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Synthesis of Isochromenones and Oxepines via Pd-Catalyzed Cascade Cyclization of Alkynes and Arynes Involving a C-H Activation

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Supporting Information

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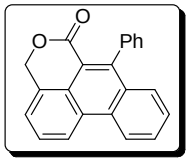
Experimental Section

General: All reactions were conducted under nitrogen atmosphere on a screw-capped sealed tube. Acetonitrile was dried with CaH_2 and distilled prior to use.[1] Aryne precursors **2a-d** were synthesized according to the reported procedure.[2] Starting materials alkynes **1a-g** was synthesized according to the literature procedures.[3] Other reagents were commercially available and used as purchased.

General Procedure for the Palladium-Catalyzed Synthesis of Isochromenone Derivatives (3): A sealed tube containing $Pd(dba)_2$ (5 mol %), CsF (4 equiv), Tl(OAc) (1.2 equiv) and alkynes (1.00 mmol) was evacuated and purged with nitrogen gas three times. Then, benzyne precursor (1.20 mmol), acetonitrile (2.0 mL) and toluene (1.0 mL) were sequentially added to the system and the reaction mixture was allowed to stir at 85 °C for 8 h. The mixture was filtered through a short Celite pad and the Celite pad was washed with CH_2Cl_2 several times. The filtrate was concentrated and the residue was purified on a silica gel column using hexanes-ethyl acetate as eluent to afford isochromenone derivative **3**. Similar procedures were also used for the preparation of oxepine derivative **4**.

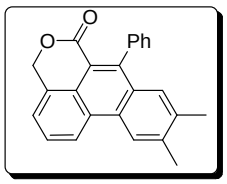
The spectral data and a copy of ^{1}H and ^{13}C NMR spectra of all compounds are listed below.

7-Phenyl-4*H*,6*H*-dibenzo[*de,g*]isochromen-6-one (3a)



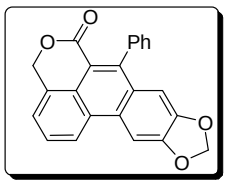
Yellow solid; m.p. 139-141 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.74 (d, *J* = 8.8 Hz, 1 H), 8.68 (d, *J* = 8.8 Hz, 1 H), 7.78-7.71 (m, 3 H), 7.61 (d, *J* = 7.2 Hz, 1 H), 7.59-7.49 (m, 4 H), 7.36 - 7.27 (m, 2 H), 5.74 (s, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.0, 146.1, 139.7, 132.3, 131.9, 130.0, 129.3, 129.2, 128.8 (2 CH), 128.5, 128.0 (2 CH), 127.8, 127.3 (2 CH), 126.9, 126.5, 122.1, 121.8, 69.0; HRMS calcd for $C_{22}H_{14}O_2$ 310.0994, found 310.0998.

9,10-Dimethyl-7-phenyl-4*H*,6*H*-dibenzo[*de,g*]isochromen-6-one (3b)



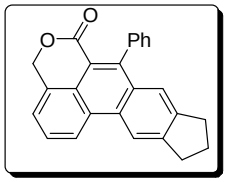
Yellow solid; m.p. 164-166 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.62 (d, *J* = 8.8 Hz, 1 H), 8.47 (s, 1 H), 7.67 (t, *J* = 8.0 Hz, 1 H), 7.53-7.47 (m, 3 H), 7.40 (d, *J* = 8.8 Hz, 1 H), 7.29-7.26 (m, 3 H), 5.69 (s, 2H), 2.52 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.1, 145.9, 139.9, 139.3, 136.9, 130.8, 130.4, 129.8, 128.7 (2 CH), 128.5, 128.0 (2 CH), 127.8, 127.2, 126.6, 126.3, 122.9, 121.9, 121.5, 115.0, 69.0, 20.6, 20.2 ; HRMS calcd for $C_{24}H_{18}O_2$ 338.1307, found 338.1311.

9,10,-Dioxolo-7-phenyl-4*H*,6*H*-dibenzo [*de,g*]isochromen-6-one (3c)



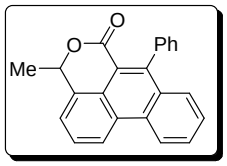
Yellow solid; m.p. 221-223 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45 (d, *J* = 8.0 Hz, 1 H), 8.04 (s, 1 H), 7.66 (t, *J* = 7.2 Hz, 1 H), 7.50-7.46 (m, 3 H), 7.40 (d, *J* = 6.0 Hz, 1 H), 7.25 (d, *J* = 6.0 Hz, 2 H), 6.89 (s, 1 H), 6.09 (s, 2 H), 5.68 (s, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.1, 150.2, 148.2, 145.4, 140.1, 129.5, 129.1, 128.8, 128.7(2CH), 128.6, 128.2(2CH), 127.3, 126.5, 126.2, 121.6, 121.4, 107.0, 101.9, 100.5, 69.0; HRMS calcd for $C_{23}H_{14}O_4$ 354.0892, found 354.0896.

7-Phenyl-9,10,11-tetrahydro-6*H*-benzo[*de*]indeno[5,6-*g*]isochromen-6-one (3d)



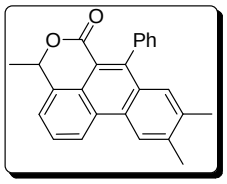
Yellow solid; m.p. 196-198 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.63 (d, *J* = 8.8 Hz, 1 H), 8.50 (s, 1 H), 7.67 (t, *J* = 8.0 Hz, 1 H), 7.54-7.48 (m, 3 H), 7.41 (d, *J* =7.2 Hz, 1 H), 7.38 (s, 1 H), 7.29-7.27 (m, 2 H), 5.69 (s, 2 H), 3.16 (t, *J* = 6.8 Hz, 2 H), 2.96 (t, *J* = 6.8 Hz, 2 H), 2.16-2.11 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.2, 147.2, 146.3, 144.7, 140.2, 131.5, 131.1, 129.2, 128.8(2CH), 128.4, 128.0, 127.7, 127.1, 126.5, 126.3, 124.6, 121.7, 121.5, 117.5, 114.9, 69.0, 33.2, 32.8, 25.8; HRMS calcd for $C_{25}H_{18}O_2$ 350.1307, found 350.1310.

4-Methyl-7-phenyl-4*H*,6*H*-dibenzo[*de*,*g*]isochromen-6-one (3e)



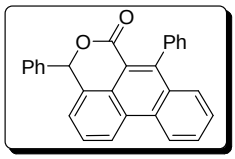
Yellow solid; m.p. 140-142 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.73 (d, *J* = 8.8 Hz, 1 H), 8.67 (d, *J* = 8.8 Hz, 1 H), 7.77-7.70 (m, 2 H), 7.59 (d, *J* = 8.0 Hz, 1 H), 7.53-7.41 (m, 4 H), 7.28 (d, *J* = 8.0 Hz, 2 H), 5.89 (q, *J* = 6.8, 13.6 Hz, 1 H), 1.77 (d, *J* = 6.8 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.9, 145.8, 139.7, 133.9, 132.2, 132.0, 129.9, 129.3, 129.1, 128.9, 128.7, 128.2, 128.1, 128.0, 127.5, 127.2, 126.9, 125.6, 122.5, 122.3, 115.9, 76.0, 24.5; HRMS calcd for $C_{23}H_{16}O_2$ 324.1150, found 324.1142.

4,9,10-Trimethyl-7-phenyl-4*H*,6*H*-dibenzo[*de*,*g*]isochromen-6-one (3f)



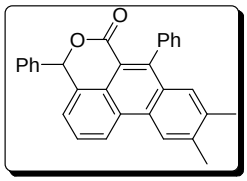
Yellow solid; m.p. 232-234 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.62 (d, *J* = 8.0 Hz, 1 H), 8.47 (s, 1 H), 7.68 (t, *J* = 8.0 Hz, 1 H), 7.53-7.47 (m, 3 H), 7.42 (d, *J* = 8.0 Hz, 1 H), 7.30-7.26 (m, 3 H), 5.87 (q, *J* = 6.4, 13.2 Hz, 1 H), 2.52 (s, 3 H), 2.30 (s, 3 H), 1.76 (d, *J* = 6.4 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.2, 145.6, 140.0, 139.2, 136.8, 133.8, 130.7, 130.5, 129.8, 129.0, 128.9, 128.7, 128.0, 127.1, 126.6, 125.5, 122.9, 121.9, 121.5, 115.1, 75.9, 24.4, 20.6, 20.1; HRMS calcd for $C_{25}H_{20}O_2$ 352.1463, found 352.1455.

4,7-Diphenyl-4*H*,6*H*-dibenzo[*de*,*g*]isochromen-6-one (3g)



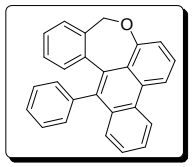
Yellow solid; m.p. 167-169 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.74 (d, *J* = 8.2 Hz, 1 H), 8.69 (d, *J* = 8.2 Hz, 1 H), 7.74 (t, *J* = 6.8 Hz, 1 H), 7.69 (t, *J* = 6.8 Hz, 1 H), 7.60 (d, *J* = 6.8 Hz, 1 H), 7.54-7.46 (m, 4 H), 7.33-7.29 (m, 8 H), 6.74 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.7, 146.2, 140.1, 139.6, 132.2, 132.0, 131.9, 129.9, 129.3, 128.9, 128.8, 128.7, 128.7, 128.6, 128.1, 128.0, 127.4, 127.3 (2CH), 126.8, 126.3, 124.5(2 CH), 122.5, 122.1, 116.2, 81.4; HRMS calcd for $C_{28}H_{18}O_2$ 386.1307, found 386.1312.

9,10-Dimethyl-4,7-diphenyl-4*H*,6*H*-dibenzo[*de*,*g*]isochromen-6-one (3h)



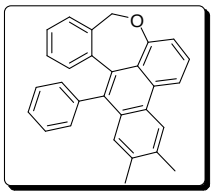
Yellow solid; m.p. 215-217 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.66 (d, *J* = 8.0 Hz, 1 H), 8.50 (s, 1 H), 7.65 (t, *J* = 11.2 Hz, 1 H), 7.55-7.48 (m, 4 H), 7.34-7.22 (m, 8 H), 6.73 (s, 1 H), 2.56 (s, 3 H), 2.54 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.0, 146.0, 140.2, 139.9, 139.3, 136.9, 131.7, 130.7, 130.5, 129.8, 129.3, 129.1, 128.9, 128.8, 128.6, 128.5, 128.1, 128.0, 127.5, 127.4, 127.2, 127.1, 126.5, 126.1, 124.0, 122.9, 121.9, 115.4, 81.4, 20.6, 20.1; HRMS calcd for $C_{30}H_{22}O_2$ 414.1620, found 414.1620.

14-Phenyl-9*H*-benzo[*e*]phenanthro[1,10-*bc*]oxepine (4a)



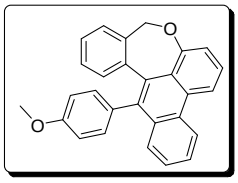
Yellow solid; m.p. 236-238 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.67 (d, *J* = 8.0 Hz, 1 H), 8.36 (d, *J* = 8.5 Hz, 1 H), 7.68 (d, *J* = 8.5 Hz, 1 H), 7.62 (t, *J* = 7.0 Hz, 1 H), 7.56 (d, *J* = 7.0 Hz, 1 H), 7.51-7.45 (m, 2 H), 7.42 (t, *J* = 7.0 Hz, 1 H), 7.27 (d, *J* = 7.5 Hz, 1 H), 7.22 (t, *J* = 7.0 Hz, 1 H), 7.12 (d, *J* = 7.0 Hz, 1 H), 7.10-7.06 (m, 2 H), 6.88 (d, *J* = 7.5 Hz, 1 H), 6.84-6.79 (m, 2 H), 5.62 (d, *J* = 12.0 Hz, 1 H), 4.97 (d, *J* = 12.0 Hz, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.6, 140.3, 140.0, 137.6, 137.4, 134.9, 133.5, 132.2, 132.0, 128.4, 127.8, 127.5, 127.2, 126.9, 126.8, 126.7, 123.1, 121.1, 116.2, 115.4, 72.4; HRMS calcd for $C_{27}H_{18}O$ 358.1358, found 358.1360.

2,3-Dimethyl-14-phenyl-9*H*-benzo[*e*]phenanthro[1,10-*bc*]oxepine (4b).



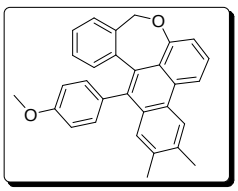
Pale yellow solid; m.p. 218-220 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45 (s, 1 H) 8.35 (d, *J* = 7.6 Hz, 1 H), 7.59 (d, *J* = 7.6 Hz, 1 H), 7.49-7.42 (m, 3 H), 7.29-7.22 (m, 2 H), 7.11-7.06 (m, 3 H), 6.89 (d, *J* = 7.2 Hz, 1 H), 6.85-6.79 (m, 3 H), 5.63 (d, *J* = 12.0 Hz, 1 H), 4.97 (d, *J* = 12.0 Hz, 1 H), 2.53 (s, 3 H), 2.34 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.8, 137.6, 136.5, 136.3, 135.2, 133.7, 132.2, 132.1, 131.2, 130.6, 129.3, 128.6, 128.1, 127.6, 127.3, 127.1, 126.8, 126.4, 123.7, 121.3, 115.8, 115.6, 72.7, 20.7, 20.4; HRMS calcd for $C_{29}H_{22}O$ 386.1671, found 386.1673.

14-(4-Methoxyphenyl)-9*H*-benzo[*e*]phenanthro[1,10-*bc*]oxepine (4c).



Yellow solid; m.p. 268-270 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.66 (d, *J* = 8.0 Hz, 1 H), 8.35 (d, *J* = 8.0 Hz, 1 H), 7.71 (d, *J* = 8.5 Hz, 1 H), 7.61 (t, *J* = 7.5 Hz, 1 H), 7.49- 7.44 (m, 3 H), 7.27 (d, *J* = 8.0 Hz, 1 H), 7.10 (t, *J* = 8.0 Hz, 1 H), 6.97 (d, *J* = 8.0 Hz, 1 H), 6.86 (t, *J* = 8.0 Hz, 1 H), 6.79 (d, *J* = 8.0 Hz, 2 H), 6.62 (d, *J* = 8.5 Hz, 1 H), 5.60 (d, *J* = 11.5 Hz, 1 H), 4.96 (d, *J* = 12.0 Hz, 1 H), 3.79 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.5, 158.3, 140.2, 137.3, 137.2, 134.9, 134.3, 133.0, 132.5, 132.2, 132.1, 131.8, 130.7, 127.8, 127.6, 127.0, 126.8, 123.1, 121.2, 116.1, 115.5, 113.6, 113.0, 72.4, 55.2; HRMS calcd for $C_{28}H_{20}O_2$ 388.1463, found 388.1464.

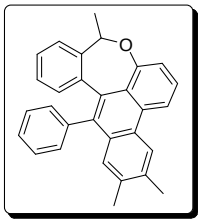
4-(4-Methoxyphenyl)-2,3-dimethyl-9*H*-benzo[*e*]phenanthro[1,10-*bc*]oxepine (4d).



Pale yellow solid; m.p. 168-170 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.41 (d, *J* = 8.0 Hz, 1 H), 8.31 (d, *J* = 8.5 Hz, 1 H), 7.45-7.41 (s, 1 H, m, 1 H merged with same value), 7.05 (d, *J* = 7.0 Hz, 2 H), 6.96 (t, *J* = 8.0 Hz, 1 H), 6.85 (t, *J* = 7.5 Hz, 1 H), 6.76 (d, *J* = 8.5 Hz, 2 H), 6.61 (d, *J* = 8.5 Hz, 1 H), 5.59 (d, *J* = 11.5 Hz, 1 H), 4.93 (d, *J* = 12.0 Hz, 1 H), 3.78 (s, 3 H), 2.51 (s, 3 H), 2.32 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.5, 158.3, 140.5, 137.4, 136.9, 134.9, 134.0, 132.7, 131.9, 130.8, 130.6,

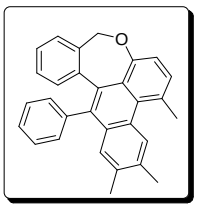
129.0, 127.8, 127.5, 126.5, 126.1, 123.5, 121.1, 115.3, 113.4, 113.1, 72.4, 55.2, 20.4, 20.1; HRMS calcd for $C_{30}H_{24}O_2$ 416.1776, found 416.1779.

2,3,9-Trimethyl-14-phenyl-9*H*-benzo[*e*]phenanthro[1,10-*bc*]oxepine (4e).



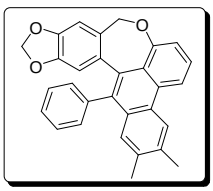
Brown solid; m.p. 165-167 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.42 (s, 1 H), 8.29 (d, *J* = 8.0 Hz, 1 H), 7.55 (d, *J* = 7.6 Hz, 1 H), 7.46 - 7.38 (s, 1 H, m, 2 H merged with same value), 7.34 (d, *J* = 7.6 Hz, 2 H), 7.23 (t, *J* = 7.6 Hz, 1 H), 7.09-7.03 (m, 3 H), 6.84 (d, *J* = 7.6 Hz, 1 H), 6.81-6.74 (m, 2 H), 6.76 (d, *J* = 8 .5 Hz, 2 H), 5.84 (q, *J* = 6.4 Hz, 1 H), 2.15 (s, 3 H), 2.31 (s, 3 H), 1.82 (d, *J* = 6.8 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.5, 140.6, 140.4, 136.0, 135.9, 134.9, 128.3, 127.8, 127.1, 127.0, 126.7, 126.6, 126.5, 123.5, 121.0, 115.4, 115.0, 75.2, 20.4, 20.0, 17.5; HRMS calcd for $C_{30}H_{24}O$ 400.1827, found 400.1828.

2,3,5-Trimethyl-14-phenyl-9*H*-benzo[*e*]phenanthro[1,10-*bc*]oxepine (4f).



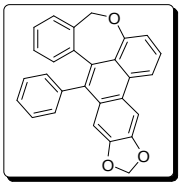
Yellow solid; m.p. 173-175 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.40 (s, 1 H), 7.53 (d, *J* = 7.6 Hz, 1 H), 7.44 (t, *J* = 7.2 Hz, 1 H), 7.37 (s, 1 H), 7.31-7.22 (m, 2 H), 7.11-7.06 (m, 2 H), 6.94 (d, *J* = 8.0 Hz, 1 H), 6.86-6.80 (m, 2 H), 6.74 (d, *J* = 7.6 Hz, 1 H), 5.57 (d, *J* = 11.6 Hz, 1 H), 4.92 (d, *J* = 12.0 Hz, 1 H), 3.02 (s, 3 H), 2.48 (s, 3 H), 2.30 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.1, 140.6, 137.4, 135.2, 134.5, 134.3, 132.9, 131.9, 131.0, 130.8, 130.1, 128.2, 127.9, 127.4, 127.3, 127.2, 126.8, 126.5, 126.1, 126.0, 114.1, 71.6, 25.5, 20.5, 20.0; HRMS calcd for $C_{30}H_{24}O$ 400.1827, found 400.1830.

2,3-Dimethyl-15-phenyl-9*H*-[1,3]dioxolo[4,5-*h*]phenanthro[1,10-*cd*][2]benzoxepin e (4g).



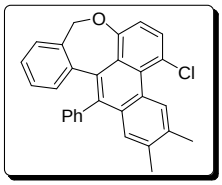
Yellow solid; m.p. 228-230 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.40 (s, 1 H), 8.27 (d, *J* =8.0 Hz, 1 H), 7.51 (d, *J* = 7.6 Hz, 1 H), 7.42 (t, *J* = 7.6 Hz, 1 H), 7.40 (s, 1 H), 7.37 - 7.20 (m, 2 H), 7.15 (t, *J* = 7.6 Hz, 1 H), 7.02 (d, *J* = 7.6 Hz, 1 H), 6.91(d, *J* = 7.6 Hz, 1 H), 6.72 (s, 1 H), 6.23 (s, 1 H), 5.72 (d, *J* = 14.8 Hz, 2 H), 5.50 (d, *J* = 12.0 Hz, 1 H), 4.80 (d, *J* = 12.0 Hz, 1 H), 2.47 (s, 3 H), 2.28 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.4, 146.9, 146.1, 140.4, 136.8, 136.0, 134.3, 133.2, 131.2, 131.3, 130.8, 130.3, 128.9, 128.5, 127.7, 127.2, 126.8, 126.6, 123.5, 120.9, 115.5, 115.3, 114.9, 106.8, 100.8, 71.9, 20.4, 20.1; HRMS calcd for $C_{30}H_{22}O_3$ 430.1569, found 430.1572.

10-Phenyl-5*H*-[1,3]dioxolo[6,7]phenanthro[1,10-*cd*][2]benzoxepine (4h).



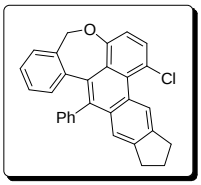
Yellow solid; m.p. 203-205 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (d, *J* = 8.4 Hz, 1 H), 8.02 (s, 1 H), 7.55 (d, *J* = 7.6 Hz, 1 H), 7.44 (t, *J* = 8.0 Hz, 2 H), 7.25 - 7.20 (m, 2 H), 7.19 - 7.03 (s, 1 H, m, 3 H merged with same value), 6.85 - 6.76 (m, 3 H), 6.05 (d, *J* = 15.6 Hz, 2 H), 5.59 (d, *J* = 11.6 Hz, 1 H), 4.93 (d, *J* = 12.0 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.6, 148.0, 147.5, 140.6, 140.0, 137.4, 137.3, 134.9, 133.3, 132.0, 131.9, 131.8, 130.4, 128.5, 127.3, 127.2, 127.1, 126.8, 126.7, 126.6, 126.1, 120.9, 115.4, 115.0, 101.4, 101.3, 72.4; HRMS calcd for $C_{28}H_{18}O_3$ 402.1256, found 402.1259.

5-Chloro-2,3-dimethyl-14-phenyl-9*H*-benzo[*e*]phenanthro[1,10-*bc*]oxepine (4i).



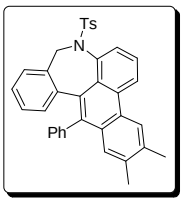
Yellow solid; m.p. 212-214 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.30 (s, 1 H), 7.55-7.43 (m, 3 H), 7.41 (s, 1 H), 7.39-7.25 (m, 3 H), 7.22-7.04 (m, 2 H), 6.93 (d, *J* = 8.0 Hz, 1 H), 6.84-6.79 (m, 2 H), 6.71 (d, *J* = 7.6 Hz, 1 H), 5.52 (d, *J* = 11.6 Hz, 1 H), 4.91 (d, *J* = 11.6 Hz, 1 H), 2.47 (s, 3 H), 2.28 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.7, 136.9, 136.5, 134.8, 134.4 (2CH), 132.8, 131.7, 131.3, 130.2, 129.9, 129.6, 128.2, 128.1(2CH), 127.5, 127.4 (2CH), 127.3, 127.0, 126.7, 126.0, 122.5, 121.7, 114.9, 71.7, 20.4, 20.0; HRMS calcd for $C_{29}H_{21}ClO$ 420.1281, found 420.1279.

