

Acid-Catalyzed Synthesis of Condensed Polycyclic Diaryl Ethers from Arenols

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Supplementary Materials

^1H , ^{13}C and ^{19}F nuclear magnetic resonance (NMR) spectra were recorded on a Varian Mercury (400 MHz), calibrated using trimethylsilane (TMS, δ 0.00 ppm), residual undeuterated solvent as an internal reference (CHCl_3 , δ 77.0 ppm), and trifluoroacetic acid (TFA, δ -79.0 ppm). IR spectra were measured on a JASCO FT/IR-410 spectrophotometer. Melting points were determined with a Yanagimoto micro melting point apparatus without correction. High- and low-resolution mass spectra were measured on a JEOL JMS-DX-303, a JEOL JMS-700, or a JMS-T100GC spectrometer. Silica gel 60 (40-50 μm , Kanto Chemical CO., INC.) was employed for flash column chromatography.

Experimental Procedures

Synthesis of Symmetric Ethers

Typical experimental procedures: Synthesis of bis(2-naphthyl) ether 2a

In a two-necked flask equipped with a magnetic stirrer bar and a reflux condenser were placed 2-naphthol (1.00 mmol, 144.2 mg) and 4-chlorobenzenesulfonic acid (10 mol%, 19.3 mg) in *o*-dichlorobenzene (1.0 mL) under an argon atmosphere, and the solution was stirred and heated at reflux for 10 h. Solvent was removed in vacuo, and the residue was purified by flash column chromatography on silica gel (Hexane/Toluene 10:1) giving bis(2-naphthyl) ether (**2a**) (85%, 115.4 mg).

Bis(2-naphthyl) ether (**2a**)^[1]

Colorless solid. Mp. 104.0-104.5 °C (Hexane), lit 104-105 °C.^[1] R_f = 0.44 (Hexane/Toluene 6:1). $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.30 (2H, d, J = 8.8 Hz), 7.36-7.42 (6H, m), 7.66 (2H, d, J = 8.0 Hz), 7.80 (2H, d, J = 6.4 Hz), 7.82 (2H, d, J = 8.4 Hz). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 114.4, 120.1, 124.8, 126.5, 127.1, 127.7, 129.9, 130.2, 134.3, 155.0. IR (KBr) 3051, 1595, 1269, 806, 742 cm^{-1} . MS (EI) m/z 270 (M^+ , 100%). HRMS Calcd for ($\text{C}_{20}\text{H}_{14}\text{O}$) $^+$: 270.1045. Found: 270.1035.

Bis(6-bromonaphth-2-yl) ether (**2b**)

Colorless solid. Mp. 178.0-179.0 °C (Hexane). R_f = 0.64 (Hexane/Toluene 6:1). $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.31 (2H, s), 7.32 (2H, dd, J = 6.4, 2.4 Hz), 7.52 (2H, dd, J = 8.8, 2.0 Hz), 7.56 (2H, d, J = 8.8 Hz), 7.77 (2H, d, J = 9.6 Hz), 7.99 (2H, s). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 114.4, 118.6, 121.0, 128.8, 129.2, 129.8, 130.0, 131.3, 132.7, 155.0. IR (KBr) 3063, 1582, 1250, 1209, 878, 808, 638 cm^{-1} . MS (EI) m/z 430 ($\text{M}^+ + 4$, 46%), 428 ($\text{M}^+ + 2$, 100%), 426 (M^+ , 47%). HRMS Calcd for ($\text{C}_{20}\text{H}_{12}\text{Br}_2\text{O}$) $^+$: 425.9255. Found: 425.9250.

Bis(7-bromonaphth-2-yl) ether (**2c**)

Colorless solid. Mp. 191.0-192.0 °C (Hexane). R_f = 0.62 (Hexane/Toluene 6:1). $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.26 (2H, d, J = 3.6 Hz), 7.31 (2H, dd, J = 8.8, 2.4 Hz), 7.50 (2H, dd, J = 8.8, 2.0 Hz), 7.71 (2H, d, J = 8.4 Hz), 7.83 (2H, d, J = 8.8 Hz), 7.87 (2H, d, J = 2.0 Hz). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 113.6, 120.4, 120.9, 128.3, 128.7, 129.2, 129.4, 130.1, 135.5, 155.5. IR (KBr) 3062, 1620, 1251, 833, 769 cm^{-1} . MS (EI) m/z 430 ($\text{M}^+ + 4$, 51%), 428 ($\text{M}^+ + 2$, 100%), 426 (M^+ , 51%). HRMS Calcd for ($\text{C}_{20}\text{H}_{12}\text{Br}_2\text{O}$) $^+$: 425.9255. Found: 425.9230.

Bis(6-methylnaphth-2-yl) ether (**2d**)

Colorless solid. Mp. 145.0-146.0 °C (Hexane/AcOEt = 10). R_f = 0.53 (Hexane/Toluene 6:1). $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 2.48 (6H, s), 7.26 (4H, dd, J = 8.8, 2.4 Hz), 7.31 (2H, d, J = 2.4 Hz), 7.57 (2H, d, J = 8.4 Hz), 7.58 (2H, s), 7.73 (2H, d, J = 9.2 Hz). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 21.5, 114.2, 120.1, 126.7, 127.0, 128.8, 129.2, 130.4, 132.4, 134.3, 154.5. IR (KBr) 3056, 2915, 1600, 1257, 1236, 811 cm^{-1} . MS (EI) m/z 298 (M^+ , 100%). HRMS Calcd for ($\text{C}_{22}\text{H}_{18}\text{O}$) $^+$: 298.1358. Found: 298.1341.

Bis(6-chloronaphth-6-yl) ether (**2e**)

Colorless solid. Mp. 163.0-164.0 °C (Hexane/Toluene = 6). R_f = 0.59 (Hexane/Toluene 6:1). $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.32-7.35 (4H, m), 7.41 (2H, dd, J = 8.8, 2.0 Hz), 7.64 (2H, d, J = 8.8 Hz), 7.78 (2H, d, J = 9.6 Hz), 7.83 (2H, d, J = 2.0 Hz). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 114.4, 121.1, 126.5, 127.5, 128.7, 129.2, 130.5, 130.8, 132.5, 155.0. IR (KBr) 3066, 1589, 1252, 1209, 879, 804 cm^{-1} . MS (EI) m/z 338 (M^+ , 100%). HRMS Calcd for ($\text{C}_{20}\text{H}_{12}\text{Cl}_2\text{O}$) $^+$: 338.0265. Found: 338.0290.

Bi(1-naphthyl) ether (**2f**)^[2]

Colorless solid. Mp. 109.0-110.0 °C (Hexane), lit 109.5 °C.^[2] R_f = 0.66 (Hexane/Toluene 6:1). $^1\text{H-NMR}$ ^[2] (400 MHz, CDCl_3) δ 6.90 (2H, d, J = 7.6 Hz), 7.36 (2H, t, J = 8.0 Hz), 7.50 (2H, td, J = 8.0, 1.2 Hz), 7.55 (2H, td, J = 8.0, 1.2 Hz), 7.64 (2H, d, J = 8.0 Hz), 7.91 (2H, d, J = 8.0 Hz), 8.31 (2H, d, J = 8.0 Hz). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 113.1, 122.0, 123.2, 125.9, 126.0, 126.5, 126.6, 127.8, 134.9, 153.4. IR (KBr) 3057, 1233, 1092, 772 cm^{-1} . MS (EI) m/z 270 (M^+ , 100%). HRMS Calcd for ($\text{C}_{20}\text{H}_{14}\text{O}$) $^+$: 270.1045. Found: 270.1028.

Bis(6,7-dimethylnaphth-5-yl) ether (**2g**)

Colorless solid. Mp. 225.0-226.0 °C (Hexane). R_f = 0.68 (Hexane/Toluene 6:1). $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 2.42 (6H, s), 2.45 (6H, s), 6.77 (2H, d, J = 7.2 Hz), 7.24 (2H, t, J = 8.0 Hz), 7.50 (2H, d, J = 8.4 Hz), 7.65 (2H, s), 8.06 (2H, s). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 20.2, 20.4, 112.2, 121.4, 122.1, 125.0, 125.4, 127.4, 133.9, 135.8, 136.4, 153.0. IR (KBr) 3060, 2939, 2916, 1565, 1230, 1140, 792, 744 cm^{-1} . MS (EI) m/z 326 (M^+ , 100%). HRMS Calcd for ($\text{C}_{24}\text{H}_{22}\text{O}$) $^+$: 326.1671. Found: 326.1676.

Bis(4,6,7-trimethylnaphth-1-yl) ether (**2h**)

Colorless solid. Mp 182.0-183.0 °C (Hexane). R_f = 0.62 (Hexane/Toluene 6:1). $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 2.41 (6H, s), 2.48 (6H, s), 2.62 (6H, s), 6.65 (2H, d, J = 7.6 Hz), 7.06 (2H, d, J = 7.6 Hz), 7.75 (2H, s), 8.09 (2H, s). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 19.0, 20.3, 20.6,

111.7, 122.0, 124.1, 125.4, 125.4, 128.2, 132.6, 135.3, 136.0, 151.8. IR (KBr) 3024, 2963, 2938, 2913, 2853, 1430, 1247, 1208, 862 cm⁻¹. MS (EI) *m/z* 354 (M⁺, 100%). HRMS Calcd for (C₂₆H₂₆O)⁺: 354.1984. Found: 354.1986.

Bis(4-methylnaphth-1-yl) ether (2i)

Colorless solid. Mp. 128.0-129.0 °C (Hexane). R_f = 0.83 (Hexane/Toluene 6:1). ¹H-NMR (400 MHz, CDCl₃) δ 2.66 (6H, d, *J* = 0.8 Hz), 6.78 (2H, d, *J* = 7.6 Hz), 7.17 (2H, dd, *J* = 8.0, 0.8 Hz), 7.50 (2H, ddd, *J* = 8.0, 6.8, 1.2 Hz), 7.59 (2H, ddd, *J* = 8.4, 6.8, 1.6 Hz), 8.02 (2H, d, *J* = 8.4 Hz), 8.34 (2H, d, *J* = 8.0 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 19.0, 112.6, 122.6, 124.3, 125.6, 126.2, 126.4, 126.6, 129.3, 133.7, 152.1. IR (KBr) 3064, 2936, 1577, 1236, 758 cm⁻¹. MS (EI) *m/z* 298 (M⁺, 100%), 283 (M⁺-CH₃, 24%), 268 (M⁺-C₂H₆, 12%), 141 (M⁺-C₁₃H₉O, 13%). HRMS Calcd for (C₂₂H₁₈O)⁺: 298.1358. Found: 298.1357.

Bis(6,7-dibromonaphth-5-yl) ether (2j)

Colorless solid. Mp. 238.5-239.5 °C (Hexane/Toluene = 2). R_f = 0.59 (Hexane/Toluene 6:1). ¹H-NMR (400 MHz, CDCl₃) δ 6.88 (2H, dd, *J* = 7.6, 1.2 Hz), 7.40 (2H, d, *J* = 8.0 Hz), 7.54 (2H, d, *J* = 8.4 Hz), 8.22 (2H, s), 8.56 (2H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 114.0, 122.5, 122.7, 123.4, 126.2, 126.7, 127.4, 132.3, 134.5, 152.2. IR (KBr) 3056, 1562, 1254, 1101, 785, 736 cm⁻¹. MS (EI) *m/z* 590 (M⁺+8, 17%), 588 (M⁺+6, 59%), 586 (M⁺+4, 100%), 584 (M⁺+2, 69%), 582 (M⁺, 15%). HRMS Calcd for (C₂₀H₁₀Br₄O)⁺: 581.7465. Found: 581.7464.

Bis(9-phenanthryl) ether (2k)^[3]

Colorless solid. Mp. 212.5-213.0 °C (Hexane), lit 202.5-203.0 °C^[3]. R_f = 0.47 (Hexane/Toluene = 6). ¹H-NMR (400 MHz, CDCl₃) δ 7.21 (2H, s), 7.51 (2H, ddd, *J* = 8.0, 8.0, 1.2 Hz), 7.58 (2H, ddd, *J* = 8.4, 6.8, 1.6 Hz), 7.63 (4H, td, *J* = 6.8, 1.2 Hz), 7.74 (2H, ddd, *J* = 8.4, 7.2, 1.6 Hz), 8.43 (2H, dd, *J* = 8.4, 1.2 Hz), 8.68 (2H, d, *J* = 8.4 Hz), 8.76 (2H, d, *J* = 8.4 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 112.1, 122.6, 122.7, 122.9, 125.5, 126.7, 126.9, 127.0, 127.5, 127.8, 127.9, 131.9, 132.2, 151.4. IR (KBr) 3054, 1625, 1227, 766, 727 cm⁻¹. MS (EI) *m/z* 370 (M⁺, 100%). HRMS Calcd for (C₂₈H₁₈O)⁺: 379.1358. Found: 370.1339.

Bis(2-phenanthryl) ether (2l)

Colorless solid. Mp. 198.5-199.5 °C (Hexane/Toluene 10:1). R_f = 0.32 (Hexane/Toluene = 6). ¹H-NMR (400 MHz, CDCl₃) δ 7.45 (2H, d, *J* = 9.2 Hz), 7.48 (2H, s), 7.54–7.65 (6H, m), 7.71 (2H, d, *J* = 9.2 Hz), 7.86 (2H, d, *J* = 8.0 Hz), 8.60 (2H, d, *J* = 8.0 Hz), 8.66 (2H, d, *J* = 8.8 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 116.1, 119.3, 122.4, 124.7, 126.2, 126.3, 126.5, 126.8, 127.8, 128.6, 130.2, 131.4, 133.4, 155.8. IR (KBr) 3047, 1613, 1266, 744 cm⁻¹. MS (EI) *m/z* 370 (M⁺, 100%). HRMS Calcd for (C₂₈H₁₈O)⁺: 370.1358. Found: 370.1378.

Bis(4-pyrenyl) ether (2m)

Colorless solid. Mp. 234.0-235.0 °C (Hexane/Toluene 3:1). R_f = 0.52 (Hexane/Toluene = 6). ¹H-NMR (400 MHz, CDCl₃) δ 7.65 (2H, s), 7.94 (2H, dd, *J* = 12.4, 8.0 Hz), 7.96 (2H, s), 8.05 (2H, t, *J* = 8.0 Hz), 8.14-8.17 (6H, m), 8.30 (2H, dd, *J* = 8.0, 1.2 Hz), 8.74 (2H, dd, *J* = 8.0, 1.2 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 112.6, 119.9, 122.3, 124.2, 124.6, 125.9, 126.0, 126.0, 126.1, 126.3, 127.4, 127.6, 131.1, 131.4, 131.4, 152.5. IR (KBr) 3041, 1593, 1269, 717 cm⁻¹. MS (EI) *m/z* 418 (M⁺, 100%). HRMS Calcd for (C₃₂H₁₈O)⁺: 418.1358. Found: 418.1329.

Synthesis of Unsymmetric Ethers

Typical experimental procedures: Synthesis of 9-(3-fluorophenoxy)phenanthrene

In a two-necked flask equipped with a magnetic stirrer bar were placed 9-phenanthrol (0.5 mmol, 97.1 mg), 3-fluorophenol (0.5 mmol, 56.1 mg), and 4-chlorobenzenesulfonic acid (0.1 mmol, 19.3 mg) in *o*-dichlorobenzene (1.0 mL) under an argon atmosphere, and the solution was stirred and heated at reflux for 10 h. Solvent was removed in vacuo, and the residue was purified by flush column chromatography on silica gel (Hexane/Toluene 10:1) giving 9-(3-fluorophenoxy)phenanthrene (**4a**) (68%, 97.9 mg).

9-(3-Fluorophenoxy)phenanthrene (4a)

Colorless solid. Mp. 82.5-83.0 °C (Hexane). R_f = 0.30 (Hexane/Toluene 6:1). ¹H-NMR (400 MHz, CDCl₃) δ 6.79-6.83 (2H, m), 6.86 (1H, d, *J* = 8.8 Hz), 7.20 (1H, s), 7.26 (1H, td, *J* = 8.4, 6.8 Hz), 7.50-7.61 (3H, m), 7.65-7.71 (2H, m), 8.22 (1H, dd, *J* = 8.4, 0.8 Hz), 8.61 (1H, d, *J* = 8.4 Hz), 8.68 (1H, d, *J* = 8.4 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 106.1 (d, *J* = 24.5 Hz), 110.1 (d, *J* = 21.6 Hz), 113.1, 114.0 (d, *J* = 3.0 Hz), 122.6, 122.6, 122.9, 125.8, 126.7, 126.8, 127.0, 127.4, 127.9, 128.0, 130.6 (d, *J* = 9.7 Hz), 131.8, 132.0, 150.6, 159.0 (d, *J* = 10.4 Hz), 163.6 (d, *J* = 245.8 Hz). ¹⁹F-NMR (376 MHz, CDCl₃) δ -114.2. IR (KBr) 3065, 1609, 1485, 1271, 1125, 738, 726 cm⁻¹. MS (EI) *m/z* 288 (M⁺, 100%). HRMS Calcd for (C₂₀H₁₃FO)⁺: 288.0950. Found: 288.0935.

9-(4-Chlorophenoxy)phenanthrene (4b)

Colorless solid. Mp. 104.0-105.0 °C (Hexane). R_f = 0.59 (Hexane/Toluene 6:1). ¹H-NMR (400 MHz, CDCl₃) δ 6.99 (2H, d, *J* = 8.8 Hz), 7.06 (1H, s), 7.26 (2H, d, *J* = 9.2 Hz), 7.48 (1H, dd, *J* = 7.2, 1.2 Hz), 7.52 (1H, dd, *J* = 7.2, 1.6 Hz), 7.57 (1H, ddd, *J* = 9.2, 8.0, 1.2 Hz), 7.64 (2H, ddd, *J* = 9.6, 8.4, 1.6 Hz), 8.25 (1H, dd, *J* = 8.0, 1.2 Hz), 8.56 (1H, d, *J* = 8.0 Hz), 8.63 (1H, d, *J* = 8.4 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 111.8, 120.2, 122.5, 122.6, 122.8, 125.6, 126.6, 126.8, 127.0, 127.4, 127.75, 127.80, 128.4, 129.8, 131.7, 132.0, 151.2, 155.9. IR (KBr) 3054, 1589, 1238, 1063, 823, 726 cm⁻¹. MS (EI) *m/z* 304 (M⁺, 100%). HRMS Calcd for (C₂₀H₁₃ClO)⁺: 304.0655. Found: 304.0647.

9-(4-Methoxyphenoxy)phenanthrene (4c)

Colorless solid. Mp. 123.5-124.5 °C (Hexane/Toluene 10:1). R_f = 0.26 (Hexane/Toluene 6:1). ¹H-NMR (400 MHz, CDCl₃) δ 3.84 (3H, s), 6.92 (1H, s), 6.95 (2H, d, *J* = 9.2 Hz), 7.12 (2H, d, *J* = 9.2 Hz), 7.50 (1H, td, *J* = 7.2, 1.6 Hz), 7.54 (1H, td, *J* = 7.2, 1.6 Hz), 7.63–7.68 (2H, m), 7.73 (1H, ddd, *J* = 8.4, 7.2, 1.6 Hz), 8.44 (1H, dd, *J* = 8.0, 1.2 Hz), 8.62 (1H, d, *J* = 8.0 Hz), 8.71 (1H, d, *J* = 8.0 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 55.7, 108.9, 115.0, 121.2, 122.5, 122.6, 122.7, 125.0, 126.6, 126.7, 126.9, 127.2, 127.3, 127.6, 131.6, 132.4, 149.9, 153.1, 156.1. IR (KBr) 3075, 2832, 1504, 1207, 834, 726 cm⁻¹. MS (EI) *m/z* 300 (M⁺, 100%). HRMS Calcd for (C₂₁H₁₆O₂)⁺: 300.1150. Found: 300.1143.

9-((6-Chloronaphthalen-2-yl)oxy)phenanthrene (4d)

Colorless solid. Mp. 122.5-123.5 °C (Hexane). R_f = 0.53 (Hexane/Toluene 6:1). ¹H-NMR (400 MHz, CDCl₃) δ 7.20 (1H, s), 7.35 (1H, d, *J* = 2.4 Hz), 7.38 (1H, dd, *J* = 8.8, 2.4 Hz), 7.43 (1H, dd, *J* = 8.8, 2.4 Hz), 7.53-7.65 (4H, m), 7.70 (1H, dd, *J* = 7.2, 1.6 Hz), 7.73 (1H, ddd, *J* = 8.4, 6.8, 1.2 Hz), 7.80 (1H, d, *J* = 9.2 Hz), 7.83 (1H, d, *J* = 2.0 Hz), 8.32 (1H, dd, *J* = 8.4, 1.2 Hz), 8.68 (1H, d, *J* = 8.4 Hz), 8.75 (1H, d, *J*

= 8.4 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 112.6, 114.0, 120.9, 122.6, 122.7, 122.9, 125.7, 126.5, 126.8, 126.9, 127.1, 127.4, 127.5, 127.9, 127.9, 128.7, 129.2, 130.4, 130.7, 131.8, 132.1, 132.6, 151.2, 155.6. IR (KBr) 3071, 1591, 1246, 1195, 1147, 726 cm⁻¹. MS (EI) *m/z* 354 (M⁺, 100%). HRMS Calcd for (C₂₄H₁₅ClO)⁺: 354.0811. Found: 354.0806.

9-((4-Chloronaphthalen-1-yl)oxy)phenanthrene (4e)

Colorless solid. Mp. 133.0-134.0 °C (Hexane). R_f = 0.60 (Hexane/Toluene 6:1). ¹H-NMR (400 MHz, CDCl₃) δ 6.96 (1H, d, *J* = 8.4 Hz), 7.07 (1H, s), 7.48 (1H, d, *J* = 8.0 Hz), 7.51-7.71 (6H, m), 7.76 (1H, ddd, *J* = 8.4, 7.2, 1.6 Hz), 8.33 (1H, d, *J* = 8.4 Hz), 8.34 (1H, d, *J* = 8.8 Hz), 8.40 (1H, dd, *J* = 8.0, 1.2 Hz), 8.67 (1H, d, *J* = 8.0 Hz), 8.76 (1H, d, *J* = 8.0 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 111.4, 113.8, 122.5, 122.5, 122.6, 122.9, 124.7, 125.6, 125.9, 126.4, 126.7, 126.9, 126.9, 127.0, 127.5, 127.6, 127.8, 127.8, 127.9, 131.8, 131.9, 132.1, 151.7, 152.0. IR (KBr) 3060, 1589, 1228, 1079, 736 cm⁻¹. MS (EI) *m/z* 354 (M⁺, 100%). HRMS Calcd for (C₂₄H₁₅ClO)⁺: 354.0811. Found: 354.0844.

9-((6,7-Dibromonaphthalen-1-yl)oxy)phenanthrene (4f)

Colorless solid. Mp. 192.0-193.0 °C (Hexane/Toluene 2:1). R_f = 0.56 (Hexane/Toluene 6:1). ¹H-NMR (400 MHz, CDCl₃) δ 6.93 (1H, dd, *J* = 7.6, 0.8 Hz), 7.17 (1H, s), 7.37 (1H, t, *J* = 8.0 Hz), 7.50 (1H, d, *J* = 8.4 Hz), 7.55 (1H, ddd, *J* = 8.4, 7.2, 1.2 Hz), 7.59 - 7.65 (2H, m), 7.69 (1H, dd, *J* = 8.0, 1.2 Hz), 7.75 (1H, ddd, *J* = 8.4, 7.2, 1.6 Hz), 8.22 (1H, s), 8.29 (1H, dd, *J* = 8.4, 0.8 Hz), 8.68 (1H, dd, *J* = 8.4, 0.4 Hz), 8.69 (1H, s), 8.76 (1H, d, *J* = 8.0 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 112.7, 113.6, 121.9, 122.5, 122.5, 122.7, 123.0, 123.3, 125.8, 126.1, 126.5, 126.9, 127.0, 127.1, 127.5, 127.6, 128.0, 128.1, 131.9, 132.0, 132.2, 134.5, 150.9, 152.7. IR (KBr) 3072, 1565, 1230, 1090, 738 cm⁻¹. MS (EI) *m/z* 480 (M⁺+4, 50%), 478 (M⁺+2, 100%), 476 (M⁺, 50%). HRMS Calcd for (C₂₄H₁₄Br₂O)⁺: 475.9411. Found: 475.9405.

2-(Phenanthren-9-yloxy)phenanthrene (4g)

Colorless solid. Mp. 151.0-152.0 °C (Hexane/Toluene 2:1). R_f = 0.44 (Hexane/Toluene 6:1). ¹H-NMR (400 MHz, CDCl₃) δ 7.22 (1H, s), 7.51-7.76 (11H, m), 7.88 (1H, dd, *J* = 8.0, 1.2 Hz), 8.40 (1H, dd, *J* = 8.0, 1.2 Hz), 8.64 (1H, d, *J* = 8.4 Hz), 8.68 (1H, d, *J* = 8.0 Hz), 8.71 (1H, d, *J* = 9.2 Hz), 8.76 (1H, d, *J* = 8.4 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 112.1, 116.1, 119.3, 122.4, 122.6, 122.8, 122.8, 124.8, 125.5, 126.2, 126.4, 126.5, 126.8 (overlapped 2 peaks), 126.9, 127.0, 127.4, 127.8, 127.8, 127.9, 128.6, 130.2, 131.4, 131.8, 132.2, 133.5, 151.6, 156.0. IR (KBr) 3057, 1611, 1599, 1252, 750, 726 cm⁻¹. MS (EI) *m/z* 370 (M⁺, 100%). HRMS Calcd for (C₂₈H₁₈O)⁺: 370.1358. Found: 370.1380.

