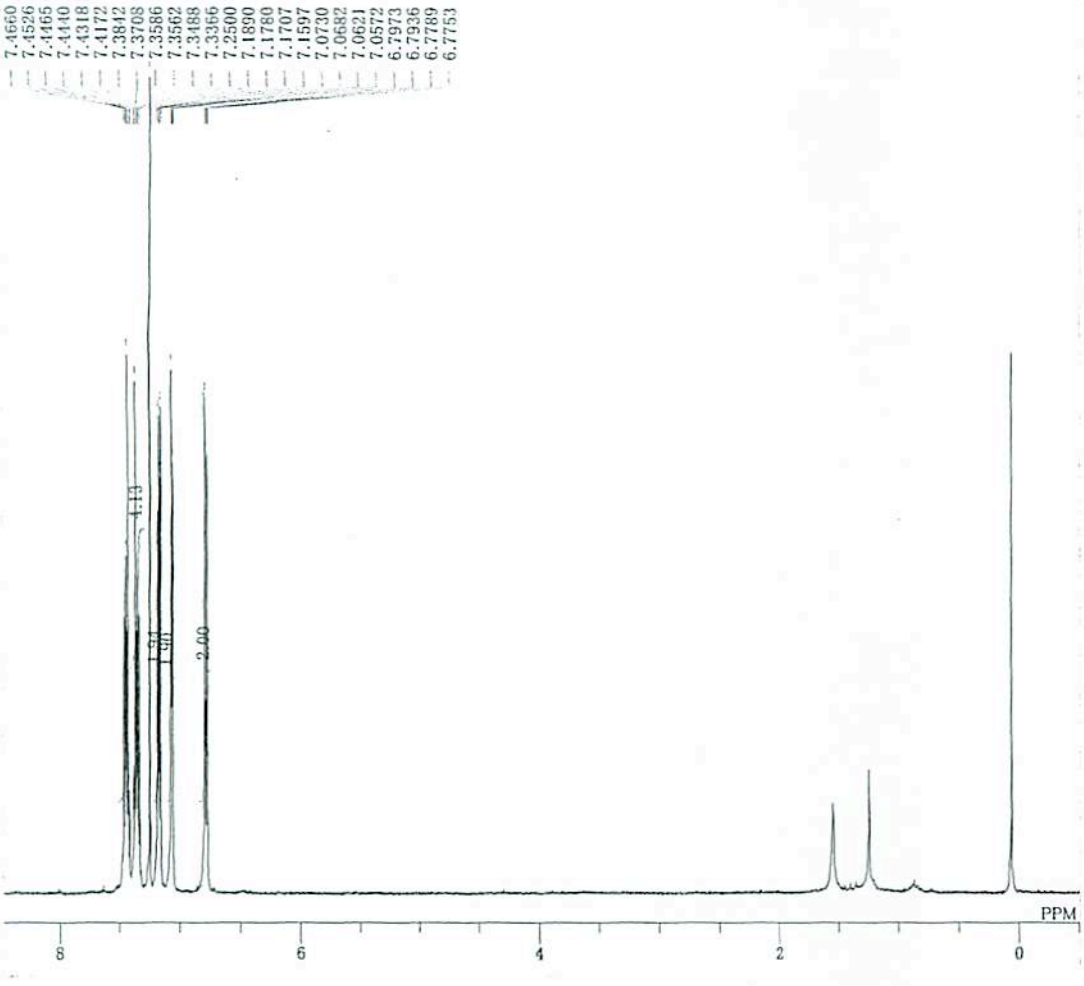
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	16
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	18.5 c
SLVNT	CDCL3
EXREF	7.25 ppm
BF	0.12 Hz
RGAIN	19



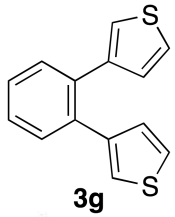
3d



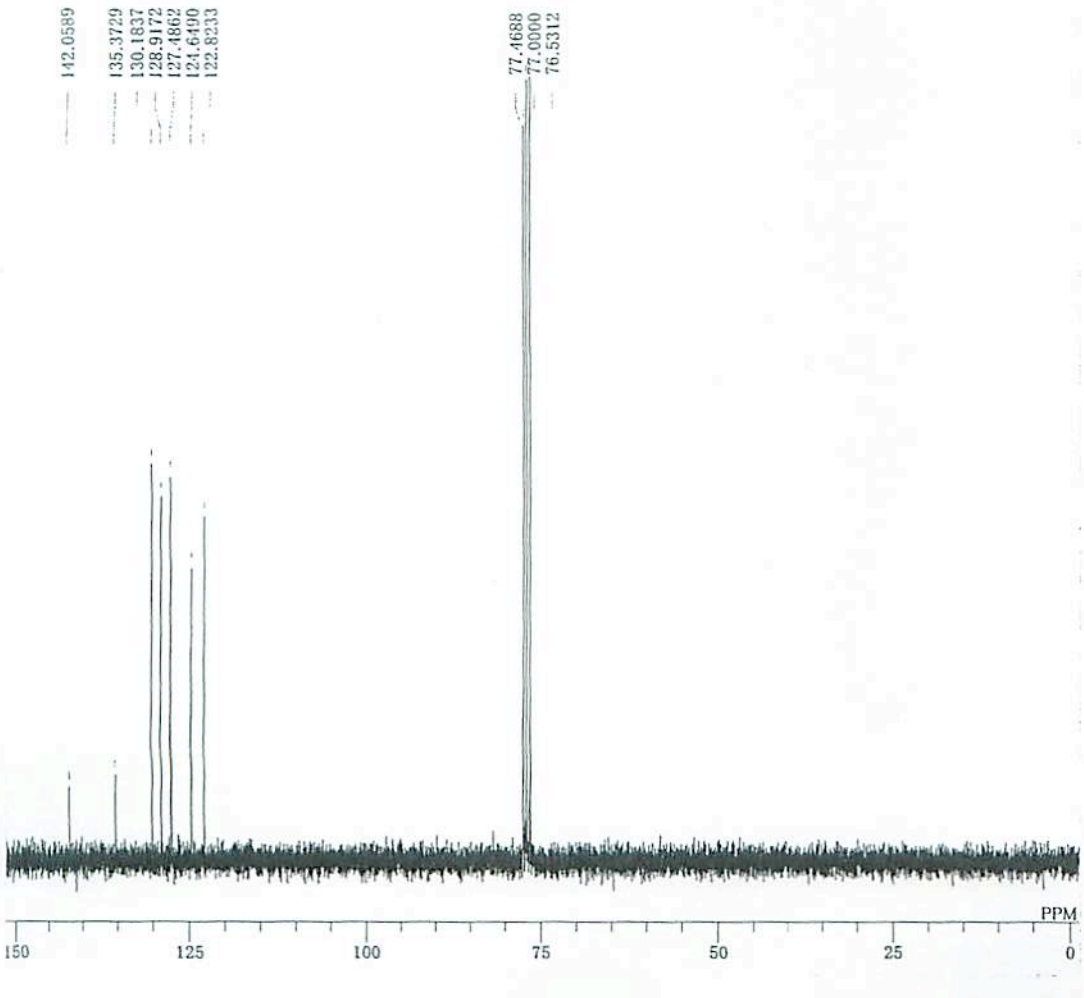
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	609
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	27



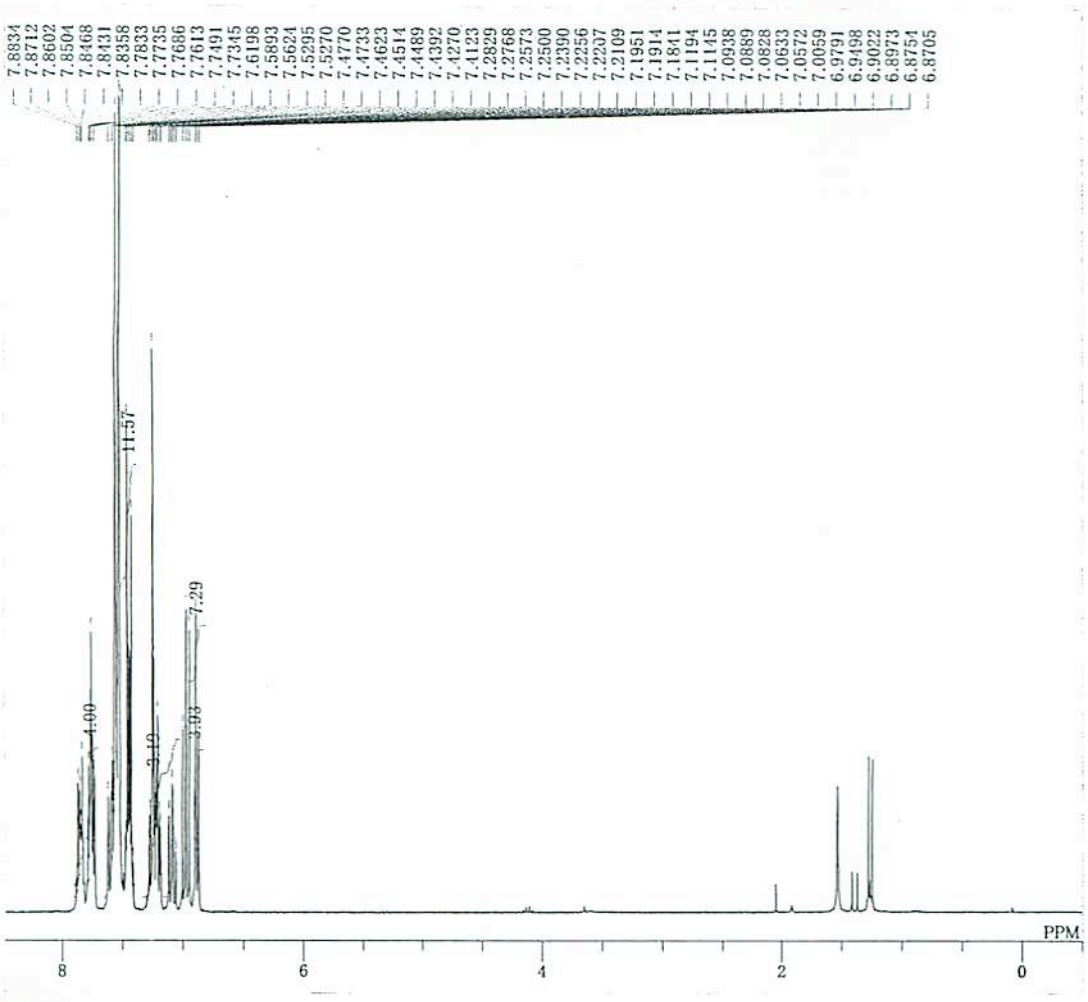
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	8
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	17.7 c
SLVNT	CDCL3
EXREF	7.25 ppm
BF	0.12 Hz
RGAIN	22