6-Chloro-2,3,4-tetrahydro-14-phenyl-9*H*-indeno[*e*]phenanthro[1,10-*bc*]oxepine (4j).



Yellow solid; m.p. 233-235 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.34 (s, 1 H), 7.56-7.42 (m, 3 H), 7.41 (s, 1 H), 7.39-7.24 (m, 3 H), 7.22-7.06 (m, 2 H), 6.90 (d, *J* = 8.4 Hz, 1 H), 6.84-6.81 (m, 2 H), 6.73 (d, *J* = 7.6 Hz, 1 H), 5.53 (d, *J* = 11.6 Hz, 1 H), 4.91 (d, *J* = 11.6 Hz, 1 H), 3.27-3.21 (m, 1 H), 3.19-2.97 (m, 2 H), 2.89-2.81 (m, 1 H), 2.21-2.04 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.6, 144.3, 142.7, 140.4, 139.0, 137.3, 137.0, 134.4, 132.8, 131.9, 131.7, 130.2, 130.1, 129.8, 128.6, 128.2, 127.5, 127.4, 127.0, 126.7, 126.0, 122.4, 122.1, 122.1, 7, 114.7, 71.7, 33.1, 32.9, 25.9; HRMS calcd for $C_{30}H_{21}ClO$ 432.1281, found 432.1283.

2,3-Dimethyl-14-phenyl-8-tolyl-8,9-dihydrobenzo[*e*]phenanthro[1,10-*bc*]azepine (4k).



Yellow solid; m.p. 173-175 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.64 (d, *J* = 8.0 Hz, 1 H), 8.44 (s, 1 H), 8.28 (d, *J* = 8.0 Hz, 1 H), 7.57 (t, *J* = 8.0 Hz, 1 H), 7.43 (t, *J* = 8.0 Hz, 1 H), 7.38-7.32 (m, 3 H), 7.14 (t, *J* = 7.6 Hz, 1 H), 7.01-6.96 (m, 2 H), 6.63 (d, *J* = 8.4 Hz, 1 H), 6.58-6.53 (m, 4 H), 6.32 (d, *J* = 7.6 Hz, 1 H), 5.17 (d, *J* = 14.8 Hz, 1 H), 4.95 (d, *J* = 14.8 Hz, 1 H), 2.49 (s, 3 H), 2.28 (s, 3 H), 2.07 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.0, 140.1 (2C), 139.5, 139.2, 139.1, 137.8, 136.3, 136.2, 135.9, 133.4, 131.7, 131.5, 131.2, 130.0, 128.8, 128.4, 128.2, 127.9, 127.3, 126.9, 126.6, 126.3, 126.2(2CH), 125.6, 54.2, 21.1, 20.4, 20.1; HRMS calcd for $C_{36}H_{29}NO_2S$ 539.1919, found 539.1921.

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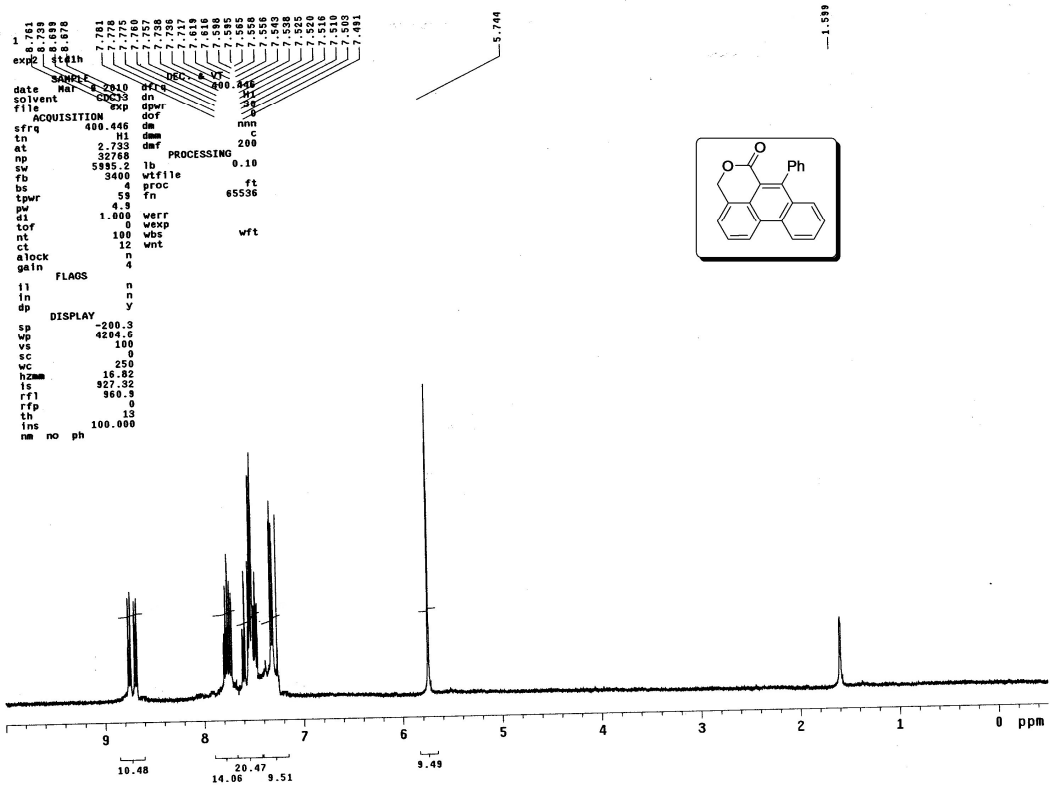
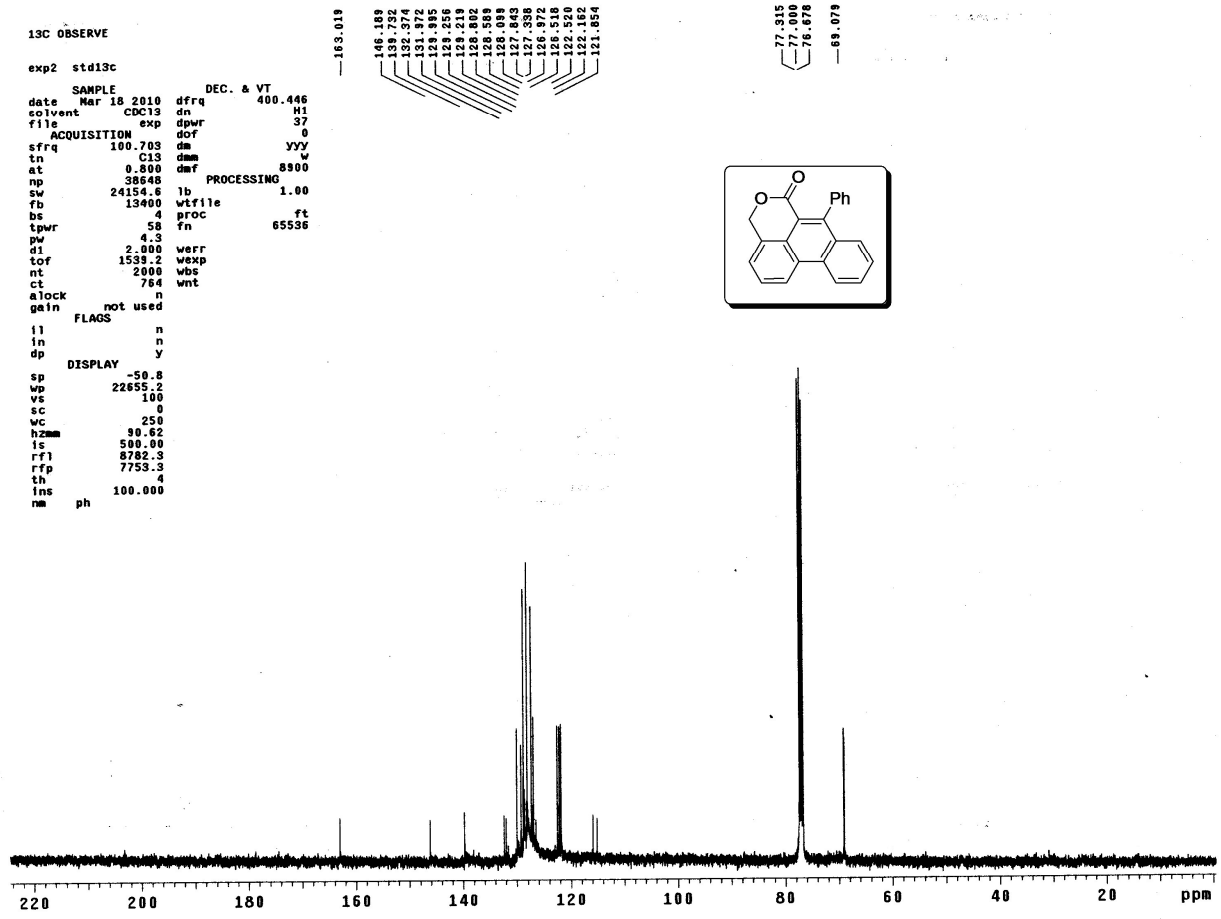
2) *(a)* D. Pena, S. Escudero, D. Perez, E. Guitian and L. Castedo, *Angew. Chem. Int. Ed.* 1998, **37**, 2659; *(b)* D. Peña, D. Pérez, E. Guitián and L. Castedo, *J. Am. Chem. Soc.* 1999, **121**, 5827.

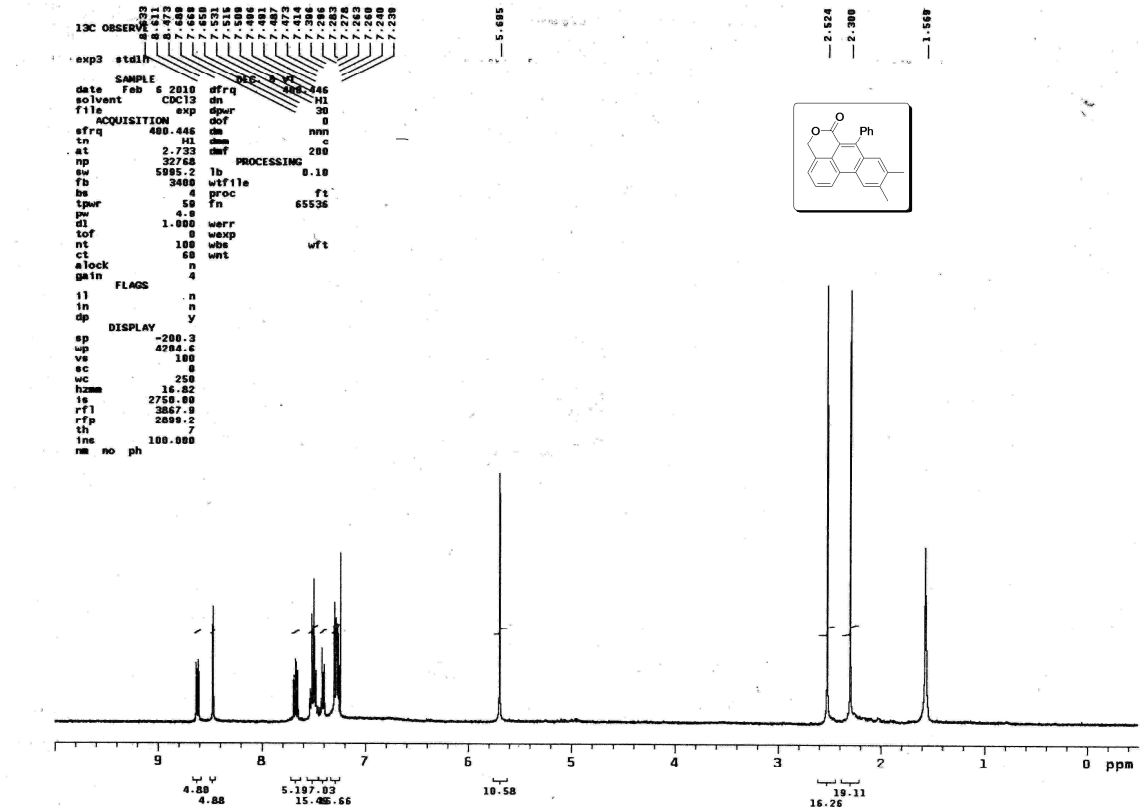
3) H. Yu, R. N. Richey, M. W. Cason and M. J. Coghlan, *Org. Lett.* 2006, **8**, 1685.

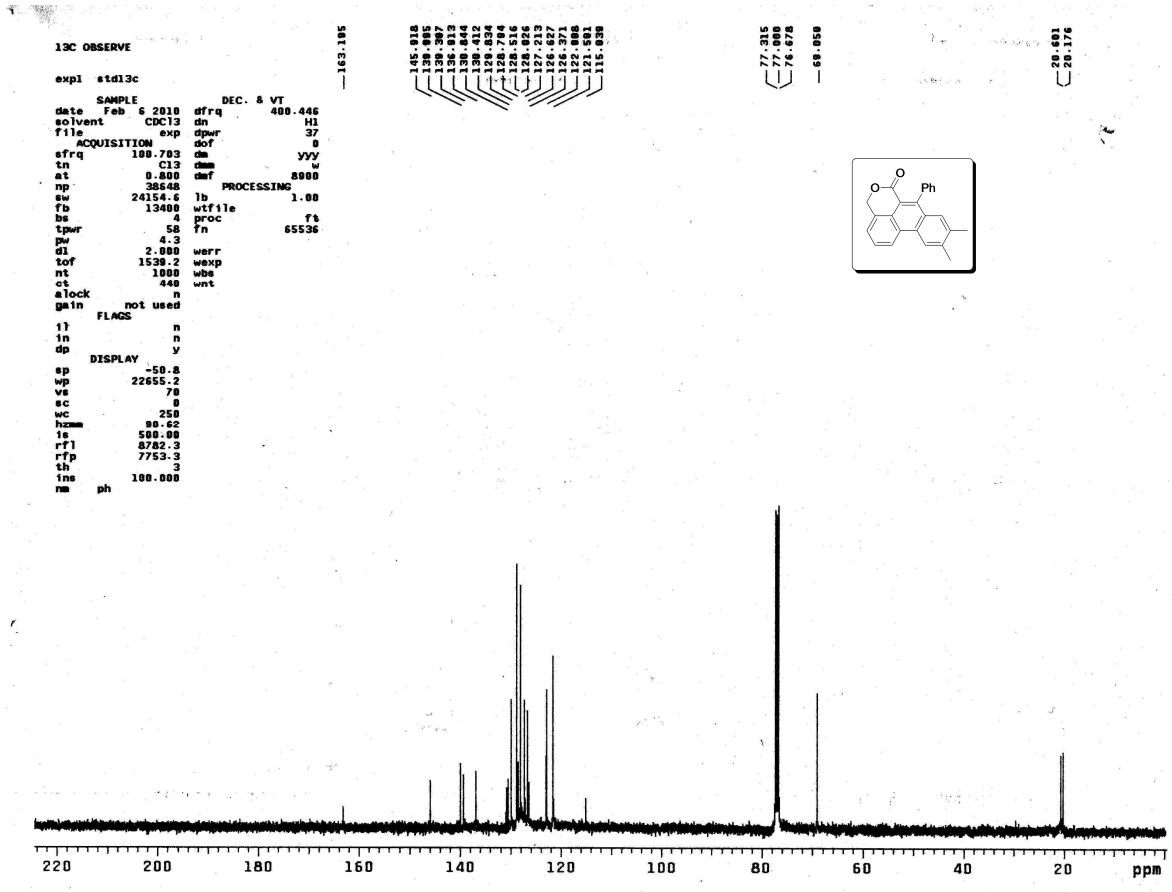
^{1}H and ^{13}C NMR spectra of compound **3a**.



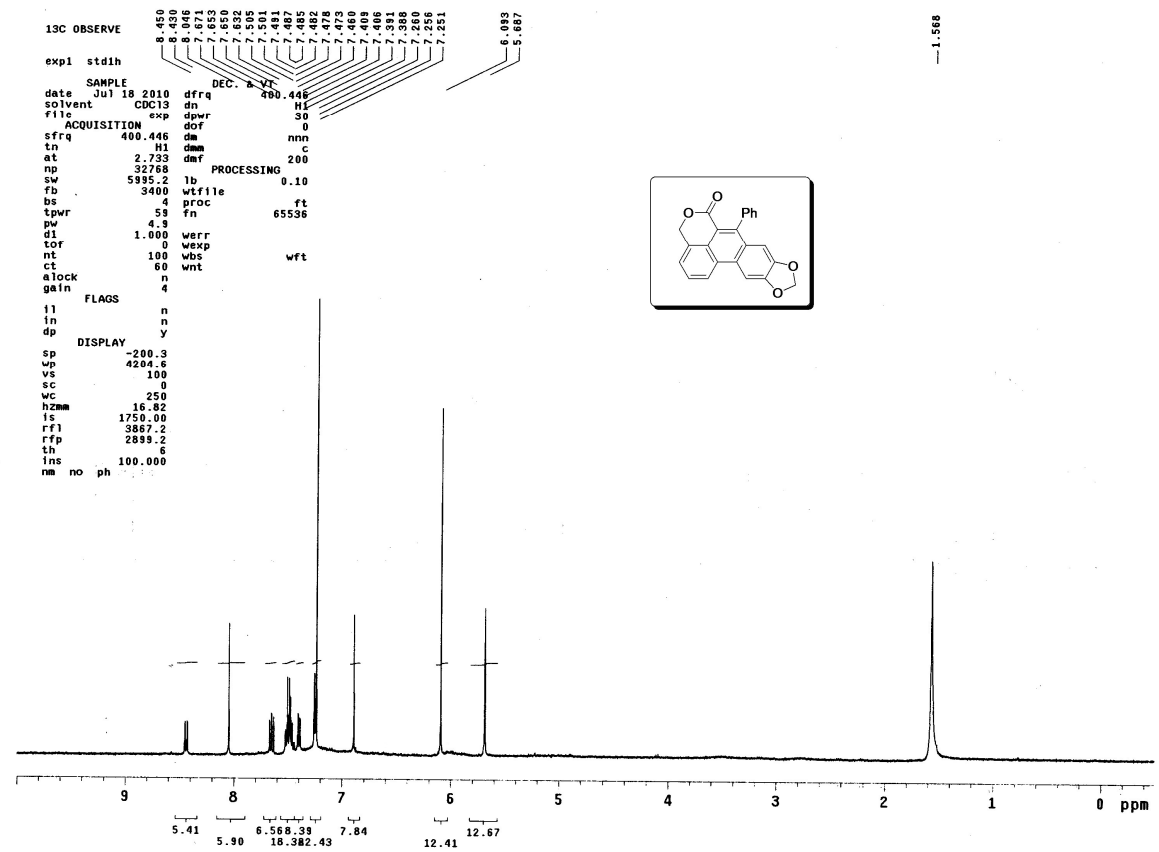


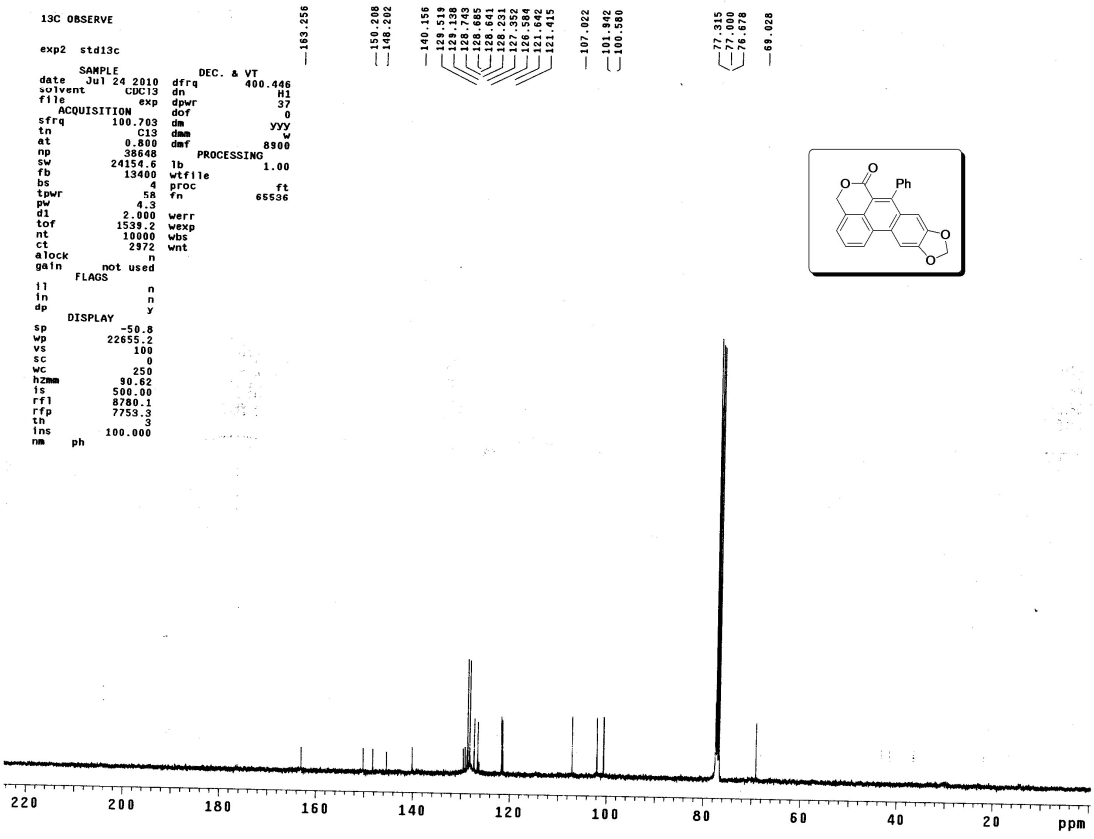
^{1}H and ^{13}C NMR spectra of compound **3b**.



