Electronic Supplementary Material (ESI) for Chemical Communications

Formation of Organic Gel/Liquid Two-Layer Systems Using Diffusion-Controlled Gelation by a Helicene Derivative

Masanori Shigeno and Masahiko Yamaguchi*[†]

Department of Organic Chemistry, Graduate School of Pharmaceutical Sciences, Tohoku University, Aoba, Sendai 980-8578, Japan

[†]*WPI Advanced Institute for Materials Research, Tohoku University, Aoba, Sendai 980-8577, Japan*

*E-mail: yama@mail.pharm.tohoku.ac.jp

Supporting Information

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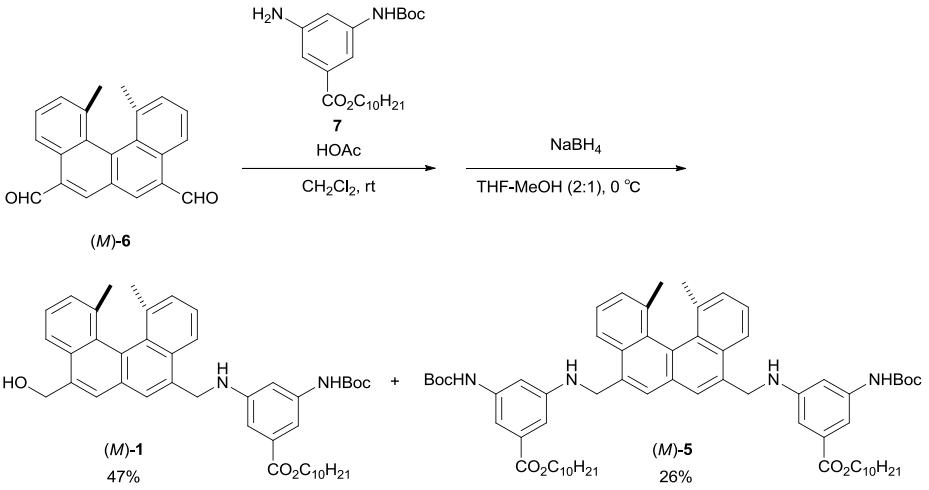
General methods. All reactions were carried out under an argon atmosphere. Column chromatography was carried out on silica gel 60N (40–50 μm). NMR spectra were measured on a Varian-MR spectrometer (^{1}H at 400 MHz and ^{13}C at 100 MHz). ^{1}H NMR spectra were recorded in $CDCl_3$ with tetramethylsilane as the internal standard. ^{1}H NMR taken in benzene-d_6 (δ 128.0) was referenced to the residual solvent. ^{13}C NMR spectra taken in $CDCl_3$ (δ 77.0) were referenced to the residual solvent. Melting points were determined with a Yanagimoto micro melting point apparatus without correction. Elemental analyses were conducted with Yanaco CHN CORDER MT-6. Optical rotations were measured on a JASCO P-1010 digital polarimeter. IR spectra were measured on a JASCO FT/IR-400 spectrophotometer. Low- and high-resolution mass spectra were recorded on a JEOL JMS DX-303, or a JEOL JMS AX-700 spectrometer. FAB mass spectra were recorded on a JEOL JMS DX-303, or a JEOL JMS AX-700 spectrometer by using 3-nitrobenzyl alcohol (NBA) matrix. Ultrasound irradiation was performed on a SMT SC-208.

Scheme S-1. Preparation of (*M*)-1 and (*M*)-2

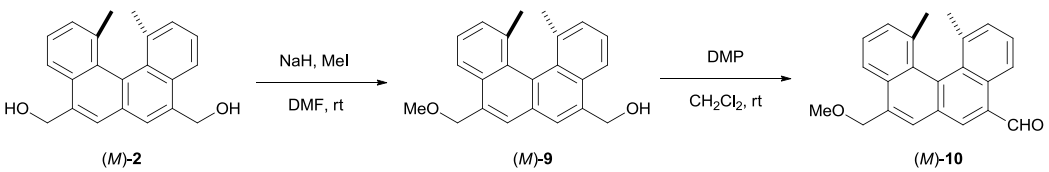


(*M*)-8-{3-(*t*-Butoxycarbonylamino)-5-(decyloxycarbonyl)anilido}-1,12-dimethylbenzo[*c*]phenanthrene-5-methanol, (*M*)-1 and (*M*)-5,8-bis{3-(*t*-Butoxycarbonylamino)-5-(decyloxycarbonyl)anilinomethyl}-1,12-dimethylbenzo[*c*]phenanthrene, (*M*)-5.

To a solution of diadehyde **(*M*)-6**[1] (462 mg, 1.48 mmol) and HOAc (102 μl, 1.78 mmol) in CH_2Cl_2 (20 mL) was added a solution of aniline derivative **7** (581 mg, 1.48 mmol) in CH_2Cl_2 (10 mL). After stirring at rt for 11 h, the mixture was concentrated. The residue was dissolved in THF–MeOH (2:1, 45 mL), to which $NaBH_4$ (1.68 g, 44.4 mmol) was

slowly added at 0 °C. After stirring at the temperature for 1 h, the reaction was quenched with saturated NH_4Cl aqueous solution, and the organic materials were extracted with AcOEt. The extract was washed with water and brine, dried over Na_2SO_4, and concentrated. The residue was purified by column chromatography on silica gel (hexane:AcOEt = 4:1) to afford **(*M*)-1** (482 mg, 0.698 mmol, 47%) and **(*M*)-5** (415 mg, 0.390 mmol, 26%). **(*M*)-1:** Mp 149-150 °C. $[\alpha]_D^{19}$ +12.5 (c 1.0, THF). MS (FAB, NBA) Calcd for $C_{44}H_{54}N_2O_5(M^+)$: 690.4033. Found: 690.4034. IR (KBr) 3396, 2954, 2925, 2854, 1708, 1691, 1232, 1163 cm^{-1}. Anal. ($C_{44}H_{54}N_2O_5$) Calcd for: C, 76.49; H, 7.88; N, 4.05%. Found: C, 76.39; H, 7.97; N, 4.06%. ^1H NMR (400 MHz, $CDCl_3$) δ 0.87 (t, *J* = 7.0 Hz, 3H), 1.19-1.44 (m, 14H), 1.50 (s, 9H), 1.71 (quint, *J* = 7.1 Hz, 2H), 1.85 (br s, 1H), 1.96 (s, 3H), 1.97 (s, 3H), 4.27 (t, *J* = 6.8 Hz, 2H), 4.31 (br s, 1H), 4.87 (d, *J* = 13.6 Hz, 1H), 4.94 (d, *J* = 13.6 Hz, 1H), 5.25 (d, *J* = 13.2 Hz, 1H), 5.33 (d, *J* = 13.2 Hz, 1H), 6.48 (s, 1H), 7.11 (s, 1H), 7.15 (s, 1H), 7.31 (br s, 1H), 7.43 (d, *J* = 7.2 Hz, 2H), 7.60 (t, *J* = 8.4 Hz, 1H), 7.62 (t, *J* = 7.8 Hz, 1H), 7.80 (s, 1H), 7.81 (s, 1H), 8.06 (d, *J* = 8.0 Hz, 1H), 8.13 (d, *J* = 8.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 22.6, 23.4, 23.5, 25.9, 28.3, 28.6, 29.23, 29.26, 29.48, 29.51, 31.8, 46.3, 63.5, 65.1, 80.6, 106.7, 108.2, 108.7, 120.7, 120.8, 124.3, 125.2, 125.4, 125.9, 126.0, 128.20, 128.21, 130.3, 130.6, 131.4, 131.6, 131.8, 131.9, 132.8, 135.4, 136.8, 136.9, 139.5, 148.9, 152.7, 166.8.

(*M*)-5: Mp 127-128 °C. $[\alpha]_D^{21}$ +36.5 (c 0.25, $CHCl_3$). MS (FAB, NBA) Calcd for $C_{66}H_{88}N_4O_8$ (M^+): 1064.6602. Found: 1064.6614. IR (KBr) 3384, 2954, 2925, 2854, 1703, 1234, 1159 cm^{-1}. Anal. ($C_{66}H_{88}N_4O_8$) Calcd for: C, 74.40; H, 8.33; N, 5.26%. Found: C, 74.34; H, 8.27; N, 5.37%. ^1H NMR (400 MHz, $CDCl_3$) δ 0.86 (t, *J* = 6.8 Hz, 6H), 1.19-1.44 (m, 28H), 1.49 (s, 18H), 1.71 (quint, *J* = 7.1 Hz, 4H), 1.97 (s, 6H), 4.25 (t, *J* = 6.8 Hz, 4H), 4.27 (br s, 2H), 4.83 (d, *J* = 13.6 Hz, 2H), 4.92 (d, *J* = 13.2 Hz, 2H), 6.51 (s, 2H), 7.10 (s, 2H), 7.16 (s, 2H), 7.29 (br s, 2H), 7.43 (d, *J* = 7.2 Hz, 2H), 7.59 (t, *J* = 7.6 Hz, 2H), 7.78 (s, 2H), 8.05 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 22.7, 23.5, 26.0, 28.3, 28.7, 29.26, 29.28, 29.51, 29.53, 31.9, 46.5, 65.1, 80.6, 106.7, 108.2, 108.8, 120.9, 125.4, 125.5, 126.1, 128.3, 130.6, 131.4, 131.8, 132.0, 133.0, 137.0, 139.5, 148.9, 152.6, 166.7.



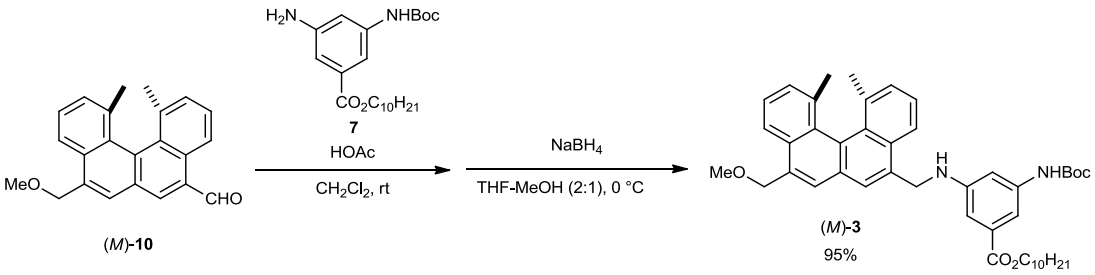
(*M*)-**2** (*M*)-**9** (*M*)-**10**

(*M*)-8-methoxymethyl-1,12-dimethylbenzo[*c*]phenanthrene-5-methanol, (*M*)-9.

To a solution of **(*M*)-2**[1] (253 mg, 0.800 mmol) in DMF (12 mL) was added NaH (60% dispersion in mineral oil, 35.0 mg, 0.875 mmol) at 0°C. After stirring at the temperature, methyl iodide (50.0 μL, 0.800 mmol) was added. The mixture was stirred at rt for 4 h, and the reaction was quenched with saturated NH_4Cl aqueous solution. The organic materials were extracted with AcOEt. The extract was washed with water and brine, dried over Na_2SO_4, and concentrated. The residue was purified by column chromatography on silica gel (hexane:AcOEt = 4:1) to afford **(*M*)-9** (125 mg, 0.38 mmol, 47%): Mp 161-163 °C (CH_2Cl_2-hexane). $[\alpha]_D^{19}$ –1571 (c 0.50, $CHCl_3$). LRMS (EI, 70 eV) *m/z* 330 (M^+, 100%), 299 (M^+–MeO, 37%). HRMS *m/z* Calcd for $C_{23}H_{22}O_2$ (M^+): 330.1620. Found: 330.1613. IR (KBr) 3438, 2960, 2925, 2868, 1119, 1088 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 1.86 (br s, 1H), 1.94 (s, 6H), 3.54 (s, 3H), 5.00 (dd, *J* = 11.8, 0.6 Hz, 1H), 5.11 (dd, *J* = 11.6, 0.8 Hz, 1H), 5.27 (d, *J* = 12.8 Hz, 1H), 5.34 (d, *J* = 12.8 Hz, 1H), 7.41 (dd, *J* = 7.2, 0.8 Hz, 1H), 7.42 (dd, *J* = 7.2, 1.2 Hz, 1H), 7.61 (dd, *J* = 8.2, 7.0 Hz, 1H), 7.62 (dd, *J* = 8.0, 7.2 Hz, 1H), 7.81 (s, 1H), 7.84 (s, 1H), 8.11 (d, *J* = 8.8 Hz, 1H), 8.13 (d, *J* = 8.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.4 (2 peaks), 58.3, 63.6, 73.2, 120.7, 121.1, 124.3, 125.6, 125.7, 125.95, 125.98, 128.17, 128.20, 130.3, 130.8, 131.2, 131.6, 131.7, 132.7, 135.3, 136.7, 136.9.

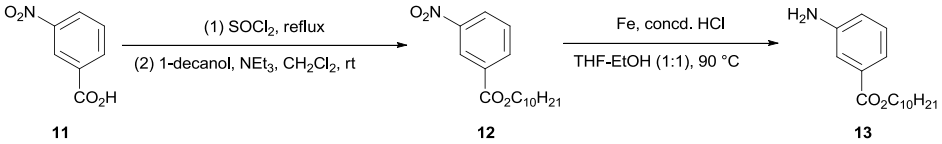
(*M*)-8-methoxymethyl-1,12-dimethylbenzo[*c*]phenanthrene-5-carbaldehyde, (*M*)-10.

To a stirred solution of **(*M*)-9** (105 mg, 0.318 mmol) in CH_2Cl_2 (20 mL) at 0 °C was added Dess-Martin periodinane (148 mg, 0.350 mmol). The mixture was stirred at room temperature for 2 h. The reaction was quenched with saturated $Na_2S_2O_3$ aqueous solution, and the orgnic materials were extracted with AcOEt. The combined organic layers were washed with water and brine, dried over anhydrous Na_2SO_4, and concentrated. The residue was purified by column chromatography on silica gel (hexane:AcOEt = 8:1) to afford **(*M*)-10** (79.1 mg, 0.241 mmol, 76%): Mp 181-183 °C. $[\alpha]_D^{20}$ +149 (c 0.25, $CHCl_3$). LRMS (EI, 70 eV) *m/z* 328 (M^+, 100%), 297 (M^+–MeO, 37%). HRMS *m/z* Calcd for $C_{23}H_{20}O_2$ (M^+): 328.1463. Found: 328.1466. IR (KBr) 2966, 2924, 1678, 1122, 1088 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 1.91 (s, 3H), 1.93 (s, 3H), 3.58 (s, 3H), 5.02 (d, *J* = 12.4 Hz, 1H), 5.11 (d, *J* = 12.2 Hz, 1H), 7.46 (t, *J* = 6.2 Hz, 2H), 7.68 (dd, *J* = 8.0, 7.2 Hz, 1H), 7.69 (dd, *J* = 8.4, 7.2 Hz, 1H), 7.92 (s, 1H), 8.11 (d, *J* = 8.4 Hz, 1H), 8.28 (s, 1H), 9.24 (d, *J* = 8.4 Hz, 1H), 10.47 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.3, 23.4, 58.6, 72.9, 121.2, 122.3, 125.1, 127.5, 127.8, 128.7, 128.8, 129.3, 130.19, 130.21, 130.7, 131.3, 131.6, 132.3, 133.8, 136.4, 137.6, 137.8, 193.0.



(*M*)-5-{3-(*t*-Butoxycarbonylamino)-5-(decyloxycarbonyl)anilinomethyl}-8-methoxy methyl-1,12-dimethylbenzo[*c*]phenanthrene, (*M*)-3.

Mp 68-69 °C. $[\alpha]_D^{19}$ +17.2 (c 0.20, $CHCl_3$). MS (FAB, NBA) Calcd for $C_{44}H_{56}N_2O_5(M^+)$: 704.4189. Found: 704.4200. IR (KBr) 3356, 2952, 2925, 2854, 1714, 1703, 1234, 1159 cm^{-1}. Anal. ($C_{44}H_{56}N_2O_5$) Calcd for: C, 76.67; H, 8.01; N, 3.97%. Found: C, 76.40; H, 7.97; N, 3.98%. ^1H NMR (400 MHz, $CDCl_3$) δ 0.86 (t, J = 6.8 Hz, 3H), 1.19-1.43 (m, 14H), 1.50 (s, 9H), 1.71 (quint, J = 7.1 Hz, 2H), 1.95 (s, 3H), 1.96 (s, 3H), 3.53 (s, 3H), 4.25 (t, J = 6.8 Hz, 2H), 4.30 (br s, 1H), 4.86 (d, J = 13.6 Hz, 1H), 4.93 (d, J = 13.6 Hz, 1H), 4.97 (d, J = 12.0 Hz, 1H), 5.09 (d, J = 12.0 Hz, 1H), 6.51 (s, 1H), 7.11 (t, J = 1.8 Hz, 1H), 7.17 (s, 1H), 7.29 (br s, 1H), 7.42 (d, J = 7.2 Hz, 2H), 7.59 (t, J = 7.6 Hz, 1H), 7.61 (t, J = 7.6 Hz, 1H), 7.78 (s, 1H), 7.81 (s, 1H), 8.05 (d, J = 8.0 Hz, 1H), 8.11 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 22.7, 23.48, 23.49, 26.0, 28.3, 28.7, 29.26, 29.28, 29.51, 29.53, 31.9, 46.5, 58.4, 65.1, 73.2, 80.6, 106.6, 108.2, 108.7, 120.7, 121.1, 125.4, 125.6, 125.7, 125.9, 126.1, 128.2, 128.3, 130.6, 130.8, 131.3, 131.6, 131.9, 132.0, 132.8 (2 peaks), 136.7, 137.0, 139.5, 148.9, 152.6, 166.7.

