Supporting Information

Development of an aquacatalytic system based on the formation of vesicles of an amphiphilic palladium NNC-pincer complex

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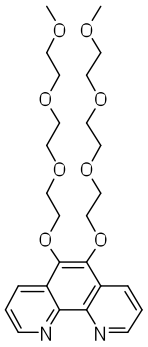
Table of Contents

General information

When manipulations were performed under a nitrogen atmosphere, nitrogen gas was dried by passage through P_2O_5. Commercially available chemicals (purchased from Sigma-Aldrich, TCI, Kanto chemical, Wako Pure Chemical Industries, Nacalai tesque, and Merck) are used without further purification unless otherwise noted. Silica gel was purchased from Kanto chemical (Silica gel 60N, spherical neutral, particle size 40-50μm) or Yamazen corporation (Hi-Flash™ Column Silica gel 40 mm 60 Å). Aluminium oxide was purchased from Merck (Aluminium oxide active basic, particle size 0.063-0.200 mm). TLC plates were purchased from Merck (TLC Silica gel 60 F_{254} and TLC Aluminium oxide 150 F_{254}). NMR spectra were recorded on a JEOL JNM A-500 spectrometer (500 MHz for ^{1}H, 125 MHz for ^{13}C) or a JEOL JNM ECS-400 spectrometer (396 MHz for ^{1}H, 100 MHz for ^{13}C). Chemical shifts are reported in δ (ppm) referenced to an internal tetramethylsilane standard for ^{1}H NMR. Chemical shifts of ^{13}C NMR are given related to $CDCl_3$ as an internal standard (δ 77.0). ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ at 25 °C. GC-MS analyses were measured with an Agilent 6890 GC/5973N MS Detector. ESI mass spectra (LRMS and HRMS) were recorded on a JEOL JMS-T100LC spectrometer. Elemental analyses were performed on a J-SCIENCE LAB MICRO CORDER JM10. Melting points were determined using a Yanaco micro melting point apparatus MP-J3 and were uncorrected. IR spectra were obtained using a JASCO FT/IR-460plus spectrometer in ATR mode. Dynamic light scatterings (DLS) were observed on an Otsuka electronics Co. DLS-6100P system using He-Ne 10 mW 632.8 nm laser. Transmission electron microscopy (TEM) images were obtained using a JEOL JEM-2100F operated at 200 kV. Atomic force microscopy (AFM) observations were performed using an Agilent Technologies Pico-Scan2500 in conventional tapping mode under air. Fluorescence microscopy images were obtained using a Keyence BZ-8000 with a x60 oil immersion objective lens. Confocal laser scanning microscopy (CLSM) images were obtained using a Nikon A1R with a x100 oil immersion objective lens. Millipore water was obtained from a Millipore Milli-Q Academic A10 purification unit. 1,10-Phenanthroline-5,6-diol (**3**),[1] *p*-bromo-[2-{2-(2-methoxyethoxy)ethoxy}ethoxy]benzene[2] and complex **14**[3] were prepared by literature methods. Theoretical calculations were carried out using the Gaussian 09 program[4] with RHF method. The geometry of palladium NNC pincer complexes **2a** and **2b** were optimized by using a STO-3G basis set.

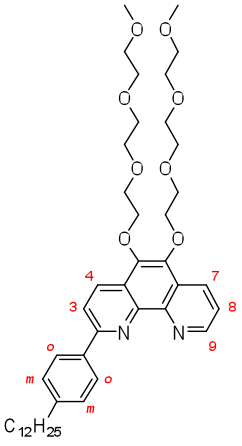
Synthesis of amphiphilic NNC pincer palladium complex 2a

5,6-Bis[2-[2-(2-methoxyethoxy)ethoxy]ethoxy]-1,10-phenanthroline (4).



Under a nitrogen atmosphere, to a mixture of 1,10-phenanthroline-5,6-diol (**3**) (1.72 g, 8.10 mmol) and sodium hydride (712.0 mg, 17.8 mmol, 60% oil) was added anhydrous DMF (70 mL) at 0 °C. After being stirred at 25 °C for 25 min, [2-[2-(2-methoxyethoxy)ethoxy]ethoxy] *p*-toluenesulfonate (5.7 g, 17.8 mmol) was slowly added at 0 °C. The reaction mixture was stirred at 80 °C for 24 h and quenched with water (100 mL). The resulting mixture was extracted with dichloromethane (20 mL, 3 times). The combined organic layer was washed with water (20 mL) and brine (20 mL), and dried over Na_2SO_4. After removal of the solvent, the resulting residue was chromatographed on aluminium oxide (eluent 1% MeOH/$CHCl_3$) to give **4** (2.3 g, 4.60 mmol, 55% yield) as brown oil. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 9.12 (dd, *J* = 1.8, 4.3 Hz, 2H, phen 2,9-H), 8.73 (dd, *J* = 1.8, 8.5 Hz, 2H, phen 4,7-H), 7.64 (dd, *J* = 4.3, 8.5 Hz, 2H, phen 3,8-H), 4.45–4.47 (m, 4H, -OC**H**$_2$CH$_2$(OCH$_2$CH$_2$)$_2$OCH$_3$), 3.84–3.86 (m, 4H, -C$_2$**H**$_4$O-), 3.64–3.72 (m, 12H, -C$_2$**H**$_4$O-), 3.54–3.55 (m, 4H, -C$_2$**H**$_4$O-), 3.38 (s, 6H, -OC**H**$_3$). ^{13}C-NMR (125 MHz, $CDCl_3$): δ 149.06, 144.08, 141.83, 130.68, 126.04, 122.73, 72.46, 71.73, 70.48, 70.41, 70.39, 58.80. IR (ATR): 2872, 1612, 1457, 1425, 1322, 1104, 1070, 1029, 810, 744 cm^{-1}. ESI-MS *m/z* 527 ([M+Na]$^+$), 505 ([M+1]$^+$). HR-ESI-MS calcd for $C_{26}H_{37}N_2O_8$ *m/z* 505.2550, found 505.2552.

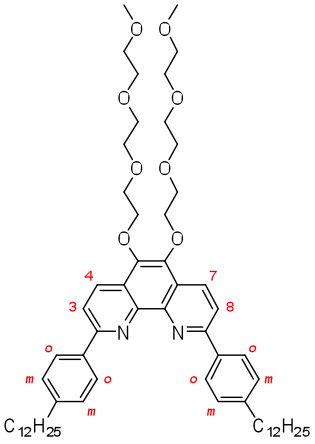
2-(4-Dodecylphenyl)-5,6-bis[2-[2-(2-methoxyethoxy)ethoxy]ethoxy]-1,10-phenanthroline (5).



Under a nitrogen atmosphere, 0.49 mL (1.1 mmol) of 2.3 M *n*-BuLi in hexane was slowly added to a degassed solution of 4-bromododecylbenzene (357.9 mg, 1.10 mmol) in anhydrous diethyl ether (8 mL) at -10 °C. After being stirred at -10 °C for 2 h, the solution was added dropwise to a degassed solution of **4** (504.6 mg, 1.00 mmol) in anhydrous toluene (20 mL). The reaction mixture was stirred at -10 °C for 30 min and quenched with water (1 mL). The resulting mixture was extracted with dichloromethane (20 mL, 3 times). The combined organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The resulting residue was dissolved in dichloromethane (30 mL). Activated MnO_2 (Merck, catalog No. 8.05958.0100, 1.5 g, 17.3 mmol) was added to the solution. After being stirred at 25 °C for 1 h, the reaction mixture was filtered through Celite and eluted with dichloromethane. The filtrate was concentrated under reduced pressure. The resulting residue was chromatographed on silica gel (eluent 0-3% MeOH/EtOAc) to give **5** (399.1 mg, 0.533 mmol, 55% yield) as light brown oil. ^{1}H-NMR (396 MHz, $CDCl_3$): δ 9.16 (dd, *J* = 1.4, 4.5 Hz, 1H, phen 9-H), 8.76 (d, *J* = 8.9 Hz, 2H, phen 4-H), 8.73 (dd, *J* = 1.4, 8.1 Hz, 1H, phen 7-H), 8.24 (d, *J* = 8.2 Hz, 2H, *o*-H), 8.10 (d, *J* = 8.9 Hz, 1H, phen 3-H), 7.63 (dd, *J* = 4.5, 8.1 Hz, 1H, phen 8-H), 7.34 (d, *J* = 8.2 Hz, 2H, *m*-H), 4.45–4.49 (m, 4H, -OC**H**$_2$CH$_2$(OCH$_2$CH$_2$)$_2$OCH$_3$), 3.85–3.88 (m, 4H, -C$_2$**H**$_4$O-), 3.65–3.71 (m, 12H, -C$_2$**H**$_4$O-), 3.54–3.56 (m, 4H, -C$_2$**H**$_4$O-), 3.38 (s, 3H, -OCH$_3$), 3.37 (s, 3H, -OCH$_3$), 2.69 (t, *J* = 7.7 Hz, 2H, -C**H**$_2$C$_{11}$H$_{23}$), 1.63–1.70 (m, 2H, -CH$_2$C**H**$_2$C$_{10}$H$_{21}$), 1.27–1.37 (m, 18 H, -CH$_2$CH$_2$(C**H**$_2$)$_9$CH$_3$), 0.88

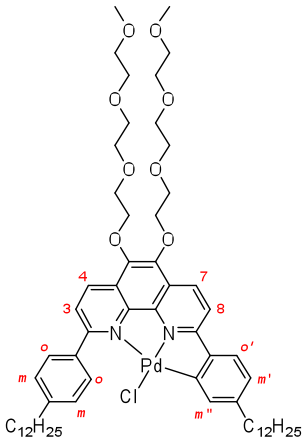
(t, J = 6.7 Hz, 3H, -(CH_2)$_{11}$C**H**$_3$). ^{13}C-NMR (96 MHz, $CDCl_3$): δ 156.75, 149.35, 144.45, 144.29, 144.18, 142.21, 141.59, 137.09, 131.64, 130.89, 128.87, 127.78, 126.55, 124.85, 122.72, 120.43, 71.97, 70.72, 70.65, 70.36, 59.06, 35.80, 31.93, 31.45, 29.69, 29.66, 29.64, 29.56, 29.37, 29.30, 22.71, 14.14. IR (ATR): 2923, 2853, 1614, 1453, 1323, 1108, 1071, 1030, 819, 762 cm^{-1}. ESI-MS m/z 771 ([M+Na]$^+$), 749 ([M+H]$^+$). Anal. Calcd for $C_{44}H_{64}N_2O_8$: C, 70.56; H, 8.61; N, 3.74; Found: C, 70.20; H, 8.60; N, 3.59.

2,9-Bis(4-dodecylphenyl)-5,6-bis[2-[2-(2-methoxyethoxy)ethoxy]ethoxy]-1,10-phenanthroline (6).



Under a nitrogen atmosphere, 64.2 μL (0.165 mmol) of 2.6 M n-BuLi in hexane was slowly added to a degassed solution of 4-bromododecylbenzene (53.7 mg, 0.165 mmol) in anhydrous diethyl ether (1.6 mL) at -10 °C. After being stirred at -10 °C for 1 h, the resulting solution was added dropwise to a degassed solution of **5** (112.3 mg, 0.150 mmol) in anhydrous toluene (5 mL). The reaction mixture was stirred at -10 °C for 1 h and quenched with water (1 mL). The resulting mixture was extracted with dichloromethane (5 mL, 3 times). The combined organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The resulting residue was dissolved in dichloromethane (15 mL). Activated MnO_2 (Merck, catalog No. 8.05958.0100, 1.5 g, 17.3 mmol) was added to the solution. After being stirred at 25 °C for 11 h, the reaction mixture was filtered through Celite and eluted with dichloromethane. The filtrate was concentrated under reduced pressure. The resulting residue was chromatographed on silica gel (eluent 5-10% MeOH/EtOAc) to give **6** (71.1 mg, 0.0716 mmol, 52% yield) as yellow oil. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.74 (d, J = 8.5 Hz, 2H, phen 4-H and 7-H), 8.37 (d, J = 8.5 Hz, 4H, o-H), 8.13 (d, J = 8.5 Hz, 2H, phen 3-H and 8-H), 7.39 (d, J = 8.5 Hz, 4H, m-H), 4.47–4.49 (m, 4H, -OC**H**$_2$CH$_2$(OCH$_2$CH$_2$)$_2$OCH$_3$), 3.87–3.86 (m, 4H, -C$_2$**H**$_4$O-), 3.73–3.66 (m, 12H, -C$_2$**H**$_4$O-), 3.57–3.55 (m, 4H, -C$_2$**H**$_4$O-), 3.37 (s, 6H, -OCH$_3$), 2.71 (t, J = 7.4 Hz, 4H, -C**H**$_2$C$_{11}$H$_{23}$), 1.72–1.67 (m, 4H, -CH$_2$C**H**$_2$C$_{10}$H$_{21}$), 1.40–1.27 (m, 36 H, -CH$_2$CH$_2$(C**H**$_2$)$_9$CH$_3$), 0.88 (t, J = 7.3 Hz, 6H, -(CH_2)$_{11}$C**H**$_3$). ^{13}C-NMR (125 MHz, $CDCl_3$): δ 155.88, 144.29, 144.21, 141.80, 136.97, 131.59, 128.85, 127.45, 125.06, 119.64, 72.61, 71.96, 70.72, 70.64, 70.63, 70.36, 70.13, 59.03, 35.82, 31.90, 31.81, 29.66, 29.62, 29.60, 29.54, 29.33, 29.31, 22.66, 14.09. IR (ATR): 2923, 2852, 1615, 1488, 1464, 1329, 1188, 1100, 1078, 1038, 830, 771 cm^{-1}. ESI-MS m/z 994 ([M+H]$^+$). Anal. Calcd for $C_{62}H_{92}N_2O_8 \cdot 0.5H_2O$: C, 74.29; H, 9.35; N, 2.79; Found: C, 74.46; H, 9.33; N, 2.87.

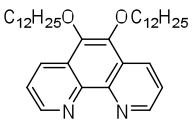
Chloro-[5-dodecyl-2-{9-(4-dodecylphenyl)-5,6-bis(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)-1,10-phenanthrolin-2-yl}phenyl]palladium (2a).



To a solution of **6** (49.7 mg, 0.050 mmol) in methanol (1.5 mL) was added $PdCl_2(MeCN)_2$ (13.0 mg, 0.0501 mmol), and the reaction mixture was stirred at 50 °C for 6 h. After removal of the solvent, the residue was chromatographed on aluminium oxide (eluent: 20% acetone/CH_2Cl_2) to give **2a** (44.9 mg, 0.0396 mmol, 79%) as yellow oil. ^{1}H-NMR (396 MHz, $CDCl_3$) δ 8.79 (d, *J* = 8.6 Hz, 1H, phen 4-H), 8.74 (d, *J* = 8.6 Hz, 1H, phen 7-H), 7.89 (d, *J* = 8.6 Hz, 1H, phen 8-H), 7.85 (d, *J* = 8.6 Hz, 1H, phen 3-H), 7.80 (d, *J* = 8.2 Hz, 2H, *o*-H), 7.75 (d, *J* = 1.6 Hz, 1H, *m*''-H), 7.38 (d, *J* = 8.0 Hz, 1H, *o*'-H), 7.37 (d, *J* = 8.2 Hz, 2H, *m*-H), 6.92 (dd, *J* = 1.6, 8.0 Hz, 1H, *m*'-H), 4.50–4.46 (m, 4H, -OC**H**$_2$CH$_2$(OC$_2$H$_4$)$_2$OCH$_3$), 3.85–3.82 (m, 4H, -(OC$_2$**H**$_4$)$_3$OCH$_3$), 3.72–3.64 (m, 12H, -(OC$_2$**H**$_4$)$_3$OCH$_3$), 3.58–3.53 (m, 4H, -(OC$_2$**H**$_4$)$_3$OCH$_3$), 3.38 (s, 3H, -OCH$_3$), 3.34 (s, 3H, -OCH$_3$), 2.72 (t, *J* = 7.9 Hz, 2H, -C**H**$_2$C$_{11}$H$_{23}$), 2.55 (t, *J* = 7.9 Hz, 2H, -C**H**$_2$C$_{11}$H$_{23}$), 1.70 (quint, *J* = 7.7 Hz, 2H, -CH$_2$C**H**$_2$C$_{10}$H$_{21}$), 1.63–1.58 (m, 2H, -CH$_2$C**H**$_2$C$_{10}$H$_{21}$), 1.41–1.25 (m, 36H, -CH$_2$CH$_2$(C**H**$_2$)$_9$CH$_3$), 0.90–0.86 (m, 6H, -(C*H*$_2$)$_{11}$C**H**$_3$). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.39, 161.71, 151.01, 146.36, 144.92, 144.71, 144.34, 142.76, 142.52, 142.34, 137.51, 135.47, 132.85, 132.66, 129.81, 128.29, 127.02, 126.07, 124.88, 124.29, 124.15, 118.15, 73.07, 71.96, 71.90, 70.69, 70.63, 70.59, 70.11, 59.06, 59.03, 36.63, 35.97, 31.93, 31.40, 29.71, 29.68, 29.65, 29.57, 29.49, 29.36, 22.70, 14.15. IR (ATR): 2922, 2852, 1615, 1581, 1448, 1335, 1103, 1083, 1051, 836, 816 cm^{-1}. ESI-MS *m*/*z* 1098 ([M-Cl]$^+$). Anal. Calcd for $C_{62}H_{91}ClN_2O_8Pd$: C, 65.65: H, 8.09; N, 2.47%. Found: C, 65.28: H, 8.03: N, 2.50%.

Synthesis of amphiphilic NNC pincer palladium complex 2b

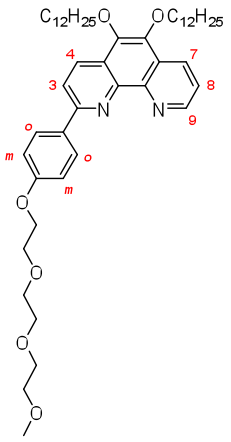
5,6-Bis(dodecyloxy)-1,10-phenanthroline (7).