2-(Phenanthren-9-yloxy)triphenylene (4h)

Colorless solid. Mp. 195.0-196.0 °C (Toluene/Hexane 5:1). R_f = 0.30 (Hexane/Toluene 6:1). ¹H-NMR (400 MHz, CDCl₃) δ 7.22 (1H, s), 7.47 (1H, dd, *J* = 9.2, 2.4 Hz), 7.53 (1H, ddd, *J* = 8.0, 7.2, 1.2 Hz), 7.58 (1H, dd, *J* = 7.2, 1.2 Hz), 7.62 (1H, dd, *J* = 7.2, 1.6 Hz), 7.64-7.70 (5H, m), 7.77 (1H, ddd, *J* = 8.4, 6.8, 1.2 Hz), 8.39 (1H, d, *J* = 2.4 Hz), 8.46 (2H, t, *J* = 8.0 Hz), 8.61 (1H, d, *J* = 9.6 Hz), 8.65-8.71 (4H, m), 8.77 (1H, d, *J* = 8.0 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 111.5, 112.8, 119.4, 122.6, 122.8, 122.8, 123.1, 123.3, 123.4, 123.5, 125.4, 125.5, 126.0, 126.9 (overlapped 3 peaks), 127.0, 127.2, 127.4, 127.5, 127.6, 127.7, 127.9, 129.2, 129.2, 129.6, 130.1, 131.7, 131.8, 132.2, 151.9, 156.5. IR (KBr) 3073, 1612, 1225, 1199, 754, 729 cm⁻¹. MS (EI) *m/z* 420 (M⁺, 100%). HRMS Calcd for (C₃₂H₂₀O)⁺: 420.1514. Found: 420.1521.

1-(Phenanthren-9-yloxy)pyrene (4i)

Yellow solid. Mp. 197.0-198.0 °C (Toluene/Hexane 2:1). R_f = 0.45 (Hexane/Toluene 6:1). ¹H-NMR (400 MHz, CDCl₃) δ 6.89 (1H, s), 7.44-7.57 (3H, m), 7.73 (1H, ddd, *J* = 8.4, 7.2, 1.2 Hz), 7.75 (1H, d, *J* = 8.4 Hz), 7.80 (1H, ddd, *J* = 8.4, 7.2, 1.6 Hz), 8.03 (1H, t, *J* = 7.6 Hz), 8.05 (1H, d, *J* = 9.6 Hz), 8.08 (2H, d, *J* = 2.4 Hz), 8.17 (2H, d, *J* = 8.8 Hz), 8.21 (1H, d, *J* = 7.6 Hz), 8.40 (1H, d, *J* = 9.2 Hz), 8.67 (2H, d, *J* = 8.0 Hz), 8.78 (1H, d, *J* = 8.4 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 109.8, 117.8, 121.1, 122.6, 122.7, 122.8, 123.0, 124.8, 125.0, 125.1, 125.2, 125.6, 126.1, 126.4, 126.5, 126.9, 126.9, 127.2, 127.4, 127.5, 127.7 (overlapped 2 peaks), 127.7, 128.1, 131.3, 131.4, 131.8, 132.3, 150.0, 153.2. IR (KBr) 3045, 1596, 1248, 831 cm⁻¹. MS (EI) *m/z* 394 (M⁺, 100%). HRMS Calcd for (C₃₀H₁₈O)⁺: 398.1358. Found: 394.1357.

Synthesis ¹⁸O-labeled phenol^[4]: Aniline (0.91 mL, 10 mmol) was dissolved in water (3.5 mL) and 50% tetrafluoroboric acid (3.5 mL) was added. The solution was cooled to 0 °C, and a solution of sodium nitrite (700 mg, 10.1 mmol) in water (1.5 mL) was added dropwise. The suspension was stirred keeping 0 °C for 30 min, and the mixture was filtered, solid materials was purified by re-precipitation from acetone/diethyl ether (5:1) solution. Benzenediazonium tetrafluoroborate was obtained in 78% yield (1.5g, 7.81 mmol) dried under vacuum. Concentrated sulfuric acid (62.5 μL) was added to a stirred paste of benzenediazonium tetrafluoroborate (0.5g, 2.6 mmol) in 98% [¹⁸O]water (0.5 mL, TAIYO NIPPON SANSO Coporation). The mixture was then heated to 65 °C until evolution of nitrogen ceased. The solution was extracted with diethyl ether several times. The ethereal layer was washed with 1 M HCl and saturated brine, dried over magnesium sulfate, and concentrated under reduced pressure. The crystalline residue was purified by sublimation to give the ¹⁸O-labelled phenol as colourless solid (158.8 mg, 64%). The ¹⁸O-enrichment of phenol was found by ESI-MS to be 85%.

The reaction of 9-phenanthrol (1k) and ¹⁸O-labeled phenol: In a two-necked flask equipped with a magnetic stirrer bar were placed 9-phenanthrol (0.125 mmol, 24.3 mg), ¹⁸O-phenol (0.125 mmol, 12.0 mg), and 4-chlorobenzenesulfonic acid (0.025 mmol, 4.8 mg) in *o*-dichlorobenzene (0.25 mL) under an argon atmosphere, and the solution was stirred and heated at reflux for 10 h. Solvent was removed in vacuo, and the residue was purified by flush column chromatography on silica gel (Hexane/Toluene 50:1) giving 9-phenoxyphenanthrene (4j) (63%, 21.4 mg) and 2k (28%, 6.4 mg). The ¹⁸O-enrichment of 4j was found by ESI-MS to be at least 83%. 2k was not labelled with ¹⁸O.

9-phenoxyphenanthrene (4j)

Colorless solid. Mp. 77.5-78.5 °C (Hexane). R_f = 0.2 (Hexane/Toluene 10:1). ¹H-NMR (400 MHz, CDCl₃) δ 7.10-7.20 (4H, m), 7.37 (2H, t, *J* = 8.0 Hz), 7.49-7.57 (2H, m), 7.57-7.72 (3H, m), 8.34 (1H, d, *J* = 8.4 Hz), 8.62 (1H, d, *J* = 8.0 Hz), 8.69 (1H, d, *J* = 8.0 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 111.4, 119.1, 122.6, 122.7, 123.5, 125.4, 126.7, 126.9 (overlapped 2 peaks), 127.3, 127.7, 127.8, 129.9, 131.7, 132.2, 151.7, 157.3. IR (KBr) 3061, 1488, 1229, 1066 cm⁻¹. MS (EI) *m/z* 270 (M⁺, 100%). HRMS Calcd for (C₂₀H₁₄O)⁺: 270.1045. Found: 270.1037. One carbon peaks were piled up in ¹³C-NMR. The data consistent with that reported in the literature.^[5]

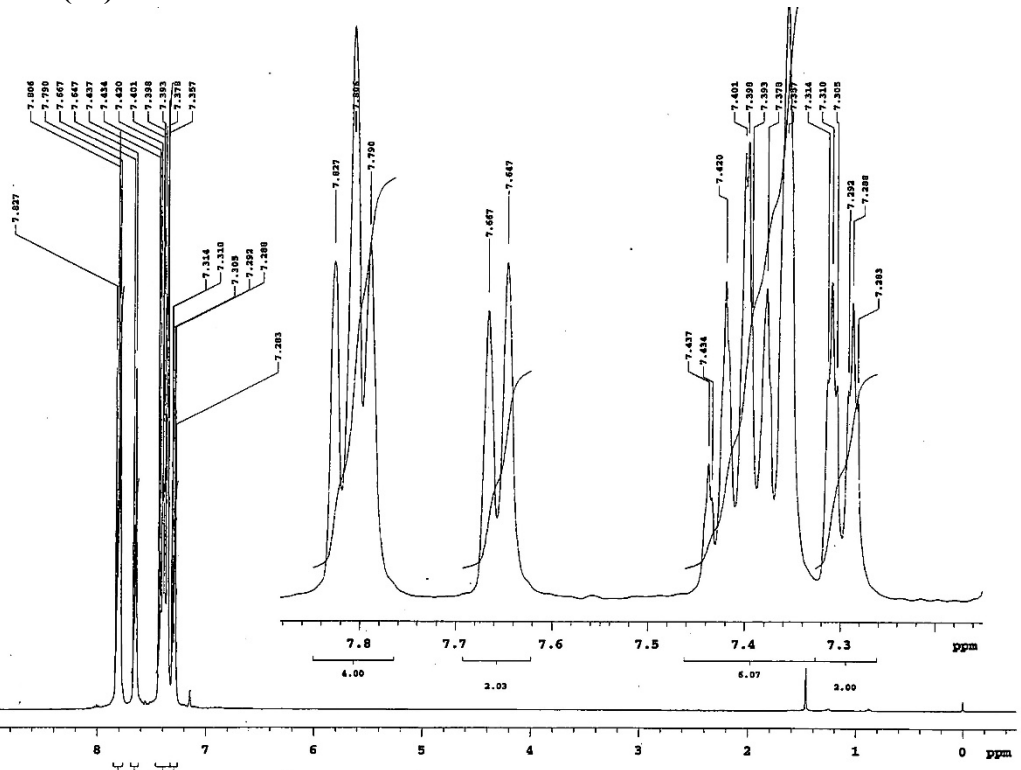
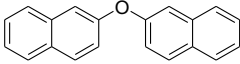
References

- [1] B. J. Morrison, O. C. Musgrave, *Phosphorus, Sulfur and Silicon*, **2002**, 177, 2725-2744.
- [2] F. Ullmann, P. Sponagel, *Justus Liebigs Ann. Chem.* **1907**, 350, 83-107.
- [3] R. C. Fuson, R. L. Talbott. *J. Org. Chem.*, **1961**, 26, 2674-2676.
- [4] A. N. Holding and J. B. Spencer, *ChemBioChem*, **2008**, 9, 2209-2214.
- [5] Y. Chen, N. Zhang, L. Ye, J. Chen, X. Sun, X. Zhang, and M. Yan, *RSC Adv.*, **2015**, 5, 48046-48049.

2,2'-Oxybisanthalene (2a) ¹H-NMR

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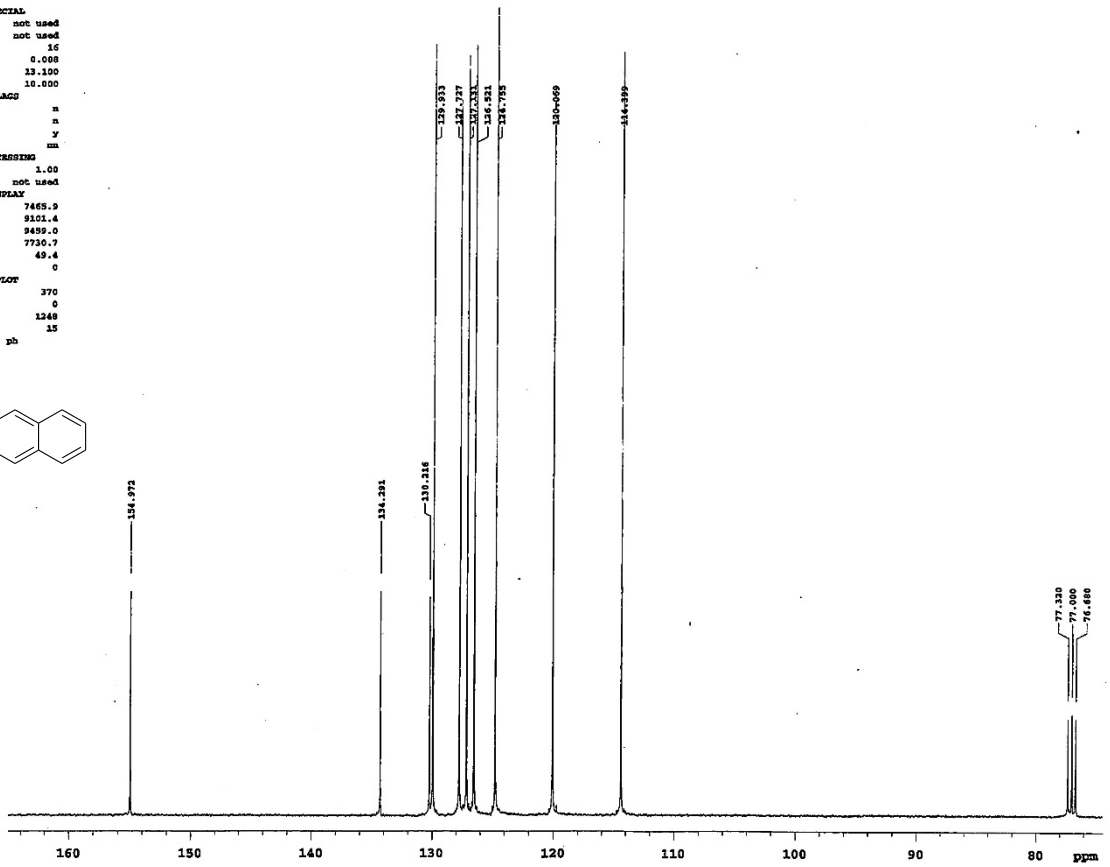
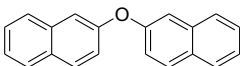
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2,2'-Oxybisanthalene (2a) ¹³C-NMR

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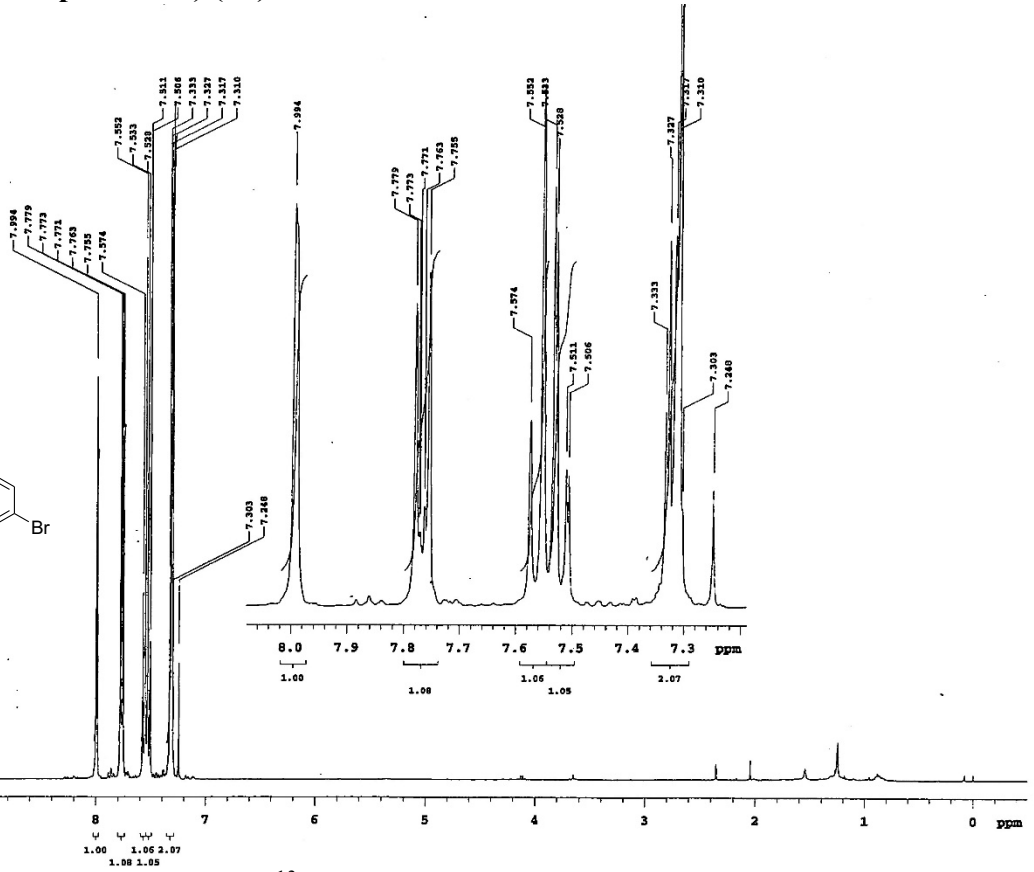
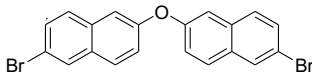
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6,6'-Oxybis(2-bromonaphthalene) (2b) ¹H-NMR

```

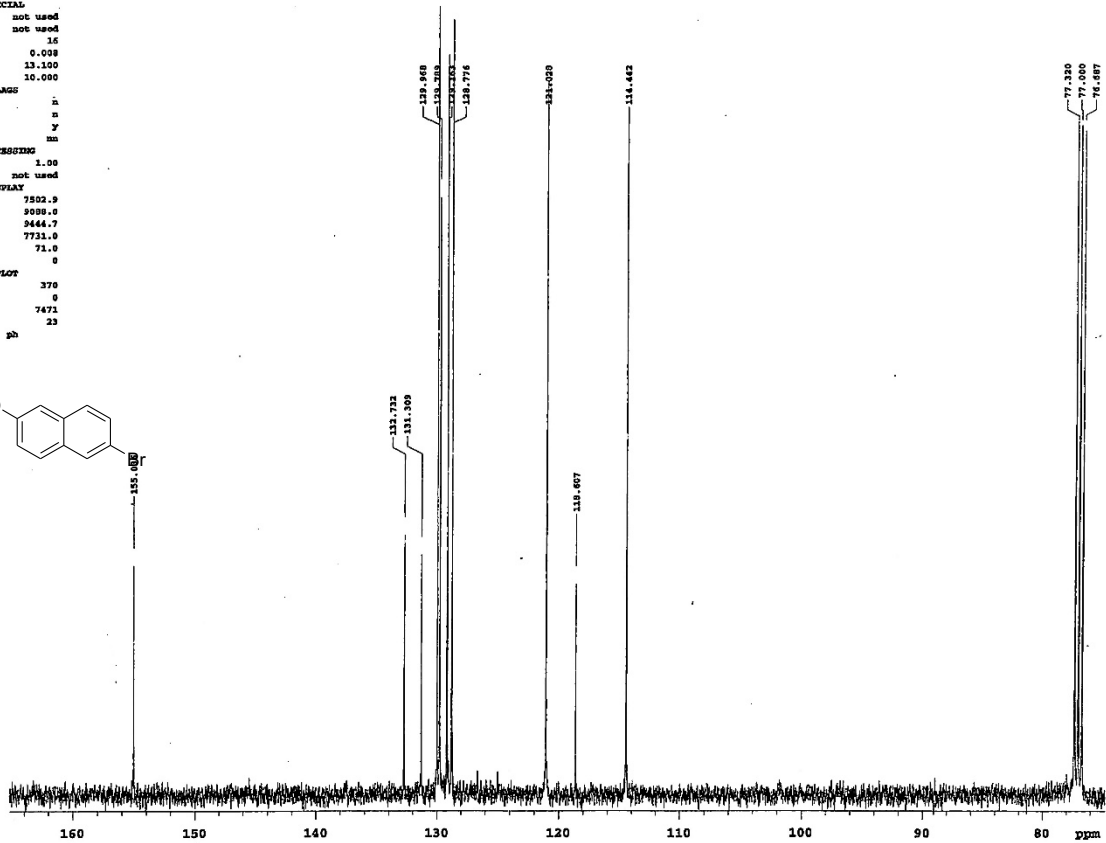
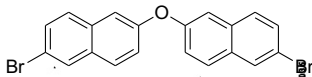
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pwr 58 lp 0
sw 7.300 FLOW
DECOUPLER      wc 180
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lof 0 vs 262
tm nm ch 16
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6,6'-Oxybis(2-bromonaphthalene) (2b) ¹³C-NMR

```

exp4 Carbon
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solvent cdcl3 gain not used
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nt 20000 DISPLAY
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TRANSMITTER    wp 5088.0
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cof 1026.8 xp 71.0
tpr 60 lp 0
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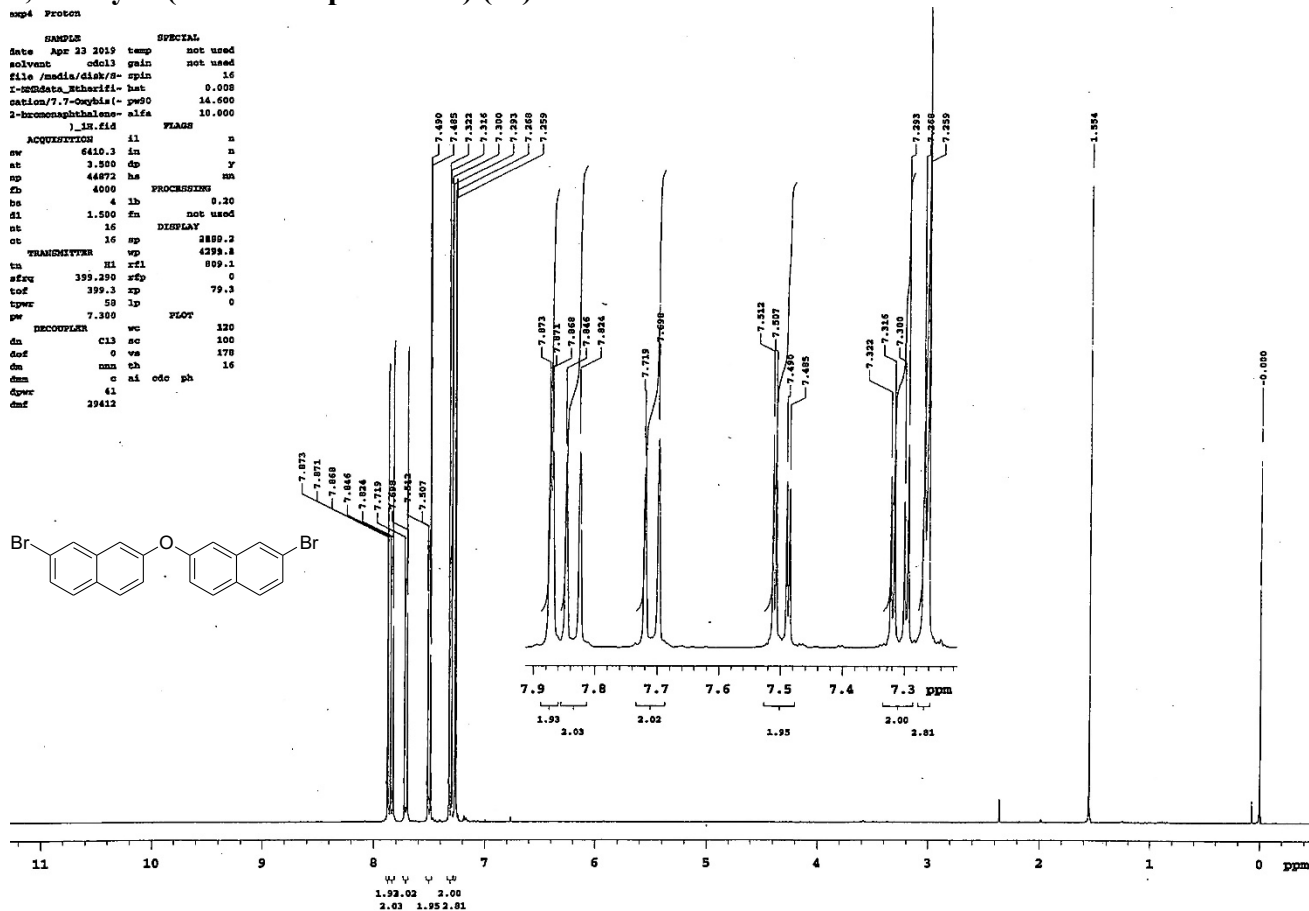
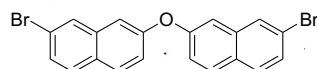


7,7'-Oxybis(2-bromonaphthalene) (2c) ¹H-NMR

exp4 Proton

```

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solvent cdcl3 gain not used
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i-bromonaphthalene- aifa 10.000
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st 1.500 sp y
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sl 1.500 fm not used
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ot 16 sp 2889.2
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sfreq 395.290 rfp 0
tof 399.3 xp 79.3
tpwr 59 lp 0
pw 7.300 PLOT
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dnt 29412
    
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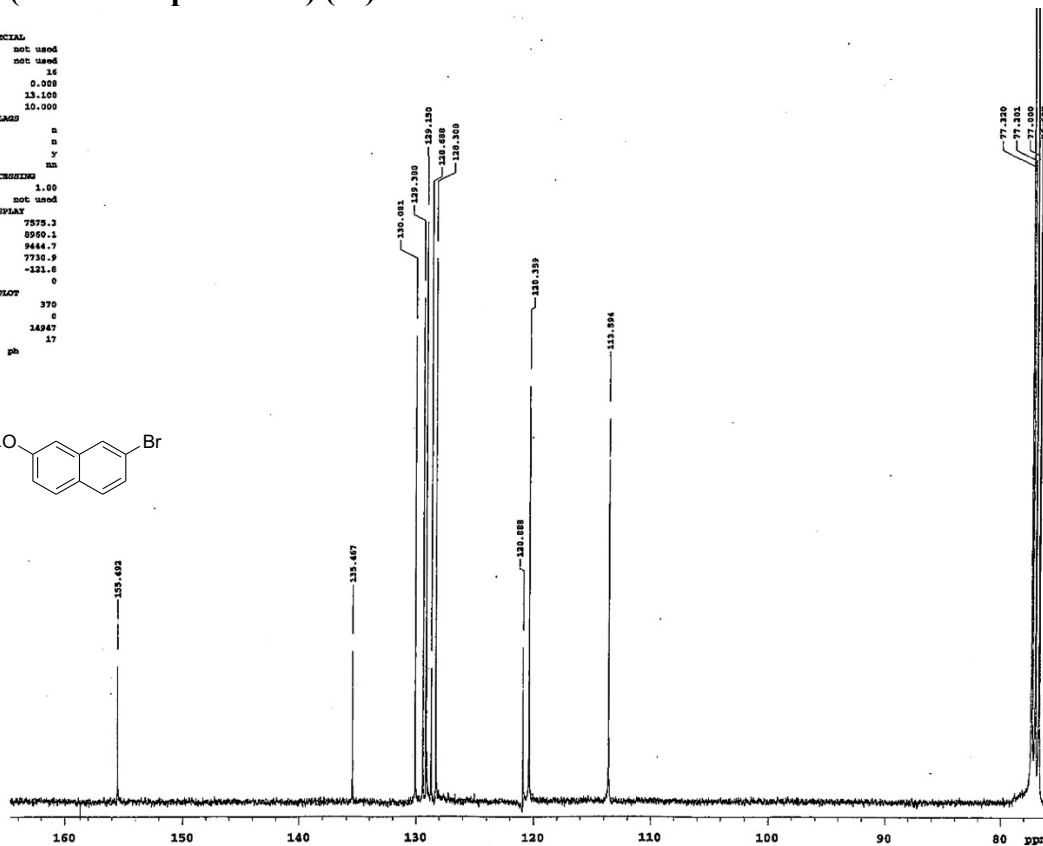
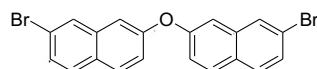


7,7'-Oxybis(2-bromonaphthalene) (2c) ¹³C-NMR

exp4 Carbon