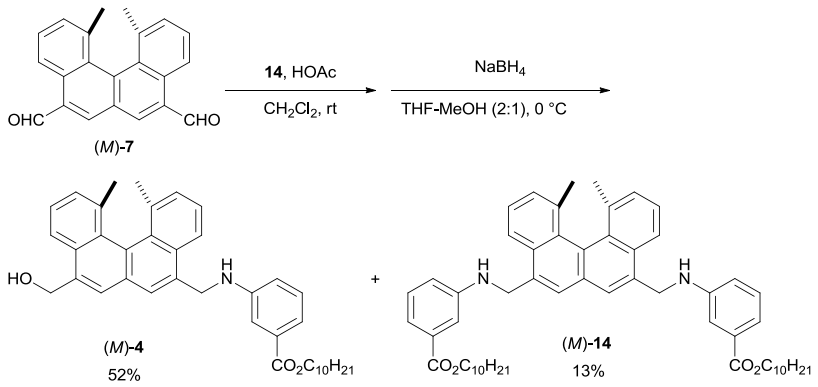


Decyl 3-nitrobenzoate, 12. A mixture of 3-nitrobenzoic acid **11** (5.01 g, 30 mmol) and thionyl chloride (60 mL) was heated at reflux for 3 h. Excess thionyl chloride was removed *in vacuo*, and the residue was azeotropically dried twice by adding and evaporating with dichloromethane (15 mL). To the residue, dichloromethane (60 mL), triethylamine (7.0 mL), and 1-decanol (6.3 mL, 33 mmol) were added successively, and the mixture was stirred at room temperature for 11 h. [1, 2] The reaction was quenched by adding 2 M hydrochloric acid, and the organic materials were extracted with CH_2Cl_2 twice. The combined organic layers were washed with water and brine, and dried over

$MgSO_4$. The solvents were evaporated, and the residue was purificated by column chromatography on silica gel (hexane:AcOEt = 8:1) to give **12** (8.48 g, 27.6 mmol, 92%): Mp 31-32 °C. LRMS (EI, 70 eV) *m/z* 308 (M^++H, 4%), 169 (M^+–decyl+3H, 48%), 168 (M^+–decyl+2H, 47%), 151 (M^+–decyloxy+H, 60%), 150 (M^+–decyloxy, 64%). HRMS *m/z* Calcd for $C_{17}H_{26}NO_4$(M^+ + H): 308.1862. Found: 308.1872. IR (KBr) 2956, 2927, 2856, 1535, 1351, 1294, 1263 cm^{-1}. Anal. ($C_{17}H_{25}NO_4$) Calcd for: C, 66.43; H, 8.20; N, 4.56%. Found: C, 66.51; H, 8.20; N, 4.55%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 7.0 Hz, 3H), 1.20-1.50 (m, 14H), 1.80 (quint, *J* = 7.1 Hz, 2H), 4.38 (t, *J* = 6.6 Hz, 2H), 7.66 (t, *J* = 8.0 Hz, 1H), 8.38 (dt, *J* = 7.6, 1.3 Hz, 1H), 8.42 (ddd, *J* = 8.0, 2.4, 1.2 Hz, 1H), 8.86 (t, *J* = 2.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ14.1, 22.7, 26.0, 28.6, 29.2, 29.3, 29.49, 29.51, 31.9, 66.1, 124.5, 127.3, 129.6, 132.3, 135.2, 148.3, 164.5.

Decyl 3-aminobenzoate, 13.

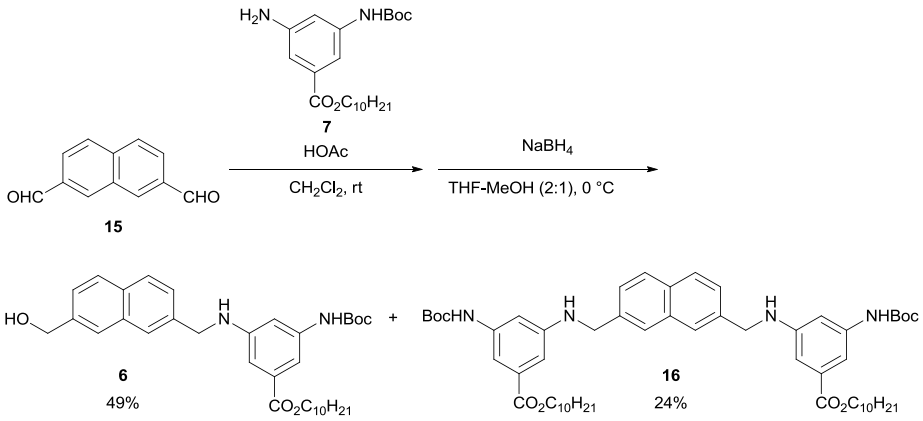
To a suspention of **12** (5.21 g, 16.9 mmol) and iron powder (25.2 g, 451 mmol) in ethanol (70 mL) and THF (70 mL) was added concentrated hydrochloric acid (8.0 mL) dropwise at room temperature. The mixture was heated at 90 °C for 18 h.[2, 3] After being cooled to room temperature, insoluble materials were filtered through Celite®, and washed with AcOEt. Saturated $NaHCO_3$ aqueous solution was added to the filtrate, and the organic materials were extracted with ethyl acetate. The organic layer was washed with water and brine, and dried over Na_2SO_4. The solvents were evaporated, and the residue was purificated by silica gel chromatography (hexane:AcOEt = 4:1) to give **13** (3.50 g, 12.6 mmol, 75%): Mp 28-29 °C. LRMS (EI, 70 eV) *m/z* 277 (M^+, 100%), 137 (M^+–decyl+H, 85%). HRMS *m/z* Calcd for $C_{17}H_{27}NO_2$ (M^+): 277.2042. Found: 277.2041. IR (KBr) 3471, 3377, 2954, 2925, 2854, 1709, 1290, 1238 cm^{-1}. Anal. ($C_{17}H_{27}NO_2$) Calcd for: C, 73.61; H, 9.81; N, 5.05%. Found: C, 73.60; H, 9.93; N, 5.05%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 3H), 1.22-1.47 (m, 14H), 1.75 (quint, *J* = 7.1 Hz, 2H), 3.78 (br s, 2H), 4.28 (t, *J* = 6.6 Hz, 2H), 6.85 (dd, *J* = 7.8, 2.2 Hz, 1H), 7.21 (t, *J* = 7.8 Hz, 1H), 7.35 (t, *J* = 1.8 Hz, 1H), 7.43 (d, *J* = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, benzene-d_6) δ14.3, 23.1, 26.3, 29.1, 29.6, 29.7, 29.89, 29.92, 32.3, 65.0, 115.9, 119.0, 119.4, 129.3, 132.2, 147.3, 166.7.



(*M*)-8-{3-(decyloxycarbonyl)anilidomethyl}-1,12-dimethylbenzo[*c*]phenanthrene-5-methanol, (*M*)-4 and (*M*)-5,8-bis{3-(decyloxycarbonyl)anilinomethyl}-1,12-dimethylbenzo[*c*]phenanthrene, (*M*)-14.

(*M*)-4: Mp 55-56 °C. $[\alpha]_D^{20}$ –21.1 (c 0.20, $CHCl_3$). MS (FAB, NBA) Calcd for $C_{39}H_{45}NO_3(M^+)$: 575.3399. Found: 575.3405. IR (KBr) 3406, 2924, 2854, 1716, 1238 cm^{-1}. Anal. ($C_{39}H_{45}NO_3$) Calcd for: C, 81.35; H, 7.88; N, 2.43%. Found: C, 81.26; H, 7.94; N, 2.41%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.87 (t, *J* = 6.8 Hz, 3H), 1.19-1.46 (m, 14H), 1.73 (quint, *J* = 7.0 Hz, 2H), 1.95 (s, 3H), 1.96 (s, 3H), 4.28 (t, *J* = 6.8 Hz, 2H), 4.86 (d, *J* = 14.0 Hz, 1H), 4.91 (d, *J* = 14.0 Hz, 1H), 5.22 (d, *J* = 13.2 Hz, 1H), 5.30 (d, *J* = 12.8 Hz, 1H), 6.86 (ddd, *J* = 8.2, 2.4, 1.2 Hz, 1H), 7.24 (t, *J* = 7.8 Hz, 1H), 7.39-7.47 (m, 4H), 7.60 (t, *J* = 7.6 Hz, 1H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.77 (s, 1H), 7.79 (s, 1H), 8.06 (d, *J* = 8.0 Hz, 1H), 8.11 (d, *J* = 8.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 22.7, 23.49, 23.51, 26.0, 28.7, 29.27, 29.29, 29.52, 29.54, 31.9, 46.4, 63.6, 65.1, 113.5, 117.0, 118.7, 120.7, 120.8, 124.3, 125.2, 125.5, 126.06, 126.08, 128.3 (2 peaks), 129.3, 130.3, 130.6, 131.4, 131.5, 131.7, 131.8, 133.0, 135.5, 136.9, 137.0, 148.2, 167.1.

(*M*)-14: Mp 111-113 °C. $[\alpha]_D^{20}$ +36.9 (c 0.25, $CHCl_3$). MS (FAB, NBA) Calcd for $C_{59}H_{70}N_2O_4(M^+)$: 834.5336. Found: 834.5330. IR (KBr) 3384, 2952, 2924, 2854, 1705 cm^{-1}. Anal. ($C_{59}H_{70}N_2O_4$) Calcd for: C, 80.53; H, 8.45; N, 3.35%. Found: C, 80.24; H, 8.45; N, 3.35%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.86 (t, *J* = 6.8 Hz, 6H), 1.19-1.46 (m, 28H), 1.73 (quint, *J* = 7.0 Hz, 4H), 1.98 (s, 6H), 4.26 (br s, 2H), 4.28 (t, *J* = 6.6 Hz, 4H), 4.86 (d, *J* = 14.0 Hz, 2H), 4.92 (d, *J* = 13.6 Hz, 2H), 6.87 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.20-7.29 (m, 2H), 7.39-7.47 (m, 6H), 7.60 (t, *J* = 7.8 Hz, 2H), 7.77 (s, 2H), 8.07 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 22.7, 23.5, 26.0, 28.7, 29.26, 29.28, 29.51, 29.52, 31.9, 46.5, 65.0, 113.5, 117.0, 118.7, 120.7, 125.2, 125.4, 126.1, 128.3, 129.3, 130.6, 131.45, 131.54, 131.8, 133.1, 137.0, 148.1, 167.0.



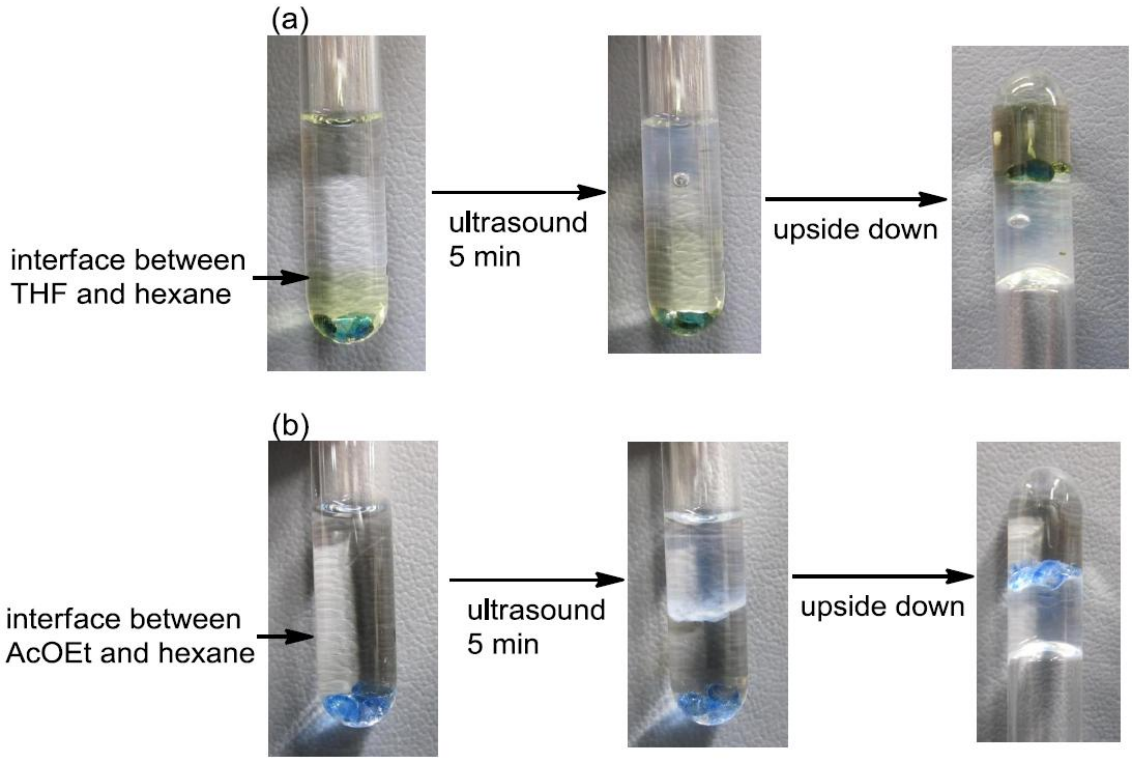
7-{3-(*t*-Butoxycarbonylamino)-5-(decyloxycarbonyl)anilinomethyl}naphthalene-2-methanol, 6 and
2,7-bis{3-(*t*-Butoxycarbonylamino)-5-(decyloxycarbonyl)anilinomethyl}naphthalene, 16.

6: Mp 120-121 °C. MS (FAB, NBA) Calcd for $C_{34}H_{46}N_2O_5(M^+)$: 562.3407. Found: 562.3405. IR (KBr) 3379, 2925, 2854, 1707, 1687, 1243, 1174 cm^{-1}. Anal. ($C_{34}H_{46}N_2O_5$) Calcd for: C, 72.57; H, 8.24; N, 4.98%. Found: C, 72.50; H, 8.46; N, 5.13%. ^1H NMR (400 MHz, $CDCl_3$) δ 0.87 (t, J = 6.8 Hz, 3H), 1.21-1.42 (m, 14H), 1.50 (s, 9H), 1.70 (quint, J = 7.1 Hz, 2H), 1.91 (br s, 1H), 4.22 (t, J = 6.8 Hz, 2H), 4.31 (br s, 1H), 4.49 (s, 2H), 4.83 (s, 2H), 6.51 (s, 1H), 7.02 (s, 1H), 7.11 (s, 1H), 7.23 (br s, 1H), 7.44 (dd, J = 8.0, 1.6 Hz, 1H), 7.45 (dd, J = 8.0, 2.0 Hz, 1H), 7.74 (s, 1H), 7.75 (s, 1H), 7.80 (d, J = 8.4 Hz, 1H), 7.81 (d, J = 8.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 22.7, 26.0, 28.3, 28.7, 29.26, 29.29, 29.52, 29.54, 31.9, 48.2, 65.1, 65.4, 80.6, 106.9, 108.4, 108.7, 125.0, 125.3, 125.8, 126.1, 128.1, 128.2, 131.9, 132.2, 133.4, 136.7, 138.7, 139.4, 148.8, 152.6, 166.7.

16: Mp 157-158 °C. MS (FAB, NBA) Calcd for $C_{56}H_{80}N_4O_8(M^+)$: 936.5976. Found: 936.5958. IR (KBr) 3425, 3332, 2954, 2927, 2854, 1716, 1699, 1232, 1163 cm^{-1}. Anal. ($C_{56}H_{80}N_4O_8$) Calcd for: C, 71.76; H, 8.60; N, 5.98%. Found: C, 71.76; H, 8.60; N, 5.98%. ^1H NMR (400 MHz, $CDCl_3$) δ 0.87 (t, J = 6.8 Hz, 6H), 1.20-1.43 (m, 28H), 1.50 (s, 18H), 1.71 (quint, J = 7.1 Hz, 4H), 4.24 (t, J = 6.6 Hz, 4H), 4.28 (br s, 2H), 4.51 (s, 4H), 6.44 (s, 2H), 7.03 (t, J = 1.8 Hz, 2H), 7.11 (s, 2H), 7.23 (br s, 2H), 7.45 (dd, J = 8.4, 1.6 Hz, 2H), 7.77 (s, 2H), 7.81 (d, J = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 22.7, 26.0, 28.3, 28.7, 29.26, 29.28, 29.52, 29.54, 31.9, 48.3, 65.1, 80.6, 106.8, 108.4, 108.7, 125.7, 126.0, 128.2, 131.9, 132.1, 133.5, 136.7, 139.5, 148.8, 152.6, 166.7.

Synthesis of organogel/organic liquid two-layer systems (Figure S1)

Typical procedures: In a glass tube (5.5 mm in spout-diameter), hexane (0.45 mL) was slowly added on a THF (0.15 mL) solution of (*M*)-**1** (4.1 mg), which formed two-layer of hexane (1.4 cm height) and THF (0.6 cm height). Then, the mixture was subjected to ultrasonication (28.0 kHz, 0.34 Wcm^{-2}) for 5 min. The upper layer (1.0 cm height) gelated, and the lower layer (1.0 cm height) remained liquid. The liquid nature of the lower layer was determined by mobility of blue silica gel initially added in the THF phase. Gel formation in the upper layer was concluded by non-flow nature of the mixture by turning upside down.