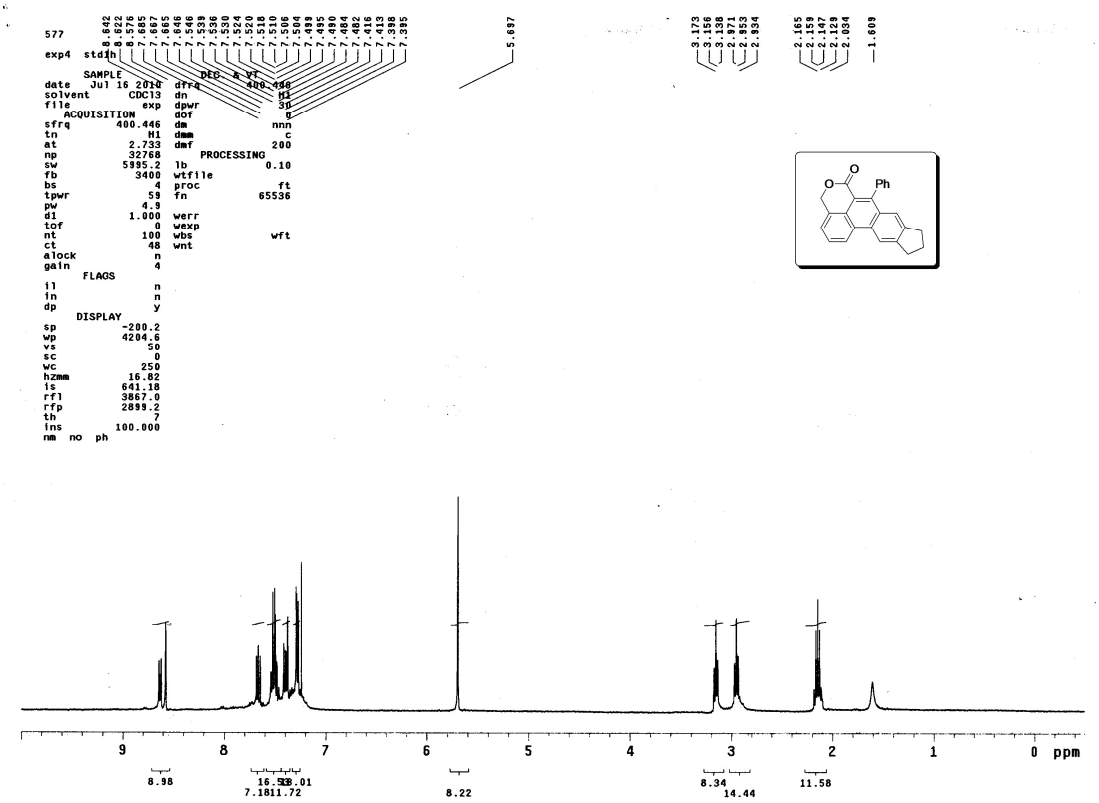


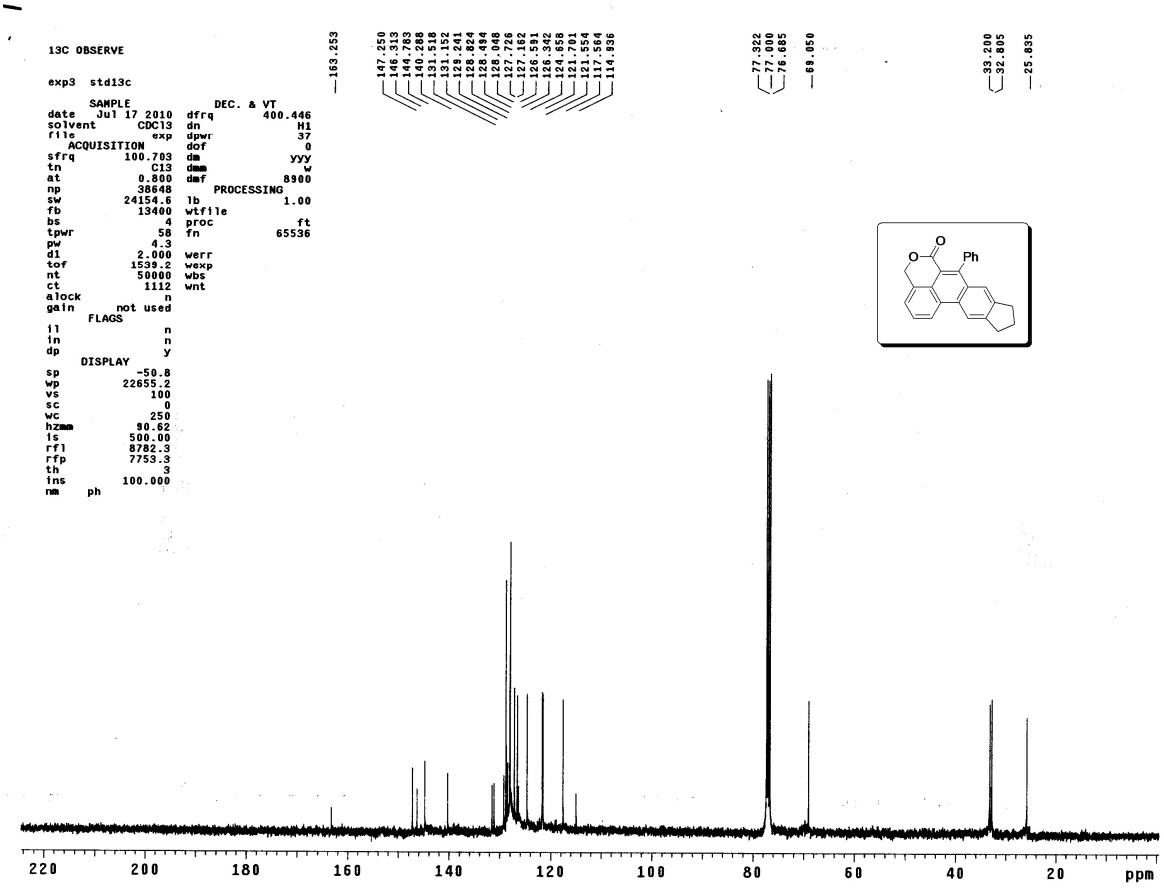
^{1}H and ^{13}C NMR spectra of compound **3c**.



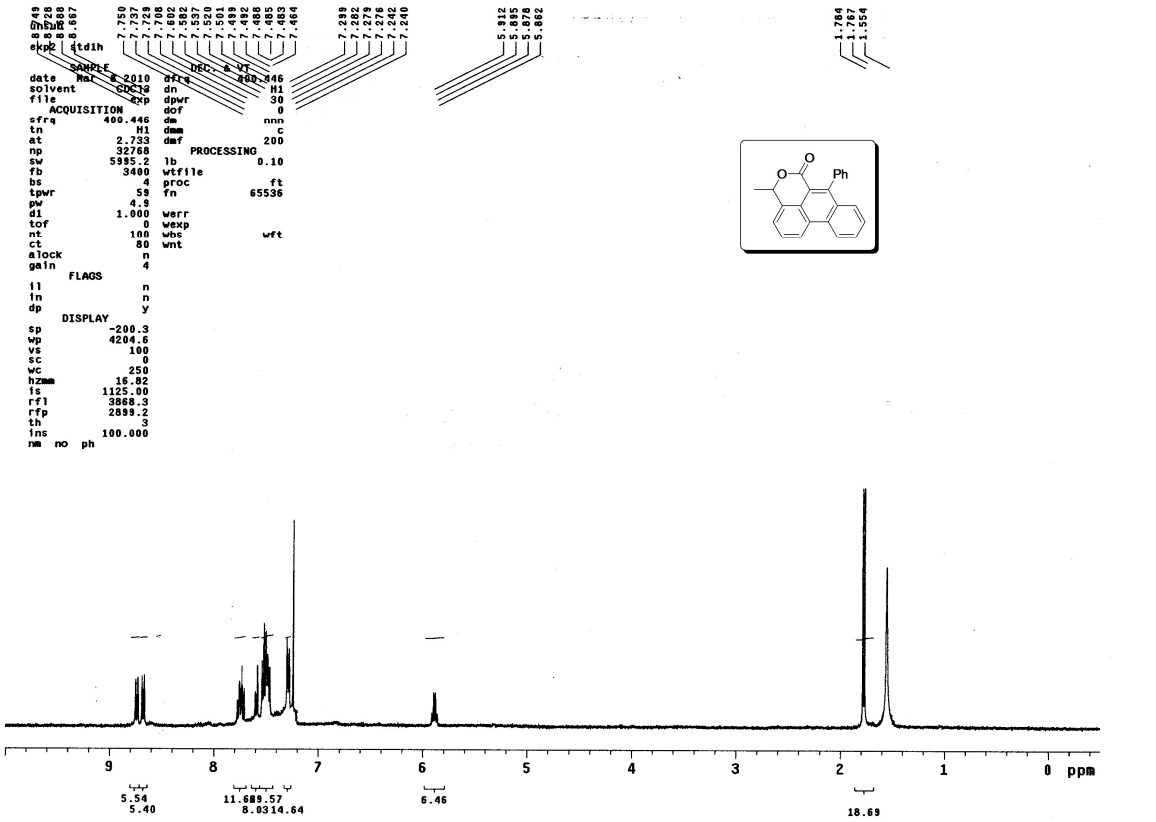


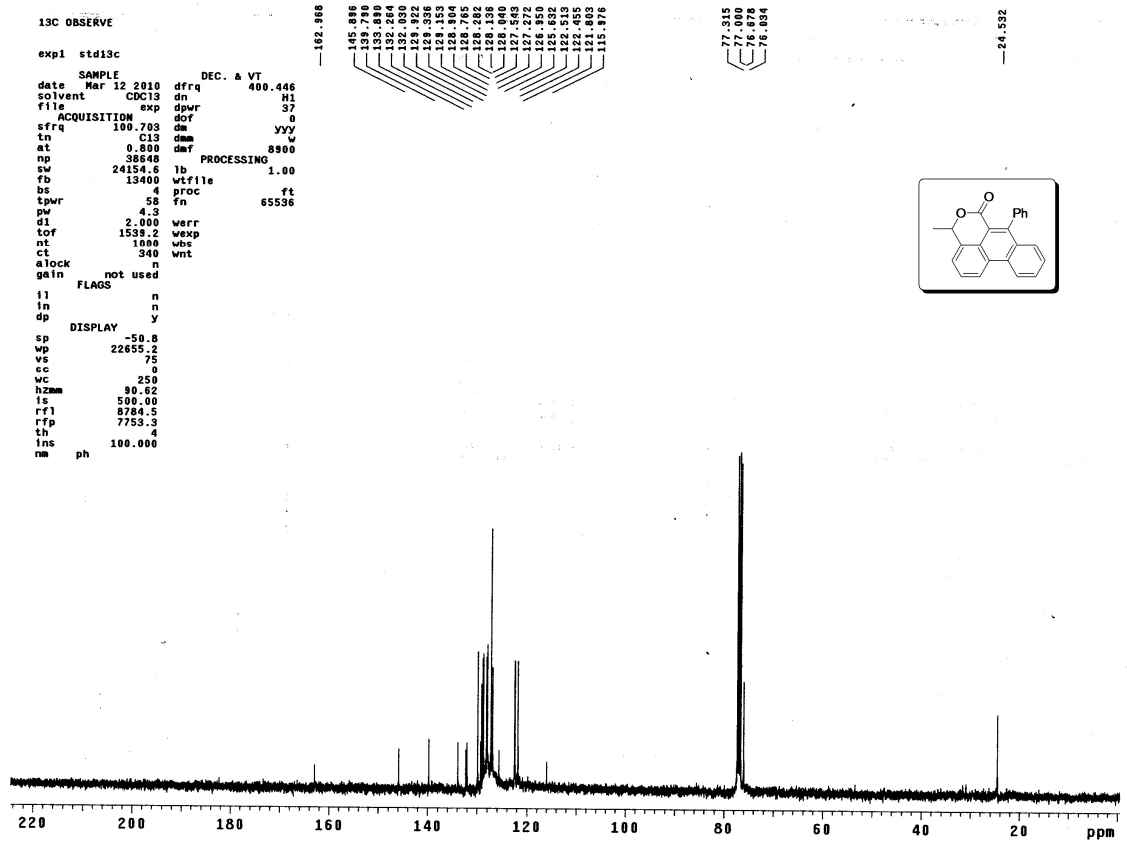
^{1}H and ^{13}C NMR spectra of compound **3d**.



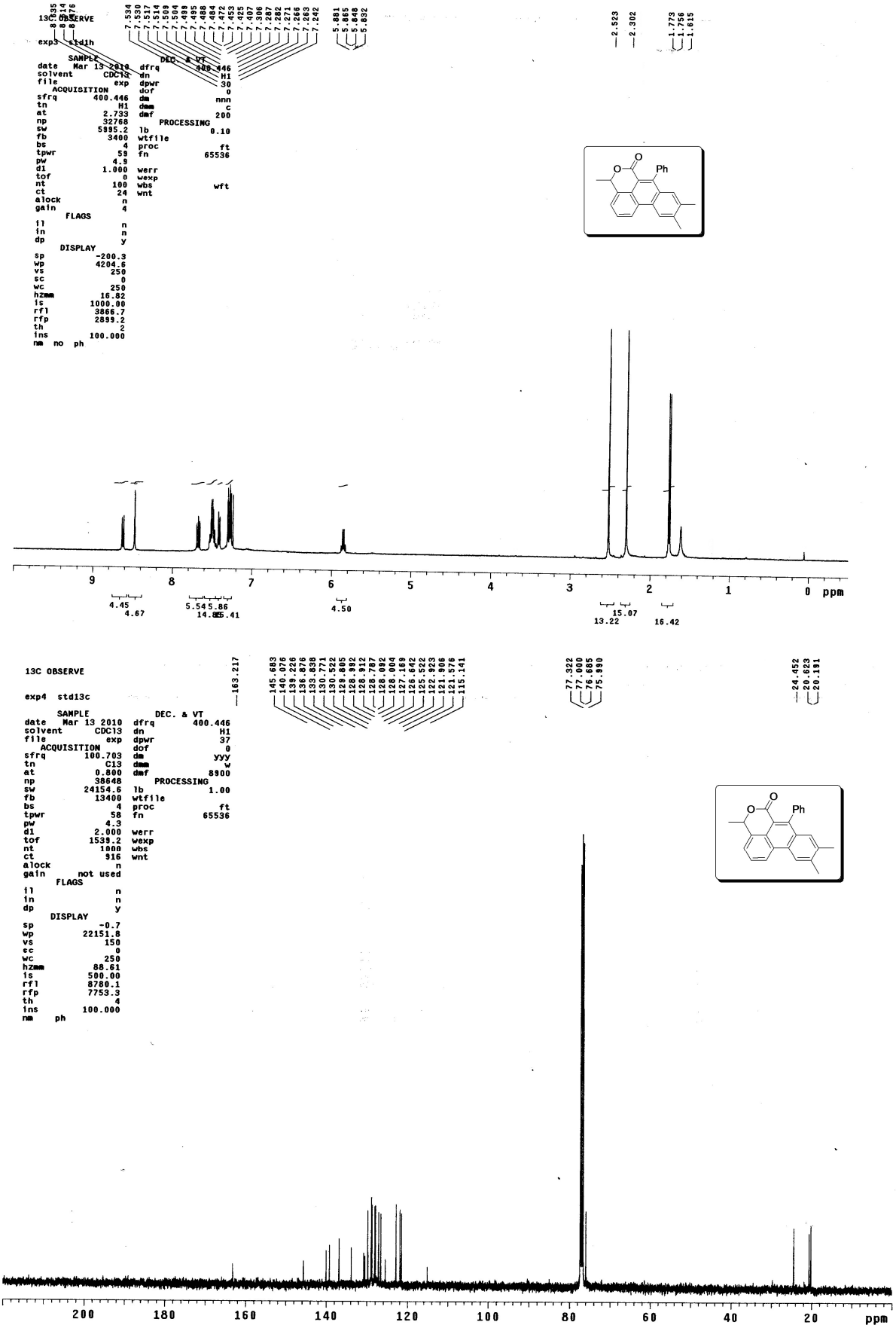


^{1}H and ^{13}C NMR spectra of compound **3e**.

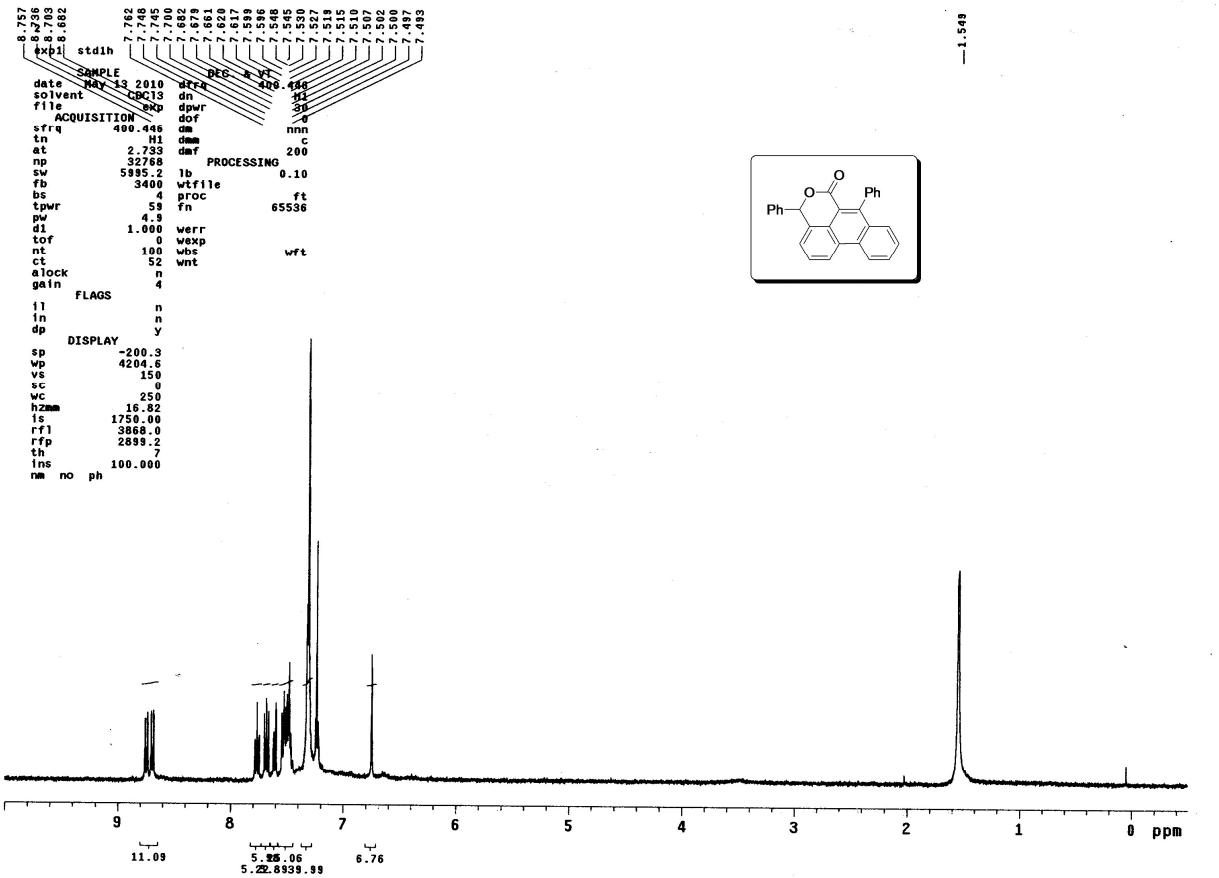


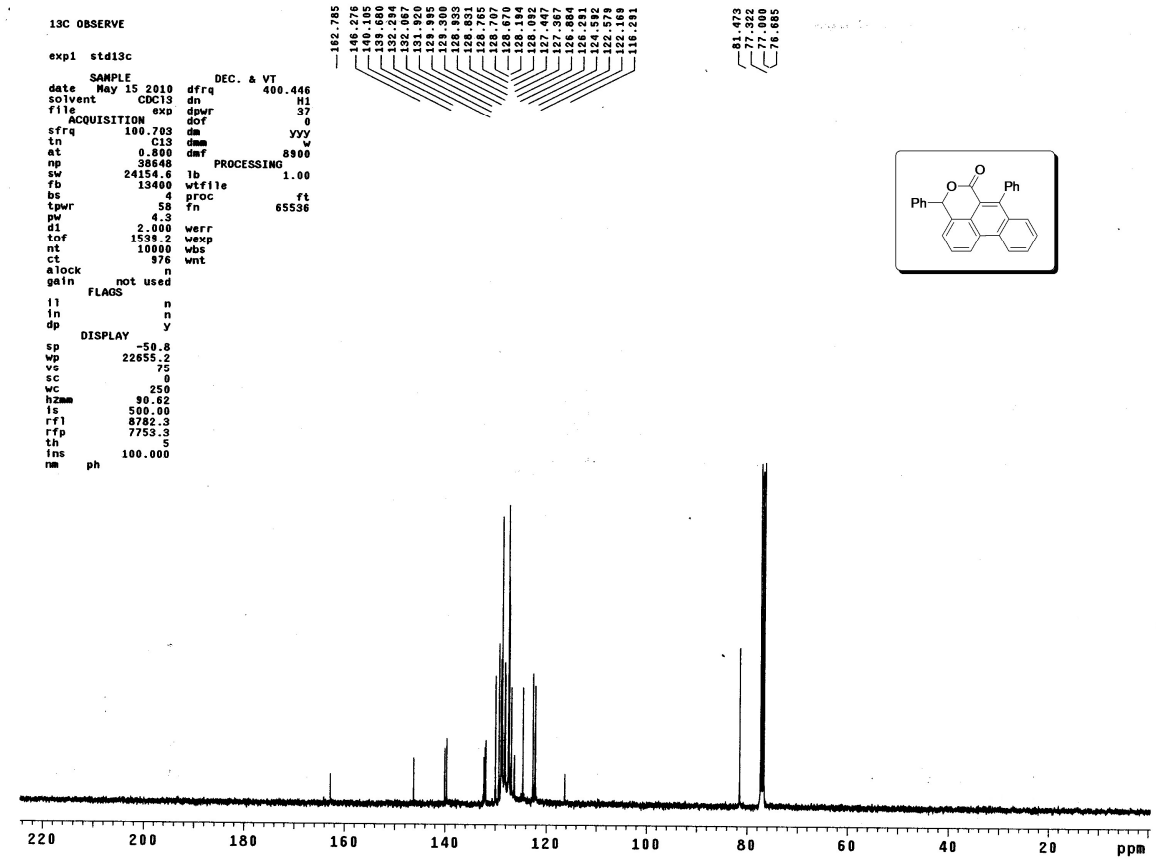


^{1}H and ^{13}C NMR spectra of compound **3f**.