```

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solvent cdcl3 gain not used
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sp 61750 hs nm
zb 17000 PROCESSING
bs 16 lb 1.00
sl 0.700 fm not used
st 102400 DISPLAY
ot 10944 sp 7575.2
TRANSMITTER    wp 8959.1
ta c13 xfl 9444.7
sfreq 100.611 rfp 7735.9
tof 1026.9 xp -121.6
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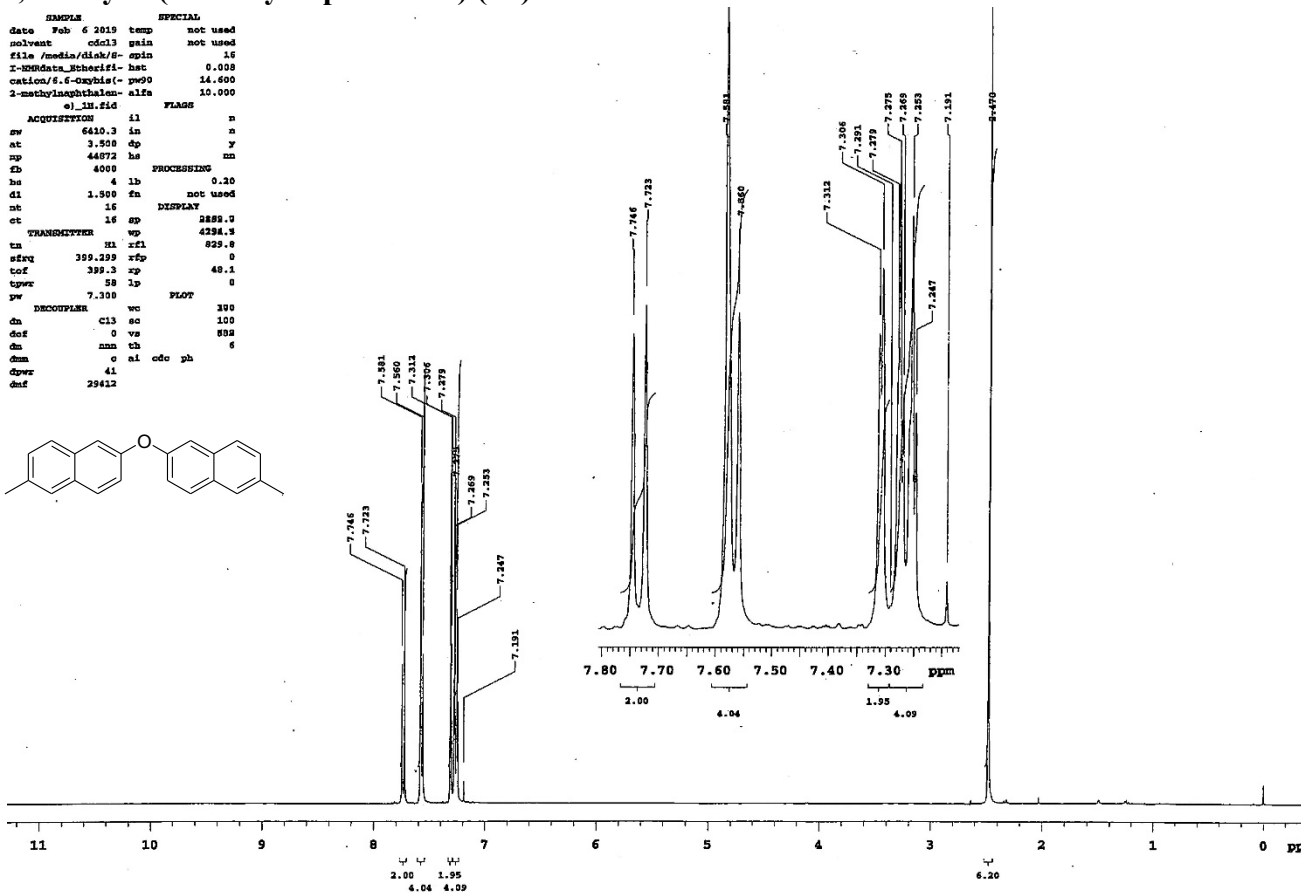
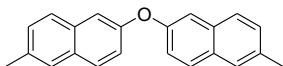


6,6'-Oxybis(2-methylnaphthalene) (2d) ¹H-NMR

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tof 399.3 sp 48.1
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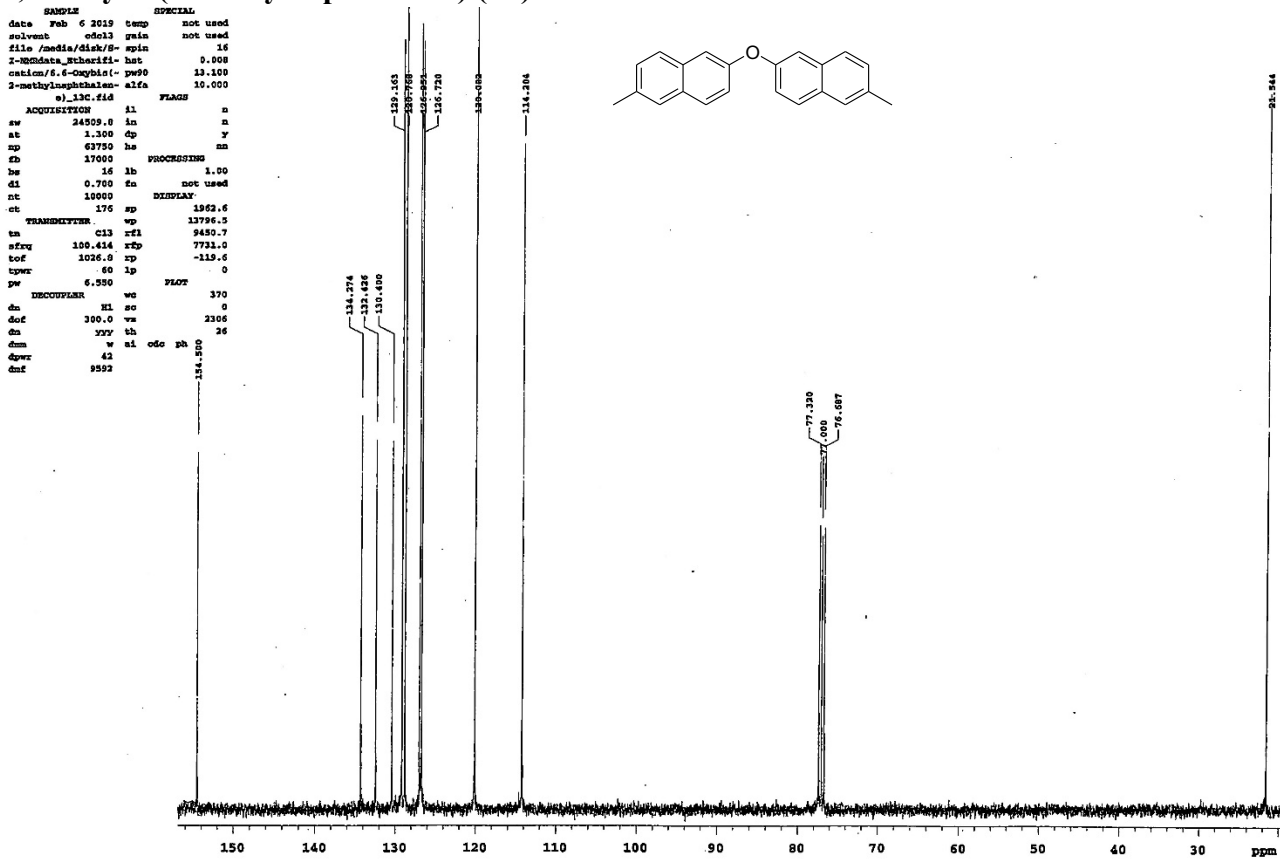
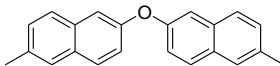


6,6'-Oxybis(2-methylnaphthalene) (2d) ¹³C-NMR

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e)_13c.fid

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tof 1026.8 sp -119.6
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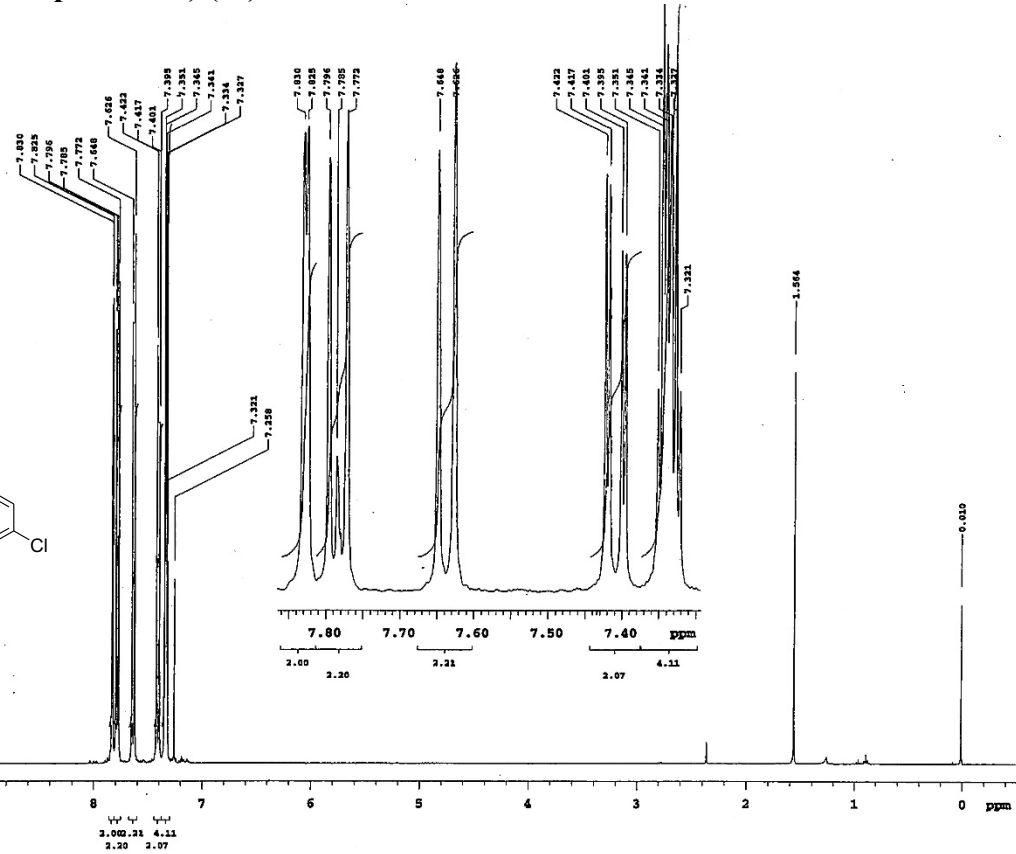
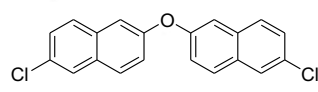


6,6'-Oxybis(2-chloronaphthalene) (2e) ¹H-NMR

exp4 Proton

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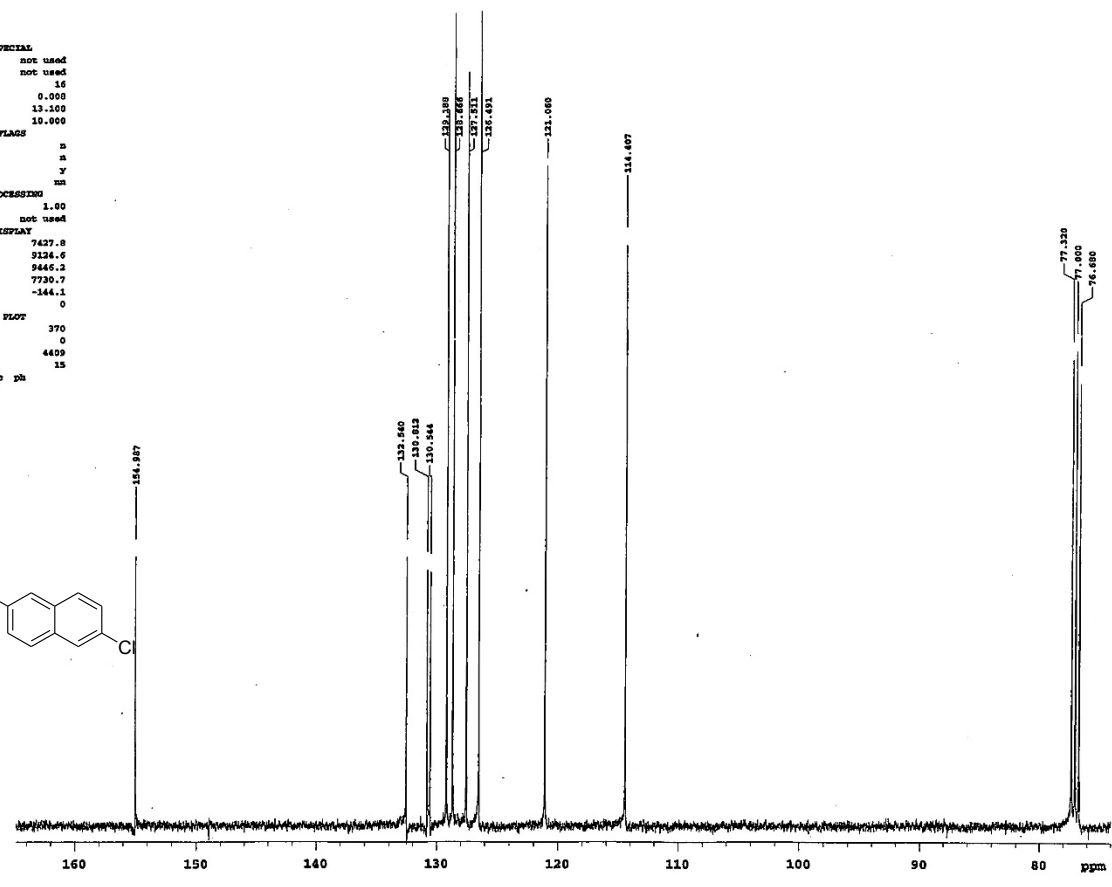
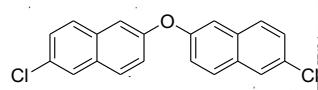


6,6'-Oxybis(2-chloronaphthalene) (2e) ¹³C-NMR

exp4 Carbon

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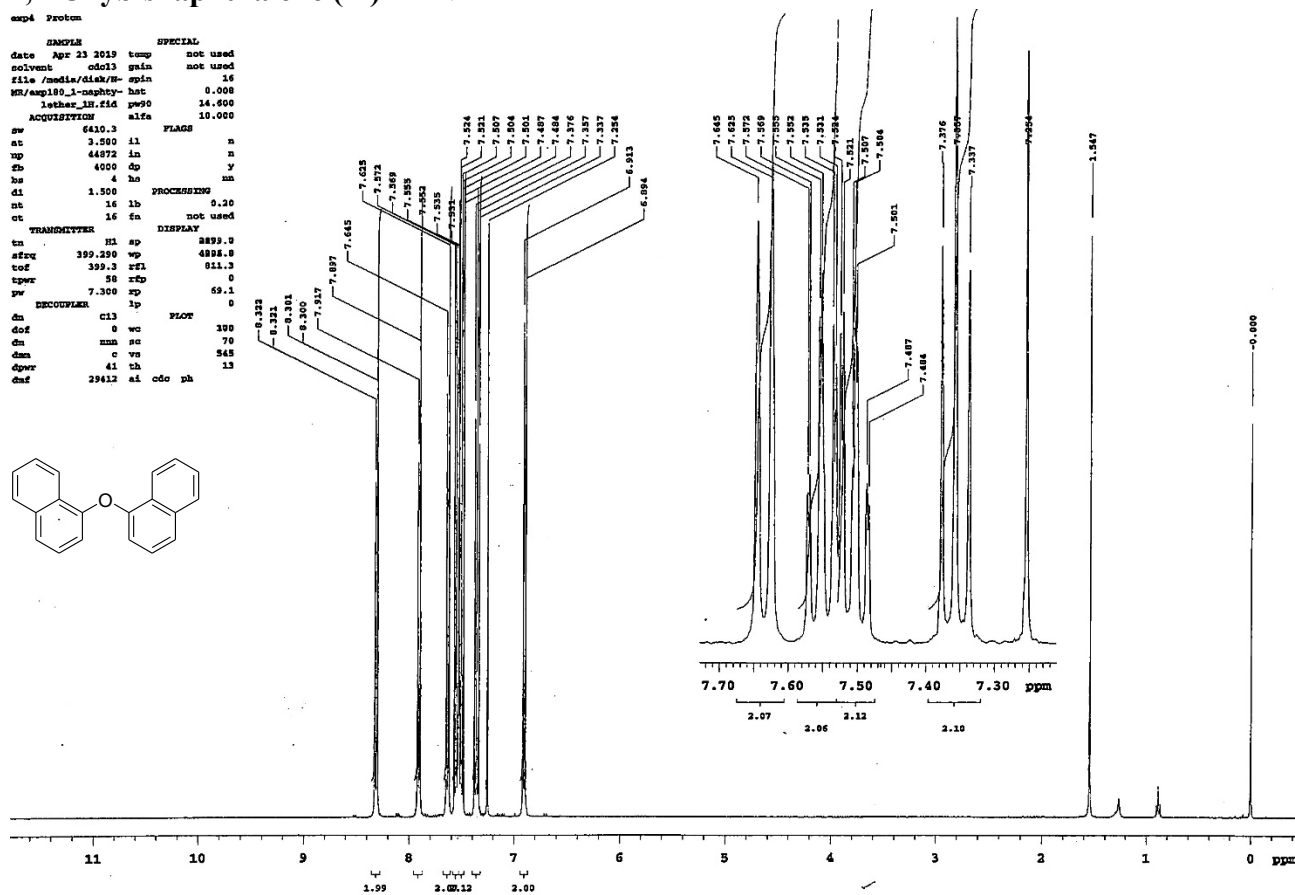
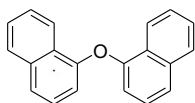
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e)_13c.fid          FLAGS
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1,1-Oxybisanthracene (2f) ¹H-NMR

exp4 Proton

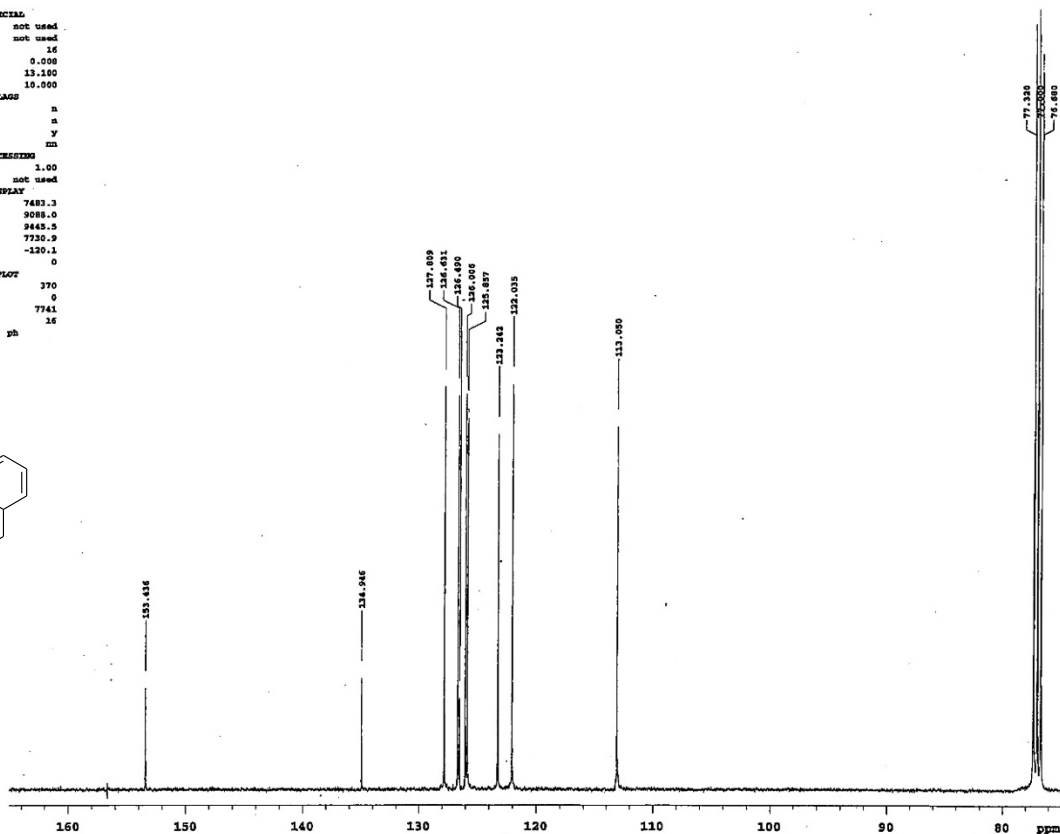
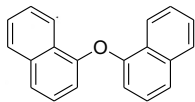
SAMPLE		SPECIAL	
date	Apr 23 2019	temp	not used
solvent	cdcl3	gain	not used
file	/media/disk/m- spin		16
MR/exp180_1-naphth-	hat		0.008
lotheser_1h.fid	pw90		14.900
ACQUISITION	aire		10.000
PROCCESSING		FLAGS	
sv	6410.3		
at	3.500	il	n
ap	44872	in	n
sb	4000	cp	y
hs	4	ho	nm
dl	1.500		
nt	16	fn	0.20
ot	16	fn	not used
TRANSMITTER		DISPLAY	
tn	399.290	sp	8899.0
afrc	399.290	wp	4884.0
toF	399.3	rfl	811.3
tpwr	50	rfp	0
pw	7.300	rp	69.1
DECOUPLER		PLOT	
dn	C13	wo	300
doF	0	wo	70
dm	c	vs	545
dpwr	41	th	13
dmf	29412	ai	odo ph



1,1-Oxybisanthracene (2f) ¹³C-NMR

exp4 Carbon

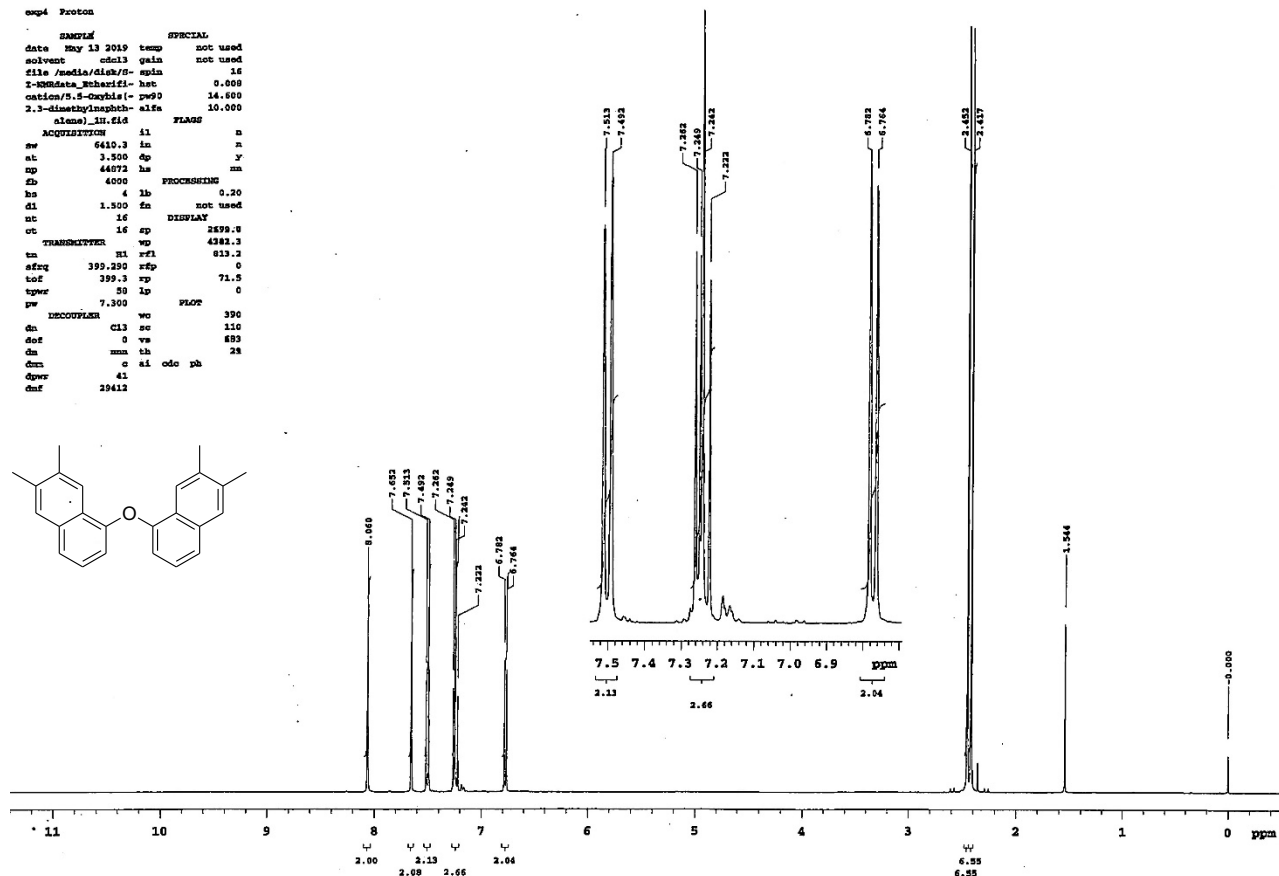
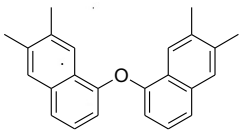
SAMPLE		SPECIAL	
date	Apr 23 2019	temp	not used
solvent	cdcl3	gain	not used
file	/media/disk/m- spin		16
MR/exp180_1-naphth-	hat		0.008
lotheser_13c.fid	pw90		15.100
ACQUISITION	aire		10.000
PROCCESSING		FLAGS	
sv	24509.8		
at	1.300	il	n
ap	83730	in	n
sb	17000	cp	y
hs	16	hs	nm
dl	0.700		
nt	28000	lb	1.00
ot	18088	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	7483.3
afrc	100.411	wp	9088.0
toF	1026.9	rfl	8445.5
tpwr	60	rfp	7730.9
pw	6.550	rp	-120.1
DECOUPLER		PLOT	
dn	1H	wo	0
doF	300.0	wo	370
dm	yyy	no	0
dm	w	vs	7141
dpwr	43	th	16
dmf	9593	ai	odo ph