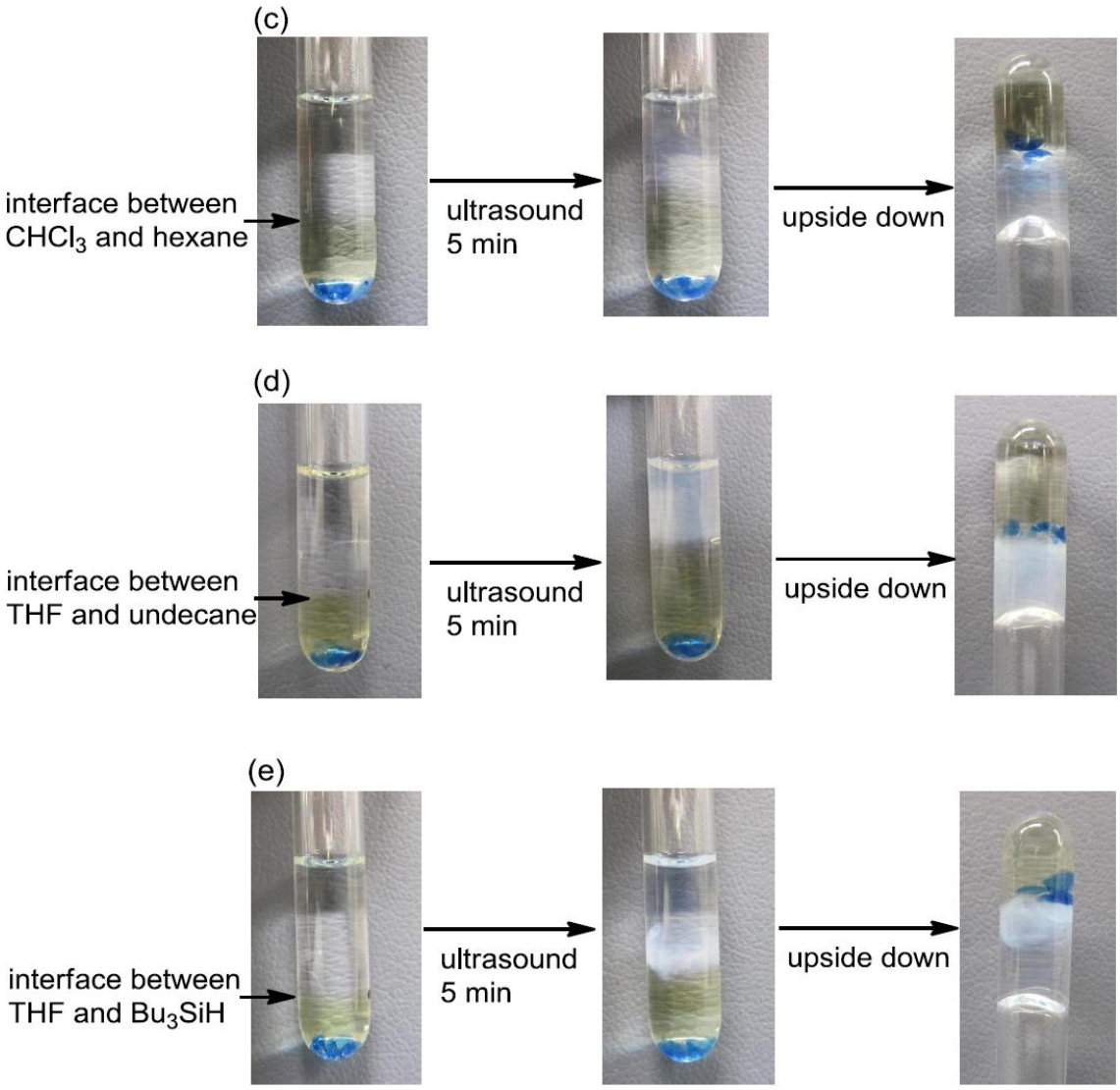


Figure S1. Synthesis of two-layered gel/liquid systems. (a) THF-hexane (1:3, v/v, total 0.60 mL; (*M*)-**1**, 4.1 mg), (b) AcOEt-hexane (1:2, v/v, total 0.60 mL; (*M*)-**1**, 2.1 mg), (c) $CHCl_3$-hexane (1:2, v/v, total 0.60 mL; (*M*)-**1**, 4.1 mg), (d) THF-undecane (1:2, v/v, total 0.60 mL; (*M*)-**1**, 8.3 mg), and (e) THF-Bu_3SiH (1:3, v/v, total 0.60 mL; (*M*)-**1**, 8.3 mg).

Homogeneous Gelation Experiments (Table S1, Figure S2, Table S2 & Figure S3)

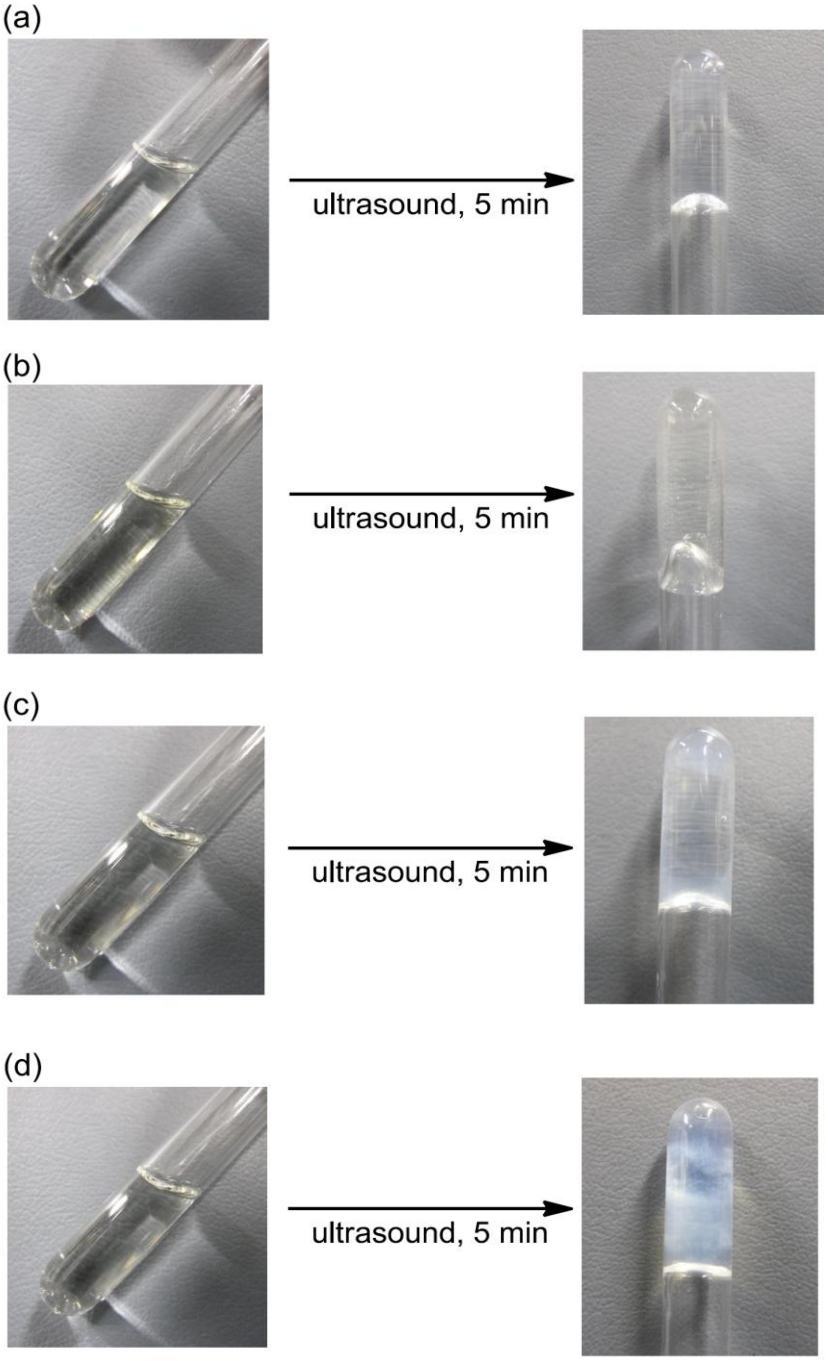
Typical procedures: A THF-hexane solution (1:3, v/v, total 0.60 mL) of (*M*)-**1** (4.1 mg) was placed in a capped glass tube (5.5 mm in spout-diameter). The solution was ultrasonicated (28.0 kHz, 0.34 Wcm^{-2}) for 5 min. A homogeneous substance, which exhibited no gravitational flow by upending the tube, was obtained.

When a homogeneous substance was obtained that exhibited no gravitational flow, it was concluded to be a stable gel (G). When clear solution retained, it was marked soluble (S). If precipitate was formed, it was marked precipitate (P). State of samples before and just after sonication (28.0 kHz, 0.34 Wcm^{-2}, 5 min) was marked as State 1 and State 2, respectively.

Table S1. Homogeneous gelation with (*M*)-1

Solvents		Solvent ratio (v/v, total 0.60 mL)	(*M*)-**1** (mg)	State 1	State 2
THF (0.30 mL)	hexane (0.30 mL)	1:1	4.1	S	S
THF (0.20 mL)	hexane (0.40 mL)	1:2	4.1	S	S
THF (0.15 mL)	hexane (0.45 mL)	1:3	4.1	S	G
THF (0.12 mL)	hexane (0.48 mL)	1:4	4.1	S	G
THF (0.15 mL)	hexane (0.45 mL)	1:3	2.1	S	S
THF (0.60 mL)	—	—	4.1	S	S
hexane (0.60 mL)	—	—	4.1	I	P
$CHCl_3$ (0.30 mL)	hexane (0.30 mL)	1:1	4.1	S	S
$CHCl_3$ (0.20 mL)	hexane (0.40 mL)	1:2	4.1	S	G
THF (0.20 mL)	undecane (0.40 mL)	1:2	4.1	S	S
THF (0.15 mL)	undecane (0.45 mL)	1:3	4.1	S	G
CpOMe (0.30 mL)	undecane (0.30 mL)	1:1	2.1	S	G
CpOMe (0.20 mL)	undecane (0.40 mL)	1:2	2.1	S	G

S: soluble. G: gel. I: insoluble. P: precipitate.



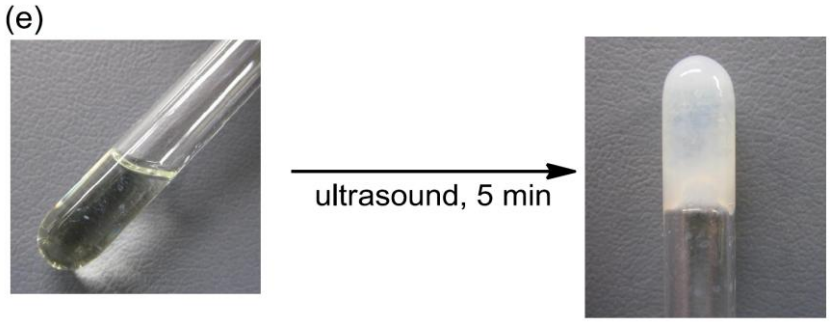


Figure S2. Homogeneous gelation. (a) THF-hexane (1:3, v/v, total 0.60 mL; (*M*)-**1**, 4.1 mg), (b) $CHCl_3$-hexane (1:2, v/v, total 0.60 mL; (*M*)-**1**, 4.1 mg), (c) THF-undecane (1:3, v/v, total 0.60 mL; (*M*)-**1**, 4.1 mg), (d) cyclopentyl methyl ether-undecane (1:2, v/v, total 0.60 mL; (*M*)-**1**, 2.1 mg), and (e) a mixure of (*M*)-**1** (4.1 mg) and $C_{20}H_{42}$ (200 mg, 0.71 mmol) in $CHCl_3$ (0.20 mL).

Table S2. Homogeneous gelation with (±)-1

Solvents		(±)-**1** (mg)	concentration (mM)	State 1	State 2
THF (0.15 mL)	hexane (0.45 mL)	4.1	10	S	S
THF (0.10 mL)	hexane (0.30 mL)	4.1	15	S	G
THF (0.075 mL)	hexane (0.225 mL)	4.1	20	S	G

S: soluble. G: gel.

Gelation of (±)-1 (Figure S3)

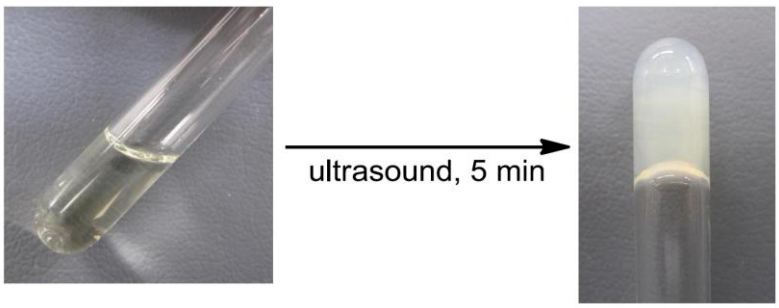


Figure S3. Gelation of **(±)-1** in THF-hexane. Homogeneous solution of THF-hexane (1:3, v/v, total 0.40 mL) containing (±)-**1** (4.1 mg) gave turbid gel by ultrasonication (28.0 kHz, 0.34 Wcm^{-2}) for 5 min. Gel exhibited no gravitational flow by upending the tube.

Atomic Force Microscopy (AFM) (Figure S4 and S5)

AFM images were recorded under ambient conditions using a Digital Instrument Multimode Nanoscope IIIa operating in the tapping mode regime. Micro-fabricated silicon cantilever tips (OMCL-AC160TS-C2) were used. (*M*)-**1** (4.1 mg) and (±)-**1** (4.1 mg) gel in THF-hexane (1:3, v/v, total 0.60 mL; 1:3, total 0.40 mL respectively) was prepared in a glass tube by ultrasonication (28.0 kHz, 0.34 Wcm^{-2}) for 5 min, and placed on freshly cleaved mica followed by the removal of the solvent *in vacuo*.

AFM analysis of (*M*)-1 gel (Figure S4)

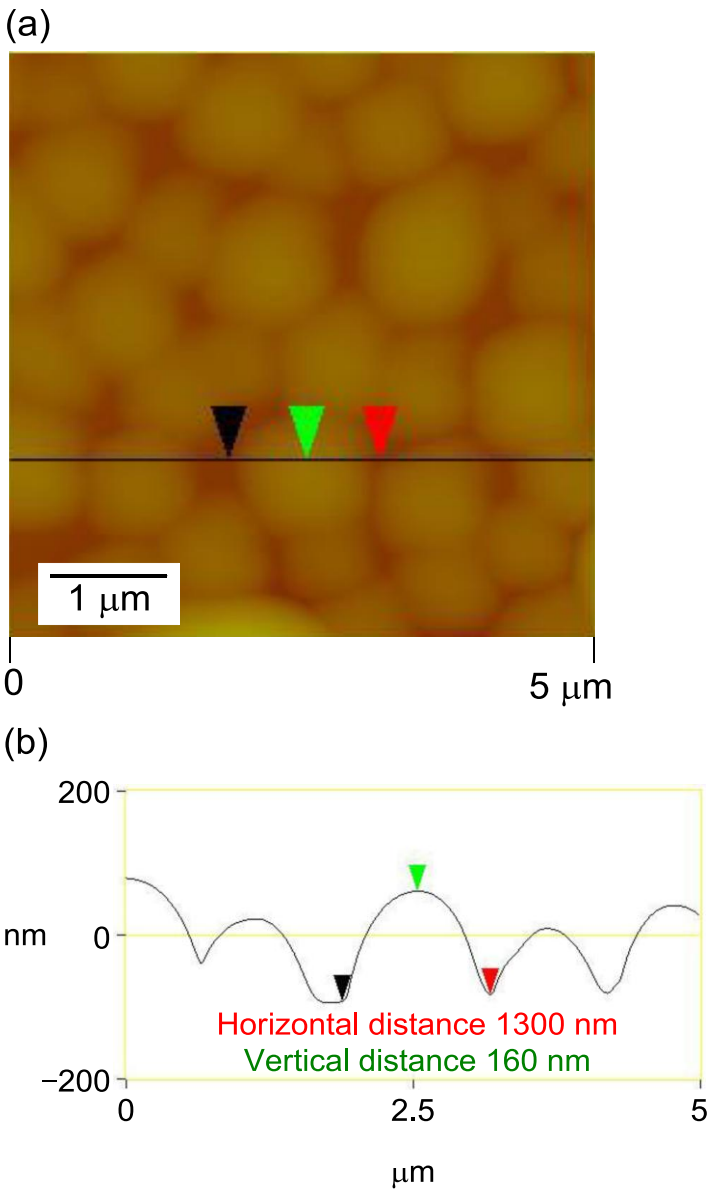


Figure S4. AFM image (height mode) of the dried homogeneous **(*M*)-1** (4.1 mg) gel (xerogel) obtained in THF-hexane (1:3, v/v, total 0.60 mL) by ultrasonication (28.0 kHz,

0.34 Wcm^{-2}, 5 min): (a) height image and (b) cross-sectional analysis. The horizontal distance (diameter) and the vertical distance (height) of one particle were 1300 and 160 nm, respectively.

AFM analysis of (±)-1 gel (Figure S5)

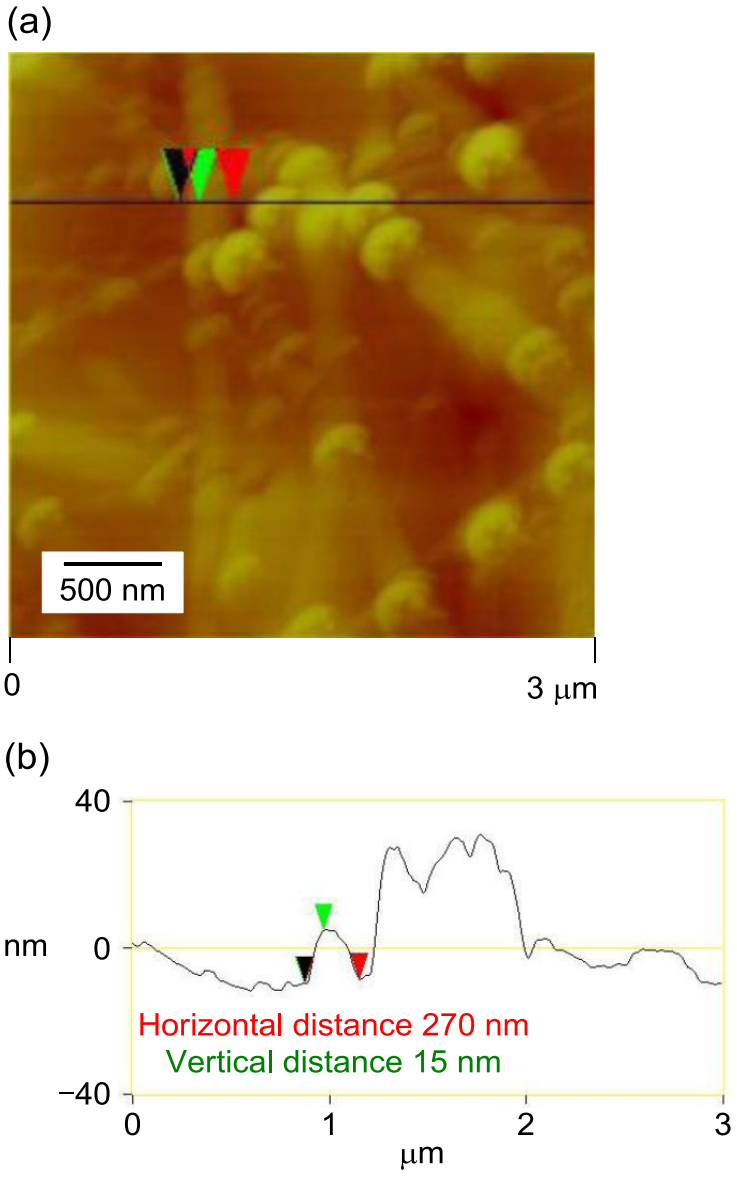


Figure S5. AFM images of the dried homogeneous **(±)-1** (4.1 mg) gel (xerogel) obtained in THF-hexane (1:3, v/v, total 0.30 mL) by ultrasonication (28.0 kHz, 0.34 Wcm^{-2}, 5 min): (a) height image and (b) cross-sectional analysis. The horizontal distance (diameter) and the vertical distance (height) of one fiber were 270 and 15 nm, respectively.