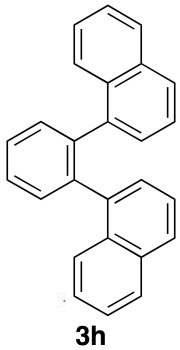
3g



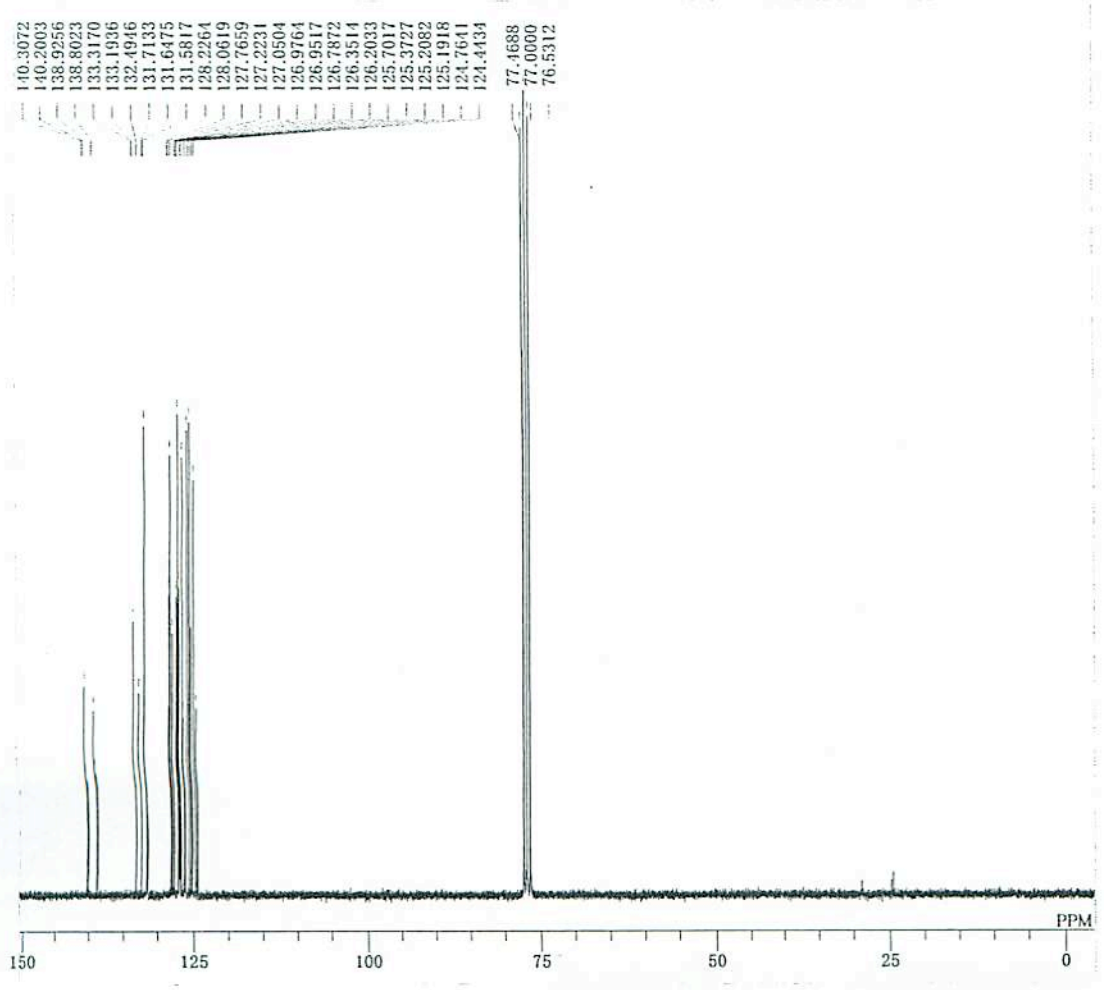
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	882
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	28



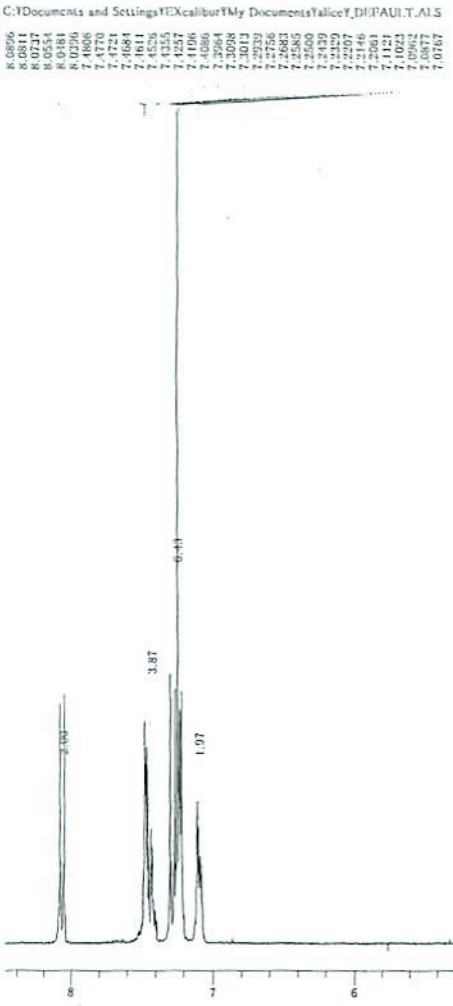
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	8
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	7.25 ppm
BF	0.12 Hz
RGAIN	19



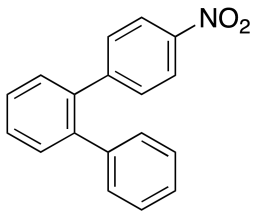
3h



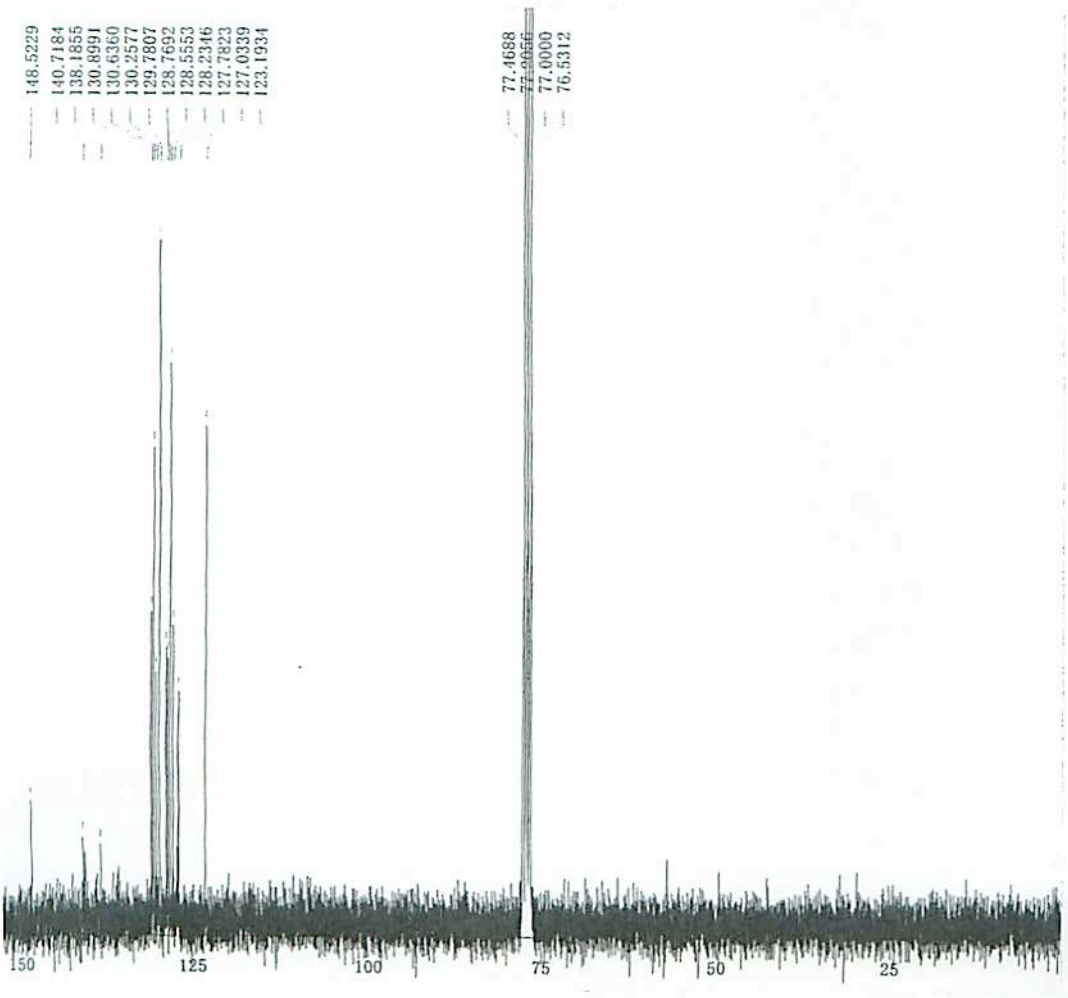
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	11064
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	28



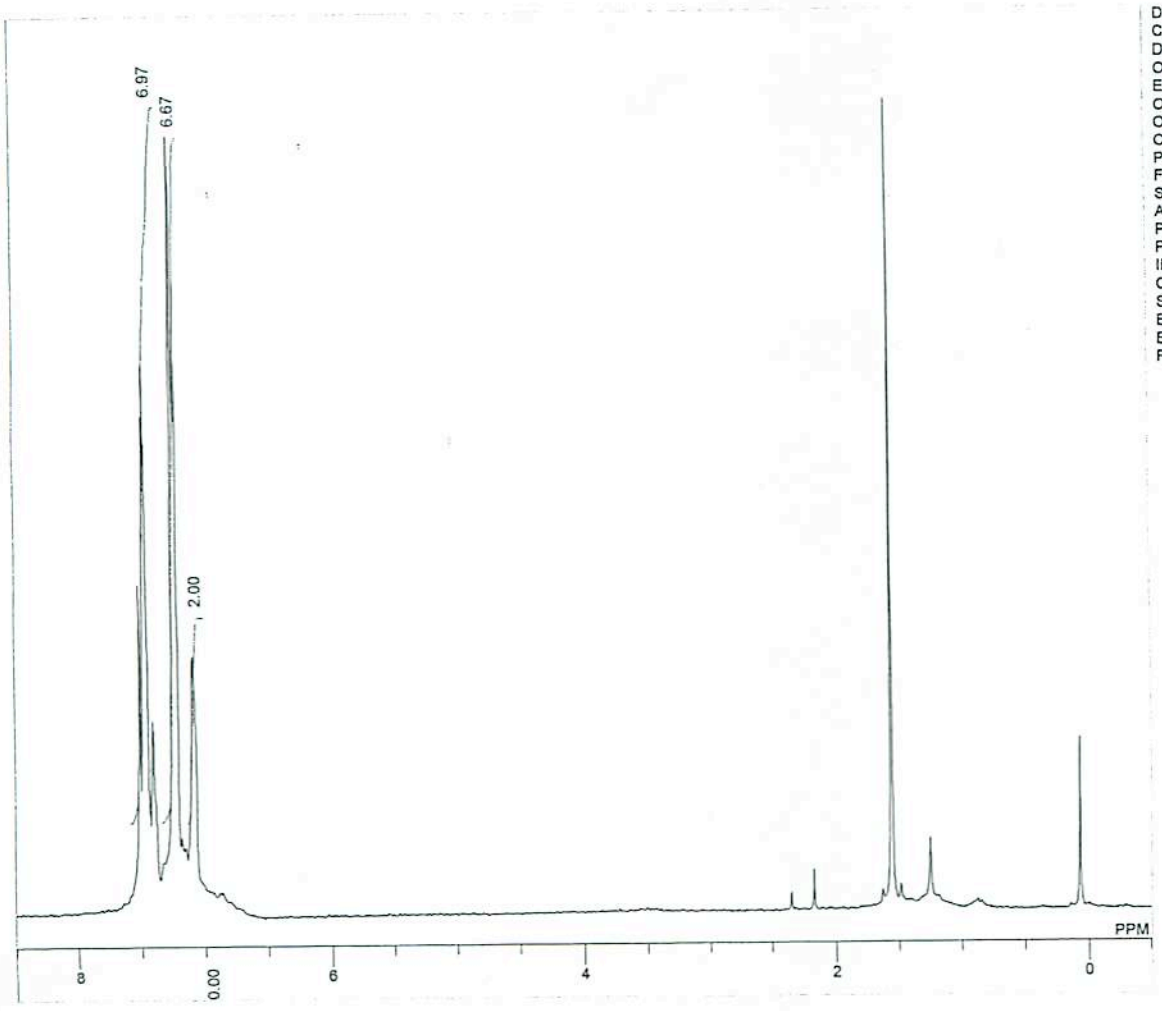
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	8
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	18.2 c
SLVNT	CDCL3
EXREF	7.25 ppm
BF	0.12 Hz
RGAIN	24