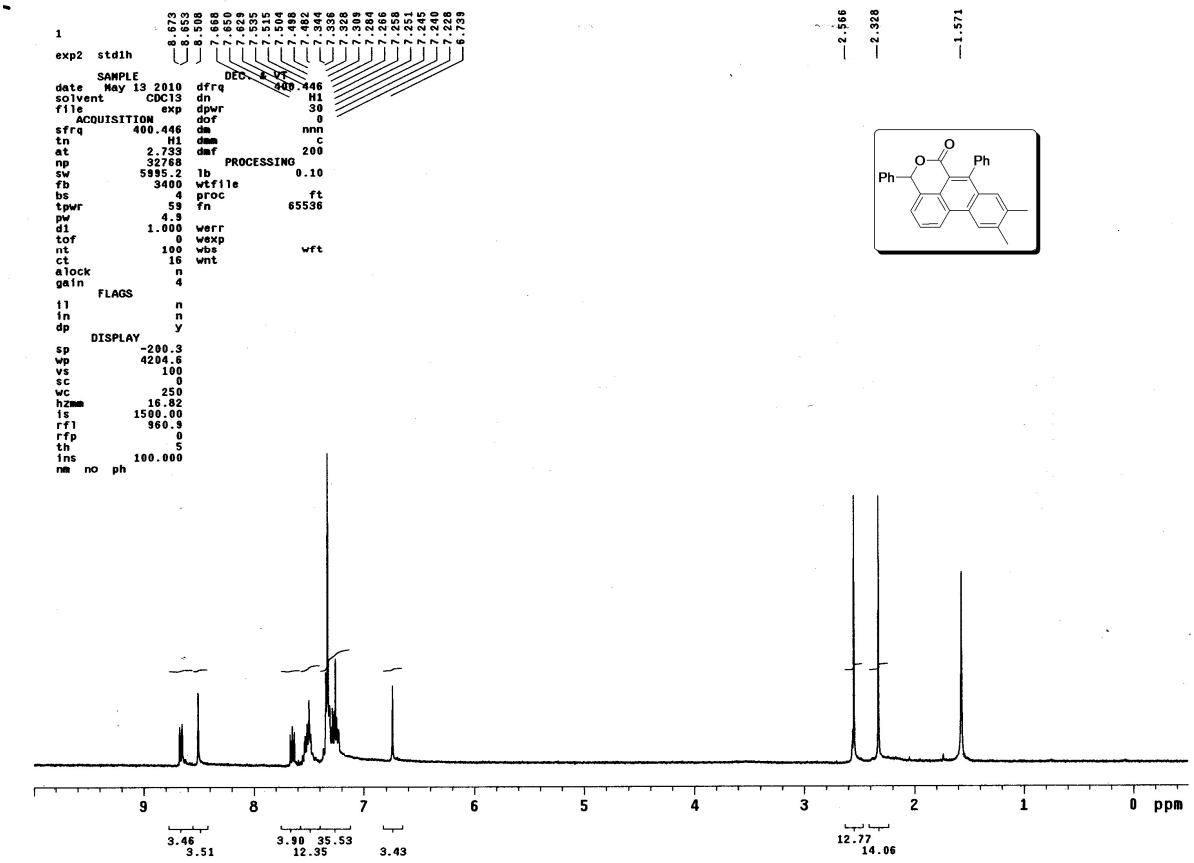


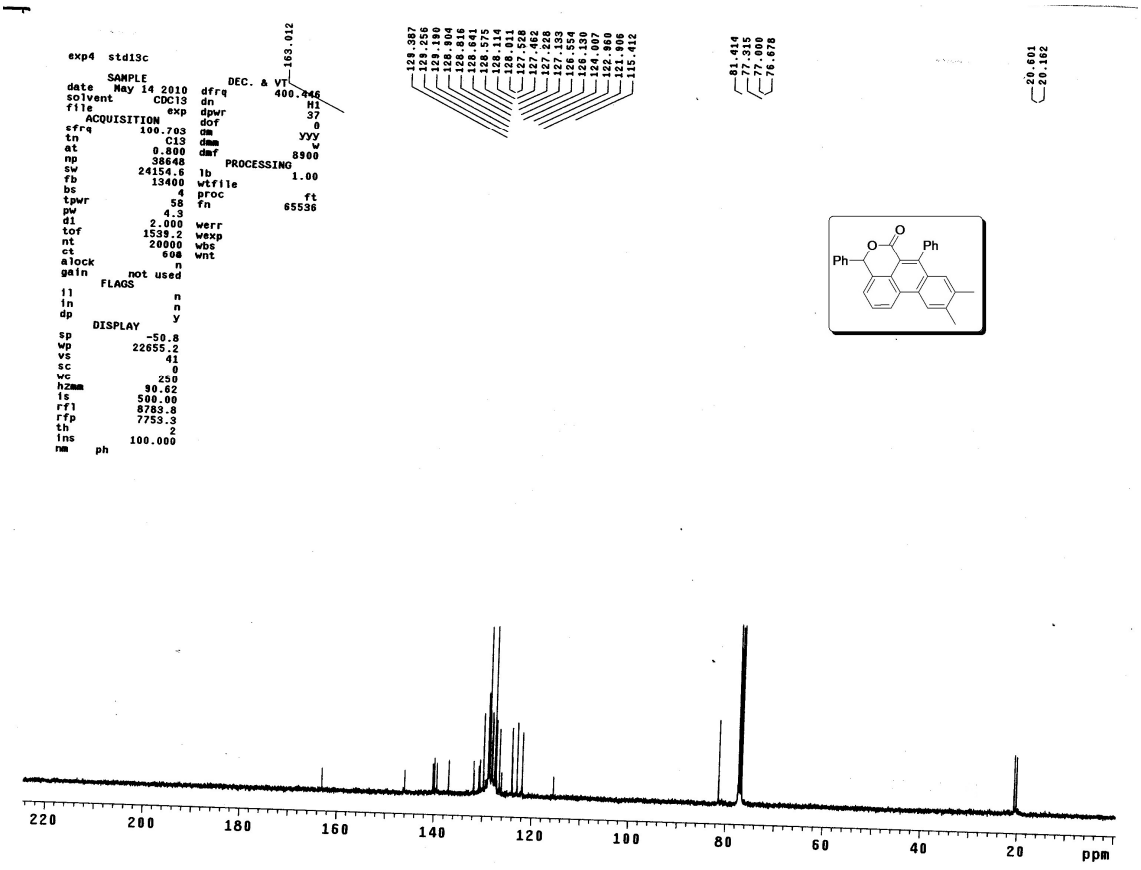
^{1}H and ^{13}C NMR spectra of compound **3g**.



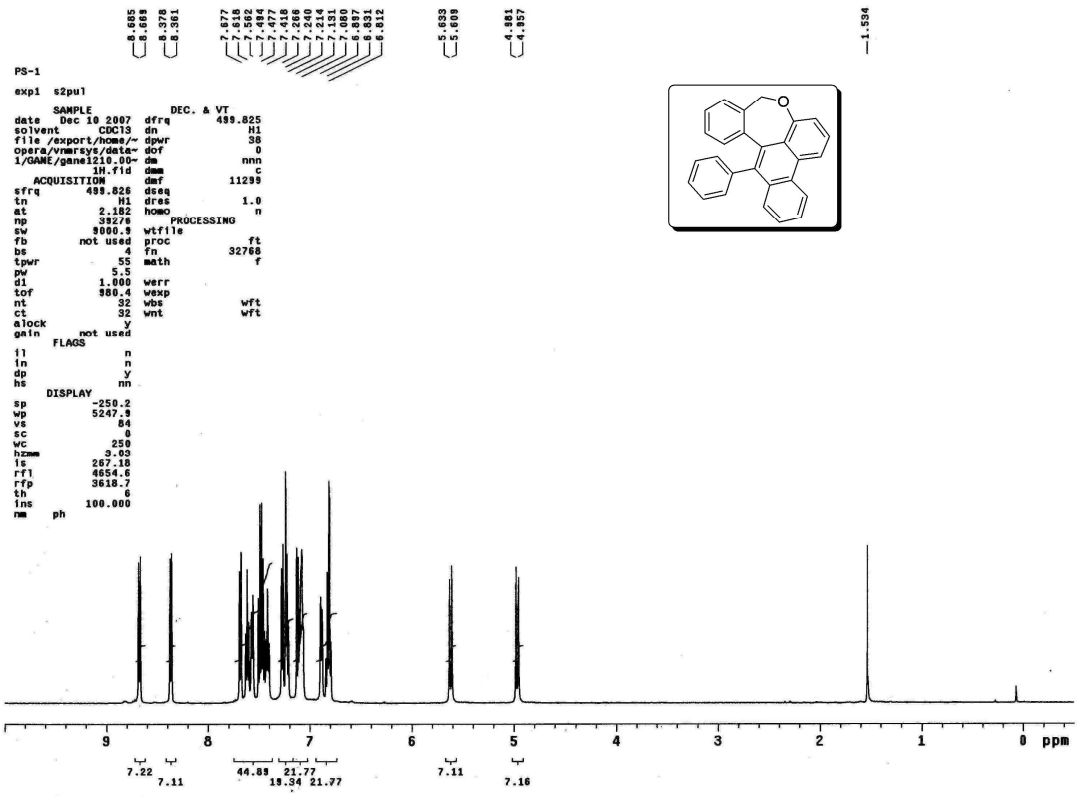


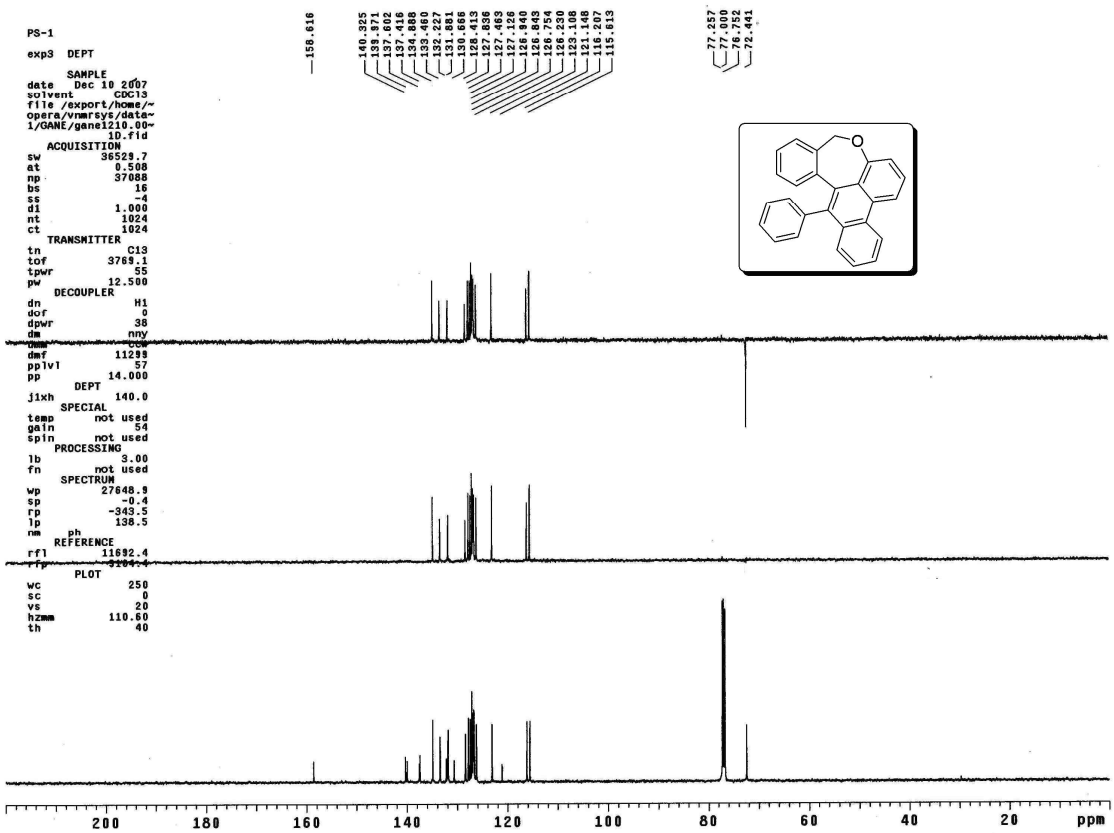
^{1}H and ^{13}C NMR spectra of compound **3h**.



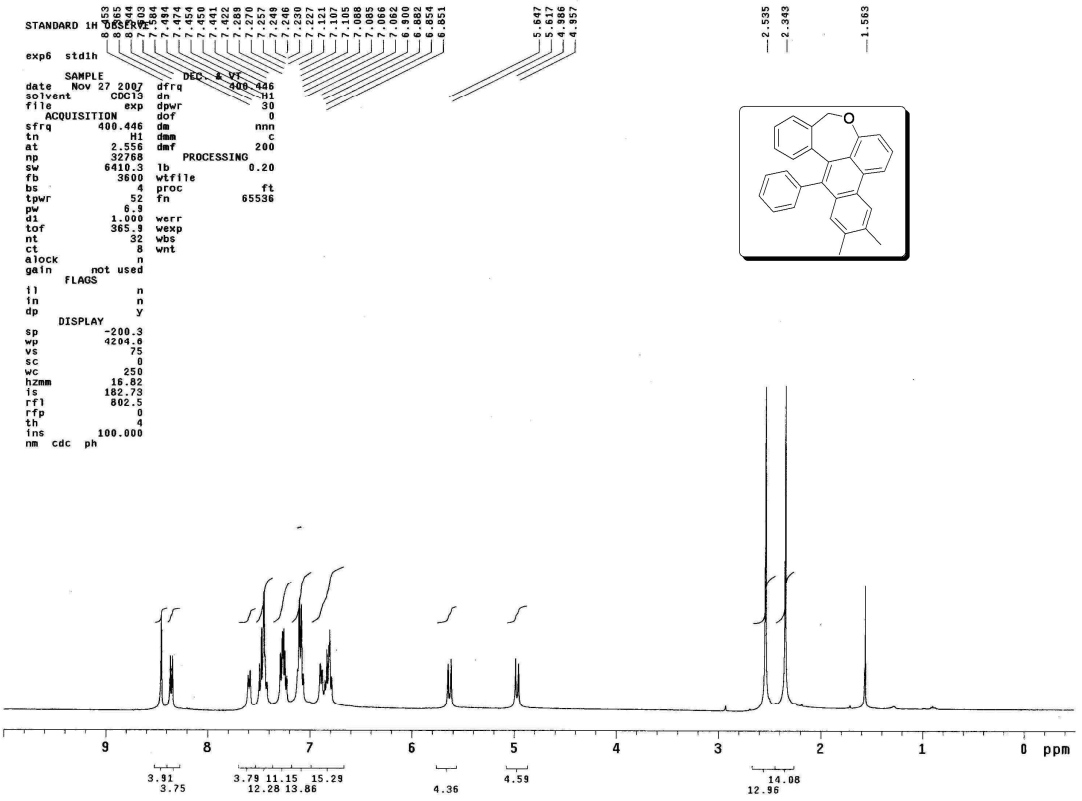


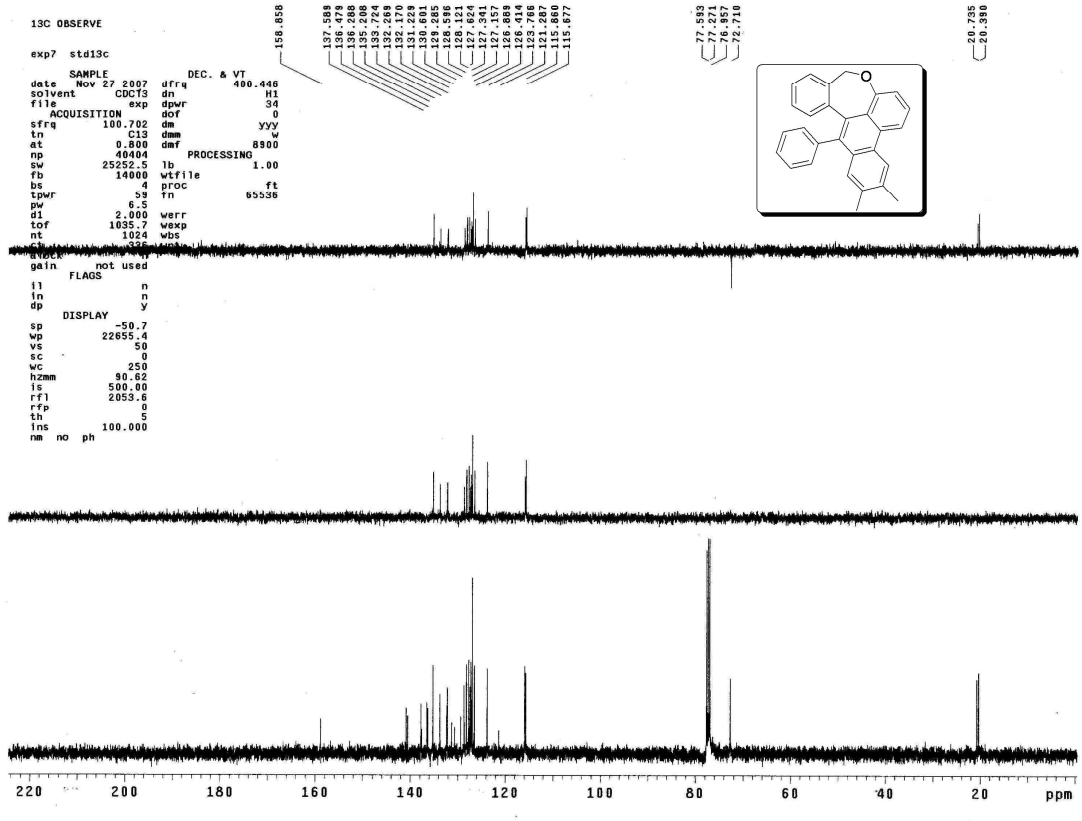
^{1}H and ^{13}C NMR spectra of compound **4a**.



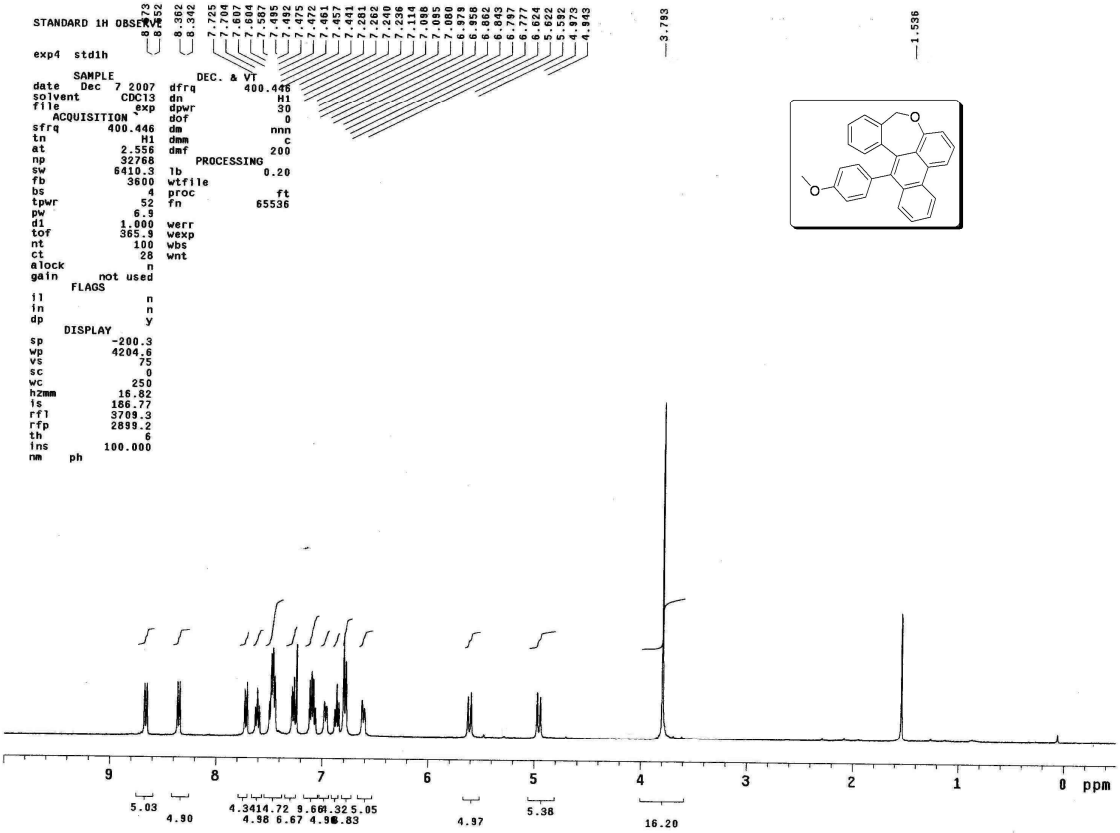


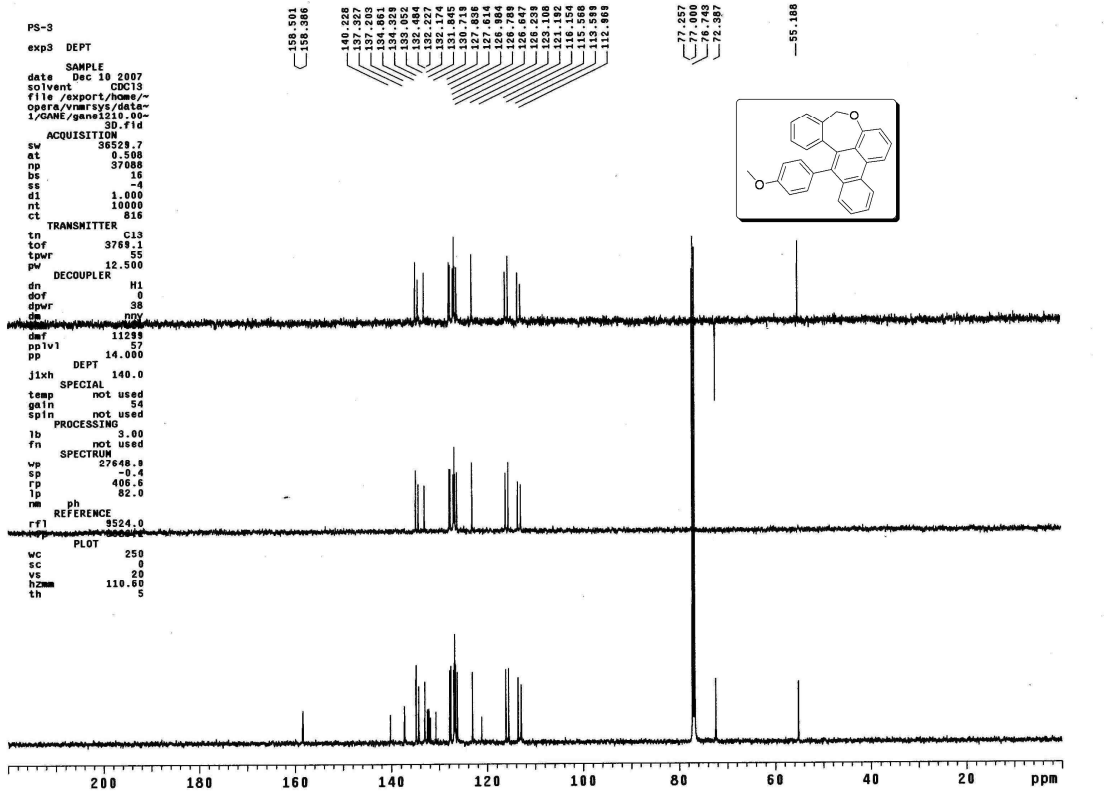
^{1}H and ^{13}C NMR spectra of compound **4b**.