Sol/gel switching (Figure S6)

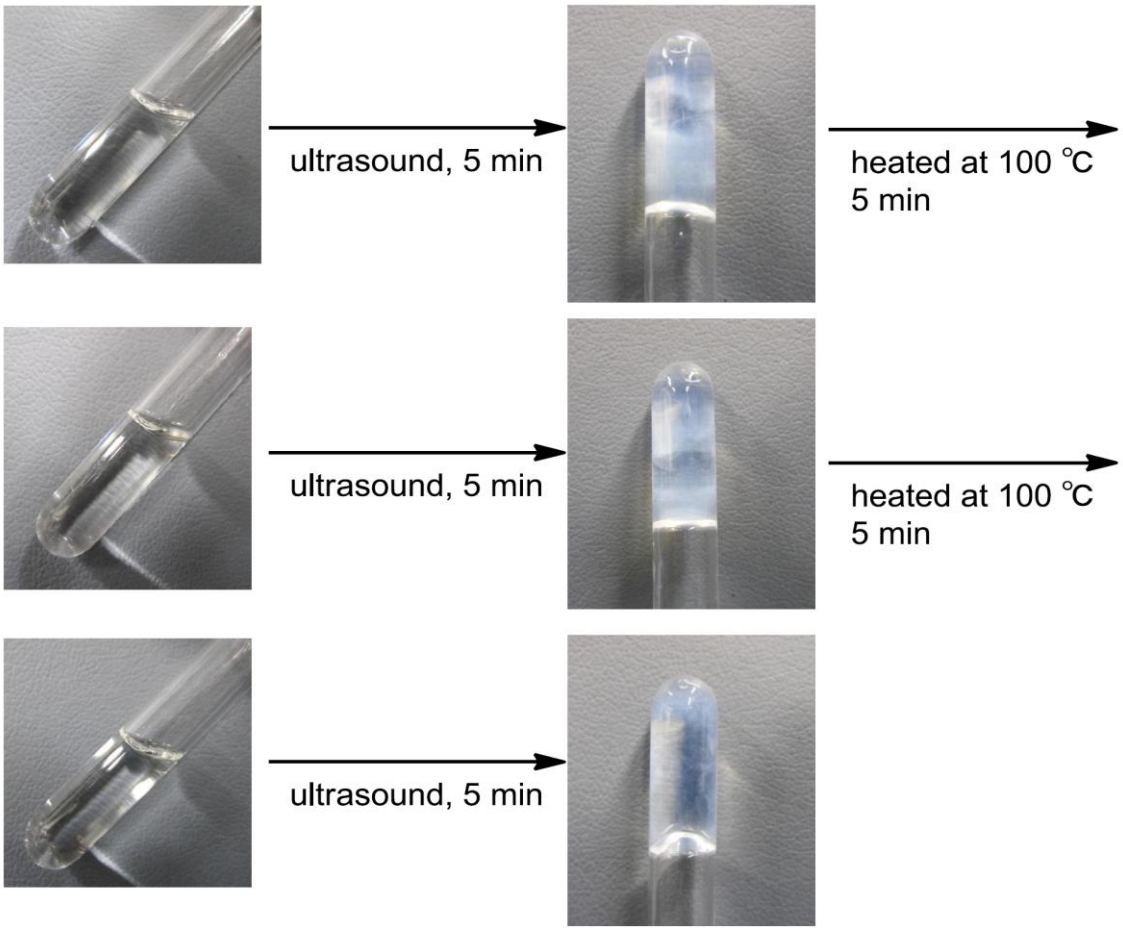


Figure S6. Sol/gel switching of homogeneous gel formed from (*M*)-**1** (2.1 mg) in cyclopentyl methyl ether/undecane (1:1, v/v, total 0.60 mL). Switching was conducted by sonication at room temperature for 5 min and heating at 100 °C for 5 min. This ultrasonic/thermo-reversible switching was repeated three times.

Formation of a metastable gel/liquid state (Figure S7)

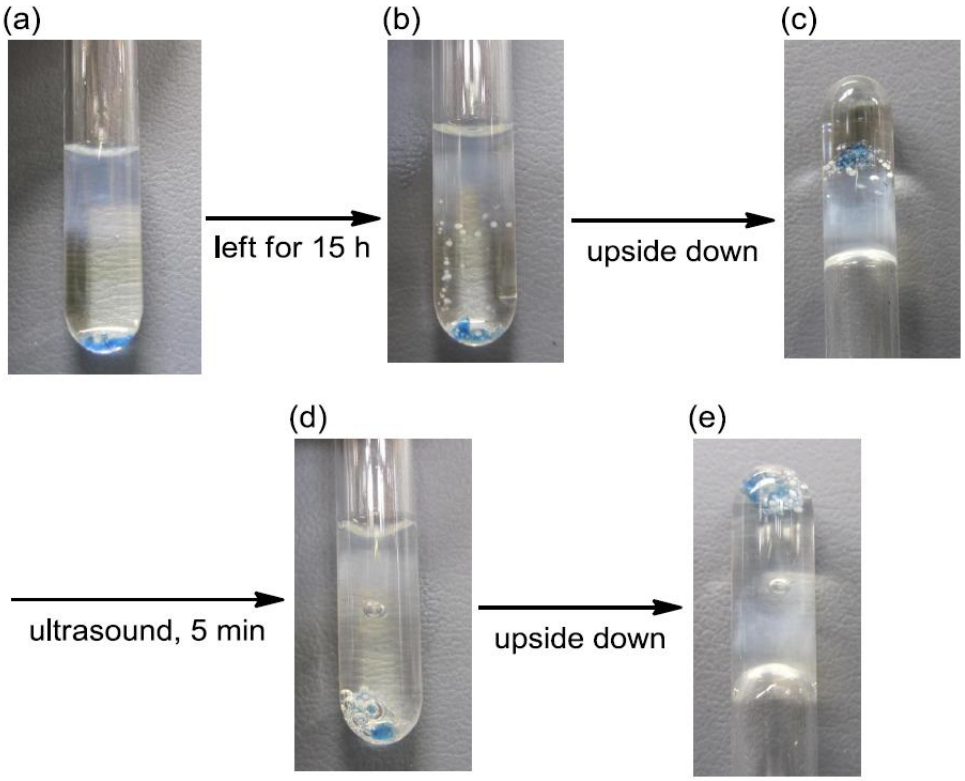


Figure S7. Experiment on the stability of two-layered gel/sol system in THF-hexane (1:3, v/v, total 0.60 mL; (*M*)-**1**, 4.1 mg). (a) Two-layer gel/liquid was formed in THF-hexane (1:3, v/v, total 0.60 mL; (*M*)-**1**, 4.1 mg)**.** (b) After 15 h, mobility of blue silica gel confirmed two-layer gel/liquid structure. (c) Upended. (d) Ultrasonication (28.0 kHz, 0.34 Wcm^{-2}) for 5 min gelated the lower layer. (e) The gel nature of the lower layer was determined by immobility of blue silica gel initially added in the THF phase.

Gelation by short-time ultrasonication (Figure S8)

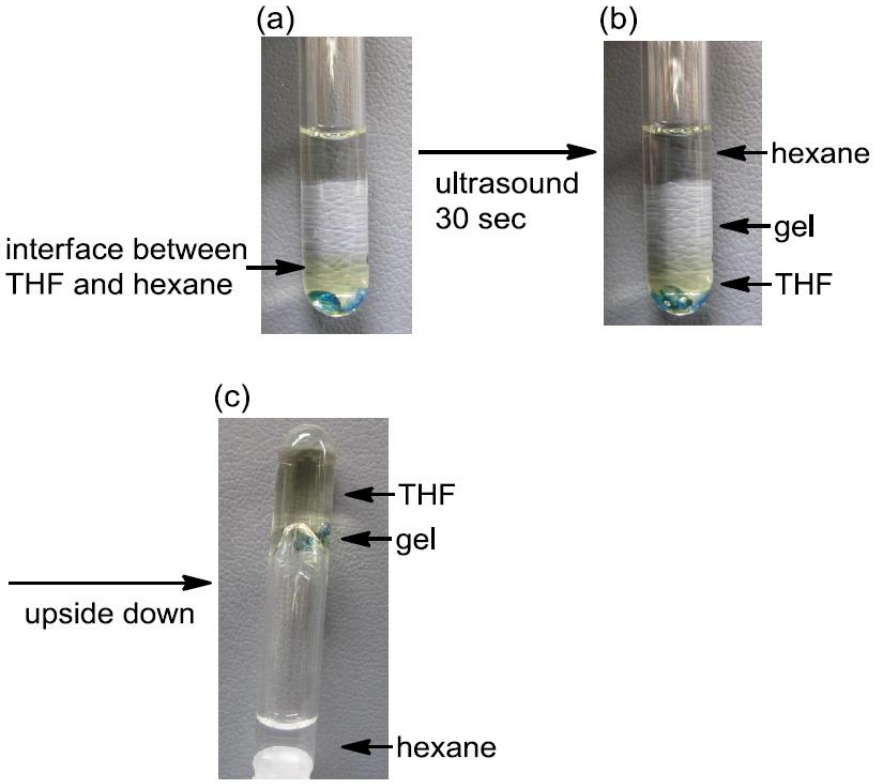


Figure S8. Short-time ultrasonication induced gelation in THF-hexane (1:3, v/v, total 0.60 mL; (*M*)-**1**, 4.1 mg). (a) Two-liquid layer of THF (0.15 mL) containing (*M*)-**1** (4.1 mg) and hexane (0.45 mL). (b) Gelation by ultrasonication (28.0 kHz, 0.34 Wcm^{-2}) for 30 sec. Gel was formed at the central part in a glass-tube. (c) Turning upside down. Liquid layer fell down.

Gelation using a large amount of hexane (Figure S9)

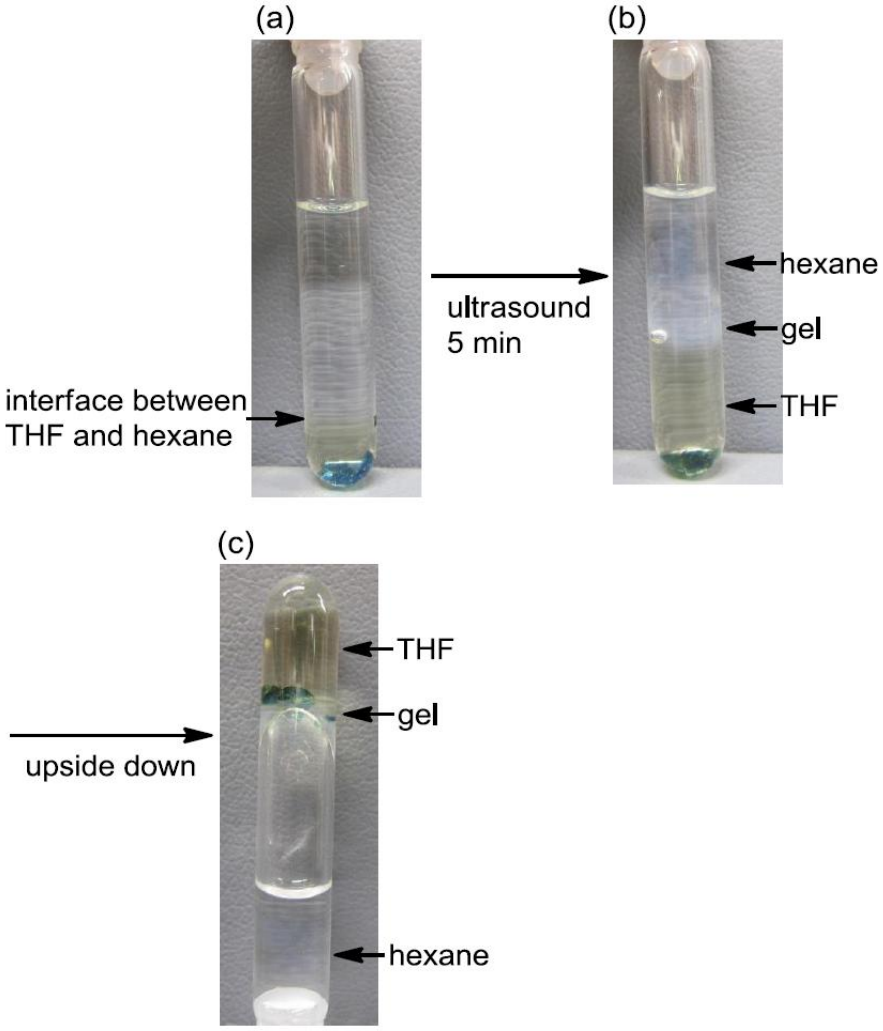


Figure S9. Gelation in THF-hexane (1:5, v/v, total 0.90 mL; (*M*)-**1**, 4.1 mg). Gel was formed at the central position in a glass-tube. (a) Two-liquid layer of THF (0.15 mL) containing (*M*)-**1** (4.1 mg) and hexane (0.75 mL). (b) Gelation by ultrasonication (28.0 kHz, 0.34 Wcm^{-2}) for 30 sec. (c) Turning upside down, and upper layer fell down.

Six-layer system of water/liquid/gel/water/liquid/gel (Figure S10)

In a glass tube (5.5 mm in spout-diameter), three-layer system was formed by a sequence of slow addition of AcOEt solution (0.30 mL; (*M*)-**1**, 3.1 mg) and hexane (0.60 mL) on $Cu(OAc)_2$ (5.0 mg) aqueous solution (0.15 mL). Then, the mixture was subjected to ultrasonication (28.0 kHz, 0.34 Wcm^{-2}) for 5 min. The upper layer gelated, and the lower layer remained liquid. Gel formation in the upper layer was concluded by non-flow nature of the mixture by turning upside down. Then, $Cu(OAc)_2$ aqueous solution (0.15 mL) was added on the three-layer. Addition of AcOEt solution (0.30 mL; (*M*)-**1**, 3.1 mg) and hexane (0.60 mL) followed by ultrasonication (28.0 kHz, 0.34 Wcm^{-2}) for 5 min provided six-layer system of water/liquid/gel/water/liquid/gel. The system was stable enough to allow turning upside down.

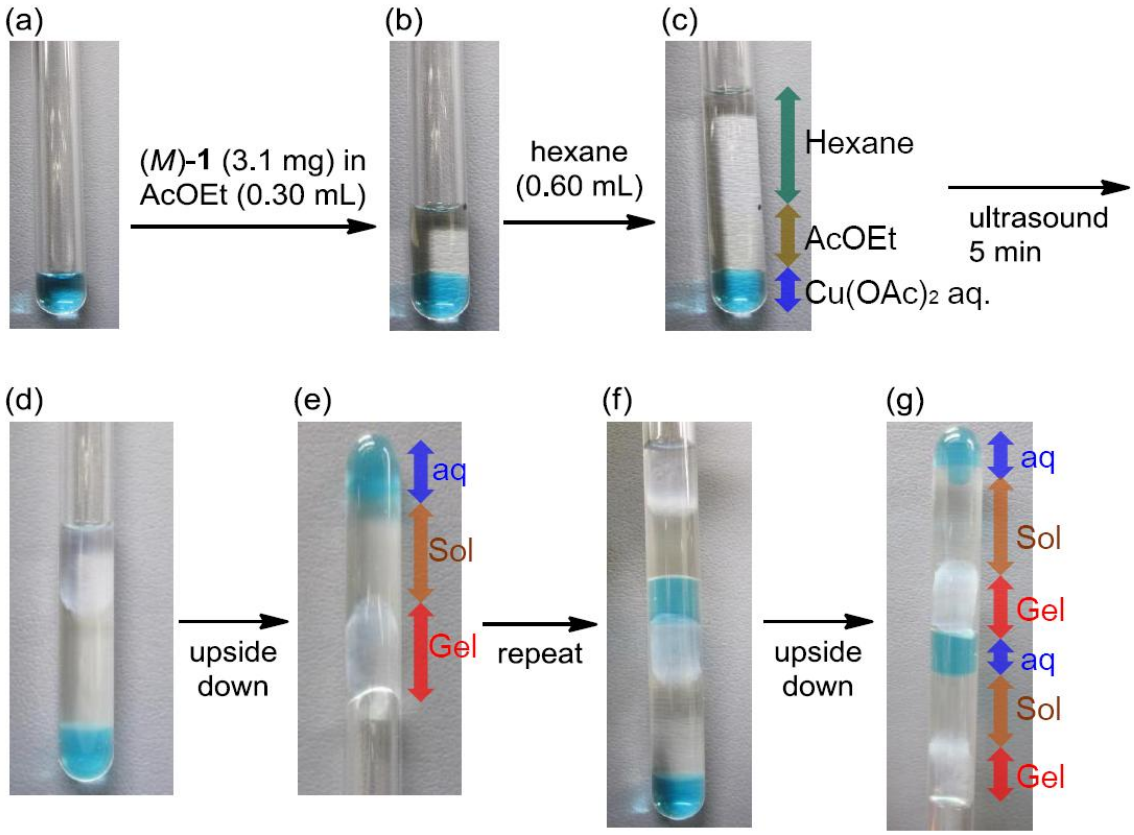
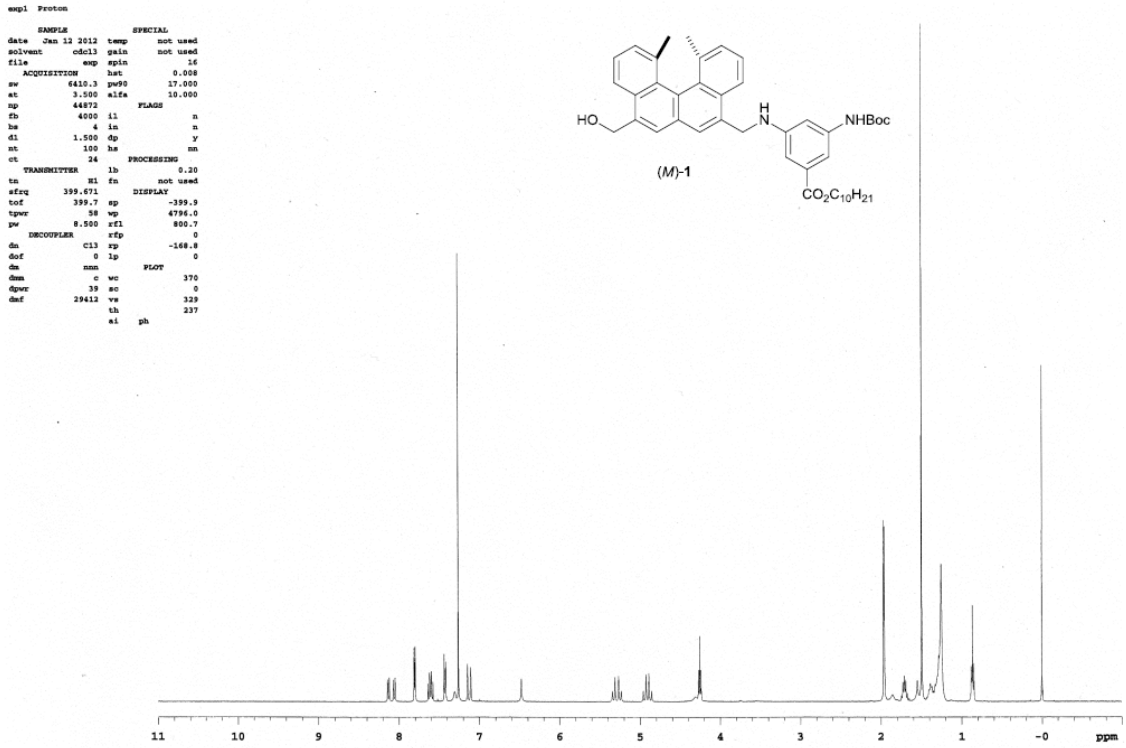