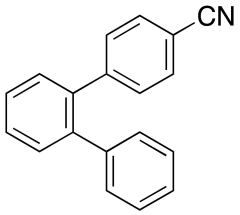
4b



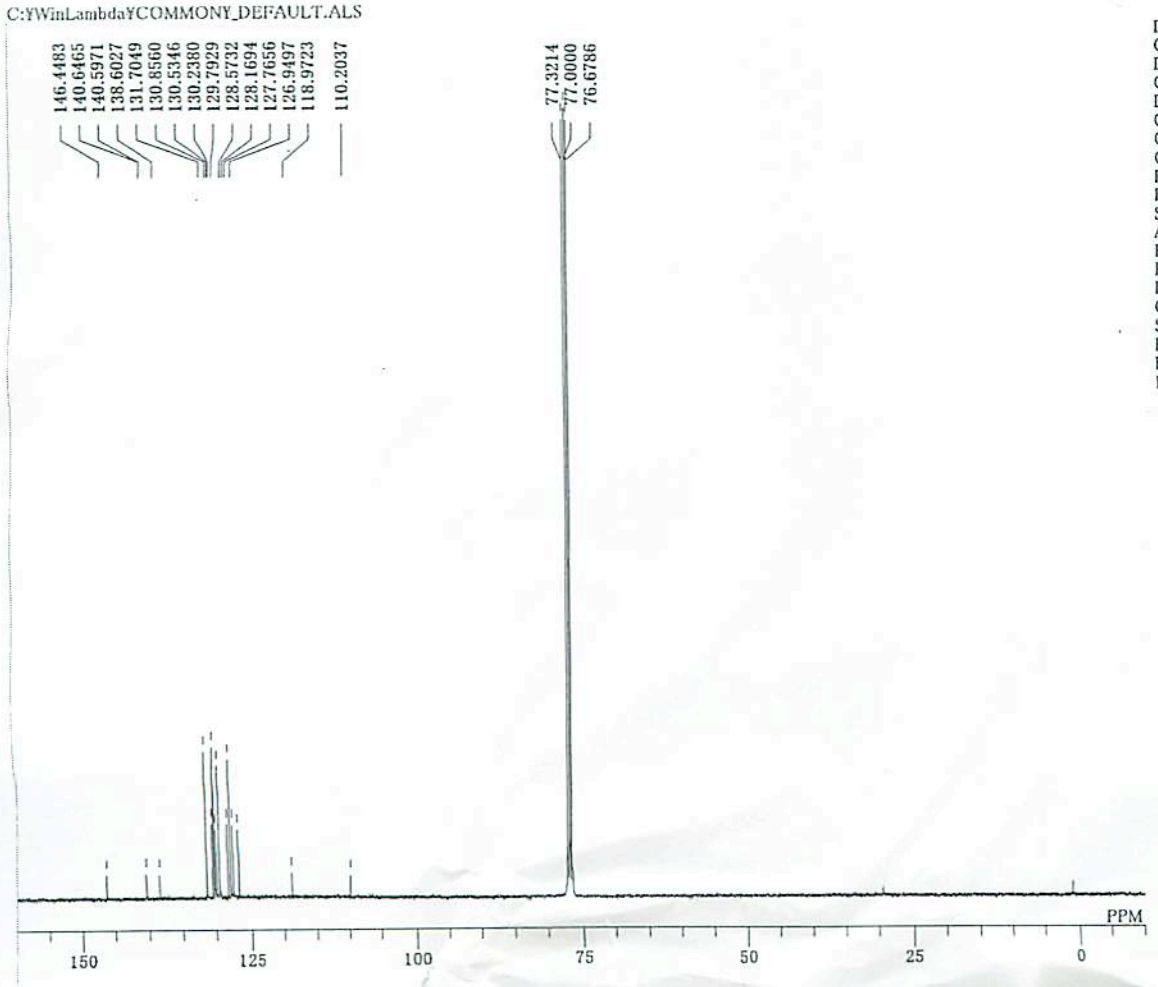
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	11431
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	12.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	27



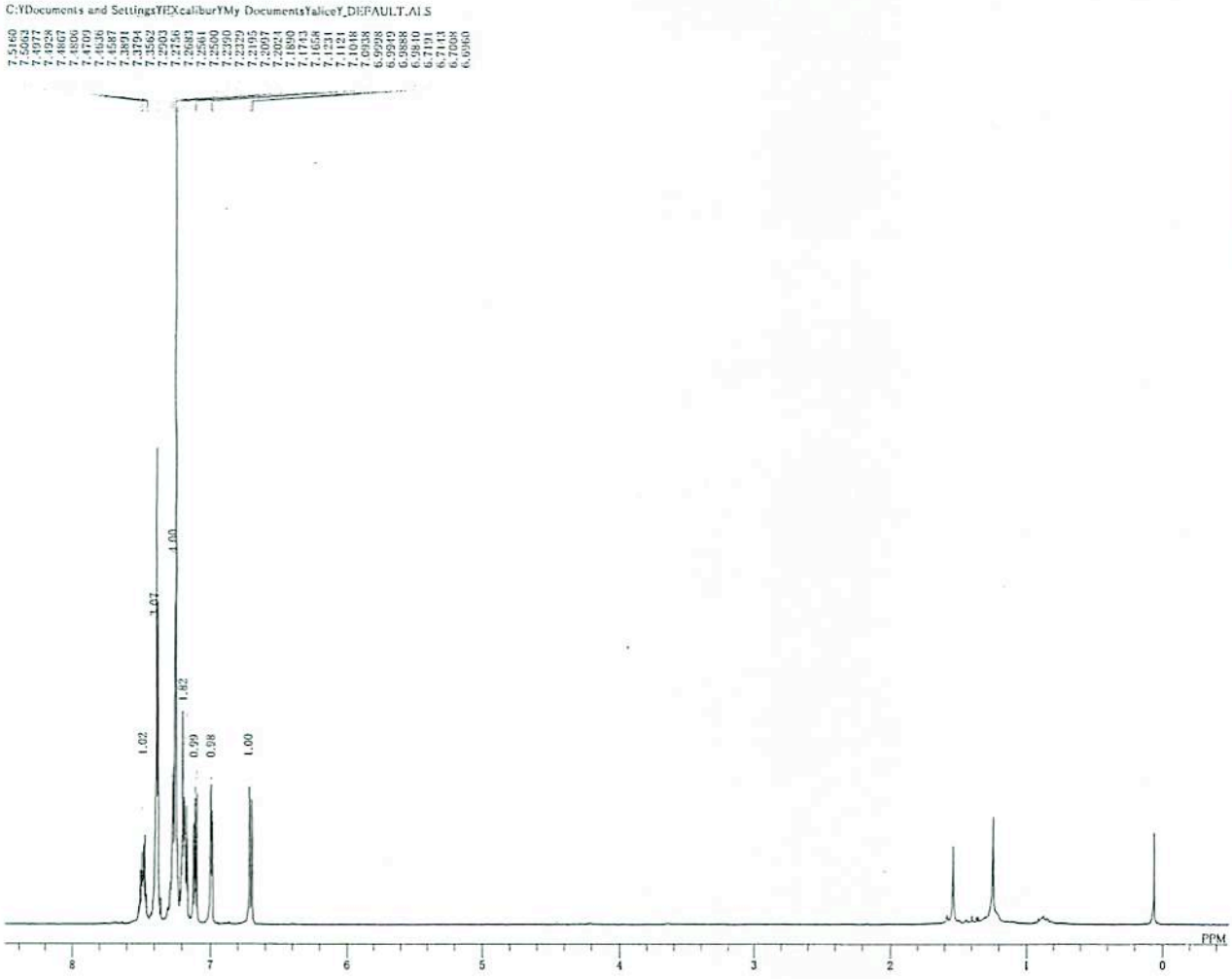
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	16
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	17.6 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	1.62 Hz
RGAIN	25



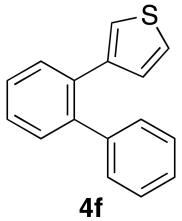
4c



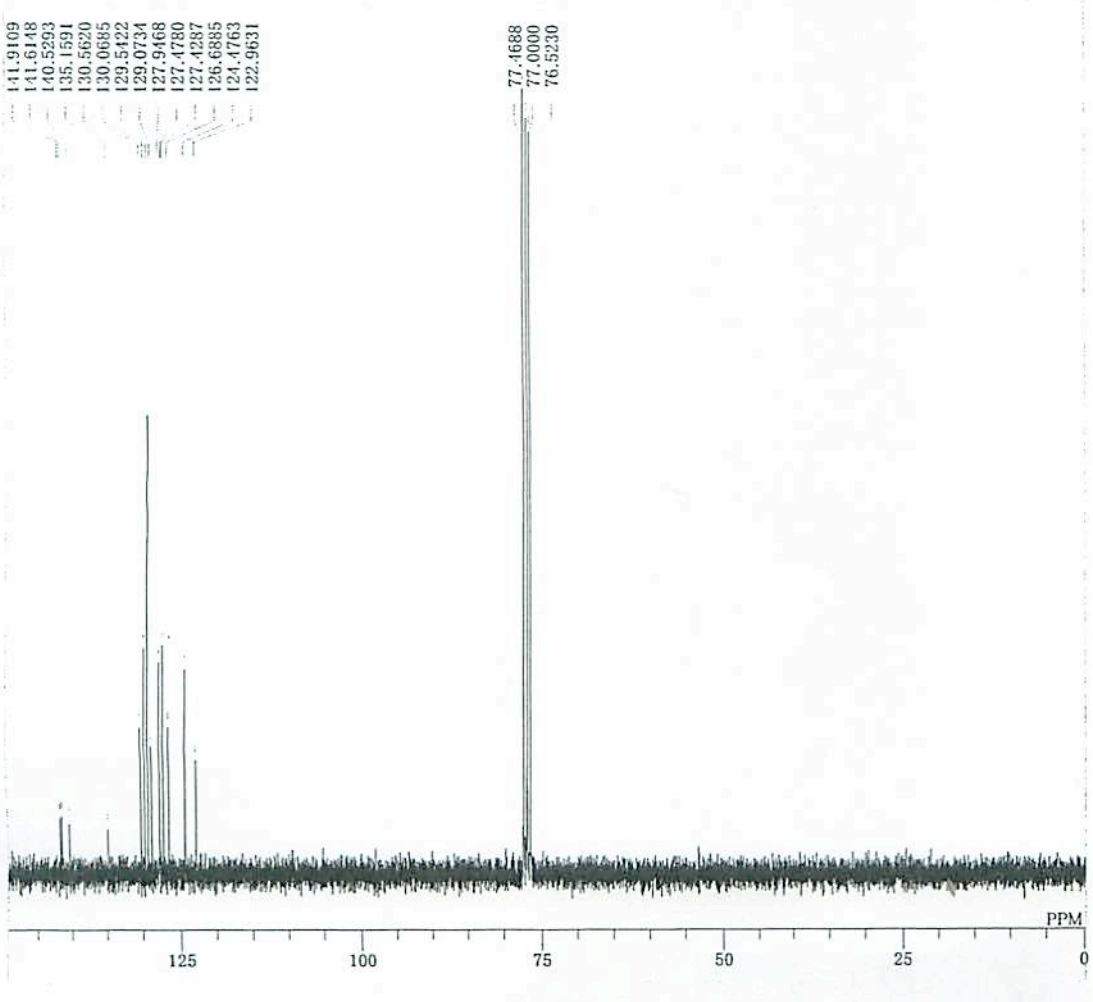
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	13C
EXMOD	bcm
OBFRQ	99.45 MHz
OBSET	0.00 KHz
OBFIN	104750.00 Hz
POINT	32768
FREQU	26881.72 Hz
SCANS	11183
ACQTM	1.2190 sec
PD	1.7810 sec
PW1	4.25 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.62 Hz
RGAIN	27