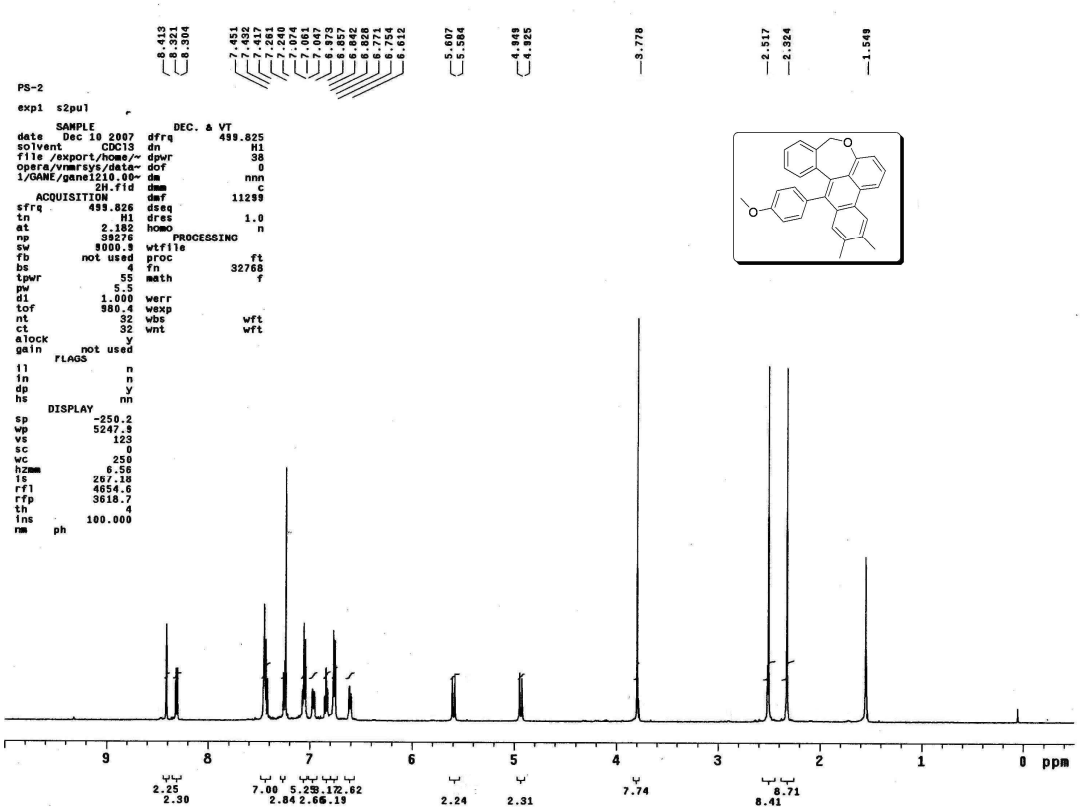


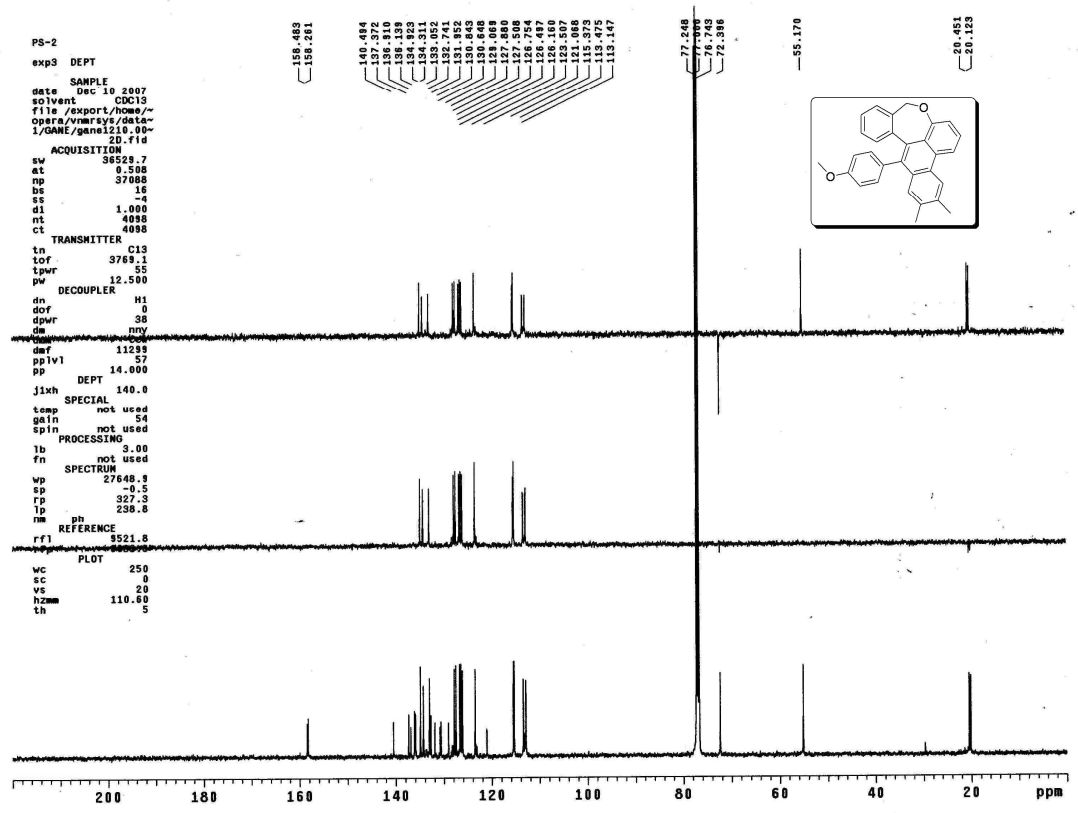
^{1}H and ^{13}C NMR spectra of compound **4c**.



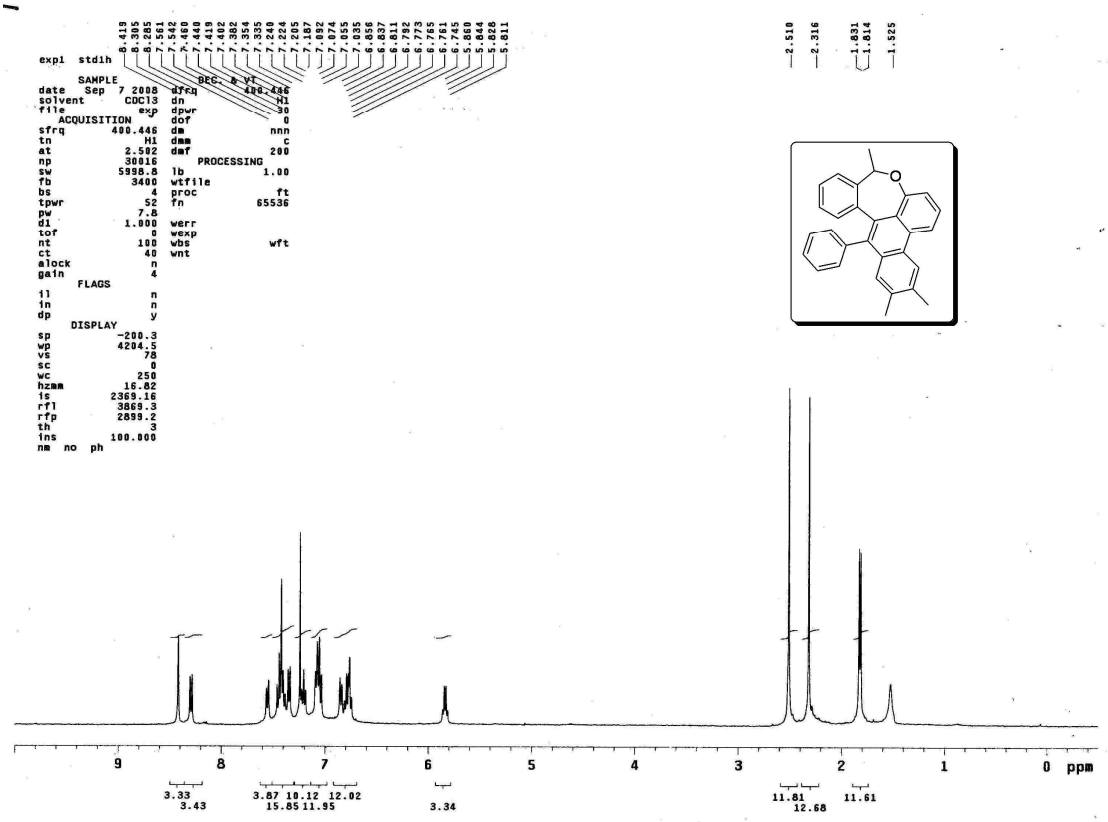


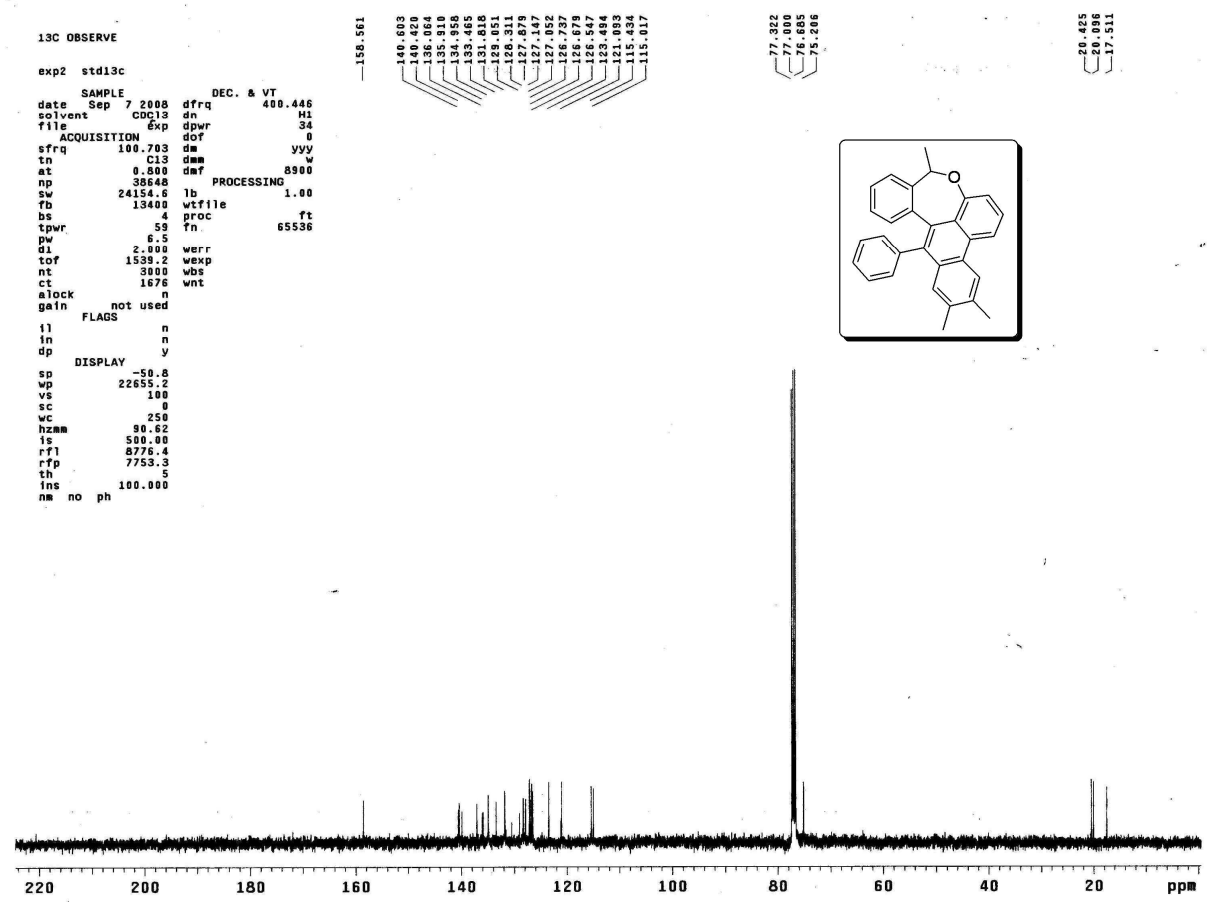
^{1}H and ^{13}C NMR spectra of compound **4d**.



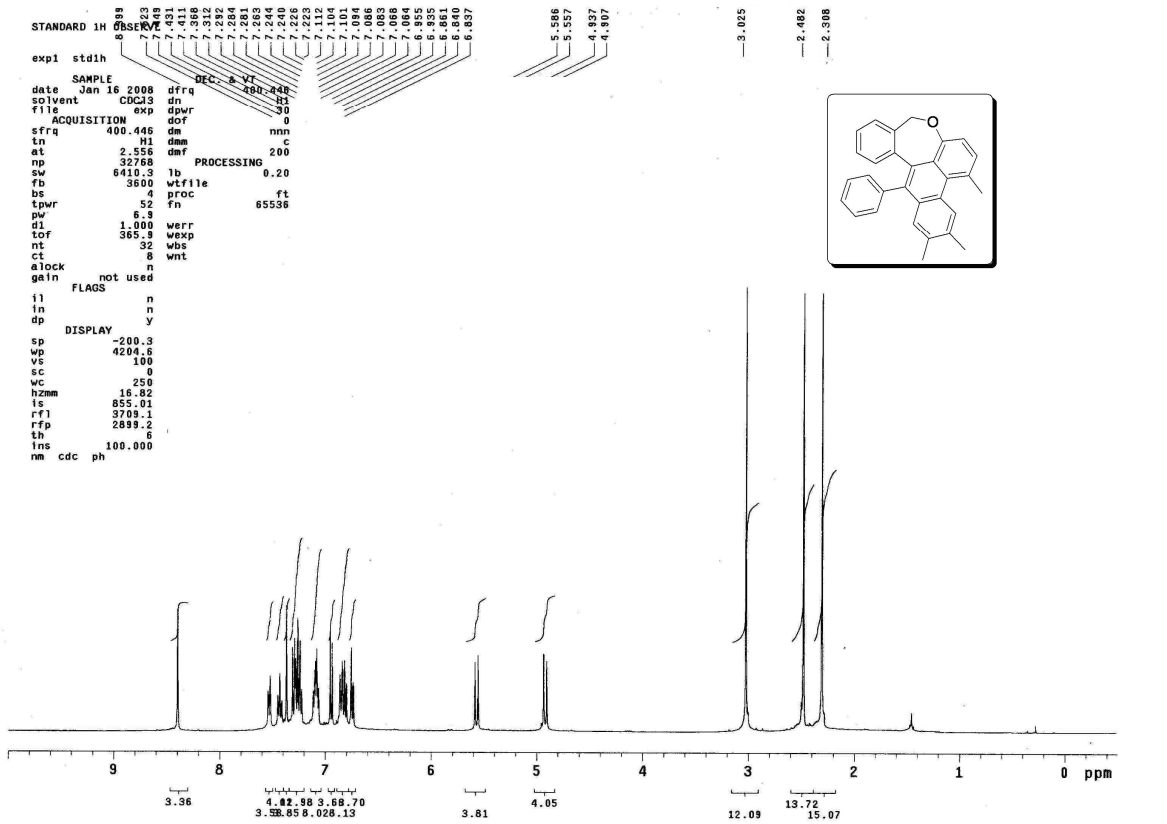


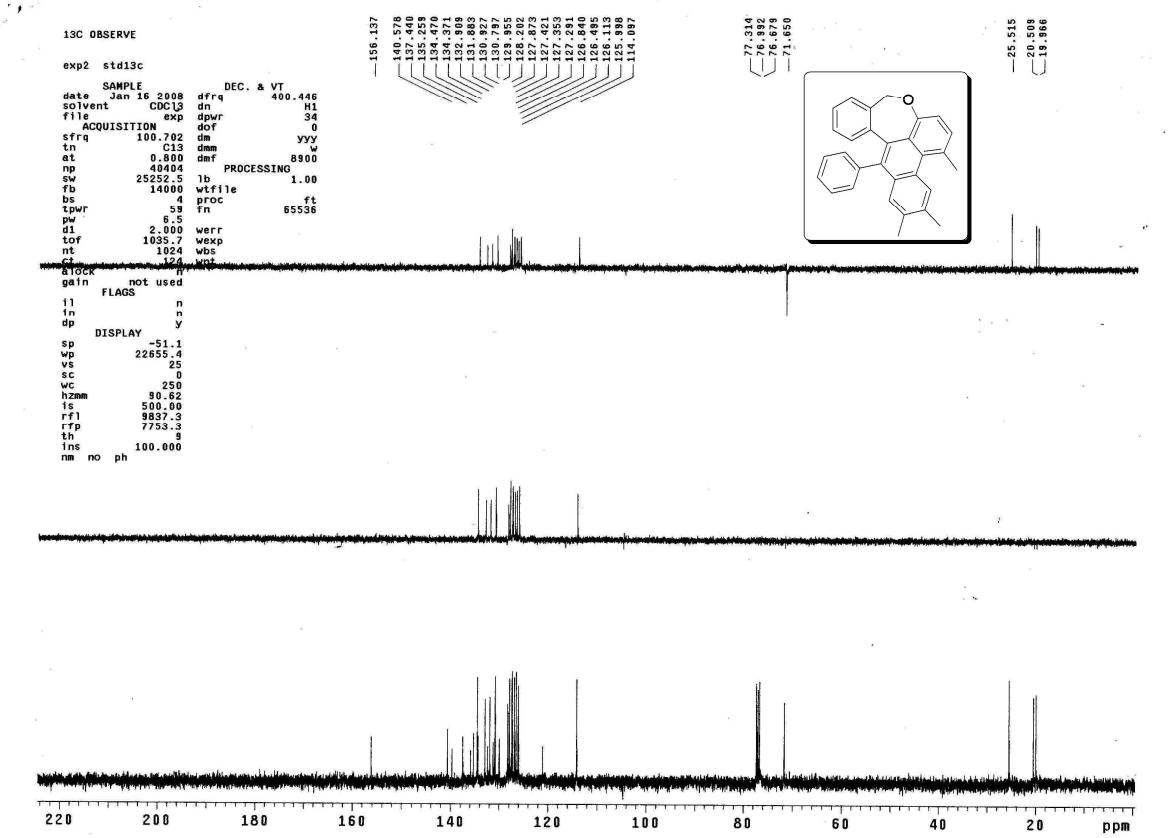
^{1}H and ^{13}C NMR spectra of compound **4e**.



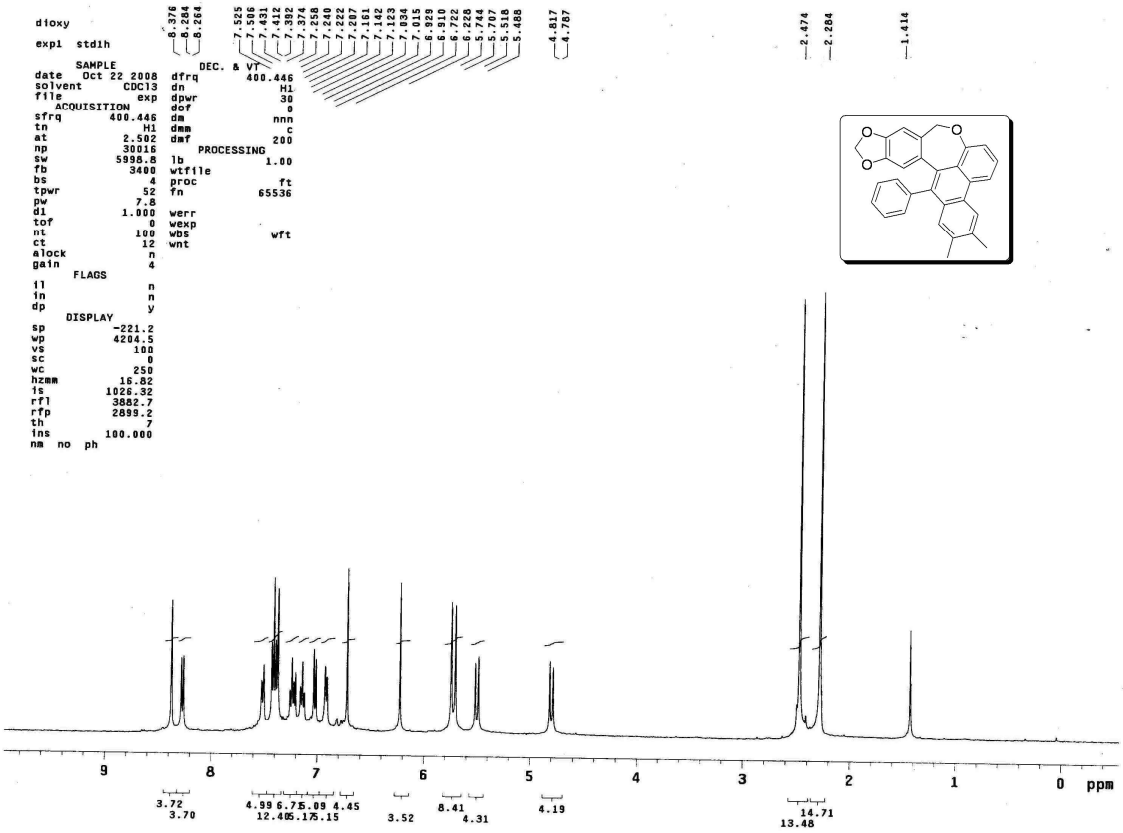


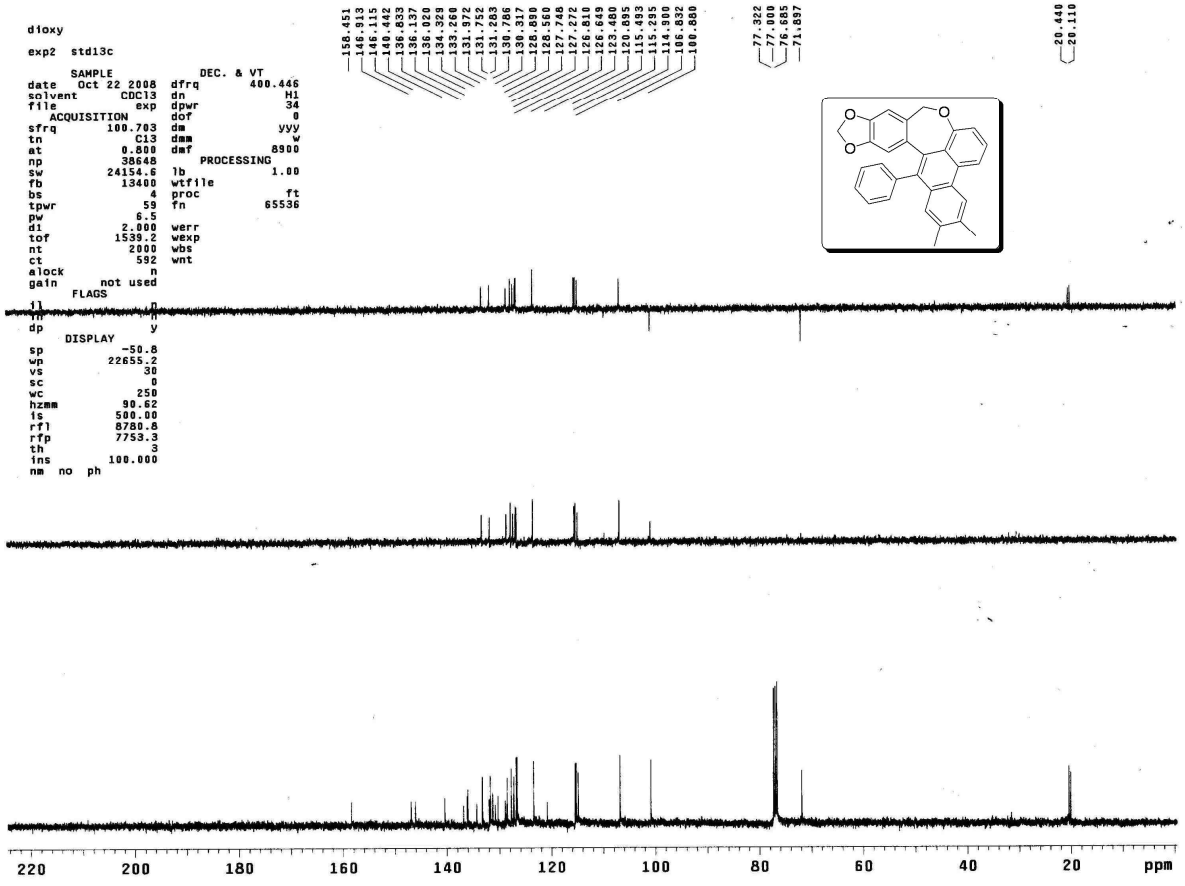
^{1}H and ^{13}C NMR spectra of compound **4f**.