Figure S10. Construction of six-layer system of water/liquid/gel/water/liquid/gel. (a) $Cu(OAc)_2$ aqueous solution (0.15 mL). (b) Added AcOEt solution (0.30 mL, (*M*)-**1** ,3.1 mg). (c) Added hexane (0.60 mL). (d) Afer sonication (28.0 kHz, 0.34 Wcm^{-2}, 5 min), three-layer of water/liquid/gel was obtained. (e) Gel in the upper layer exhibited no gravitational flow by upending the tube. (f) Repeating this procedure afforded a

six-layer system of water/liquid/gel/water/liquid/gel. (g) This system was stable enough to allow turning upside down.

References.

1) K. Nakamura, H. Okubo, M. Yamaguchi, *Org. Lett.* **2001**, *3*, 1097-1099.
2) R. Amemiya, W. Ichinose, M. Yamaguchi, *Bull. Chem. Soc. Jpn.* **2010**, *83*, 809-815.
3) Y. Saiki, H. Sugiura, K. Nakamura, M. Yamaguchi, T. Hoshi, J. Anzai, *J. Am. Chem. Soc.* **2003**, *125*, 9268-9269.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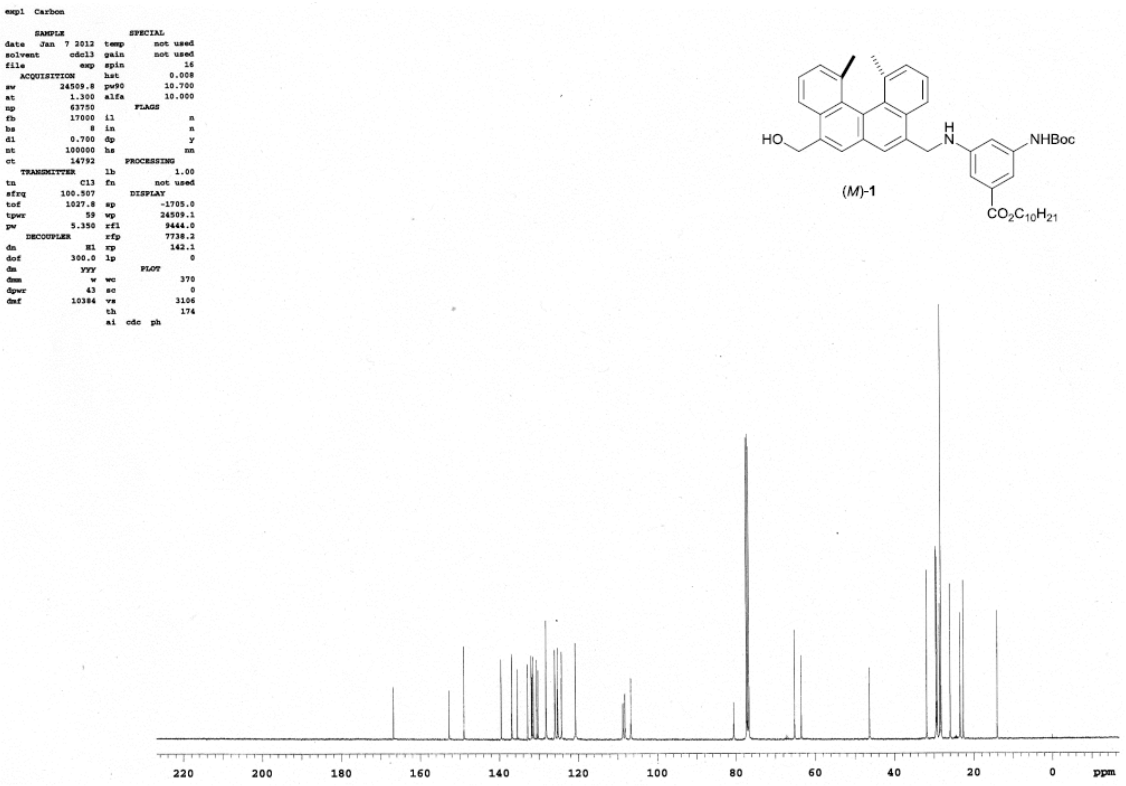
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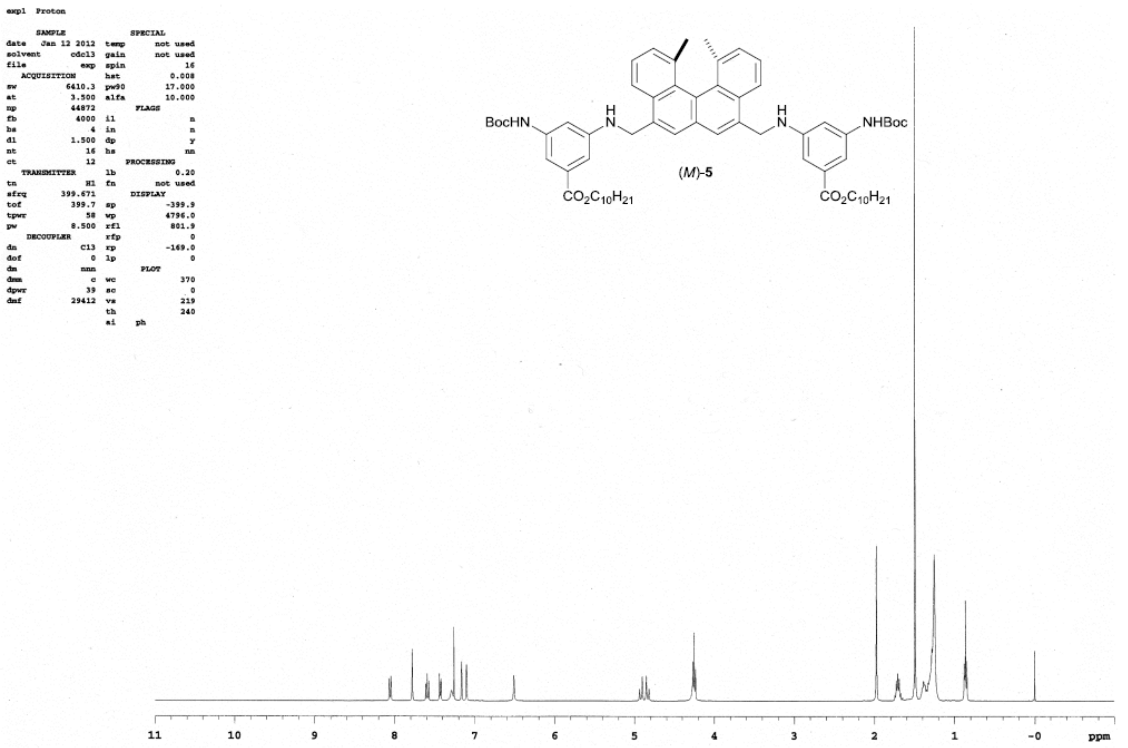
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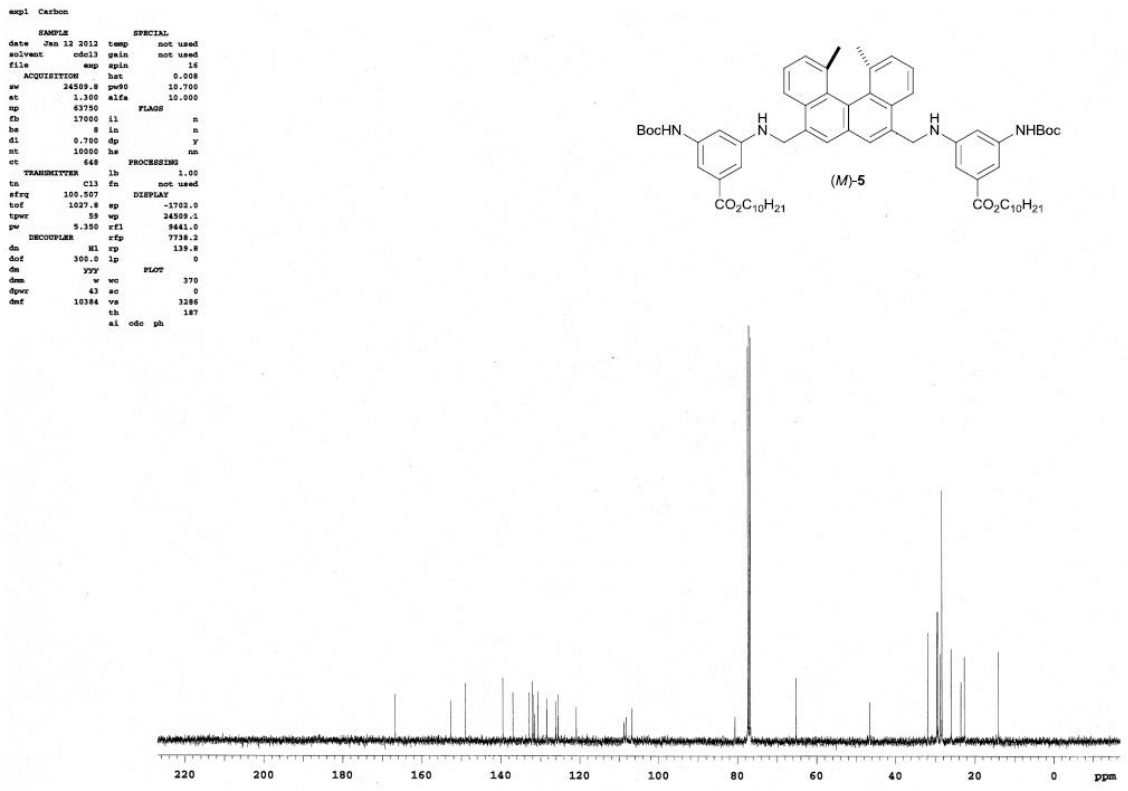
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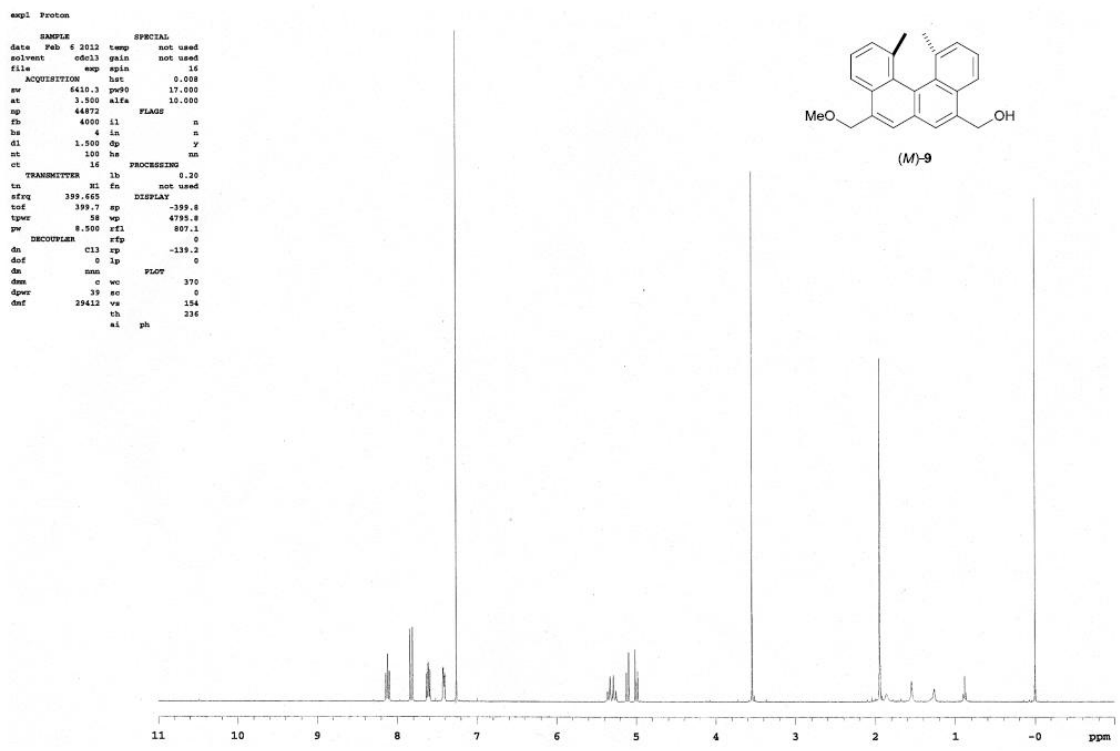
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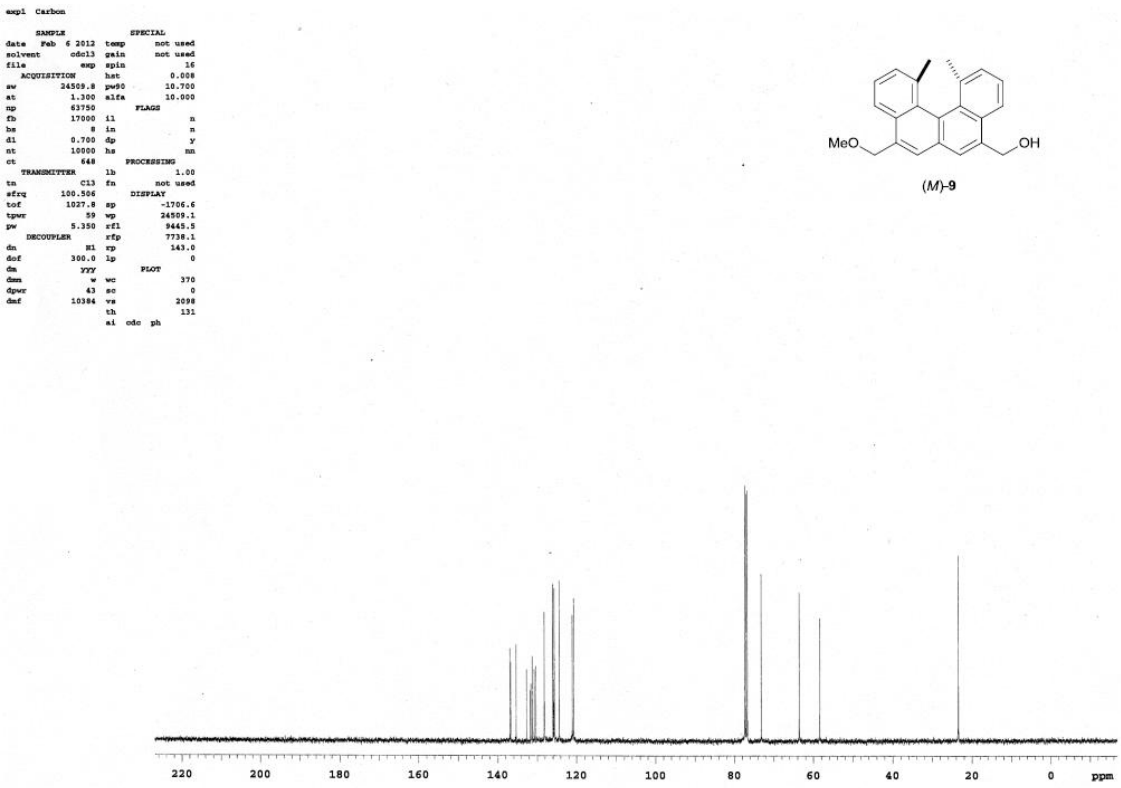
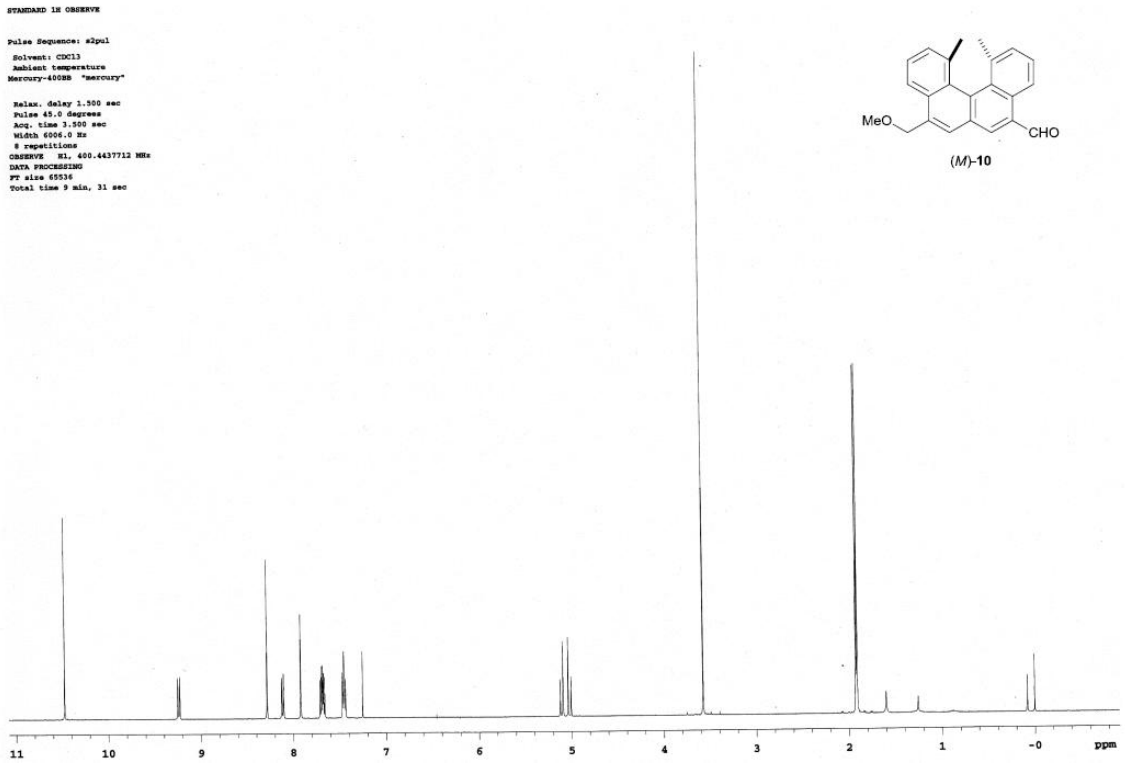
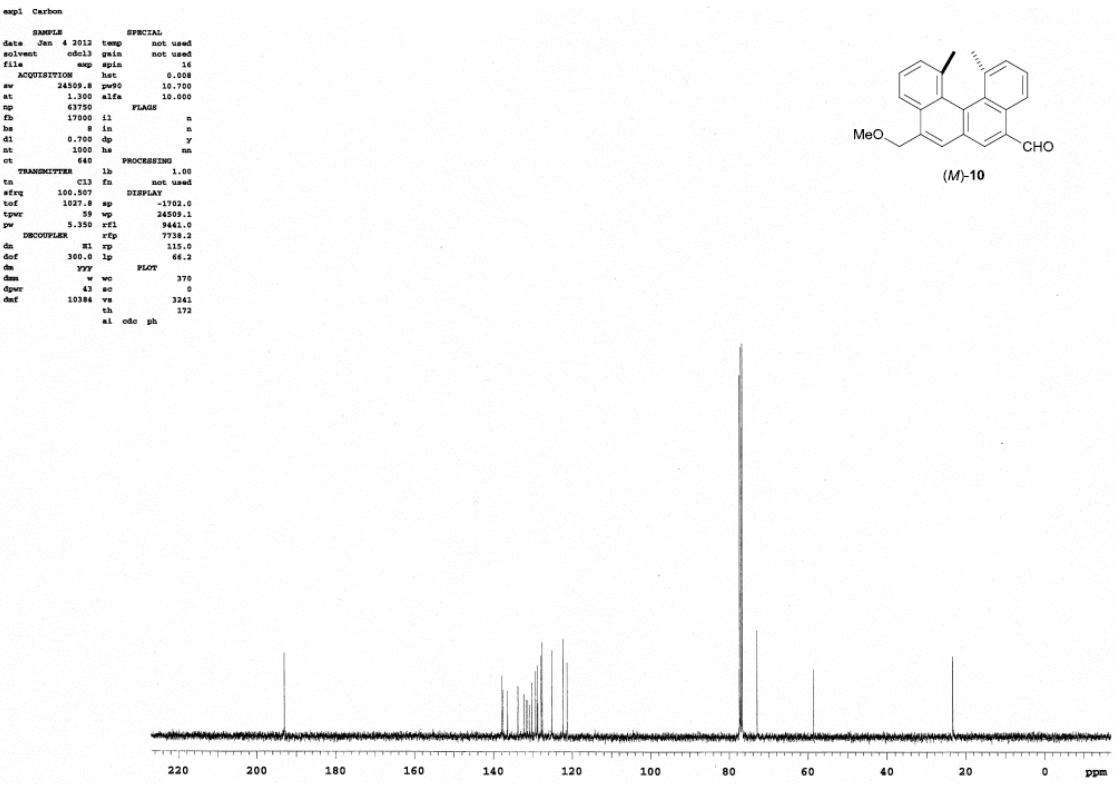
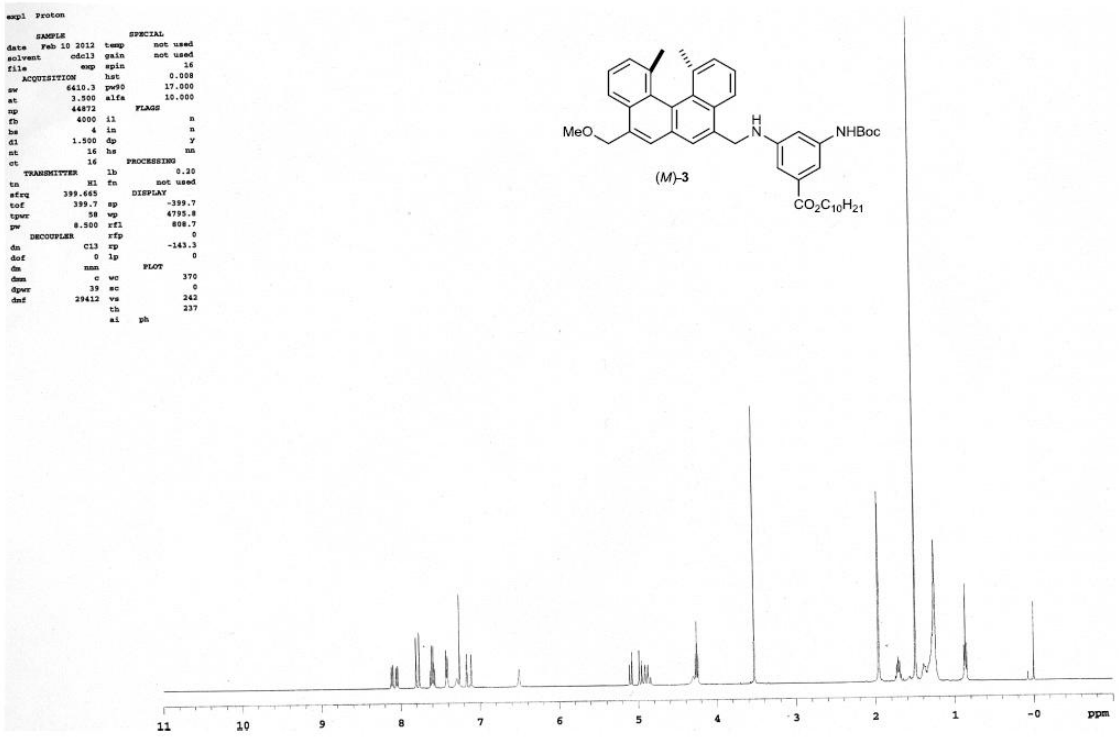
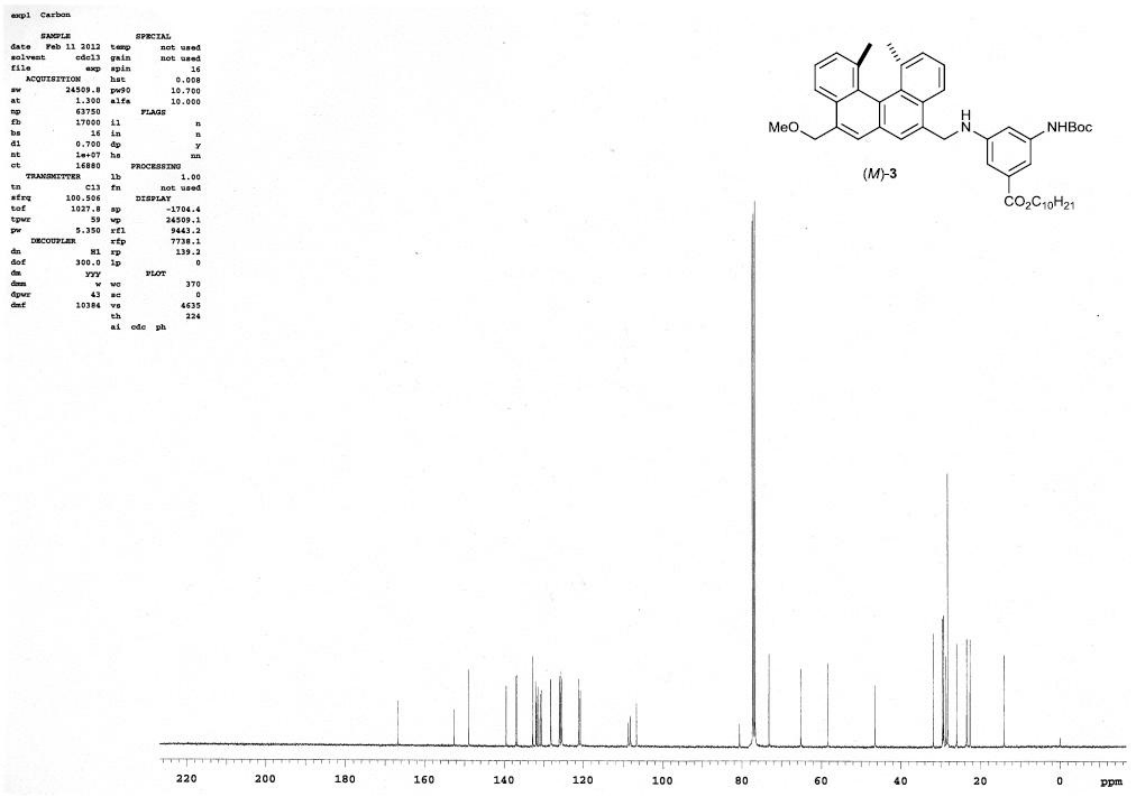
rfl 807.1
rfp 0
rp -139.2
lp 0
PLOT
wc 370
sc 0
vs 154
th 236
ai ph
MeO
OH
(M)-9
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