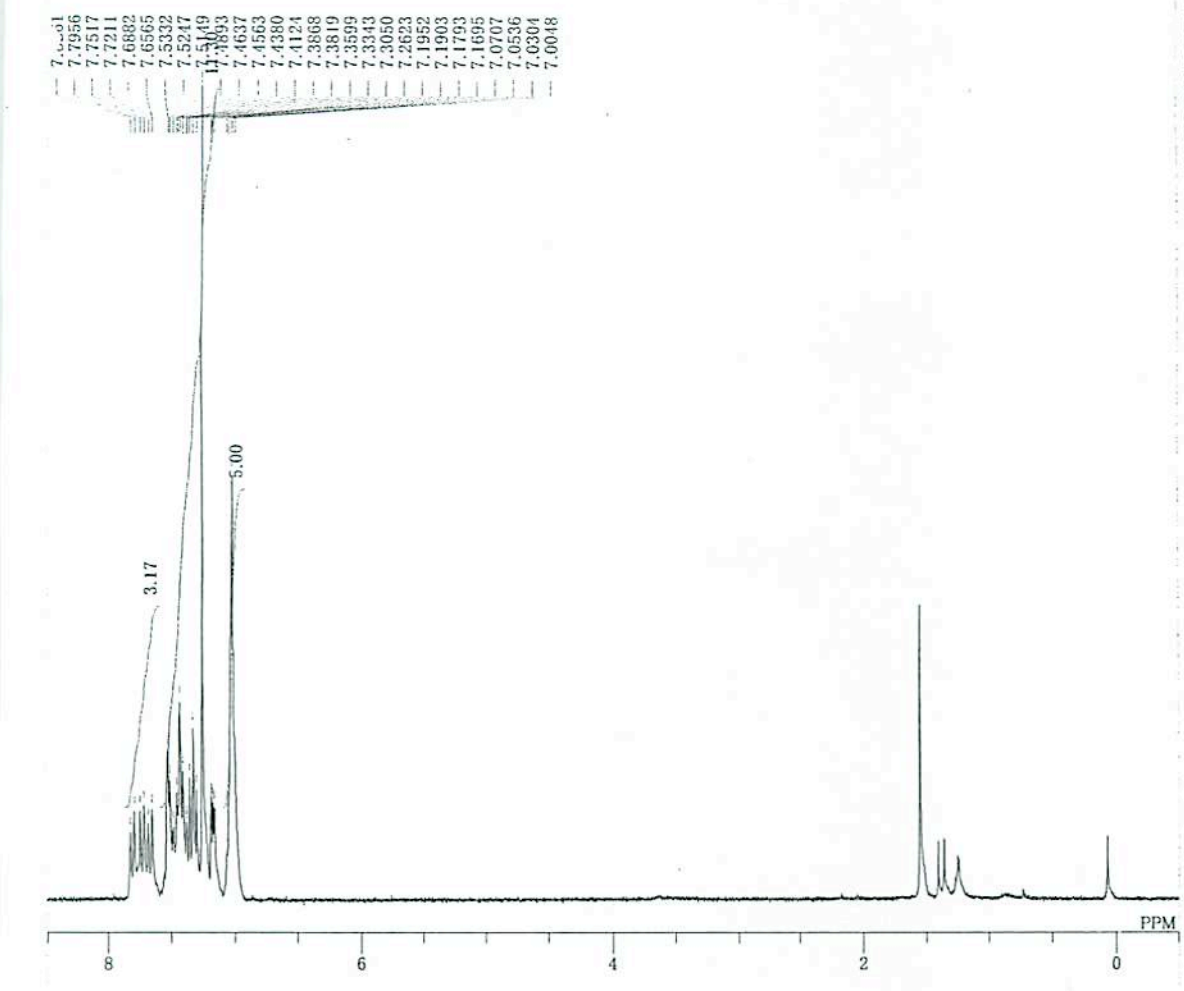
DFILE _DEFAULT.ALS
COMNT
DATIM
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 16384
FREQU 5401.76 Hz
SCANS 16
ACQTM 3.0331 sec
PD 3.9670 sec
PW1 5.40 usec
IRNUC 1H
CTEMP 18.1 c
SLVNT CDCL3
EXREF 7.25 ppm
BF 0.12 Hz
RGAIN 22



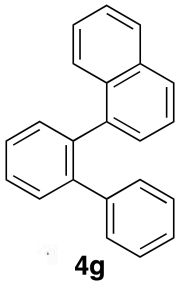
4f



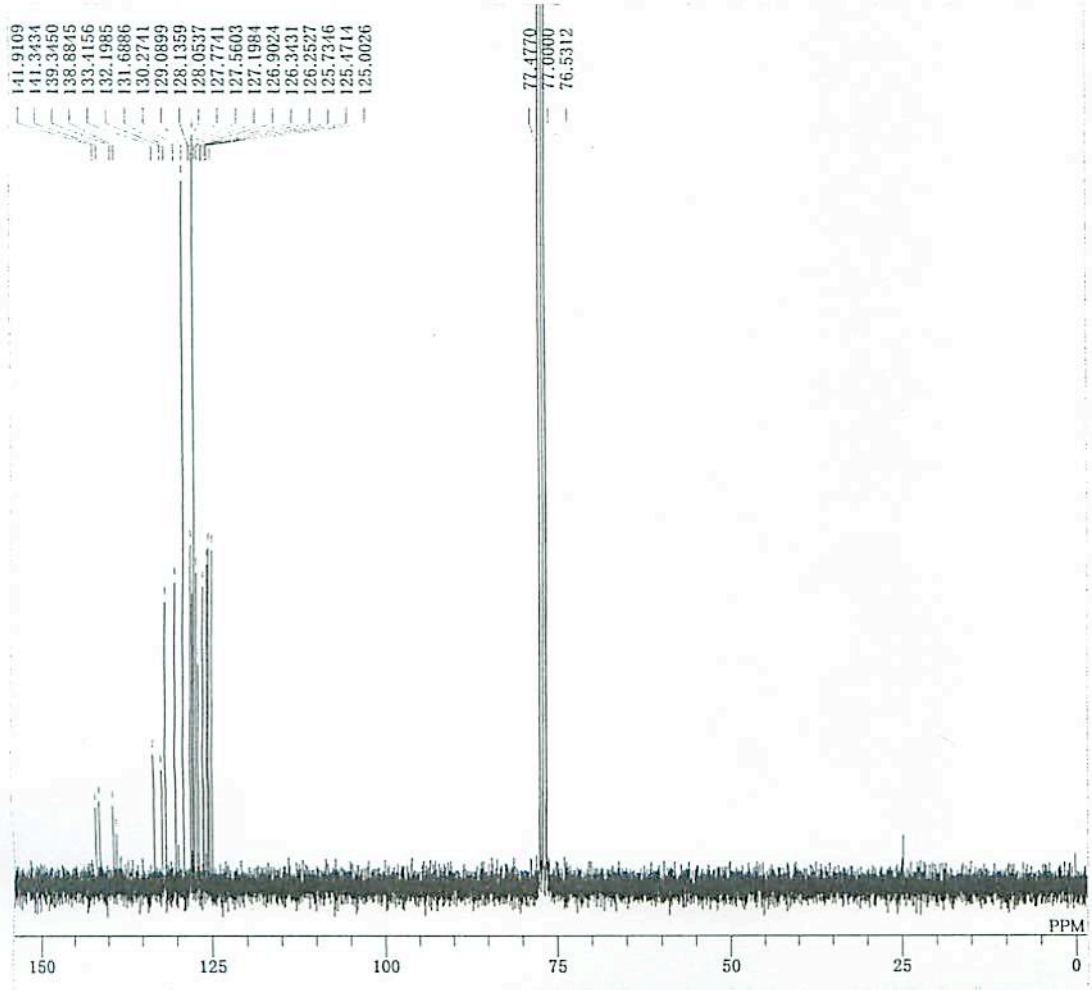
DFILE _DEFAULT.ALS
COMNT
DATIM
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.00 Hz
POINT 32768
FREQU 18306.64 Hz
SCANS 1047
ACQTM 1.7900 sec
PD 1.2100 sec
PW1 3.50 usec
IRNUC 1H
CTEMP 14.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 27



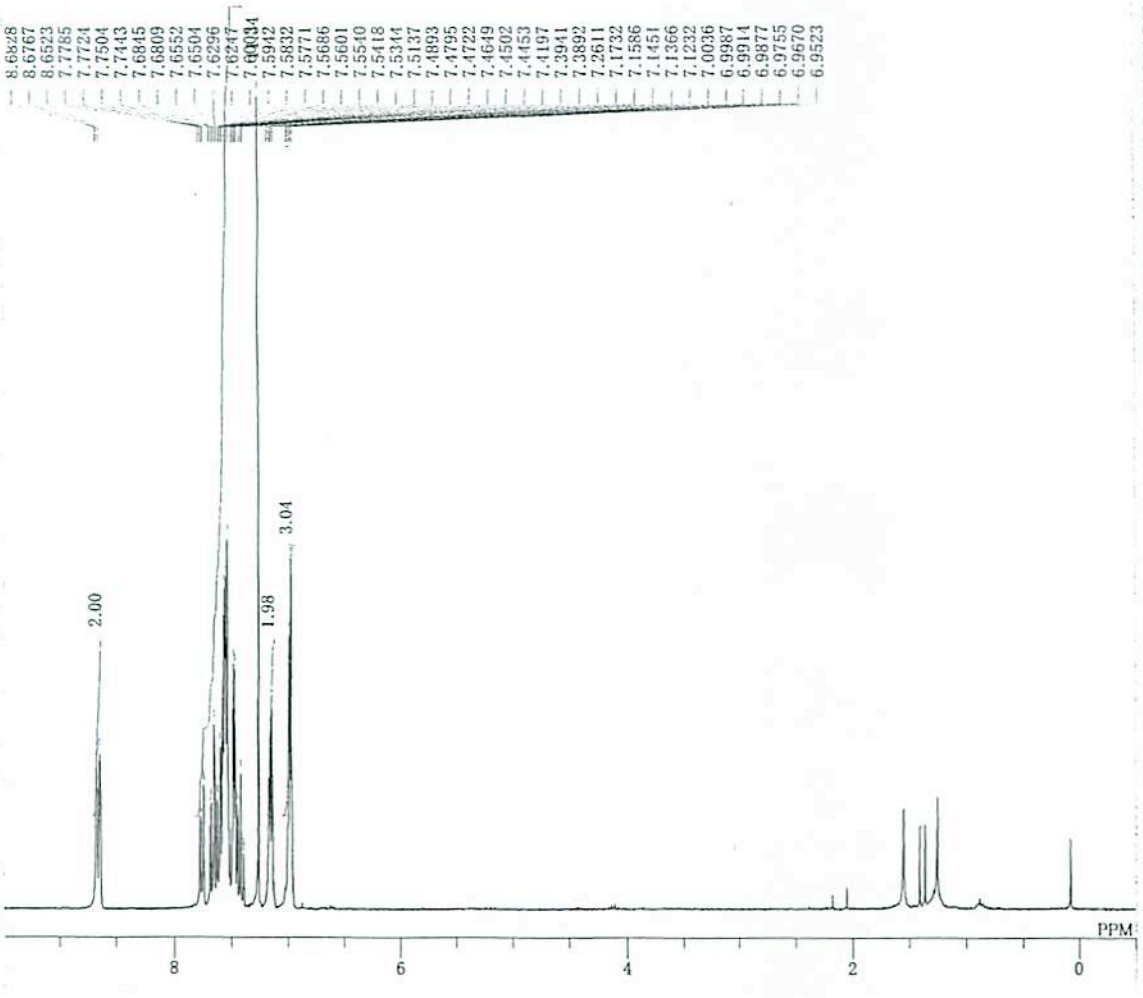
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	16
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	17.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	25