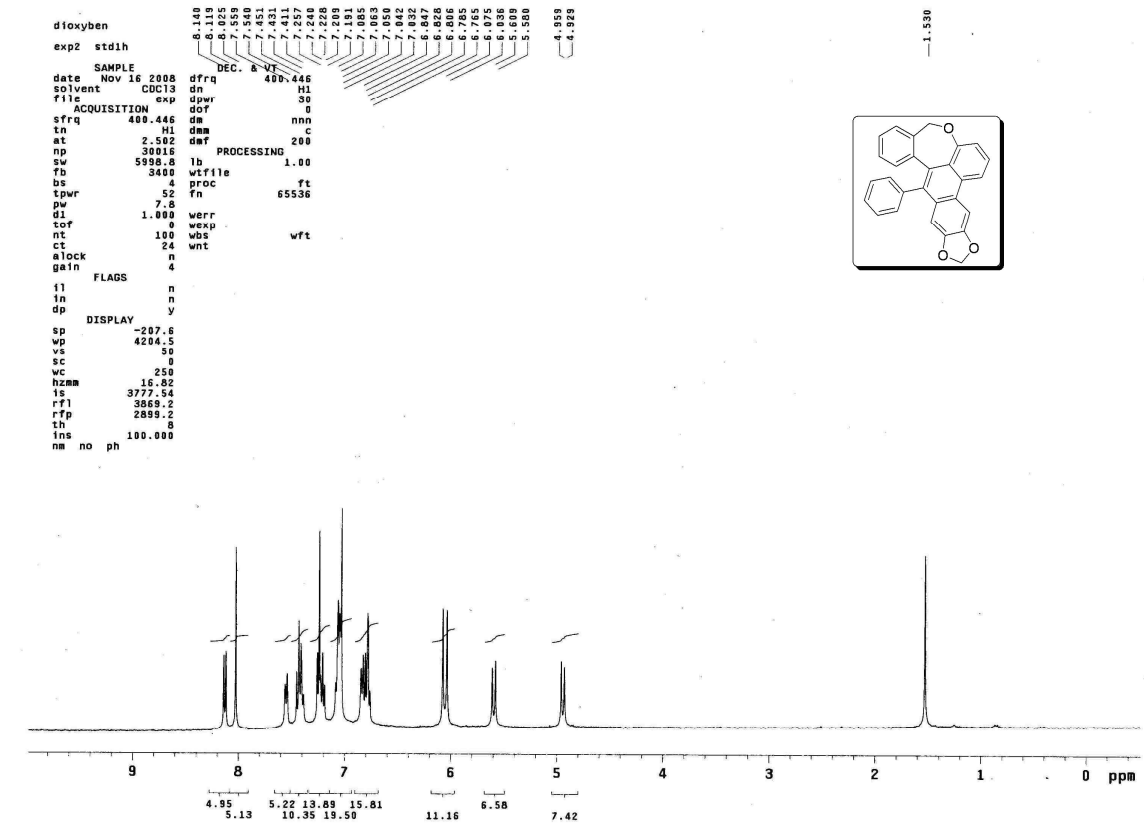


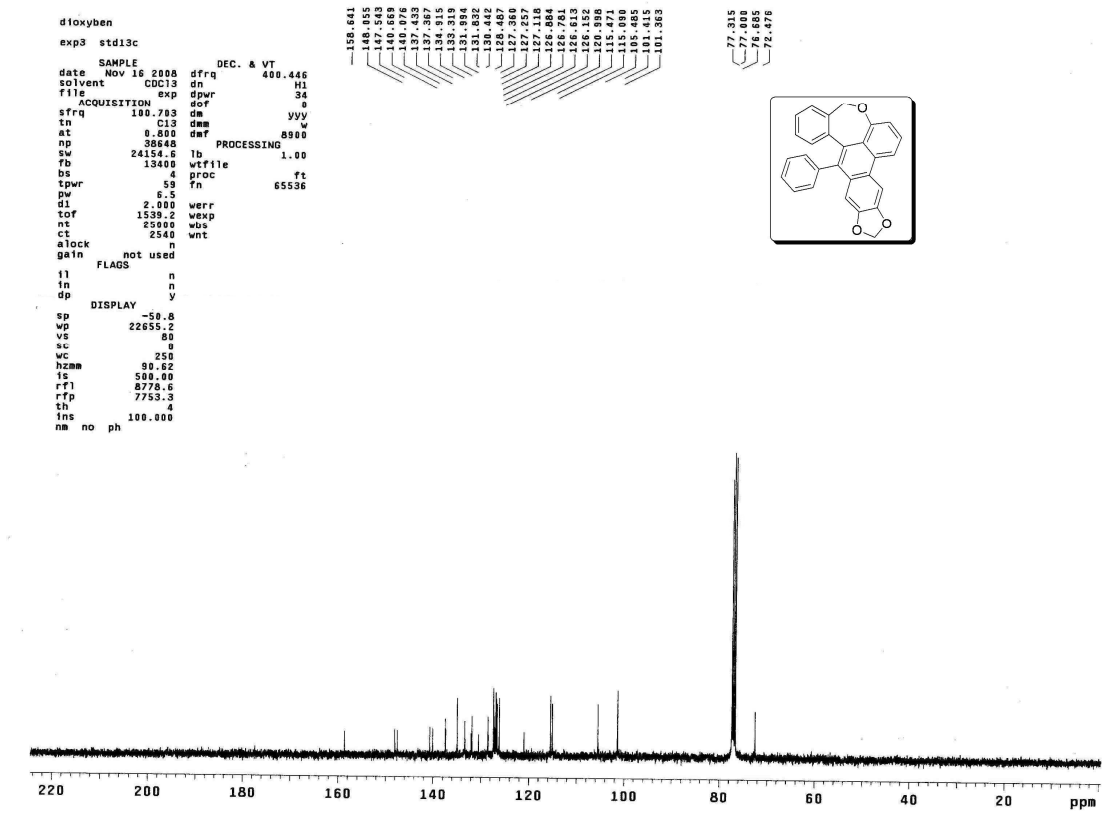
^{1}H and ^{13}C NMR spectra of compound **4g**.



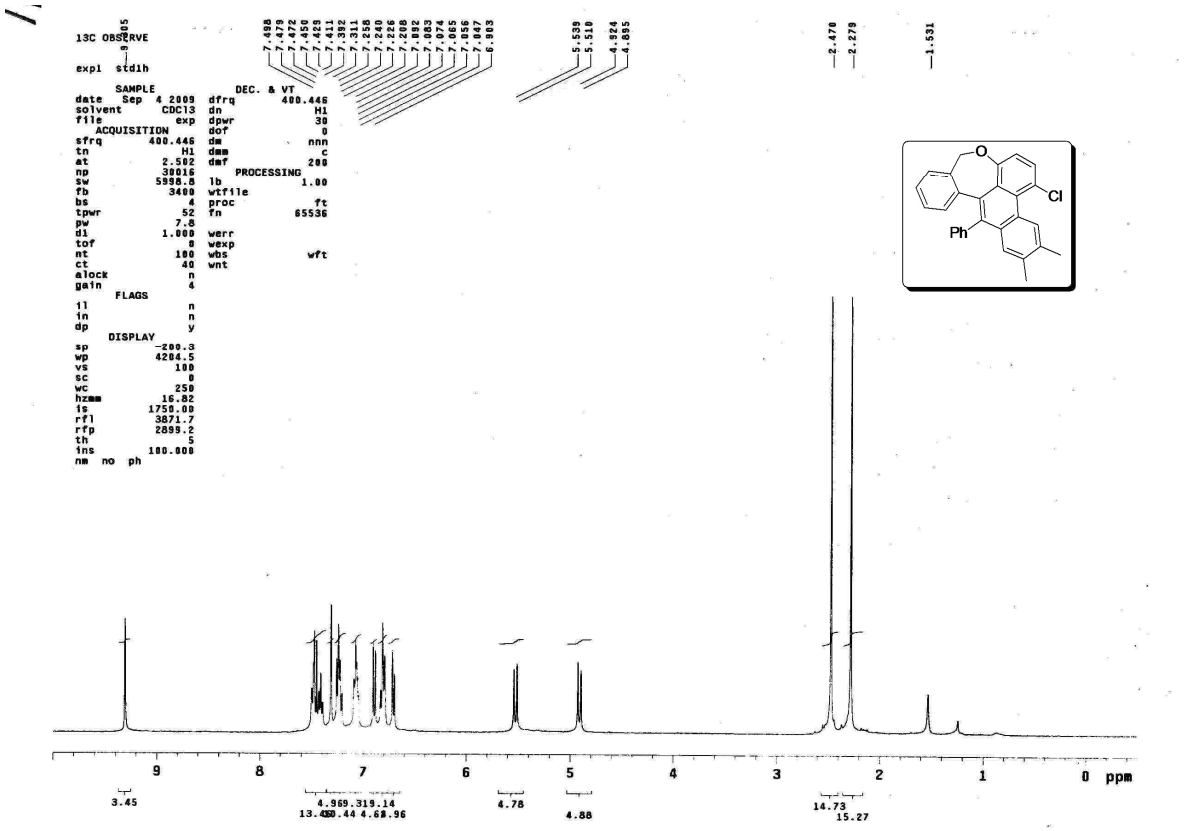


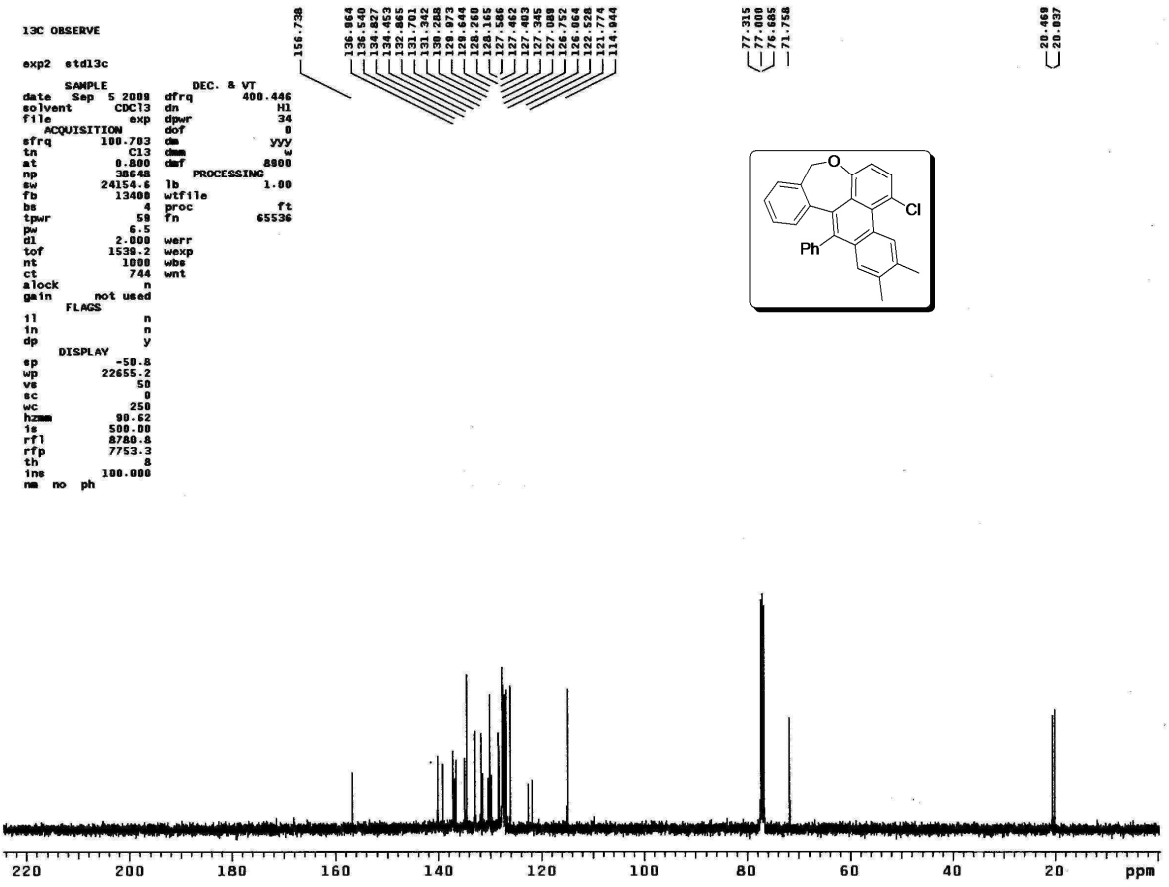
^{1}H and ^{13}C NMR spectra of compound **4h**.



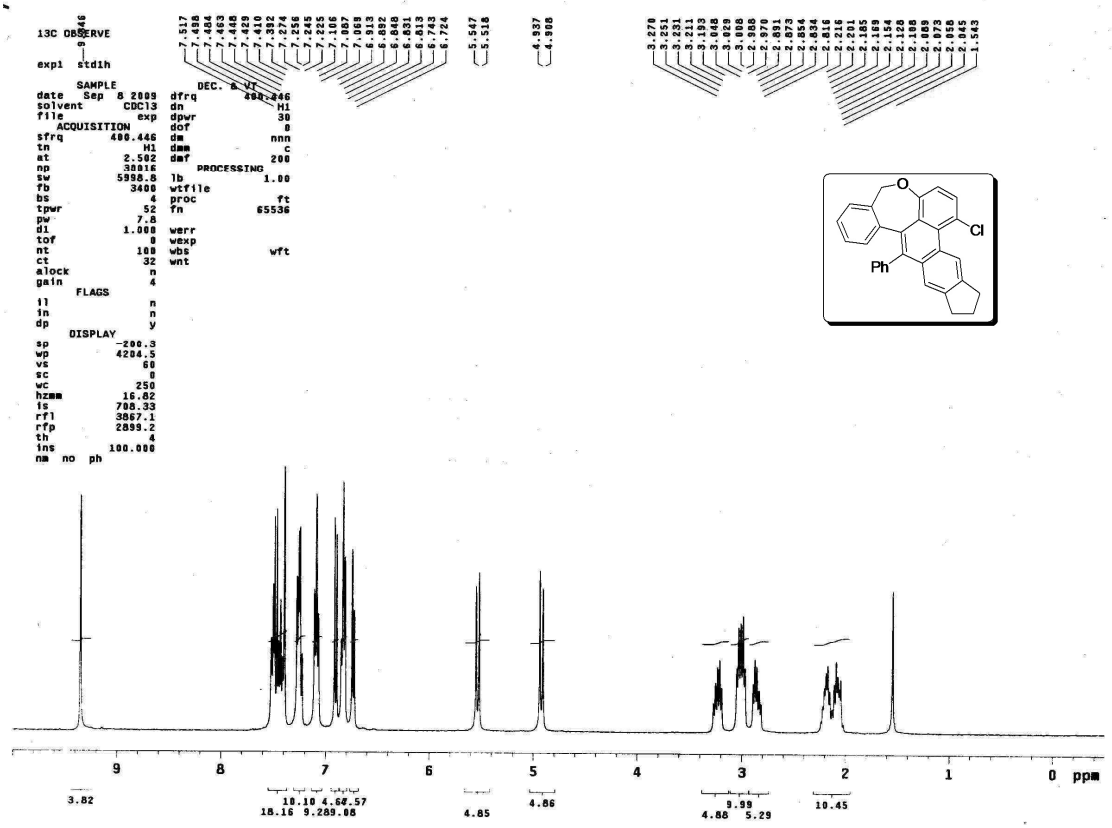


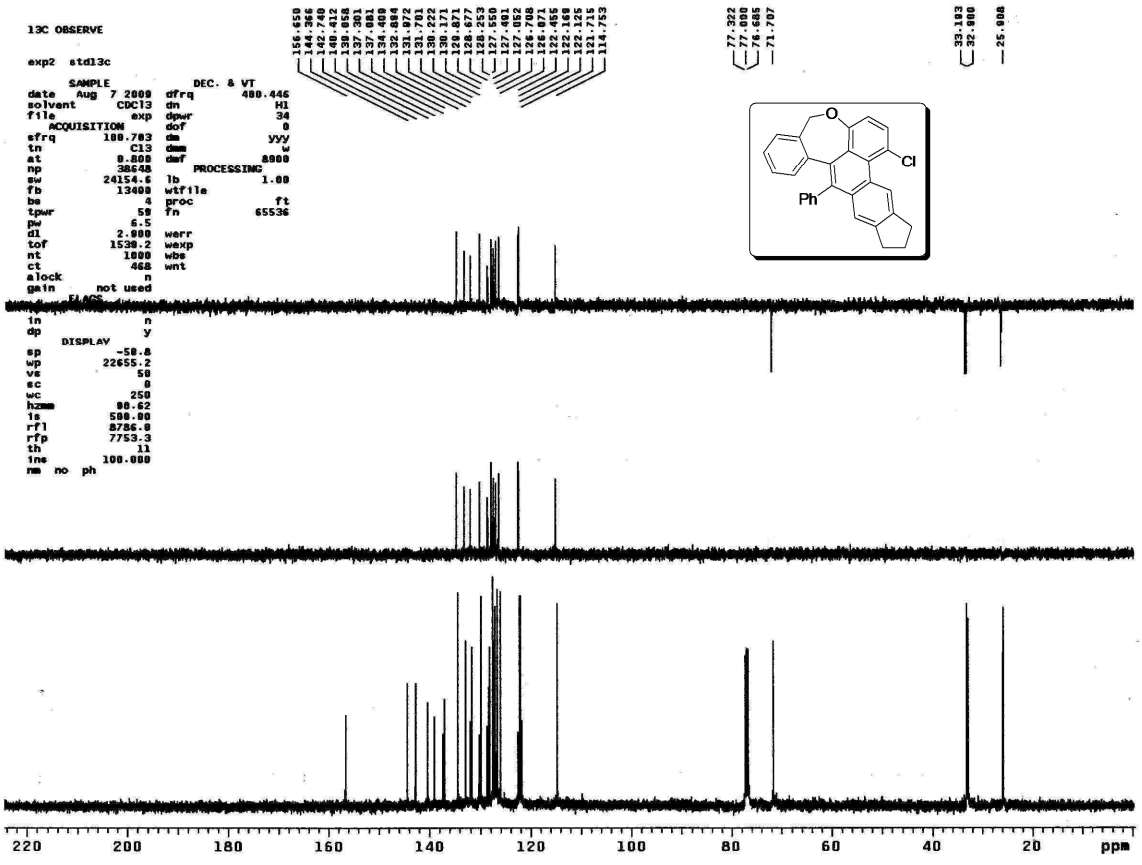
^{1}H and ^{13}C NMR spectra of compound **4i**.



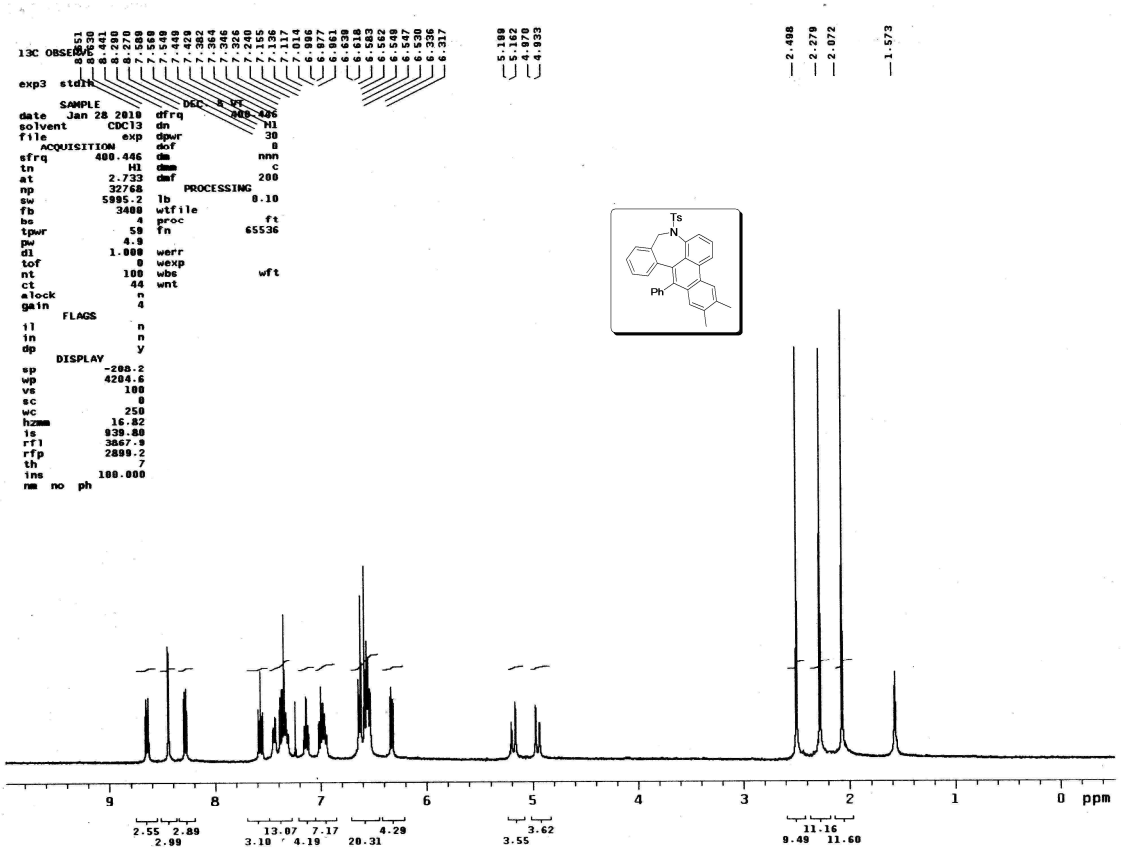


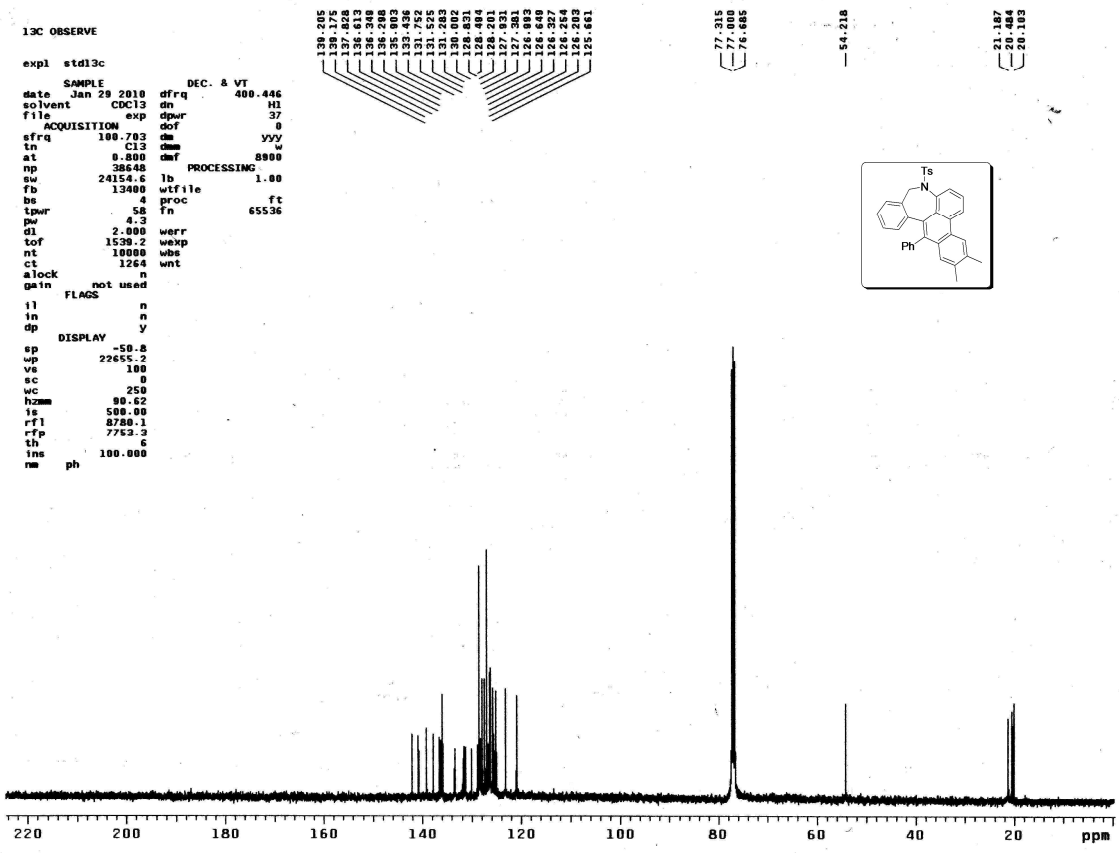
^{1}H and ^{13}C NMR spectra of compound **4j**.