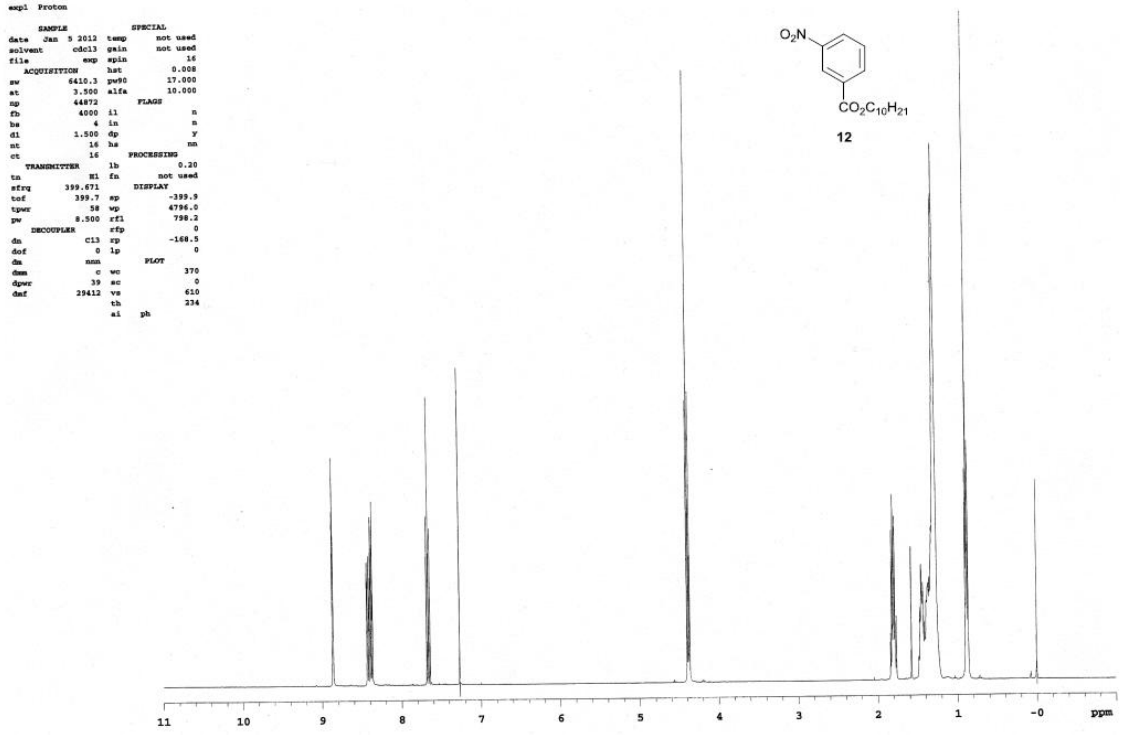

expl Carbon
SAMPLE
date Feb 6 2012
solvent cdcl3
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 8
d1 0.700
nt 10000
ct 648
TRANSMITTER
tn C13
sfrq 100.506
tof 1027.8
tpwr 59
pw 5.350
DECOUPLER
dn H1
dof 300.0
dm YYY
dmm w
dpwr 43
dmf 10384
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 10.700
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1706.6
wp 24509.1
rfl 9445.5
rfp 7738.1
rp 143.0
lp 0
PLOT
wc 370
sc 0
vs 2098
th 131
ai cdc ph
MeO
OH
(M)-9
220
200
180
160
140
120
100
80
60
40
20
0
ppm

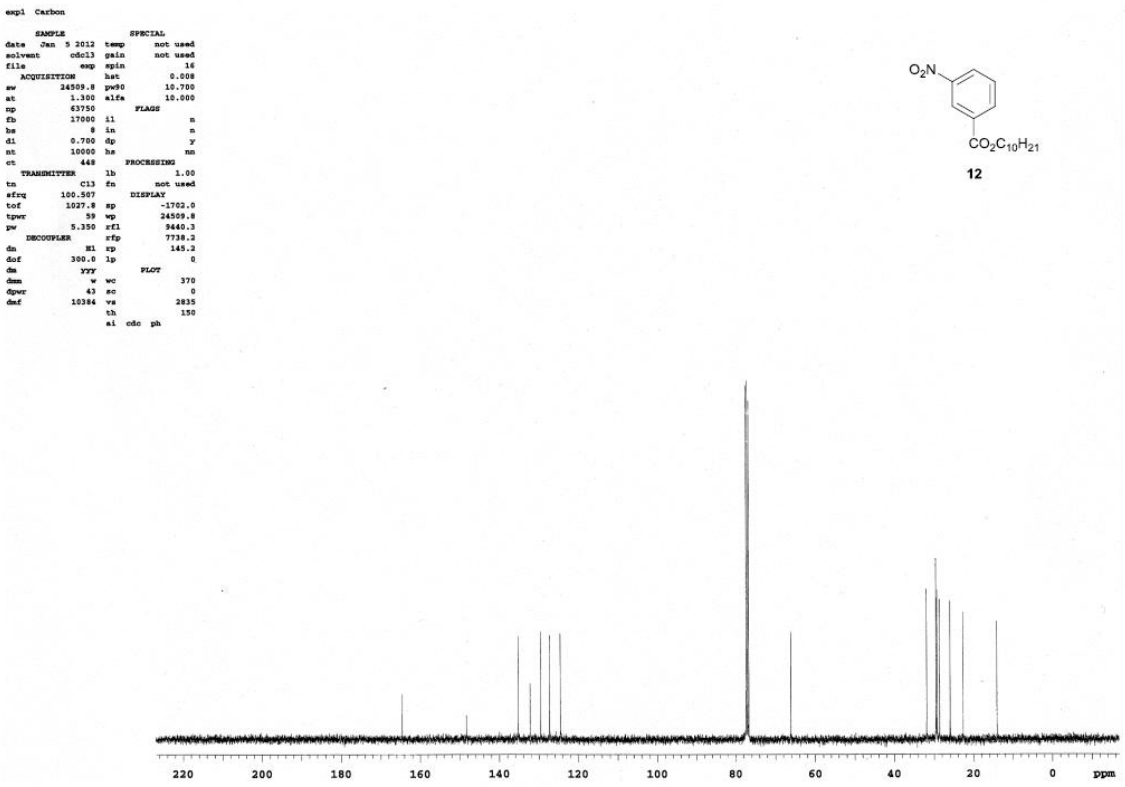

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "mercury"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6006.0 Hz
8 repetitions
OBSERVE H1, 400.4437712 MHz
DATA PROCESSING
FT size 65536
Total time 9 min, 31 sec
MeO
CHO
(M)-10
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

