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Supporting Information

Au (I)-Catalyzed Synthesis of 8-Oxabicyclo[3.2.1]oct-2-enes and 9-Oxabicyclo[3.3.1]nona-2,6-dienes from En-yne-ol via Oxonium/Prins-type Cyclization

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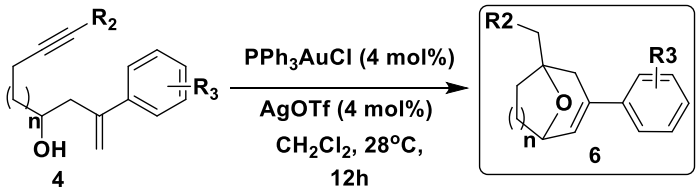
I. General Information

Unless otherwise noted, all the reactions for the preparations of the substrates were performed in oven-dried glassware under nitrogen atmosphere with freshly distilled solvents. Solvents were dried under standard methods. All other commercial reagents were used without further purification. ^{1}H NMR and ^{13}C NMR spectra were recorded on 400 MHz Spectrometers using chloroform-d (CDCl3) as the internal standards. Multiplicities were denoted as (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or overlap of nonequivalent resonances, integration). Aluminum-backed plates precoated with silica gel 60F254 were used for thin layer chromatography and were visualized with a UV lamp. Melting points are uncorrected. Mass spectra and high resolution mass spectra (HRMS) were obtained by electron impact at 70 eV in an ion trap. Flash column chromatography was carried out over silica gel 60 (230-400 mesh).

II. Standard protocol



Compound 6 Synthesis protocol:

To a solution of Compound **3** (50 mg, 0.18 mmol) in dichloromethane (6 mL) at 28 °C, PPh_3AuCl (1.78 mg, 0.0072 mmol), and AgOTf (1.85 mg, 0.0072 mmol). The resulting solution was stirred for 12 h at 28 °C. The reaction mixture was directly purified by column chromatography technique (hexane/EtOAc: 10/1) to obtain compound **4** (41 mg, 82%).

1-benzyl-3-(p-tolyl)-8-oxabicyclo[3.2.1]oct-2-ene (6a): Yellow oil; IR $(neat)_{\nu max}$: 3026, 2953, 1514, 1496, 1454, 1330, 1022, 804 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34-7.20 (m, 7H), 7.09 (d, *J* = 8.4 Hz, 2H), 6.29 (dt, *J* = 3.2, 1.6 Hz, 1H), 4.68-4.66 (m, 1H), 3.12-3.03 (m, 2H), 2.76 (d, *J* = 16.8 Hz, 1H), 2.31 (s, 3H), 2.20 (dd, *J* = 16.4, 1.2 Hz, 1H), 1.99-1.86 (m, 3H), 1.81-1.73 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.5, 137.0, 136.9, 133.2, 130.3, 128.9, 128.0, 126.7, 126.3, 124.7, 81.1, 74.2, 46.2, 40.7, 35.4, 33.7, 21.0. HRMS calcd for $C_{21}H_{22}NaO$ [M+H]: 313.1562 found 313.1563.

1-(2-methoxybenzyl)-3-phenyl-8-oxabicyclo[3.2.1]oct-2-ene (6b): Light Yellow oil; IR $(neat)_{\nu max}$: 3031, 2926, 2361, 1732, 1651, 1599, 1493, 1243, 1127 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38–7.20 (m, 7H), 6.94-6.88 (m, 2H), 6.33 (dt, *J* = 3.2, 1.6 Hz, 1H), 4.69-4.66 (m, 1H), 3.85 (s, 3H), 3.16 (s, 2H), 2.80 (d, *J* = 16.8 Hz, 1H), 2.27 (dd, *J* = 16.8, 1.2 Hz, 1H), 2.00-1.91 (m, 3H), 1.84-1.76 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.7, 139.9, 133.6, 132.2, 128.2, 127.5, 127.1, 126.0, 124.8, 120.3, 110.4, 81.7, 74.2, 55.2, 40.5, 38.6, 35.4, 33.6. HRMS calcd for $C_{21}H_{22}NaO_2$ [M+Na]: 329.1517 found 329.1518.

1-(2-methoxybenzyl)-3-(p-tolyl)-8-oxabicyclo[3.2.1]oct-2-ene (6c): yellow oil; IR (neat)$_{\nu max}$: 3026, 2954, 2359, 1601, 1587, 1493, 1244, 1117 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 7.44 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.30-7.25 (m, 3H), 7.16 (d, *J* = 8 Hz, 2H), 7.00-6.92 (m, 2H), 6.34 (dt, *J* = 3.2, 1.6 Hz, 1H), 4.73-4.72 (m, 1H), 3.89 (s, 3H), 3.22 (s, 2H), 2.85 (d, *J* = 17.2 Hz, 1H), 2.37 (s, 3H), 2.32 (dd, *J* = 16.8, 1.2 Hz, 1H), 2.03-1.97 (m, 3H), 1.89-1.80 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.6, 137.0, 136.8, 133.3, 132.1, 128.8, 127.4, 126.6, 125.9, 124.6, 120.2, 110.2, 81.6, 74.1, 55.1, 40.5, 38.5, 35.4, 33.5, 20.9. HRMS calcd for $C_{22}H_{24}NaO_2$ [M+Na]: 343.1668 found 343.1667.

1-benzyl-3-phenyl-8-oxabicyclo[3.2.1]oct-2-ene (6d): White solid, m.p: 9-101 ºC; IR (neat)$_{\nu max}$: 3028, 2954, 2362, 1600, 1494, 1454, 1362 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34-7.20 (m, 10H), 6.33 (dt, *J* = 3.6, 1.6 Hz, 1H), 4.68 (t, *J* = 4.0 Hz, 1H), 3.13-3.03 (m, 2H), 2.78 (d, *J* = 16.8 Hz, 1H), 2.21 (dt, *J* = 16.8, 1.6 Hz, 1H), 2.01-1.87 (m, 3H), 1.82-1.74 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.8, 137.4, 133.4, 130.3, 129.6, 128.2, 128.0, 127.6, 127.2, 126.3, 124.8, 81.1, 74.2, 46.1, 43.1, 40.7, 39.6, 35.4, 33.7, 30.6. HRMS calcd for $C_{20}H_{20}NaO$ [M+Na]: 299.1406 found 299.1405.

1-(2-methoxybenzyl)-3-(p-tolyl)-9-oxabicyclo[3.3.1]non-2-ene (6e): Colorless oil; IR (neat)$_{\nu max}$: 2946, 2346, 1599, 1498, 1458, 1240 cm^{-1};^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 (dd, *J* = 7.2, 1.6 Hz, 1H), 7.30-7.25 (m, 2H), 7.20 (td, *J* = 8.0, 2.0 Hz, 1H), 7.12 (d, *J* = 7.6 Hz, 2H), 6.91-6.85 (m, 2H), 6.07 (dt, *J* = 4.0, 2.0 Hz, 1H), 4.64 (s, 1H), 3.81 (s, 3H), 3.0 (d, *J* = 13.6 Hz, 1H), 2.88 (d, *J* = 13.2 Hz, 1H), 2.74 (d, *J* = 17.6 Hz, 1H), 2.33 (s, 3H), 2.25 (dd, *J* = 17.6, 1.2 Hz, 1H), 1.85-1.72 (m, 2H), 1.65-1.58 (m, 3H), 1.49-1.41 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.4, 136.9, 136.3, 132.6, 128.9, 127.4, 125.8, 124.7, 123.8, 120.1, 110.4, 72.8, 69.7, 55.2, 42. 9, 36.5, 35.9, 27.9, 21.0, 16.3. HRMS calcd for $C_{23}H_{26}NaO_2$ [M+Na]: 357.1830 found 357.1832.

1-(2-methoxybenzyl)-3-phenyl-9-oxabicyclo[3.3.1]non-2-ene (6f): Yellow oil; IR (neat)$_{\nu max}$: 2926, 2348, 1601, 1492, 1460, 1244 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42–7.20 (m, 7H), 6.94-6.88 (m, 2H), 6.13 (dt, *J* = 4.0, 2.0 Hz, 1H), 4.67 (t, *J* = 6.4 Hz, 1H), 3.83 (s, 3H), 3.05-2.90 (m, 2H), 2.78 (d, *J* = 17.6 Hz, 1H), 2.29 (dt, *J* = 18.0, 1.2 Hz, 1H), 1.85-1.79 (m, 2H), 1.66-1.63 (m, 2H), 1.52-1.44 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9, 140.2, 136.4, 132.6, 128.2, 127.4, 127.1, 125.7, 124.8, 124.7, 120.1, 110.3, 72.8, 69.7, 55.2, 42.9, 36.4, 35.8, 27.8, 16.3. HRMS calcd for $C_{22}H_{24}NaO_2$ [M+Na]: 343.1673 found 343.1674.

5-methyl-3-phenyl-8-oxabicyclo[3.2.1]oct-2-ene (6g) (two isomers are inseparable): Colorless oil; IR (neat)$_{\nu max}$: 3028, 2954, 2362, 1600, 1494, 1454, 1362 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, *J* = 2.0 Hz, 2H), 7.43 (d, *J* = 2.0 Hz, 2H), 7.35 (t, *J* = 7.2 Hz, 4H), 7.28 (d, *J* = 6.8 Hz, 2H), 6.14 (t, *J* = 2.4 Hz, 1H), 5.98 (s, 1H), 4.64 (s, 1H), 4.48 (t, *J* = 6.8 Hz, 1H), 2.98-2.92 (m, 1H), 2.57 (d, *J* = 17.6 Hz, 1H), 2.38 (d, *J* = 18.0 Hz, 1H), 2.19 (d, J = 18.0 Hz, 1H), 1.92-1.80 (m, 3H), 1.64-1.52 (m, 5H), 1.37 (s, 2H), 1.35 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.9, 139.7, 136.0, 135.4, 128.6, 128.1, 127.0, 124.6, 124.4, 69.7, 69.5, 67.9, 38.7, 37.5, 34.9, 31.6, 31.5, 30.5, 28.0, 27.5, 17.1, 16.3. HRMS calcd for $C_{15}H_{18}NaO$ [M+Na]: 237.1249 found 237.1251.

5-methyl-3-phenyl-8-oxabicyclo[3.2.1]oct-2-ene (6h) (two isomers are inseparable): Colorless oil; IR (neat)$_{\nu max}$: 2926, 2348, 1601, 1492, 1460, 1244 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31-7.27 (m, 4H), 7.23 (t, *J* = 7.6 Hz, 4H), 7.18-7.14 (m, 2H), 6.28 (dt, *J* = 3.2, 1.6 Hz, 1H), 6.14 (t, *J* = 2.0 Hz, 1H), 4.65 (t, 7.2 Hz, 1H), 4.59 (t, *J* = 5.6 Hz, 1H), 2.92-2.87 (m, 1H), 2.66 (d, *J* = 16.8 Hz, 1H), 2.26-2.15 (m, 2H), 2.08-2.00 (m, 3H), 1.94-1.88 (m, 1H), 1.86-1.78 (m, 1H), 1.72-1.62 (m, 3H), 1.43 (s, 3H), 1.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.7, 139.6, 133.3, 132.2, 131.0, 128.2, 127.4, 127.2, 124.7, 78.7, 78.0, 74.5, 73.9, 42.5, 41.5, 35.8, 35.6, 35.5, 31.1, 26.9, 23.2. HRMS calcd for $C_{14}H_{16}NaO$ [M+Na]: 223.1098 found 223.1096.

5-(4-methylbenzyl)-3-phenyl-9-oxabicyclo[3.3.1]non-2-ene (6i): Colorless oil; IR (neat)$_{\nu max}$: 3028, 2954, 2362, 1600, 1494, 1454, 1362 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25-7.19 (m, 4H), 7.16-7.12 (m, 3H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.25 (dt, *J* = 3.2, 1.6 Hz, 1H), 4.60 (t, *J* = 6.0 Hz, 1H), 3.00-2.92 (m, 2H), 2.69 (d, *J* = 17.2 Hz, 1H), 2.25 (s, 3H), 2.13 (dd, *J* = 16.4, 1.6 Hz, 1H), 1.91-1.81 (m, 3H), 1.72-1.65 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.8, 135.8, 134.3, 133.4, 130.1, 128.7, 128.2, 127.6, 127.2, 124.8, 81.2, 74.2, 45.7, 40.6, 35.4, 33.7, 21.0. HRMS calcd for $C_{21}H_{22}NaO$ [M+Na]: 313.1568 found 313.1566.

3-methoxy-9-phenyl-7-(p-tolyl)-5,6,9,10-tetrahydro-5,9-epoxybenzo[8]annulene (7a): Light yellow solid, m.p: 150-152 ^{o}C; IR (neat)$_{\nu max}$: 3028, 2923, 2346, 1614, 1504, 1251 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67-7.64 (m, 2H), 7.44-7.39 (m, 2H), 7.31 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.20-7.17 (m, 2H), 7.07-7.03 (m, 3H), 6.75 (dd, *J* = 8.4, 2.8 Hz, 1H), 6.67 (d, *J* = 2.4 Hz, 1H), 6.17 (d, *J* = 2.0 Hz, 1H), 5.48 (d, *J* = 6.0 Hz, 1H), 3.79 (s, 3H), 3.28-3.11 (m, 3H), 2.52 (d, *J* = 16.8 Hz, 1H), 2.28 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.7, 145.0, 138.8, 137.2, 136.9, 131.6, 129.6, 128.9, 128.4, 127.5, 127.2, 125.1, 124.9, 124.8, 112.7, 110.0, 74.0, 71.3, 55.2, 38.6, 33.7, 21.0. HRMS calcd for $C_{26}H_{25}O_2$ [M+H]: 369.1854 found 369.1855.

3-methoxy-7,9-diphenyl-5,6,9,10-tetrahydro-5,9-epoxybenzo[8]annulene (7b): Light brown solid, m.p: 146-148 ^{o}C; IR (neat)$_{\nu max}$: 3028, 2924, 2346, 1615, 1504, 1252 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67-7.63 (m, 2H), 7.43-7.39 (m, 2H), 7.32-7.16 (m, 6H), 7.04 (d, *J* = 8.0 Hz, 1H), 6.75 (dd, *J* = 8.0, 2.4 Hz, 1H), 6.67 (d, *J* = 2.4 Hz, 1H), 6.20 (d, *J* = 1.6 Hz, 1H), 5.48 (d, *J* = 5.6 Hz, 1H), 3.79 (s, 3H), 3.32-3.22 (m, 2H), 3.14 (d, *J* = 16.0 Hz, 1H), 2.53 (d, *J* = 16.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.7, 144.9, 139.8, 138.8, 131.8, 128.4, 128.2, 127.7, 127.4, 127.2, 125.1, 124.9, 112.8, 110.1, 74.1, 71.3, 55.2, 38.6, 33.8. HRMS calcd for $C_{25}H_{23}O_2$ [M+H]: 355.1692 found 355.1690.

7,9-diphenyl-5,6,9,10-tetrahydro-5,9-epoxybenzo[8]annulene (7c): Yellow solid, m.p: 110-112 ^{o}C; IR (neat)$_{\nu max}$: 3027, 2922, 2358, 1599, 1494, 1447, 1351 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.37-7.31 (m, 2H), 7.26-7.04 (m, 10H), 6.15 (d, *J* = 2.0 Hz, 1H), 5.46 (d, *J* = 6.0 Hz, 1H), 3.32 (d, *J* = 16.0 Hz, 1H), 3.22-3.11 (m, 2H), 2.46 (d, *J* = 16.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9, 139.8, 137.7, 133.0, 131.9, 128.8, 128.6, 128.5, 128.4, 128.3, 128.2, 127.4, 127.2, 126.7, 126.0, 124.9, 73.9, 71.2, 39.3, 33.8, 31.6, 29.6. HRMS calcd for $C_{24}H_{21}O$ [M+H]: 325.1586 found 325.1588.

7,9-diphenyl-5,6,9,10-tetrahydro-5,9-epoxycycloocta[4,5]benzo[1,2-d][1,3]dioxole(7d): White solid, m.p: 179-181 ^{o}C; IR (neat)$_{\nu max}$: 3028, 2896, 2378, 1484, 1343, 1236 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (dd, *J* = 7.2 Hz, 2H), 7.35 (t, *J* = 7.2 Hz, 2H), 7.26-7.11 (m, 6H), 6.53 (d, *J* = 9.2 Hz, 2H), 6.14 (d, *J* = 2.0 Hz, 1H), 5.82 (dd, *J* = 12.8, 1.2 Hz, 2H), 5.35 (d, *J* = 5.6 Hz, 1H), 3.22 (d, *J* = 16.0 Hz, 1H), 3.17-3.10 (m, 1H), 3.01 (d, *J* = 16.0 Hz, 1H), 2.41 (d, *J* = 16.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.4, 145.9, 144.7, 139.8, 131.8, 130.6, 128.4, 128.3, 128.2, 127.4, 127.2, 126.0, 124.9, 108.5, 105.0, 100.7, 73.7, 71.2, 39.4, 33.6. HRMS calcd for $C_{25}H_{21}O_3$ [M+H]: 369.1486 found 369.1485.