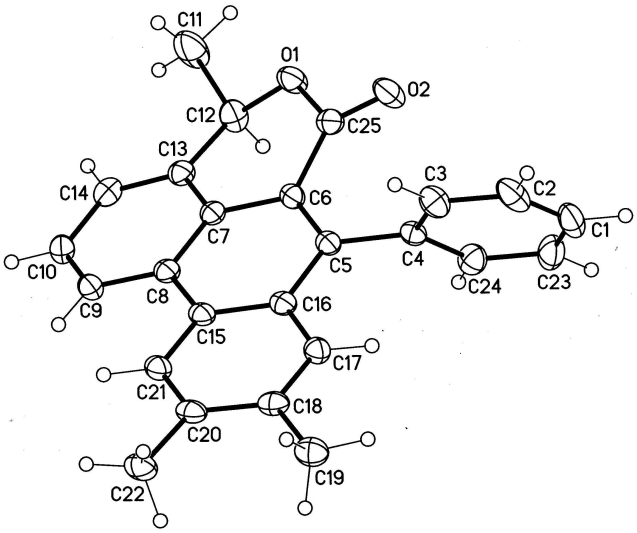


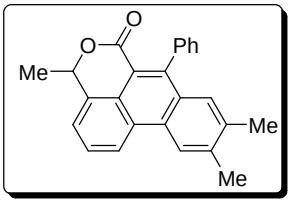
. ^{1}H and ^{13}C NMR spectra of compound **4k**.



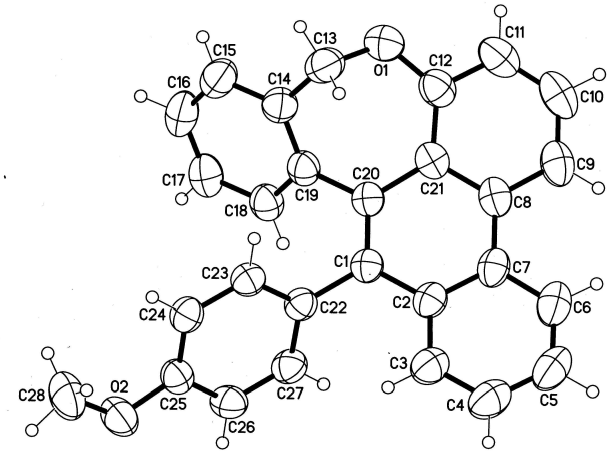


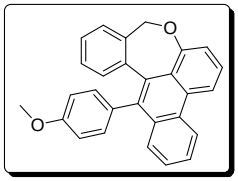
ORTEP diagram of compound **3f**.



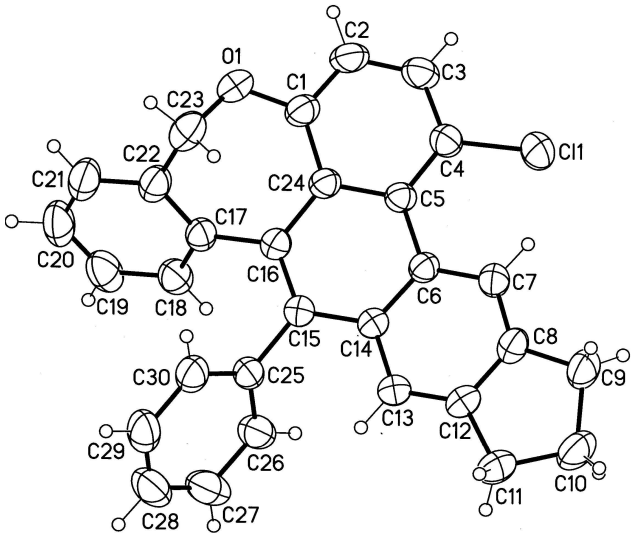


ORTEP diagram of compound **4c**.





ORTEP diagram of compound **4j**.



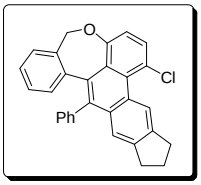


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Table 1. Crystal data and structure refinement for **3f**.

Identification code	111103lt	
Empirical formula	C25 H20 O2	
Formula weight	352.41	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P n a 21	
Unit cell dimensions	a = 18.289(4) Å	α= 90°.
	b = 17.374(4) Å	β= 90°.
	c = 5.6562(12) Å	γ = 90°.
Volume	1797.3(7) $Å^3$	
Z	4	
Density (calculated)	1.302 Mg/m^3	
Absorption coefficient	0.081 mm^{-1}	
F(000)	744	
Crystal size	0.25 x 0.07 x 0.06 mm^3	
Theta range for data collection	1.62 to 26.54°.	
Index ranges	-22<=h<=22, -21<=k<=21, -6<=l<=7	
Reflections collected	14636	
Independent reflections	3186 [R(int) = 0.0308]	
Completeness to theta = 26.54°	99.4 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9486 and 0.8891	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3186 / 1 / 247	
Goodness-of-fit on F^2	1.041	
Final R indices [I>2sigma(I)]	R1 = 0.0319, wR2 = 0.0755	
R indices (all data)	R1 = 0.0366, wR2 = 0.0781	
Absolute structure parameter	2.0(12)	
Largest diff. peak and hole	0.229 and -0.167 $e.Å^{-3}$	

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Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 111103LT. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	2206(1)	6898(1)	12855(2)	27(1)
O(2)	2960(1)	6279(1)	10538(2)	31(1)
C(1)	5035(1)	5849(1)	7228(4)	36(1)
C(2)	4500(1)	6033(1)	5597(4)	34(1)
C(3)	4015(1)	6627(1)	6056(3)	28(1)
C(4)	4055(1)	7040(1)	8151(3)	20(1)
C(5)	3546(1)	7700(1)	8630(3)	19(1)
C(6)	2985(1)	7652(1)	10248(3)	19(1)
C(7)	2557(1)	8326(1)	10835(3)	19(1)
C(8)	2644(1)	9018(1)	9552(3)	20(1)
C(9)	2233(1)	9668(1)	10236(3)	24(1)
C(10)	1777(1)	9639(1)	12165(3)	26(1)
C(11)	1310(1)	7426(1)	15380(5)	49(1)
C(12)	2036(1)	7579(1)	14235(3)	28(1)
C(13)	2071(1)	8302(1)	12780(3)	22(1)
C(14)	1696(1)	8953(1)	13438(3)	25(1)
C(15)	3179(1)	9041(1)	7650(3)	20(1)
C(16)	3652(1)	8403(1)	7299(3)	20(1)
C(17)	4202(1)	8465(1)	5552(3)	22(1)
C(18)	4269(1)	9098(1)	4113(3)	23(1)
C(19)	4851(1)	9133(1)	2236(3)	30(1)
C(20)	3771(1)	9720(1)	4396(3)	23(1)
C(21)	3250(1)	9683(1)	6160(3)	23(1)
C(22)	3808(1)	10415(1)	2812(4)	30(1)
C(23)	5086(1)	6260(1)	9311(4)	35(1)
C(24)	4600(1)	6859(1)	9773(3)	27(1)
C(25)	2748(1)	6896(1)	11216(3)	21(1)

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Table 3. Bond lengths [Å] and angles [°] for 111103LT.

O(1)-C(25)	1.3561(19)
O(1)-C(12)	1.451(2)
O(2)-C(25)	1.2027(19)
C(1)-C(23)	1.382(3)
C(1)-C(2)	1.383(3)
C(1)-H(1)	0.9500
C(2)-C(3)	1.385(2)
C(2)-H(2)	0.9500
C(3)-C(4)	1.387(2)
C(3)-H(3)	0.9500
C(4)-C(24)	1.390(2)
C(4)-C(5)	1.502(2)
C(5)-C(6)	1.377(2)
C(5)-C(16)	1.448(2)
C(6)-C(7)	1.447(2)
C(6)-C(25)	1.489(2)
C(7)-C(8)	1.413(2)
C(7)-C(13)	1.415(2)
C(8)-C(9)	1.412(2)
C(8)-C(15)	1.455(2)
C(9)-C(10)	1.375(2)
C(9)-H(9)	0.9500
C(10)-C(14)	1.400(2)
C(10)-H(10)	0.9500
C(11)-C(12)	1.499(2)
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800
C(11)-H(11C)	0.9800
C(12)-C(13)	1.504(2)
C(12)-H(12)	1.0000
C(13)-C(14)	1.374(2)
C(14)-H(14)	0.9500
C(15)-C(21)	1.404(2)
C(15)-C(16)	1.419(2)
C(16)-C(17)	1.415(2)
C(17)-C(18)	1.374(2)

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C(17)-H(17)	0.9500
C(18)-C(20)	1.421(2)
C(18)-C(19)	1.504(2)
C(19)-H(19A)	0.9800
C(19)-H(19B)	0.9800
C(19)-H(19C)	0.9800
C(20)-C(21)	1.381(2)
C(20)-C(22)	1.505(2)
C(21)-H(21)	0.9500
C(22)-H(22A)	0.9800
C(22)-H(22B)	0.9800
C(22)-H(22C)	0.9800
C(23)-C(24)	1.393(2)
C(23)-H(23)	0.9500
C(24)-H(24)	0.9500

C(25)-O(1)-C(12)	121.88(12)
C(23)-C(1)-C(2)	119.84(16)
C(23)-C(1)-H(1)	120.1
C(2)-C(1)-H(1)	120.1
C(1)-C(2)-C(3)	120.0(2)
C(1)-C(2)-H(2)	120.0
C(3)-C(2)-H(2)	120.0
C(2)-C(3)-C(4)	120.76(17)
C(2)-C(3)-H(3)	119.6
C(4)-C(3)-H(3)	119.6
C(3)-C(4)-C(24)	119.00(15)
C(3)-C(4)-C(5)	121.06(14)
C(24)-C(4)-C(5)	119.88(15)
C(6)-C(5)-C(16)	119.71(13)
C(6)-C(5)-C(4)	122.44(13)
C(16)-C(5)-C(4)	117.85(13)
C(5)-C(6)-C(7)	120.47(14)
C(5)-C(6)-C(25)	120.98(13)
C(7)-C(6)-C(25)	118.15(13)
C(8)-C(7)-C(13)	119.66(14)
C(8)-C(7)-C(6)	120.59(14)
C(13)-C(7)-C(6)	119.68(14)

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C(9)-C(8)-C(7)	118.71(14)
C(9)-C(8)-C(15)	122.54(14)
C(7)-C(8)-C(15)	118.69(13)
C(10)-C(9)-C(8)	120.75(15)
C(10)-C(9)-H(9)	119.6
C(8)-C(9)-H(9)	119.6
C(9)-C(10)-C(14)	120.24(15)
C(9)-C(10)-H(10)	119.9
C(14)-C(10)-H(10)	119.9
C(12)-C(11)-H(11A)	109.5
C(12)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
C(12)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
O(1)-C(12)-C(11)	106.19(14)
O(1)-C(12)-C(13)	112.19(14)
C(11)-C(12)-C(13)	114.95(14)
O(1)-C(12)-H(12)	107.7
C(11)-C(12)-H(12)	107.7
C(13)-C(12)-H(12)	107.7
C(14)-C(13)-C(7)	120.03(15)
C(14)-C(13)-C(12)	121.23(15)
C(7)-C(13)-C(12)	118.45(13)
C(13)-C(14)-C(10)	120.57(16)
C(13)-C(14)-H(14)	119.7
C(10)-C(14)-H(14)	119.7
C(21)-C(15)-C(16)	118.67(14)
C(21)-C(15)-C(8)	121.94(14)
C(16)-C(15)-C(8)	119.39(14)
C(17)-C(16)-C(15)	118.14(14)
C(17)-C(16)-C(5)	121.48(14)
C(15)-C(16)-C(5)	120.32(14)
C(18)-C(17)-C(16)	122.54(14)
C(18)-C(17)-H(17)	118.7
C(16)-C(17)-H(17)	118.7
C(17)-C(18)-C(20)	119.01(15)
C(17)-C(18)-C(19)	120.87(15)

C(20)-C(18)-C(19)	120.12(15)
C(18)-C(19)-H(19A)	109.5
C(18)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
C(18)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
C(21)-C(20)-C(18)	119.20(15)
C(21)-C(20)-C(22)	119.83(14)
C(18)-C(20)-C(22)	120.96(15)
C(20)-C(21)-C(15)	122.31(14)
C(20)-C(21)-H(21)	118.8
C(15)-C(21)-H(21)	118.8
C(20)-C(22)-H(22A)	109.5
C(20)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5
C(20)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5
C(1)-C(23)-C(24)	120.20(18)
C(1)-C(23)-H(23)	119.9
C(24)-C(23)-H(23)	119.9
C(4)-C(24)-C(23)	120.16(18)
C(4)-C(24)-H(24)	119.9
C(23)-C(24)-H(24)	119.9
O(2)-C(25)-O(1)	117.20(14)
O(2)-C(25)-C(6)	125.11(15)
O(1)-C(25)-C(6)	117.47(13)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 111103LT. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	31(1)	19(1)	30(1)	3(1)	6(1)	-3(1)
O(2)	40(1)	18(1)	36(1)	1(1)	9(1)	-1(1)
C(1)	36(1)	22(1)	50(1)	12(1)	21(1)	9(1)
C(2)	49(1)	20(1)	33(1)	3(1)	15(1)	1(1)
C(3)	36(1)	24(1)	24(1)	2(1)	0(1)	1(1)
C(4)	22(1)	18(1)	22(1)	4(1)	2(1)	-1(1)
C(5)	20(1)	17(1)	18(1)	0(1)	-5(1)	-1(1)
C(6)	21(1)	17(1)	20(1)	1(1)	-5(1)	0(1)
C(7)	18(1)	19(1)	21(1)	0(1)	-6(1)	-1(1)
C(8)	19(1)	19(1)	23(1)	1(1)	-6(1)	-2(1)
C(9)	23(1)	17(1)	30(1)	2(1)	-4(1)	0(1)
C(10)	23(1)	21(1)	35(1)	-2(1)	-3(1)	4(1)
C(11)	53(1)	28(1)	65(2)	13(1)	31(1)	5(1)
C(12)	31(1)	26(1)	27(1)	2(1)	3(1)	4(1)
C(13)	20(1)	22(1)	24(1)	2(1)	-4(1)	-2(1)
C(14)	20(1)	29(1)	25(1)	-1(1)	0(1)	0(1)
C(15)	20(1)	18(1)	22(1)	1(1)	-6(1)	-3(1)
C(16)	21(1)	19(1)	18(1)	1(1)	-6(1)	-3(1)
C(17)	23(1)	20(1)	24(1)	0(1)	-3(1)	0(1)
C(18)	24(1)	24(1)	21(1)	1(1)	-4(1)	-7(1)
C(19)	32(1)	29(1)	28(1)	4(1)	2(1)	-5(1)
C(20)	25(1)	21(1)	22(1)	3(1)	-7(1)	-7(1)
C(21)	23(1)	18(1)	27(1)	1(1)	-7(1)	-2(1)
C(22)	32(1)	24(1)	32(1)	9(1)	-4(1)	-6(1)
C(23)	27(1)	35(1)	44(1)	14(1)	2(1)	8(1)
C(24)	27(1)	29(1)	25(1)	4(1)	-2(1)	2(1)
C(25)	21(1)	21(1)	22(1)	2(1)	-2(1)	-2(1)

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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 111103LT.

	x	y	z	U(eq)
H(1)	5366	5440	6917	43
H(2)	4465	5752	4160	40
H(3)	3650	6753	4924	34
H(9)	2273	10132	9353	28
H(10)	1515	10086	12636	31
H(11A)	925	7439	14177	73
H(11B)	1214	7822	16574	73
H(11C)	1319	6919	16135	73
H(12)	2409	7622	15519	33
H(14)	1379	8938	14767	30
H(17)	4538	8053	5366	26
H(19A)	5143	8661	2284	44
H(19B)	5167	9578	2522	44
H(19C)	4620	9182	679	44
H(21)	2929	10107	6372	27
H(22A)	3420	10777	3250	44
H(22B)	3742	10255	1165	44
H(22C)	4285	10665	2991	44
H(23)	5454	6135	10432	42
H(24)	4641	7145	11199	32

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Table 1. Crystal data and structure refinement for **4c**.

Identification code	07dc31m	
Empirical formula	C28 H20 O2	
Formula weight	388.44	
Temperature	295(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*-1	
Unit cell dimensions	a = 9.8053(15) Å	□= 82.137(2)°.
	b = 10.1884(16) Å	□= 65.417(2)°.
	c = 11.1732(17) Å	□ =
77.710(2)°.		
Volume	990.3(3) $Å^3$	
Z	2	
Density (calculated)	1.303 Mg/m^3	
Absorption coefficient	0.081 mm^{-1}	
F(000)	408	
Crystal size	0.25 x 0.20 x 0.20 mm^3	
Theta range for data collection	2.01 to 28.31°.	
Index ranges	$-13 \leq h \leq 13$, $-13 \leq k \leq 13$, $-14 \leq l \leq 14$	
Reflections collected	11736	
Independent reflections	4907 [R(int) = 0.0605]	
Completeness to theta = 28.31°	99.6 %	
Absorption correction	Empirical	
Max. and min. transmission	0.95104 and 0.76198	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4907 / 0 / 272	
Goodness-of-fit on F^2	0.909	
Final R indices [I>2sigma(I)]	R1 = 0.0437, wR2 = 0.1073	
R indices (all data)	R1 = 0.0675, wR2 = 0.1180	
Largest diff. peak and hole	0.199 and -0.187 $e.Å^{-3}$	

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Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 07dc31m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

_____	x	y	z	U(eq)
C(1)	2474(1)	464(1)	4854(1)	43(1)
C(2)	2801(1)	154(1)	3524(1)	46(1)
C(3)	3371(2)	-1170(1)	3092(1)	59(1)
C(4)	3626(2)	-1461(2)	1846(1)	69(1)
C(5)	3314(2)	-450(2)	992(1)	70(1)
C(6)	2792(2)	848(2)	1368(1)	64(1)
C(7)	2543(1)	1194(1)	2632(1)	49(1)
C(8)	2152(1)	2590(1)	2992(1)	49(1)
C(9)	1838(2)	3654(2)	2150(1)	62(1)
C(10)	1597(2)	4967(2)	2453(2)	69(1)
C(11)	1784(2)	5262(2)	3537(1)	66(1)
C(12)	2109(2)	4243(1)	4374(1)	55(1)
C(13)	3259(2)	3739(1)	5954(1)	61(1)
C(14)	2203(2)	2946(1)	7004(1)	52(1)
C(15)	1725(2)	3159(2)	8335(1)	62(1)
C(16)	621(2)	2510(2)	9276(1)	65(1)
C(17)	-41(2)	1664(1)	8903(1)	58(1)
C(18)	426(1)	1444(1)	7585(1)	50(1)
C(19)	1578(1)	2065(1)	6619(1)	47(1)
C(20)	2121(1)	1776(1)	5197(1)	44(1)
C(21)	2143(1)	2880(1)	4210(1)	47(1)
C(22)	2592(1)	-675(1)	5822(1)	43(1)
C(23)	3583(1)	-749(1)	6435(1)	45(1)
C(24)	3744(1)	-1805(1)	7317(1)	47(1)
C(25)	2896(1)	-2817(1)	7598(1)	47(1)
C(26)	1883(1)	-2754(1)	7011(1)	51(1)
C(27)	1738(1)	-1704(1)	6140(1)	49(1)
C(28)	4071(2)	-4093(2)	8983(2)	76(1)
O(1)	2420(1)	4686(1)	5328(1)	72(1)
O(2)	2976(1)	-3924(1)	8435(1)	64(1)

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Table 3. Bond lengths [Å] and angles [°] for 07dc31m.