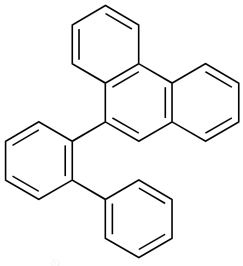
4g



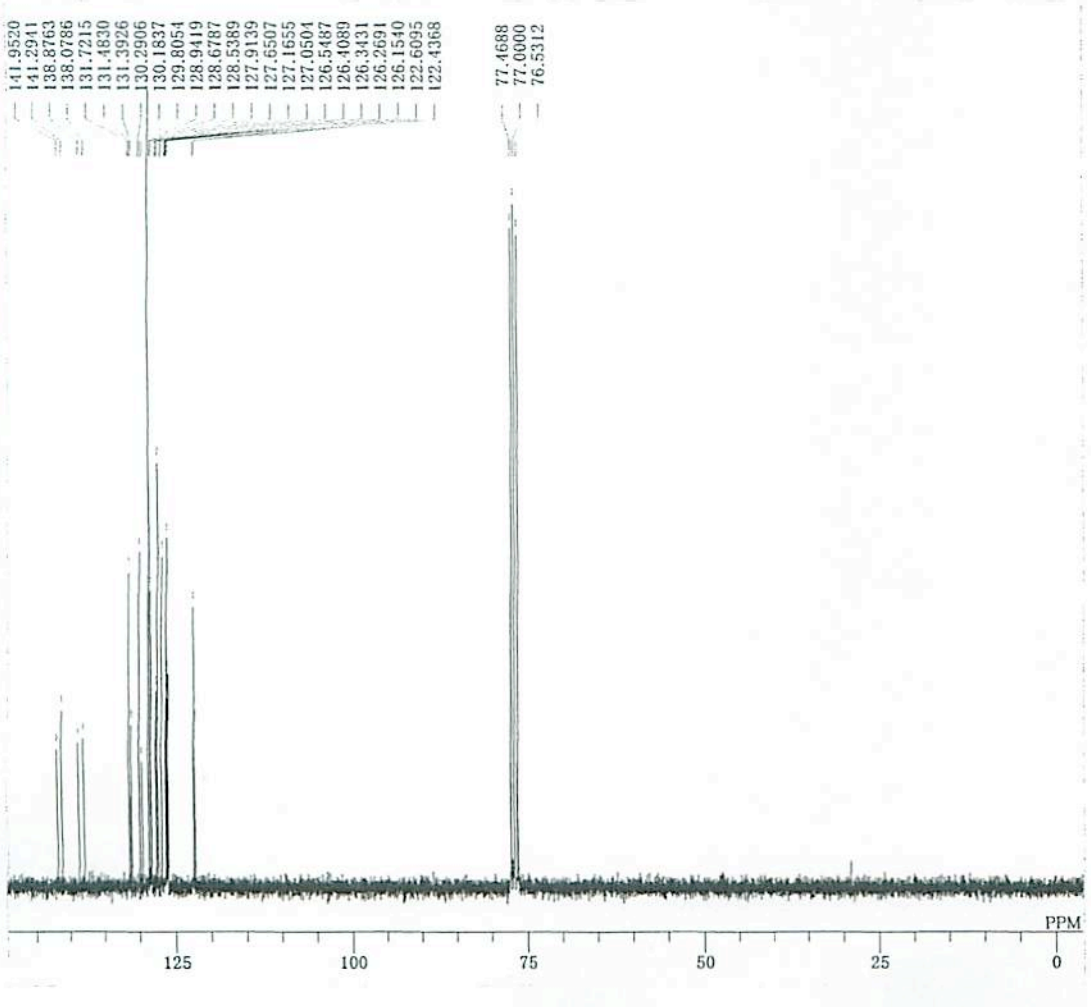
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	1475
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	26



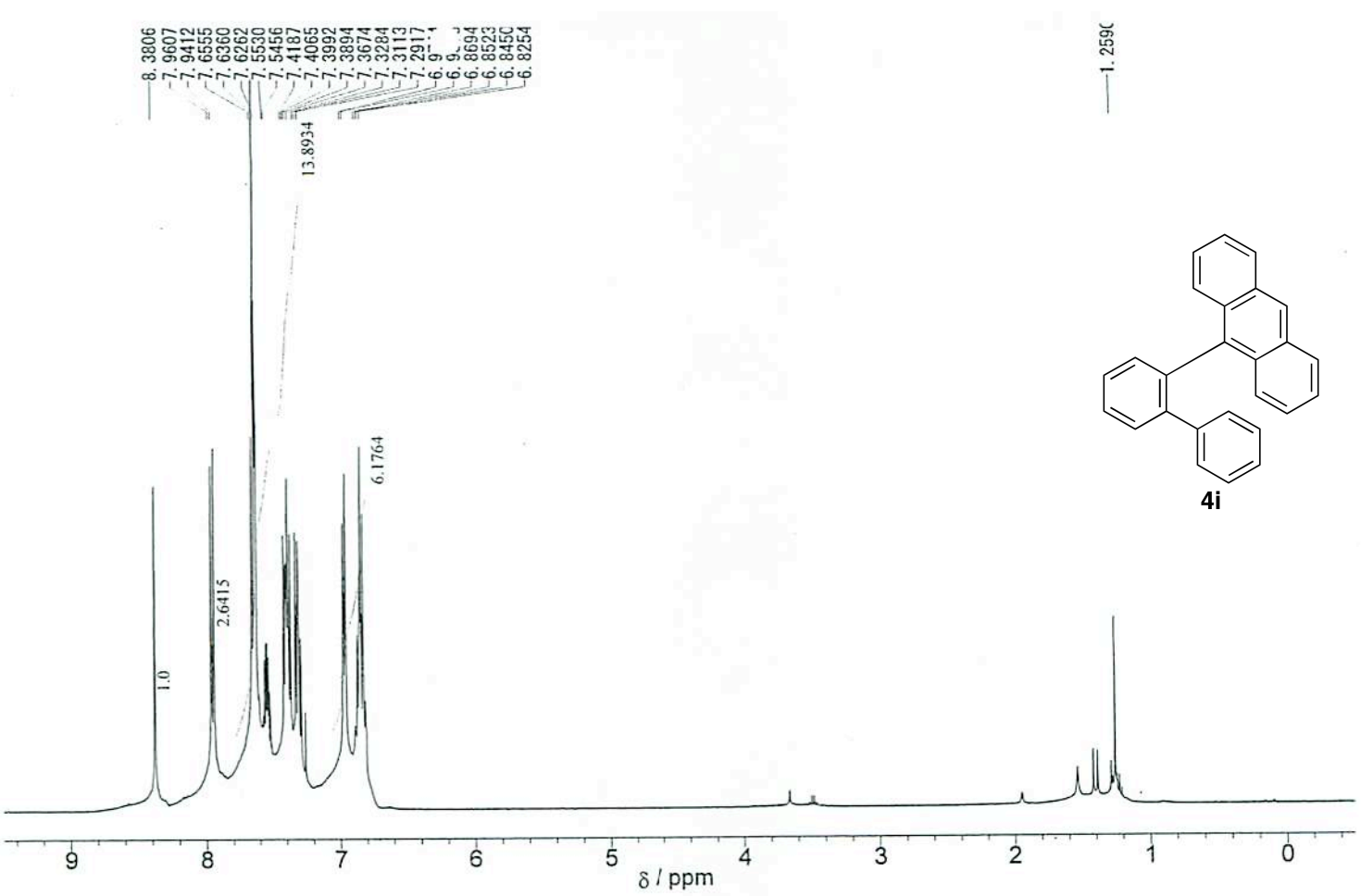
DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	16
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	17.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	22