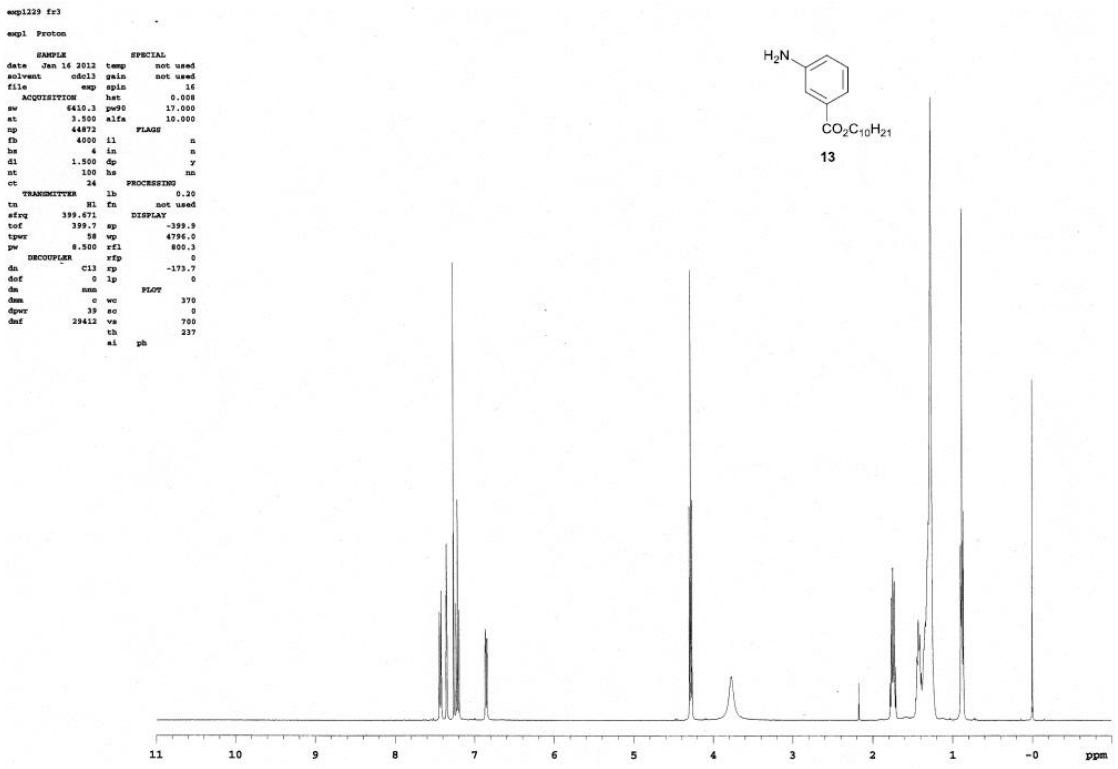

expl Carbon
SAMPLE
date Jan 4 2012
solvent cdcl3
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 8
d1 0.700
nt 1000
ct 640
TRANSMITTER
tn C13
sfrq 100.507
tof 1027.8
tpwr 59
pw 5.350
DECOUPLER
dn H1
dof 300.0
dm yyy
dmm w
dpwr 43
dmf 10384
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 10.700
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1703.0
wp 24509.1
rfl 9441.0
rfp 7738.2
rp 115.0
lp 66.2
PLOT
wc 370
sc 0
vs 3241
th 172
ai cdc ph
MeO
CHO
(M)-10
220
200
180
160
140
120
100
80
60
40
20
0
ppm

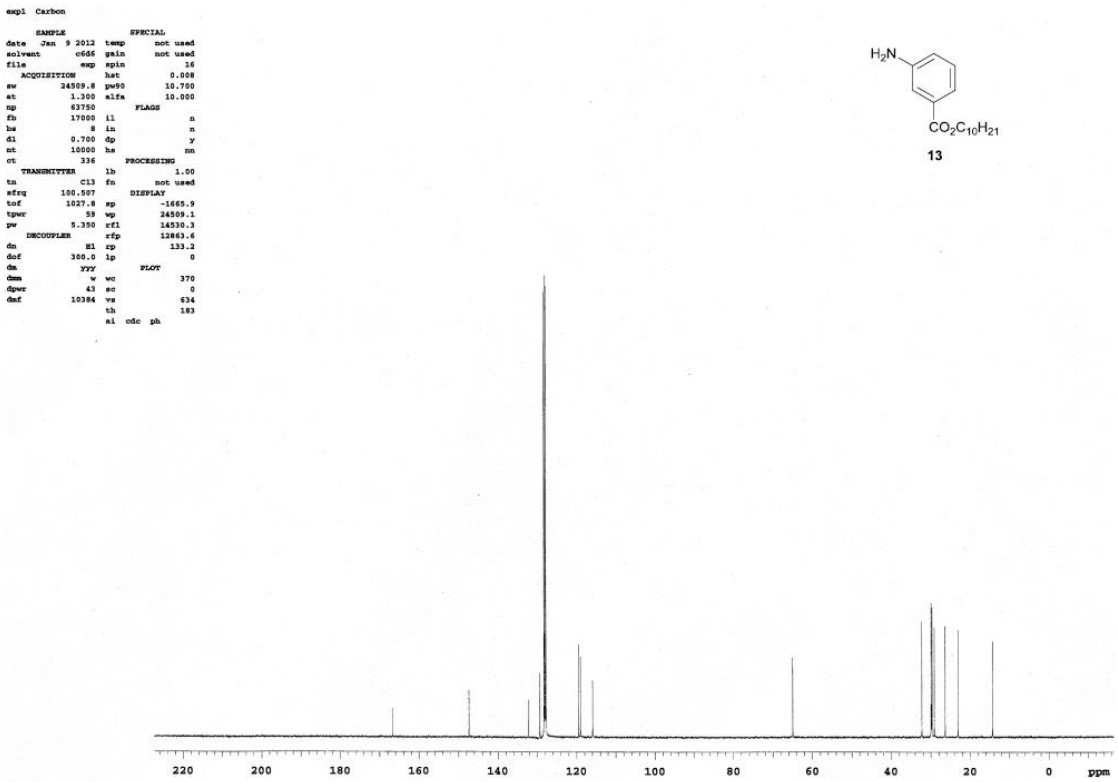

expl Proton
SAMPLE
date Feb 10 2012
solvent cdcl3
file exp
ACQUISITION
sw 6410.3
at 2.500
np 44872
fb 4000
bs 4
d1 1.500
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 399.665
tof 399.7
tpwr 58
pw 8.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 39
dmf 29412
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 17.000
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.20
fn not used
DISPLAY
sp -399.7
wp 4795.8
rfl 808.7
rfp 0
rp -143.3
lp 0
PLOT
wc 370
sc 0
vs 242
th 237
ai ph
MeO
NHBoc
(M)-3
CO2C10H21
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

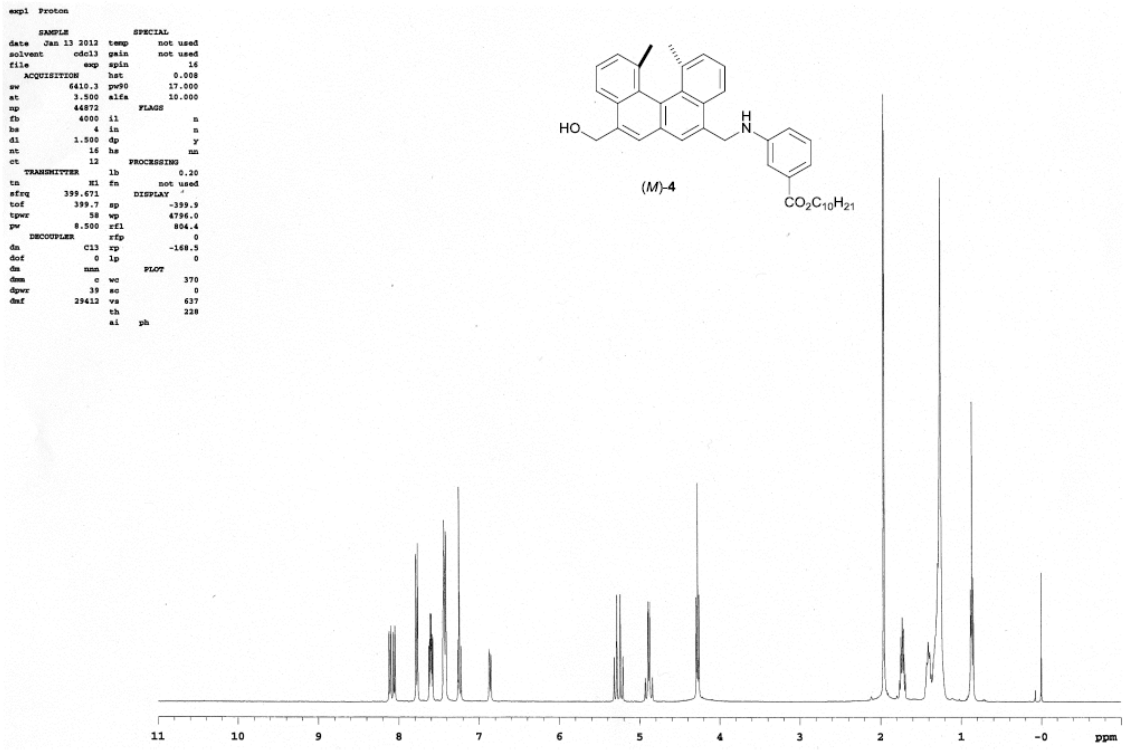

expl Carbon
SAMPLE
date Feb 11 2012
solvent cdcl3
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 16
d1 0.700
nt 1e+07
ct 16880
TRANSMITTER
tn C13
sfrq 100.506
tof 1027.8
tpwr 59
pw 5.350
DECOUPLER
dn H1
dof 300.0
dm yyy
dmm w
dpwr 43
dmf 10384
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 10.700
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1704.4
wp 24509.1
rfl 9443.2
rfp 7738.1
rp 139.2
lp 0
PLOT
wc 370
sc 0
vs 4635
th 224
ai cdc ph
MeO
NHBoc
(M)-3
CO2C10H21
220
200
180
160
140
120
100
80
60
40
20
0
ppm

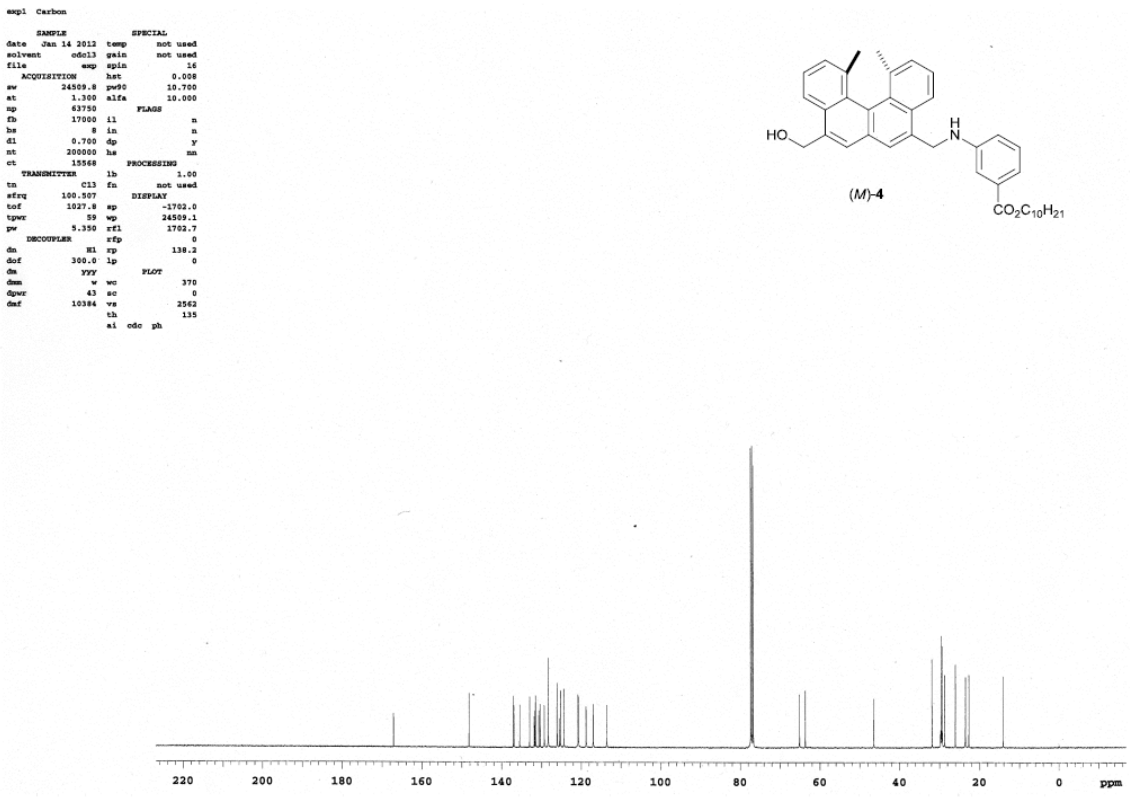

expl Proton
SAMPLE
date Jan 5 2012
solvent cdcl3
file exp
ACQUISITION
sw 6410.3
at 3.500
np 44872
fb 4000
bs 4
d1 1.500
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 399.671
tof 399.7
tpwr 58
pw 8.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 39
dmf 29412
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 17.000
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.20
fn not used
DISPLAY
sp -399.9
wp 4796.0
rfl 798.2
rfp 0
rp -168.5
lp 0
PLOT
wc 370
sc 0
vs 610
th 234
ai ph
O2N
CO2C10H21
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

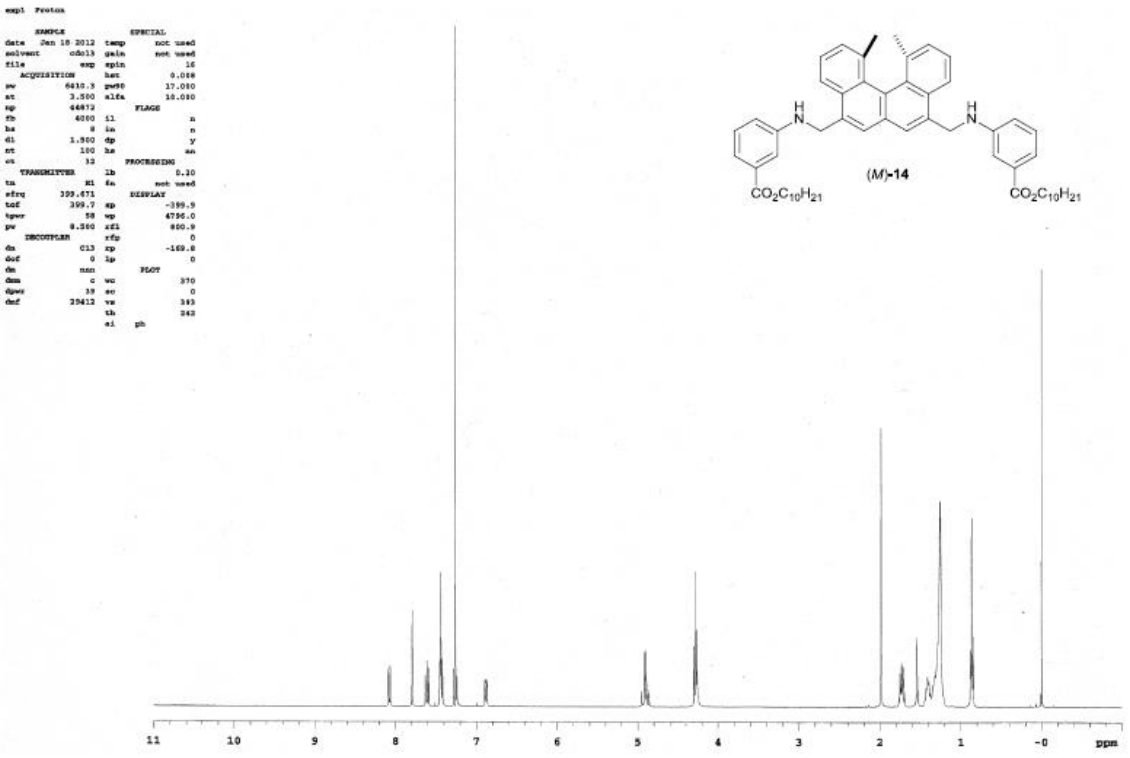

expl Carbon
SAMPLE
date Jan 5 2012
solvent cdcl3
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 8
d1 0.700
nt 10000
ct 448
TRANSMITTER
tn C13
sfrq 100.507
tof 1027.8
tpwr 59
pw 5.350
DECOUPLER
dn H1
dof 300.0
dm yyy
dmm w
dpwr 43
dmf 10384
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 10.700
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1702.0
wp 24509.8
rfl 9440.3
rfp 7738.2
rp 145.2
lp 0
PLOT
wc 370
sc 0
vs 2835
th 150
ai cdc ph
O2N
CO2C10H21
12
220
200
180
160
140
120
100
80
60
40
20
0
ppm

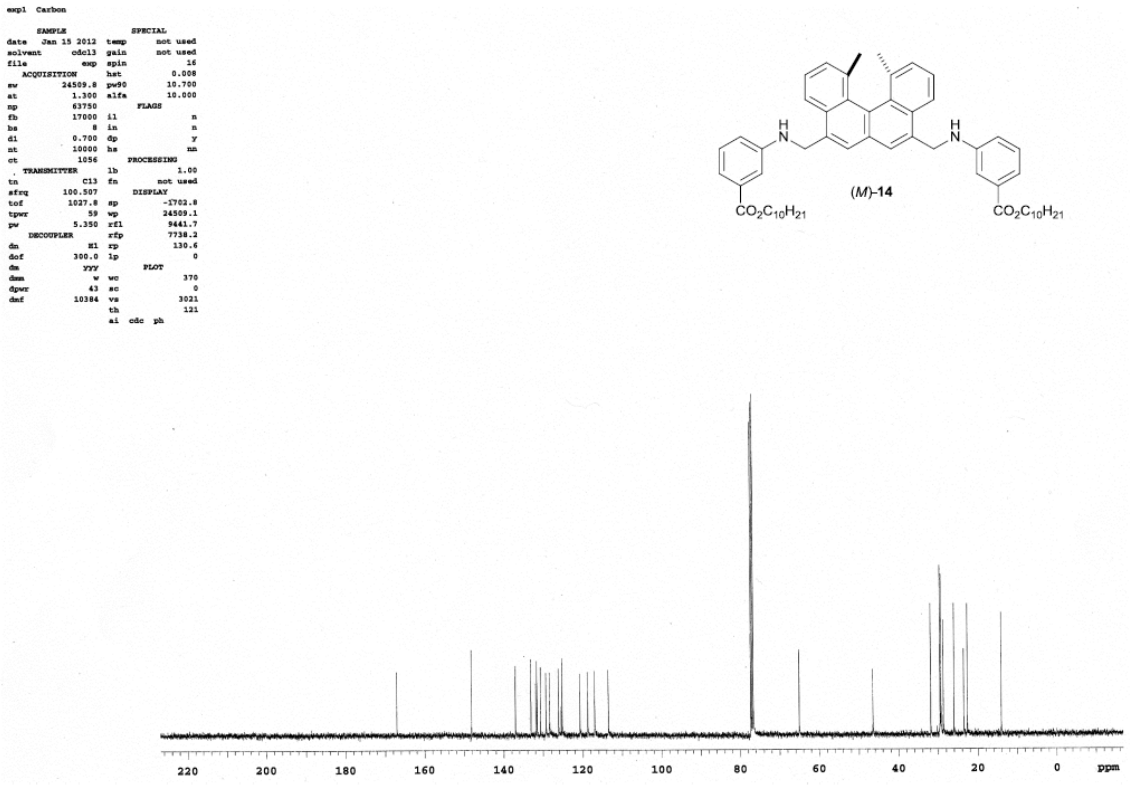

exp1229 fr3
exp1 Proton
SAMPLE
date Jan 16 2012
solvent cdcl3
file exp
ACQUISITION
sw 6410.3
at 3.500
np 44872
fb 4000
bs 4
d1 1.500
nt 100
ct 24
TRANSMITTER
tn H1
sfrq 399.671
tof 399.7
tpwr 58
pw 8.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 39
dmf 29412
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 17.000
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.20
fn not used
DISPLAY
sp -399.9
wp 4796.0
rfl 800.3
rfp 0
rp -173.7
lp 0
PLOT
wc 370
sc 0
vs 700
th 237
ai ph
H2N
CO2C10H21
13
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