9-(cyclohex-1-en-1-yl)-7-phenyl-5,6,9,10-tetrahydro-5,9-epoxybenzo[8]annulene (7e): Light yellow oil; IR (neat)$_{\nu max}$: 3023, 2925, 2364, 1730, 1600, 1493, 1446, 1349, 1264, 1073 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32-7.06 (m, 9H), 6.02 (d, *J* = 2.4 Hz, 1H), 6.00-5.98 (m, 1H), 5.37 (d, *J* = 5.6 Hz, 1H), 3.25 (d, *J* = 16.0 Hz, 1H), 3.19-3.13 (m, 1H), 2.85 (d, *J* = 16.0 Hz, 1H), 2.44 (d, *J* = 16.4 Hz, 1H), 2.31-2.26 (m, 1H), 2.15-2.10 (m, 3H), 1.75-1.58 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.2, 140.1, 137.9, 133.4, 132.7, 128.8, 128.2, 127.9, 127.2, 126.5, 125.7, 124.8, 122.0, 70.9, 36.7, 33.8, 31.6, 29.6, 25.3, 24.1, 22.9, 22.4. HRMS calcd for $C_{24}H_{25}O$ [M+H]: 329.1901 found 329.1899.

9-cyclopropyl-7-phenyl-5,6,9,10-tetrahydro-5,9-epoxybenzo[8]annulene (7f): Yellow solid, m.p: 90-92 ^{o}C; IR (neat)$_{\nu max}$: 3006, 2918, 1599, 1493, 1453, 1421, 1348, 1244, 1080 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29-7.17 (m, 5H), 7.14-7.10 (m, 2H), 7.07-7.02 (m, 2H), 5.93 (d, *J* = 2.0 Hz, 1H), 5.30 (d, *J* = 5.6 Hz, 1H), 3.15-3.09 (m, 1H), 3.05 (d, *J* = 16.4 Hz, 1H), 2.65 (d, *J* = 16.0 Hz, 1H), 2.40 (16.4 Hz, 1H), 1.29-1.20 (m, 1H), 0.62-0.59 (m, 2H), 0.56-0.49 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.0, 137.9, 133.2, 132.9, 128.7, 128.2, 127.3, 126.9, 126.6, 125.7, 124.9, 124.8, 71.7, 70.8, 37.4, 33.9, 20.0, 0.32, 0.29. HRMS calcd for $C_{21}H_{21}O$ [M+H]: 289.1584 found 289.1586.

9-cyclopropyl-3-methoxy-7-phenyl-5,6,9,10-tetrahydro-5,9-epoxybenzo[8]annulene (7g): Light yellow solid, m.p: 122-124 ^{o}C; IR (neat)$_{\nu max}$: 3005, 2915, 2348, 1613, 1504, 1426, 1268, 1152, 1080 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.17 (m, 5H), 6.95 (d, *J* = 8.4 Hz, 1H), 6.70 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.59 (d, *J* = 2.8 Hz, 1H), 5.91 (d, *J* = 2.0 Hz, 1H), 5.26 (d, *J* = 6.0 Hz, 1H), 3.75 (s, 3H), 3.14-3.08 (m, 1H), 2.97 (d, *J* = 16.0 Hz, 1H), 2.59 (d, *J* = 15.6 Hz, 1H), 2.40 (d, *J* = 16.4 Hz, 1H), 1.29-1.19 (m, 1H), 0.62-0.60 (m, 2H), 0.57-0.48 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.5, 140.0, 139.0, 132.8, 129.5, 128.2, 127.3, 126.9, 125.2, 124.8, 112.7, 110.0, 72.0, 70.9, 55.2, 36.5, 33.9, 20.0, 0.32, 0.29. HRMS calcd for $C_{22}H_{22}NaO_2$ [M+Na]: 341.1510 found 341.1512.

9-cyclopropyl-7-phenyl-5,6,9,10-tetrahydro-5,9-epoxycycloocta[4,5]benzo[1,2-d][1,3]dioxole (7h): Light brown solid, m.p: 124-126 ^{o}C; IR (neat)$_{\nu max}$: 3006, 2914, 2361, 1504, 1483, 1379, 1235, 1085 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31-7.18 (m, 5H), 6.51 (d, *J* = 9.2 Hz, 2H), 5.90 (d, *J* = 2.0 Hz, 1H), 5.87 (d, *J* = 1.2 Hz, 1H), 5.83 (d, *J* = 1.2 Hz, 1H), 5.19 (d, *J* = 6.0 Hz, 1H), 3.09-3.04 (m, 1H), 2.96 (d, *J* = 16.0 Hz, 1H), 2.53 (d, *J* = 16.0 Hz, 1H), 2.34 (d, *J* = 16.8 Hz, 1H), 1.24-1.17 (m, 1H), 0.58-0.56 (m, 2H), 0.0.53-0.48 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$)

δ 146.2, 145.7, 140.0, 132.8, 130.8, 128.2, 127.3, 126.7, 126.2, 124.8, 108.4, 105.0, 100.6, 71.6, 70.8, 37.5, 33.7, 19.9, 0.30, 0.28. HRMS calcd for $C_{22}H_{21}O_3$ [M+H]: 333.1485 found 333.1485.

9-cyclopropyl-7-(p-tolyl)-5,6,9,10-tetrahydro-5,9-epoxybenzo[8]annulene (7i): Light yellow solid, m.p: 138-140 ^oC; IR (neat)$_{\nu max}$: 3006, 2918, 2360, 1607, 1513, 1454, 1421, 1348, 1244, 1080 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.21 (d, *J* = 8.4 Hz, 2H), 7.15-7.05 (m, 6H), 5.91 (d, *J* = 2.4 Hz, 1H), 5.32 (d, *J* = 5.6 Hz, 1H), 3.16-3.11 (m, 1H), 3.08 (d, *J* = 16.8 Hz, 1H), 2.67 (d, *J* = 16.4 Hz, 1H), 2.41 (d, *J* = 16.8 Hz, 1H), 2.31 (s, 3H), 1.30-1.23 (m, 1H), 0.64-0.61 (m, 2H), 0.59-0.52 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.0, 137.1, 137.0, 133.2, 132.6, 128.9, 128.6, 126.5, 125.9, 125.6, 124.9, 124.6, 71.7, 70.8, 37.4, 33.9, 20.9, 20.0, 0.32, 0.29. HRMS calcd for $C_{22}H_{21}O_3$ [M+H]: 333.1485 found 333.1485.

9-cyclopropyl-2-methyl-7-phenyl-5,6,9,10-tetrahydro-5,9-epoxybenzo[8]annulene (7j): Light yellow solid, m.p: 76-78 ^oC ; IR (neat)$_{\nu max}$: 3005, 2919, 2352, 1599, 1495, 1446, 1422, 1346, 1246, 1079 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.32-7.21 (m, 5H), 6.97 (s, 2H), 6.88 (s, 1H), 5.95 (d, *J* = 2.0 Hz, 1H), 5.31 (d, *J* = 5.6 Hz, 1H), 3.16-3.09 (m, 1H), 3.05 (d, *J* = 16.4 Hz, 1H), 2.64 (d, *J* = 16.4 Hz, 1H), 2.43-2.31 (m, 1H), 2.29 (s, 3H), 1.31-1.23 (m, 1H), 0.64-0.61 (m, 2H), 0.55-0.53 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.0, 135.0, 132.9, 129.1, 128.2, 127.2, 126.9, 126.5, 124.85, 124.80, 71.7, 70.7, 37.4, 34.0, 21.0, 20.0, 0.32, 0.28. HRMS calcd for $C_{22}H_{21}O_3$ [M+H]: 303.1749 found 303.1750.

9-butyl-7-phenyl-5,6,9,10-tetrahydro-5,9-epoxybenzo[8]annulene (7k): Light yellow oil; IR (neat)$_{\nu max}$: 3025, 2926, 2956, 1493, 1456, 1378, 1075 cm^{-1};^1H NMR (400 MHz, $CDCl_3$): δ 7.33-7.04 (m, 9H), 6.03 (d, *J* = 2.0 Hz, 1H), 5.33 (d, *J* = 5.6 Hz, 1H), 3.17-3.11 (m, 1H), 3.05 (d, *J* = 16.4 Hz, 1H), 2.69 (d, *J* = 16.0 Hz, 1H), 2.43 (d, *J* = 16.8 Hz, 1H), 1.83-1.78 (m, 2H), 1.47-1.31 (m, 4H), 0.96 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.0, 138.0, 133.2, 132.6, 128.7, 128.2, 128.1, 127.2, 126.5, 125.7, 124.9, 124.8, 72.8, 70.7, 40.7, 38.4, 33.8, 25.4, 23.2, 14.1. HRMS calcd for $C_{22}H_{25}O$ [M+H]: 305.1899 found 305.1901.

5-butyl-7-phenyl-4,5,8,9-tetrahydro-5,9-epoxycycloocta[b]thiophene (7l): Light yellow solid, m.p: 66-68 ^oC ; IR (neat)$_{\nu max}$: 3027, 2955, 2928, 2341, 1733, 1599, 1494, 1446, 1348, 1259, 1070 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.33-7.19 (m, 5H), 7.10 (d, *J* = 4.8 Hz, 1H), 6.73 (d, *J* = 4.8 Hz, 1H), 5.97 (d, *J* = 2.0 Hz, 1H), 5.44 (d, *J* = 5.6 Hz, 1H), 3.11-3.04 (m, 1H), 2.83 (d, *J* = 16.0 Hz, 1H), 2.59 (d, *J* = 16.0 Hz, 1H), 2.42 (d, *J* = 16.4 Hz, 1H), 1.79 (t, *J* = 13.6 Hz, 2H), 1.46-1.42 (m, 4H), 0.93 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.1, 136.1, 132.8, 132.5, 128.3, 127.8, 127.3, 126.6, 124.9, 122.4, 72.8, 68.6, 40.4, 35.7, 33.6, 31.6, 29.6, 25.5, 23.2, 14.0. HRMS calcd for $C_{20}H_{22}NaOS$ [M+Na]: 333.1283 found 333.1284.

9-methyl-7-phenyl-6,9-dihydro-5H-5,9-epoxybenzo[7]annulene (7m) (two isomers are inseperable): Yellow solid, m.p: 118-120 ^oC; IR (neat)$_{\nu max}$: 3025, 2927, 2360, 1950, 1732, 1599, 1494, 1445, 1376, 1286, 1030 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.29-7.16 (m, 16H), 7.09-7.06 (m, 2H), 6.63-6.61 (m, 1H), 6.42-6.41 (m, 1H), 5.54 (d, *J* = 6.0 Hz, 1H), 5.41 (d, *J* = 4.8 Hz, 1H), 3.21-3.15 (m, 1H), 2.90 (dd, *J* = 17.2, 1.6 Hz, 1H), 2.39 (dd, *J* = 17.2, 1.6 Hz, 1H), 2.28 (dd, *J* = 17.6, 1.6 Hz, 1H), 1.78 (s, 3H), 1.76 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.7, 148.0,

145.1, 142.7, 139.5, 131.7, 131.2, 128.2, 127.4, 126.9, 126.8, 126.7, 126.5, 124.7, 120.8, 120.1, 118.1, 117.0, 81.8, 80.5, 38.3, 30.9, 29.6, 24.3, 20.5. HRMS calcd for $C_{18}H_{17}O$ [M+H]: 249.1273 found 249.1274.

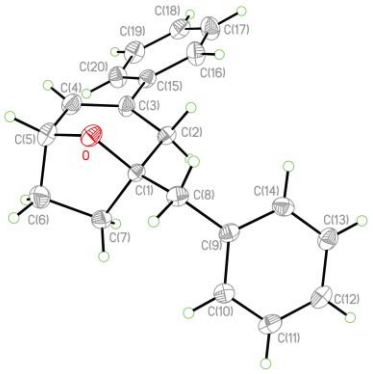
2,9-dimethyl-7-phenyl-6,9-dihydro-5H-5,9-epoxybenzo[7]annulene (7n) (two isomers are inseperable): Yellow solid, m.p: 68-70 ^{o}C; IR (neat)$_{\nu max}$: 3030, 2924, 1765, 1687, 1599, 1494, 1446, 1376, 1288, 1031 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26-7.13 (m, 10H), 6.93 (m, 4H), 6.85 (s, 1H), 6.58 (dt, *J* = 4.8, 1.6 Hz, 2H), 6.37 (t, *J* = 1.6 Hz, 1H), 5.47 (d, *J* = 6.0 Hz, 1H), 5.34 (d, *J* = 4.8 Hz, 1H), 3.15-3.09 (m, 1H), 2.85 (dt, *J* = 17.2, 1.2 Hz, 1H), 2.32 (s, 3H), 2.30 (s, 3H), 1.73 (s, 3H), 1.71 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.0, 145.4, 139.9, 139.6, 136.4, 133.5, 131.8, 131.3, 128.2, 127.4, 127.3, 127.2, 127.0, 124.7, 124.6, 121.0, 120.5, 117.9, 117.8, 81.7, 80.4, 38.3, 31.4, 30.1, 29.6, 24.3, 21.4, 20.5. HRMS calcd for $C_{19}H_{18}NaO$ [M+Na]: 285.1249 found 285.1249.

7-phenyl-9-(p-tolyl)-5,6,9,10-tetrahydro-5,9-epoxybenzo[8]annulene (7o): Light yellow oil; IR (neat)$_{\nu max}$: 3029, 2920, 2362, 1594, 1496, 1443, 1356 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, *J* = 8.4 Hz, 2H), 7.19-7.14 (m, 7H), 7.11-7.05 (m, 4H), 6.13 (d, *J* = 1.6 Hz, 1H), 5.44 (d, *J* = 5.6 Hz, 1H), 3.32-3.14 (m, 2H), 3.10 (d, *J* = 16.8 Hz, 1H), 2.45 (d, *J* = 16.8 Hz, 1H), 2.29 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.9, 139.8, 137.8, 136.9, 133.1, 131.7, 129.1, 128.8, 128.6, 128.2, 127.3, 126.7, 125.9, 124.9, 124.9, 73.7, 71.2, 39.3, 33.8, 21.0. HRMS calcd for $C_{25}H_{22}NaO$ [M+Na]: 361.1568 found 361.1569.

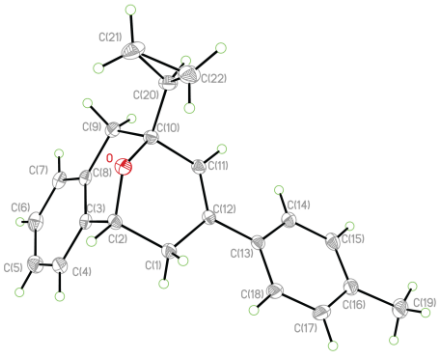
9-(2-fluorophenyl)-7-phenyl-5,6,9,10-tetrahydro-5,9-epoxybenzo[8]annulene (7p): Colorless oil; IR (neat)$_{\nu max}$: 3032, 2926, 2342, 1618, 1506, 1258 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 (td, *J* = 7.6, 1.6 Hz, 1H), 7,25 (d, *J* = 6.4 Hz, 1H), 7.16-7.06 (m, 9H), 6.98-6.93 (m, 2H), 6.49 (d, *J* = 4.8 Hz, 1H), 5.37 (d, *J* = 4.8 Hz, 1H), 3.51 (d, *J* = 14.4 Hz, 1H), 3.29 (d, *J* = 0.8 Hz, 1H), 2.83 (d, *J* = 17.6 Hz, 1H), 2.32 (dd, *J* = 16.8, 1.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.5, 160.1, 148.1, 143.9, 133.7, 133.1 (d, J_F = 3.8 Hz), 128.3, 128.3, 128.2, 127.4, 126.9 (d, J_F = 9.5 Hz), 126.7, 124.7, 123.8 (d, J_F = 3.8 Hz), 123.2, 123.0, 120.5, 118.1, 115.1, 114.9, 84.2, 76.2, 35.4, 35.2. HRMS calcd for $C_{24}H_{19}NaO$ [M+Na]: 365.1317 found 365.1319.