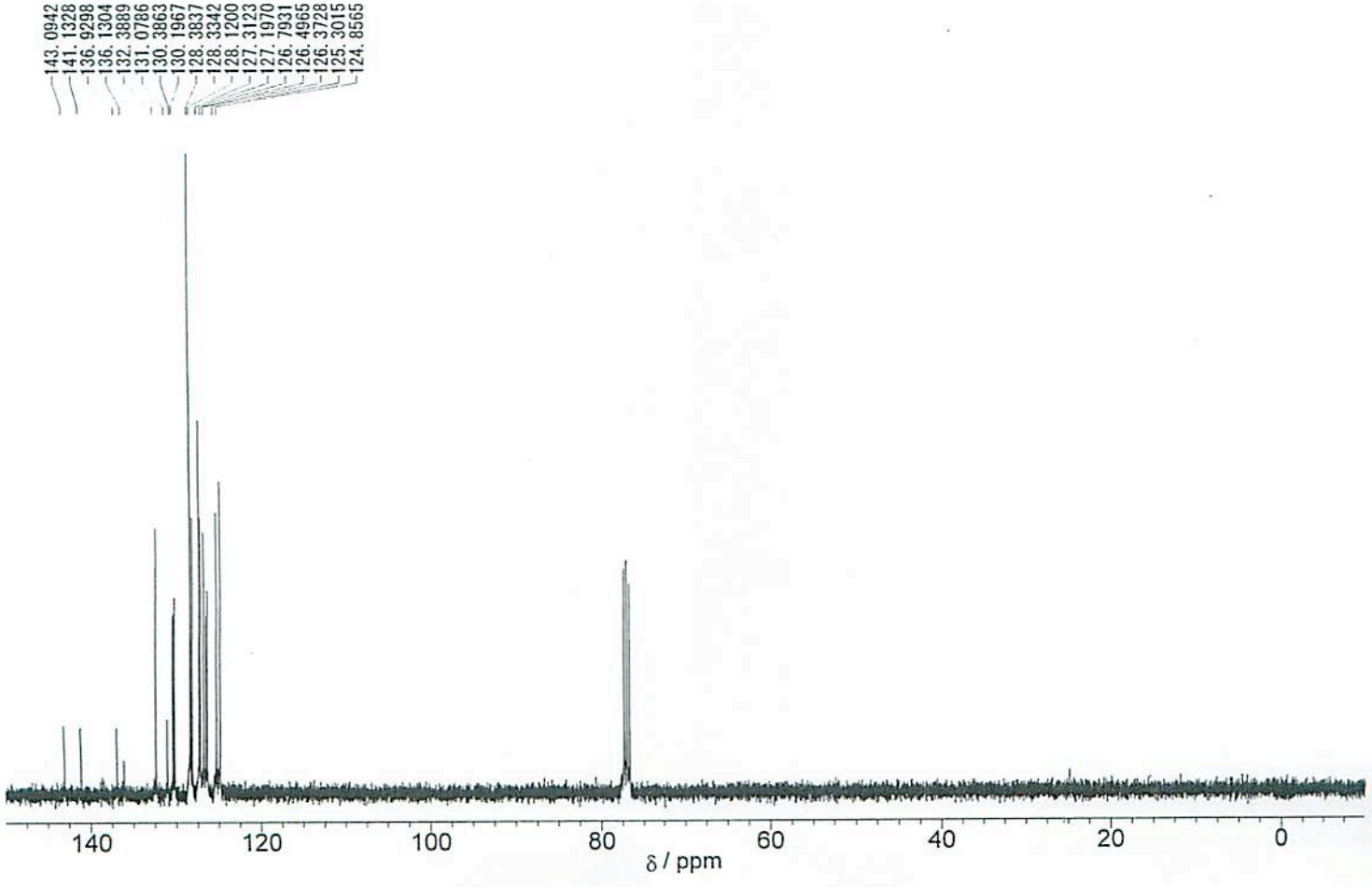
4h

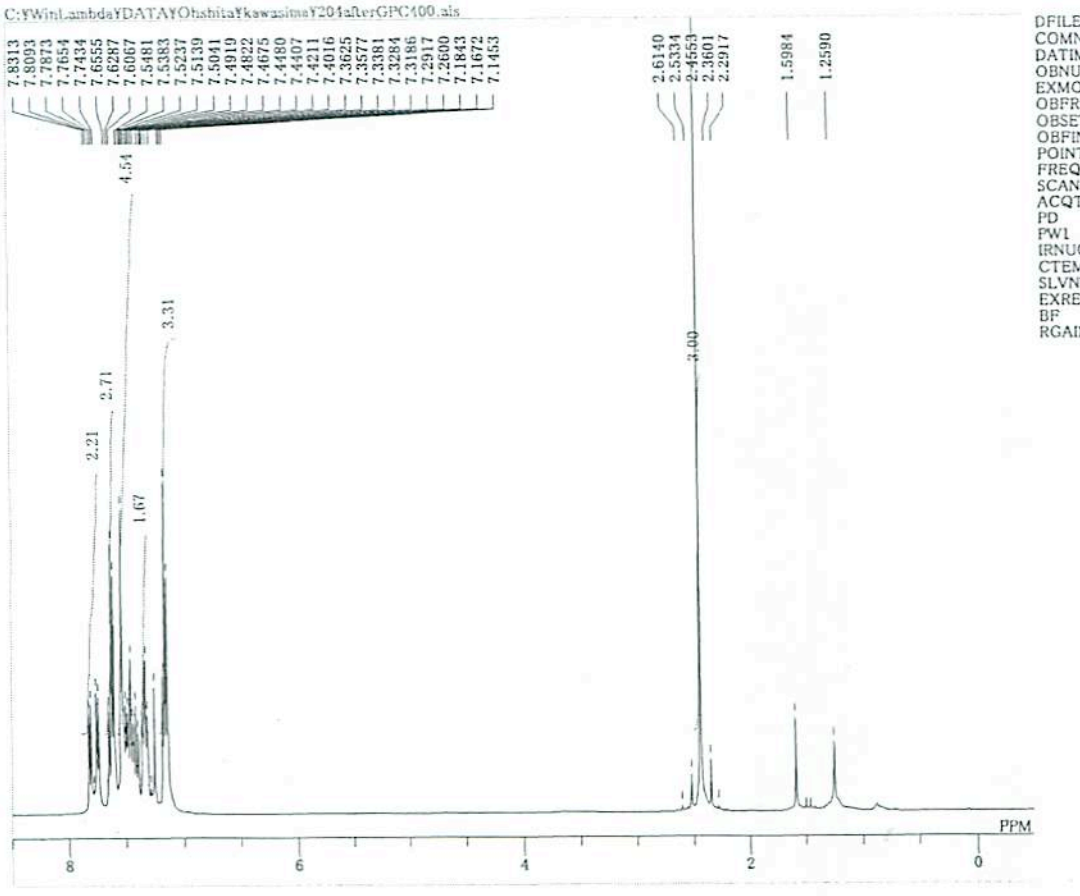


DFILE	_DEFAULT.ALS
COMNT	
DATIM	
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	1870
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	26

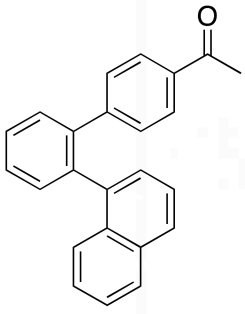

8.3806
7.9607
7.9412
7.6555
7.6360
7.6262
7.5530
7.5456
7.4187
7.4065
7.3992
7.3894
7.3674
7.3284
7.3113
7.2917
6.8694
6.8523
6.8450
6.8254
13.8934
6.1764
2.6415
1.0
δ / ppm

4i

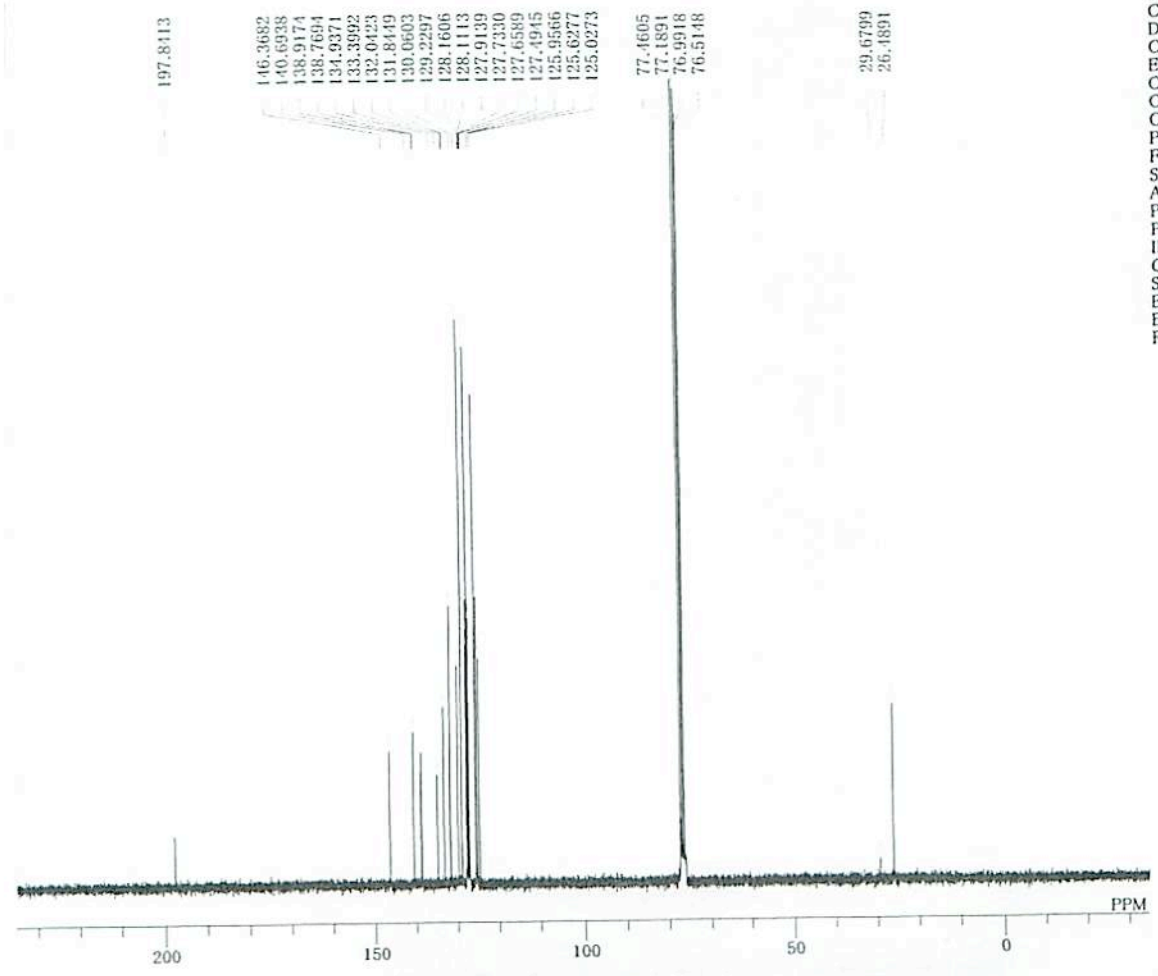

143.0942
141.1328
136.9298
136.1304
132.3889
131.0786
130.3863
130.1967
128.3837
128.3342
128.1200
127.3123
127.1970
126.7931
126.4965
126.3728
125.3015
124.8565
δ / ppm



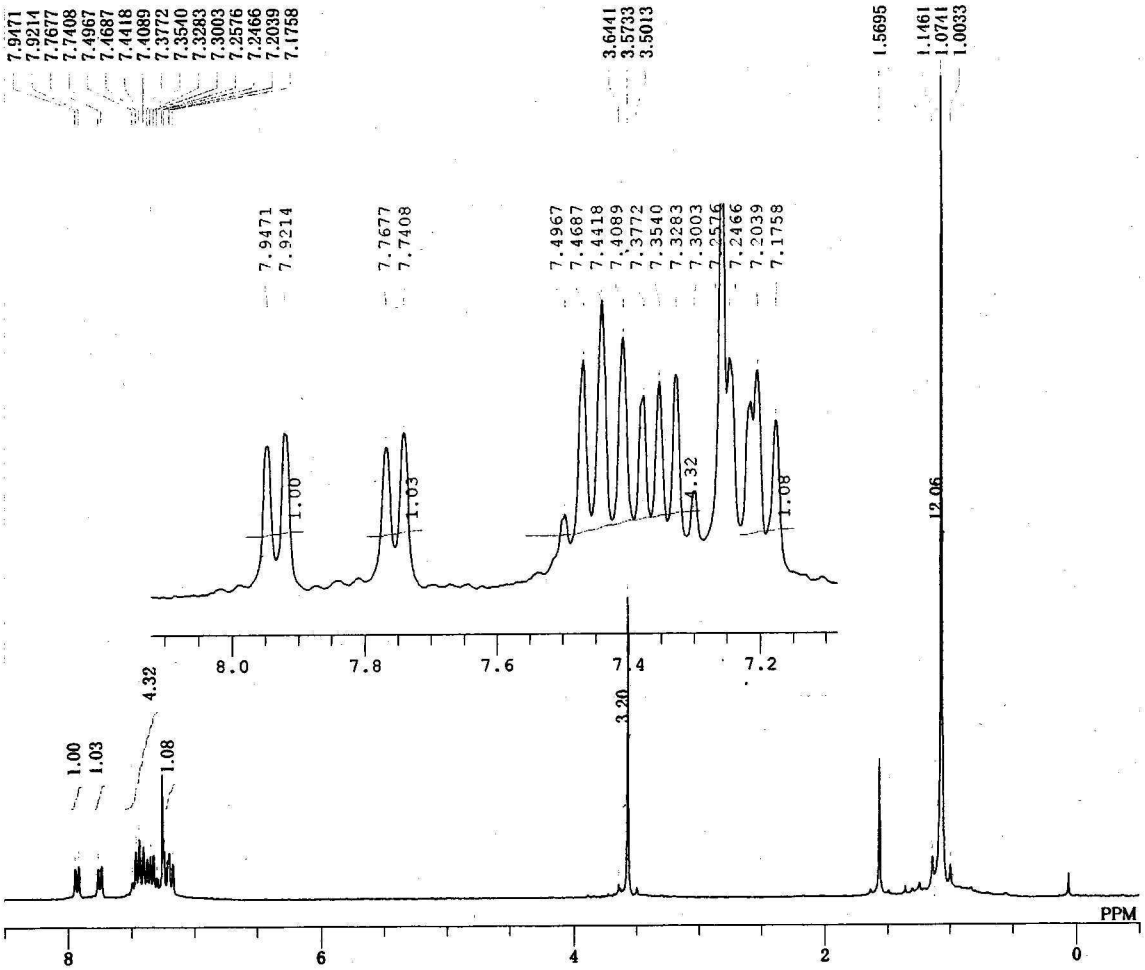
DFILE	204afterGPC400.als
COMNT	
DATIM	
OBNUC	1H
EXMOD	non
OBFRQ	395.75 MHz
OBSET	0.00 KHz
OBFIN	134498.00 Hz
POINT	8192
FREQU	7917.66 Hz
SCANS	8
ACQTM	1.0346 sec
PD	5.9650 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	18.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	1.62 Hz
RGAIN	16