5,5'-Oxybis(2,3-dimethylnaphthalene) (2g) ¹H-NMR

```

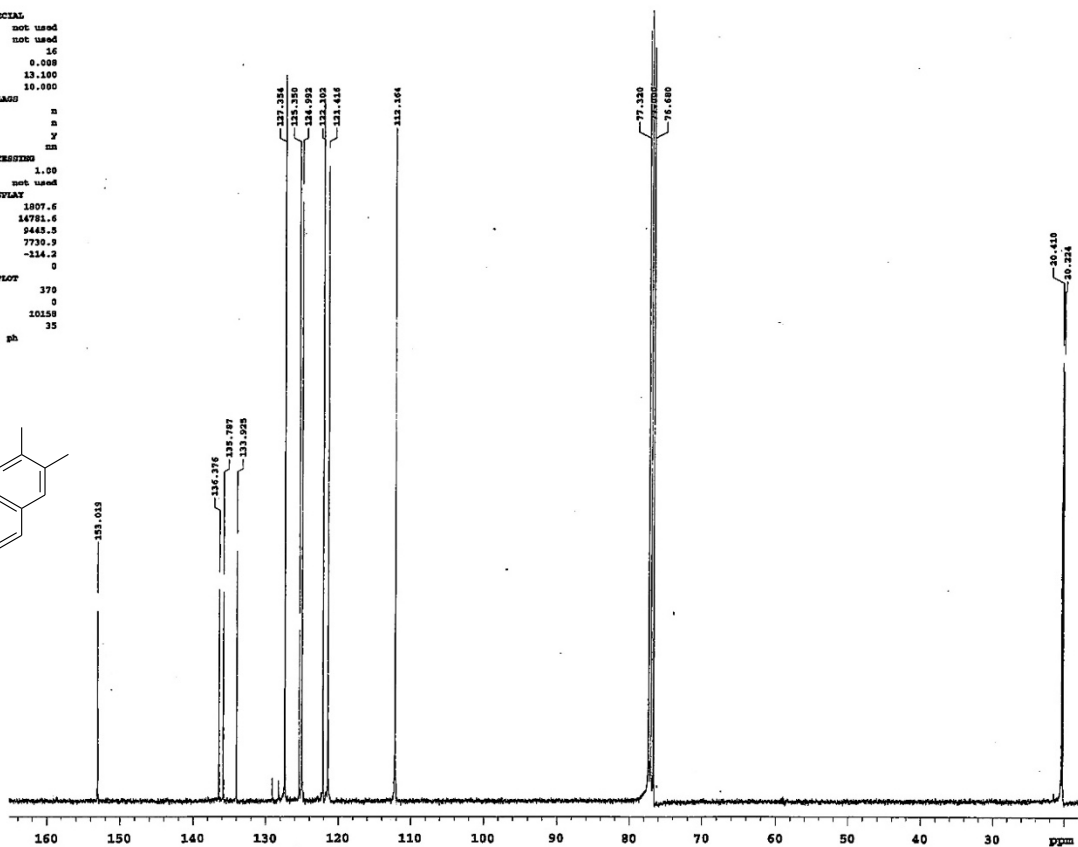
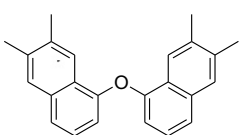
exp4 Proton
===== SPECIAL
date May 13 2019 temp not used
solvent cdcl3 gain not used
file /media/alex/8- spin 16
I-MWDATA_Rchariffi- hat 0.008
cation/5,5'-Oxybis(- pp90 14.608
2,3-dimethylnaphth- alfa 10.000
alene)_1H.fid
=====
ACQUISITION
sc 6410.3 in n
st 3.500 sp y
sp 44872 hs
zb 4000
hs 4 lb PROCESSING
di 1.500 fa not used
nt 16 DISPLAY 2599.8
ot 16 sp 4382.3
=====
TRANSMITTER
na HI rfl 813.2
afsq 399.290 rfp 0
tof 399.3 sp 71.5
tqwr 50 lp 0
pw 7.300
=====
RECOUPLER
dn HI sc 390
dof 0 ve 110
dm mm th 28
dmn c ai odc ph
dpr 41
dnt 29412
    
```



5,5'-Oxybis(2,3-dimethylnaphthalene) (2g) ¹³C-NMR

```

exp4 Carbon
===== SPECIAL
date May 13 2019 temp not used
solvent cdcl3 gain not used
file /media/alex/8- spin 16
I-MWDATA_Rchariffi- hat 0.008
cation/5,5'-Oxybis(- pp90 13.100
2,3-dimethylnaphth- alfa 10.000
alene)_13C.fid
=====
ACQUISITION
sc 24509.8 in n
st 1.380 sp y
sp 43750 hs
zb 17000
hs 16 lb PROCESSING
di 0.750 fa not used
nt 20000 DISPLAY 1807.5
ot 18024 sp 14781.6
=====
TRANSMITTER
na HI rfl 2445.3
afsq 100.411 rfp 7730.9
tof 1026.9 sp -114.2
tqwr 50 lp 0
pw 6.550
=====
RECOUPLER
dn HI sc 370
dof 300.0 ve 10158
dm mm th 35
dmn c ai odc ph
dpr 42
dnt 9592
    
```



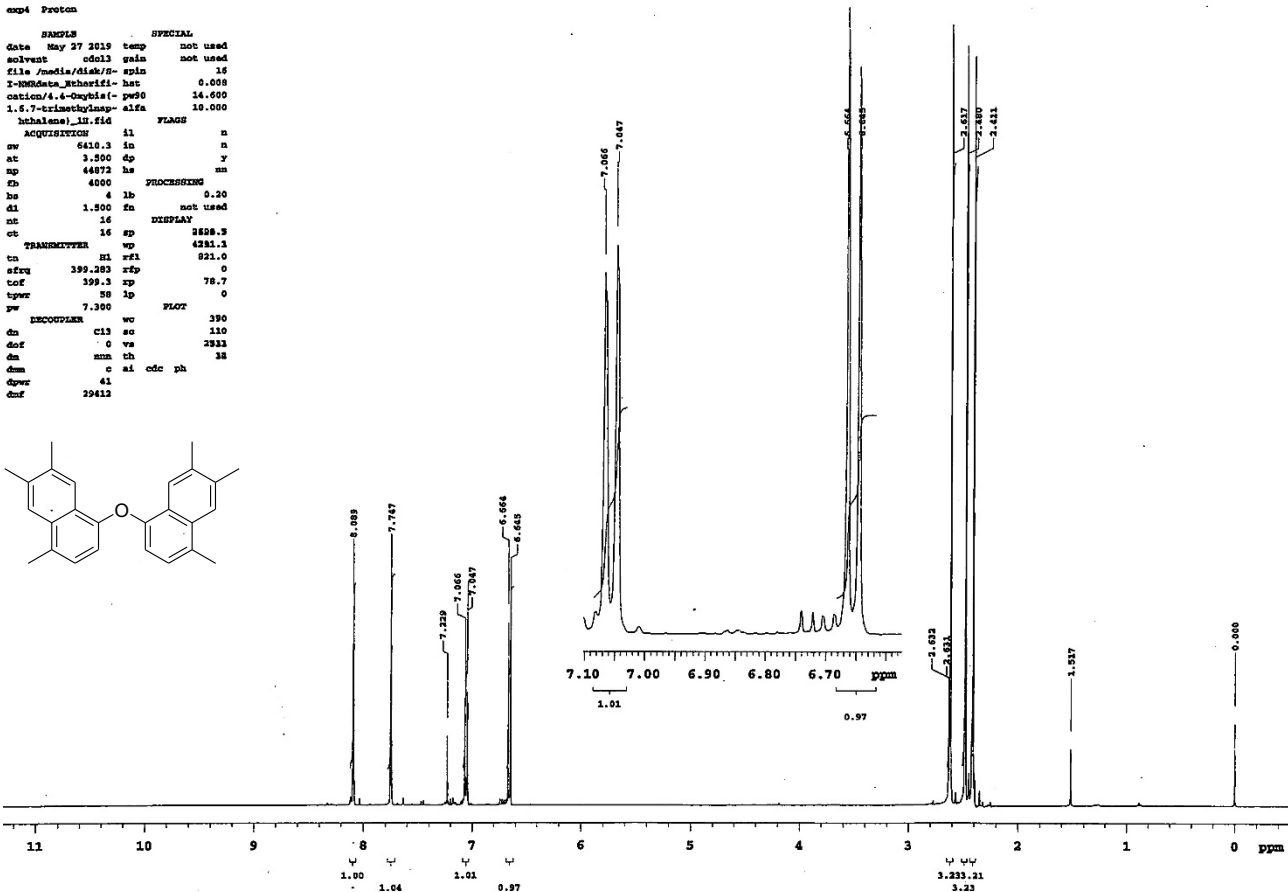
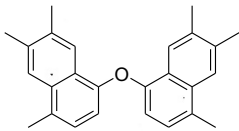
4,4'-Oxybis(1,6,7-trimethylnaphthalene) (2h) ¹H-NMR

exp4 Proton

```

SAMPLE          SPECIAL
date    May 27 2019 temp    not used
solvent  cdcl3 gain    not used
file  /media/disk/g- spin    16
I-NMRdata_Rhbarfi- hart    0.008
cation/4,4'-Oxybis(-pw90  14.000
1,6,7-trimethylnap- alfa  10.000
hthalene)_1h.fid          FLAGS

ACQUISITION    il    n
sv    6410.3 in    n
sc    1.500 sp    y
ap    44872 hs    nm
sh    4000          PROCESSING
bs    4 lb    0.20
dl    1.500 fm    not used
ss    16          DISPLAY
ct    16 sp    2888.3
TRANSMITTER    wp    4281.1
tn    hl rfl    821.0
sfreq  399.283 rfp    0
conf   399.5 xp    78.7
tpwr   58 lp    0
pw     7.300          PLOT
RECOVERER      wo    370
dn      hl    0
dof     300.0 vs    5695
dn      yyf th    30
dm      w    ai odc ph
dpr     43
dnt     9592
    
```



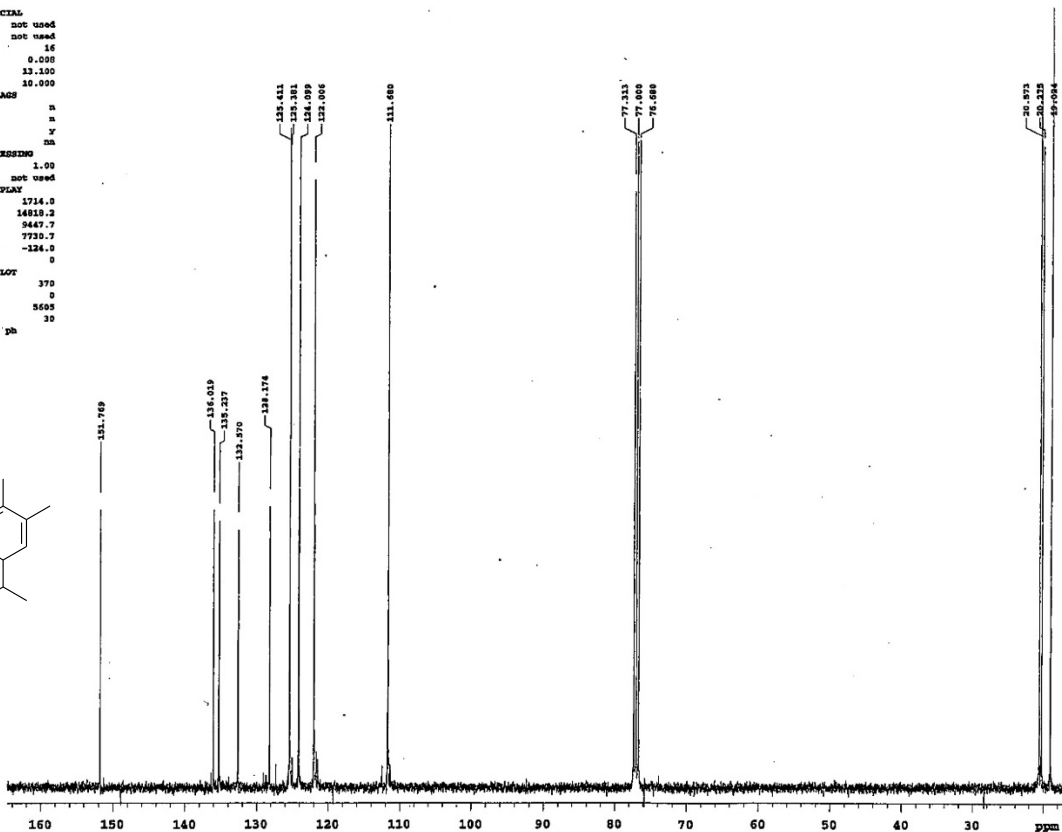
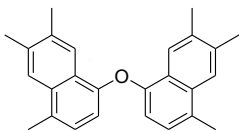
4,4'-Oxybis(1,6,7-trimethylnaphthalene) (2h) ¹³C-NMR

exp4 Carbon

```

SAMPLE          SPECIAL
date    May 27 2019 temp    not used
solvent  cdcl3 gain    not used
file  /media/disk/g- spin    16
I-NMRdata_Rhbarfi- hart    0.008
cation/4,4'-Oxybis(-pw90  13.100
1,6,7-trimethylnap- alfa  10.000
hthalene)_13c.fid          FLAGS

ACQUISITION    il    n
sv    24509.8 in    n
at    1.300 sp    y
ap    63750 hs    nm
sh    17000          PROCESSING
bs    16 lb    1.00
dl    0.700 fm    not used
nt    20000          DISPLAY
ct    1616 sp    1714.0
TRANSMITTER    wp    14819.2
tn    cl3 rfl    9447.7
sfreq  100.410 rfp    7730.3
conf   1026.0 rp    -124.0
tpwr   60 lp    0
pw     6.550          PLOT
RECOVERER      wo    370
dn      hl    0
dof     300.0 vs    5695
dn      yyf th    30
dm      w    ai odc ph
dpr     43
dnt     9592
    
```

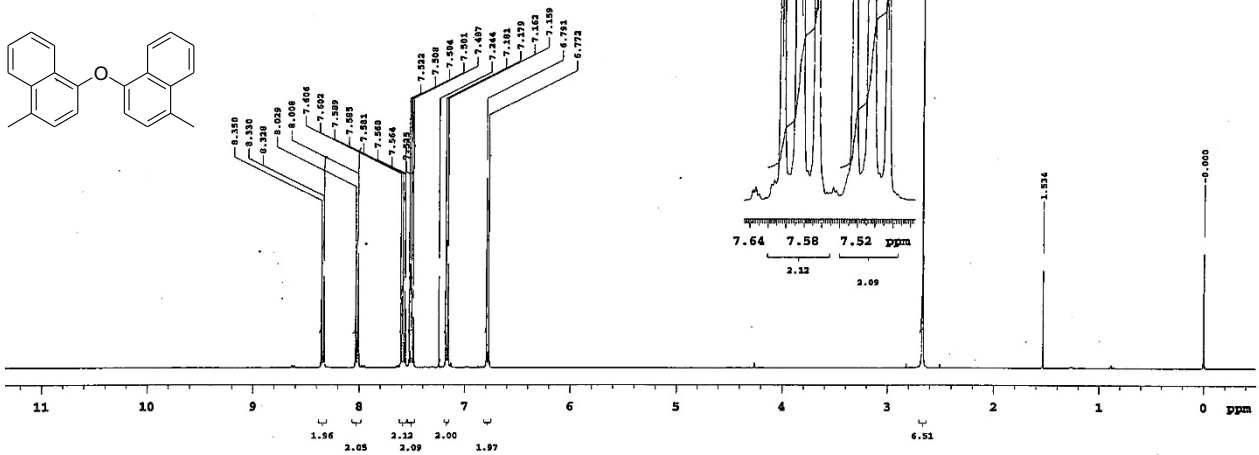
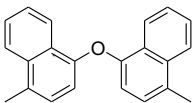


4,4'-Oxybis(1-methylnaphthalene) (2i) ¹H-NMR

exp4 Proton

```

SAMPLE          SPECIAL
date    May 24 2019    temp    not used
solvent  cdcl3         gain    not used
file    /media/disk/s- spin    16
X-NAMEdata_Rhbarf1- hat    0.000
cation/4,4'-Oxybis(= pw90    16.000
1-methylnaphthalen= a1fa    10.000
  e)_13c.fid          FLAGS
ACQUISITION      il      n
sw    6400.0       in      5
at    3.500       dq      y
mp    44872      hs      nm
fb    4000
hs    4           lb      0.20
dl    1.800       dn      not used
nt    16          DISPLAY
ot    16         sp      8899.3
TRANSMITTER      wp      4794.2
tu    H1         rf1     813.2
sfreq 399.283    rfp     0
tof    399.3     xp      72.7
tproc  30        ld      0
pw     7.300
RECOUPLER        wo      370
dn     C13       so      100
dof    0         vn      880
dm     mm        th      16
dmm    c         ai      ods  ph
dprc   41
dmf    28412
    
```

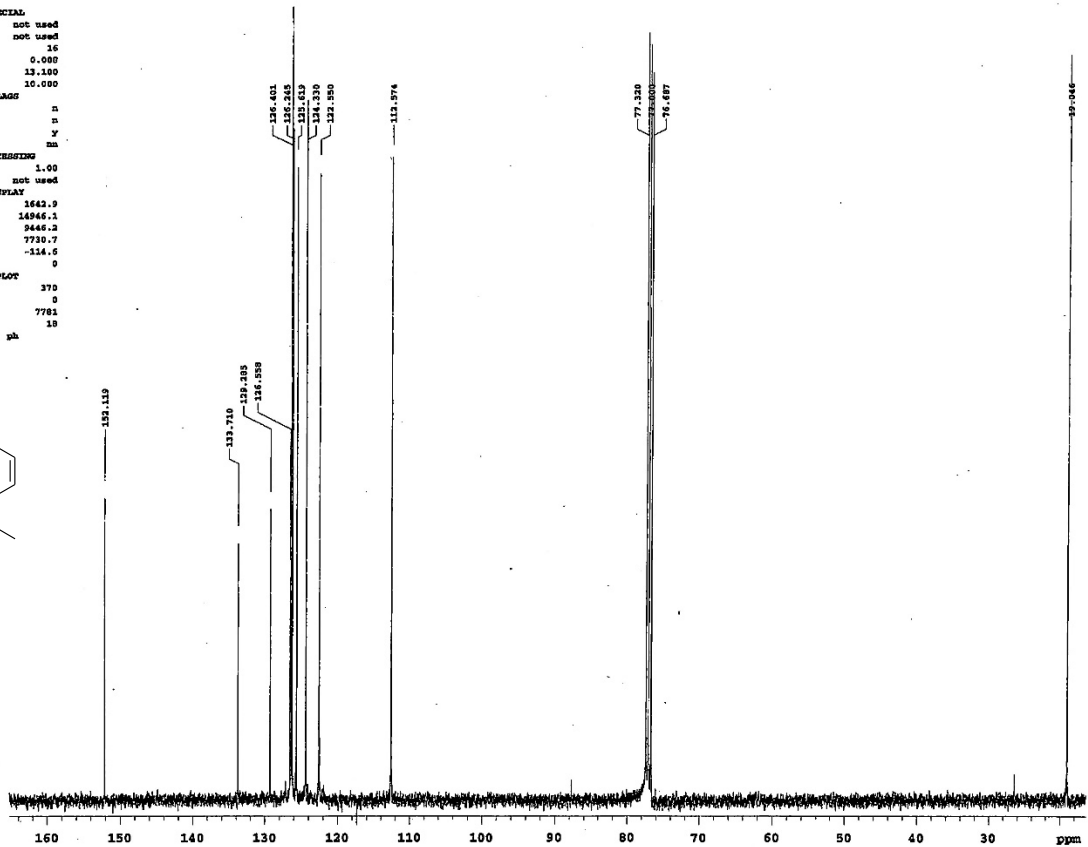
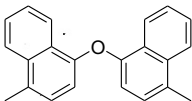


4,4'-Oxybis(1-methylnaphthalene) (2i) ¹³C-NMR

exp4 Carbon

```

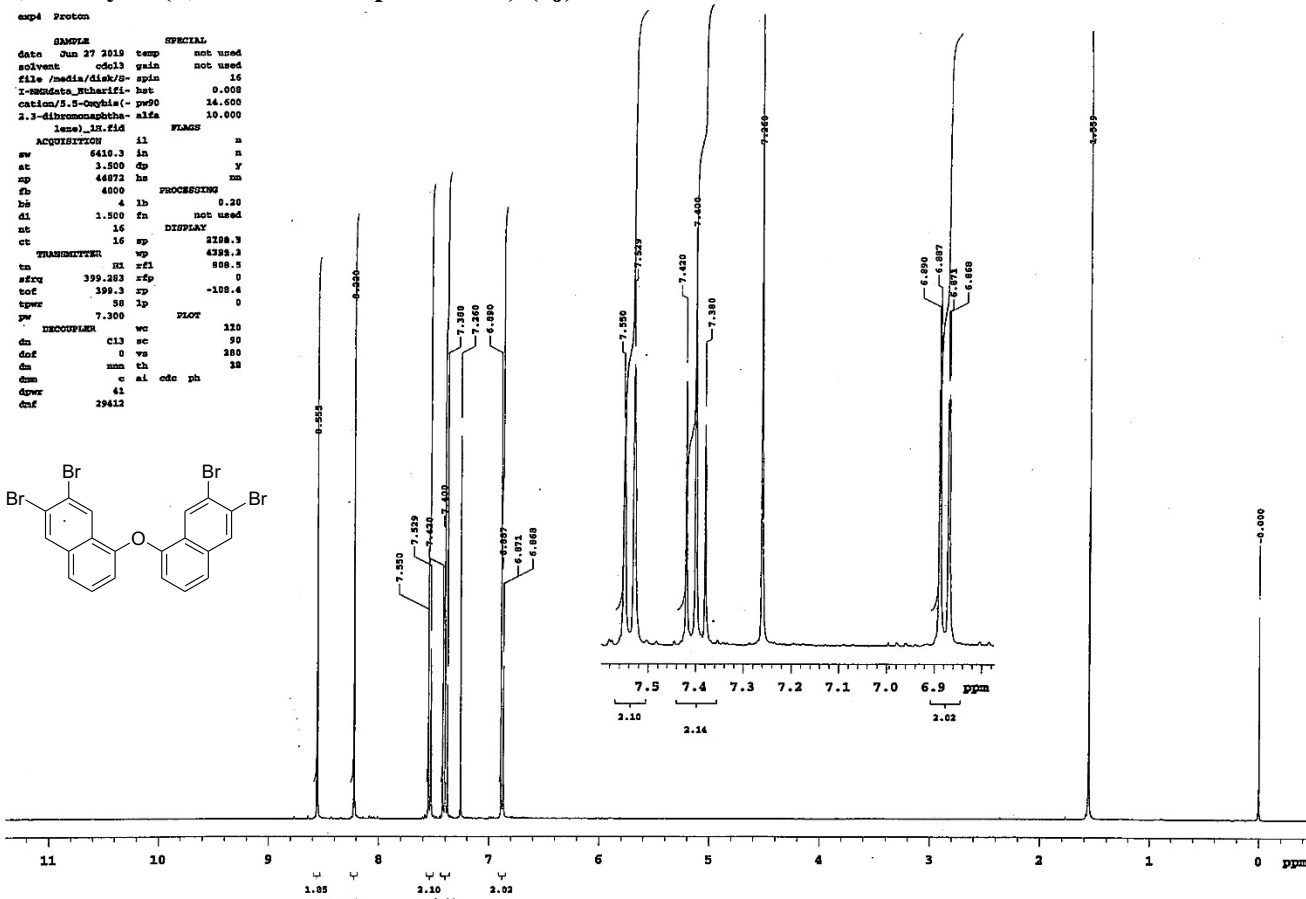
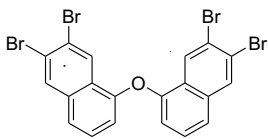
SAMPLE          SPECIAL
date    May 24 2019    temp    not used
solvent  cdcl3         gain    not used
file    /media/disk/s- spin    16
X-NAMEdata_Rhbarf1- hat    0.000
cation/4,4'-Oxybis(= pw90    12.100
1-methylnaphthalen= a1fa    10.000
  e)_13c.fid          FLAGS
ACQUISITION      il      n
sw    24500.0      in      n
at    1.300       dq      y
mp    43750      hs      nm
fb    17000
hs    16         lb      1.00
dl    0.700       dn      not used
nt    20000       DISPLAY
ot    1520       sp      1642.9
TRANSMITTER      wp      14946.1
tu    C13        rf1     9446.2
sfreq 100.610    rfp     7730.7
tof    1026.8    xp      -124.6
tproc  40        ld      0
pw     6.550
RECOUPLER        wo      370
dn     H1         so      0
dof    300.0     vn      7782
dm     Y2Y       th      18
dmm    w         ai      ods  ph
dprc   42
dmf    3552
    
```