X-ray Structures

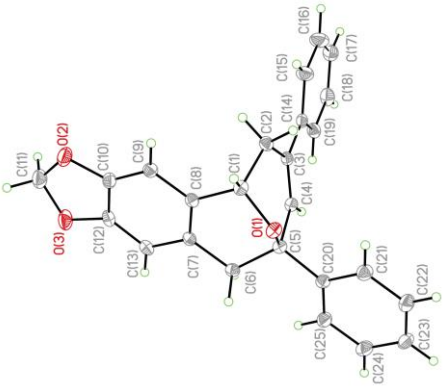
1. ***Compound 6d***



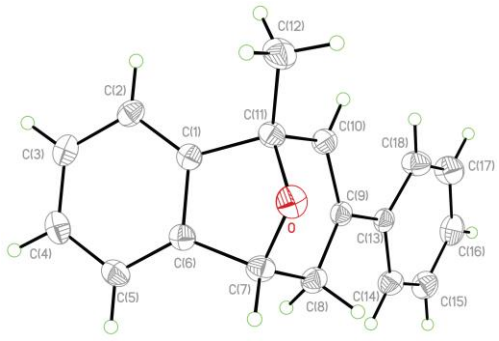
2. ***Compound 7i***



3. ***Compound 7d***



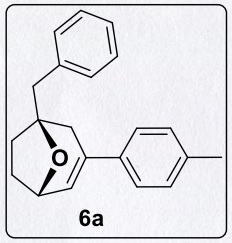
4. ***Compound 7m***



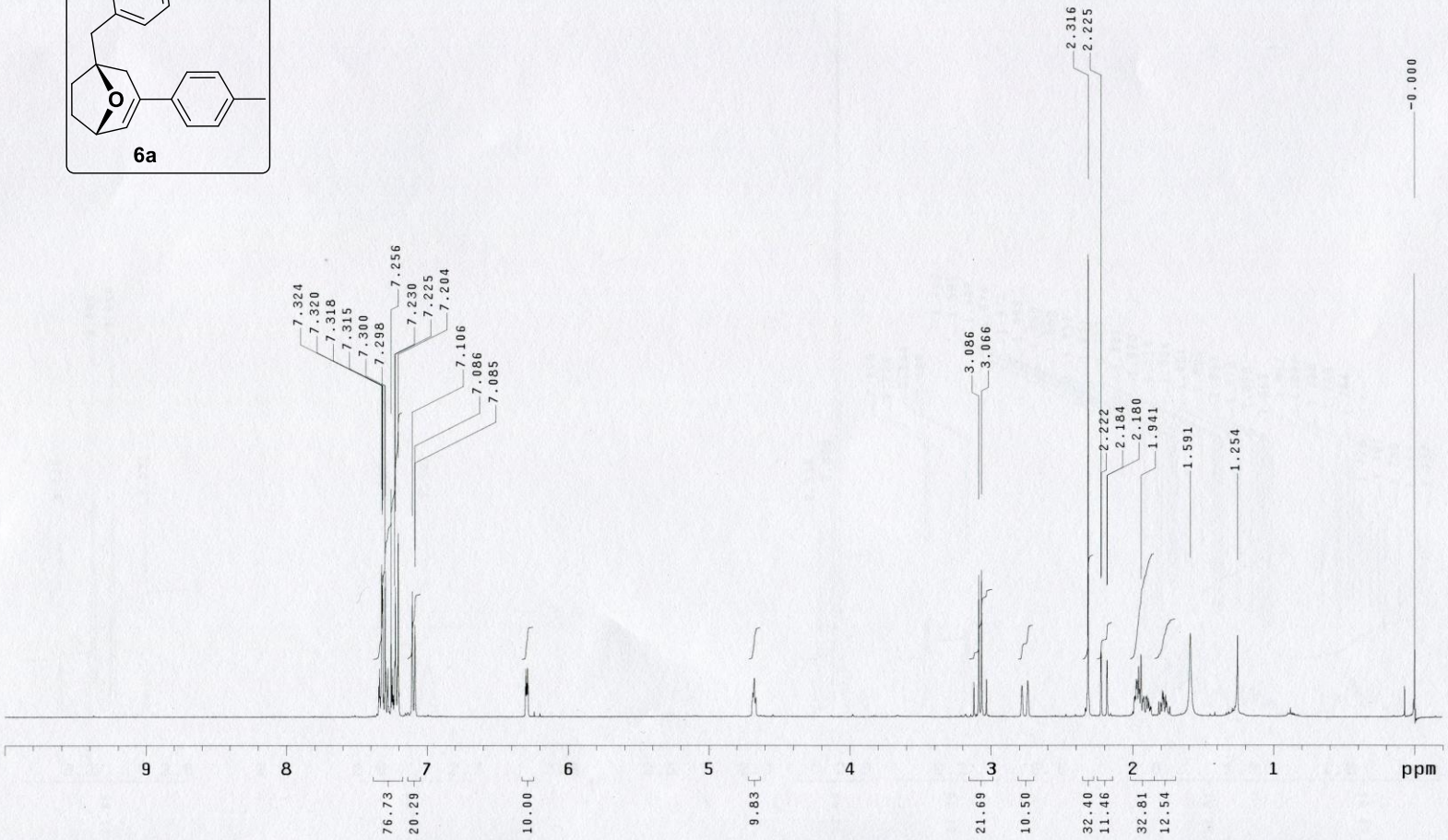
Jaya-BC2014-100

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Jun 8 2015
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



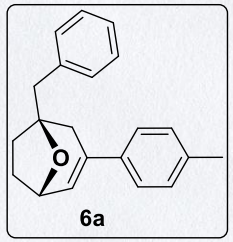
6a



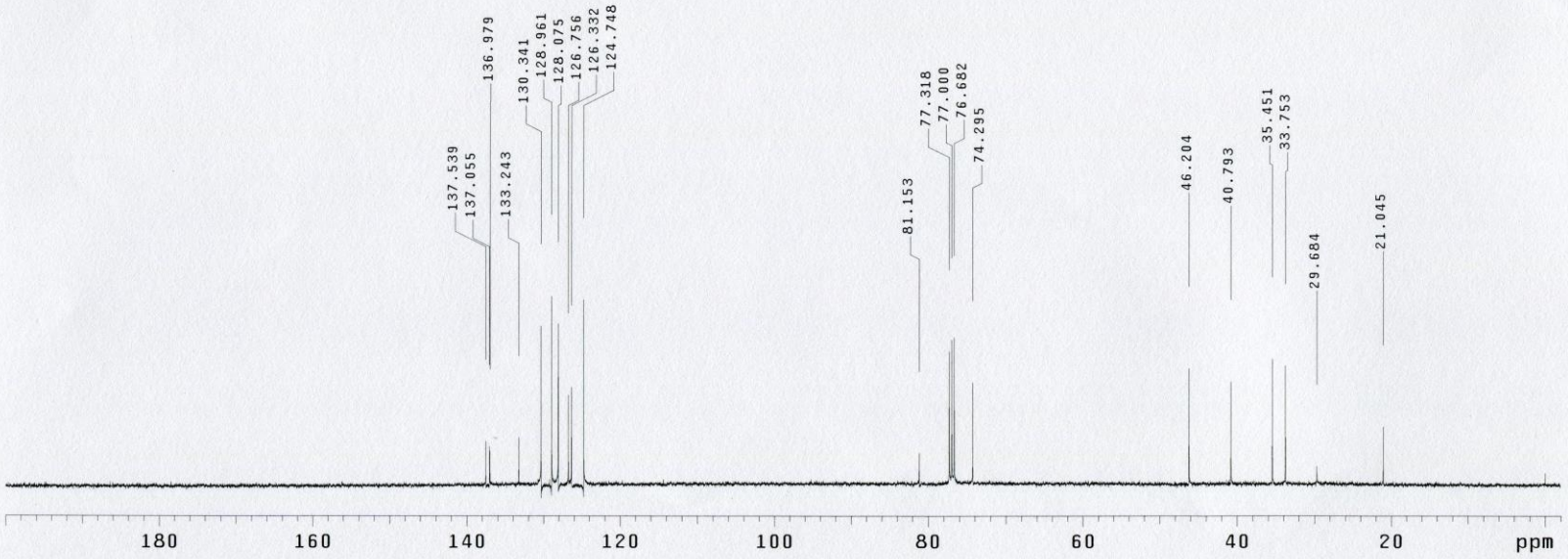
Jaya-BC2014-100

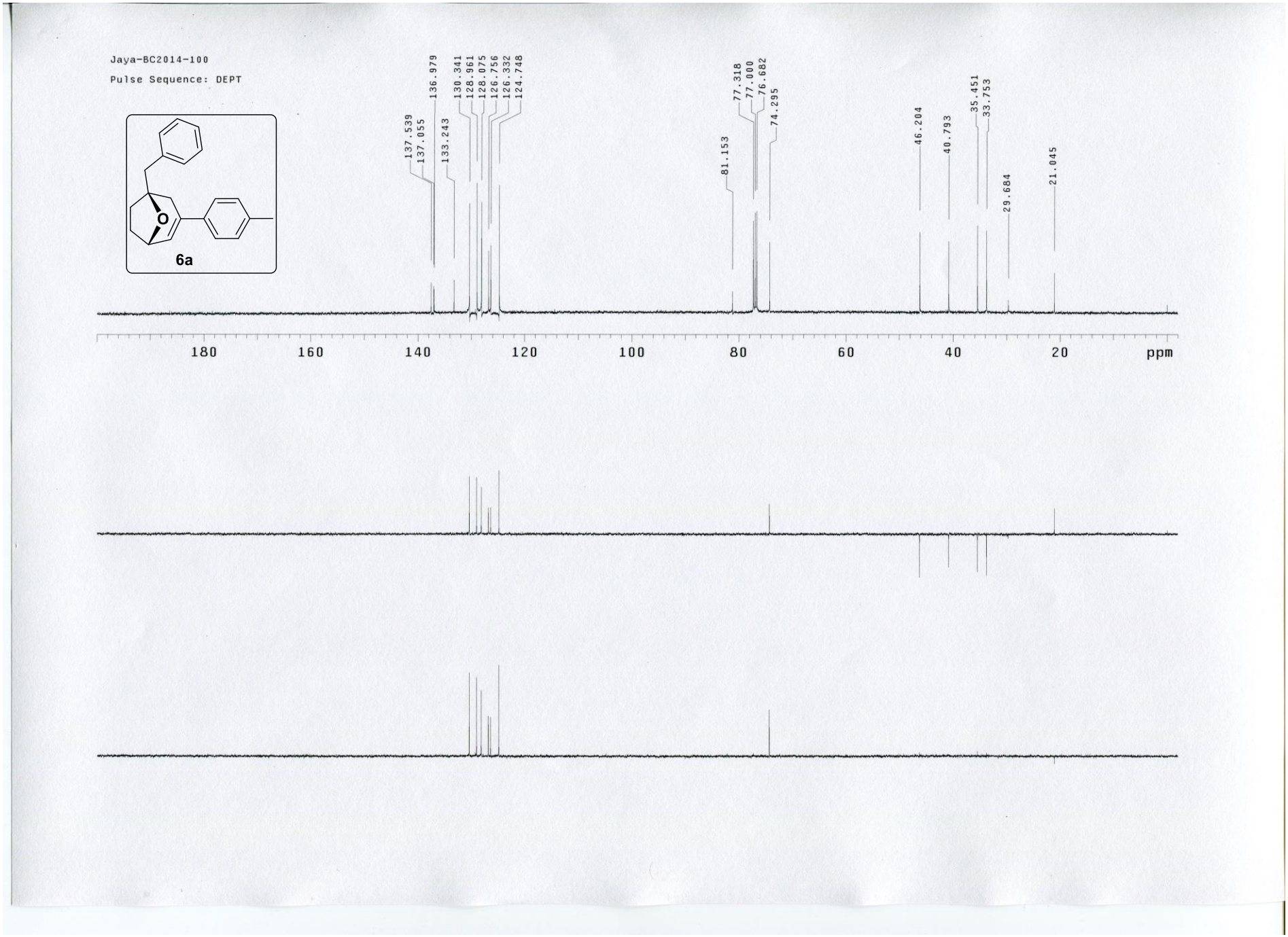
Pulse Sequence: s2pul

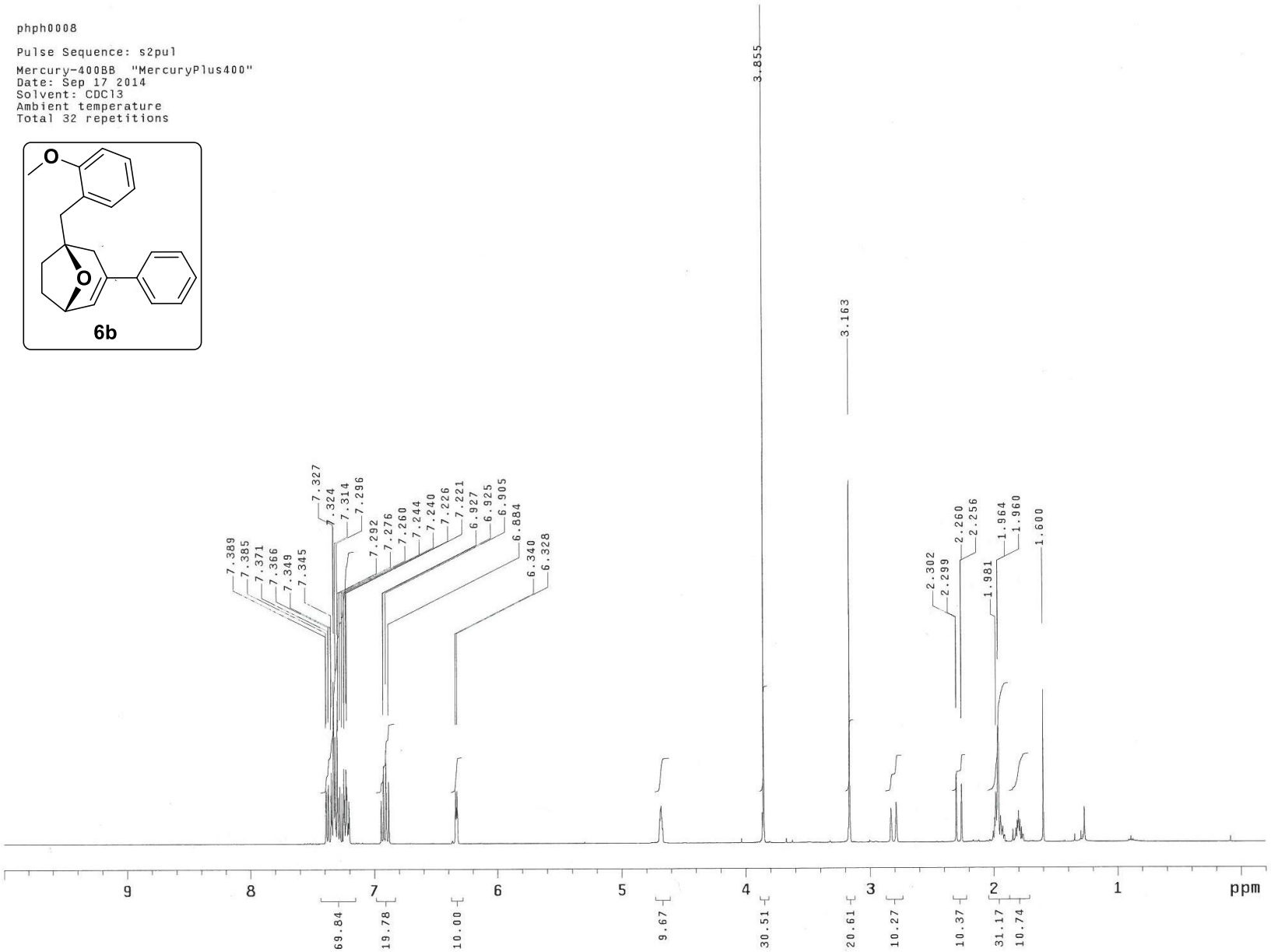
Mercury-400BB "MercuryPlus400"
Date: Jun 8 2015
Solvent: CDCl3
Ambient temperature
Total 3360 repetitions



6a



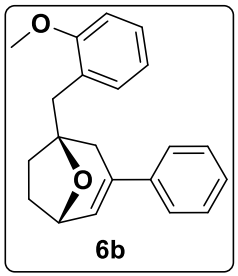

Jaya-BC2014-100
Pulse Sequence: DEPT
6a
137.539
137.055
136.979
133.243
130.341
128.961
128.075
126.756
126.332
124.748
81.153
77.318
77.000
76.682
74.295
46.204
40.793
35.451
33.753
29.684
21.045
180
160
140
120
100
80
60
40
20
ppm


phph0008
Pulse Sequence: s2pul
Mercury-400BB "MercuryPlus400"
Date: Sep 17 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions
6b
3.855
3.163
7.389
7.385
7.371
7.366
7.349
7.345
7.327
7.324
7.314
7.296
7.292
7.276
7.260
7.244
7.240
7.226
7.221
6.927
6.925
6.905
6.884
6.340
6.328
2.302
2.299
2.260
2.256
1.981
1.964
1.960
1.600
9
8
7
6
5
4
3
2
1
ppm
69.84
19.78
10.00
9.67
30.51
20.61
10.27
10.37
31.17
10.74