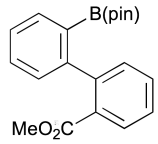
4j



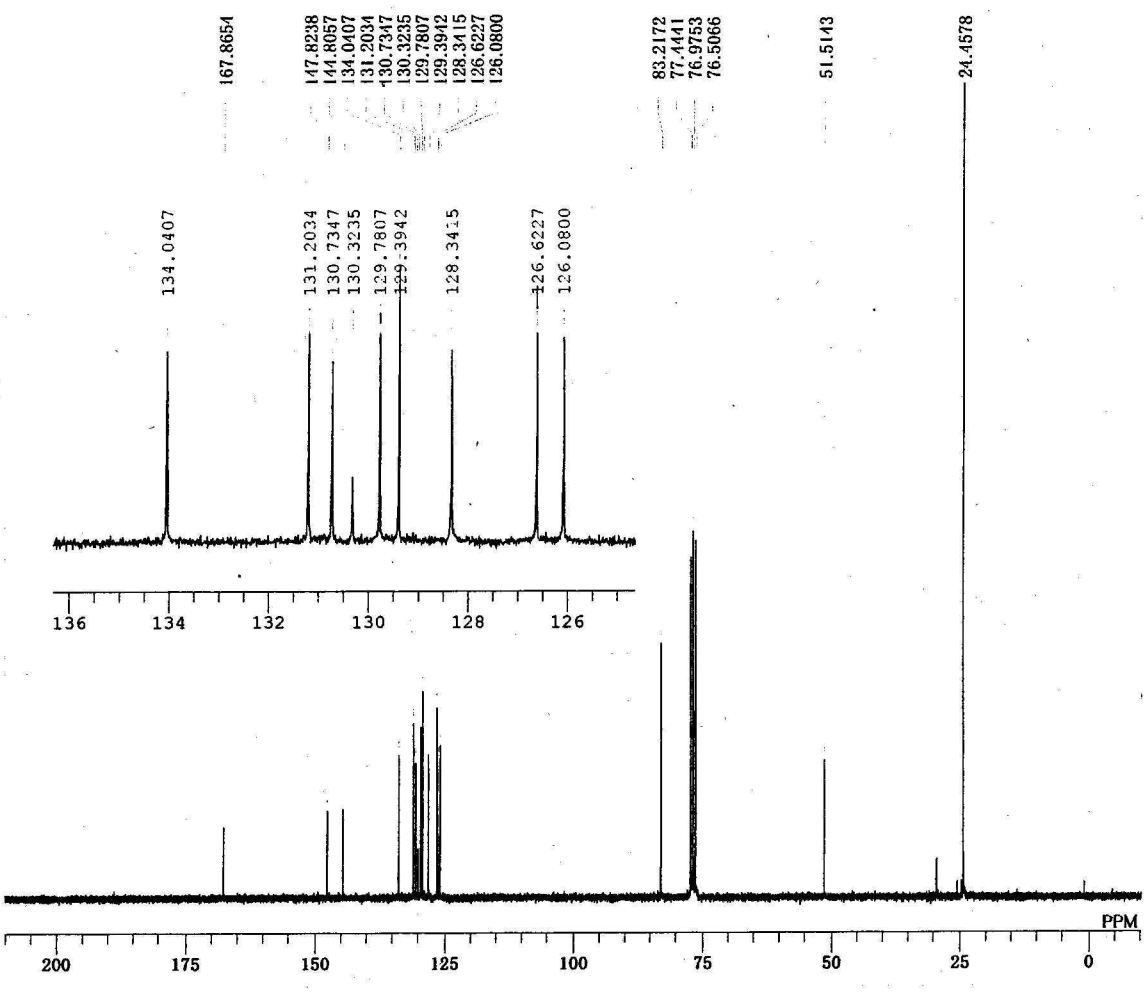
DFILE	_DEFAULTナフチル4アセトフ
COMNT	
DATIM	
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	11673
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	16.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	27



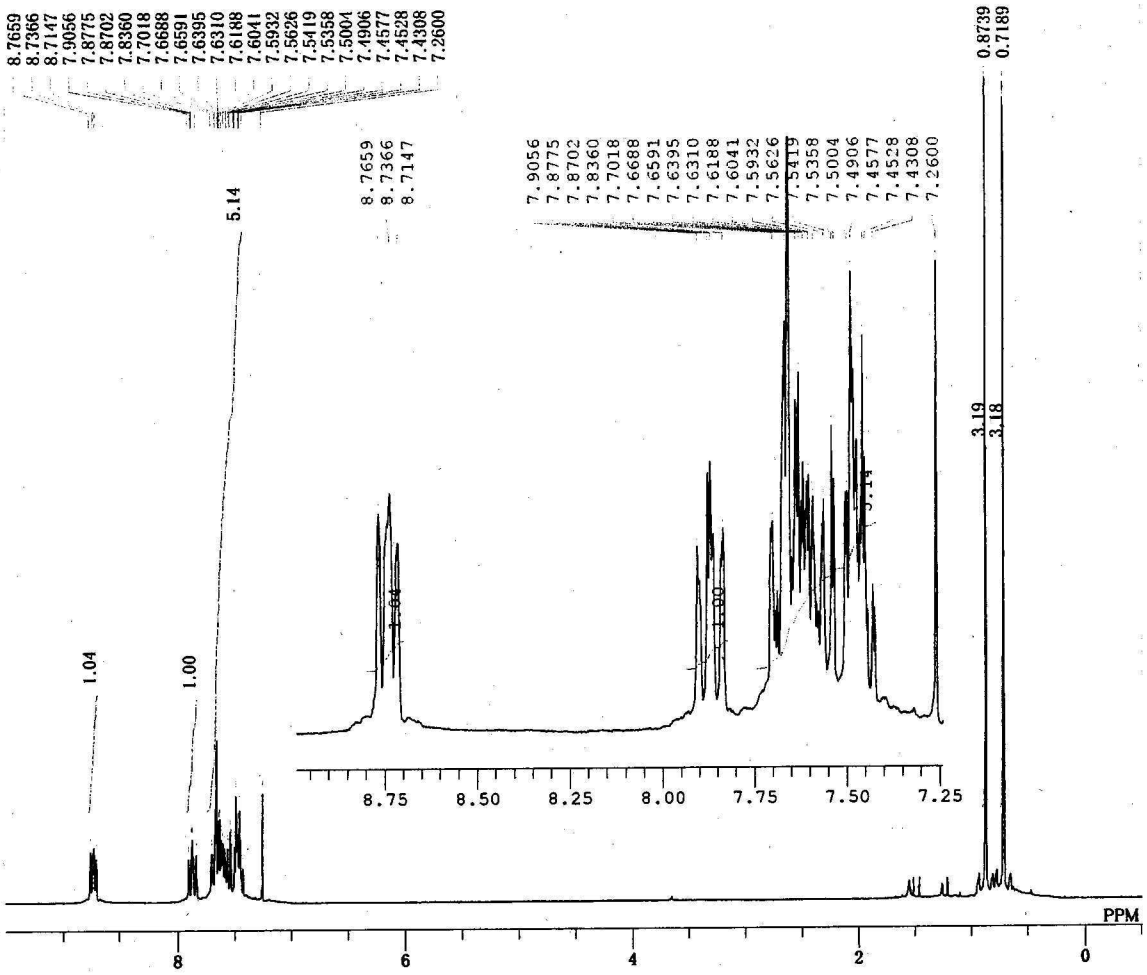
DFILE _DEFAULT.ALS
COMNT
DATIM Fri Nov 13 19:55:56 2009
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 16384
FREQU 5401.76 Hz
SCANS 8
ACQTM 3.0331 sec
PD 3.9670 sec
PW1 5.40 usec
IRNUC 1H
CTEMP 17.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 1.20 Hz
RGAIN 22



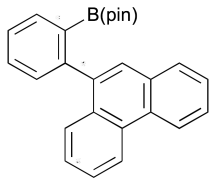
5a



DFILE 29913C.als
COMNT
DATIM Tue Nov 17 08:49:41 2009
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.00 Hz
POINT 32768
FREQU 18306.64 Hz
SCANS 10946
ACQTM 1.7900 sec
PD 1.2100 sec
PW1 3.50 usec
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28