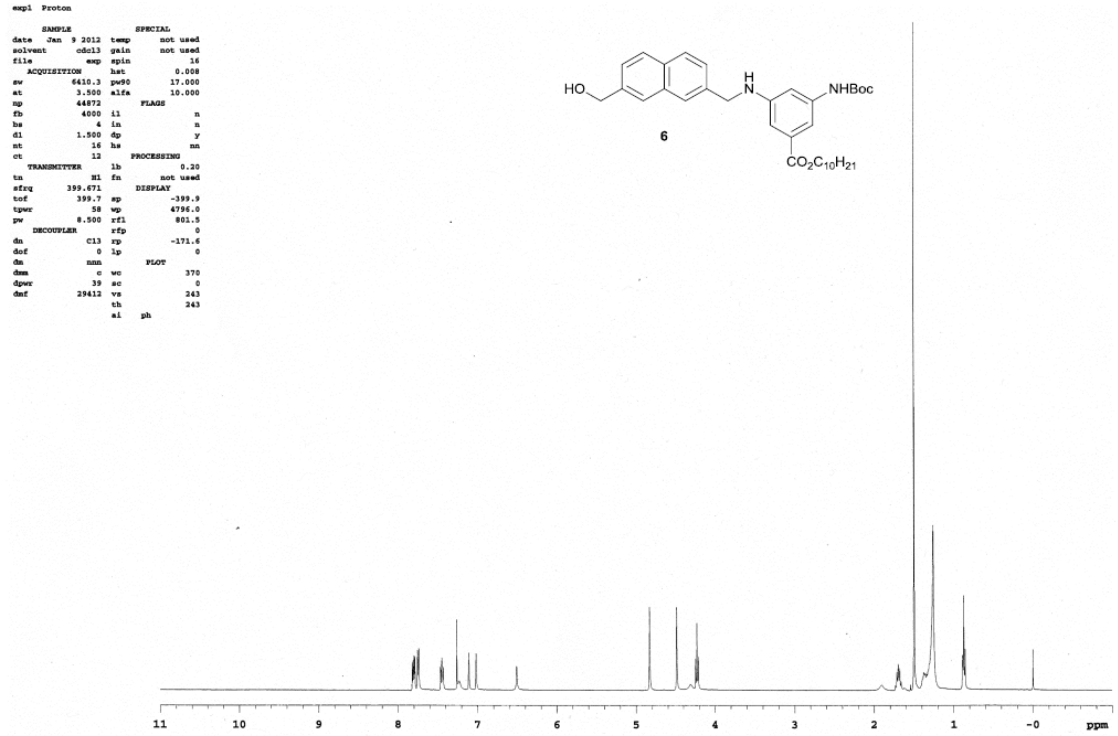

exp1 Carbon
SAMPLE
date Jan 9 2012
solvent cdcl3
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 8
d1 0.700
nt 10000
ct 336
TRANSMITTER
tn C13
sfrq 100.507
tof 1027.8
tpwr 59
pw 5.350
DECOUPLER
dn H1
dof 300.0
dm yyy
dmm w
dpwr 43
dmf 10384
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 10.700
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1665.9
wp 24509.1
rfl 14530.3
rfp 12863.6
rp 133.2
lp 0
PLOT
wc 370
sc 0
vs 634
th 183
ai cdc ph
H2N
CO2C10H21
13
220
200
180
160
140
120
100
80
60
40
20
0
ppm

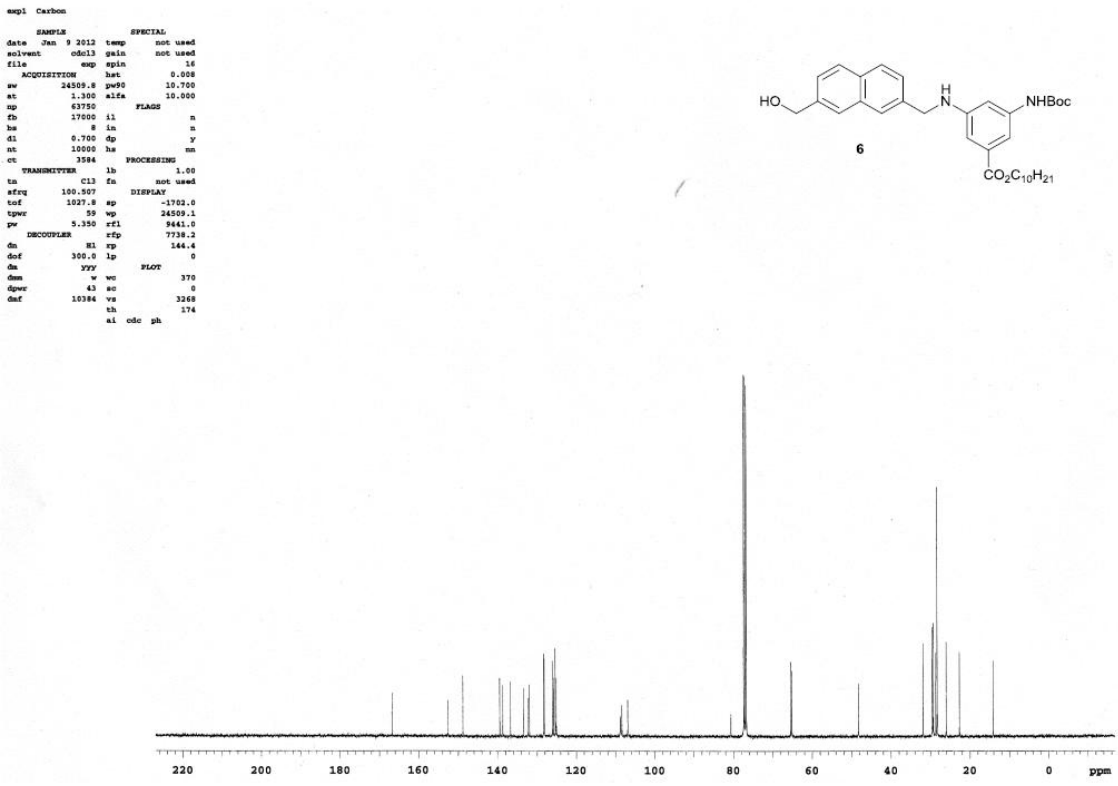

expl Proton
SAMPLE
date Jan 13 2012
solvent cdcl3
file exp
ACQUISITION
sw 6410.3
at 3.500
np 44872
fb 4000
bs 4
d1 1.500
nt 16
ct 12
TRANSMITTER
tn H1
sfrq 399.671
tof 399.7
tpwr 58
pw 8.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 39
dmf 29412
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 17.000
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.20
fn not used
DISPLAY
sp -399.9
wp 4796.0
rfl 804.4
rfp 0
rp -168.5
lp 0
PLOT
wc 370
sc 0
vs 637
th 228
ai ph
HO
H
N
(M)-4
CO2C10H21
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

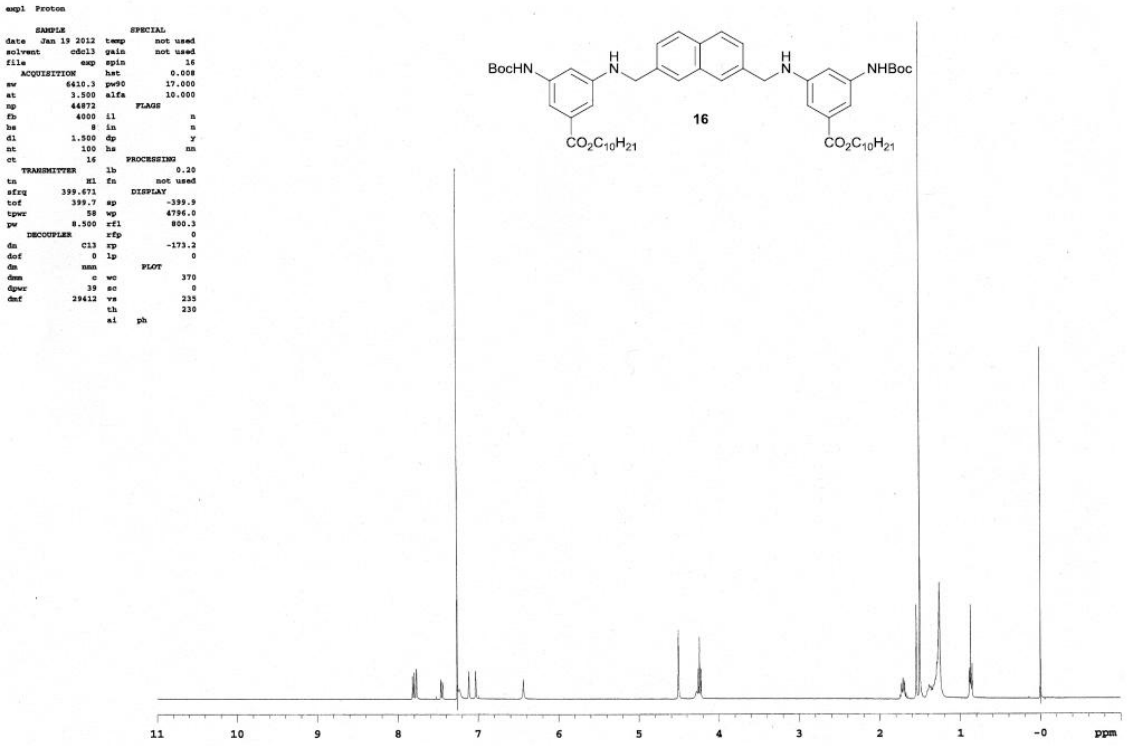

expl Carbon
SAMPLE
date Jan 14 2012
solvent cdcl3
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 8
d1 0.700
nt 200000
ct 15568
TRANSMITTER
tn C13
sfrq 100.507
tof 1027.8
tpwr 59
pw 5.350
DECOUPLER
dn H1
dof 300.0
dm yyy
dmm w
dpwr 43
dmf 10384
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 10.700
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1702.0
wp 24509.1
rfl 1702.7
rfp 0
rp 138.2
lp 0
PLOT
wc 370
sc 0
vs 2562
th 135
ai odc ph
HO
H
N
(M)-4
CO2C10H21
220
200
180
160
140
120
100
80
60
40
20
0
ppm

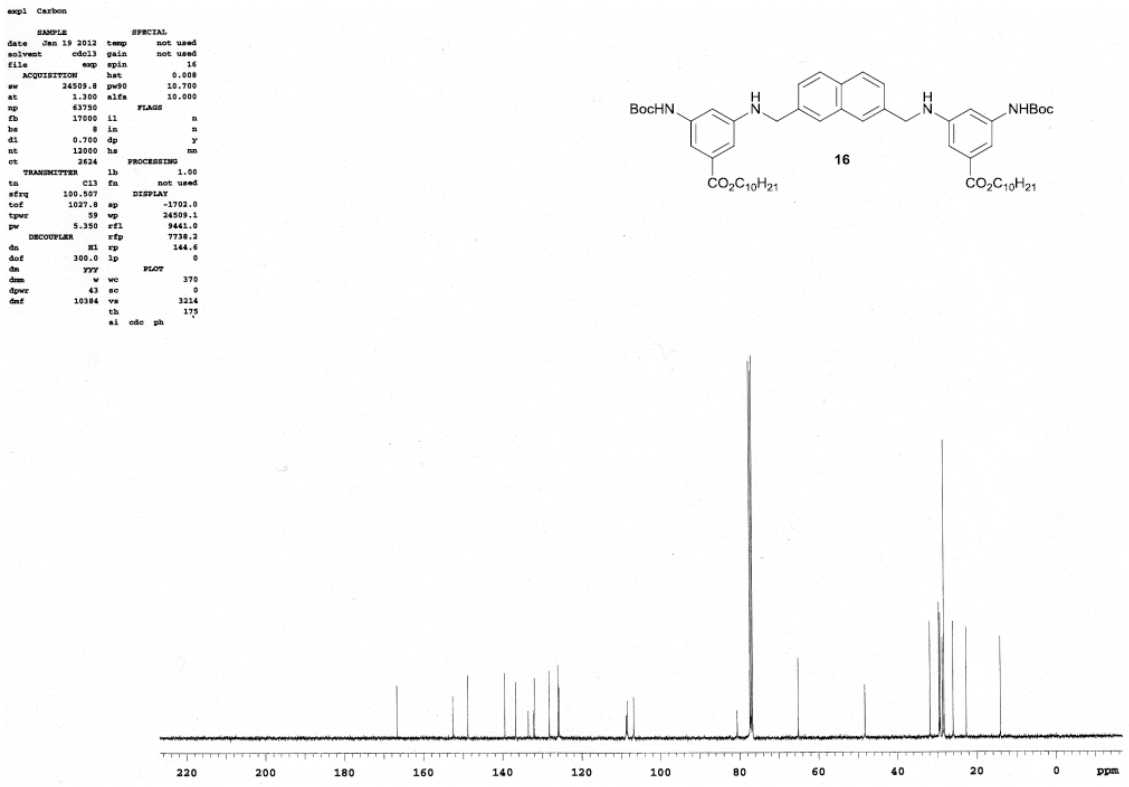

expl Proton
SAMPLE
date Jan 18 2012
solvent cdcl3
file exp
ACQUISITION
sw 6410.3
at 3.500
np 44872
fb 4000
bs 8
d1 1.500
nt 100
ct 32
TRANSMITTER
tn H1
sfrq 399.671
tof 399.7
tpwr 58
pw 8.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 39
dmf 25412
SPECIAL
temp not used
gain not used
spin 16
hst 0.088
pw90 17.050
alfa 16.050
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.30
fn not used
DISPLAY
sp -399.9
wp 4796.0
rfl 800.9
rfp 0
rp -169.8
lp 0
PLOT
wc 370
sc 0
vs 393
th 343
ai ph
(M)-14
CO2C10H21
CO2C10H21
11
10
9
8
7
6
5
4
3
2
1
-0
ppm


expl Carbon
SAMPLE
date Jan 15 2012
solvent cdcl3
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 8
d1 0.700
nt 10000
ct 1056
TRANSMITTER
tn C13
sfrq 100.507
tof 1027.8
tpwr 59
pw 5.350
DECOUPLER
dn H1
dof 300.0
dm yyy
dmm w
dpwr 43
dmf 10384
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 10.700
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -3702.8
wp 24509.1
rfl 9441.7
rfp 7738.2
rp 130.6
lp 0
PLOT
wc 370
sc 0
vs 3021
th 121
ai cdc ph
(M)-14
CO2C10H21
CO2C10H21
220
200
180
160
140
120
100
80
60
40
20
0
ppm


exp1 Proton
SAMPLE
date Jan 9 2012
solvent cdcl3
file exp
ACQUISITION
sw 6410.3
at 3.500
np 44872
fb 4000
bs 4
d1 1.500
nt 16
ct 12
TRANSMITTER
tn H1
sfrq 399.671
tof 399.7
tpwr 58
pw 8.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 39
dmf 29412
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 17.000
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.20
fn not used
DISPLAY
sp -399.9
wp 4796.0
rfl 801.5
rfp 0
rp -171.6
lp 0
PLOT
wc 370
sc 0
vs 243
th 243
ai ph
HO
H
N
NHBoc
6
CO2C10H21
11
10
9
8
7
6
5
4
3
2
1
-0
ppm


exp1 Carbon
SAMPLE
date Jan 9 2012
solvent cdcl3
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 8
d1 0.700
nt 10000
ct 3584
TRANSMITTER
tn C13
sfrq 100.507
tof 1027.8
tpwr 59
pw 5.350
DECOUPLER
dn H1
dof 300.0
dm yyy
dmm w
dpwr 43
dmf 10384
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 10.700
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1702.0
wp 24509.1
rfl 9641.0
rfp 7738.2
rp 144.4
lp 0
PLOT
wc 370
sc 0
vs 3268
th 174
ai cdc ph
HO
H
N
NHBoc
6
CO2C10H21
220
200
180
160
140
120
100
80
60
40
20
0
ppm


expl Proton
SAMPLE
date Jan 19 2012
solvent cdcl3
file exp
ACQUISITION
sw 6410.3
at 3.500
np 44872
fb 4000
bs 8
d1 1.500
nt 100
ct 16
TRANSMITTER
tn H1
sfrq 399.671
tof 399.7
tpwr 58
pw 8.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 39
dmf 29412
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 17.000
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.20
fn not used
DISPLAY
sp -399.9
wp 4796.0
rfl 800.3
rfp 0
rp -173.2
lp 0
PLOT
wc 370
sc 0
vs 235
th 230
ai ph
BocHN
NHBoc
16
CO2C10H21
CO2C10H21
11
10
9
8
7
6
5
4
3
2
1
-0
ppm


expl Carbon
SAMPLE
date Jan 19 2012
solvent cdcl3
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 8
d1 0.700
nt 12000
ct 2624
TRANSMITTER
tn C13
sfrq 100.507
tof 1027.8
tpwr 59
pw 5.350
DECOUPLER
dn H1
dof 300.0
dm yyy
dmm w
dpwr 43
dmf 10384
SPECIAL
temp not used
gain not used
spin 16
hst 0.008
pw90 10.700
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1702.0
wp 24509.1
rfl 9441.0
rfp 7738.2
rp 144.6
lp 0
PLOT
wc 370
sc 0
vs 3214
th 175
ai cdc ph
BocHN
NHBoc
16
CO2C10H21
CO2C10H21
220
200
180
160
140
120
100
80
60
40
20
0
ppm