Under a nitrogen atmosphere, to a mixture of 1,10-phenanthroline-5,6-diol (**3**) (1.73 g, 8.20 mmol) and sodium hydride (716.0 mg, 17.9 mmol, 60% oil) was added anhydrous DMF (70 mL) at 0 °C. After being stirred at 25 °C for 2.5 h, 1-bromododecane (4.46 g, 17.9 mmol) was slowly added. The reaction mixture was stirred at 80 °C for 15 h and quenched with water (100 mL). The resulting mixture was extracted with dichloromethane (20 mL, 3 times). The combined organic layer was washed with water (20 mL) and brine (20 mL), and dried over Na_2SO_4. After removal of the solvent, the resulting residue was chromatographed on aluminium oxide (eluent 0–5% MeOH/EtOAc) to give **7** (3.2 g, 5.83 mmol, 72% yield) as brown solids. Mp. 54-55 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 9.11 (dd, *J* = 1.8, 4.3 Hz, 2H, phen 2,9-H), 8.56 (dd, *J* = 1.8, 8.2 Hz, 2H, phen 4,7-H), 7.63 (dd, *J* = 4.3, 8.2 Hz, 2H, phen 3,8-H), 4.24 (t, *J* = 6.7 Hz, 4H, -OC**H**$_2$C$_{11}$H$_{23}$), 1.87–1.92 (m, 4H, -OCH$_2$C**H**$_2$(CH$_2$)$_9$CH$_3$), 1.52–1.61 (m, 4H, -OCH$_2$CH$_2$(C**H**$_2$)$_9$CH$_3$), 1.27–1.42 (m, 32H, -OCH$_2$CH$_2$(C**H**$_2$)$_9$CH$_3$), 0.88 (t, *J* = 7.0 Hz, 3H, -O(CH$_2$)$_{11}$C**H**$_3$). ^{13}C-NMR (125 MHz, $CDCl_3$): δ 149.06, 144.26, 142.20, 130.30, 126.30, 122.84, 73.92, 31.88, 30.35, 29.64, 29.61, 29.60, 29.59, 29.46, 29.32,

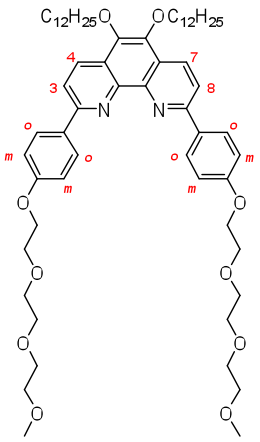
26.16, 22.65, 14.07. IR (ATR): 2915, 2849, 1613, 1463, 1427, 1398, 1322, 1110, 1075, 1065, 1025, 804, 740, 720 cm^{-1}. ESI-MS *m/z* 571 ($[M+Na]^+$), 549 ($[M+H]^+$). HR-ESI-MS calcd for $C_{36}H_{58}N_2O_2$ *m/z* 549.4420, found 549.44241.

5,6-Bis(dodecyloxy)-2-[4-[2-[2-(2-methoxyethoxy)ethoxy]ethoxy]phenyl]-1,10-phenanthroline (8).



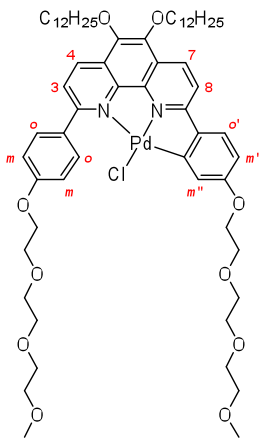
Under a nitrogen atmosphere, 1.16 mL (1.97 mmol) of 1.7 M *n*-BuLi in hexane was slowly added to a degassed solution of *p*-bromo-[2-{2-(2-methoxyethoxy)ethoxy}ethoxy]benzene (628.8 mg, 1.97 mmol) in anhydrous diethyl ether (10 mL) at -10 °C. After being stirred at 0 °C for 1 h, to the solution was added dropwise a degassed solution of **7** (900 mg, 1.64 mmol) in anhydrous toluene (10 mL). The reaction mixture was stirred at 0 °C for 30 min and quenched with water (5 mL). The resulting mixture was extracted with dichloromethane (20 mL, 3 times). The combined organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The resulting residue was dissolved in dichloromethane (40 mL) and activated MnO_2 (Merck, catalog No. 8.05958.0100, 2.0 g, 23.0 mmol) was added. After being stirred at 25 °C for 1 h, the reaction mixture was filtered through Celite and eluted with dichloromethane. The filtrate was concentrated under reduced pressure. The resulting residue was chromatographed on silica gel (eluent 70-100% EtOAc/hexane) to give **8** (745.3 mg, 0.920 mmol, 58% yield) as brown solids. Mp. 38-39 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 9.14 (dd, *J* = 1.6, 4.1 Hz, 1H, phen 9-H), 8.58 (d, *J* = 8.8 Hz, 1H, phen 4-H), 8.57 (dd, *J* = 1.6, 8.2 Hz, 1H, phen 7-H), 8.30 (d, *J* = 8.8 Hz, 2H, *o*-H), 8.05 (d, *J* = 8.8 Hz, 1H, phen 3-H), 7.62 (dd, *J* = 4.1, 8.2 Hz, 1H, phen 8-H), 7.07 (d, *J* = 8.8 Hz, 2H, *m*-H), 4.22–4.27 (m, 6H, -OC**H**$_2$C$_{11}$H$_{23}$, -OC$_2$H$_4$O-), 3.90–3.92 (m, 2H, -OC$_2$H$_4$O-), 3.77–3.78 (m, 2H, -OC$_2$H$_4$O-), 3.67–3.72 (m, 4H, -OC$_2$H$_4$O-), 3.56–3.58 (m, 2H, -OC$_2$H$_4$O-), 3.11 (s, 3H, -O(C$_2$H$_4$O)$_3$C**H**$_3$), 1.87–1.94 (m, 4H, -OCH$_2$C**H**$_2$(CH$_2$)$_9$CH$_3$), 1.53–1.60 (m, 4H, -OCH$_2$CH$_2$(C**H**$_2$)$_9$CH$_3$), 1.27–1.40 (m, 32H, -OCH$_2$CH$_2$(C**H**$_2$)$_9$CH$_3$), 0.88 (t, *J* = 7.0 Hz, 3H, -O(CH$_2$)$_{11}$C**H**$_3$). ^{13}C-NMR (125 MHz, $CDCl_3$): δ 159.87, 155.84, 148.93, 144.26, 143.98, 142.37, 141.59, 132.30, 131.03, 130.30, 129.02, 126.56, 124.59, 122.55, 119.78, 114.71, 73.85, 71.85, 70.79, 70.58, 70.49, 69.65, 67.40, 58.93, 31.83, 30.33, 29.60, 29.55, 29.42, 29.28, 26.13, 22.60, 14.03. IR (ATR): 2922, 2852, 1611, 1453, 1382, 1324, 1250, 1174, 1110, 1083, 1066, 1026, 830, 820, 764, 722 cm^{-1}. ESI-MS *m/z* 810 ($[M+Na]^+$). Anal. Calcd for $C_{49}H_{74}N_2O_6 \cdot H_2O$: C, 73.10; H, 9.51; N, 3.48; Found: C, 73.46; H, 9.53; N, 3.37.

5,6-Bis(dodecyloxy)-2,9-bis[4-[2-[2-(2-methoxyethoxy)ethoxy]ethoxy]phenyl]-1,10-phenanthroline (9).



Under a nitrogen atmosphere, 0.30 mL (0.56 mmol) of 1.9 M *n*-BuLi in hexane was slowly added to a degassed solution of *p*-bromo(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)benzene (178.7 mg, 0.560 mmol) in anhydrous THF (6 mL) at -78 °C. After being stirred at -78 °C for 1.5 h, to the solution was added dropwise a degassed solution of **8** (220.0 mg, 0.280 mmol) in anhydrous THF (1 mL). The reaction mixture was stirred at -78 °C for 1 h and quenched with water (1 mL). The resulting mixture was extracted with dichloromethane (10 mL, 3 times). The combined organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The resulting residue was dissolved in dichloromethane (20 mL) and activated MnO_2 (Merck, catalog No. 8.05958.0100, 1.0 g, 11.5 mmol) was added. After being stirred at 25 °C for 11 h, the reaction mixture was filtered through Celite and eluted with dichloromethane. The filtrate was concentrated under reduced pressure. The resulting residue was chromatographed on silica gel (eluent 5% acetone/CH_2Cl_2) to give **9** (78.8 mg, 0.0769 mmol, 27% yield) as yellow solids. Mp. 40-41 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.57 (d, J = 8.5 Hz, 2H, phen 4,7-H), 8.40 (d, J = 9.3 Hz, 4H, *o*-H), 8.07 (d, J = 8.5 Hz, 2H, phen 3,8-H), 7.12 (d, J = 9.3 Hz, 4H, *m*-H), 4.24–4.27 (m, 8H, -OC**H**$_2$C$_{11}$H$_{23}$, -OC$_2$H$_4$O-), 3.92–3.94 (m, 4H, -OC$_2$H$_4$O-), 3.78–3.80 (m, 4H, -OC$_2$H$_4$O-), 3.68–3.73 (m, 8H, -OC$_2$H$_4$O-), 3.57–3.58 (m, 4H, -OC$_2$H$_4$O-), 3.39 (s, 6H, -O(C$_2$H$_4$O)$_3$C**H**$_3$), 1.91 (t, J = 7.5 Hz, -OCH$_2$C**H**$_2$(CH$_2$)$_9$CH$_3$), 1.54–1.60 (m, 4H, -OCH$_2$CH$_2$(C**H**$_2$)$_9$CH$_3$), 1.27–1.44 (m, 32H, -OCH$_2$CH$_2$(C**H**$_2$)$_9$CH$_3$), 0.88 (t, J = 7.0 Hz, 6H, -O(CH$_2$)$_{11}$C**H**$_3$). ^{13}C-NMR (125 MHz, $CDCl_3$): δ 159.90, 155.13, 143.98, 141.90, 132.26, 131.09, 128.73, 124.84, 119.16, 114.77, 73.86, 71.87, 70.82, 70.61, 70.51, 69.69, 67.43, 58.97, 31.86, 30.37, 29.63, 29.60, 29.58, 29.46, 29.30, 26.17, 22.62, 14.06. IR (ATR): 2920, 2850, 1610, 1572, 1487, 1329, 1251, 1185, 1129, 1111, 1078, 950, 826, 771, 721 cm^{-1}. ESI-MS m/z 1048 ([M+Na]$^+$). Anal. Calcd for $C_{62}H_{92}N_2O_{10}\cdot H_2O$: C, 71.37; H, 9.08; N, 2.68; Found: C, 71.58; H, 9.08; N, 2.65.

Chloro-[2-[5,6-bis(dodecyloxy)-9-{4-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)phenyl}-1,10-phenanthrolin-2-yl]-5-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)phenyl]palladium (2b).



To a solution of **9** (20.5 mg, 0.0191 mmol) in methanol (1.0 mL) was added $PdCl_2(MeCN)_2$ (5.2 mg, 0.0200 mmol), and the reaction mixture was stirred at 50 °C for 6 h. After removal of the solvent, the residue was washed with hexane to afford **2b** (20.2 mg, 0.0173 mmol, 87%) as yellow solids. Mp. 70-71 °C. ^{1}H-NMR (396 MHz, $CDCl_3$) δ 8.55 (d, J = 8.6 Hz, 1H, phen 4-H), 8.48 (d, J = 8.6 Hz, 1H, phen 7-H), 7.83 (d, J = 8.6 Hz, 1H, phen 8-H), 7.81 (d, J = 8.5 Hz, 2H, *o*-H), 7.71 (d, J = 8.6 Hz, 1H, phen 3-H), 7.51 (d, J = 2.4 Hz, 1H, *m*''-H), 7.36 (d, J = 8.6 Hz, 1 H, *o*'-H), 7.08 (d, J = 8.5 Hz, 2H, *m*-H), 6.67 (dd, J = 2.4, 8.6 Hz, 1H, *m*'-H), 4.29–4.20 (m, 8H, -OC**H**$_2$C$_{11}$H$_{23}$, -OC$_2$H$_4$O-), 3.92–3.65 (m, 16H, -OC$_2$H$_4$O-), 3.60–3.55 (m, 4H, -OC$_2$H$_4$O-), 3.40 (s, 3H, -OCH$_3$), 3.38

(s, 3H, -OCH_3), 1.93–1.86 (m, 4H, -$OCH_2C\mathbf{H}_2C_{10}H_{21}$), 1.58–1.51 (m, 4H, -$O(CH_2)_2C\mathbf{H}_2C_9H_{19}$), 1.42–1.27 (m, 32H, -$O(CH_2)_3(C\mathbf{H}_2)_8CH_3$), 0.90–0.87 (m, 6H, -$OC_{11}H_{22}C\mathbf{H}_3$). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 161.88, 161.01, 160.26, 159.25, 153.27, 144.17, 142.65, 142.13, 139.82, 132.05, 131.83, 131.33, 130.88, 126.75 125.87, 125.27, 123.73, 121.34, 117.78, 114.38, 112.71, 71.89, 71.88, 70.81, 70.70, 70.61, 70.57, 70.52, 70.51, 69.63, 69.59, 67.40, 67.30, 59.02, 31,88, 30.28, 29.64, 29.62, 29.59, 29.43, 29.33, 26.11, 22.65, 14.10. IR (ATR): 2922, 2852, 1613, 1577, 1449, 1421, 1337, 1245, 1129, 1102, 1082, 1060, 1044, 950, 850, 830 cm^{-1}. ESI-MS *m/z* 1130 ([M-Cl]$^+$). Anal. Calcd for $C_{62}H_{91}ClN_2O_{10}Pd{\cdot}H_2O$: C, 62.88: H, 7.92; N, 2.37%. Found: C, 63.05: H, 7.75: N, 2.41%.

Preparation of vesicles $\mathbf{2a}_{vscl}$ and $\mathbf{2b}_{vscl}$

Experimental procedure for the preparation of vesicles $\mathbf{2a}_{vscl}$

A chloroform solution of **2a** (0.1 mL, 10 mg/mL) was charged in a 4 mL vial equipped with a screw cap. After evaporation of the chloroform, a thin film of **2a** was formed on the inner glass surface of the vial. Then, 1 mL of Millipore water was added to the vial followed by heating the resulting mixture at 60 °C for 4 h without stirring. After standing at 25 °C for overnight, the resulting mixture was sonicated for 10 min to generate a yellow suspension of vesicles $\mathbf{2a}_{vscl}$. The suspension was characterized by DLS and microscopic analyses (vide infra).

Experimental procedure for the preparation of vesicles $\mathbf{2b}_{vscl}$

A chloroform solution of **2b** (0.1 mL, 10 mg/mL) was charged in a 4 mL vial equipped with a screw cap. After evaporation of the chloroform, a thin film of **2b** was formed on the inner glass surface of the vial. Then, 1 mL of Millipore water was added to the vial followed by heating the resulting mixture at 80 °C for 12 h without stirring. After standing at 25 °C for overnight, the resulting mixture was sonicated for 10 min to generate a yellow suspension of vesicles $\mathbf{2b}_{vscl}$. The suspension was characterized by DLS and microscopic analyses (vide infra).

Dynamic light scattering (DLS) analysis

The suspension of vesicle $\mathbf{2a}_{vscl}$ or $\mathbf{2b}_{vscl}$ was placed in a glass tube. The DLS measurement was performed with 200 times repetition of signal scans. Particle sizes are estimated using the Marquardt method of data analysis.

Transmission electron microscopy (TEM) analysis

Samples for TEM analysis were prepared by the following method: suspensions of vesicle $\mathbf{2a}_{vscl}$ and $\mathbf{2b}_{vscl}$ (**2a** or **2b**/water = 1 mg/1 mL) were centrifuged (1000 rpm, 15 min) to give a precipitate and a supernatant. After removal of the supernatant by decantation, the resulting aqueous suspensions of the precipitate were diluted with 0.4 mL of water. The suspensions were dropped onto a copper grid covered with a carbon membrane and then air-dried. The obtained samples were measured with TEM.

Atomic Force Microscopy (AFM) analysis

Samples for AFM analysis were prepared by the following method: suspensions of vesicle **2a**$_{vscl}$ and **2b**$_{vscl}$ (**2a** or **2b**/water = 1 mg/1 mL) were centrifuged (1000 rpm, 15 min) to give a precipitate and a supernatant. After removal of the supernatant by decantation, the resulting aqueous suspensions of the precipitate were diluted with 0.4 mL of water. The suspensions were dropped onto a silicon wafer (Nilaco, P, Low, <0.02 Ωcm) and then air-dried. AFM analyses were performed under air in a conventional tapping mode.

Fluorescence microscopy and Confocal laser scanning microscopy (CLSM) analyses (diffusion of a hydrophobic compound to vesicles with fluorescein)

Samples for fluorescence microscopy and CLSM were prepared by the following method: to suspensions of **2a**$_{vscl}$ and **2b**$_{vscl}$ in water (**2a**/water = 2 mg/1 mL, **2b**/water = 0.5 mg/1 mL) were added 1 μL of an ethanol solution of fluorescein (1.5 mM) to give stained **2a**$_{vscl}$/fluorescein and **2b**$_{vscl}$/fluorescein, respectively. The stained **2a**$_{vscl}$ and **2b**$_{vscl}$ were cast (1 drop) onto microscope slides, and then covered with cover glasses. The edges of the cover glasses were sealed with glue to prevent drying. The resulting slides were subjected to fluorescence microscopy and CLSM.

Fluorescence microscopy and Confocal laser scanning microscopy (CLSM) analyses (diffusion of a hydrophobic compound to vesicles with Nile Red)

To the suspensions of **2a**$_{vscl}$/fluorescein and **2b**$_{vscl}$/fluorescein was added 3μL of a $CHCl_3$ solution of Nile Red (4.5 mM). The suspension was centrifuged at 2500 rpm for 10 min to give a supernatant and a precipitate. The precipitate was removed by decantation. The precipitate was washed via centrifugation-decantation with Millipore water (**2a**$_{vscl}$: 0.3 mL x 3 times, **2b**$_{vscl}$: 0.6 mL x 3 times) to give **2a**$_{vscl}$/Nile Red and **2b**$_{vscl}$/Nile Red, respectively. The stained **2a**$_{vscl}$ and **2b**$_{vscl}$ were cast (1 drop) onto microscope slides, and then covered with cover glasses. The edges of the cover glasses were sealed with glue to prevent drying. The resulting slides were subjected to fluorescence microscopy.