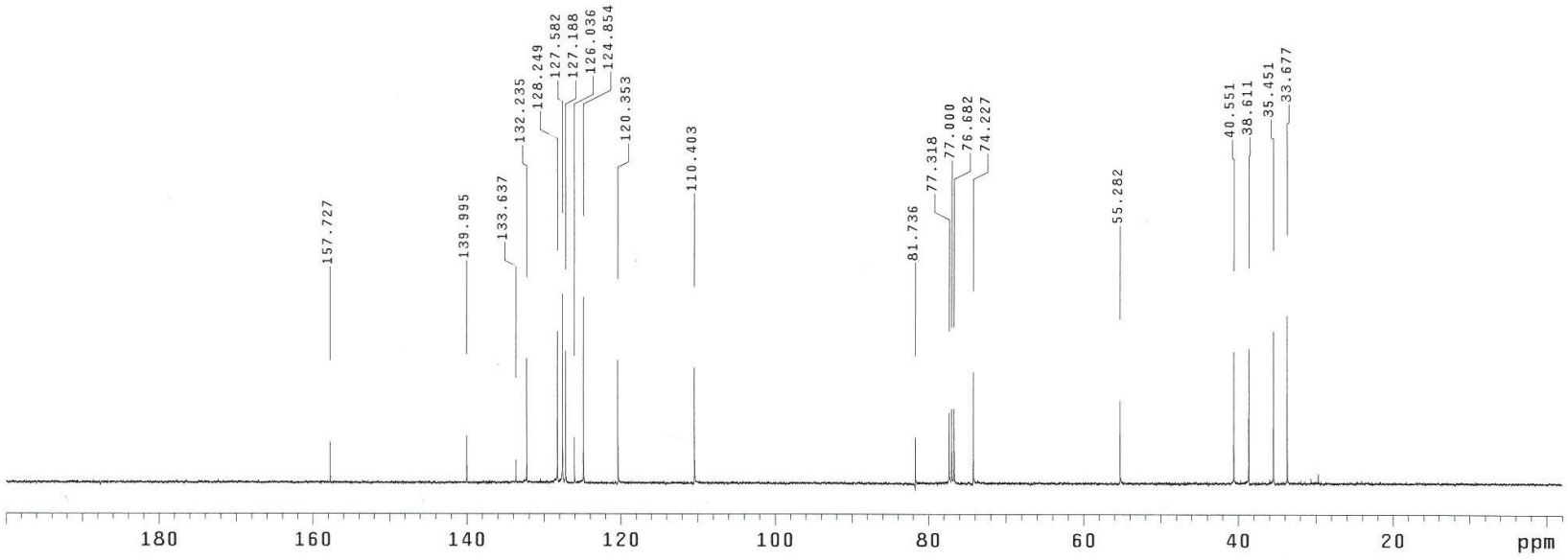
phph0008

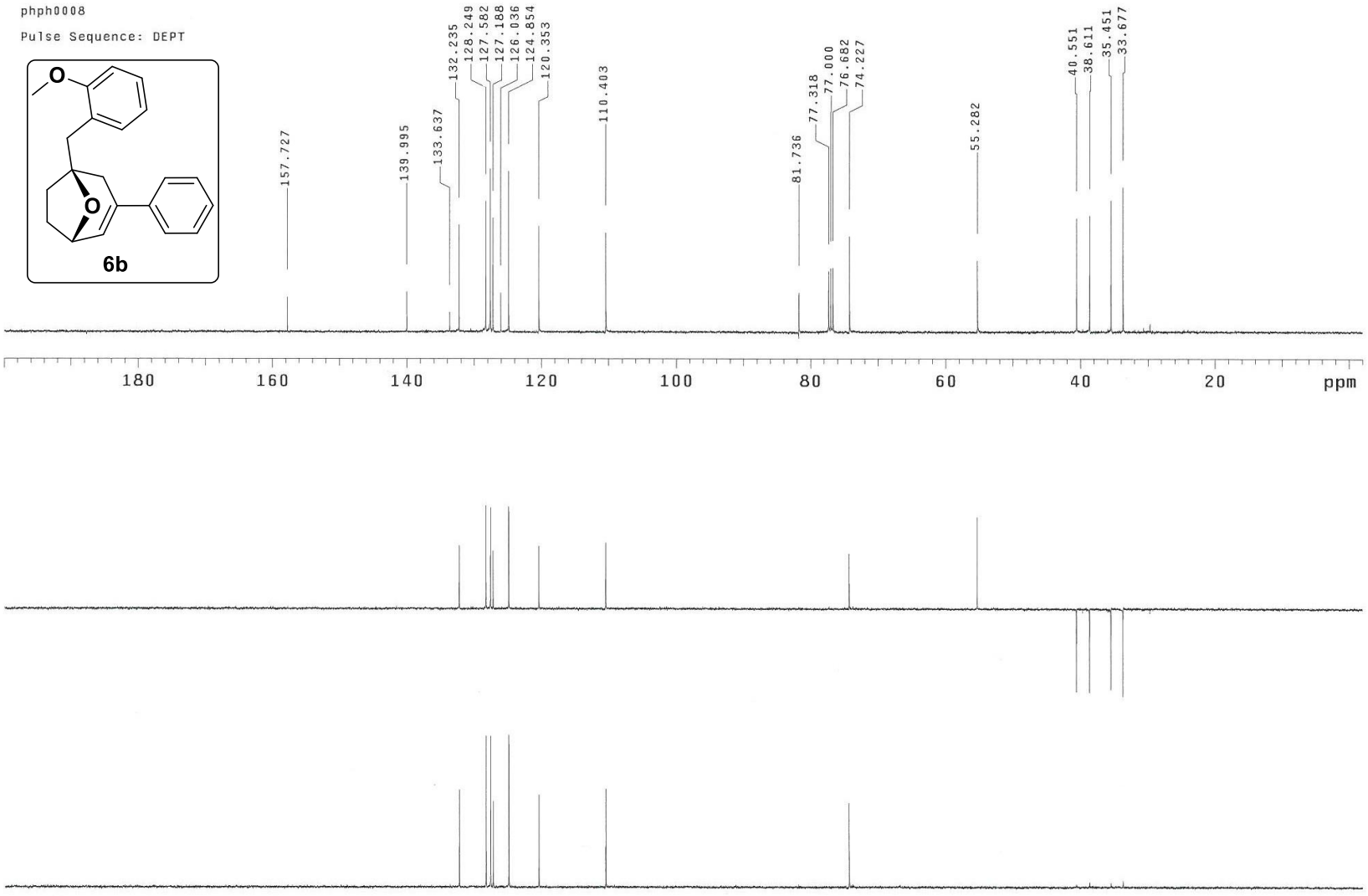
Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 17 2014
Solvent: CDCl3
Ambient temperature
Total 1504 repetitions



6b

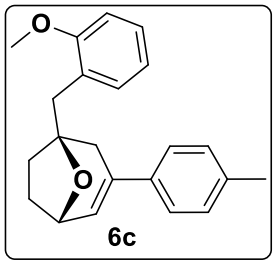



phph0008
Pulse Sequence: DEPT
O
O
6b
157.727
139.995
133.637
132.235
128.249
127.582
127.188
126.036
124.854
120.353
110.403
81.736
77.318
77.000
76.682
74.227
55.282
40.551
38.611
35.451
33.677
180
160
140
120
100
80
60
40
20
ppm

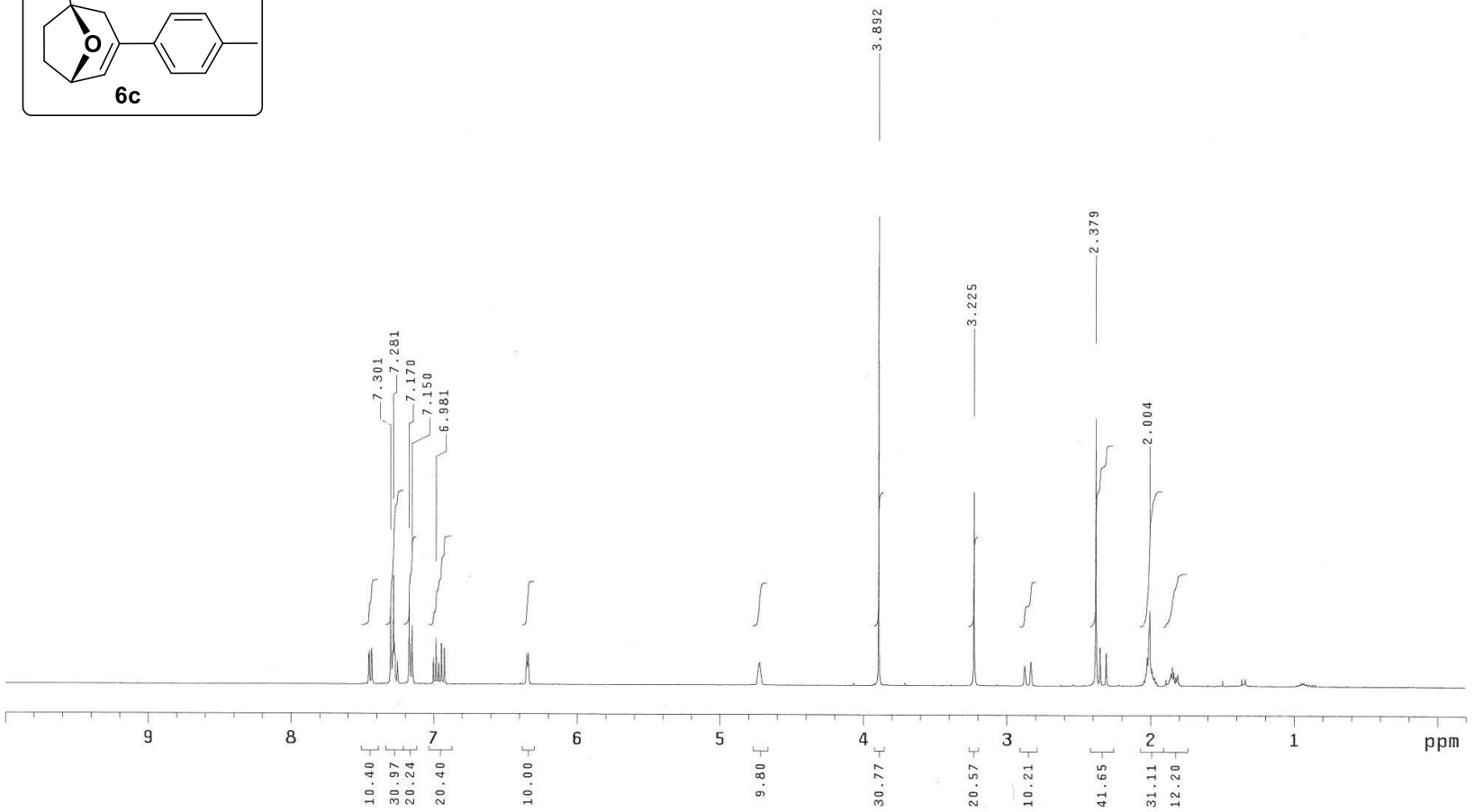
Jaya-BC2014-98-1

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Dec 25 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



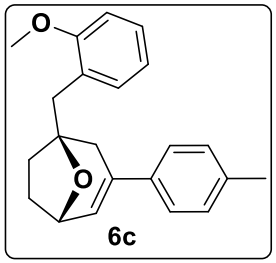
6c



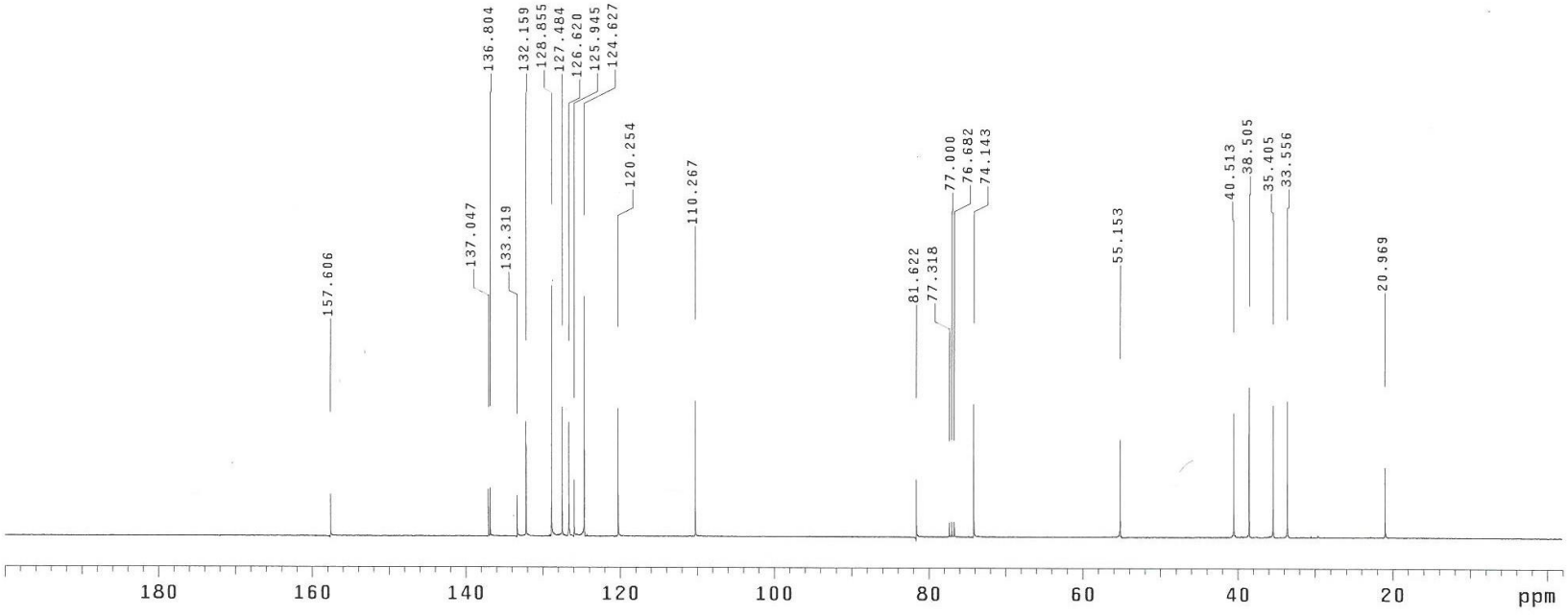
Jaya-BC2014-98-1

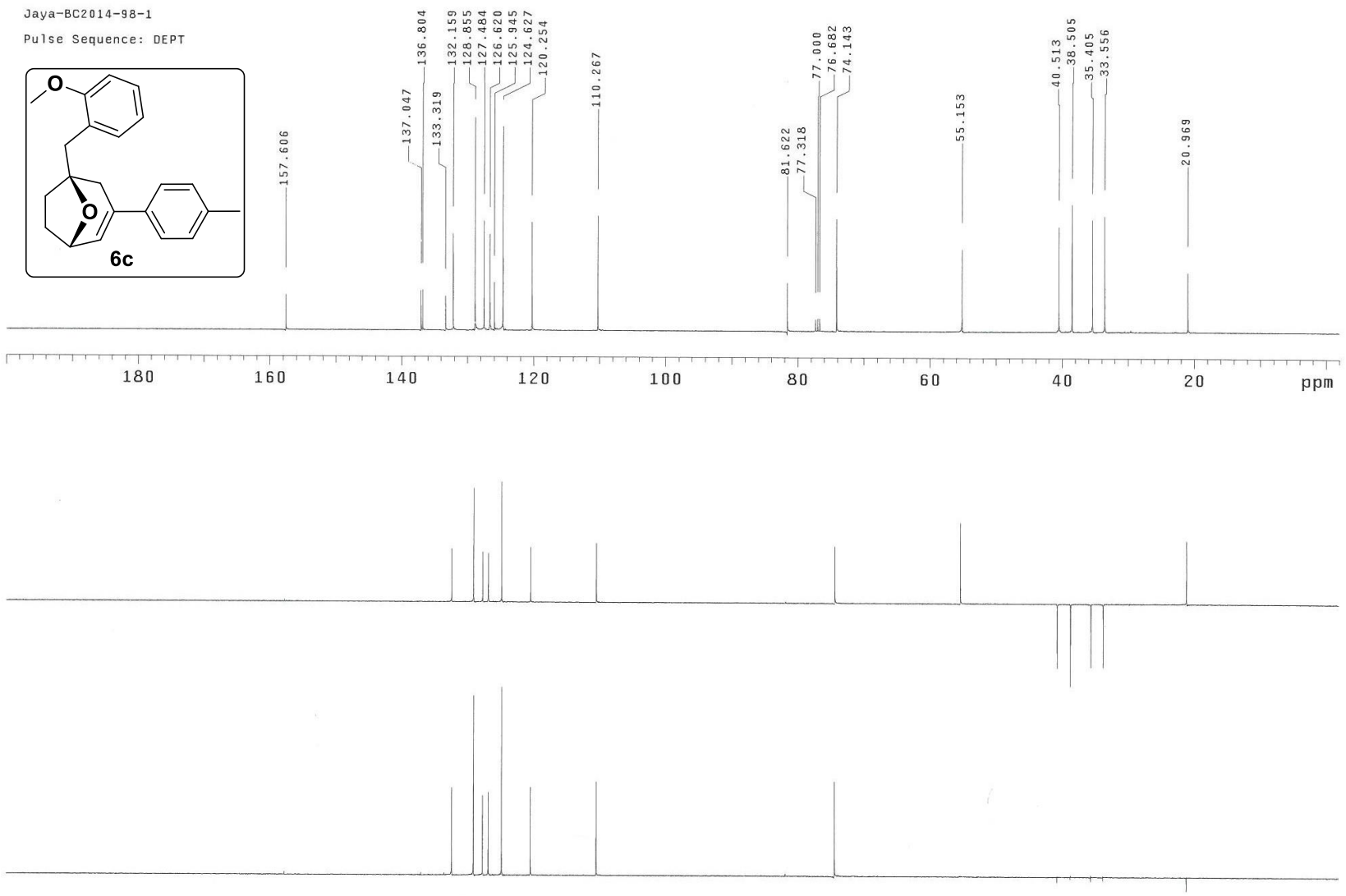
Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Dec 25 2014
Solvent: CDCl3
Ambient temperature
Total 64000 repetitions



6c

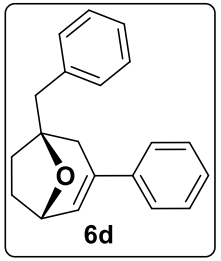



Jaya-BC2014-98-1
Pulse Sequence: DEPT
6c
157.606
137.047
136.804
133.319
132.159
128.855
127.484
126.620
125.945
124.627
120.254
110.267
81.622
77.318
77.000
76.682
74.143
55.153
40.513
38.505
35.405
33.556
20.969
180
160
140
120
100
80
60
40
20
ppm