5,5'-Oxybis(2,3-dibromonaphthalene) (2j) ¹H-NMR

```

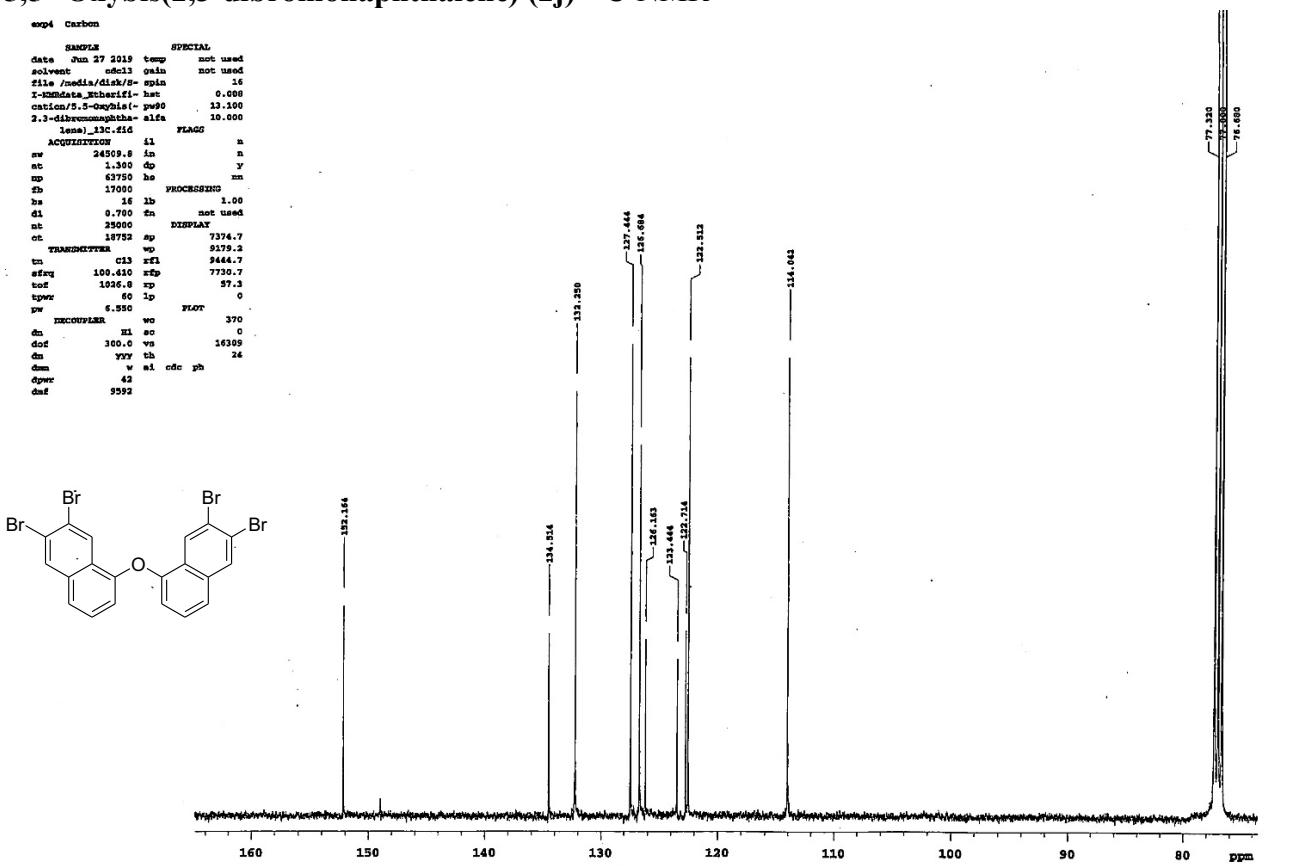
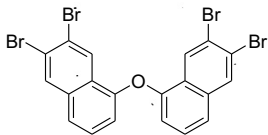
exp4 Proton
=====
SAMPLE          SPECIAL
date Jun 27 2019 temp not used
solvent cdcl3 gain not used
file /media/disk/ spin 16
T-NMRdata_Rbherfi- het 0.008
cation/5,5'-Oxybis(- pu90 14.600
2,3-dibromonaphtha- alfa 10.000
(less)_13c.fid
=====
ACQUISITION    il n
sw 6410.3 in n
st 3.500 sp y
sp 46072 hs nm
fs 4500 PROCESSING
hb 4 lb 0.20
dl 1.500 fn not used
nt 16 DISPLAY 2108.3
ct 16 sp 4398.2
=====
TRANSMITTER    wp 808.5
tn H1 xfl 0
sfreq 399.283 rfp 0
toF 399.3 sp -108.4
tprz 18 lp 0
pw 7.300 PLOT
=====
DECOUPLER      wc 120
dn C13 sc 90
dof 0 vs 180
dm th 18
dms c al cdc ph
dprz 41
dmf 29412
    
```



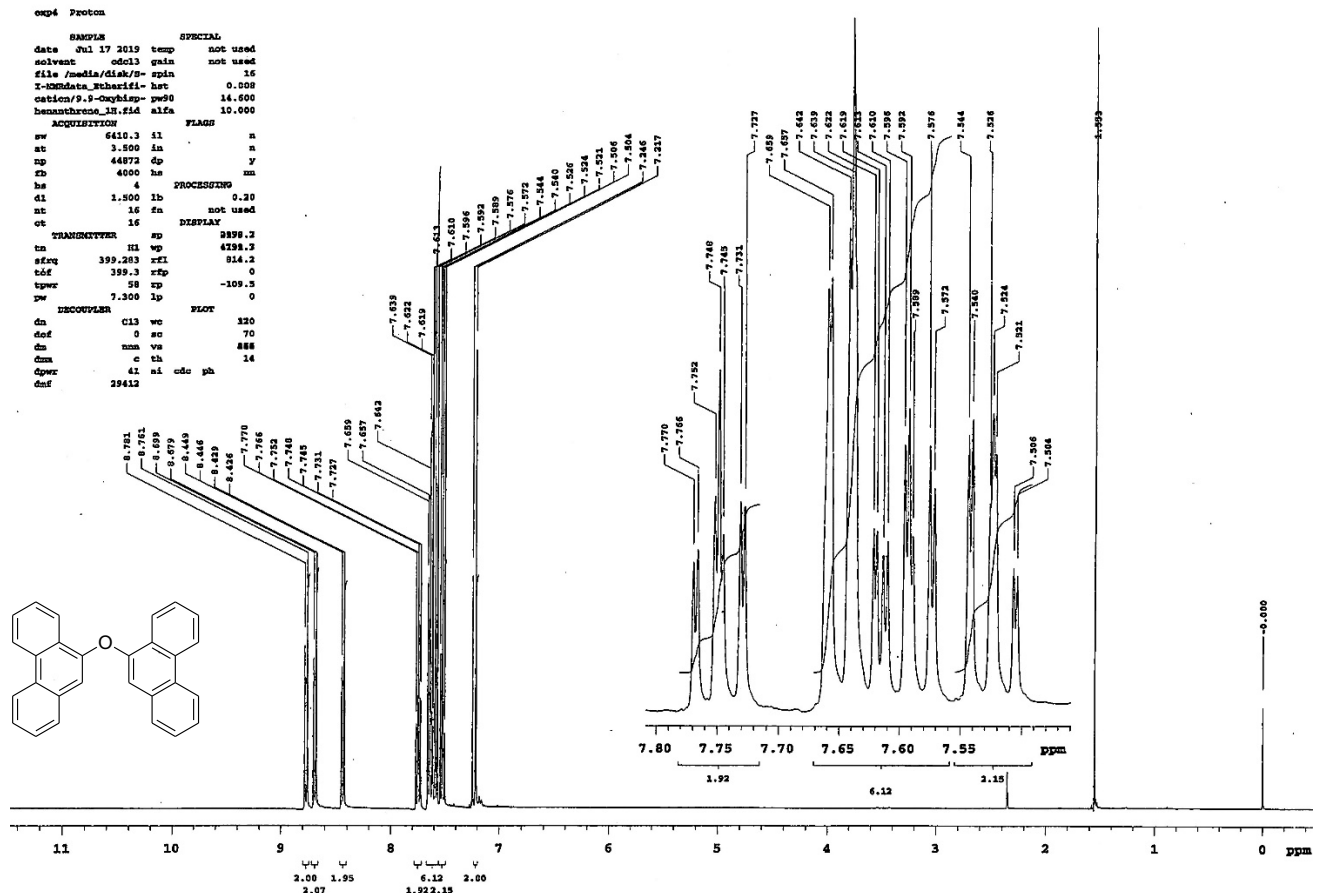
5,5'-Oxybis(2,3-dibromonaphthalene) (2j) ¹³C-NMR

```

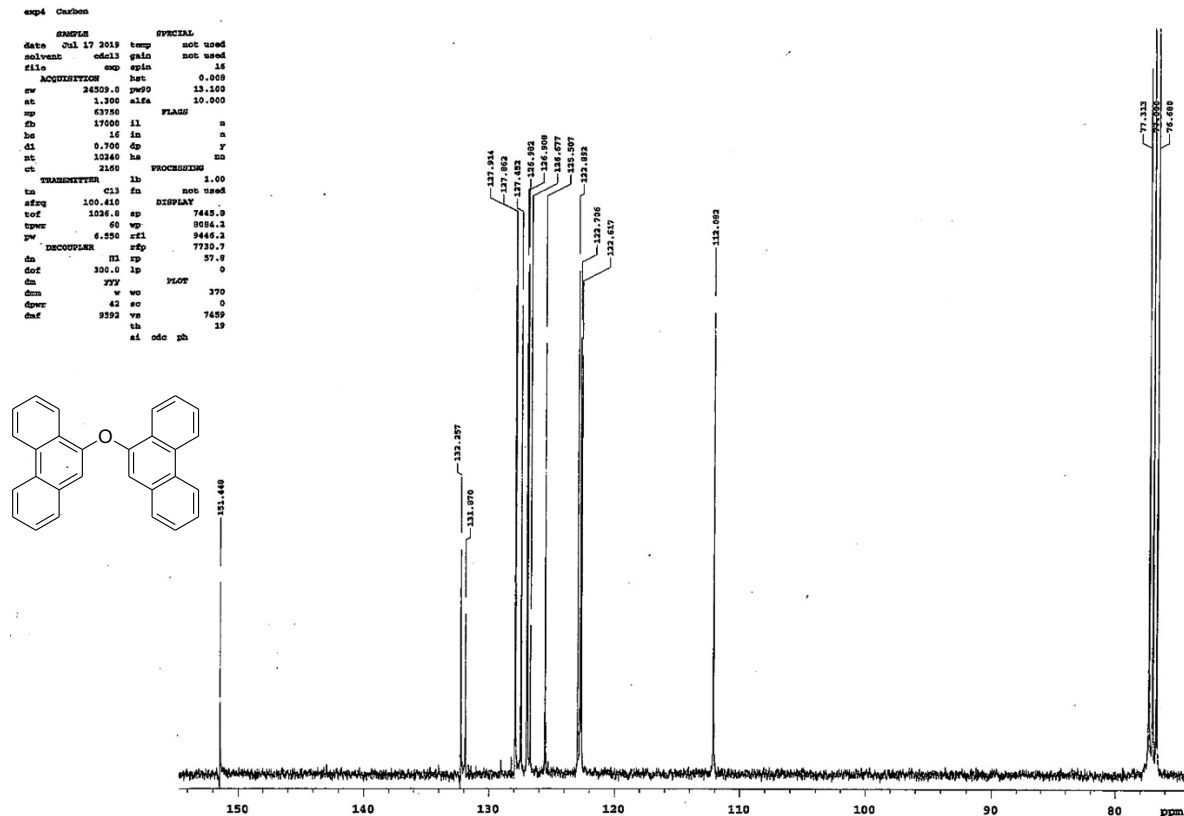
exp4 Carbon
=====
SAMPLE          SPECIAL
date Jun 27 2019 temp not used
solvent cdcl3 gain not used
file /media/disk/ spin 16
T-NMRdata_Rbherfi- het 0.008
cation/5,5'-Oxybis(- pu90 13.500
2,3-dibromonaphtha- alfa 10.000
(less)_13c.fid
=====
ACQUISITION    il n
sw 24509.8 in n
st 1.300 sp y
sp 83750 hs nm
fs 17000 PROCESSING
hb 16 lb 1.00
dl 0.700 fn not used
nt 25000 DISPLAY 7374.7
ct 18752 sp 8179.2
=====
TRANSMITTER    wp 8464.7
tn C13 xfl 9730.7
sfreq 100.440 rfp 57.3
toF 1026.8 sp 0
tprz 60 lp 0
pw 6.850 PLOT
=====
DECOUPLER      wc 370
dn H1 sc 0
dof 100.0 vs 16309
dm th 24
dms v al cdc ph
dprz 42
dmf 5592
    
```



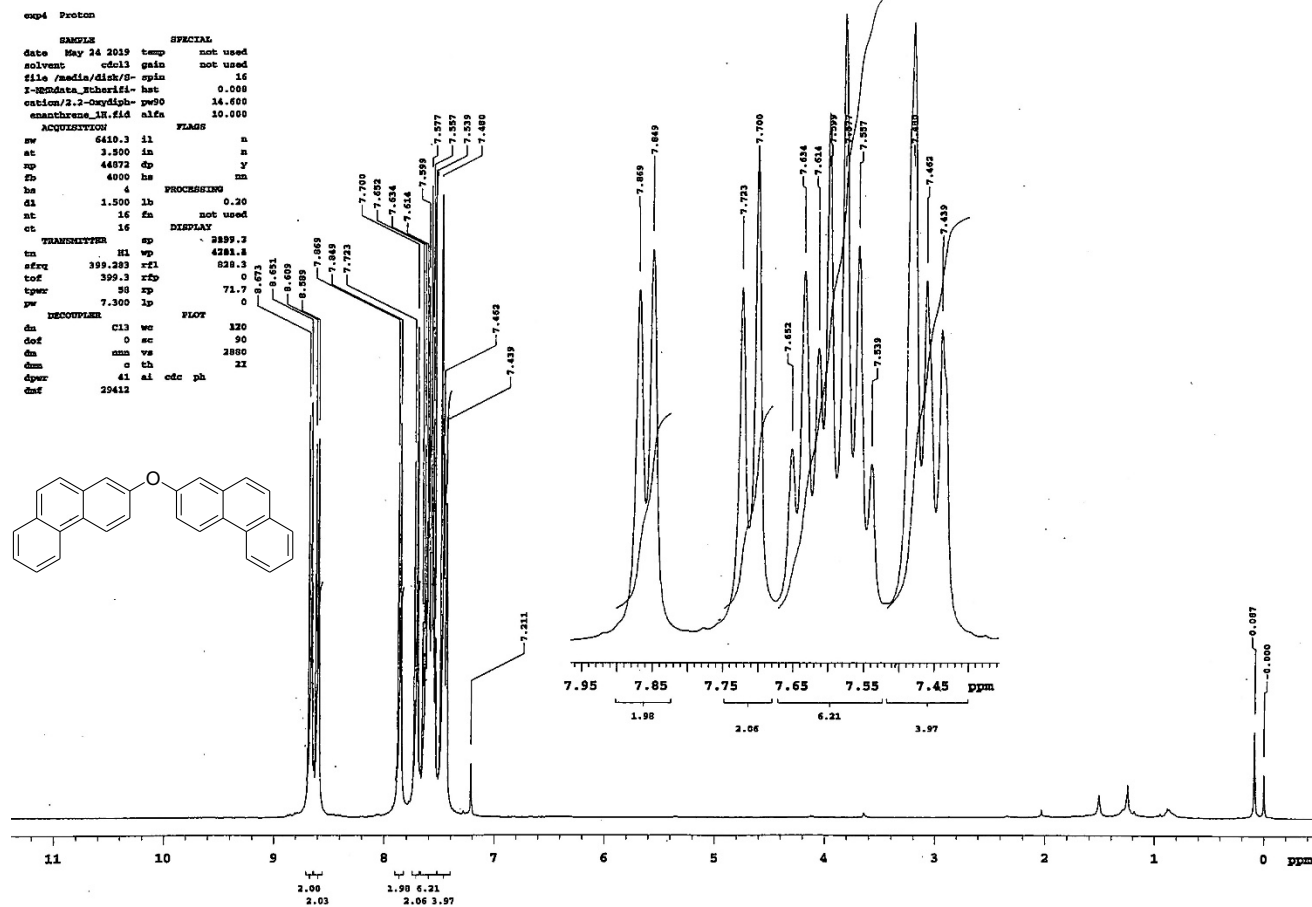
9,9'-Oxybisphenanthrene (2k) ¹H-NMR



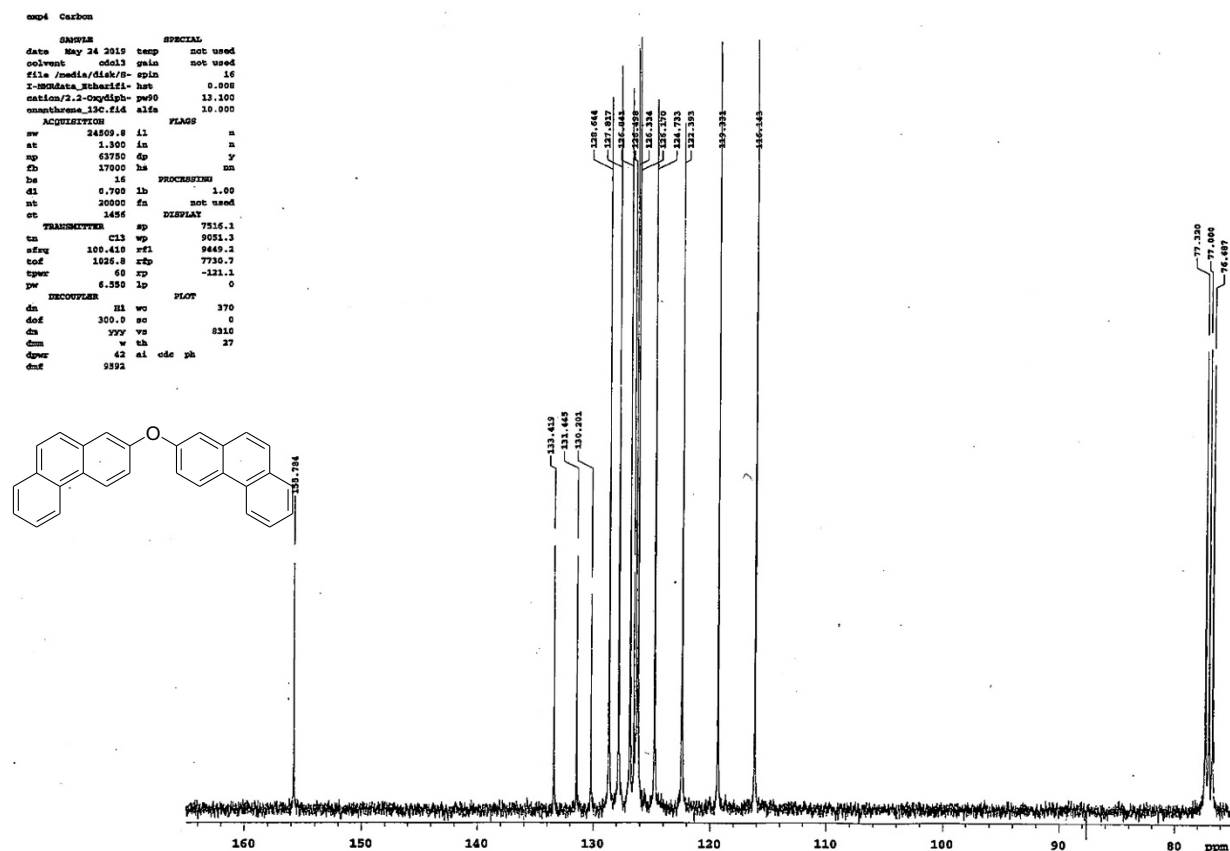
9,9'-Oxybisphenanthrene (2k) ¹³C-NMR



2,2'-Oxybisphenanthrene (2l) ¹H-NMR



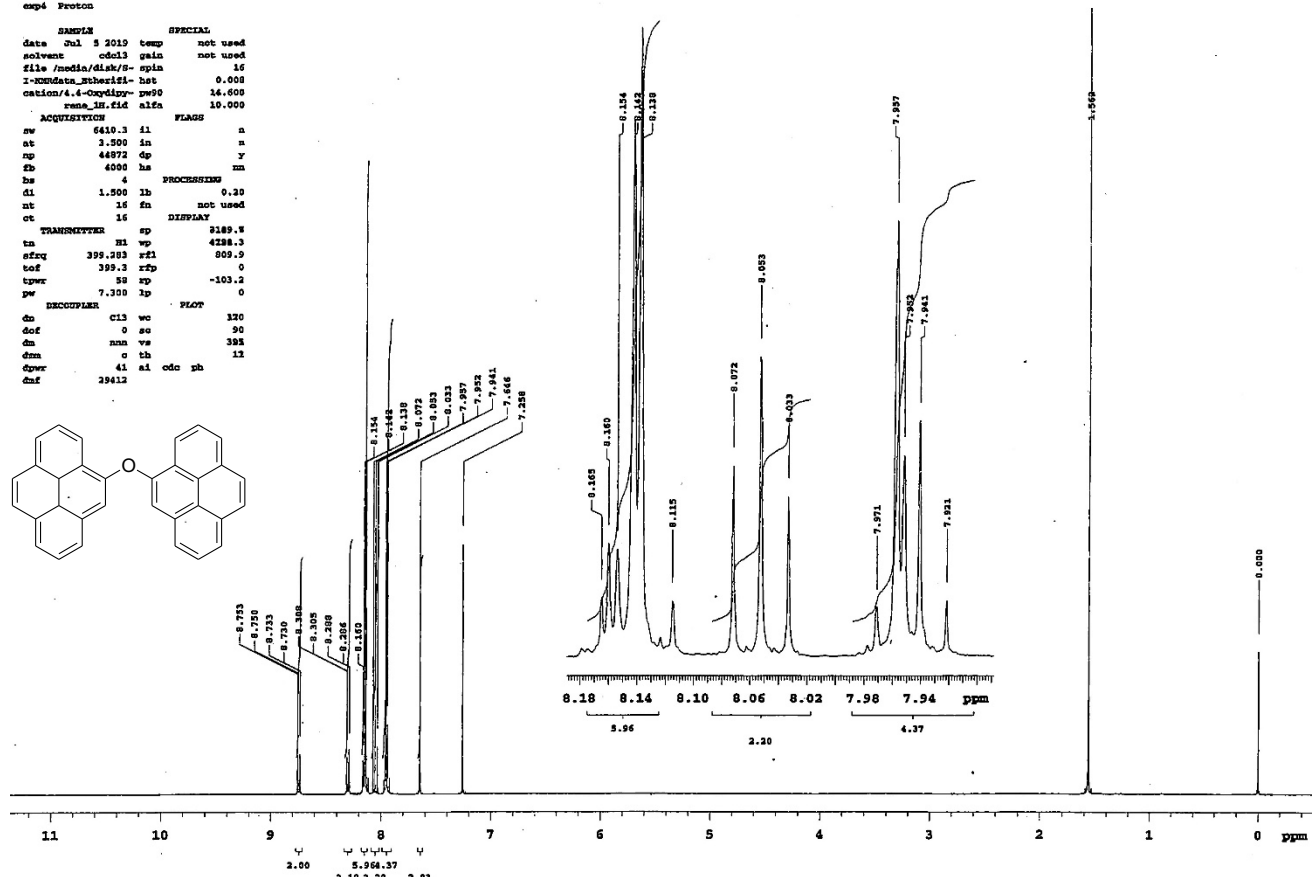
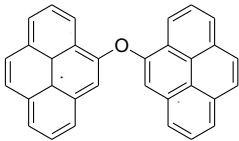
2,2'-Oxybisphenanthrene (2l) ¹³C-NMR