Additional TEM images

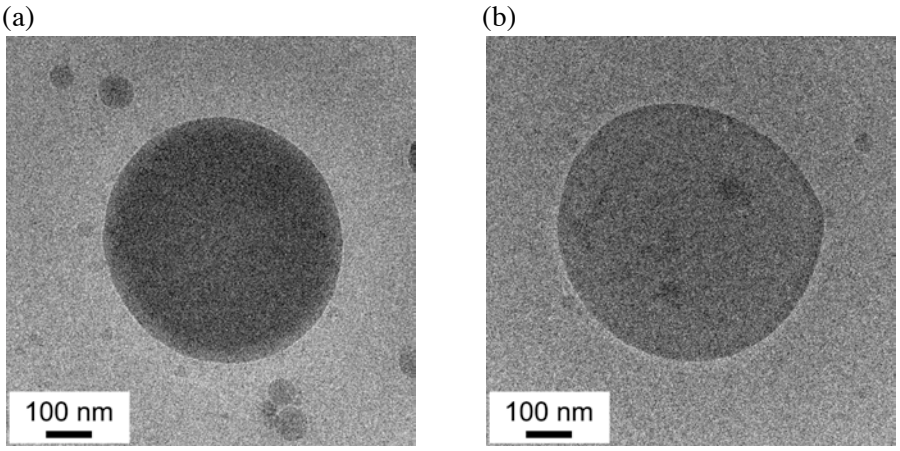


Figure S1. TEM images of vesicles **2a**$_{vscl}$ (a) after heating at 80 °C for 1 h in water, (b) after heating at 100 °C for 1h in water.

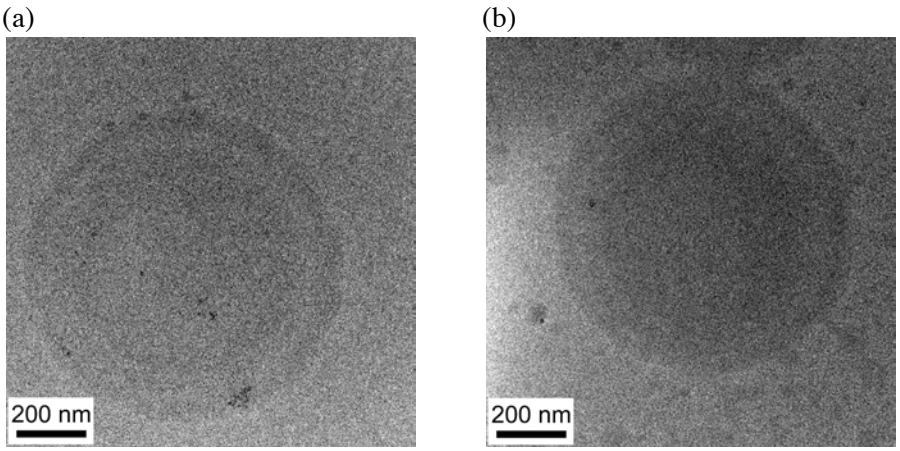


Figure S2. TEM images of vesicles **2b**$_{vscl}$ (a) after heating at 80 °C for 1 h, in water (b) after heating at 100 °C for 1h in water.

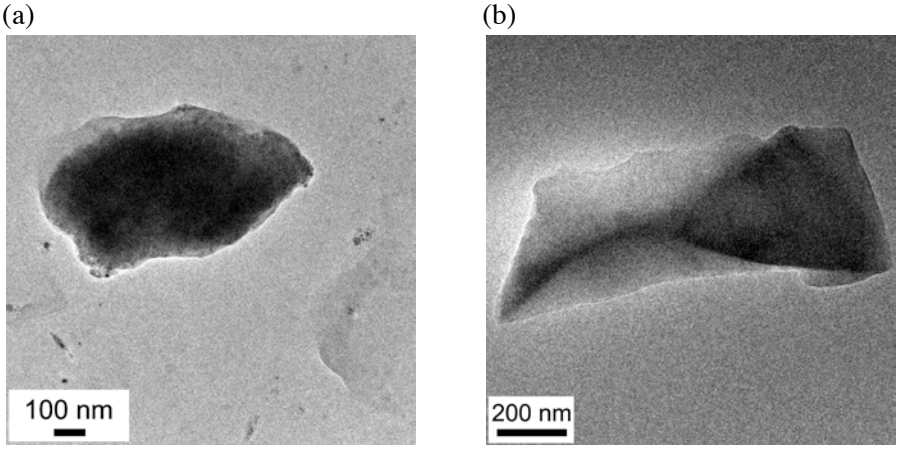


Figure S3. TEM images of amorphous complexes **2a**$_{amps}$ (a) and **2b**$_{amps}$ (b).

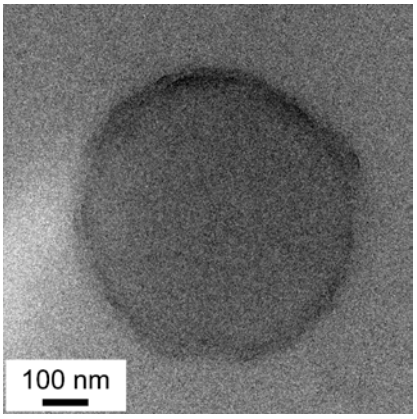


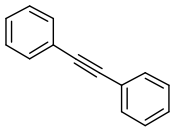
Figure S4. TEM images of vesicles **2a**$_{vscl}$ after the reaction of iodobenzene (**10a**) with ethynylbenzene (**11a**) in water at 40 °C for 1 h.

Typical procedure and characterization of the products for the arylation of terminal alkynes

Typical procedure for the arylation of terminal alkynes using 2a$_{vscl}$

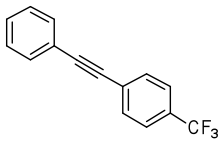
To a vial equipped with a screw cap, 1 mL aqueous suspension of **2a**$_{vscl}$ (1.0 mg, 8.5 x 10^{-4} mmol), triethylamine (10.3 mg, 0.10 mmol), ethynylbenzene (**11a**) (7.0 mg, 0.068 mmol), and iodobenzene (**10a**) (6.9 mg, 0.034 mmol) were added. The reaction mixture was agitated with shaking at 40 °C for 1 h and allowed to cool to 25 °C. The reaction mixture was extracted with *tert*-butyl-methyl ether (1.0 mL, 5 times). The combined organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The resulting residue was chromatographed on silica gel (eluent: hexane) to give 1,1'-ethyne-1,2-diyldibenzene **12a** (11.5 mg, 0.063 mmol, 92%) as white solids.

1,1'-ethyne-1,2-diyldibenzene (12a)[5] [CAS: 64666-02-0]



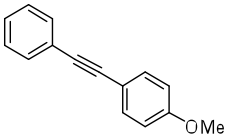
^{1}H NMR (396 MHz, $CDCl_3$) δ 7.55–7.52 (m, 4H, ArH), 7.37–7.33 (m, 6H, ArH). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 131.57, 128.31, 128.23, 123.22, 89.33. EI-MS *m/z* 178 (M^+).

1-Phenyl-2-(*p*-trifluoromethylphenyl)acetylene (12b)[5] [CAS: 370-99-0]



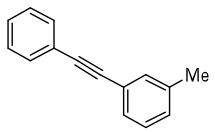
^{1}H NMR (396 MHz, $CDCl_3$) δ 7.65–7.54 (m, 6H, ArH), 7.38–7.37 (m, 3H, ArH).
^{13}C-NMR (100 MHz, $CDCl_3$) δ 131.76, 131.70, 129.84 (q, *J* = 32.7 Hz), 128.79, 128.42, 127.06, 125.23 (q, *J* = 3.5 Hz), 123.92 (q, *J* = 272.0 Hz), 122.51, 91.72, 87.94. EI-MS *m/z* 246 (M^+).

1-(*p*-Methoxyphenyl)-2-phenylacetylene (12c)[5] [CAS: 7380-78-1]



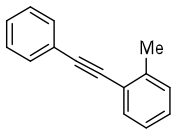
^{1}H NMR (396 MHz, $CDCl_3$) δ 7.52–7.46 (m, 4H, ArH), 7.36–7.30 (m, 3H, ArH), 6.88 (d, *J* = 8.7 Hz, 2H, ArH), 3.82 (s, 3H, -OCH_3). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 159.56, 133.02, 131.42, 128.28, 127.91, 123.53, 115.31, 113.95, 89.33, 88.03, 55.27. EI-MS *m/z* 208 (M^+).

1-Phenyl-2-(*m*-tolyl)acetylene (12d)[6] [CAS: 14635-91-7]



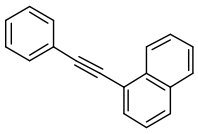
^{1}H NMR (396 MHz, $CDCl_3$) δ 7.54–7.51 (m, 2H, ArH), 7.37–7.32 (m, 5H, ArH), 7.24 (t, *J* = 7.8 Hz, 1H, ArH), 7.14 (d, *J* = 7.8 Hz, 1H, ArH), 2.35 (s, 3H, -CH_3). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 138.00, 132.17, 131.58, 129.15, 128.67, 128.31, 128.23, 128.15, 123.36, 123.04, 89.53, 89.00, 21.22. EI-MS *m/z* 192 (M^+).

1-Phenyl-2-(*o*-tolyl)acetylene (12e)[5] [CAS: 14309-60-5]



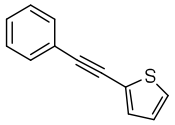
^{1}H NMR (396 MHz, $CDCl_3$) δ 7.55–7.49 (m, 3H, ArH), 7.38–7.33 (m, 3H, ArH), 7.24–7.15 (m, 3H, ArH), 2.52 (s, 3H, -CH_3). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 140.17, 131.80, 131.48, 129.44, 128.43, 128.33, 128.28, 128.16, 125.56, 123.50, 93.30, 88.29, 20.74. EI-MS *m/z* 192 (M^+).

1-(1-Naphthyl)-2-phenyl acetylene (12f)[5] [CAS: 4044-57-9]



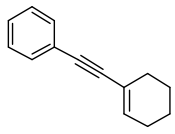
^{1}H NMR (396 MHz, $CDCl_3$) δ 8.45 (d, *J* = 9.1 Hz, 1H), 7.86 (t, *J* = 8.9 Hz, 2H), 7.77 (dd, *J* = 1.2, 7.2 Hz, 1H), 7.67–7.65 (m, 2H), 7.63–7.58 (m, 1H), 7.56–7.52 (m, 1H), 7.47 (dd, *J* = 7.2, 8.1 Hz, 1H), 7.43–7.36 (m, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 133.24, 133.18, 131.65, 130.43, 128.74, 128.41, 128.37, 128.29, 126.76, 126.41, 126.20, 125.26, 123.37, 120.86, 94.29, 87.50. EI-MS *m/z* 228 (M^+).

1-Phenyl-2-(2-thienyl)acetylene (12g)[7] [CAS: 4805-17-8]



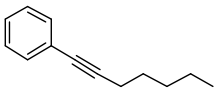
^{1}H NMR (396 MHz, $CDCl_3$) δ 7.54–7.49 (m, 2H, ArH), 7.38–7.33 (m, 3H, ArH), 7.30–7.28 (m, 2H, ArH), 7.01 (dd, *J* = 3.6, 5.1 Hz, 1H, 4-thienyl-H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 131.87, 131.39, 128.40, 128.35, 127.23, 127.08, 123.29, 122.89, 92.99, 82.57. EI-MS *m/z* 184 (M^+).

(1-Cyclohyxenylethynyl)benzene (12h)[7] [CAS: 13456-84-3]



^{1}H NMR (396 MHz, $CDCl_3$) δ 7.43–7.40 (m, 2H, ArH), 7.32–7.25 (m, 3H, ArH), 6.23–6.20 (m, 1H, CH), 2.25–2.20 (m, 2H, -CH_2-), 2.17–2.12 (m, 2H,-CH_2-), 1.70–1.60 (m, 4H, -CH_2-). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 135.18, 131.40, 128.19, 127.69, 123.69, 120.67, 91.21, 86.72, 29.19, 25.74, 22.32, 21.49. EI-MS *m/z* 182 (M^+).

1-Heptyn-1-yl-benzene[8] [CAS: 14374-45-9]



^{1}H NMR (396 MHz, CDCl3) δ 7.41–7.38 (m, 2H, ArH), 7.29–7.25 (m, 3H, ArH), 2.40 (t, *J* = 6.9 Hz, 2H), 1.63–1.54 (m, 2H), 1.49–1.32 (m, 4H), 0.92 (t, J = 6.9 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 135.53, 128.18, 127.48, 124.06, 90.50, 80.51, 31.15, 28.42, 22.26, 19.39, 14.03. EI-MS *m/z* 172 (M^+).