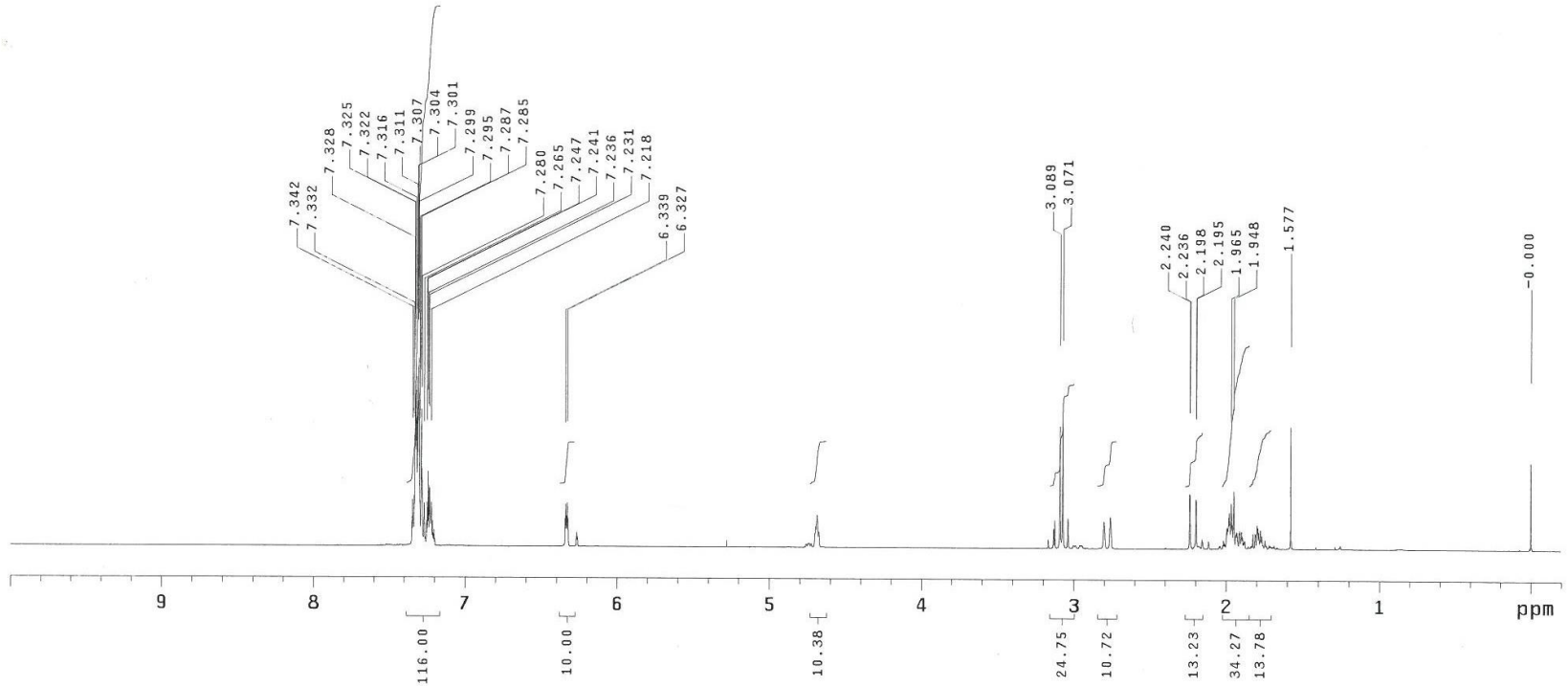
Milu-2014-P088A

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Jun 26 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



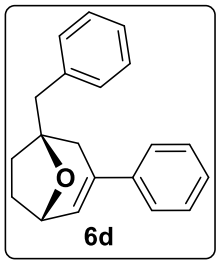
6d

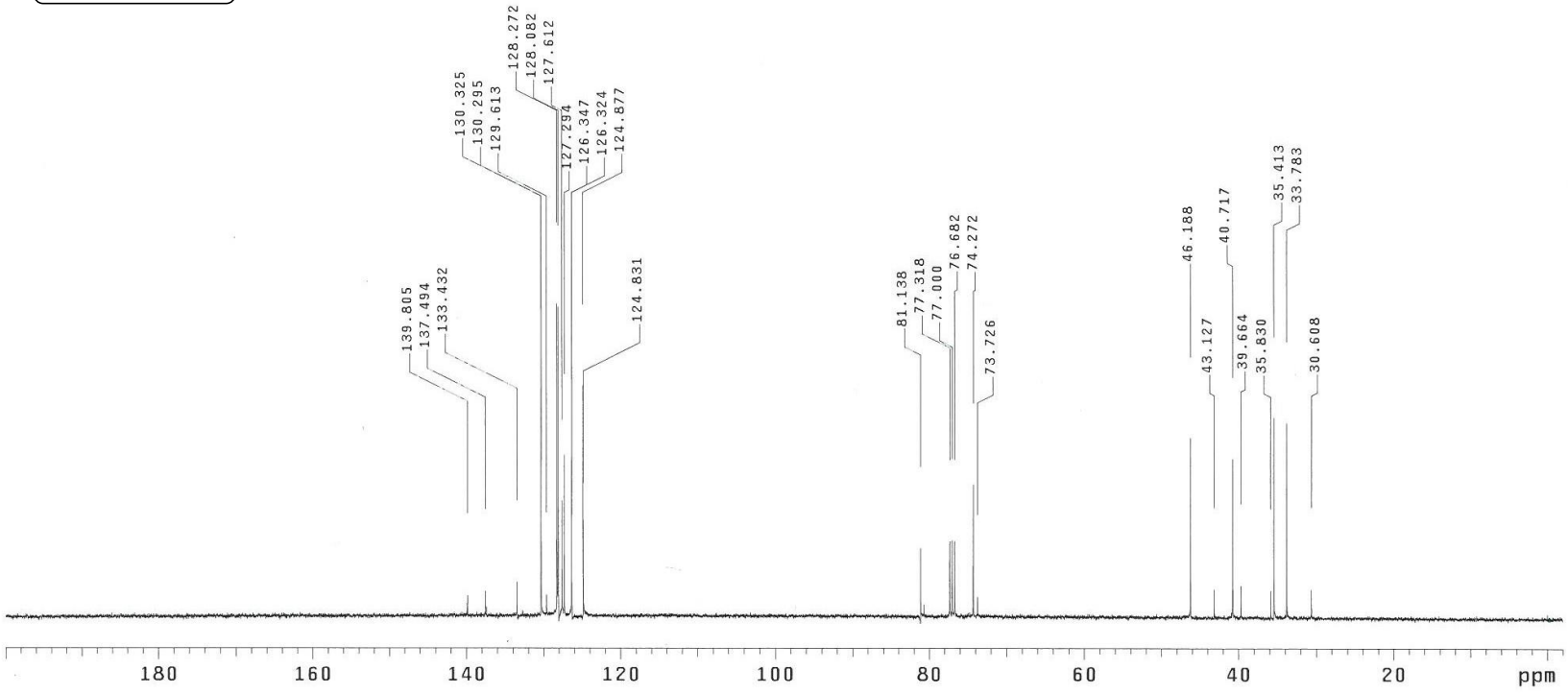


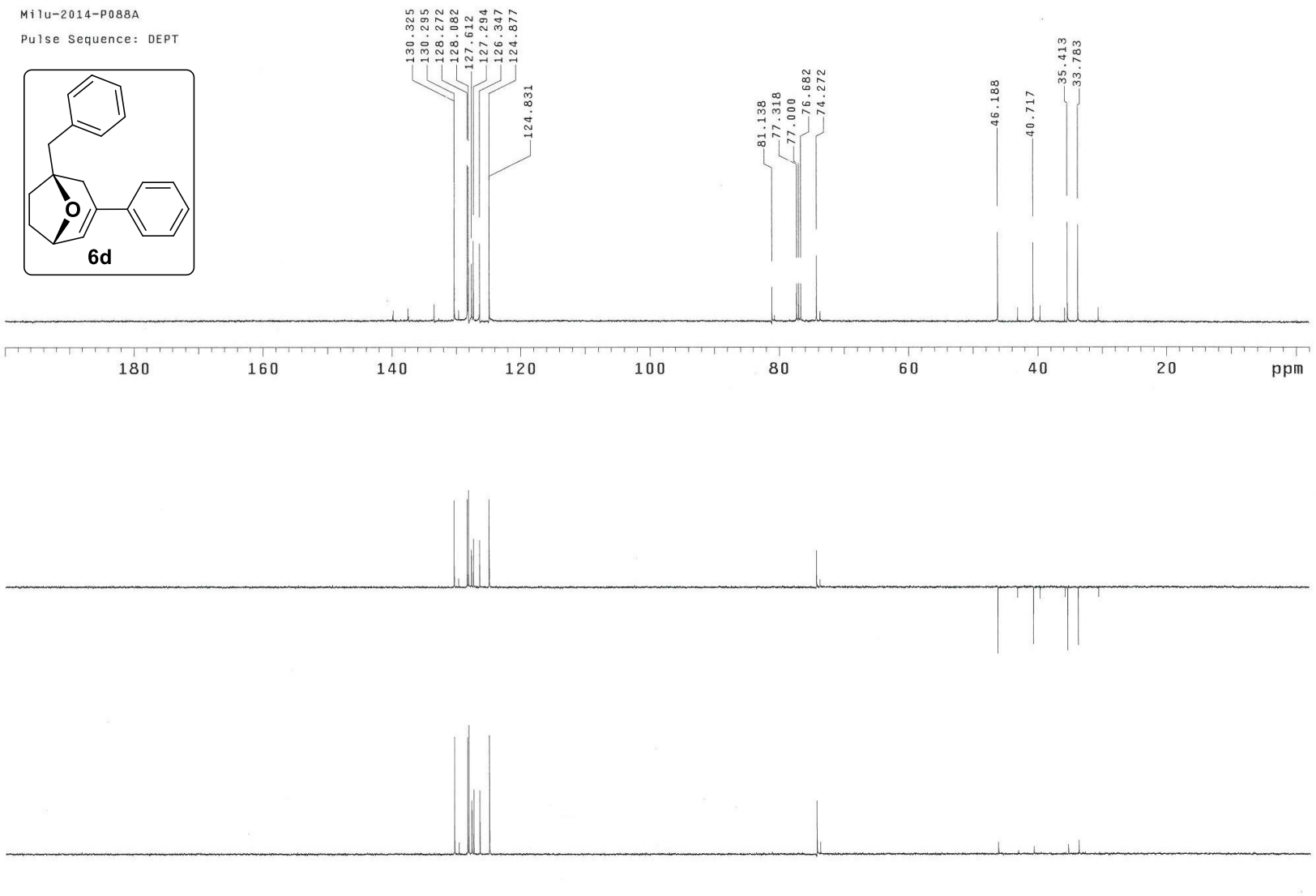
Milu-2014-P088A

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Jun 26 2014
Solvent: CDCl3
Ambient temperature
Total 1072 repetitions





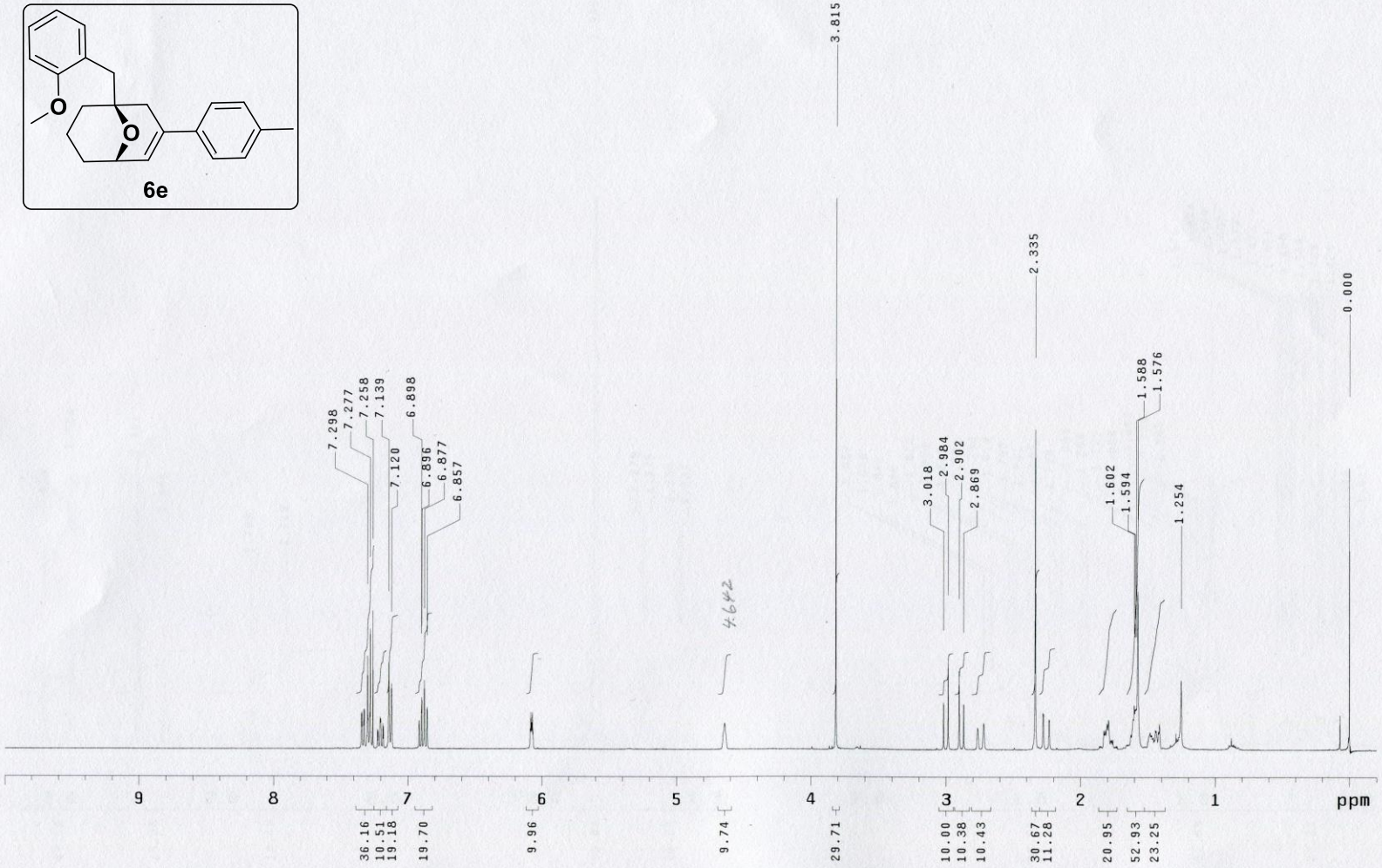

Milu-2014-P088A
Pulse Sequence: DEPT
O
6d
130.325
130.295
128.272
128.082
127.612
127.294
126.347
124.877
124.831
81.138
77.318
77.000
76.682
74.272
46.188
40.717
35.413
33.783
180
160
140
120
100
80
60
40
20
ppm

Jaya-20141225-1

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Jun 8 2015
Solvent: CDCl3
Ambient temperature
Total 48 repetitions