4,4'-Oxybispyrene (2m) ¹H-NMR

exp4 Proton

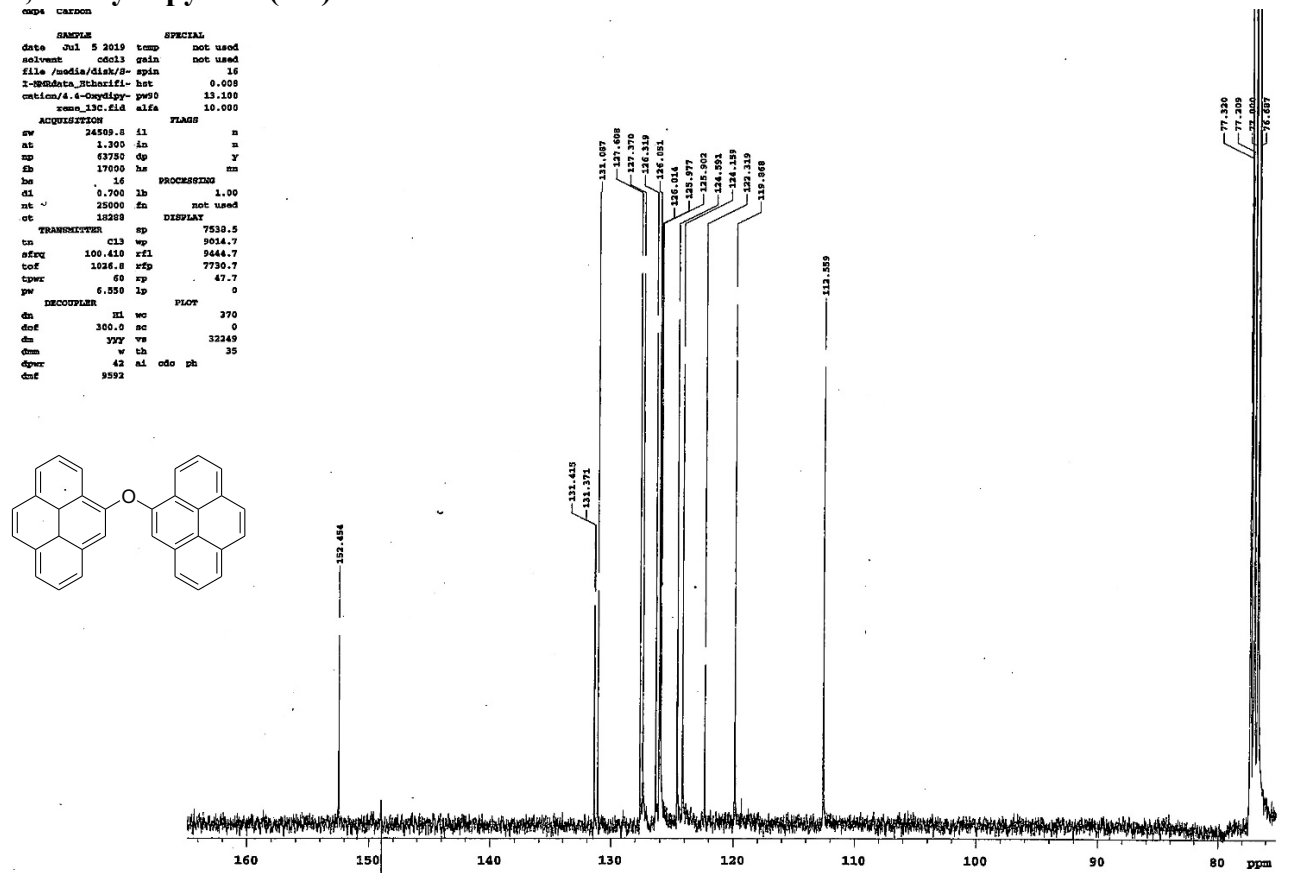
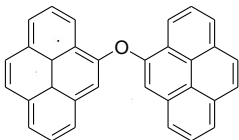
SAMPLE		SPECIAL	
date	Jul 5 2019	temp	not used
solvent	cdcl3	gain	not used
file	/media/disk/8- spin		16
2-nmdata_chnrifi-	hut		0.008
cation/4,4'-Oxybipy-	pw50		14.600
name_in.fid	alfa		10.000
ACQUISITION		FLAGS	
sv	6410.3	fl	n
at	1.500	in	n
sp	44872	dp	y
sb	4000	hs	nm
bs	4		
di	1.500	lb	0.30
nt	16	fn	not used
ct	16	display	
TRANSMITTER		SP	
tn	81	wp	3189.5
afreq	399.213	rfl	4288.3
tof	399.3	rfd	0
tpwr	58	sp	-103.2
pw	7.300	lp	0
DECOUPLER		F10F	
dn	C13	wo	320
dcf		so	90
dm	nn	vs	398
dmm	o	th	12
dqwr	41	ni	cdc
dmf	28412	ph	



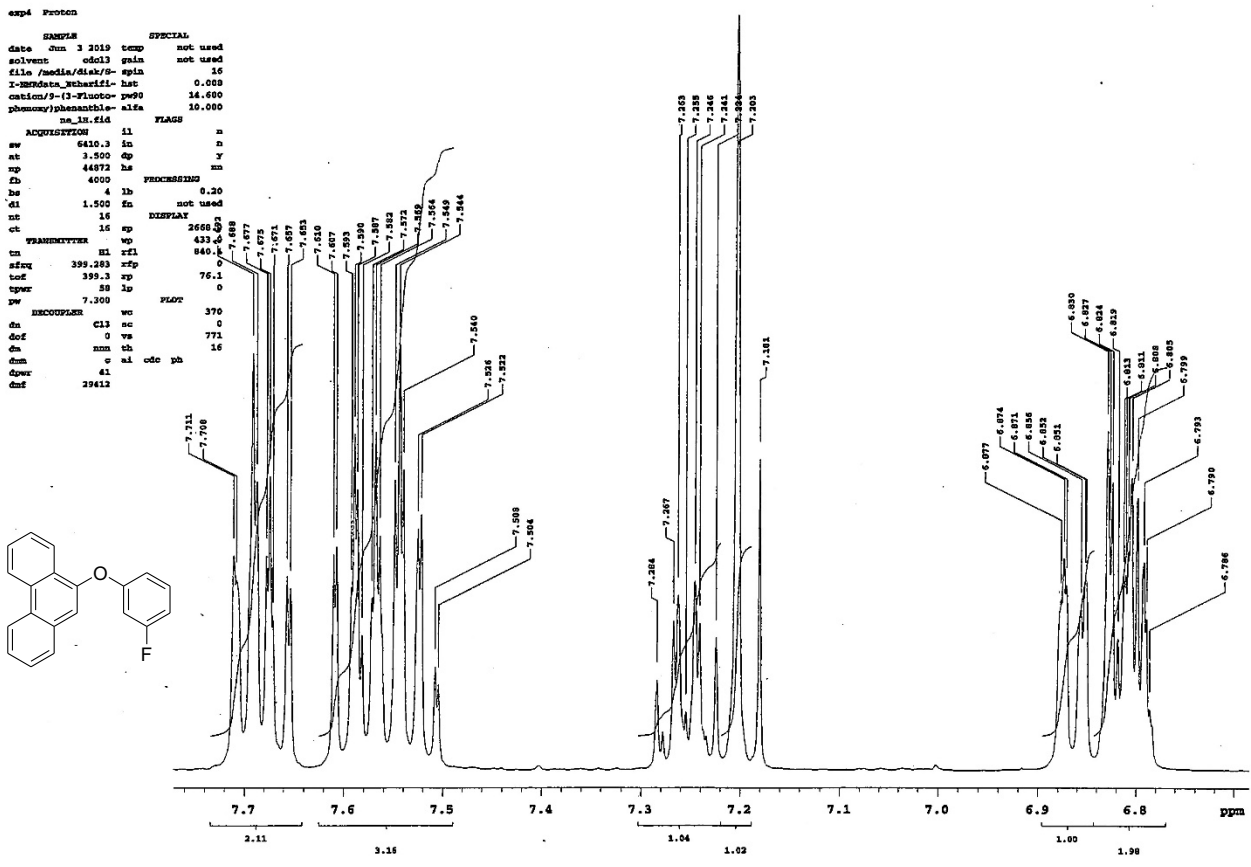
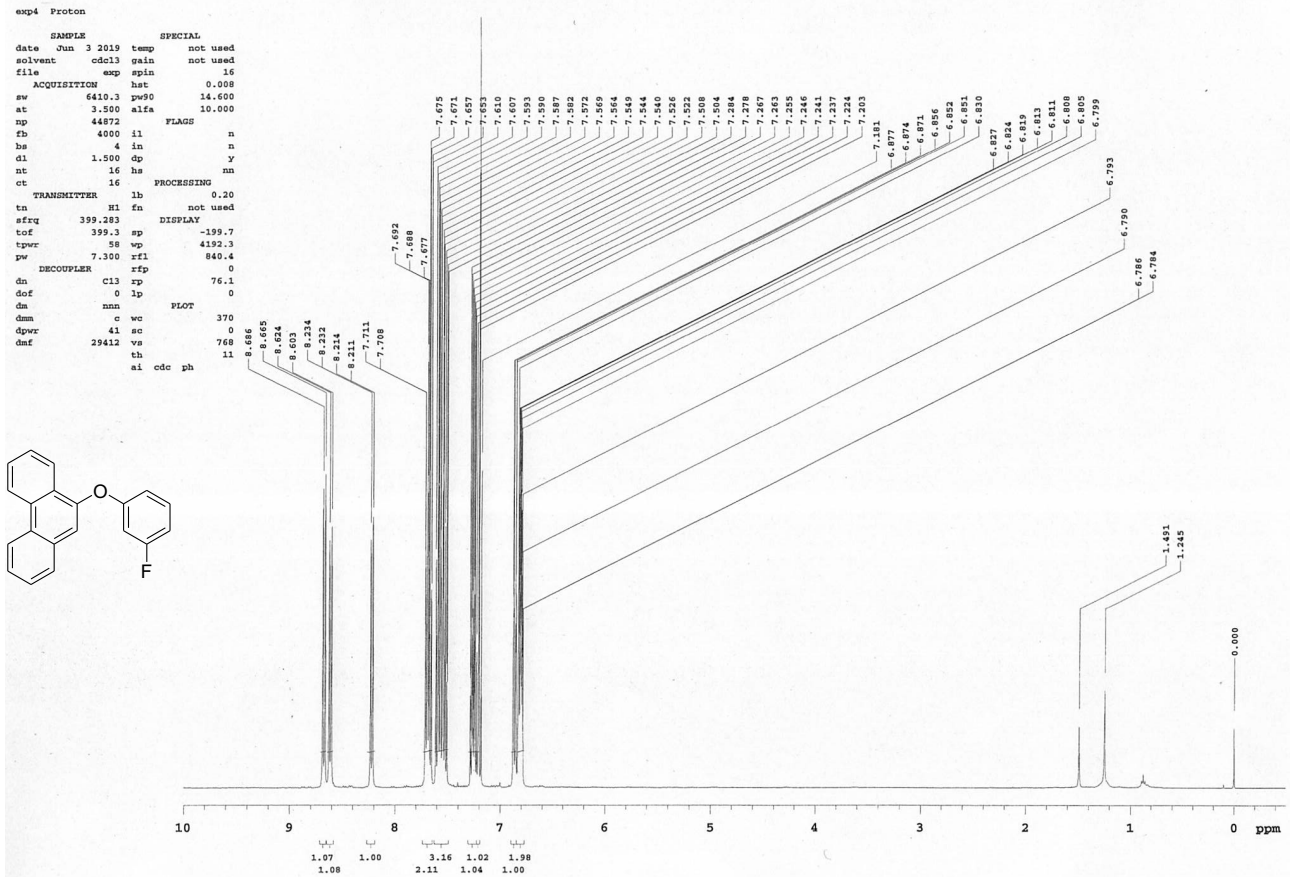
4,4'-Oxybispyrene (2m) ¹³C-NMR

exp4 Carbon

SAMPLE		SPECIAL	
date	Jul 5 2019	temp	not used
solvent	cdcl3	gain	not used
file	/media/disk/8- spin		16
2-nmdata_chnrifi-	hut		0.008
cation/4,4'-Oxybipy-	pw50		13.100
name_13c.fid	alfa		10.000
ACQUISITION		FLAGS	
sv	24509.8	fl	n
at	1.300	in	n
sp	63750	dp	y
sb	17000	hs	nm
bs	16		
di	0.700	lb	1.00
nt	25000	fn	not used
ct	18289	display	
TRANSMITTER		SP	
tn	C13	wp	7530.5
afreq	100.610	rfl	9444.7
tof	1028.8	rfd	7730.7
tpwr	60	sp	47.7
pw	6.850	lp	0
DECOUPLER		F10F	
dn	H1	wo	270
dcf	100.0	so	9
dm	YY	vs	32249
dmm	w	th	35
dqwr	42	ni	cdc
dmf	3592	ph	



9-(3-Fluorophenoxy)phenanthrene (4a) ¹H-NMR



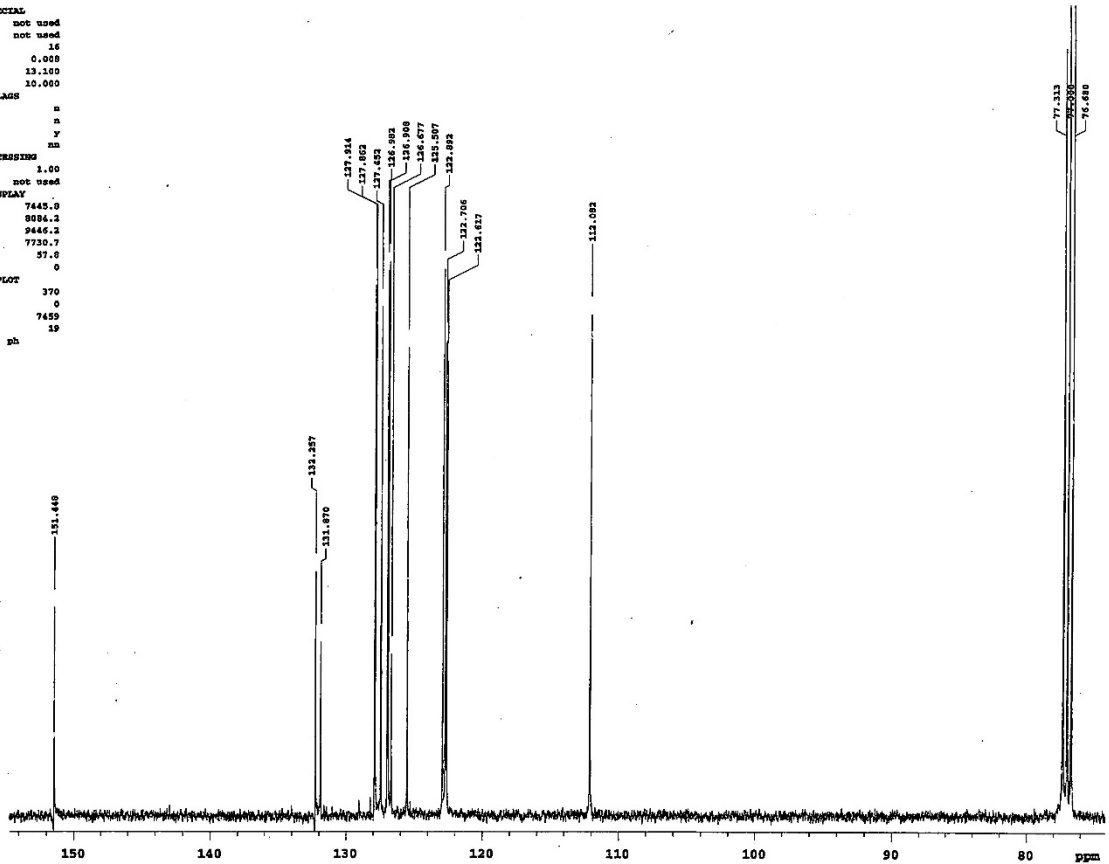
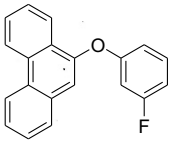
9-(3-Fluorophenoxy)phenanthrene (4a) ¹³C-NMR

exp4 Carbon

```

SAMPLE          SPECIAL
date Jul 17 2019 temp not used
solvent cdcl3 gain not used
file          exp spin 16
ACQUISITION   has 0.000
nu 24509.8 pw90 13.100
at 1.300 alfa 10.000
mp 63750 FLAGS
ch 17000 il n
hs 16 in n
dl 0.700 dp y
nt 10240 hs mn
ct 2100

TRANSMITTER lb PROCESSING 1.00
tn C13 fn not used
sfrq 100.610 DISPLAY
tof 1026.8 sp 7445.0
tprz 50 wp 8024.2
pw 6.550 rfl 9446.2
DECOUPLER rfp 7730.7
dn H1 xp 57.0
dof 100.0 lp 0
dm YYY PLOT
dms w wc 370
dprz 42 sc 0
dmf 9592 vs 7450
al cdc ph 19
  
```

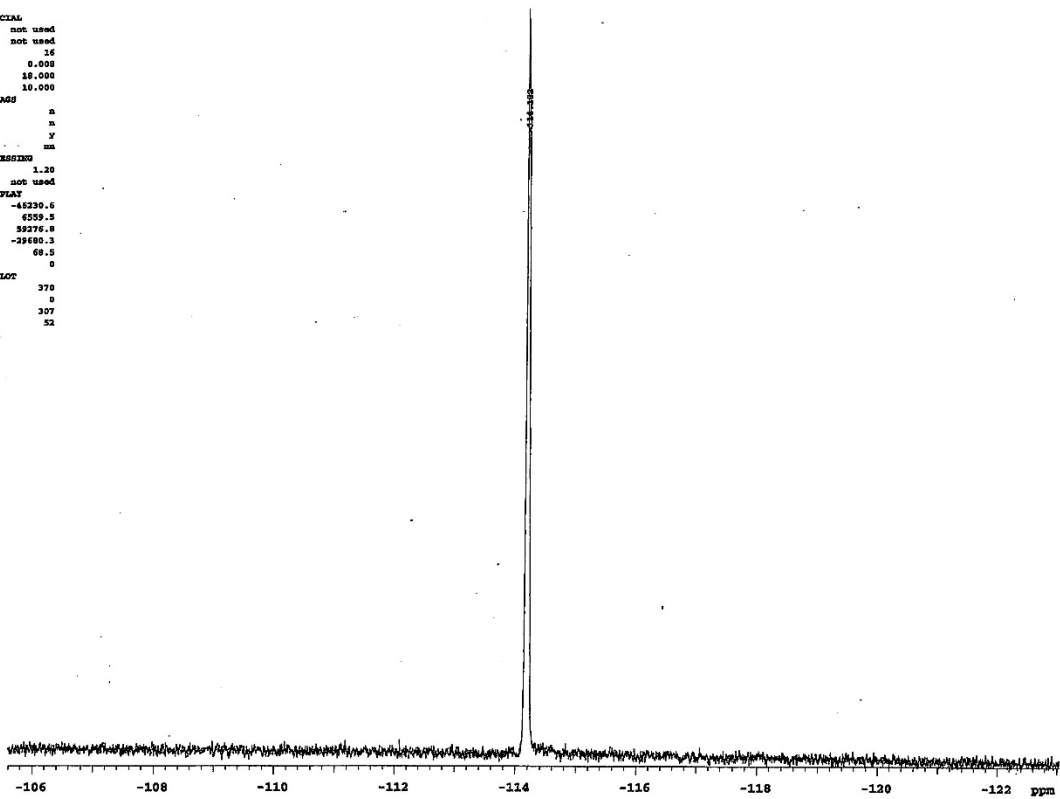
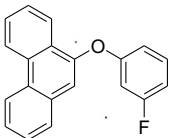


9-(3-Fluorophenoxy)phenanthrene (4a) ¹⁹F-NMR

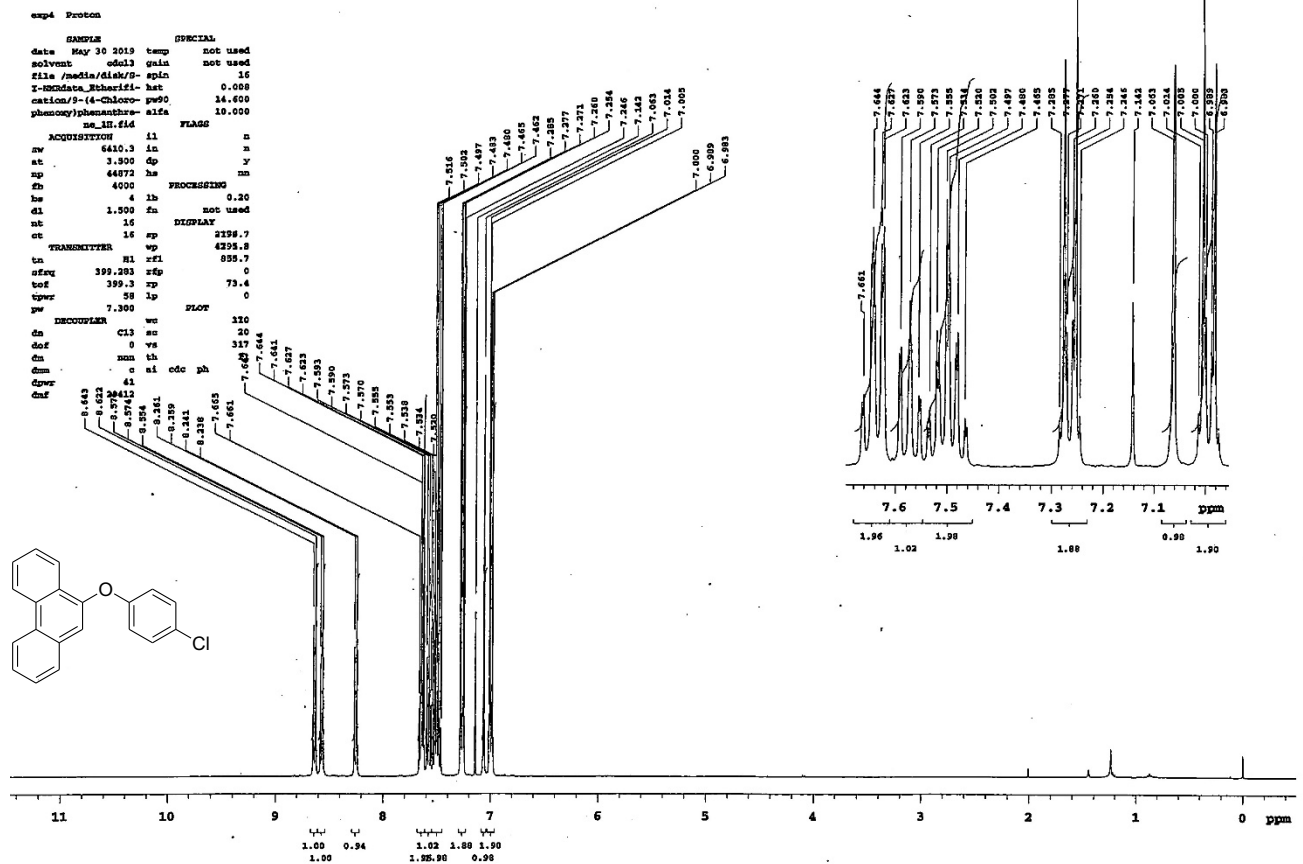
exp4 Fluorine

```

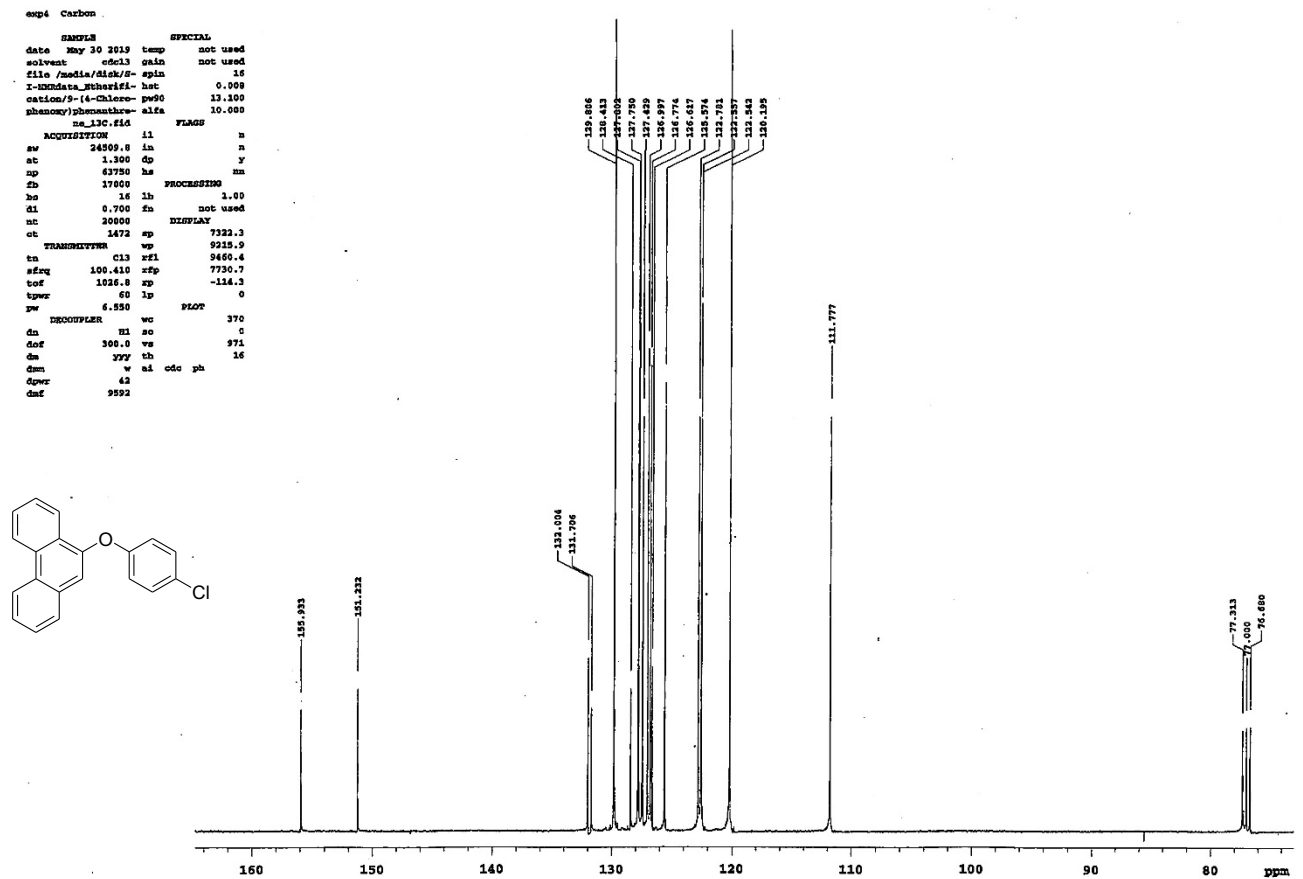
SAMPLE          SPECIAL
date Jul 17 2019 temp not used
solvent cdcl3 gain not used
file /media/disk/19- spin 16
X-MODEData_Identifier has 0.000
cation/3-(3-Fluoro- pw90 10.000
phenoxy)phenanthre- alfa 10.000
no_19F_fid FLAGS
ACQUISITION il n
nu 119047.6 in n
at 0.600 dp y
mp 142850 hs mn
ch 51800 PROCESSING 1.20
hs 4 lb not used
dl 4.400 dn
nt 16 DISPLAY
ct 16 sp -45239.6
TRANSMITTER wp 6559.2
tn F19 rfl 59276.0
sfrq 378.671 rfp -29480.3
tof 13604.2 sp 69.3
tprz 61 lp 0
pw 6.000 PLOT
dn H1 xc 370
dof 0 vs 307
dm mma th 52
dms 0 al ph
dprz 41
dmf 29413
  