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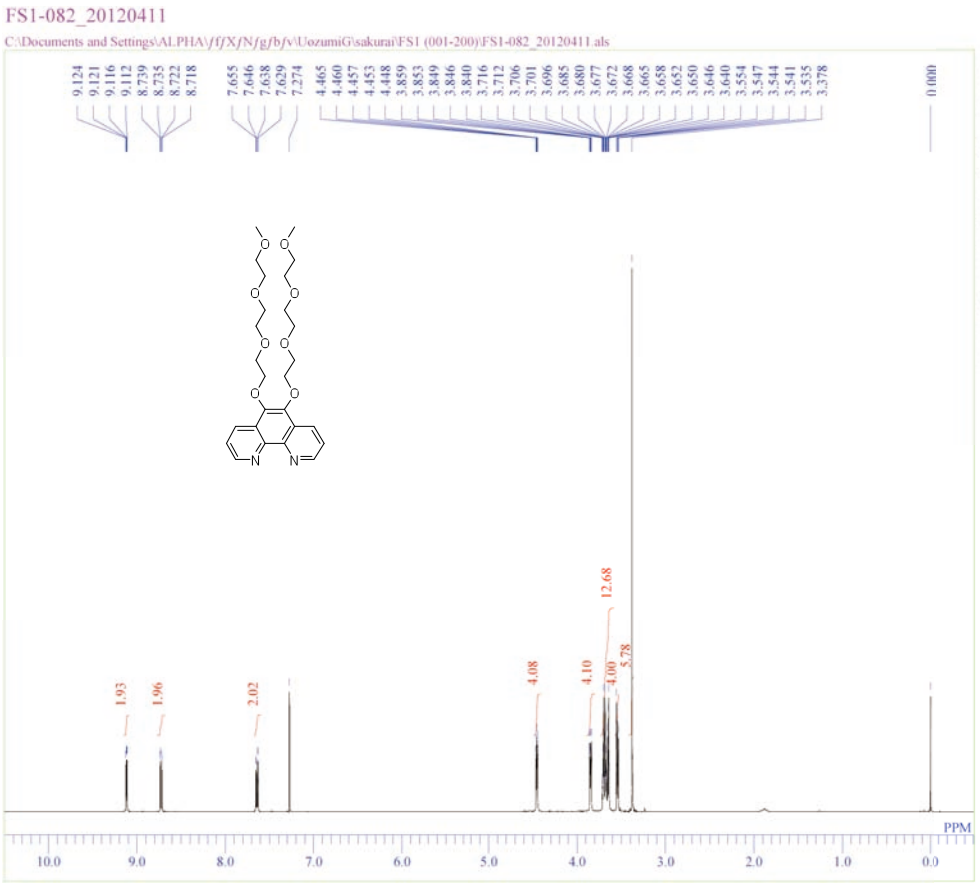
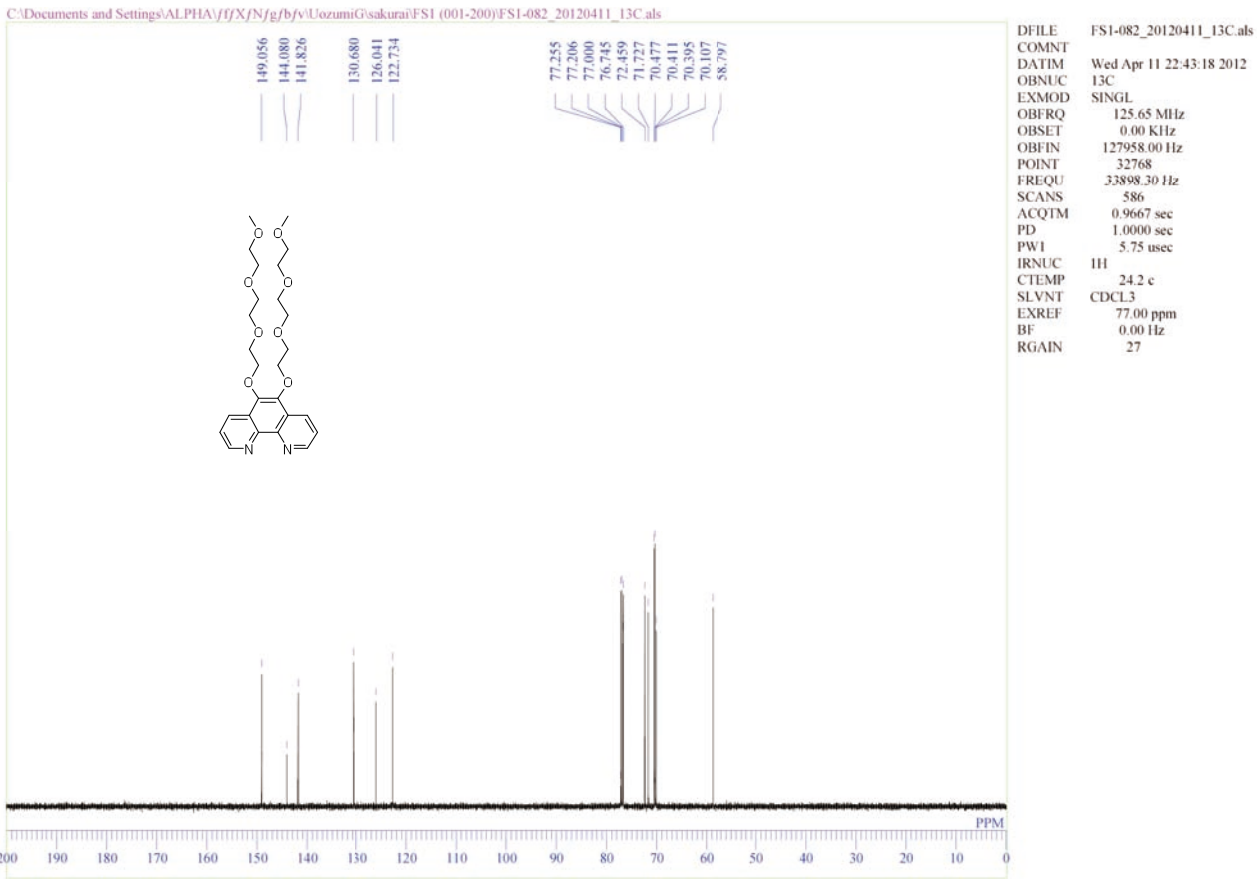
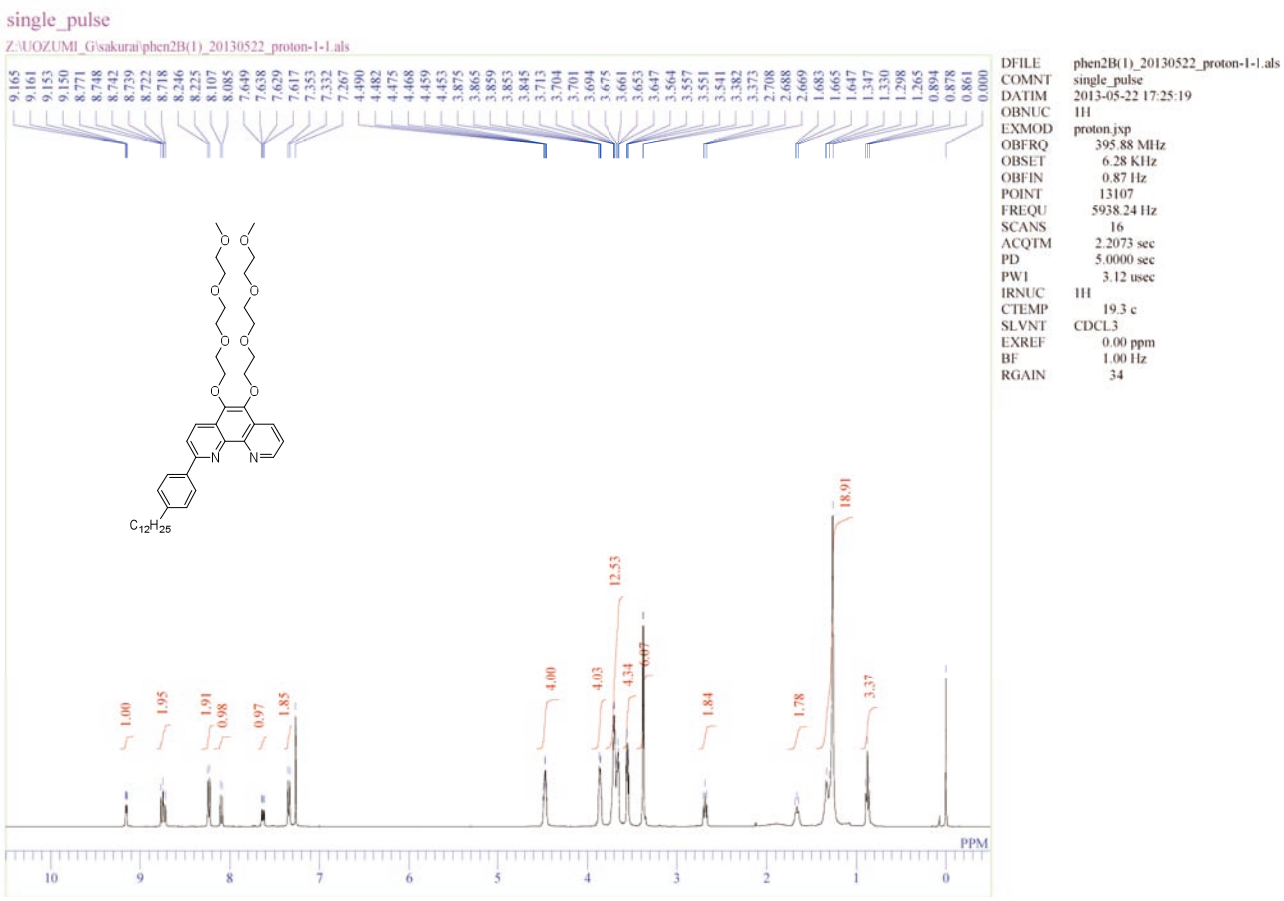
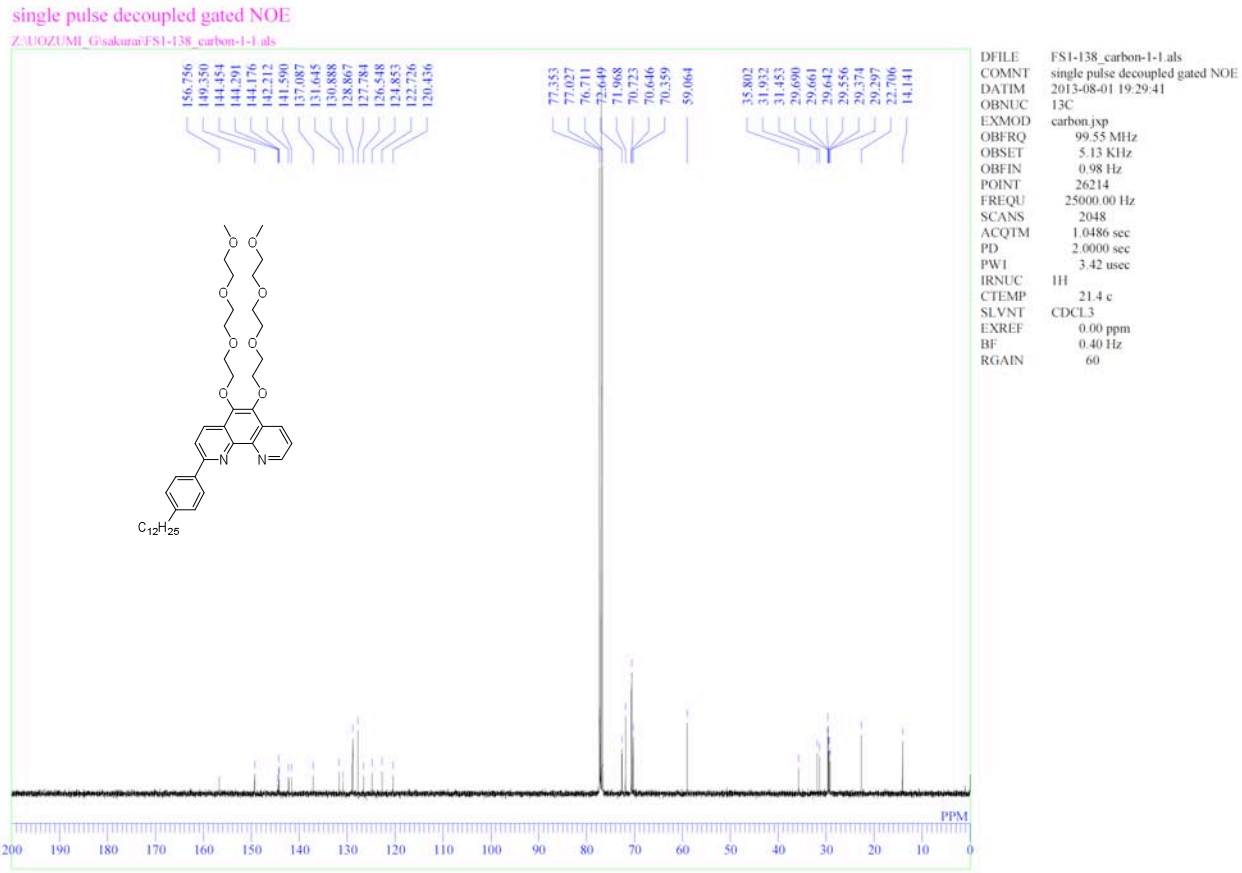
5,6-Bis[2-[2-(2-methoxyethoxy)ethoxy]ethoxy]-1,10-phenanthroline (4)





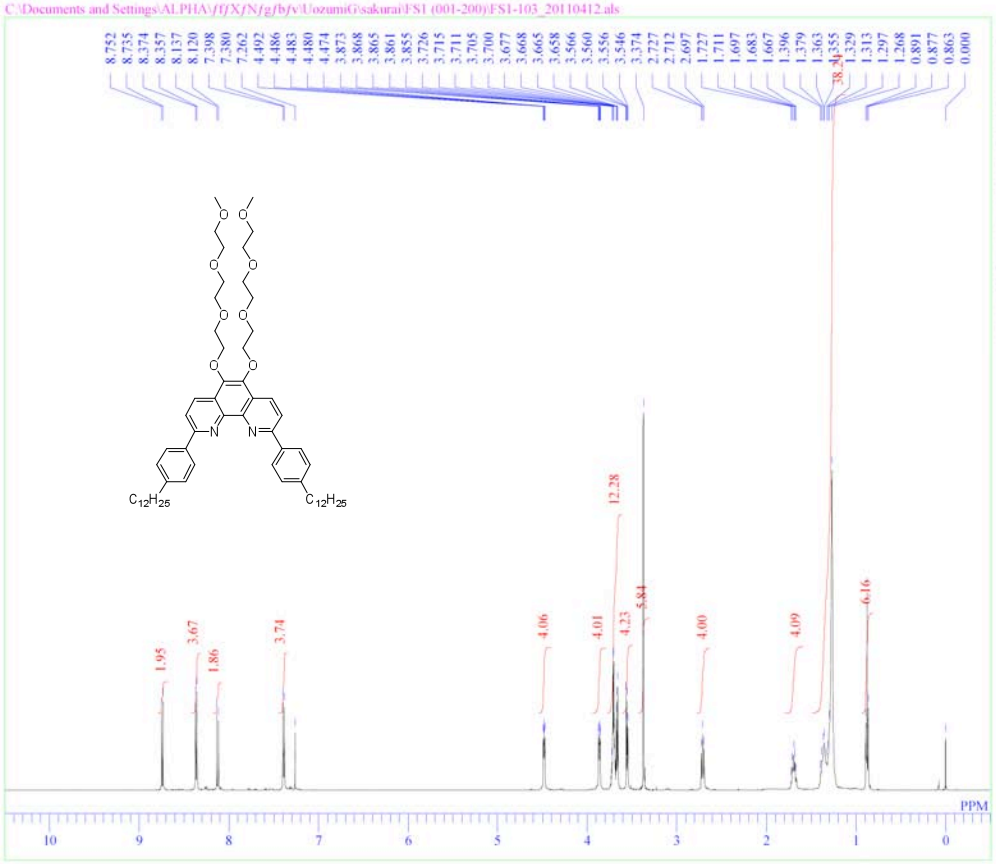
2-(4-Dodecylphenyl)-5,6-bis[2-[2-(2-methoxyethoxy)ethoxy]ethoxy]-1,10-phenanthroline (5)



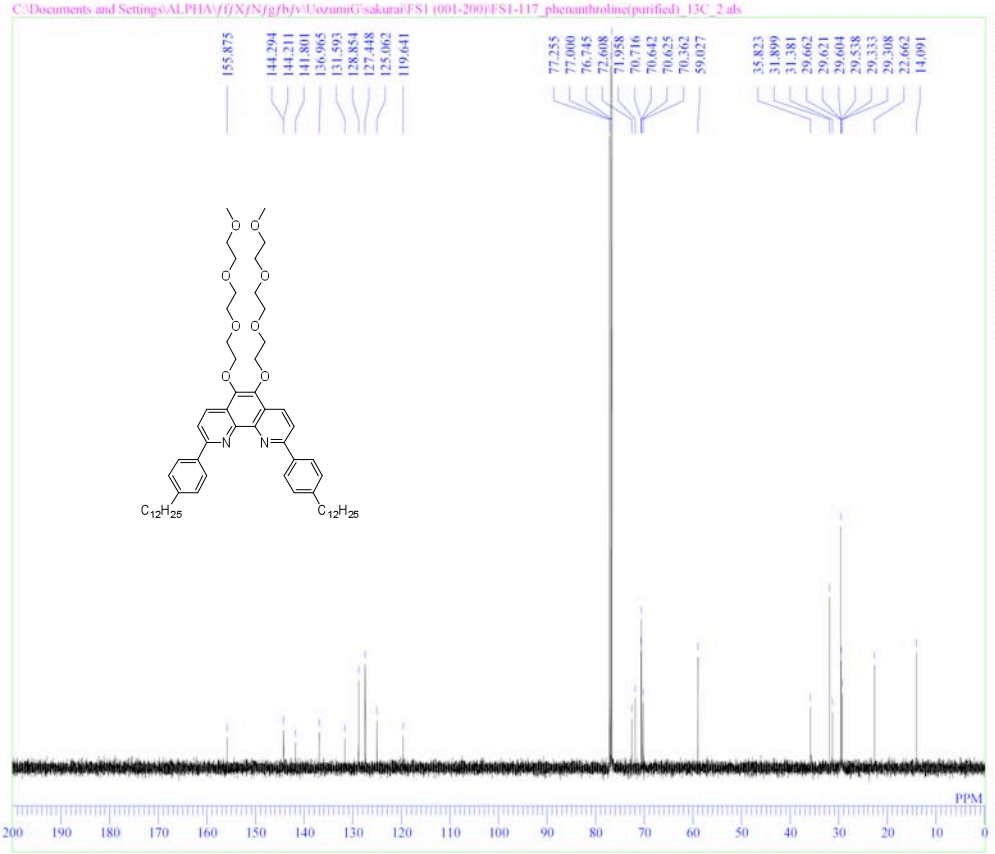


2,9-Bis(4-dodecylphenyl)-5,6-bis[2-[2-(2-methoxyethoxy)ethoxy]ethoxy]-1,10-phenanthroline (6)

FS1-103_20110412

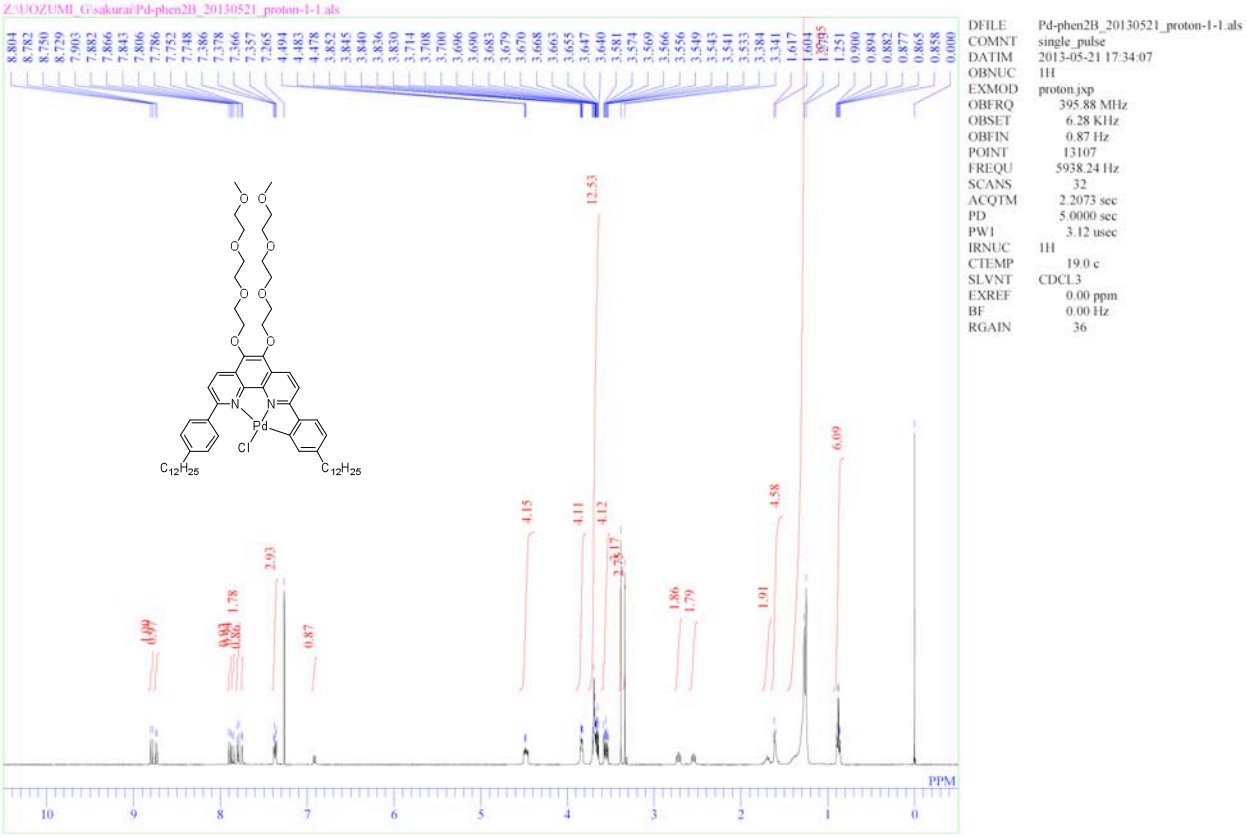


FS1-117_phenanthroline(purified)