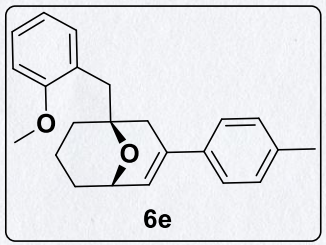
6e



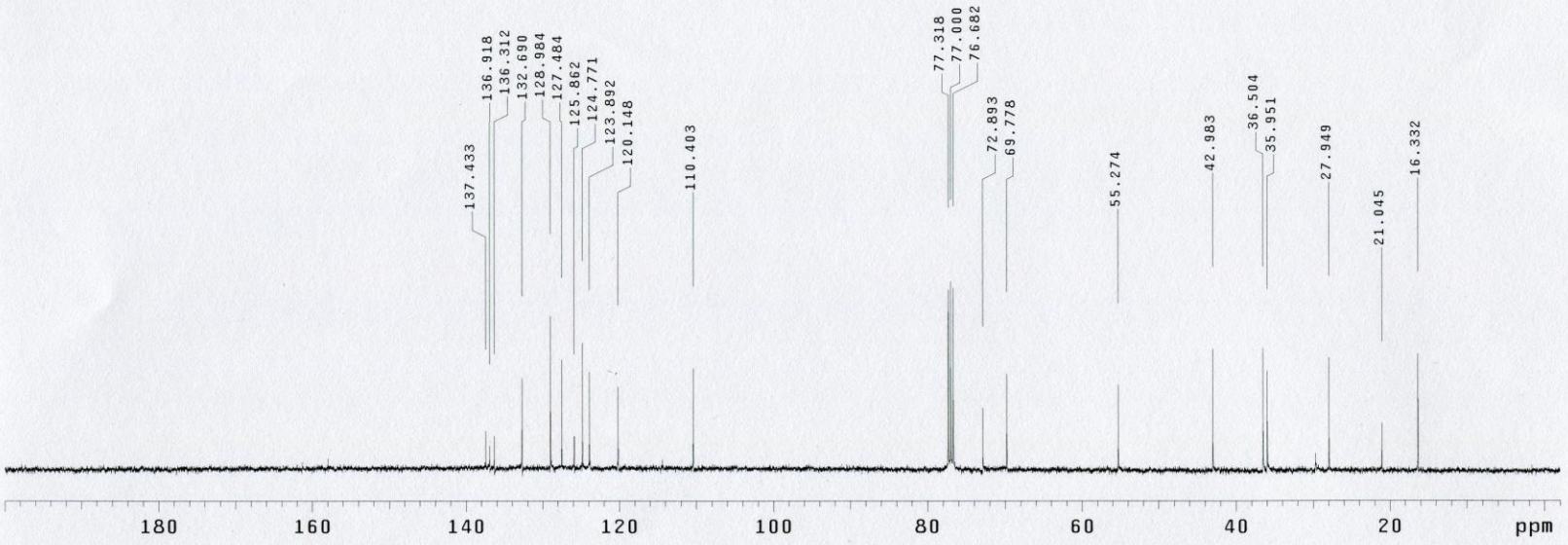
Jaya-20141225-1

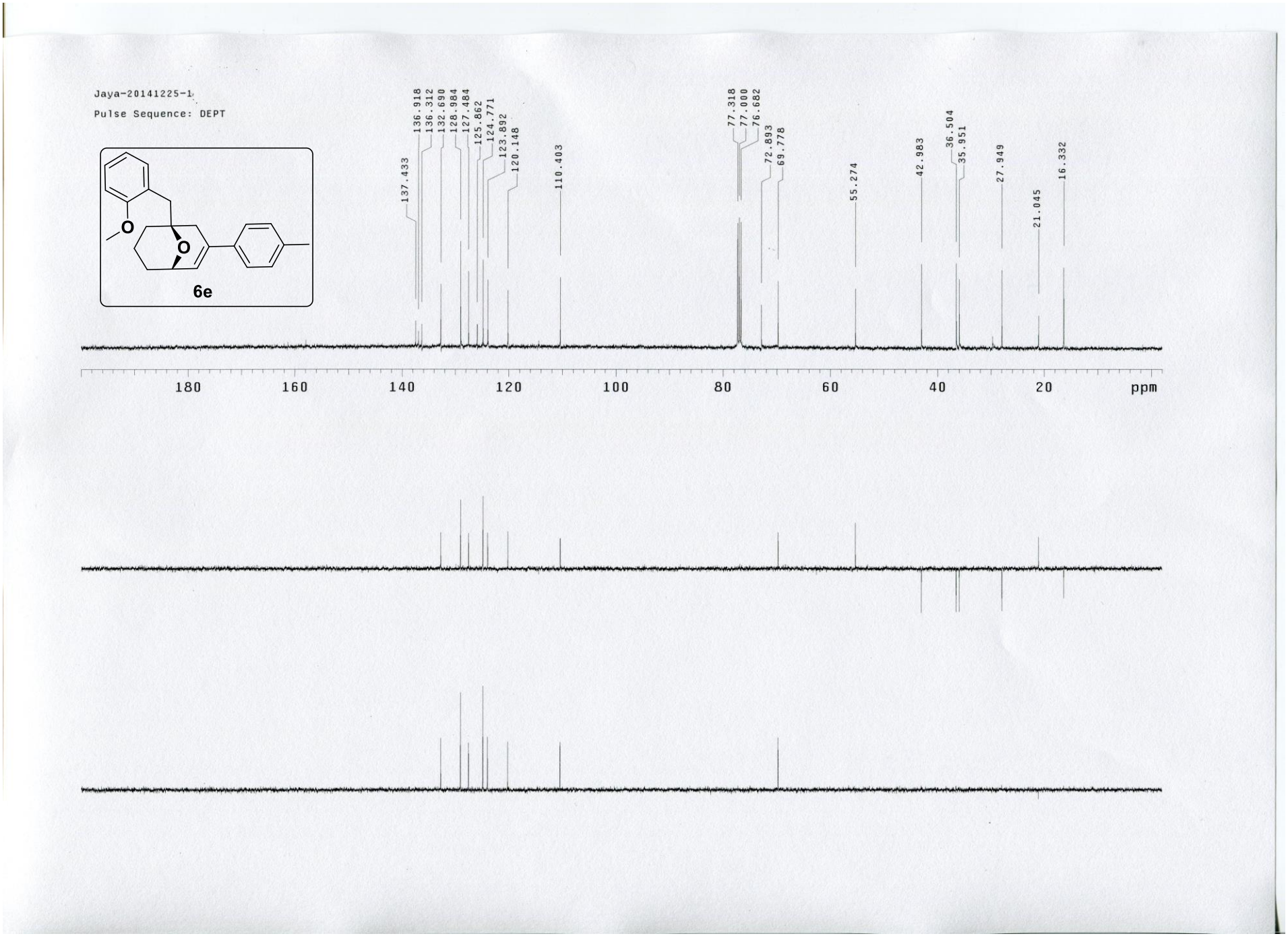
Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Jun 8 2015
Solvent: CDCl3
Ambient temperature
Total 3584 repetitions



6e

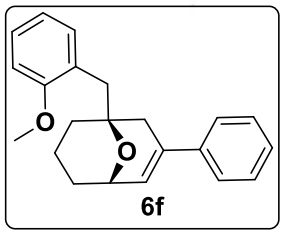



Jaya-20141225-1
Pulse Sequence: DEPT
6e
137.433
136.918
136.312
132.690
128.984
127.484
125.862
124.771
123.892
120.148
110.403
77.318
77.000
76.682
72.893
69.778
55.274
42.983
36.504
35.951
27.949
21.045
16.332
180
160
140
120
100
80
60
40
20
ppm

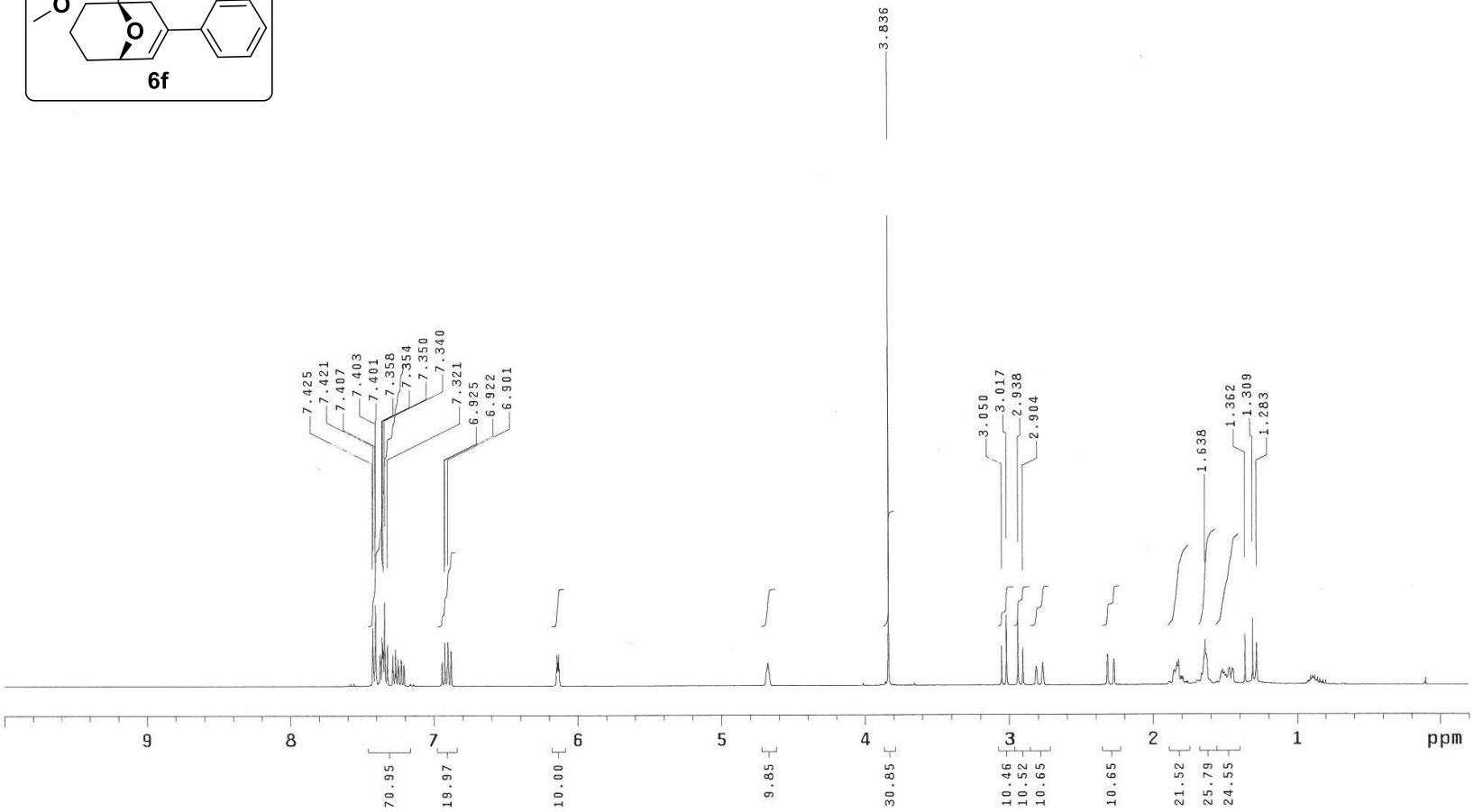
phph0009

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 17 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



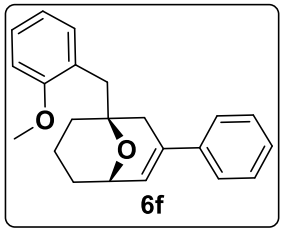
6f



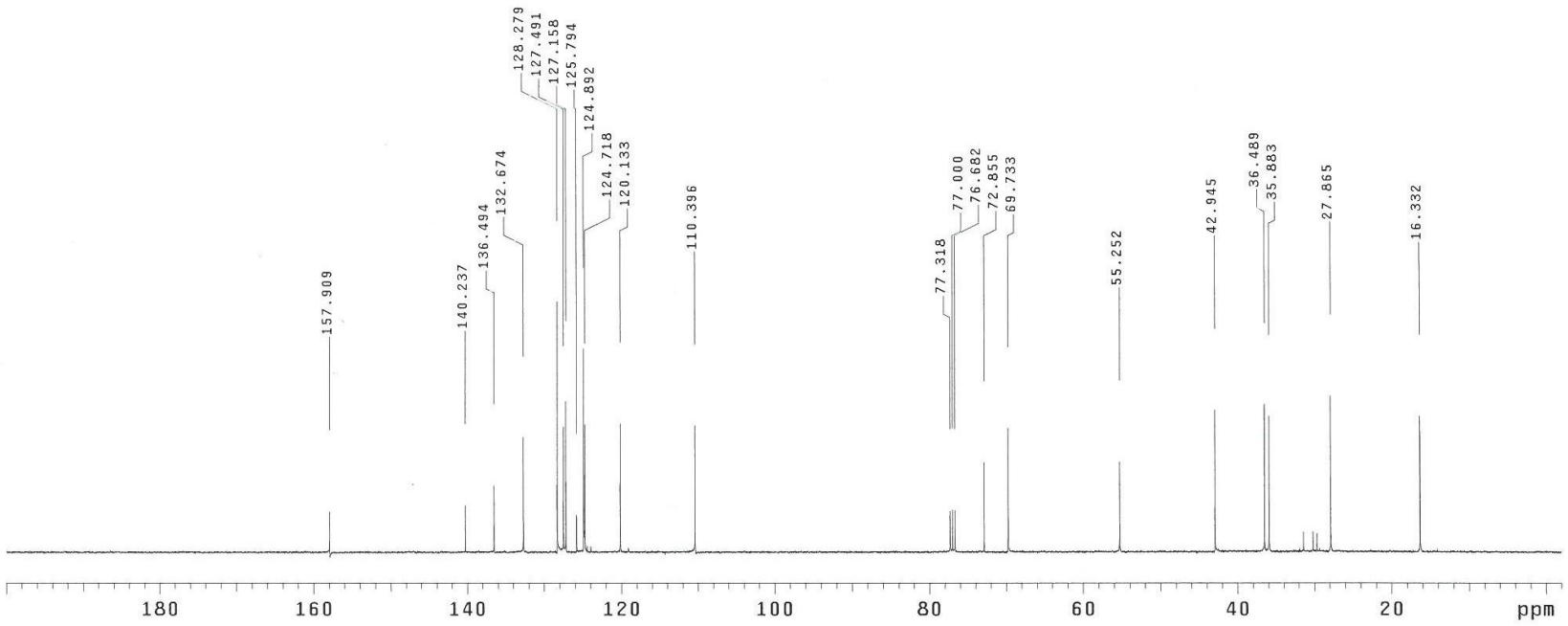
phph0009

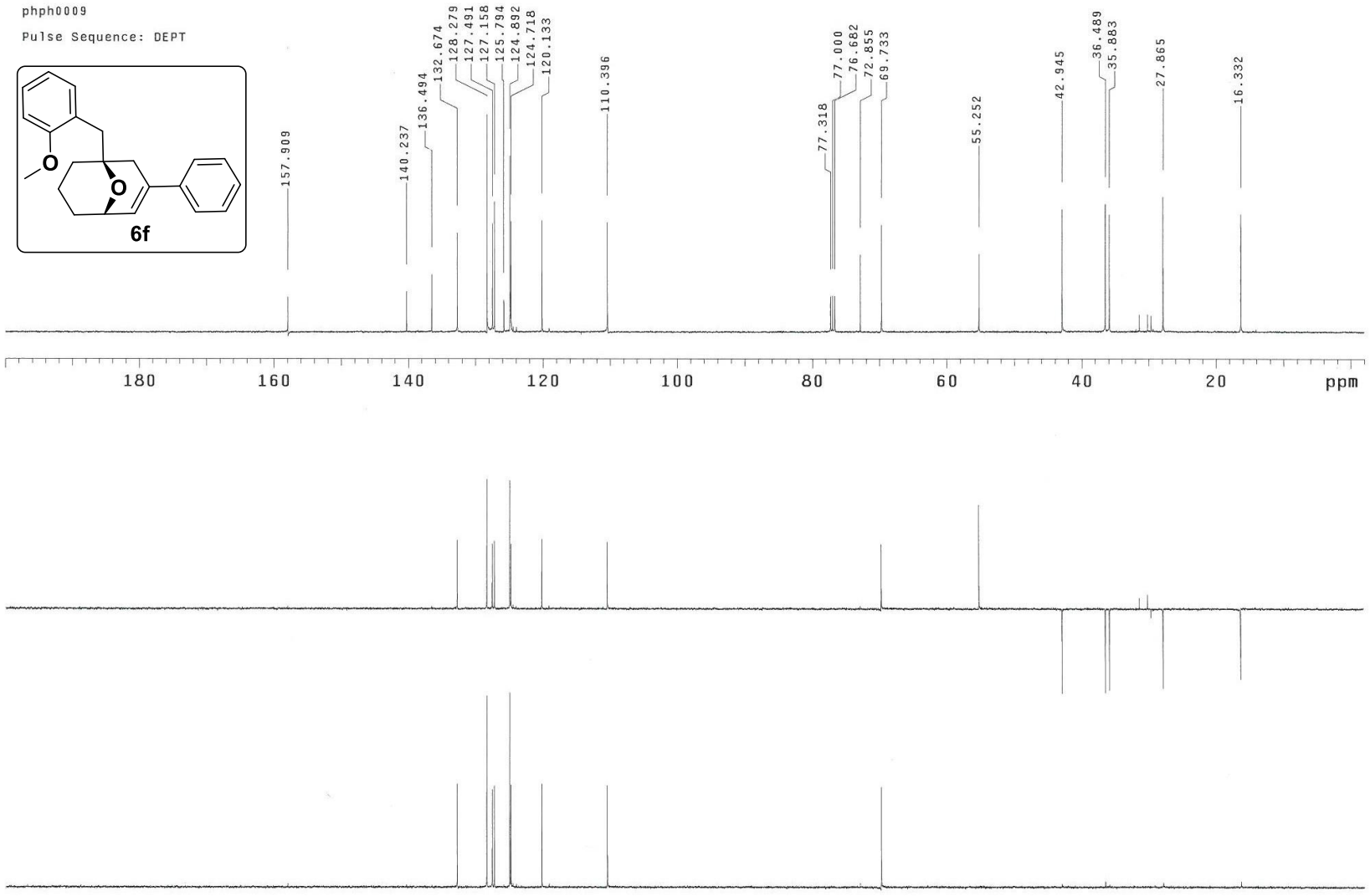
Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 17 2014
Solvent: CDCl3
Ambient temperature
Total 64000 repetitions