```



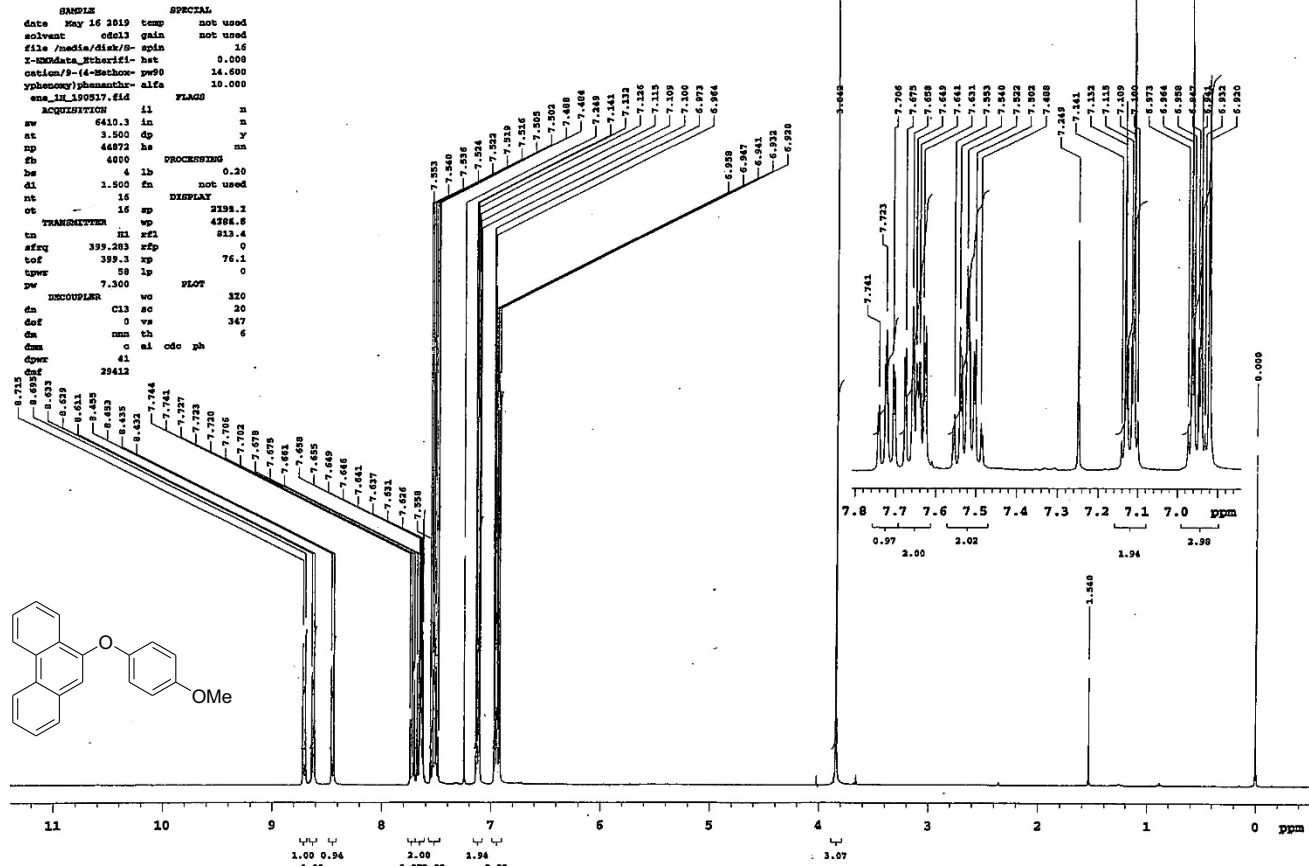
9-(4-Chlorophenoxy)phenanthrene (4b) ¹H-NMR



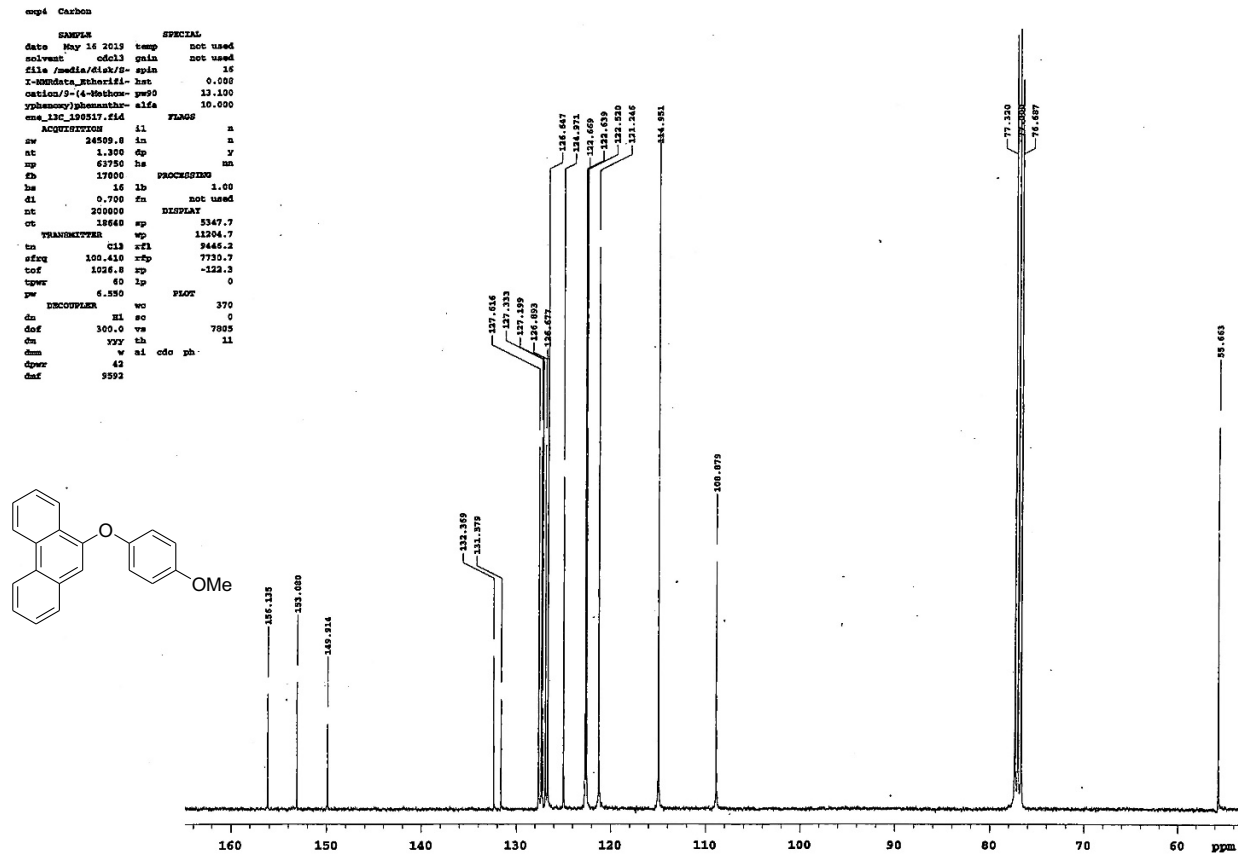
9-(4-Chlorophenoxy)phenanthrene (4b) ¹³C-NMR



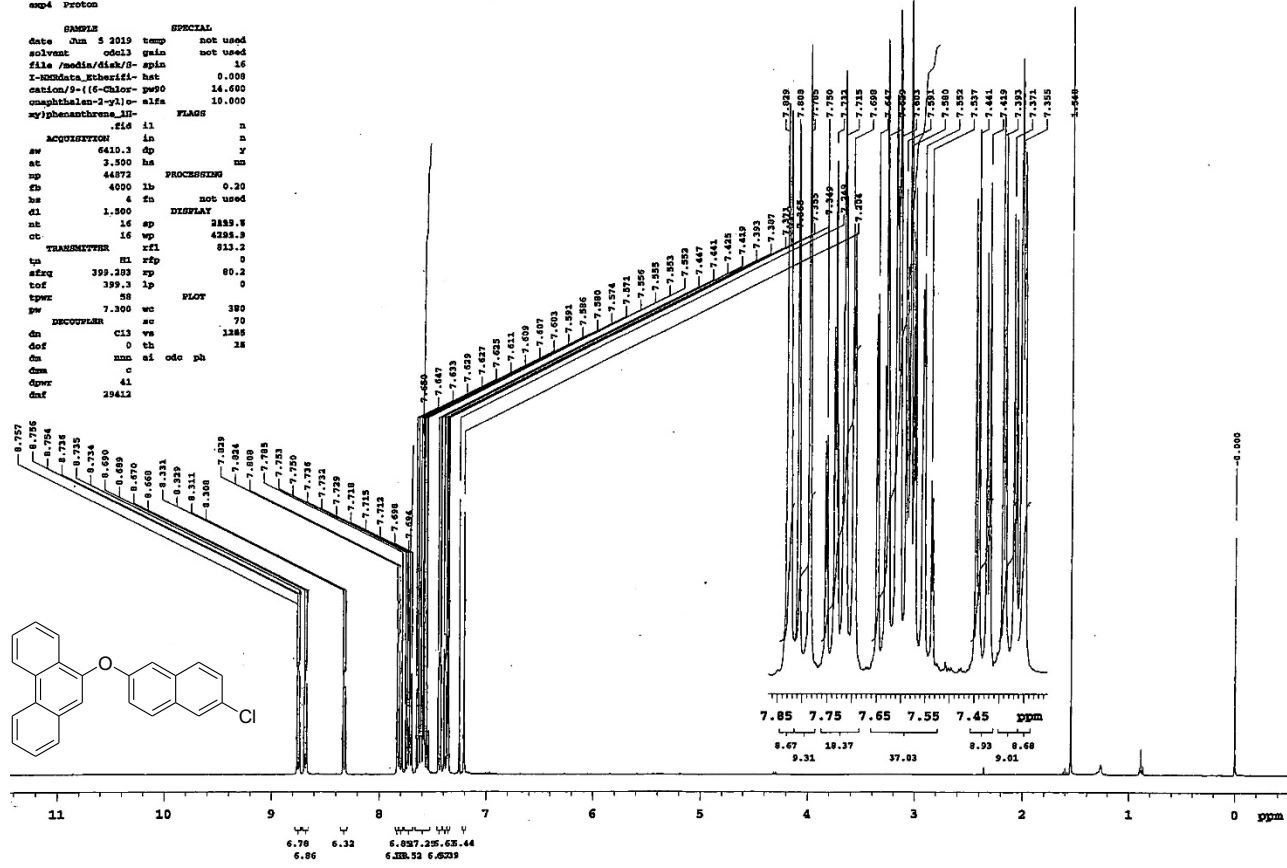
9-(4-Methoxyphenoxy)phenanthrene (4c) ¹H-NMR



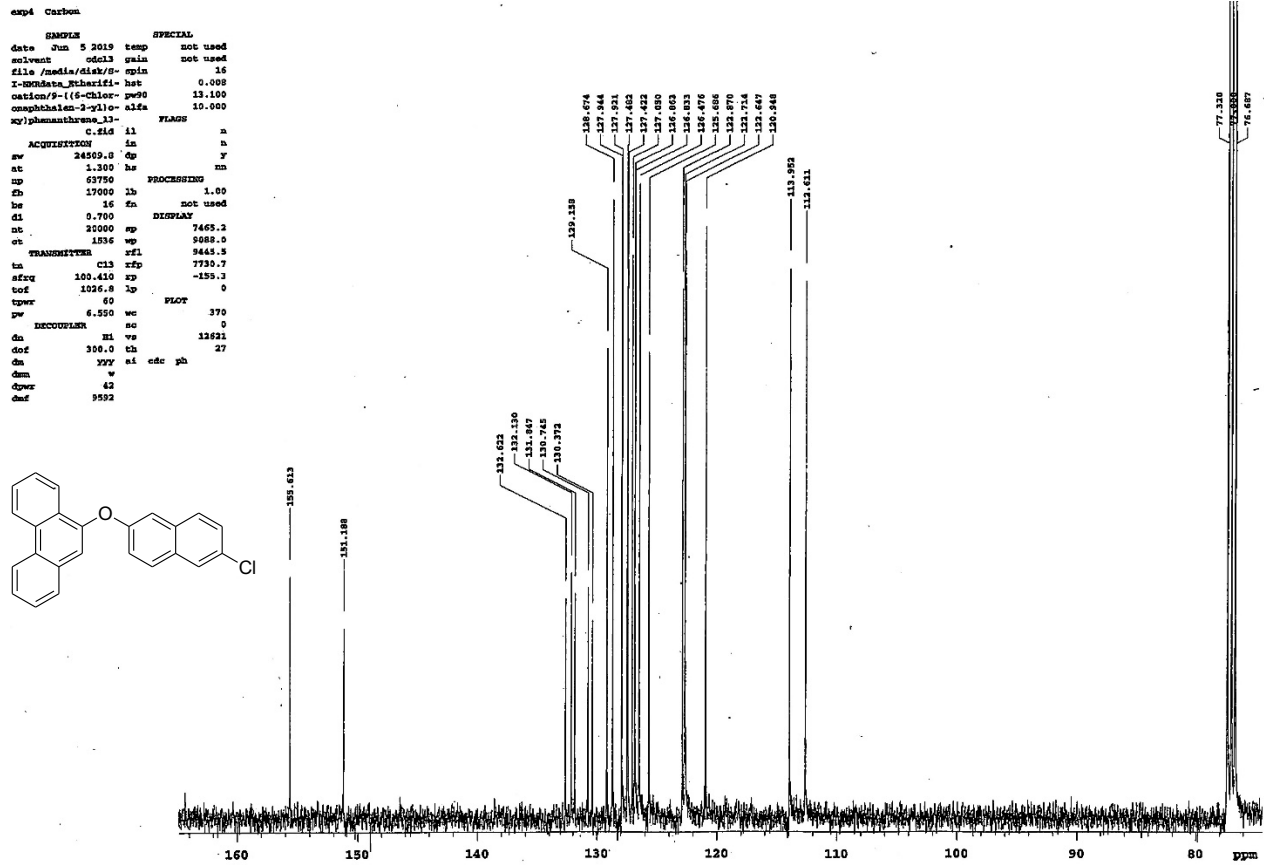
9-(4-Methoxyphenoxy)phenanthrene (4c) ¹³C-NMR



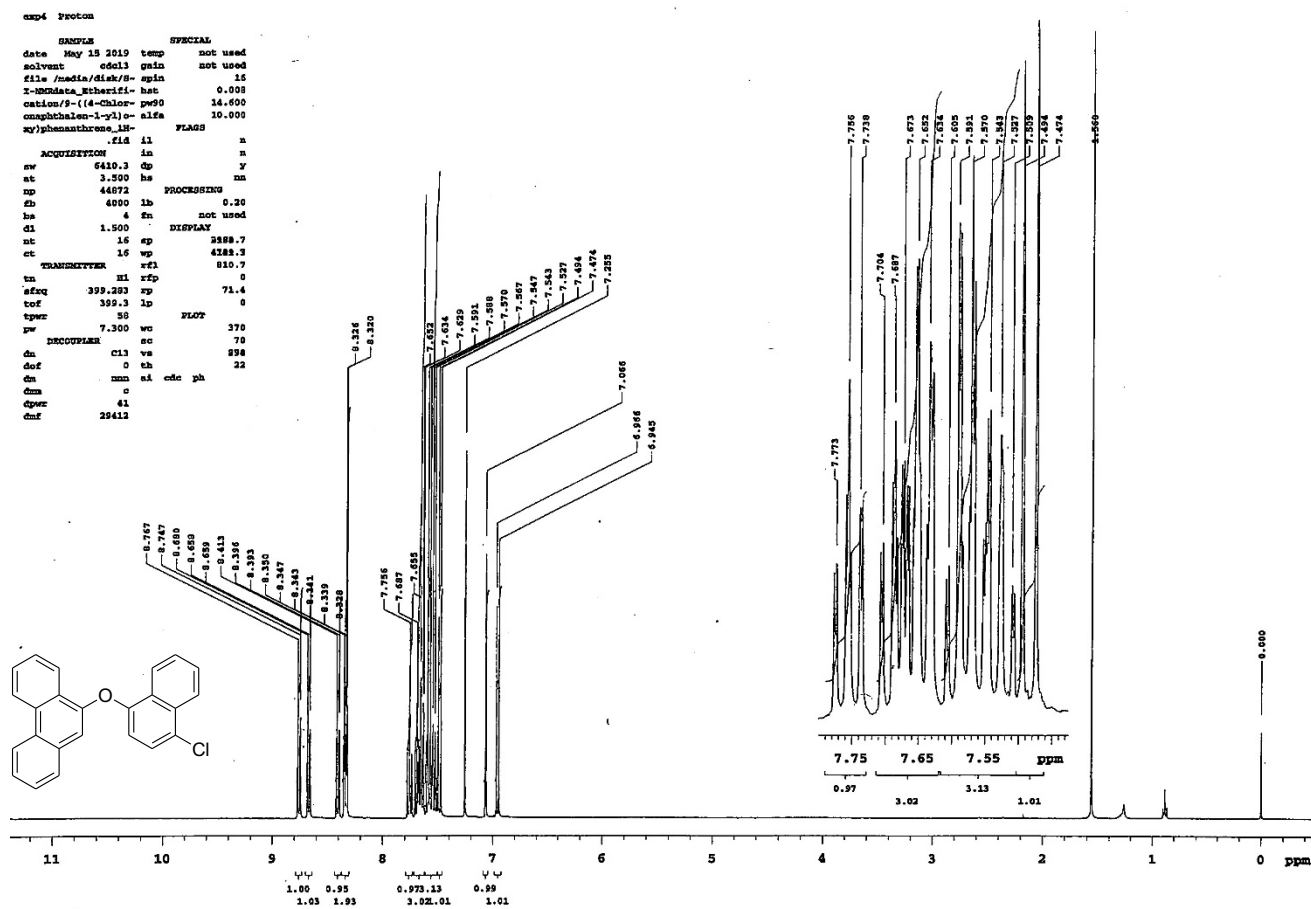
9-((6-Chloronaphthalen-2-yl)oxy)phenanthrene (4d) ¹H-NMR



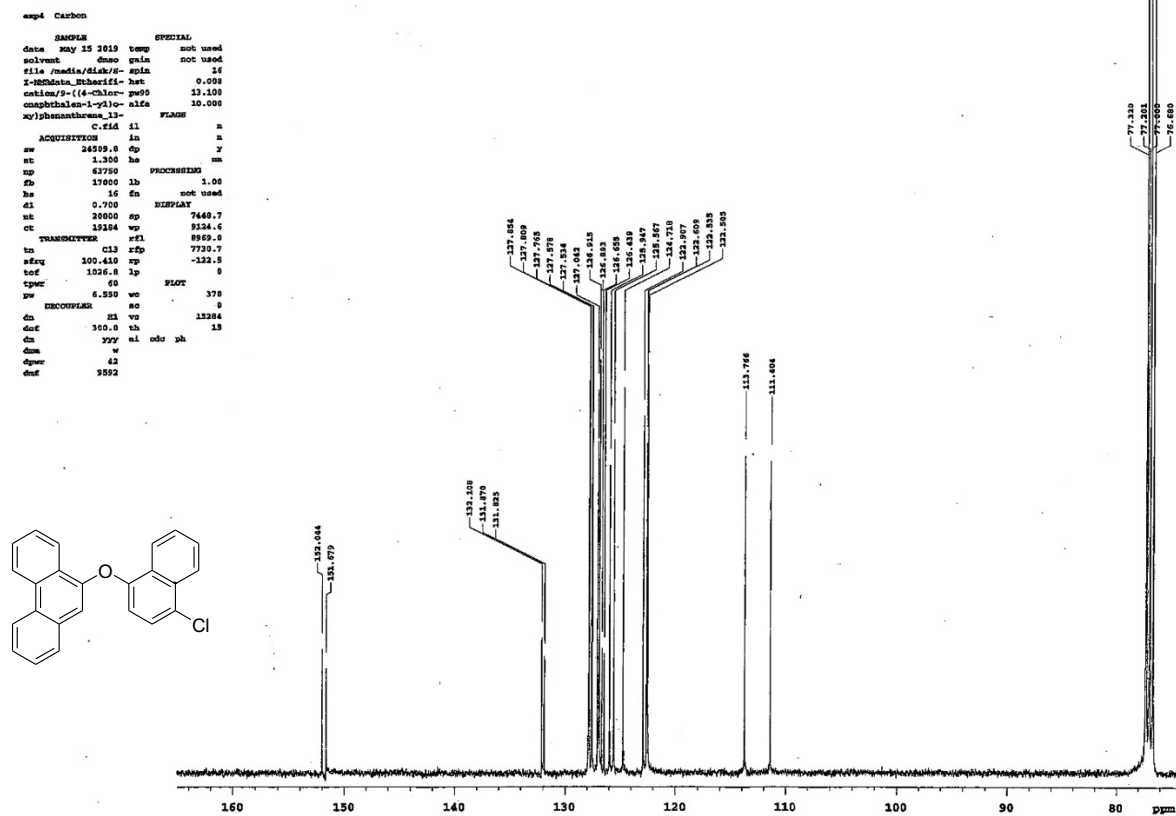
9-((6-Chloronaphthalen-2-yl)oxy)phenanthrene (4d) ¹³C-NMR



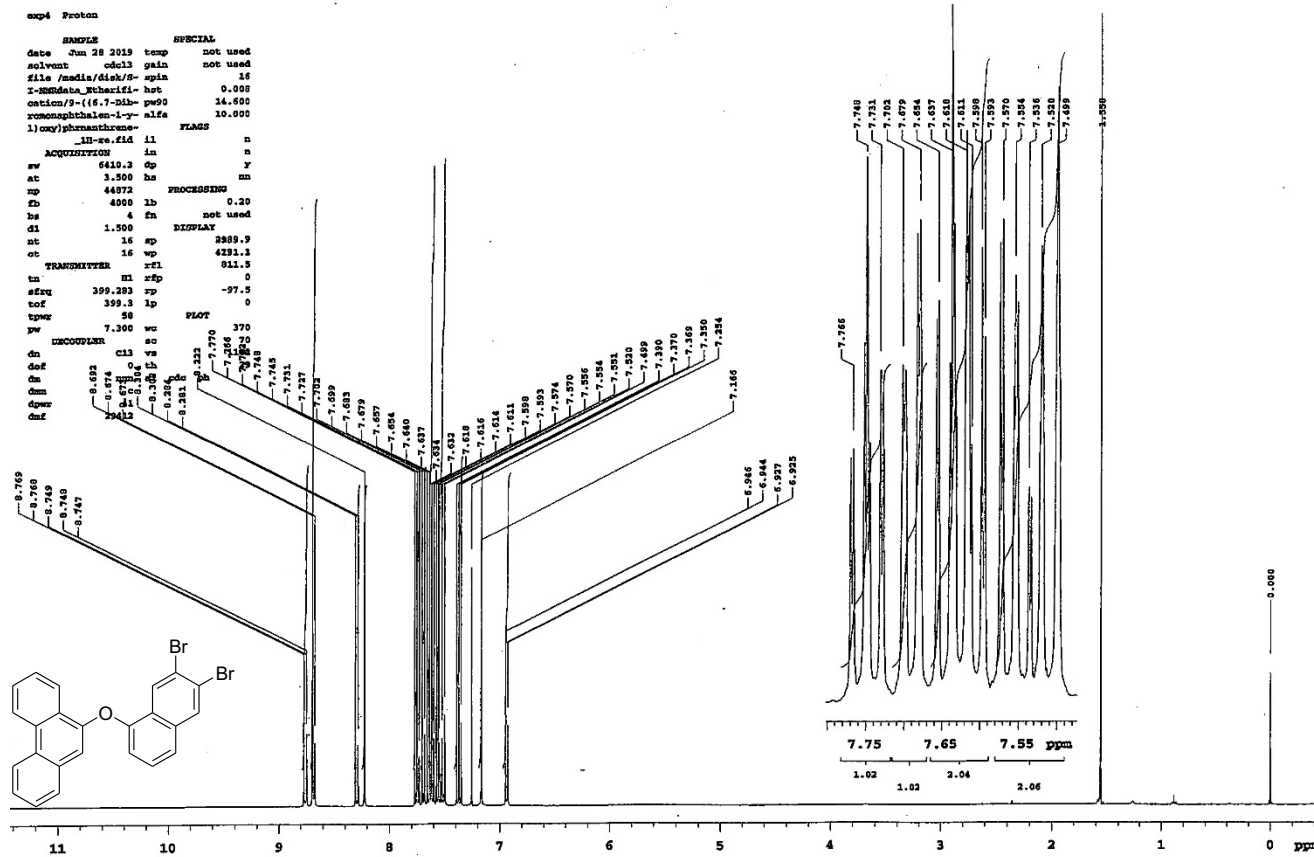
9-((4-Chloronaphthalen-1-yl)oxy)phenanthrene (4e) ¹H-NMR