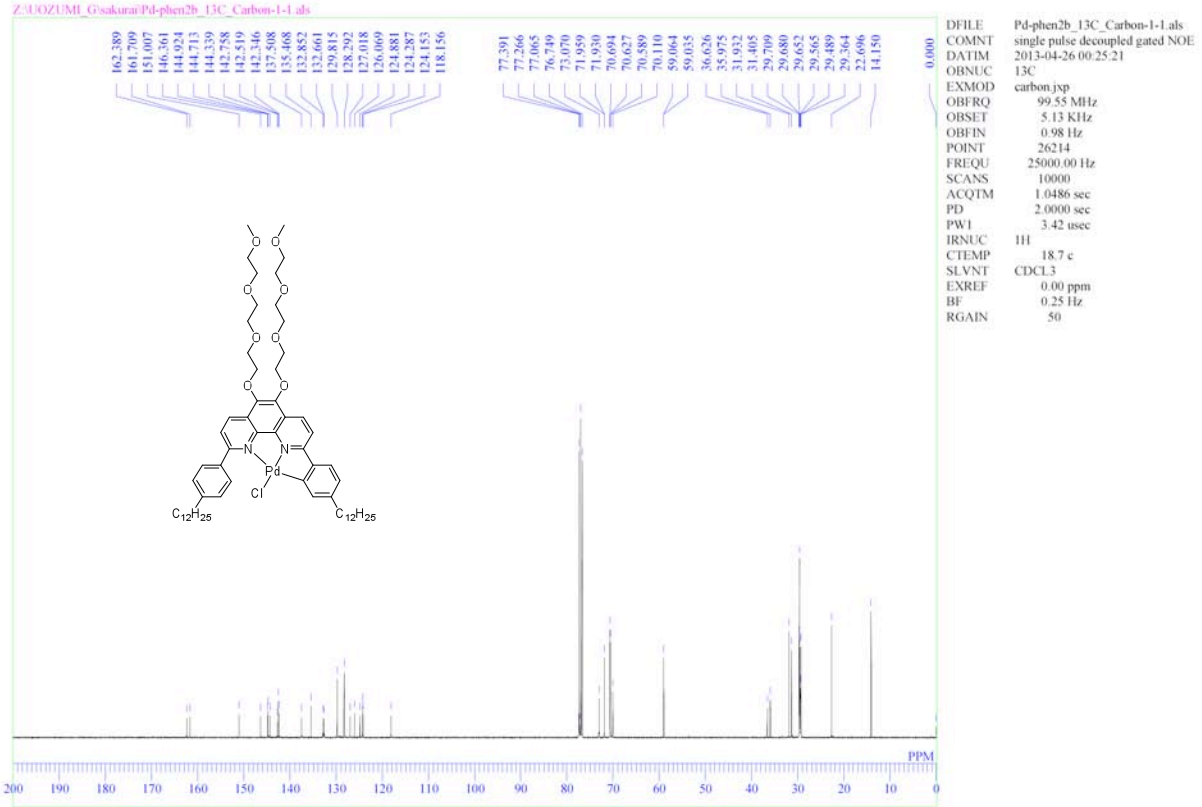


Chloro-[5-dodecyl-2-{9-(4-dodecylphenyl)-5,6-bis(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)-1,10-phenanthrolin-2-yl}phenyl]palladium (2a)

single_pulse



single pulse decoupled gated NOE



5,6-Bis(dodecyloxy)-1,10-phenanthroline (7)

FS2-133_phen2b(0)

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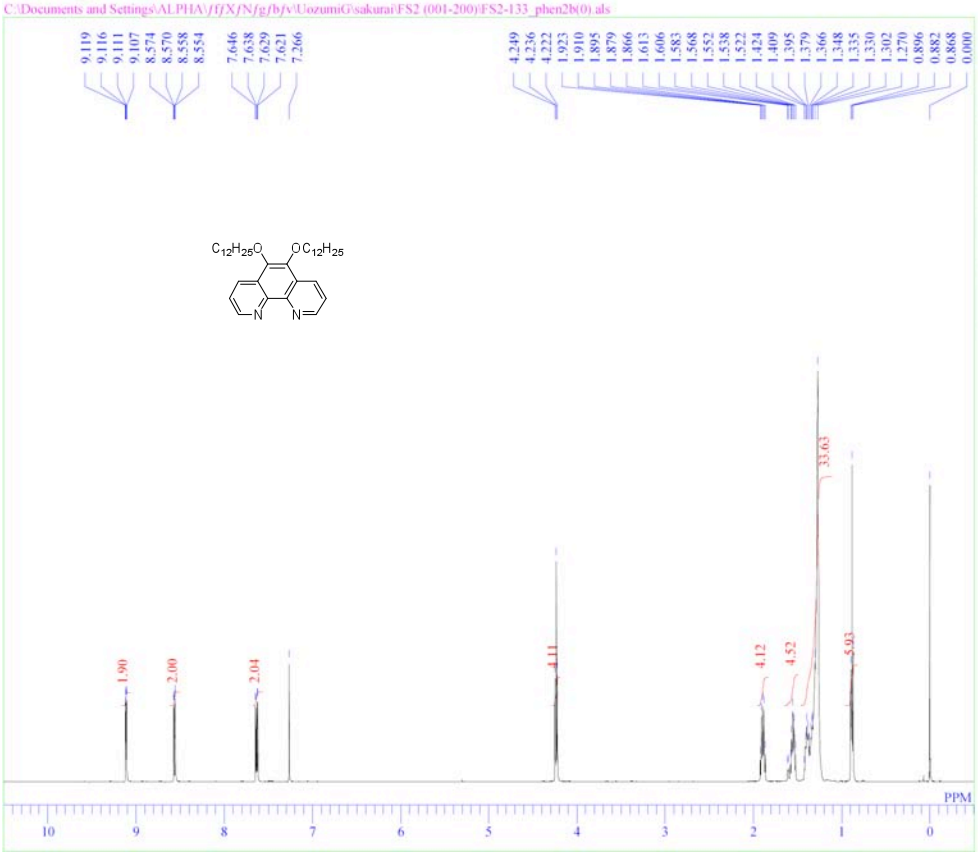

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EXMOD SINGL
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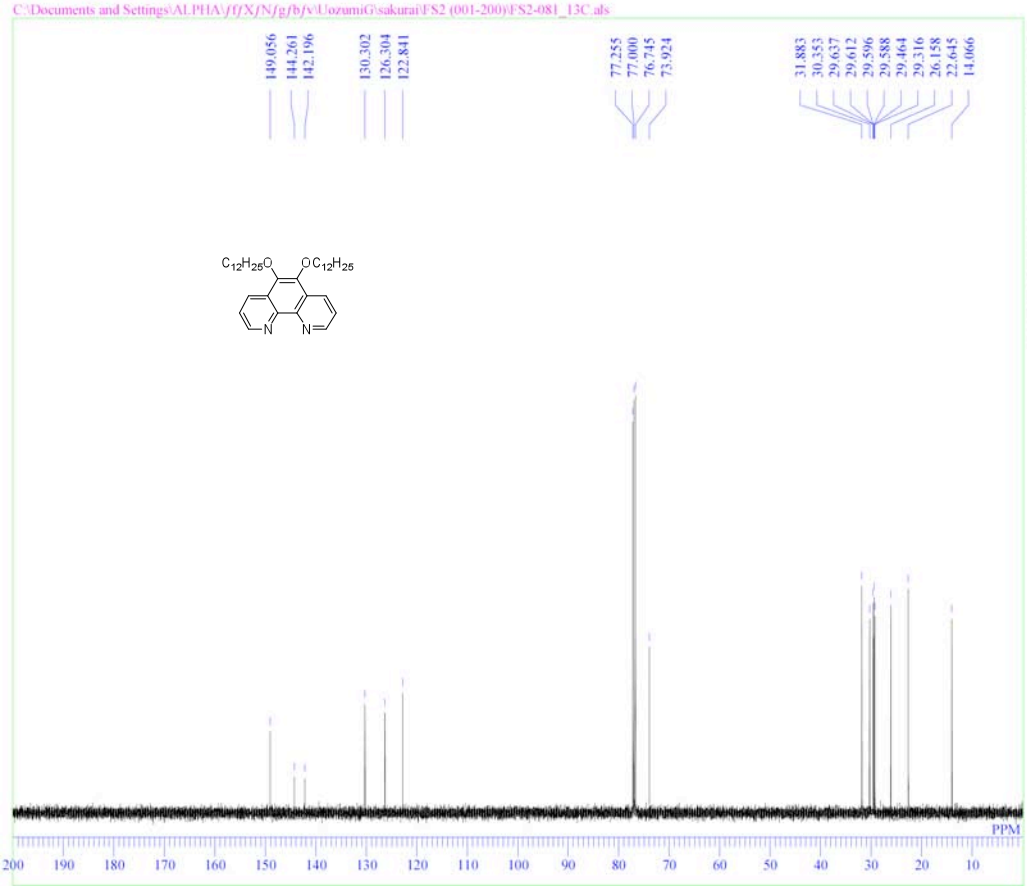
OBSET 0.00 KHz
OBFIN 162410.00 Hz
POINT 16384
FREQU 10000.00 Hz
SCANS 16
ACQTM 1.6384 sec
PD 2.0000 sec
PW1 6.40 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 16

FS2-081_13C

C:\Documents and Settings\ALPHA\ƒƒX/N/g/b/v\UozumiG\sakurai\FS2 (001-200)\FS2-081_13C.als



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DATIM Tue Sep 11 21:33:17 2012
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EXMOD SINGL
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OBSET 0.00 KHz
OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 512
ACQTM 0.9667 sec
PD 1.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 25.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 26

5,6-Bis(dodecyloxy)-2-[4-[2-[2-(2-methoxyethoxy)ethoxy]ethoxy]phenyl]-1,10-phenanthroline (8)

FS2-105_20120926

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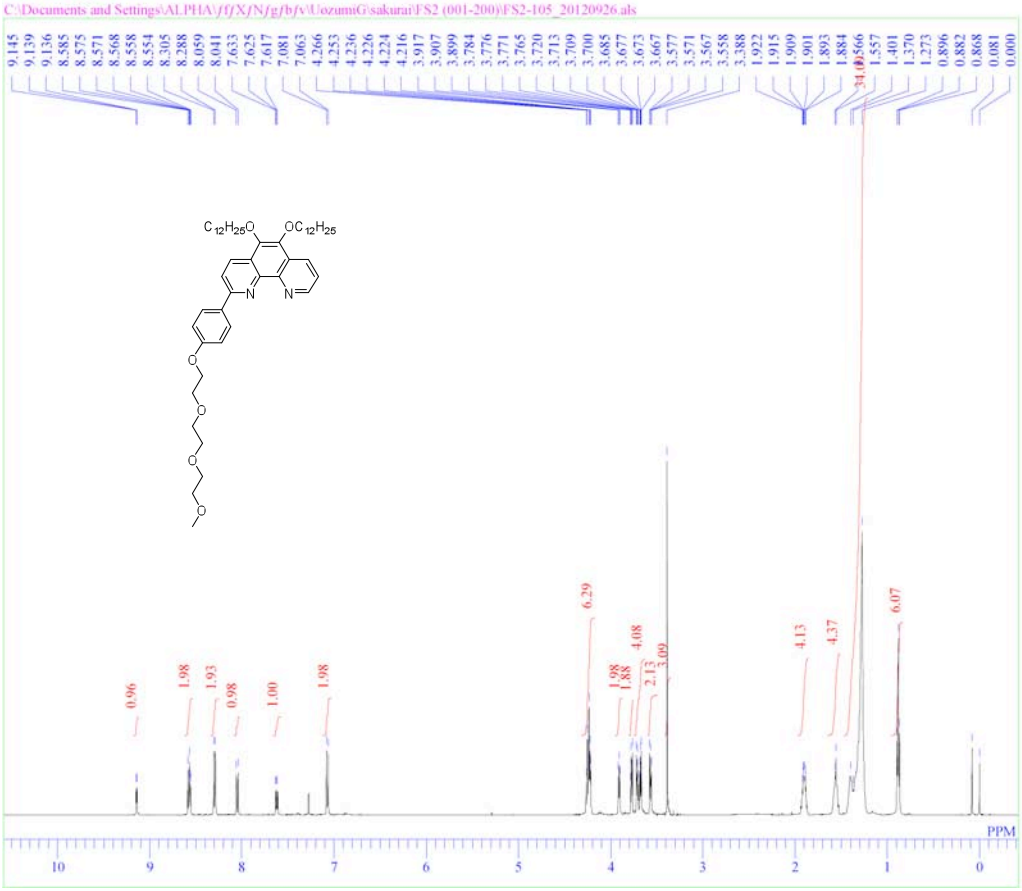

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DATIM	Wed Sep 26 11:21:06 2012
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EXMOD	SINGL
OBFRQ	500.00 MHz
OBSET	0.00 KHz
OBFIN	162410.00 Hz
POINT	16384
FREQU	10000.00 Hz
SCANS	8
ACQTM	1.6384 sec
PD	2.0000 sec
PW1	6.40 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.00 Hz
RGAIN	12

FS2-099_20120915_13C

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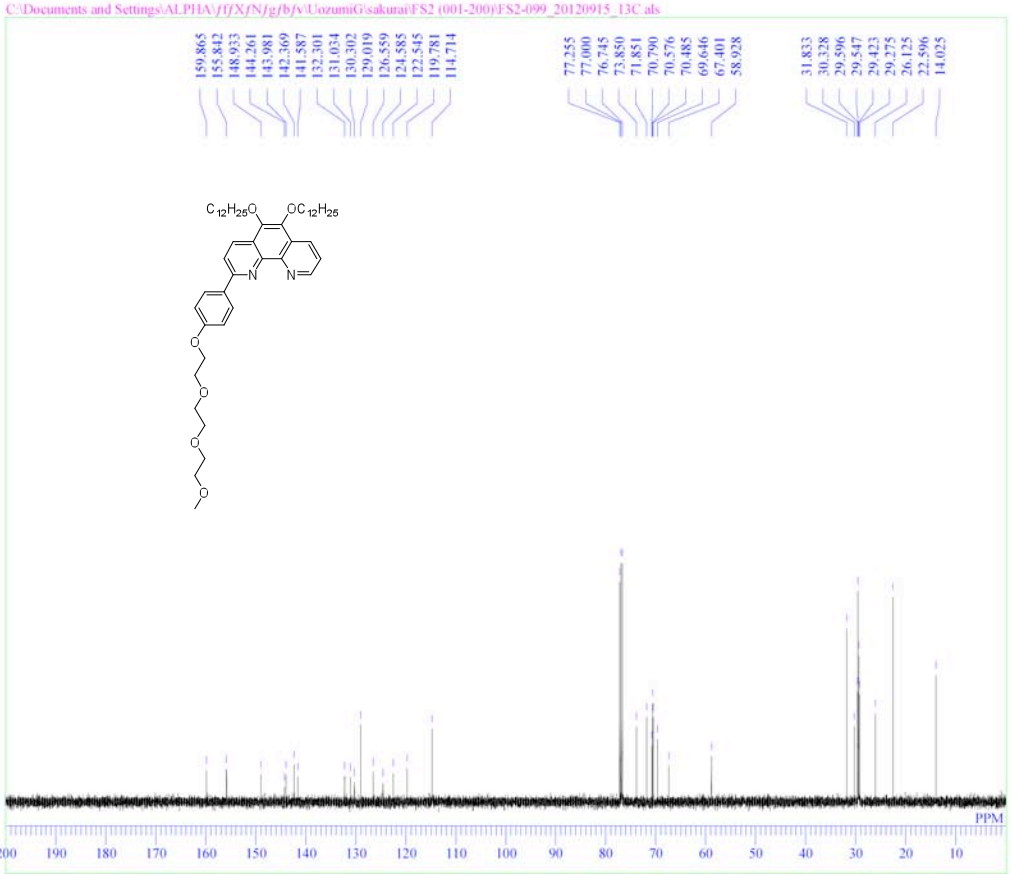

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OBSET	0.00 KHz
OBFIN	127958.00 Hz
POINT	32768
FREQU	33898.30 Hz
SCANS	258
ACQTM	0.9667 sec
PD	1.0000 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	25.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.00 Hz
RGAIN	27

5,6-Bis(dodecyloxy)-2,9-bis[4-[2-[2-(2-methoxyethoxy)ethoxy]ethoxy]phenyl]-1,10-phenanthroline (9)

FS2-118_phen2b_inversion

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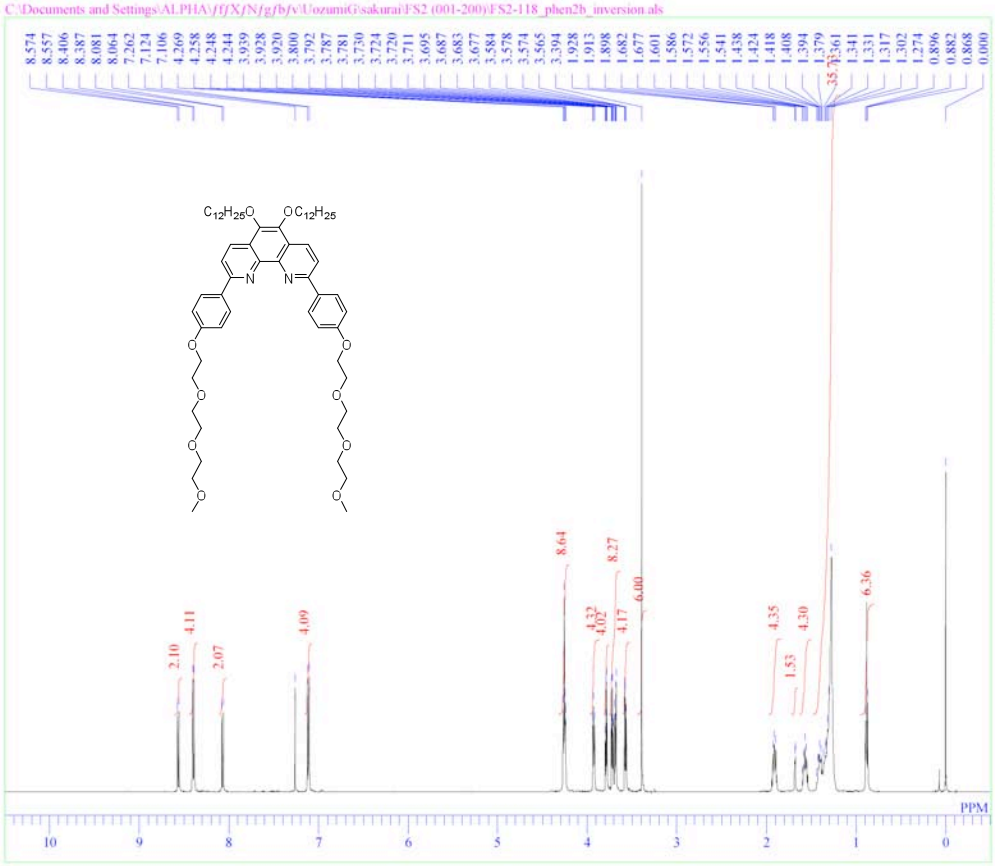