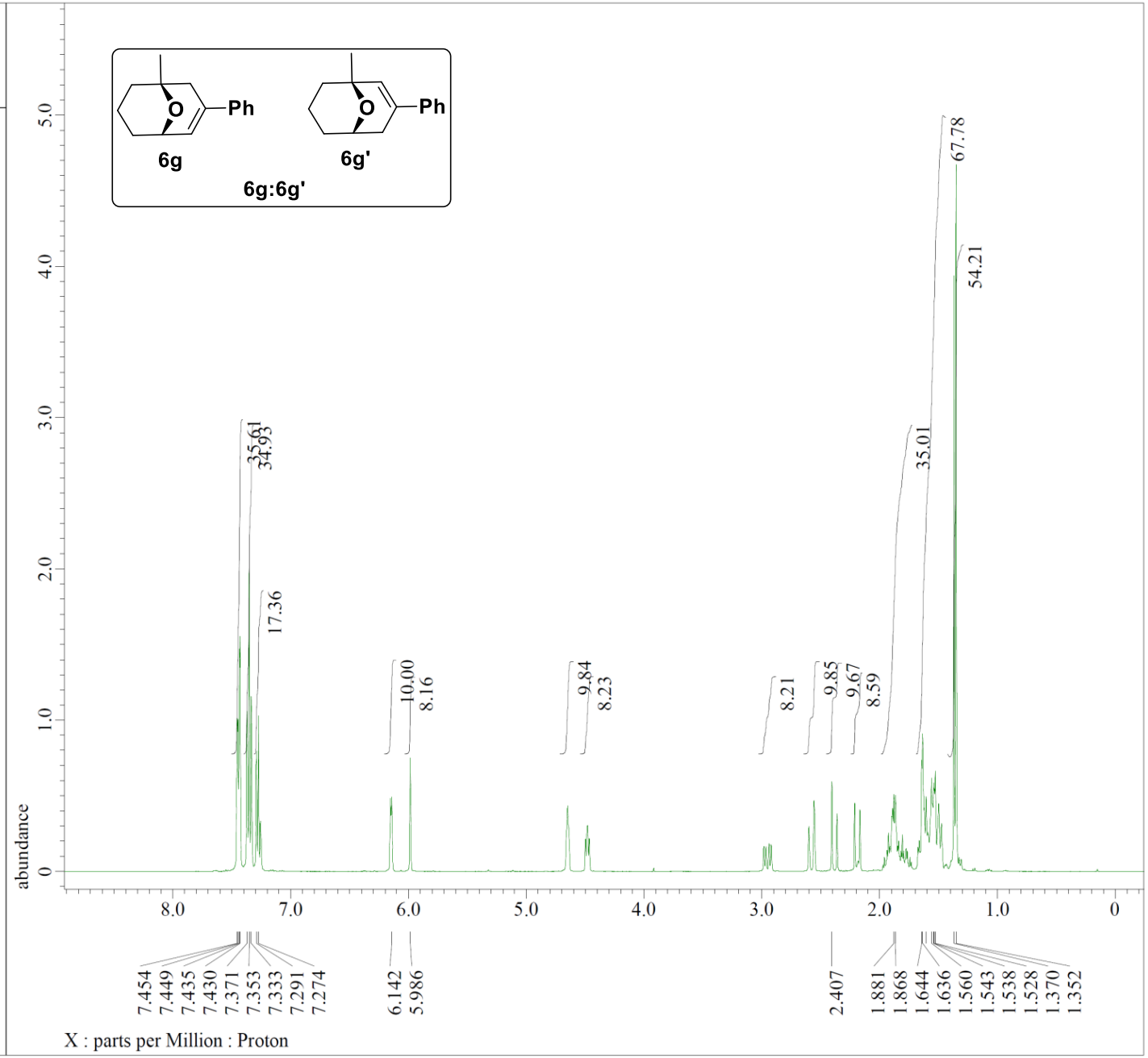
6f




phph0009
Pulse Sequence: DEPT
6f
157.909
140.237
136.494
132.674
128.279
127.491
127.158
125.794
124.892
124.718
120.133
110.396
77.318
77.000
76.682
72.855
69.733
55.252
42.945
36.489
35.883
27.865
16.332
180
160
140
120
100
80
60
40
20
ppm

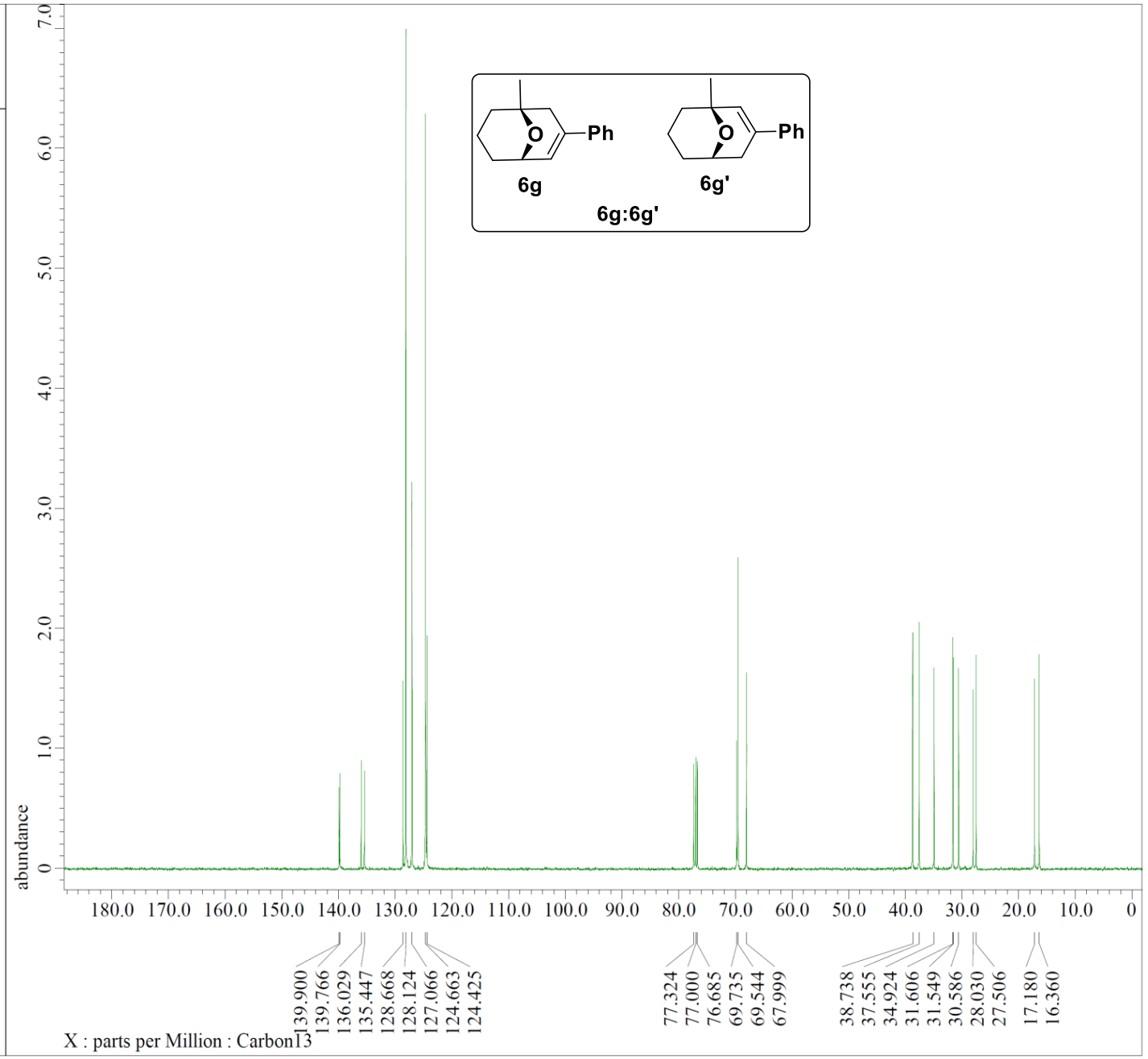
JEOL RESONANCE

```
Filename          = 104-106-106A_Proton-1-3.jd
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 104-106-106A
Solvent           = CHLOROFORM-D
Creation_Time     =  1-JUN-2015 14:22:05
Revision_Time     =  1-JUN-2015 14:47:45
Current_Time      =  1-JUN-2015 14:47:57

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 16
Temp_Get          = 22.4[dC]
X_90_Width        = 12.031[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2.4[dB]
X_Pulse           = 6.0155[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



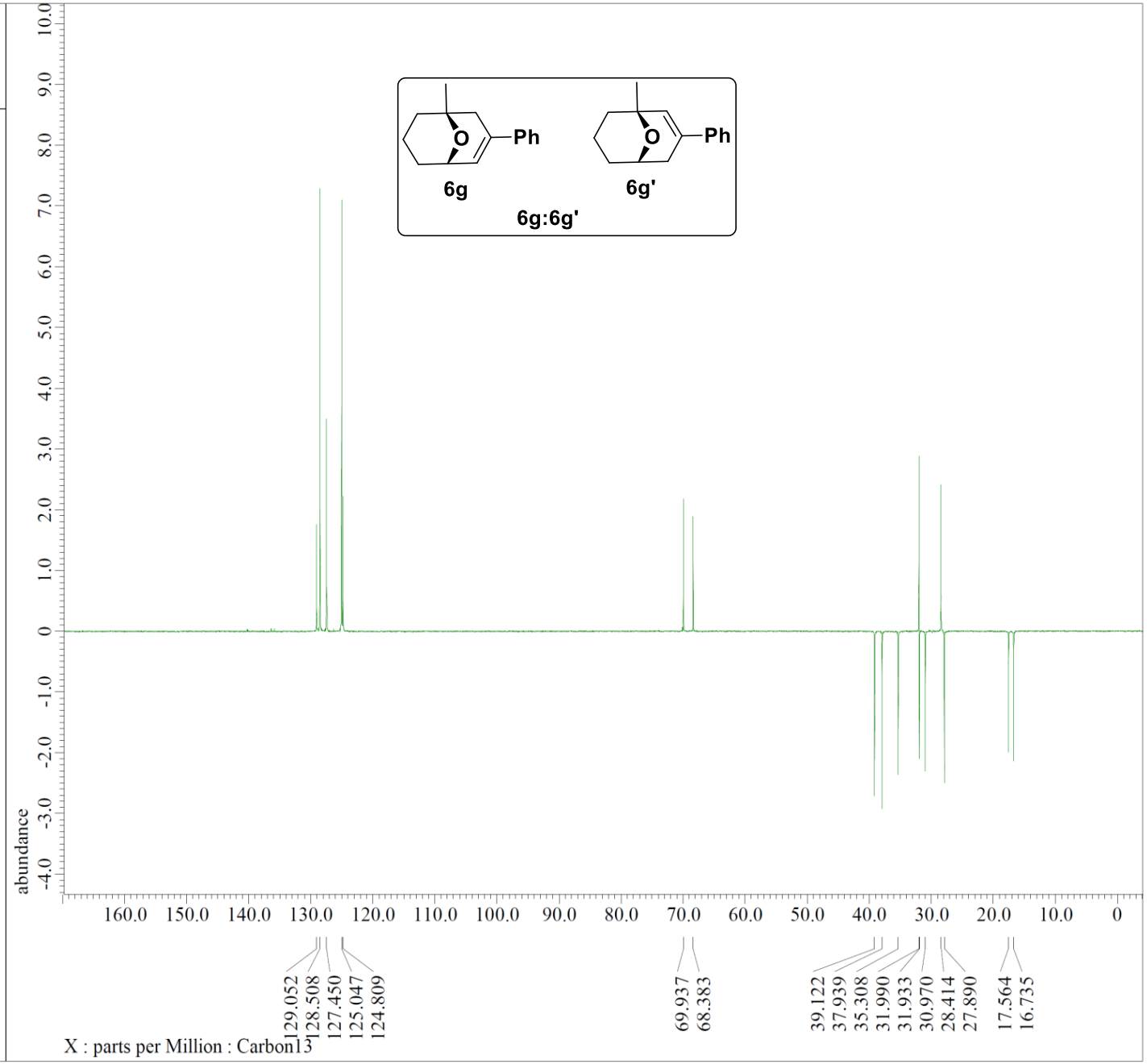
JEOL RESONANCE

```
Filename          = 104-106-106A_Carbon-1-3.jd
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 104-106-106A
Solvent           = CHLOROFORM-D
Creation_Time     =  1-JUN-2015 14:25:15
Revision_Time     =  1-JUN-2015 14:30:13
Current_Time      =  1-JUN-2015 14:31:08

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 177
Total_Scans       = 177

Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 22.7[dC]
X_90_Width        = 9.56[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 5.3[dB]
X_Pulse           = 3.18666667[us]
Irr_Atn_Dec       = 22.008[dB]
Irr_Atn_Noe       = 22.008[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



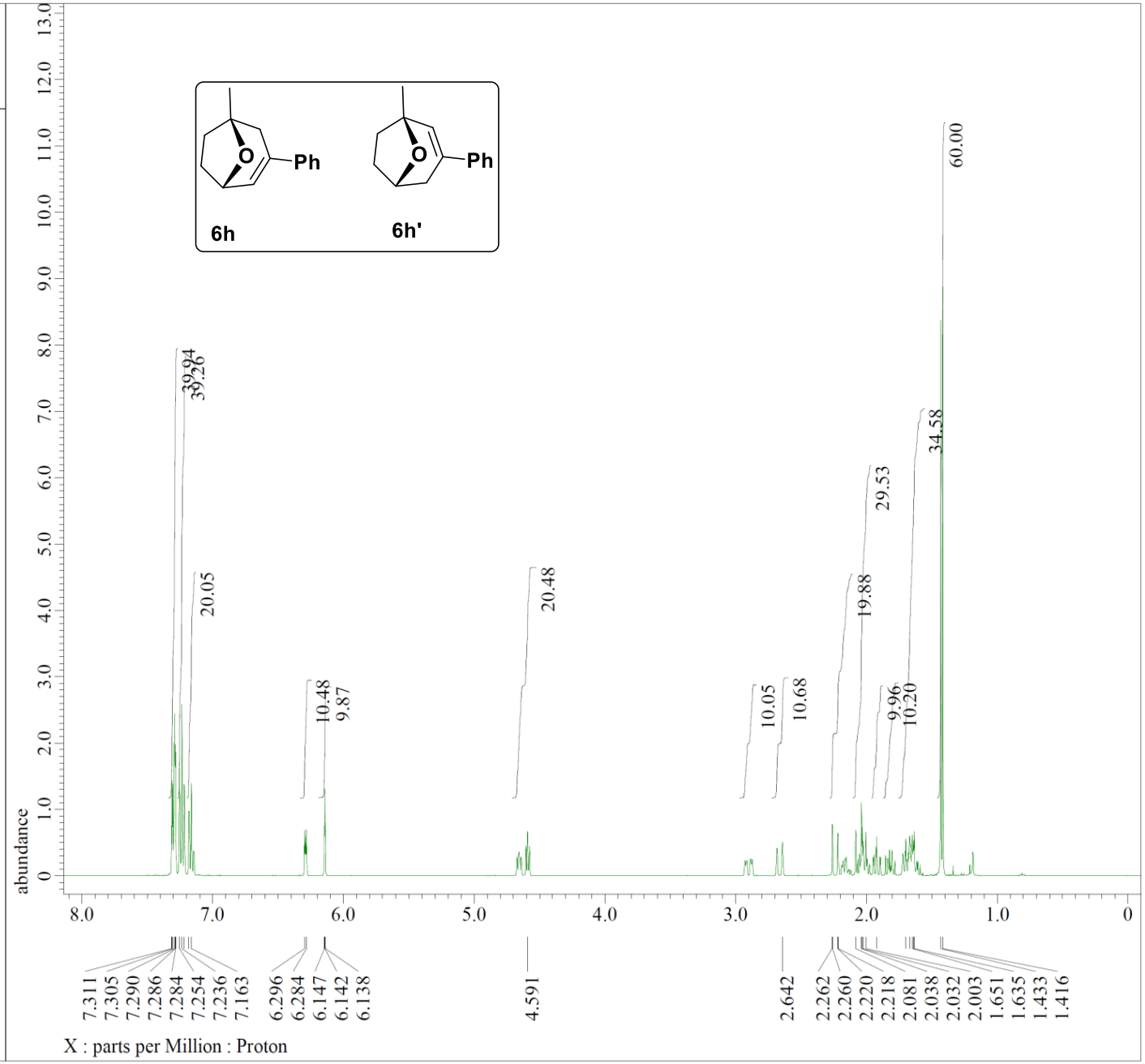
JEOL RESONANCE

```
Filename            = 104-106-106A_DEPT135deg-1
Author              = delta
Experiment          = dept.jxp
Sample_Id           = 104-106-106A
Solvent             = CHLOROFORM-D
Creation_Time       =  1-JUN-2015 14:36:31
Revision_Time       =  1-JUN-2015 14:40:29
Current_Time        =  1-JUN-2015 14:40:50

Comment             = DEPT with decoupling
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 143
Total_Scans         = 143

Relaxation_Delay    = 2[s]
Recvr_Gain          = 56
Temp_Get            = 22.5[dC]
X_Acq_Time          = 1.04333312[s]
X_Atn               = 5.3[dB]
X_Pulse             = 9.56[us]
Irr_Atn             = 2.4[dB]
Irr_Atn_Dec         = 22.008[dB]
Irr_Noise           = WALTZ
Irr_Pulse           = 12.031[us]
Irr_Pwidth          = 0.115[ms]
Base_Line_Correct   = TRUE
Decoupling          = TRUE
Initial_Wait        = 1[s]
J_Constant          = 140[Hz]
Selection_Angle     = 135[deg]
Selection_Factor    = 1
Selection_Pulse     = 18.0465[us]
```