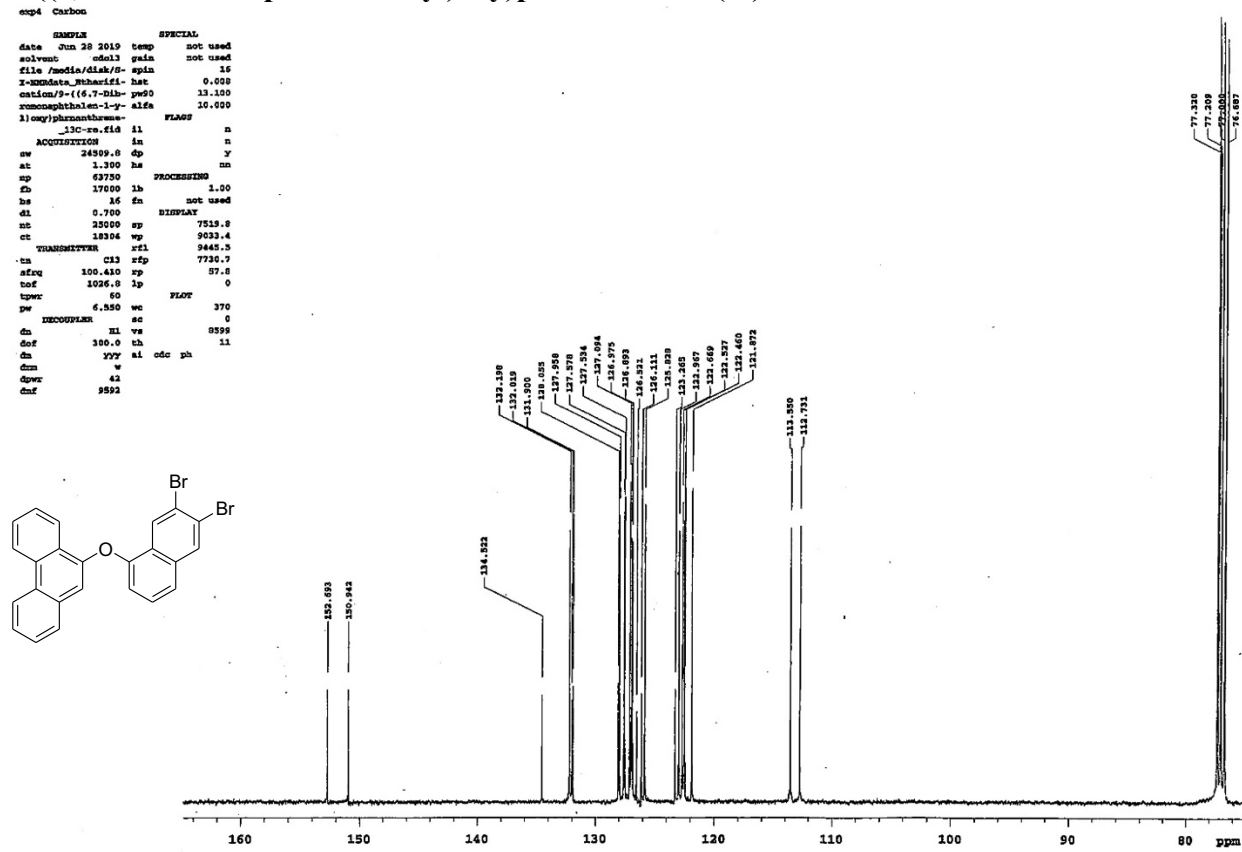
9-((4-Chloronaphthalen-1-yl)oxy)phenanthrene (4e) ¹³C-NMR



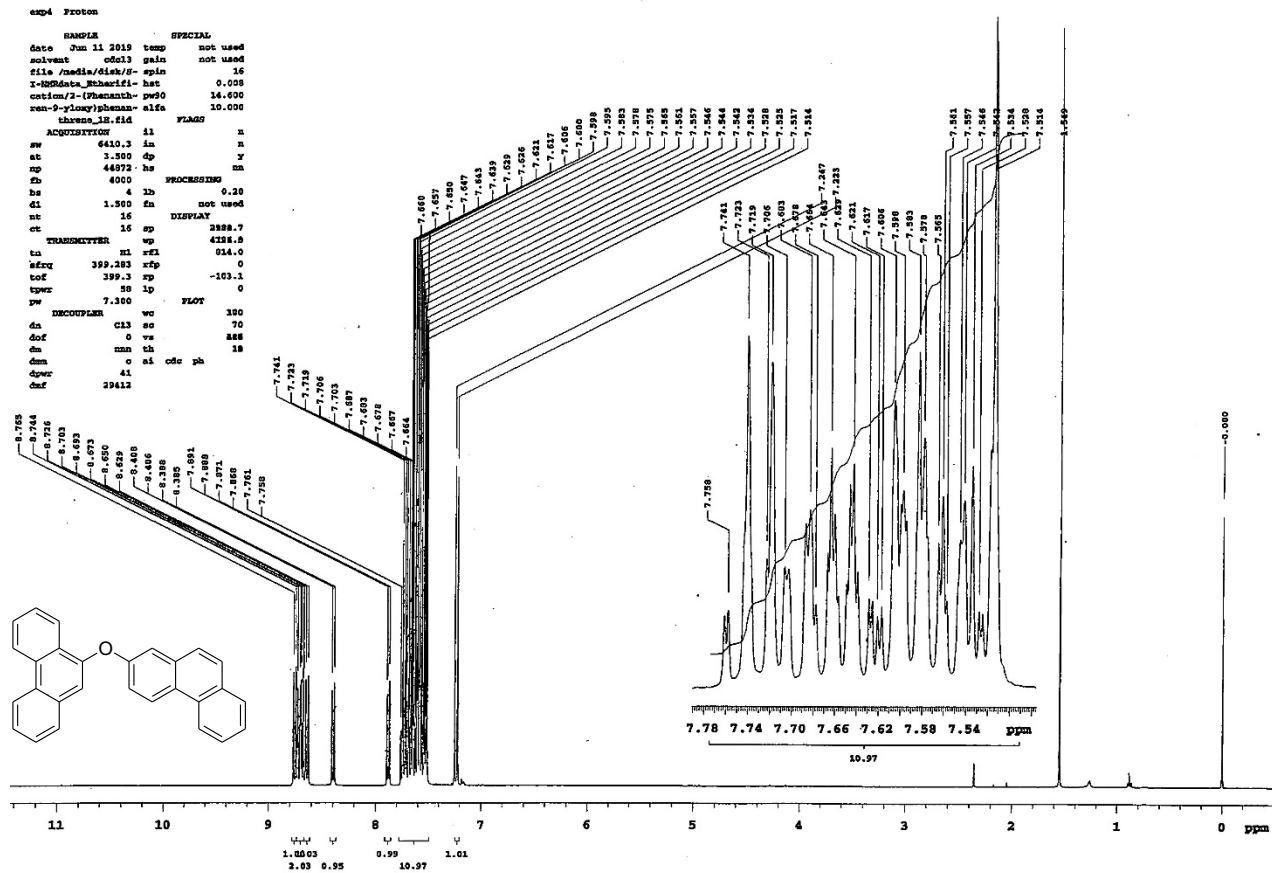
9-((6,7-Dibromonaphthalen-1-yl)oxy)phenanthrene (4f) ¹H-NMR



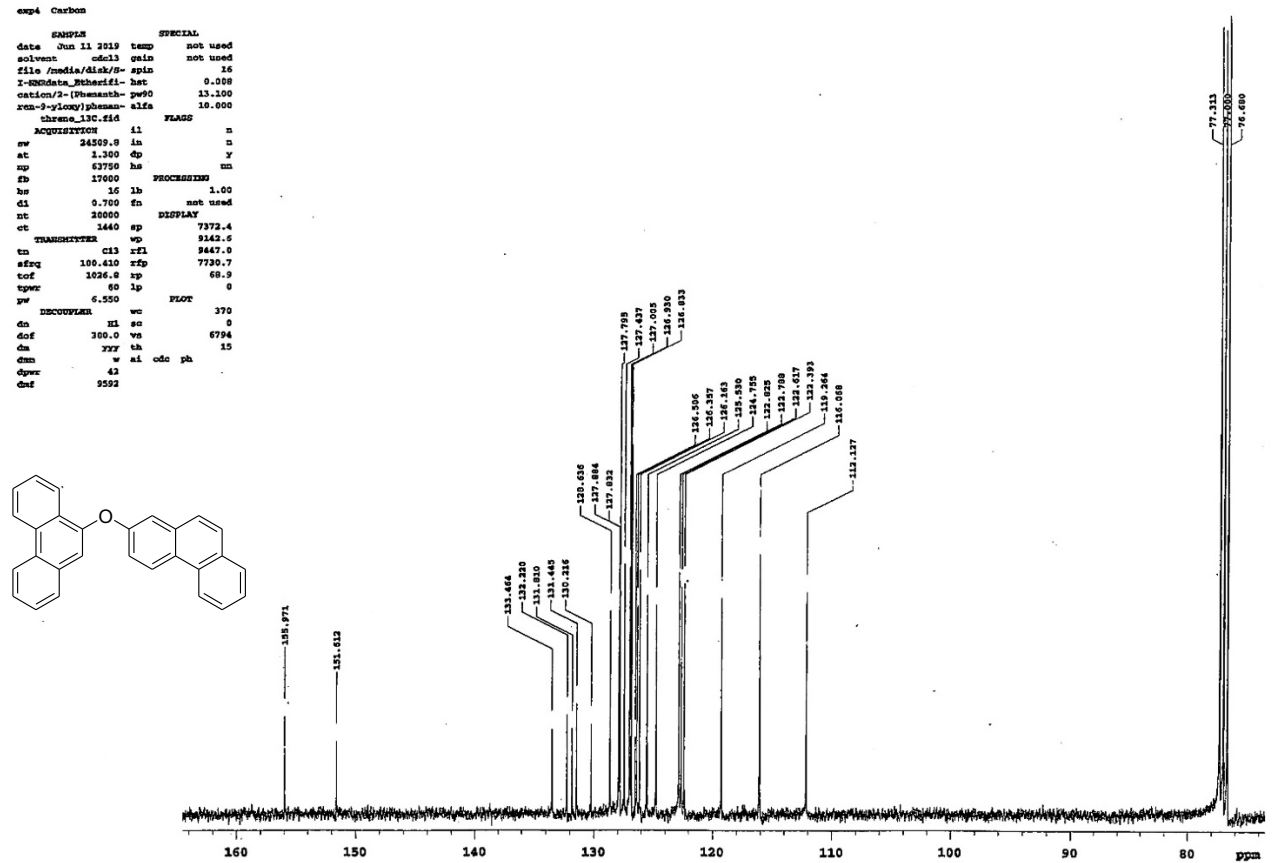
9-((6,7-Dibromonaphthalen-1-yl)oxy)phenanthrene (4f) ¹³C-NMR



2-(Phenanthren-9-yloxy)phenanthrene (4g) ¹H-NMR



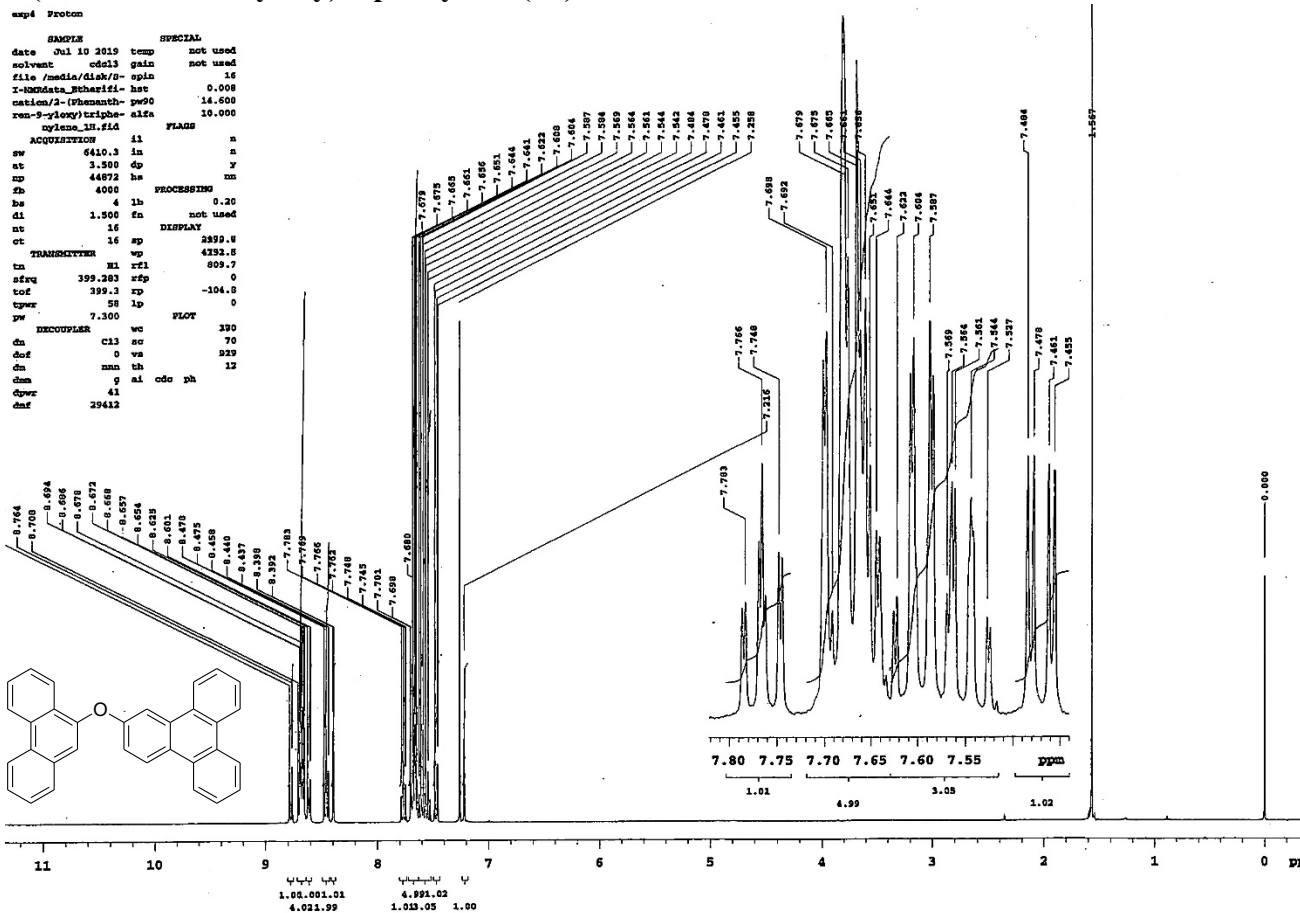
2-(Phenanthren-9-yloxy)phenanthrene (4g) ¹³C-NMR



2-(Phenanthren-9-yloxy)triphenylene (4h) ¹H-NMR

exp4 Proton

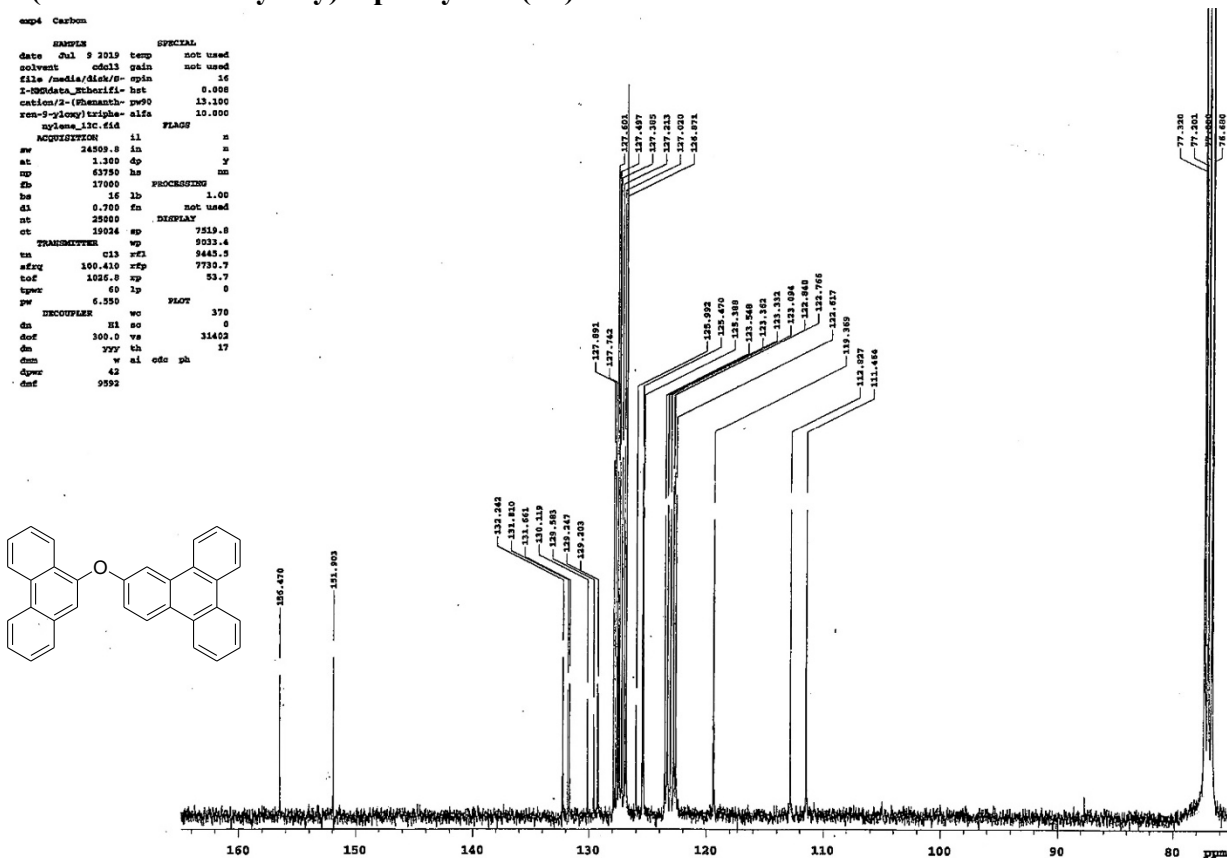
```
SAMPLE SPECIAL
date Jul 10 2019 temp not used
solvent cdcl3 gain not used
file /media/disk/r- spin 16
I-NOData_Rcharif- hst 0.008
cation/2-(Phenanthren-9-yloxy)triphenylene_1h.fid
ren-9-yloxy)triphenylene_1h.fid
ACQUISITION FLAG n
sw 6410.3 in n
at 3.300 dp y
f2 44872 hs nm
f3 4000 PROCESSING
hs 4 lb 0.20
di 1.500 fn not used
nt 16 DISPLAY
ot 16 sp 2392.5
TRANSMITTER wp 4352.5
tn H1 rf1 809.7
sfreq 399.283 rfp 0
tof 399.3 rp -104.0
tqwr 58 lp 0
pw 7.300 F1OT
RECOUPLER wc 390
dn C13 so 70
dof 0 ve 239
dn mm kh 12
dnn g ai cdo ph
dpcw 41
dmf 29412
```



2-(Phenanthren-9-yloxy)triphenylene (4h) ¹³C-NMR

exp4 Carbon

```
SAMPLE SPECIAL
date Jul 9 2019 temp not used
solvent cdcl3 gain not used
file /media/disk/r- spin 16
I-NOData_Rcharif- hst 0.008
cation/2-(Phenanthren-9-yloxy)triphenylene_1h.fid
ren-9-yloxy)triphenylene_1h.fid
ACQUISITION FLAG n
sw 24509.8 in n
at 1.300 dp y
f2 63750 hs nm
f3 17000 PROCESSING
hs 16 lb 1.00
di 0.700 fn not used
nt 25000 DISPLAY
ot 19024 sp 7519.8
TRANSMITTER wp 9233.4
tn H1 rf1 9445.3
sfreq 100.610 rfp 7730.7
tof 1026.8 rp 53.7
tqwr 60 lp 0
pw 6.550 F1OT
RECOUPLER wc 370
dn H1 so 0
dof 300.0 ve 31402
dn mm kh 17
dnn w ai cdo ph
dpcw 43
dmf 9392
```

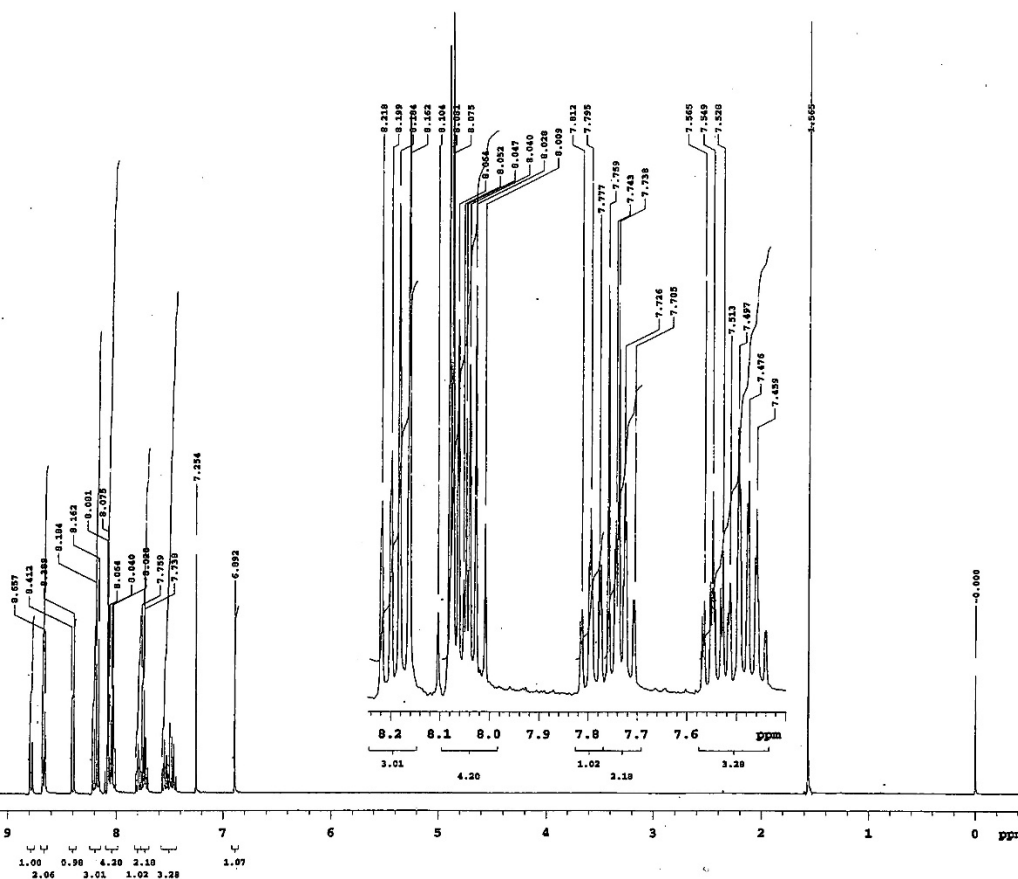
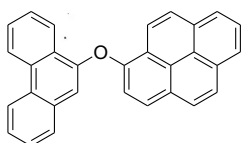


1-(Phenanthren-9-yloxy)pyrene (4i) ¹H-NMR

exp4 Proton

```

SAMPLE          SPECIAL
date Jul 9 2019 temp not used
solvent cdcl3 gain not used
file /media/disk/s- spin 16
X-MMRData_Michaeli-hat 0.008
cation/1-(Phenanth- py90 14.600
ren-9-yloxy)pyrene- alfa 10.000
   .fid          FLAGS
ACQUISITION    i1      n
pw 6410.3 in      n
at 3.300 dp      y
sp 44872 hs      m
zb 4000          PROCESSING
bs 4 lb         not used
dl 1.500 fm      DISPLAY
nt 16          2898.1
ct 16          4798.8
TRANSMITTER    vp      813.3
tn 81 rfl
sfreq 399.283 rfp 0
tof 399.3 rp    -109.7
tpwr 80 lp      0
pw 7.300          PLOT
RECOUPLER      wc      320
dn C13 ac       70
dof 0 va        309
dm mm th
dmn w nl cdc ph 21
dpcr 41
dmf 29412
    
```



1-(Phenanthren-9-yloxy)pyrene (4i) ¹³C-NMR

exp4 Carbon

```

SAMPLE          SPECIAL
date Jul 9 2019 temp not used
solvent cdcl3 gain not used
file /media/disk/s- spin 16
X-MMRData_Michaeli-hat 0.008
cation/1-(Phenanth- py90 14.600
ren-9-yloxy)pyrene- alfa 10.000
   .13c.fid      FLAGS
ACQUISITION    i1      n
pw 24509.8 in   n
at 1.300 dp     y
sp 63750 hs     m
zb 17000          PROCESSING
bs 15 lb        1.00
dl 0.700 fm     not used
nt 29000          DISPLAY
ct 10944 ap     7555.7
TRANSMITTER    wo     8030.9
tn 813 rfl      9446.2
sfreq 100.410 rfp 7730.7
tof 1026.0 rps 33.9
tpwr 60 lp      0
pw 6.550          PLOT
RECOUPLER      wc     370
dn C13 ac       20999
dof 300.0 va    20999
dm xxy th
dmn w nl cdc ph 20
dpcr 41
dmf 8592
    
```