C(1)-C(20)	1.3735(17)
C(1)-C(2)	1.4495(15)
C(1)-C(22)	1.4946(16)
C(2)-C(7)	1.4088(17)
C(2)-C(3)	1.4138(18)
C(3)-C(4)	1.3711(18)
C(3)-H(3)	0.9300
C(4)-C(5)	1.380(2)
C(4)-H(4)	0.9300
C(5)-C(6)	1.368(2)
C(5)-H(5)	0.9300
C(6)-C(7)	1.4111(17)
C(6)-H(6)	0.9300
C(7)-C(8)	1.4588(19)
C(8)-C(9)	1.4057(17)
C(8)-C(21)	1.4274(17)
C(9)-C(10)	1.371(2)
C(9)-H(9)	0.9300
C(10)-C(11)	1.378(2)
C(10)-H(10)	0.9300
C(11)-C(12)	1.3770(18)
C(11)-H(11)	0.9300
C(12)-O(1)	1.3753(16)
C(12)-C(21)	1.4177(18)
C(13)-O(1)	1.4336(17)
C(13)-C(14)	1.4784(18)
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(14)-C(19)	1.3880(18)
C(14)-C(15)	1.3943(18)
C(15)-C(16)	1.371(2)
C(15)-H(15)	0.9300
C(16)-C(17)	1.378(2)
C(16)-H(16)	0.9300
C(17)-C(18)	1.3835(17)
C(17)-H(17)	0.9300

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C(18)-C(19)	1.3944(16)
C(18)-H(18)	0.9300
C(19)-C(20)	1.5004(16)
C(20)-C(21)	1.4604(16)
C(22)-C(23)	1.3869(16)
C(22)-C(27)	1.3973(17)
C(23)-C(24)	1.3861(16)
C(23)-H(23)	0.9300
C(24)-C(25)	1.3828(17)
C(24)-H(24)	0.9300
C(25)-O(2)	1.3735(13)
C(25)-C(26)	1.3877(17)
C(26)-C(27)	1.3702(16)
C(26)-H(26)	0.9300
C(27)-H(27)	0.9300
C(28)-O(2)	1.4135(17)
C(28)-H(28A)	0.9600
C(28)-H(28B)	0.9600
C(28)-H(28C)	0.9600

C(20)-C(1)-C(2)	120.54(10)
C(20)-C(1)-C(22)	121.02(10)
C(2)-C(1)-C(22)	118.40(10)
C(7)-C(2)-C(3)	118.76(11)
C(7)-C(2)-C(1)	119.51(11)
C(3)-C(2)-C(1)	121.73(11)
C(4)-C(3)-C(2)	121.28(13)
C(4)-C(3)-H(3)	119.4
C(2)-C(3)-H(3)	119.4
C(3)-C(4)-C(5)	119.83(14)
C(3)-C(4)-H(4)	120.1
C(5)-C(4)-H(4)	120.1
C(6)-C(5)-C(4)	120.40(13)
C(6)-C(5)-H(5)	119.8
C(4)-C(5)-H(5)	119.8
C(5)-C(6)-C(7)	121.54(13)
C(5)-C(6)-H(6)	119.2
C(7)-C(6)-H(6)	119.2

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C(2)-C(7)-C(6)	118.11(12)
C(2)-C(7)-C(8)	119.77(11)
C(6)-C(7)-C(8)	121.93(12)
C(9)-C(8)-C(21)	119.39(13)
C(9)-C(8)-C(7)	121.64(12)
C(21)-C(8)-C(7)	118.93(10)
C(10)-C(9)-C(8)	120.91(13)
C(10)-C(9)-H(9)	119.5
C(8)-C(9)-H(9)	119.5
C(9)-C(10)-C(11)	120.10(13)
C(9)-C(10)-H(10)	120.0
C(11)-C(10)-H(10)	120.0
C(12)-C(11)-C(10)	120.34(14)
C(12)-C(11)-H(11)	119.8
C(10)-C(11)-H(11)	119.8
O(1)-C(12)-C(11)	113.51(12)
O(1)-C(12)-C(21)	125.14(11)
C(11)-C(12)-C(21)	121.33(13)
O(1)-C(13)-C(14)	108.96(12)
O(1)-C(13)-H(13A)	109.9
C(14)-C(13)-H(13A)	109.9
O(1)-C(13)-H(13B)	109.9
C(14)-C(13)-H(13B)	109.9
H(13A)-C(13)-H(13B)	108.3
C(19)-C(14)-C(15)	120.32(12)
C(19)-C(14)-C(13)	117.50(11)
C(15)-C(14)-C(13)	121.89(13)
C(16)-C(15)-C(14)	120.30(14)
C(16)-C(15)-H(15)	119.8
C(14)-C(15)-H(15)	119.8
C(15)-C(16)-C(17)	119.90(12)
C(15)-C(16)-H(16)	120.0
C(17)-C(16)-H(16)	120.0
C(16)-C(17)-C(18)	120.29(13)
C(16)-C(17)-H(17)	119.9
C(18)-C(17)-H(17)	119.9
C(17)-C(18)-C(19)	120.54(13)
C(17)-C(18)-H(18)	119.7

C(19)-C(18)-H(18)	119.7
C(14)-C(19)-C(18)	118.58(11)
C(14)-C(19)-C(20)	121.26(11)
C(18)-C(19)-C(20)	120.16(11)
C(1)-C(20)-C(21)	120.42(10)
C(1)-C(20)-C(19)	119.20(10)
C(21)-C(20)-C(19)	120.16(10)
C(12)-C(21)-C(8)	116.51(11)
C(12)-C(21)-C(20)	125.17(11)
C(8)-C(21)-C(20)	118.29(11)
C(23)-C(22)-C(27)	117.38(11)
C(23)-C(22)-C(1)	120.42(11)
C(27)-C(22)-C(1)	122.20(11)
C(24)-C(23)-C(22)	121.93(11)
C(24)-C(23)-H(23)	119.0
C(22)-C(23)-H(23)	119.0
C(25)-C(24)-C(23)	119.29(11)
C(25)-C(24)-H(24)	120.4
C(23)-C(24)-H(24)	120.4
O(2)-C(25)-C(24)	124.68(11)
O(2)-C(25)-C(26)	115.54(11)
C(24)-C(25)-C(26)	119.78(11)
C(27)-C(26)-C(25)	120.18(11)
C(27)-C(26)-H(26)	119.9
C(25)-C(26)-H(26)	119.9
C(26)-C(27)-C(22)	121.43(11)
C(26)-C(27)-H(27)	119.3
C(22)-C(27)-H(27)	119.3
O(2)-C(28)-H(28A)	109.5
O(2)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5
O(2)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
C(12)-O(1)-C(13)	118.22(10)
C(25)-O(2)-C(28)	117.96(10)

Symmetry transformations used to generate equivalent atoms:

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Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for 07dc31m. The anisotropic displacement factor exponent takes the form: $-2 \cdot {}^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	39(1)	47(1)	44(1)	-2(1)	-18(1)	-8(1)
C(2)	41(1)	55(1)	45(1)	-4(1)	-18(1)	-10(1)
C(3)	62(1)	60(1)	55(1)	-10(1)	-22(1)	-11(1)
C(4)	73(1)	75(1)	59(1)	-22(1)	-19(1)	-15(1)
C(5)	72(1)	97(1)	49(1)	-15(1)	-22(1)	-27(1)
C(6)	62(1)	86(1)	49(1)	1(1)	-27(1)	-20(1)
C(7)	40(1)	66(1)	44(1)	-1(1)	-19(1)	-13(1)
C(8)	39(1)	60(1)	48(1)	7(1)	-18(1)	-10(1)
C(9)	54(1)	77(1)	57(1)	14(1)	-29(1)	-13(1)
C(10)	59(1)	67(1)	71(1)	23(1)	-26(1)	-6(1)
C(11)	65(1)	51(1)	67(1)	7(1)	-17(1)	-5(1)
C(12)	54(1)	51(1)	50(1)	0(1)	-13(1)	-6(1)
C(13)	68(1)	60(1)	60(1)	-9(1)	-26(1)	-17(1)
C(14)	55(1)	49(1)	51(1)	-6(1)	-22(1)	-4(1)
C(15)	69(1)	67(1)	58(1)	-13(1)	-33(1)	-5(1)
C(16)	69(1)	81(1)	45(1)	-7(1)	-26(1)	-2(1)
C(17)	53(1)	67(1)	47(1)	4(1)	-17(1)	-4(1)
C(18)	51(1)	49(1)	48(1)	-1(1)	-20(1)	-3(1)
C(19)	48(1)	44(1)	44(1)	-2(1)	-19(1)	-1(1)
C(20)	41(1)	47(1)	42(1)	-1(1)	-17(1)	-7(1)
C(21)	41(1)	48(1)	46(1)	2(1)	-14(1)	-5(1)
C(22)	42(1)	43(1)	43(1)	-5(1)	-18(1)	-5(1)
C(23)	41(1)	47(1)	47(1)	-4(1)	-17(1)	-10(1)
C(24)	43(1)	52(1)	48(1)	-4(1)	-22(1)	-6(1)
C(25)	48(1)	45(1)	44(1)	-1(1)	-17(1)	-5(1)
C(26)	52(1)	47(1)	58(1)	-1(1)	-22(1)	-15(1)
C(27)	49(1)	52(1)	55(1)	-5(1)	-26(1)	-10(1)
C(28)	80(1)	80(1)	76(1)	21(1)	-46(1)	-14(1)
O(1)	100(1)	51(1)	65(1)	-3(1)	-33(1)	-13(1)
O(2)	70(1)	55(1)	71(1)	15(1)	-36(1)	-15(1)

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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for 07dc31m.

_	x	y	z	U(eq)
H(3)	3577	-1858	3664	70
H(4)	4007	-2337	1577	83
H(5)	3461	-652	155	85
H(6)	2597	1517	777	76
H(9)	1793	3466	1377	74
H(10)	1307	5658	1926	83
H(11)	1690	6153	3704	79
H(13A)	3736	4211	6330	73
H(13B)	4052	3145	5311	73
H(15)	2157	3743	8587	75
H(16)	319	2642	10164	78
H(17)	-805	1240	9540	70
H(18)	-32	877	7341	60
H(23)	4156	-71	6248	54
H(24)	4416	-1833	7715	56
H(26)	1301	-3427	7208	61
H(27)	1056	-1675	5752	59
H(28A)	5060	-4073	8288	114
H(28B)	4074	-4942	9476	114
H(28C)	3824	-3379	9556	114

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Table 1. Crystal data and structure refinement for **4j**.

Identification code	090839m
Empirical formula	C30 H21 Cl O
Formula weight	432.92
Temperature	295(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	*P*-1
Unit cell dimensions	a = 8.1591(4) Å □= 87.4450(10)°.
	b = 10.8269(6) Å □= 80.6820(10)°.
	c = 12.5067(7) Å □ = 81.2890(10)°.
Volume	1077.43(10) $Å^3$
Z	2
Density (calculated)	1.334 Mg/m^3
Absorption coefficient	0.198 mm^{-1}
F(000)	452
Crystal size	0.25 x 0.20 x 0.15 mm^3
Theta range for data collection	1.65 to 28.35°.
Index ranges	$-10 \le h \le 10$, $-14 \le k \le 14$, $-16 \le l \le 15$
Reflections collected	12771
Independent reflections	5353 [R(int) = 0.0197]
Completeness to theta = 28.35°	99.3 %
Absorption correction	Empirical
Max. and min. transmission	0.7457 and 0.6604
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5353 / 0 / 289
Goodness-of-fit on F^2	1.051
Final R indices [I>2sigma(I)]	R1 = 0.0462, wR2 = 0.1263
R indices (all data)	R1 = 0.0580, wR2 = 0.1375
Largest diff. peak and hole	0.283 and -0.216 $e.Å^{-3}$

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Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 090839m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	7859(2)	4939(1)	9482(1)	43(1)
C(2)	8692(2)	3803(2)	9766(1)	49(1)
C(3)	9512(2)	2978(2)	8980(1)	47(1)
C(4)	9369(2)	3236(1)	7906(1)	41(1)
C(5)	8492(2)	4380(1)	7564(1)	35(1)
C(6)	8111(2)	4672(1)	6468(1)	35(1)
C(7)	8115(2)	3738(1)	5709(1)	42(1)
C(8)	7653(2)	4048(1)	4711(1)	42(1)
C(9)	7531(2)	3189(2)	3814(1)	55(1)
C(10)	7348(3)	4077(2)	2848(1)	62(1)
C(11)	6657(2)	5374(2)	3302(1)	50(1)
C(12)	7141(2)	5289(1)	4421(1)	40(1)
C(13)	7068(2)	6213(1)	5146(1)	40(1)
C(14)	7536(2)	5931(1)	6180(1)	36(1)
C(15)	7373(2)	6907(1)	6958(1)	36(1)
C(16)	7548(2)	6619(1)	8018(1)	37(1)
C(17)	7540(2)	7656(1)	8773(1)	40(1)
C(18)	8548(2)	8583(1)	8472(1)	48(1)
C(19)	8467(3)	9592(2)	9138(2)	60(1)
C(20)	7386(3)	9683(2)	10113(2)	66(1)
C(21)	6434(2)	8748(2)	10440(2)	59(1)
C(22)	6517(2)	7728(2)	9786(1)	47(1)
C(23)	5698(2)	6619(2)	10156(1)	56(1)
C(24)	7920(2)	5320(1)	8373(1)	36(1)
C(25)	6900(2)	8235(1)	6598(1)	40(1)
C(26)	7960(2)	8815(2)	5811(1)	50(1)
C(27)	7530(3)	10053(2)	5506(2)	64(1)
C(28)	6034(3)	10723(2)	5963(2)	69(1)
C(29)	4952(3)	10153(2)	6720(2)	63(1)
C(30)	5380(2)	8912(2)	7037(1)	49(1)
Cl(1)	10498(1)	2088(1)	7020(1)	57(1)
O(1)	6982(2)	5608(1)	10347(1)	56(1)

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Table 3. Bond lengths [Å] and angles [°] for 090839m.

C(1)-O(1)	1.3669(18)
C(1)-C(2)	1.373(2)
C(1)-C(24)	1.4248(19)
C(2)-C(3)	1.376(2)
C(2)-H(2)	0.9300
C(3)-C(4)	1.379(2)
C(3)-H(3)	0.9300
C(4)-C(5)	1.4191(19)
C(4)-Cl(1)	1.7512(15)
C(5)-C(24)	1.4388(19)
C(5)-C(6)	1.4657(18)
C(6)-C(7)	1.4164(19)
C(6)-C(14)	1.4223(18)
C(7)-C(8)	1.375(2)
C(7)-H(7)	0.9300
C(8)-C(12)	1.395(2)
C(8)-C(9)	1.513(2)
C(9)-C(10)	1.522(3)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-C(11)	1.530(2)
C(10)-H(10A)	0.9700
C(10)-H(10B)	0.9700
C(11)-C(12)	1.509(2)
C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700
C(12)-C(13)	1.368(2)
C(13)-C(14)	1.4150(19)
C(13)-H(13)	0.9300
C(14)-C(15)	1.4452(18)
C(15)-C(16)	1.3722(19)
C(15)-C(25)	1.5003(19)
C(16)-C(24)	1.4583(19)
C(16)-C(17)	1.4976(18)
C(17)-C(18)	1.393(2)
C(17)-C(22)	1.399(2)

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C(18)-C(19)	1.390(2)
C(18)-H(18)	0.9300
C(19)-C(20)	1.382(3)
C(19)-H(19)	0.9300
C(20)-C(21)	1.376(3)
C(20)-H(20)	0.9300
C(21)-C(22)	1.390(2)
C(21)-H(21)	0.9300
C(22)-C(23)	1.482(3)
C(23)-O(1)	1.436(2)
C(23)-H(23A)	0.9700
C(23)-H(23B)	0.9700
C(25)-C(30)	1.385(2)
C(25)-C(26)	1.395(2)
C(26)-C(27)	1.385(2)
C(26)-H(26)	0.9300
C(27)-C(28)	1.373(3)
C(27)-H(27)	0.9300
C(28)-C(29)	1.377(3)
C(28)-H(28)	0.9300
C(29)-C(30)	1.393(2)
C(29)-H(29)	0.9300
C(30)-H(30)	0.9300

O(1)-C(1)-C(2)	113.75(13)
O(1)-C(1)-C(24)	125.51(13)
C(2)-C(1)-C(24)	120.73(14)
C(1)-C(2)-C(3)	120.44(14)
C(1)-C(2)-H(2)	119.8
C(3)-C(2)-H(2)	119.8
C(2)-C(3)-C(4)	120.07(14)
C(2)-C(3)-H(3)	120.0
C(4)-C(3)-H(3)	120.0
C(3)-C(4)-C(5)	121.94(14)
C(3)-C(4)-Cl(1)	113.85(11)
C(5)-C(4)-Cl(1)	124.03(11)
C(4)-C(5)-C(24)	116.66(12)
C(4)-C(5)-C(6)	125.49(12)

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C(24)-C(5)-C(6)	117.85(12)
C(7)-C(6)-C(14)	117.67(12)
C(7)-C(6)-C(5)	122.78(12)
C(14)-C(6)-C(5)	119.21(12)
C(8)-C(7)-C(6)	120.95(13)
C(8)-C(7)-H(7)	119.5
C(6)-C(7)-H(7)	119.5
C(7)-C(8)-C(12)	121.03(13)
C(7)-C(8)-C(9)	128.59(14)
C(12)-C(8)-C(9)	110.33(13)
C(8)-C(9)-C(10)	103.32(14)
C(8)-C(9)-H(9A)	111.1
C(10)-C(9)-H(9A)	111.1
C(8)-C(9)-H(9B)	111.1
C(10)-C(9)-H(9B)	111.1
H(9A)-C(9)-H(9B)	109.1
C(9)-C(10)-C(11)	106.96(14)
C(9)-C(10)-H(10A)	110.3
C(11)-C(10)-H(10A)	110.3
C(9)-C(10)-H(10B)	110.3
C(11)-C(10)-H(10B)	110.3
H(10A)-C(10)-H(10B)	108.6
C(12)-C(11)-C(10)	103.23(13)
C(12)-C(11)-H(11A)	111.1
C(10)-C(11)-H(11A)	111.1
C(12)-C(11)-H(11B)	111.1
C(10)-C(11)-H(11B)	111.1
H(11A)-C(11)-H(11B)	109.1
C(13)-C(12)-C(8)	119.71(13)
C(13)-C(12)-C(11)	129.57(14)
C(8)-C(12)-C(11)	110.65(13)
C(12)-C(13)-C(14)	120.87(13)
C(12)-C(13)-H(13)	119.6
C(14)-C(13)-H(13)	119.6
C(13)-C(14)-C(6)	119.69(12)
C(13)-C(14)-C(15)	120.33(12)
C(6)-C(14)-C(15)	119.94(12)
C(16)-C(15)-C(14)	120.70(12)

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C(16)-C(15)-C(25)	120.76(12)
C(14)-C(15)-C(25)	118.42(11)
C(15)-C(16)-C(24)	120.37(12)
C(15)-C(16)-C(17)	119.08(12)
C(24)-C(16)-C(17)	120.21(12)
C(18)-C(17)-C(22)	118.37(14)
C(18)-C(17)-C(16)	120.38(13)
C(22)-C(17)-C(16)	121.24(14)
C(19)-C(18)-C(17)	120.69(17)
C(19)-C(18)-H(18)	119.7
C(17)-C(18)-H(18)	119.7
C(20)-C(19)-C(18)	120.20(18)
C(20)-C(19)-H(19)	119.9
C(18)-C(19)-H(19)	119.9
C(21)-C(20)-C(19)	119.69(16)
C(21)-C(20)-H(20)	120.2
C(19)-C(20)-H(20)	120.2
C(20)-C(21)-C(22)	120.61(17)
C(20)-C(21)-H(21)	119.7
C(22)-C(21)-H(21)	119.7
C(21)-C(22)-C(17)	120.27(17)
C(21)-C(22)-C(23)	122.81(16)
C(17)-C(22)-C(23)	116.61(14)
O(1)-C(23)-C(22)	108.13(14)
O(1)-C(23)-H(23A)	110.1
C(22)-C(23)-H(23A)	110.1
O(1)-C(23)-H(23B)	110.1
C(22)-C(23)-H(23B)	110.1
H(23A)-C(23)-H(23B)	108.4
C(1)-C(24)-C(5)	117.87(12)
C(1)-C(24)-C(16)	123.38(12)
C(5)-C(24)-C(16)	118.60(12)
C(30)-C(25)-C(26)	118.38(14)
C(30)-C(25)-C(15)	120.30(14)
C(26)-C(25)-C(15)	121.31(13)
C(27)-C(26)-C(25)	120.76(17)
C(27)-C(26)-H(26)	119.6
C(25)-C(26)-H(26)	119.6

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C(28)-C(27)-C(26)	120.32(19)
C(28)-C(27)-H(27)	119.8
C(26)-C(27)-H(27)	119.8
C(27)-C(28)-C(29)	119.61(17)
C(27)-C(28)-H(28)	120.2
C(29)-C(28)-H(28)	120.2
C(28)-C(29)-C(30)	120.47(17)
C(28)-C(29)-H(29)	119.8
C(30)-C(29)-H(29)	119.8
C(25)-C(30)-C(29)	120.41(17)
C(25)-C(30)-H(30)	119.8
C(29)-C(30)-H(30)	119.8
C(1)-O(1)-C(23)	118.66(12)

Symmetry transformations used to generate equivalent atoms:

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Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 090839m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	51(1)	46(1)	33(1)	0(1)	-9(1)	-12(1)
C(2)	63(1)	53(1)	35(1)	7(1)	-15(1)	-11(1)
C(3)	55(1)	42(1)	47(1)	7(1)	-19(1)	-6(1)
C(4)	45(1)	37(1)	42(1)	0(1)	-11(1)	-4(1)
C(5)	38(1)	35(1)	34(1)	1(1)	-9(1)	-8(1)
C(6)	36(1)	37(1)	32(1)	-2(1)	-6(1)	-5(1)
C(7)	49(1)	38(1)	40(1)	-6(1)	-11(1)	0(1)
C(8)	43(1)	48(1)	36(1)	-9(1)	-7(1)	-4(1)
C(9)	67(1)	56(1)	43(1)	-14(1)	-15(1)	-1(1)
C(10)	79(1)	73(1)	37(1)	-11(1)	-11(1)	-17(1)
C(11)	56(1)	62(1)	35(1)	0(1)	-14(1)	-13(1)
C(12)	41(1)	50(1)	31(1)	-1(1)	-7(1)	-8(1)
C(13)	47(1)	40(1)	34(1)	2(1)	-10(1)	-4(1)
C(14)	38(1)	36(1)	32(1)	-1(1)	-7(1)	-5(1)
C(15)	39(1)	35(1)	35(1)	-2(1)	-6(1)	-4(1)
C(16)	39(1)	37(1)	35(1)	-2(1)	-6(1)	-6(1)
C(17)	44(1)	39(1)	38(1)	-5(1)	-14(1)	0(1)
C(18)	56(1)	42(1)	50(1)	-3(1)	-19(1)	-6(1)
C(19)	76(1)	41(1)	72(1)	-6(1)	-34(1)	-8(1)
C(20)	85(1)	49(1)	68(1)	-24(1)	-34(1)	8(1)
C(21)	65(1)	62(1)	48(1)	-20(1)	-17(1)	12(1)
C(22)	48(1)	52(1)	39(1)	-10(1)	-12(1)	3(1)
C(23)	55(1)	67(1)	42(1)	-9(1)	2(1)	-5(1)
C(24)	41(1)	38(1)	32(1)	0(1)	-8(1)	-7(1)
C(25)	49(1)	35(1)	38(1)	-3(1)	-14(1)	-2(1)
C(26)	56(1)	44(1)	50(1)	6(1)	-12(1)	-5(1)
C(27)	82(1)	48(1)	68(1)	16(1)	-24(1)	-15(1)
C(28)	94(2)	41(1)	74(1)	3(1)	-37(1)	4(1)
C(29)	73(1)	53(1)	61(1)	-16(1)	-28(1)	19(1)
C(30)	54(1)	50(1)	43(1)	-8(1)	-14(1)	3(1)
Cl(1)	67(1)	47(1)	53(1)	-4(1)	-14(1)	13(1)
O(1)	76(1)	56(1)	31(1)	-1(1)	-3(1)	-4(1)

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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 090839m.

	x	y	z	U(eq)
H(2)	8702	3589	10493	59
H(3)	10162	2247	9173	57
H(7)	8435	2904	5889	51
H(9A)	6563	2756	3998	66
H(9B)	8536	2578	3667	66
H(10A)	6582	3812	2416	74
H(10B)	8428	4090	2393	74
H(11A)	7167	6019	2864	60
H(11B)	5448	5549	3339	60
H(13)	6705	7038	4956	49
H(18)	9284	8526	7820	58
H(19)	9141	10207	8927	73
H(20)	7303	10373	10546	80
H(21)	5728	8799	11105	71
H(23A)	4910	6802	10818	67
H(23B)	5087	6394	9608	67
H(26)	8967	8366	5487	60
H(27)	8257	10432	4989	77
H(28)	5752	11556	5763	82
H(29)	3930	10601	7021	76
H(30)	4639	8535	7547	59