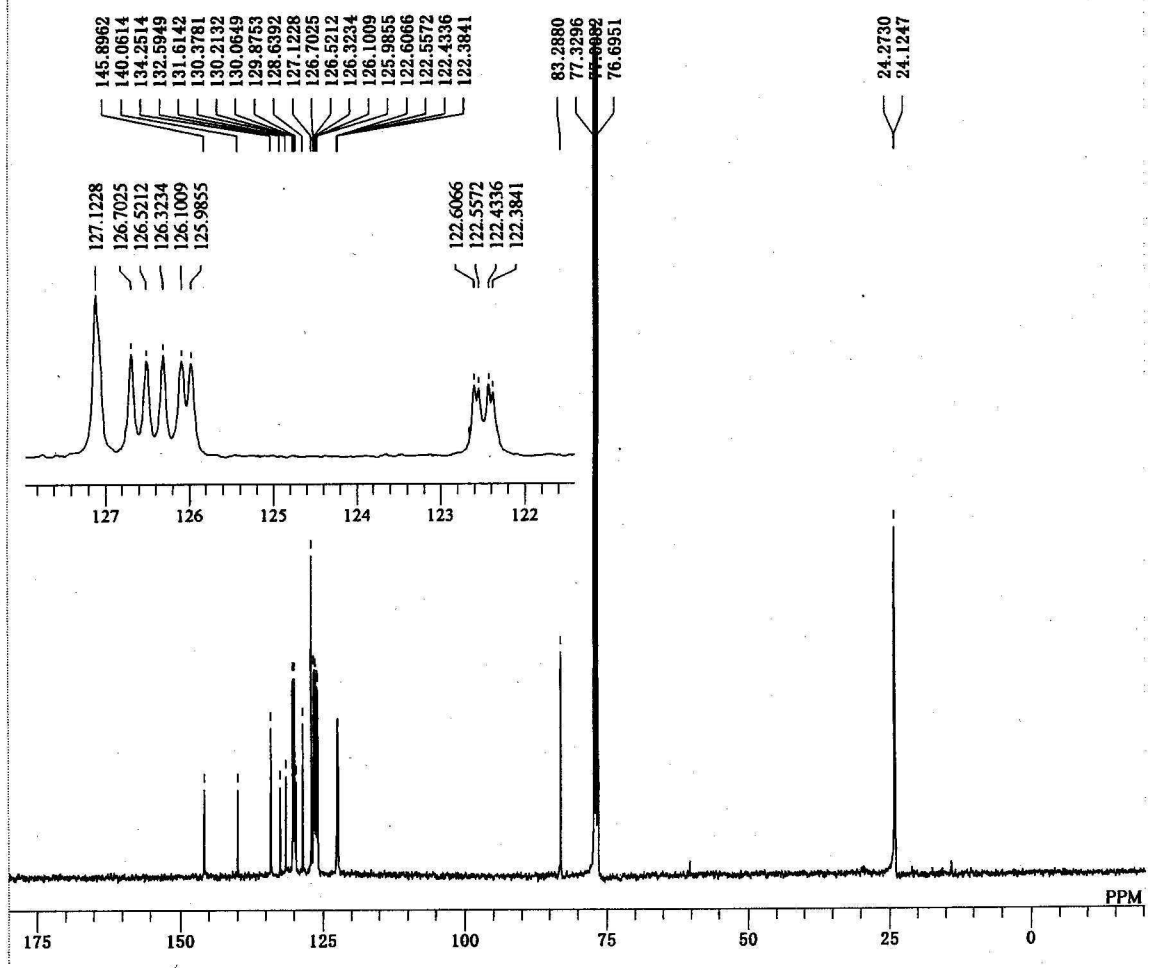


DFILE 292afterGPCピーク①.als
COMNT
DATIM Wed Nov 04 14:12:40 2009
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 16384
FREQU 5401.76 Hz
SCANS 8
ACQTM 3.0331 sec
PD 3.9670 sec
PW1 5.40 usec
IRNUC 1H
CTEMP 18.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 17

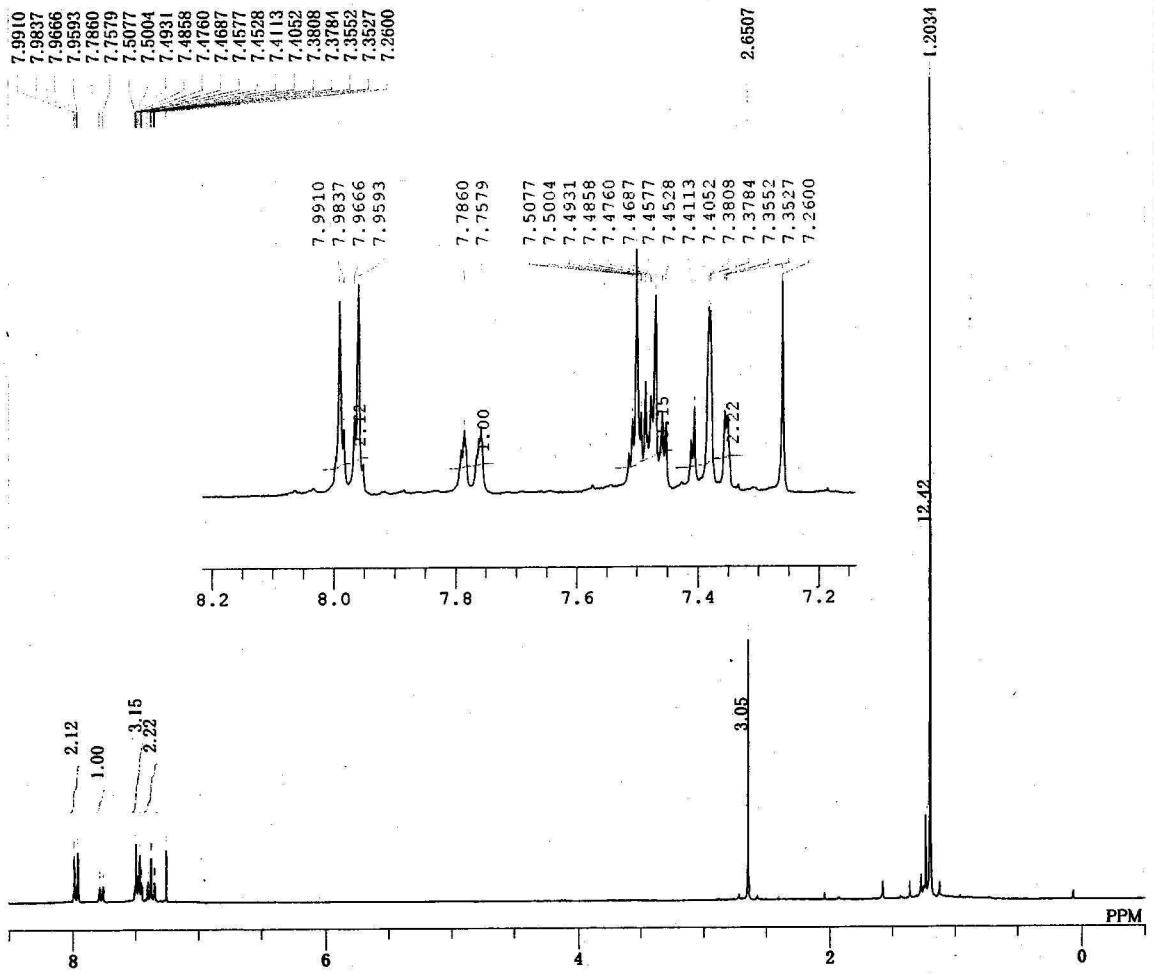


5b

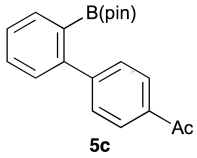
C:¥WinLambda¥DATA¥Ohshita¥kawasima¥292afterGPCピーク①13C.als



DFILE 292afterGPCピーク①13C.als
COMNT
DATIM Thu Nov 05 03:12:55 2009
OBNUC 13C
EXMOD bcm
OBFRQ 99.45 MHz
OBSET 0.00 KHz
OBFIN 104750.00 Hz
POINT 32768
FREQU 26881.72 Hz
SCANS 5888
ACQTM 1.2190 sec
PD 1.7810 sec
PW1 4.25 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 3.72 Hz
RGAIN 28

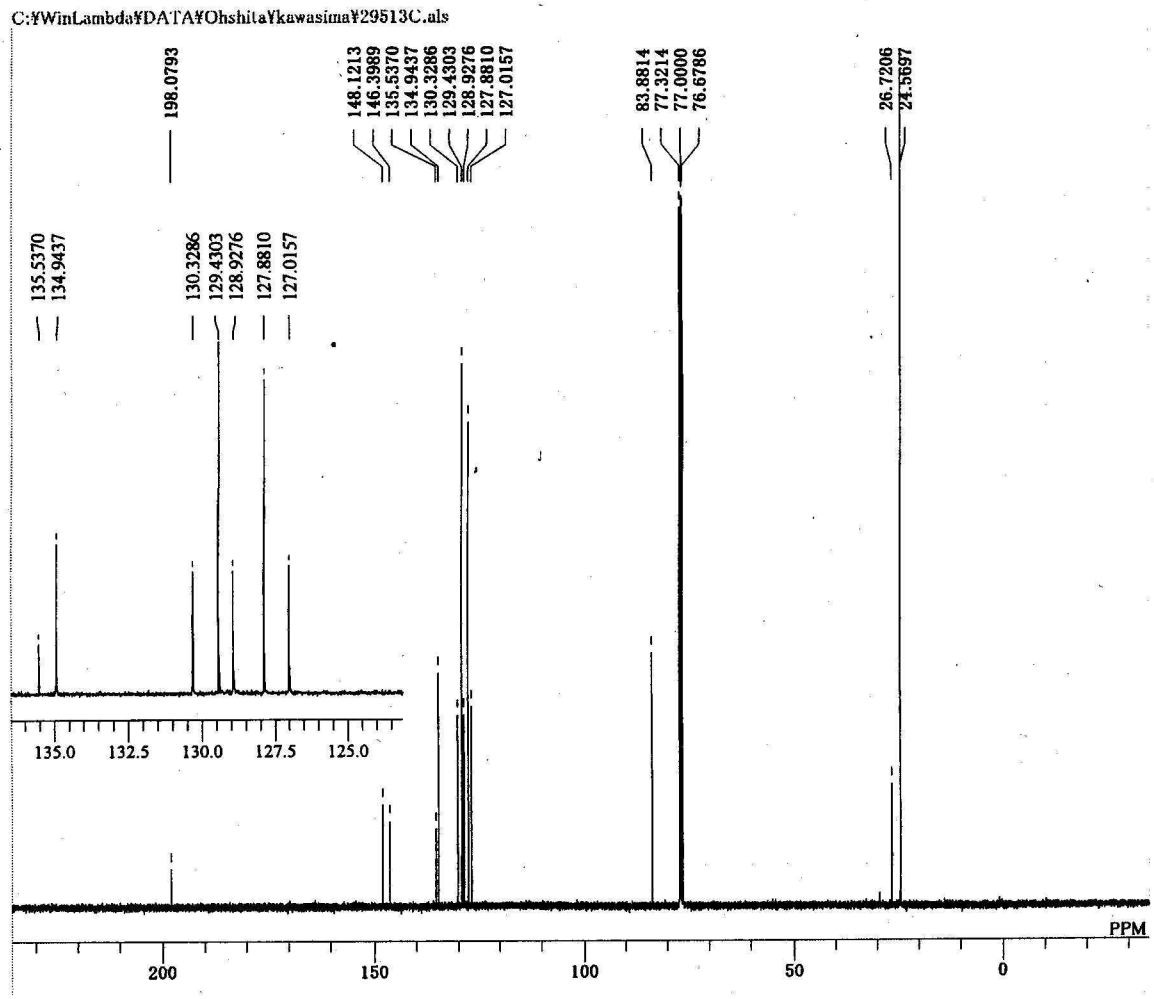


DFILE 295afterプレパラ.als
COMNT
DATIM Mon Nov 16 18:03:23 2009
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 16384
FREQU 5401.76 Hz
SCANS 8
ACQTM 3.0331 sec
PD 3.9670 sec
PW1 5.40 usec
IRNUC 1H
CTEMP 18.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 20



5c

C:¥WinLambda¥DATA¥Ohshita¥kawasima¥29513C.als



DFILE 29513C.als
COMNT
DATIM Tue Nov 17 08:53:17 2009
OBNUC 13C
EXMOD bcm
OBFRQ 99.45 MHz
OBSET 0.00 KHz
OBFIN 104750.00 Hz
POINT 32768
FREQU 26881.72 Hz
SCANS 12445
ACQTM 1.2190 sec
PD 1.7810 sec
PW1 4.25 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 27