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EXMOD SINGL
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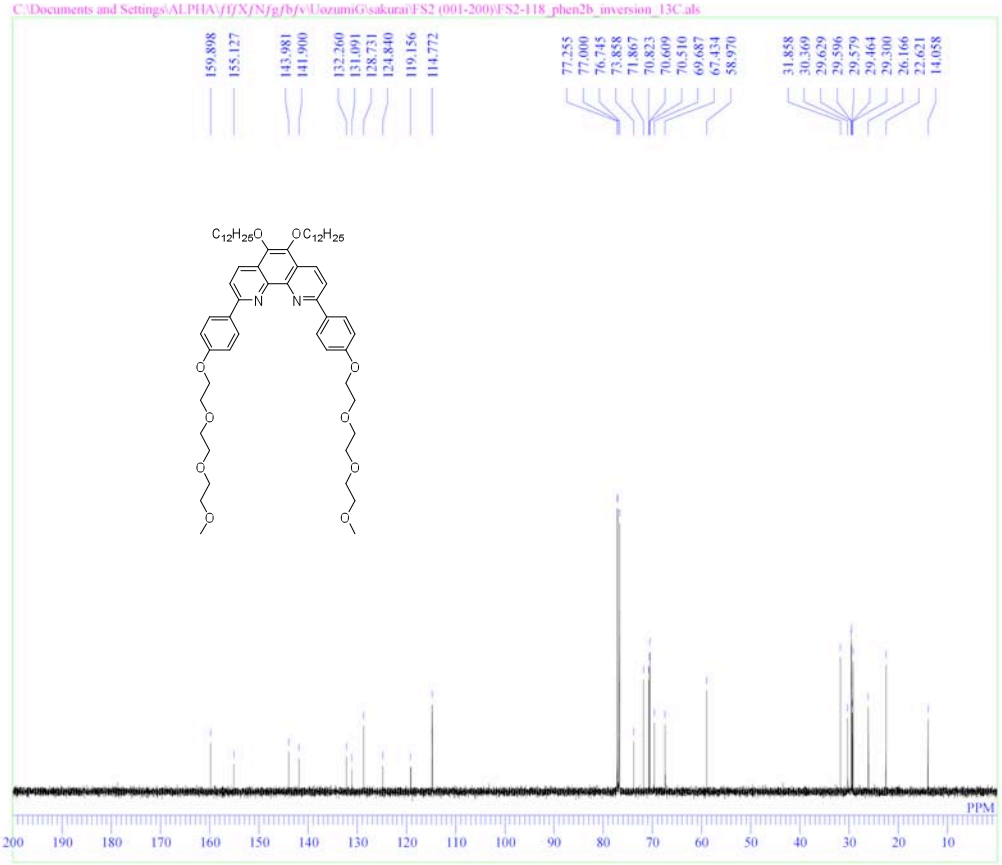
OBSET 0.00 KHz
OBFIN 162410.00 Hz
POINT 16384
FREQU 10000.00 Hz
SCANS 16
ACQTM 1.6384 sec
PD 2.0000 sec
PW1 6.40 usec
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 18

C:\Documents and Settings\ALPHA\ffjXfNfgfbfv\UozumiG\sakurai\FS2 (001-200)\FS2-118_phen2b_inversion_13C.als



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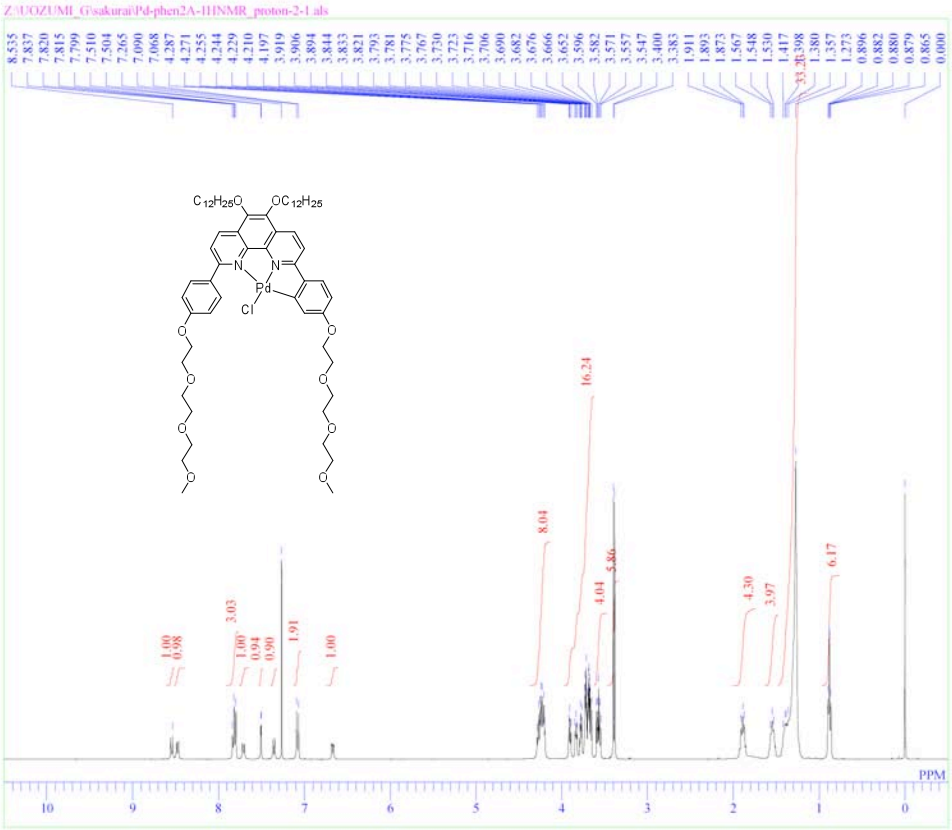
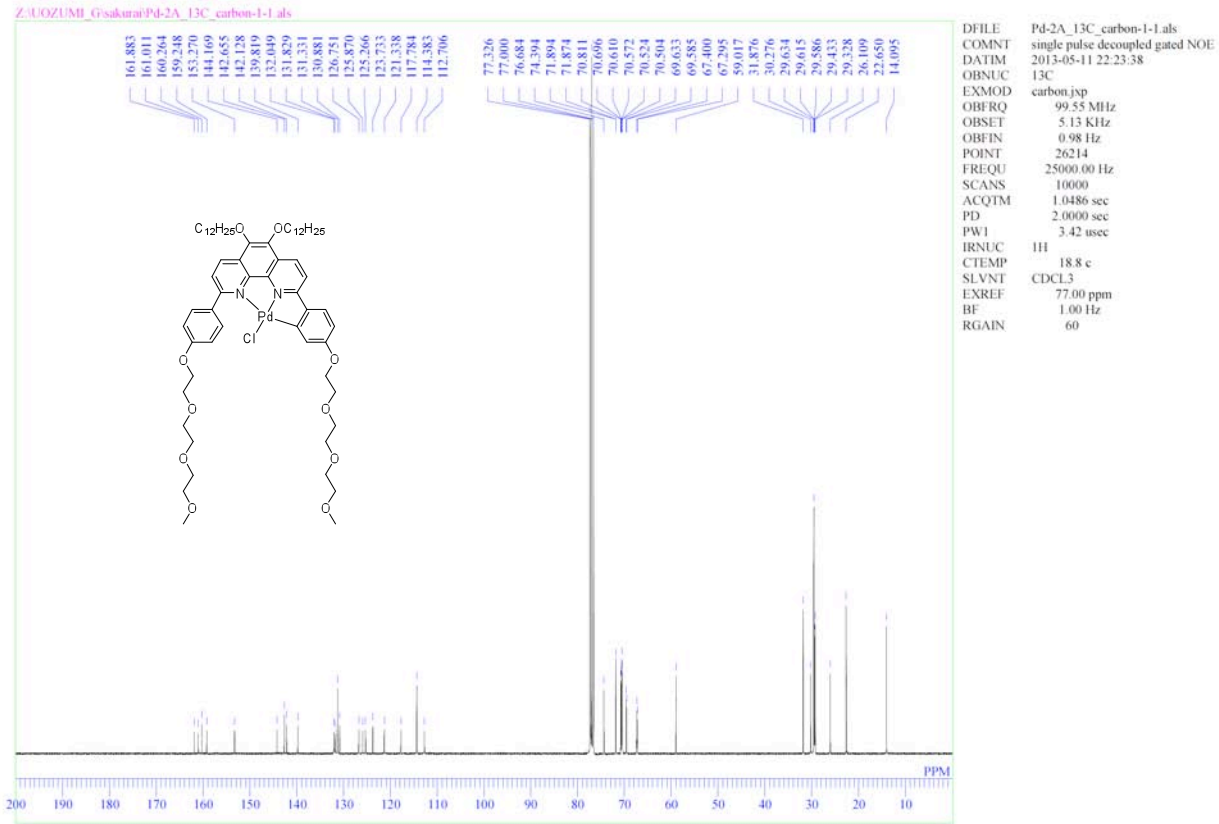
COMNT
DATIM Mon Sep 24 15:28:42 2012
OBNUC 13C
EXMOD SINGL
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 127958.00 Hz
POINT 32768
FREQU 33898.30 Hz
SCANS 427
ACQTM 0.9667 sec
PD 1.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 26

Chloro-[2-[5,6-bis(dodecyloxy)-9-{4-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)phenyl}-1,10-phenanthrolin-2-yl]-5-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)phenyl]palladium (2b)

single_pulse



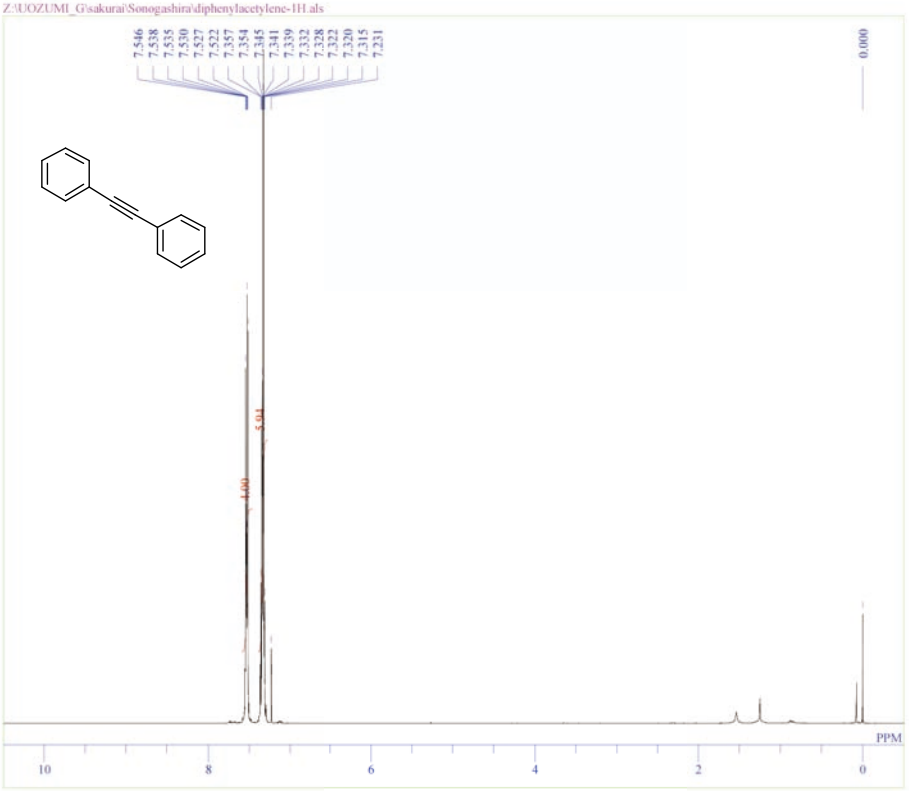
single pulse decoupled gated NOE



Diphenylacetylene (12a)

single_pulse

Z:\UOZUMI_G\sakurai\Sonogashira\diphenylacetylene-1H.als



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EXMOD	proton.jxp
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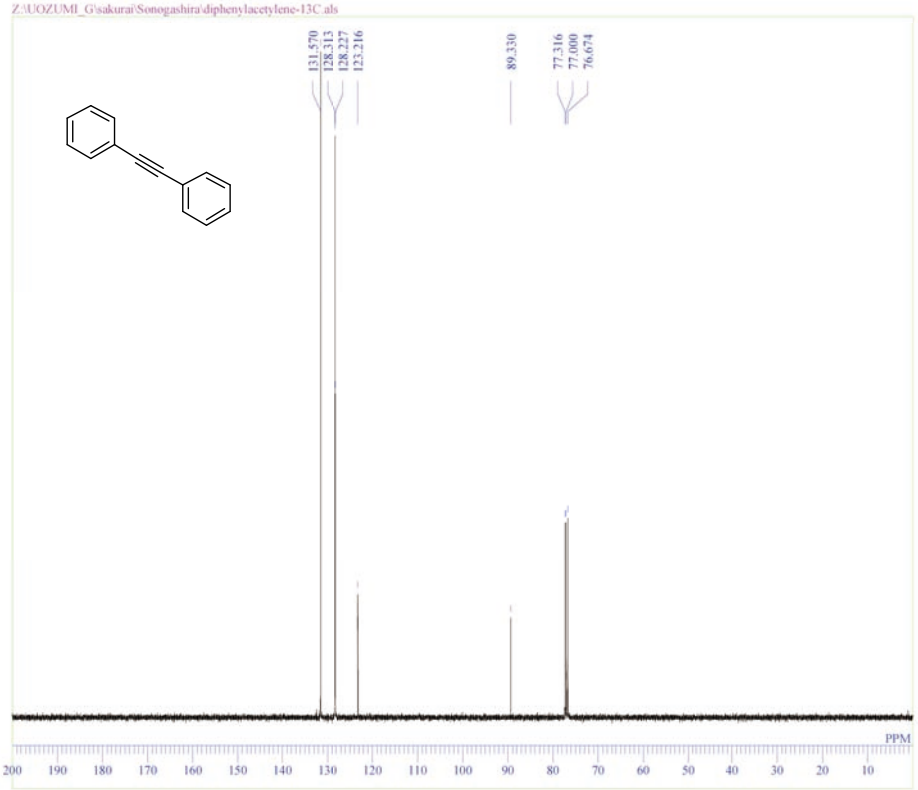
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	16384
FREQU	7422.80 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	19.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.00 Hz
RGAIN	28

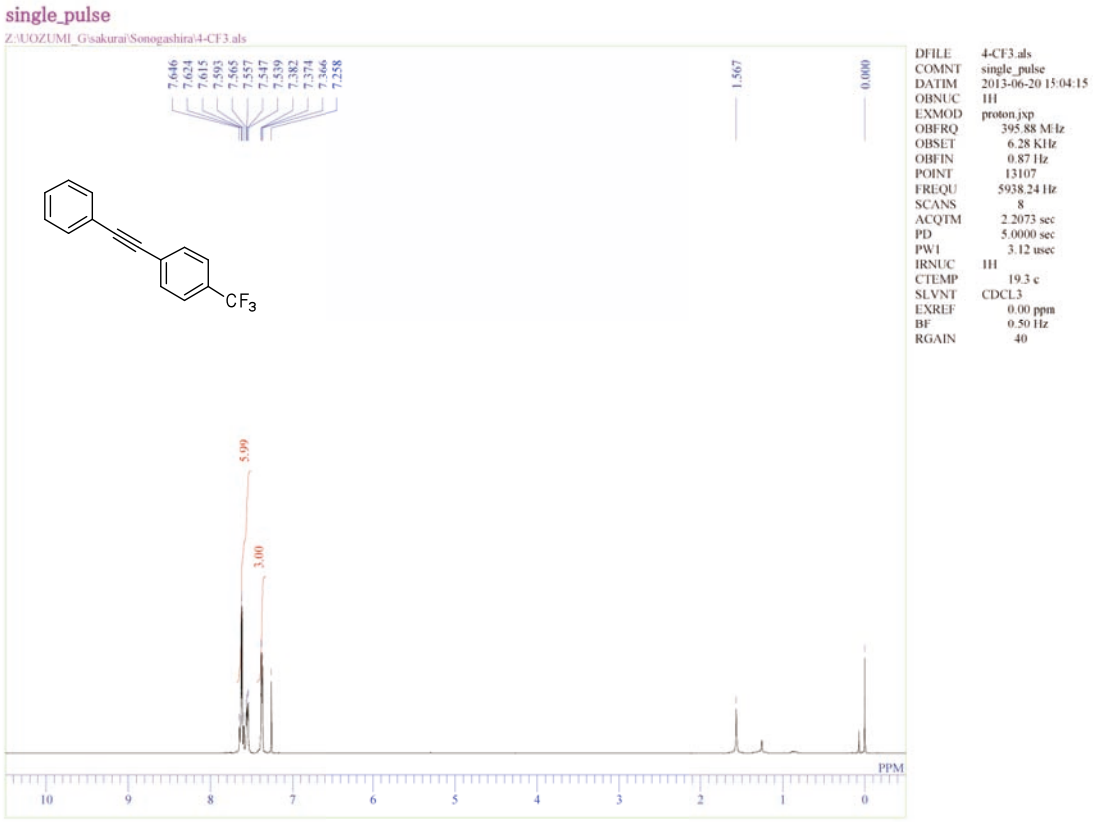
single pulse decoupled gated NOE

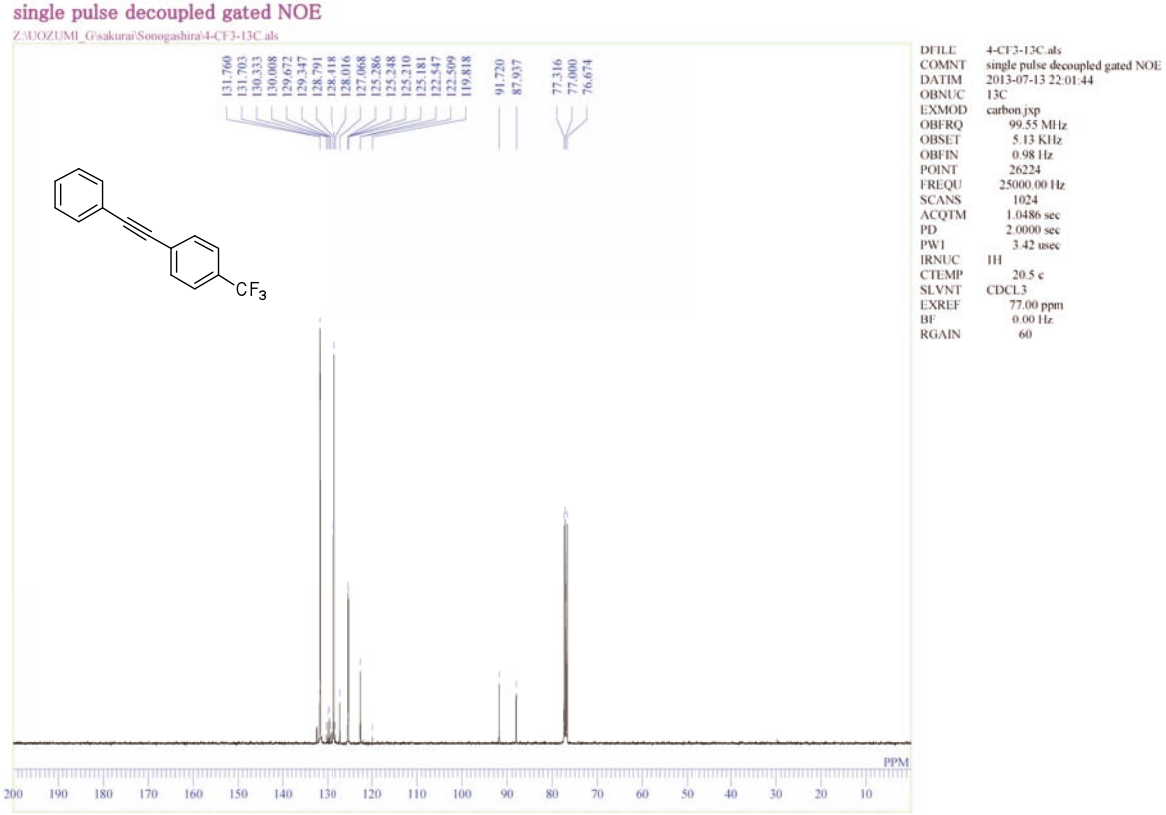
Z:\UOZUMI_G\sakurai\Sonogashira\diphenylacetylene-13C.als



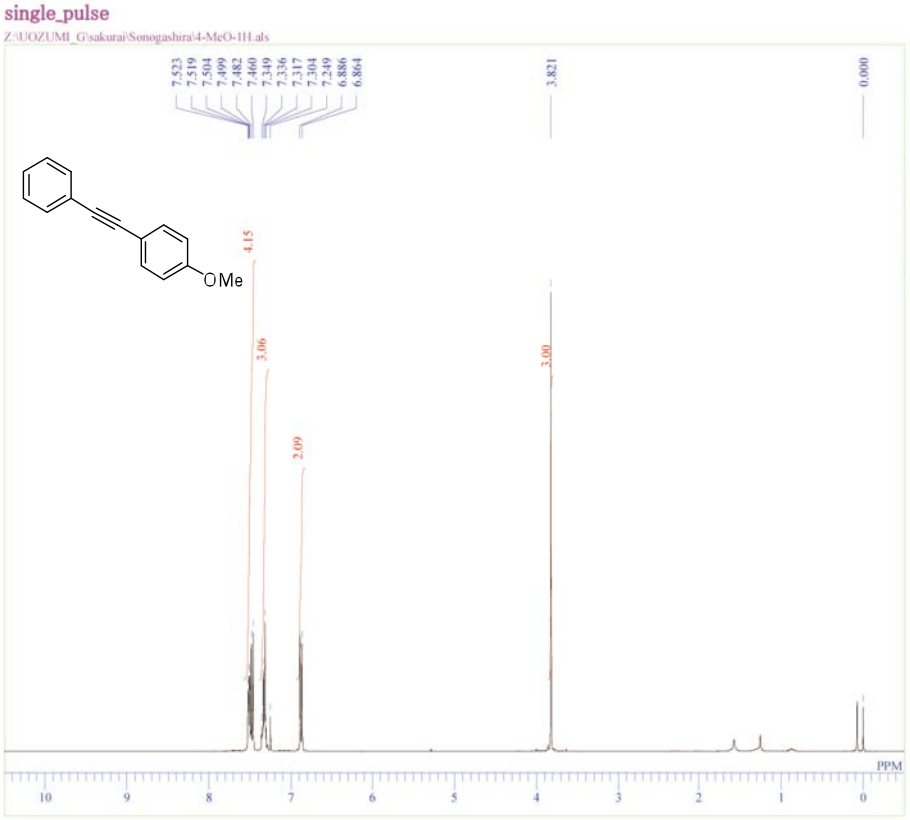
DFILE	diphenylacetylene-13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2013-06-20 15:29:46
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	161
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.42 usec
IRNUC	1H
CTEMP	19.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.50 Hz
RGAIN	60

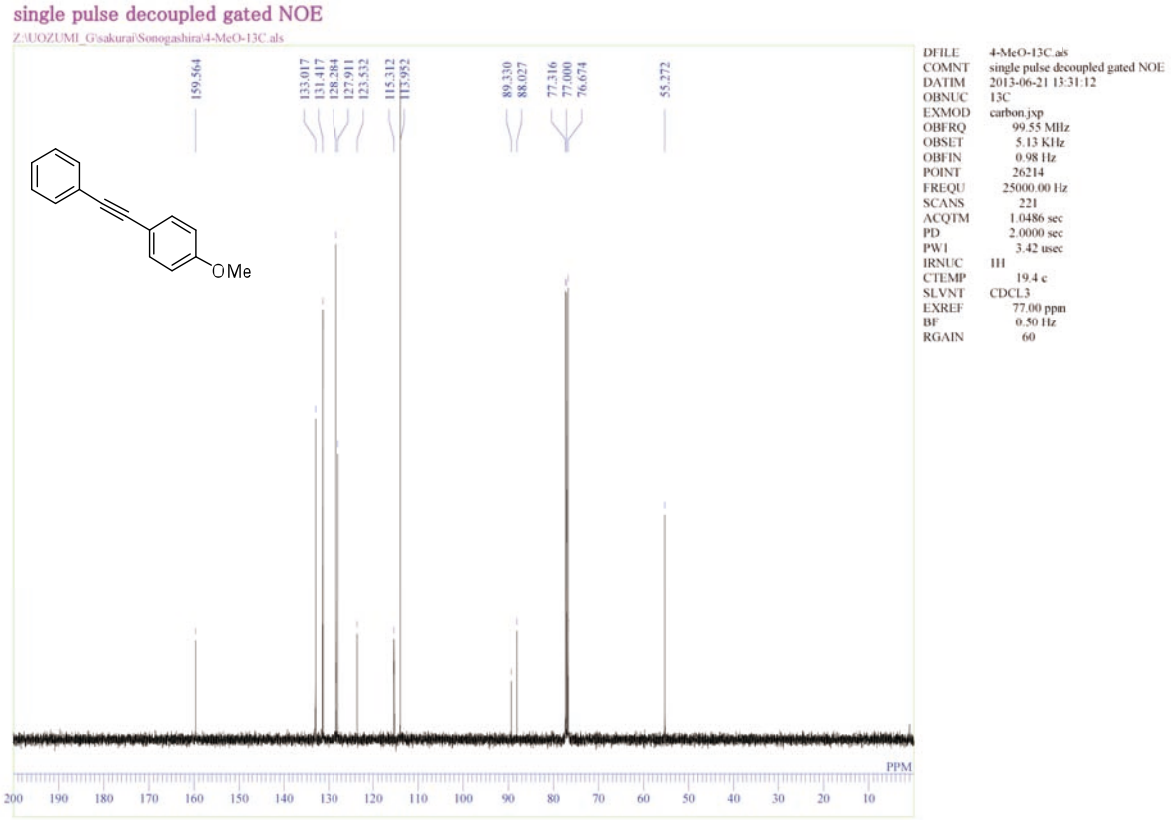
1-Phenyl-2-(*p*-trifluoromethylphenyl)acetylene (12b)



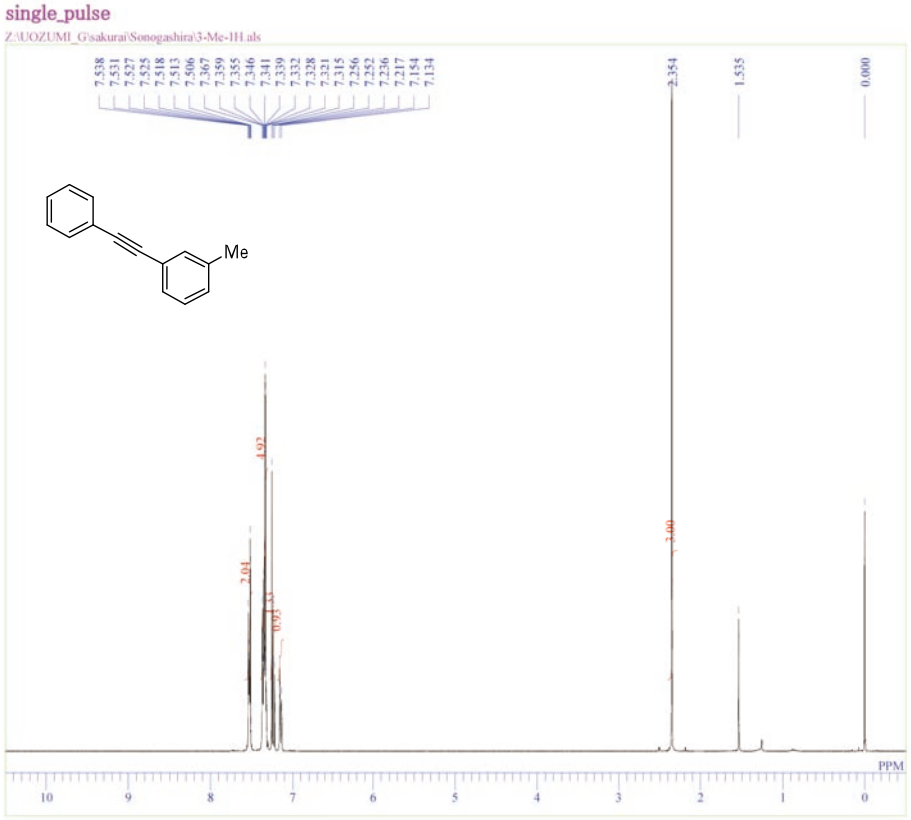


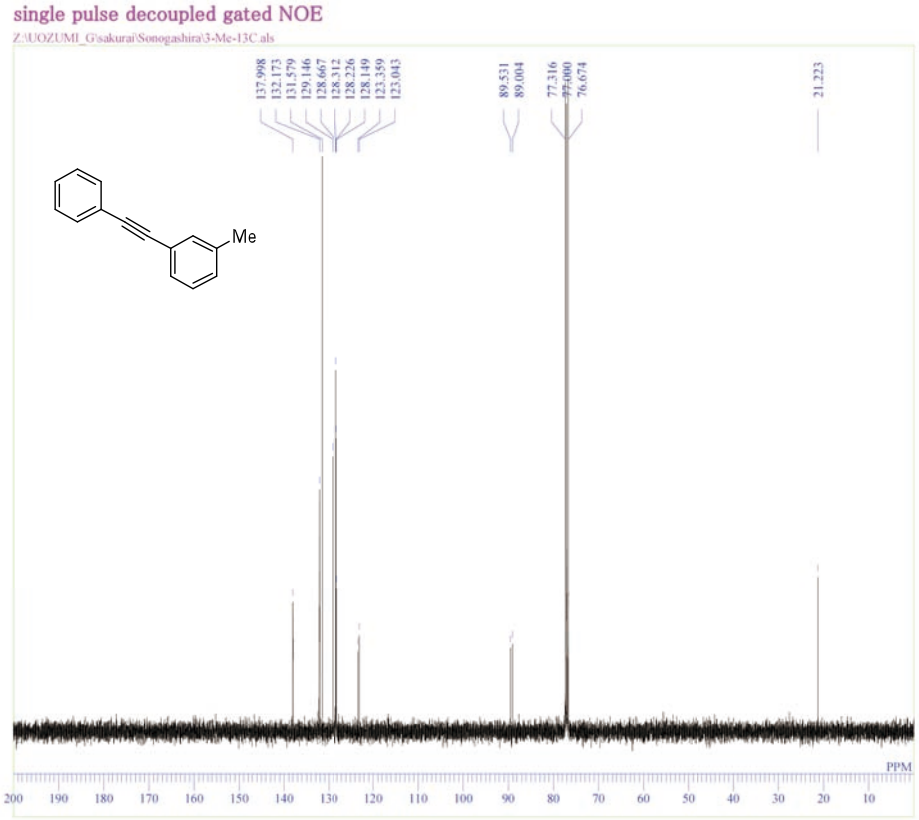
1-(*p*-Methoxyphenyl)-2-phenylacetylene (12c)



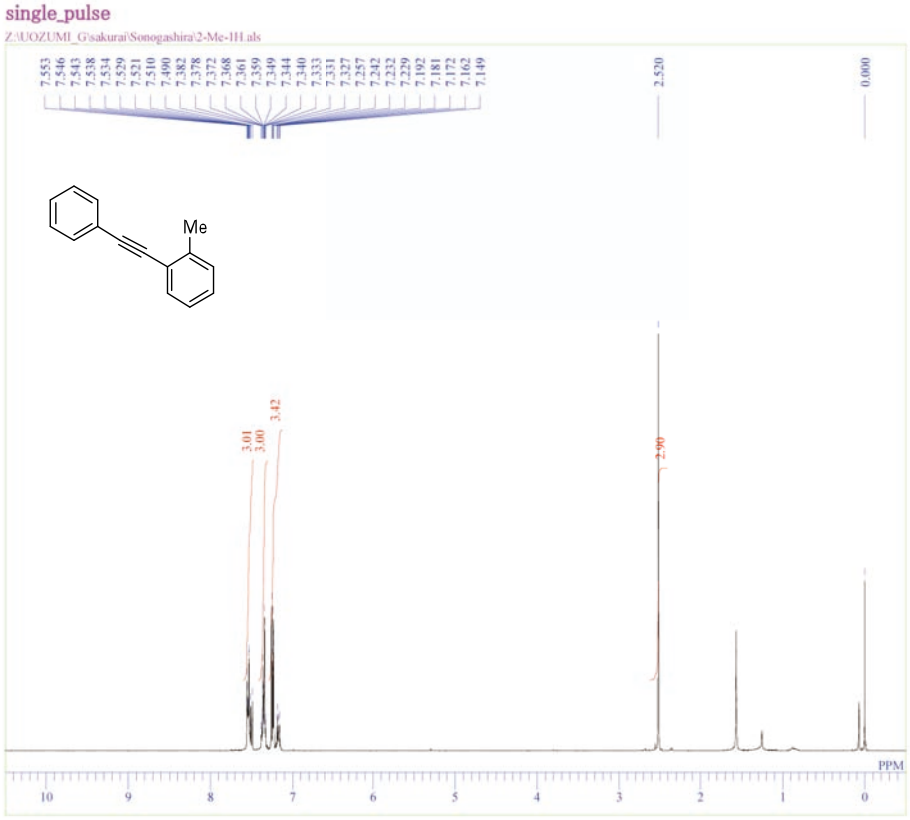


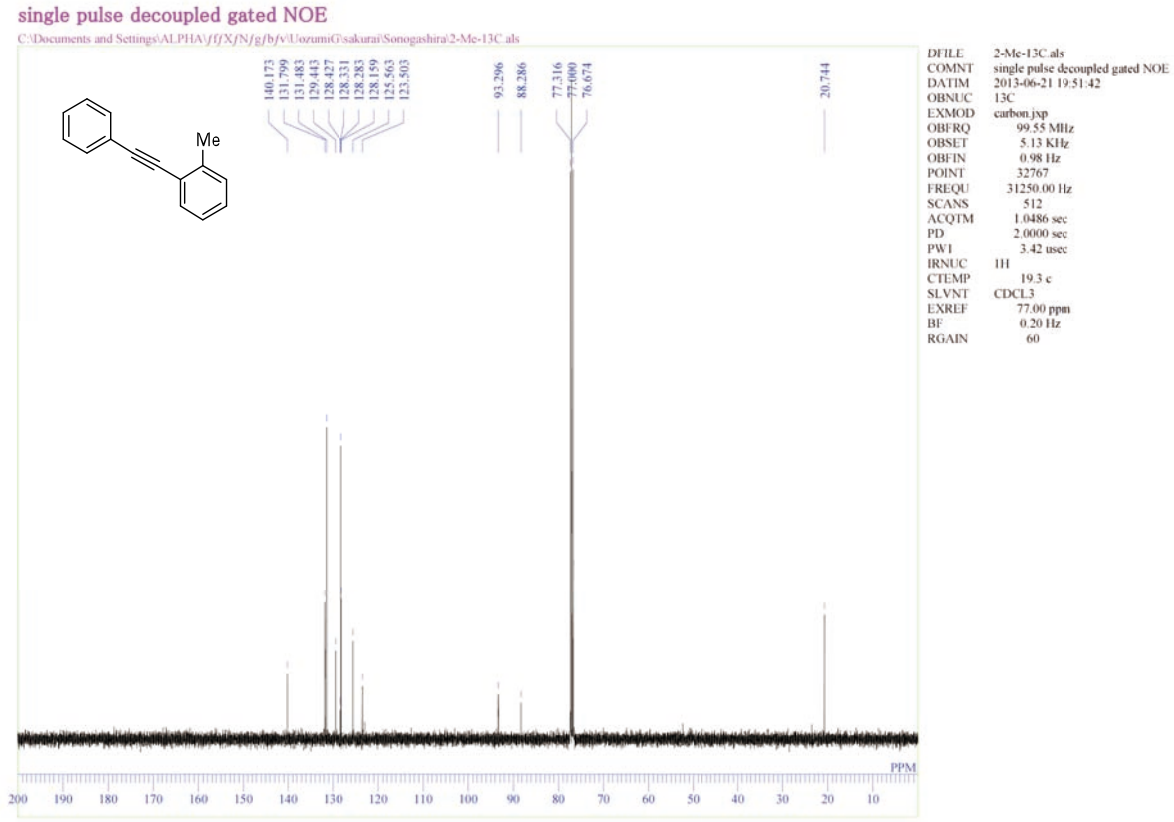
1-Phenyl-2-(*m*-tolyl)acetylene (12d)



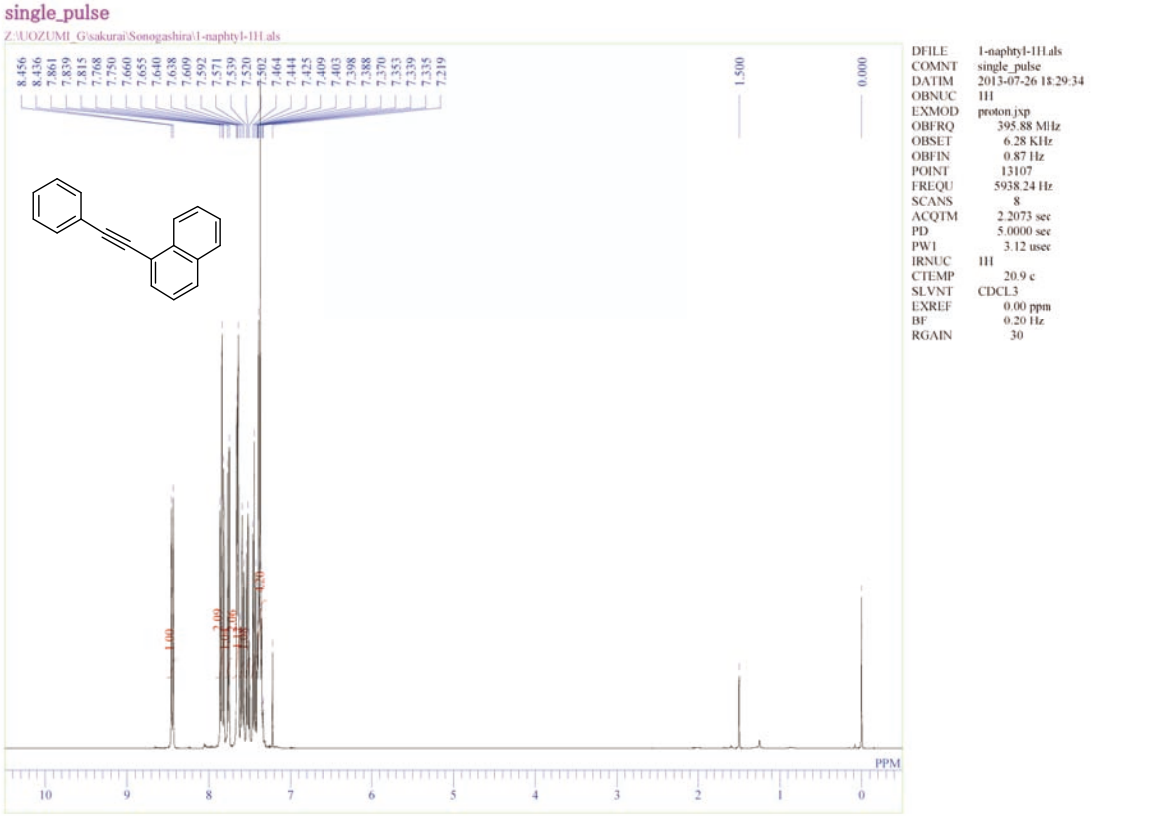


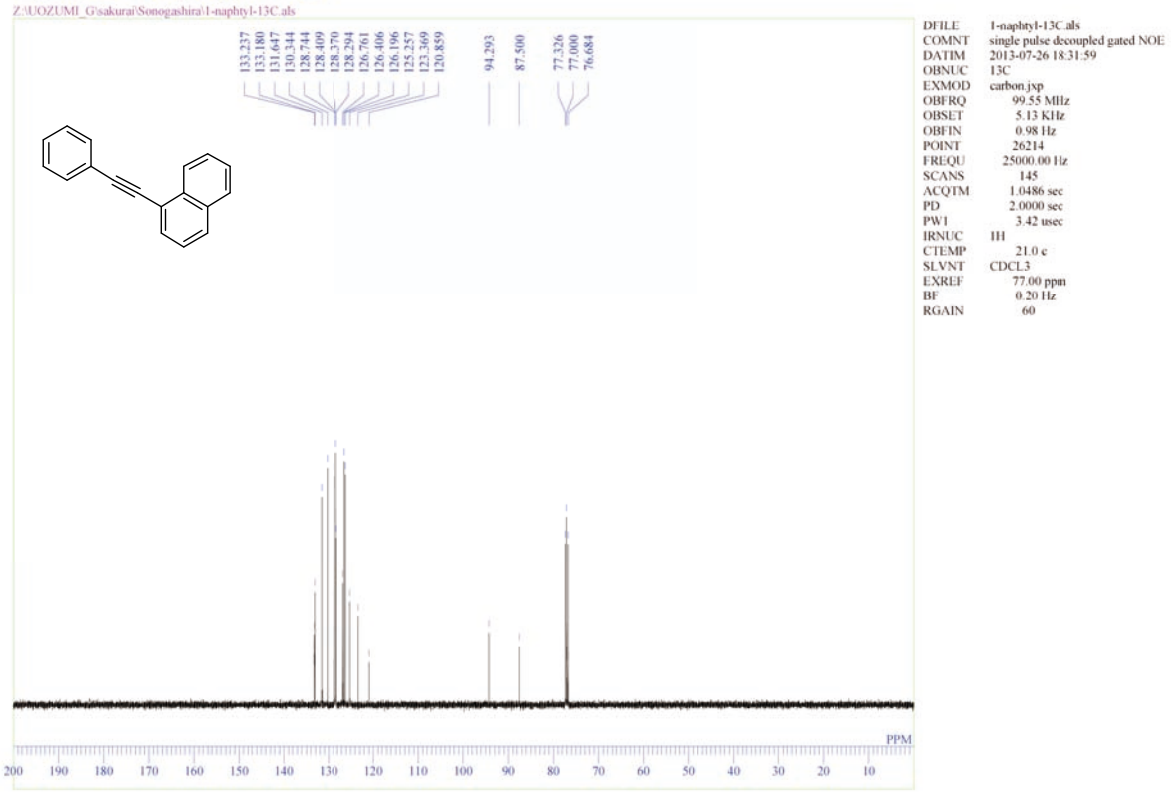
1-Phenyl-2-(*o*-tolyl)acetylene (12e)



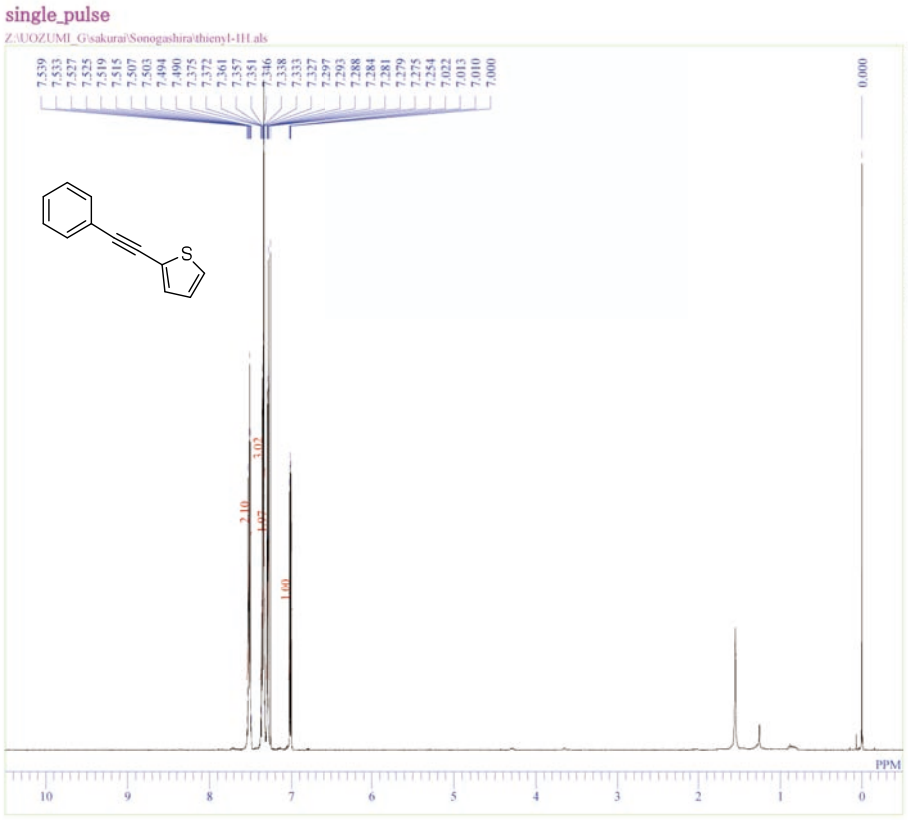


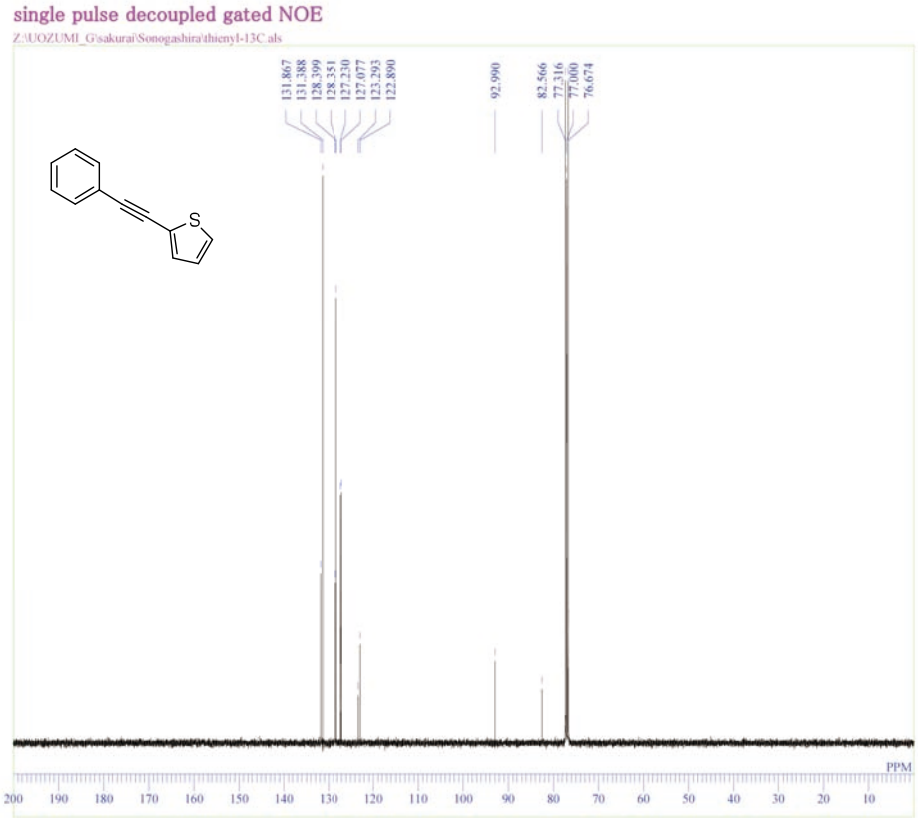
1-(1-Naphthyl)-2-phenyl acetylene (12f)



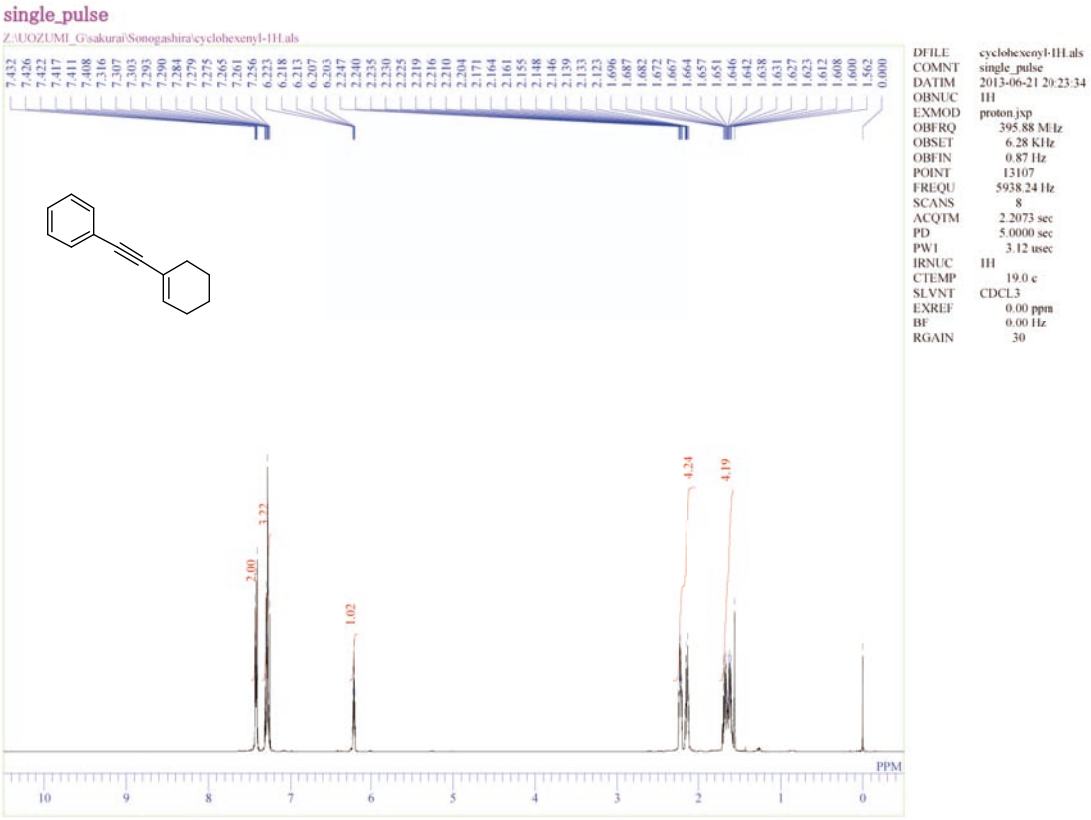


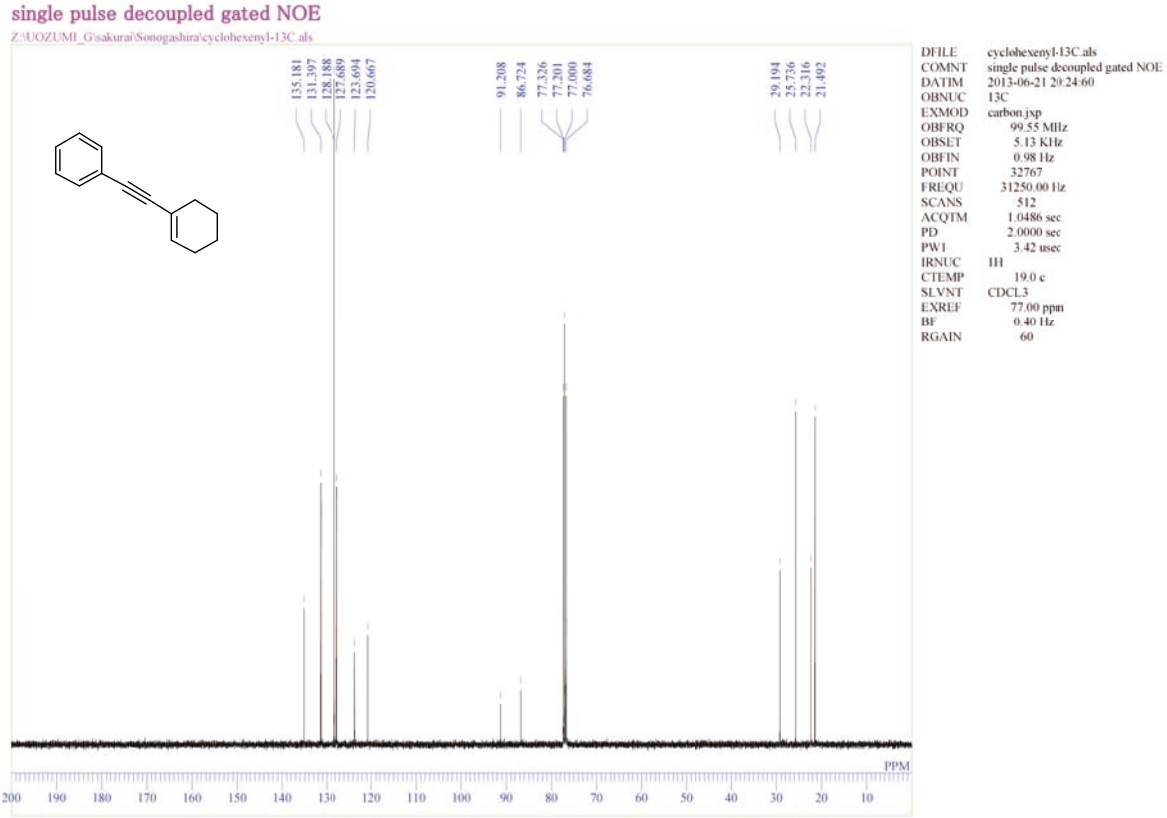
1-Phenyl-2-(2-thienyl)acetylene (12g)





(1-Cyclohyxenylethynyl)benzene (12h)





1-Heptyn-1-yl-benzene