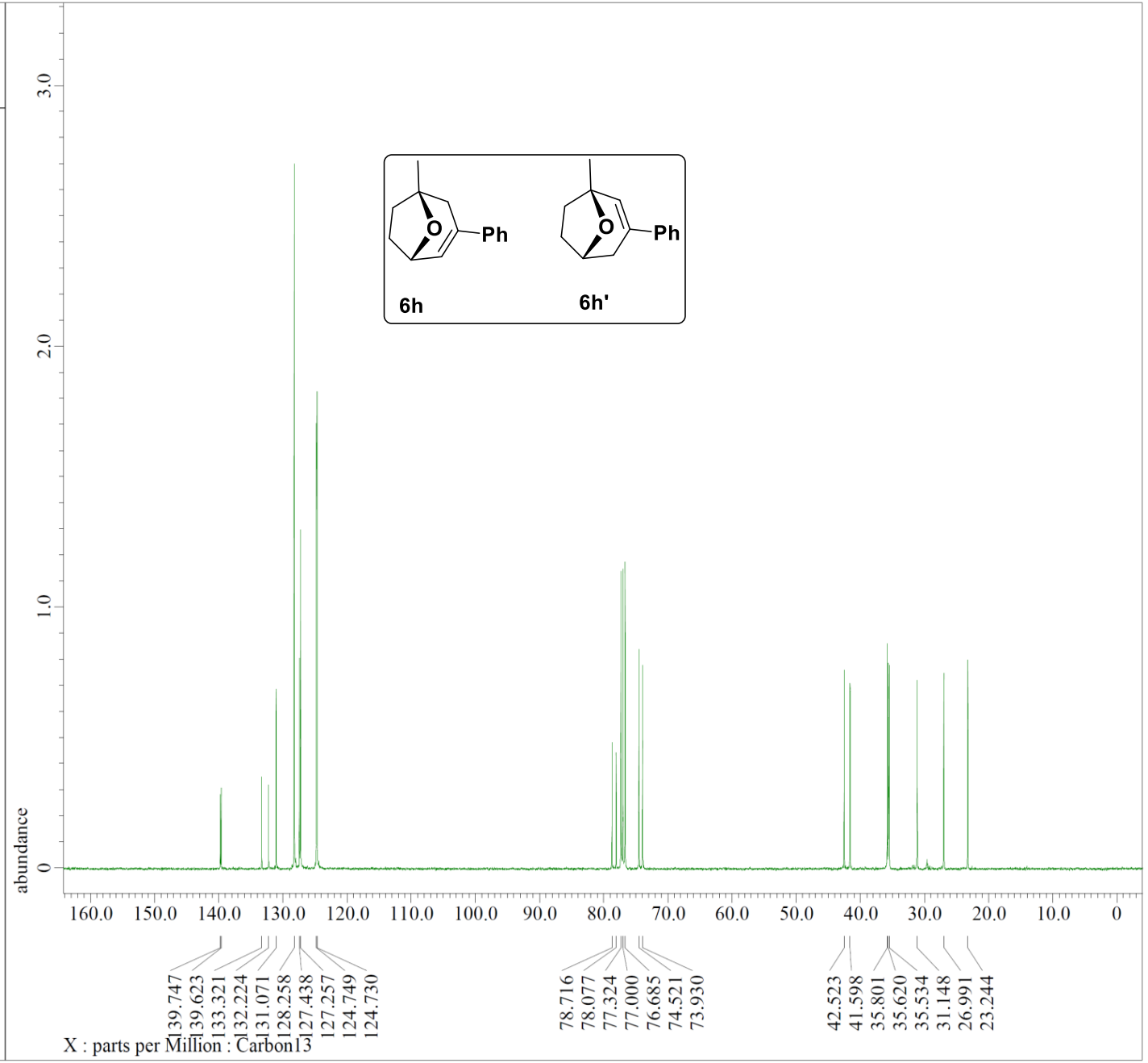
JEOL RESONANCE

```
Filename          = 104-96-96A_Proton-2-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 104-96-96A
Solvent           = CHLOROFORM-D
Creation_Time     = 25-MAY-2015 17:03:10
Revision_Time     = 25-MAY-2015 18:18:57
Current_Time      = 25-MAY-2015 18:19:21

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 21.8[dC]
X_90_Width        = 12.031[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2.4[dB]
X_Pulse           = 6.0155[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



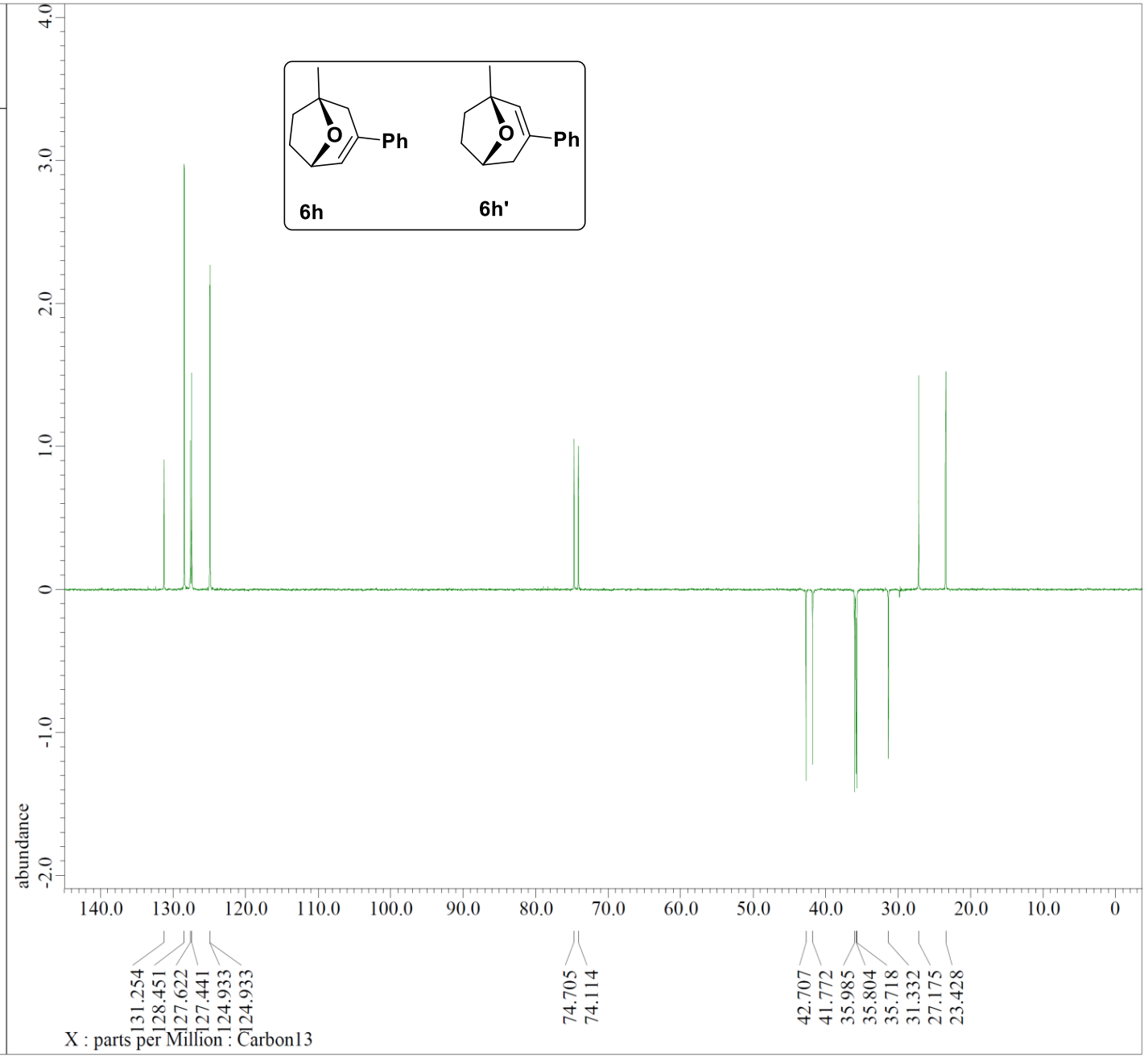
JEOL RESONANCE

```
Filename          = 104-96-96A_Carbon-2-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 104-96-96A
Solvent           = CHLOROFORM-D
Creation_Time     = 25-MAY-2015 17:06:19
Revision_Time     = 25-MAY-2015 18:23:13
Current_Time      = 25-MAY-2015 18:23:47

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22[dC]
X_90_Width        = 9.56[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 5.3[dB]
X_Pulse           = 3.18666667[us]
Irr_Atn_Dec       = 22.008[dB]
Irr_Atn_Noe       = 22.008[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



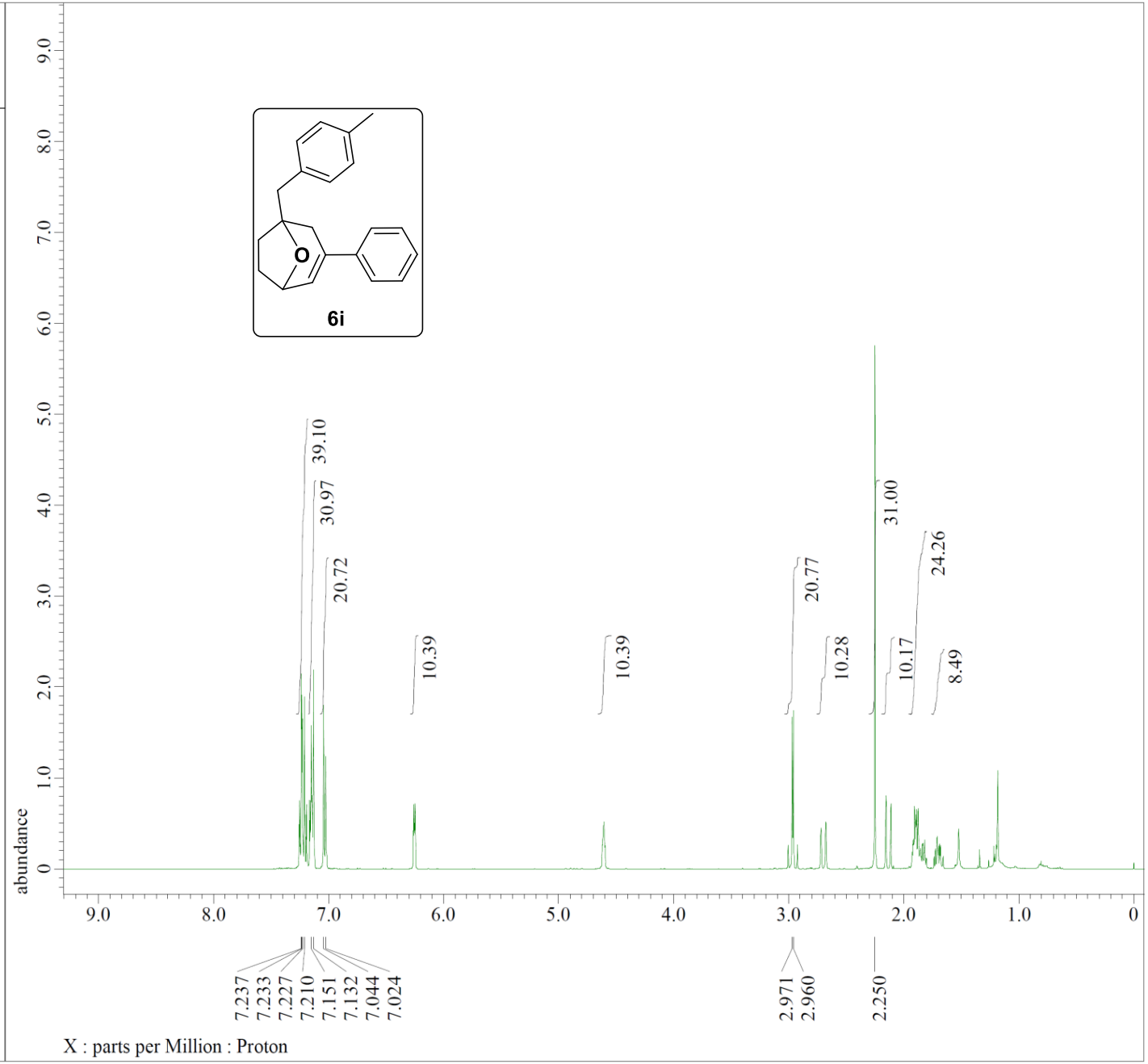
JEOL RESONANCE

```
Filename           = 104-96-96A_DEPT135deg-2-3
Author             = delta
Experiment         = dept.jxp
Sample_Id          = 104-96-96A
Solvent            = CHLOROFORM-D
Creation_Time      = 25-MAY-2015 18:00:25
Revision_Time      = 25-MAY-2015 18:24:35
Current_Time       = 25-MAY-2015 18:24:53

Comment            = DEPT with decoupling
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 512
Total_Scans        = 512

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 21.7[dC]
X_Acq_Time         = 1.04333312[s]
X_Atn              = 5.3[dB]
X_Pulse            = 9.56[us]
Irr_Atn            = 2.4[dB]
Irr_Atn_Dec        = 22.008[dB]
Irr_Noise          = WALTZ
Irr_Pulse          = 12.031[us]
Irr_Pwidth         = 0.115[ms]
Base_Line_Correct  = TRUE
Decoupling         = TRUE
Initial_Wait       = 1[s]
J_Constant         = 140[Hz]
Selection_Angle    = 135[deg]
Selection_Factor   = 1
Selection_Pulse    = 18.0465[us]
```



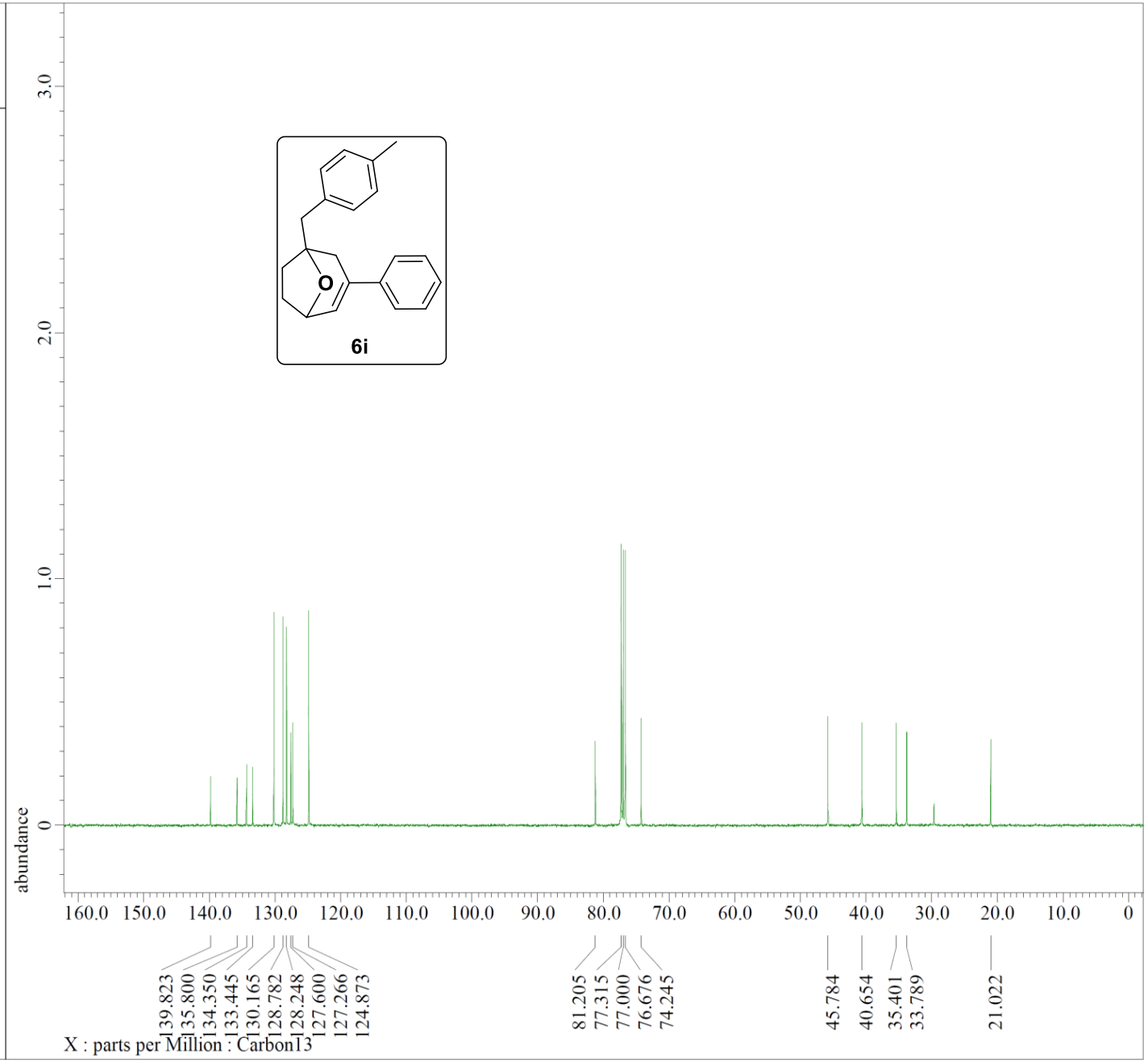
JEOL RESONANCE

```
Filename          = 104-108-108A_Proton-1-5.jd
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 104-108-108A
Solvent           = CHLOROFORM-D
Creation_Time     =  1-JUN-2015 16:15:54
Revision_Time     =  1-JUN-2015 16:15:40
Current_Time      =  1-JUN-2015 16:16:08

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 32
Temp_Get          = 22.5[dC]
X_90_Width        = 12.031[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2.4[dB]
X_Pulse           = 6.0155[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



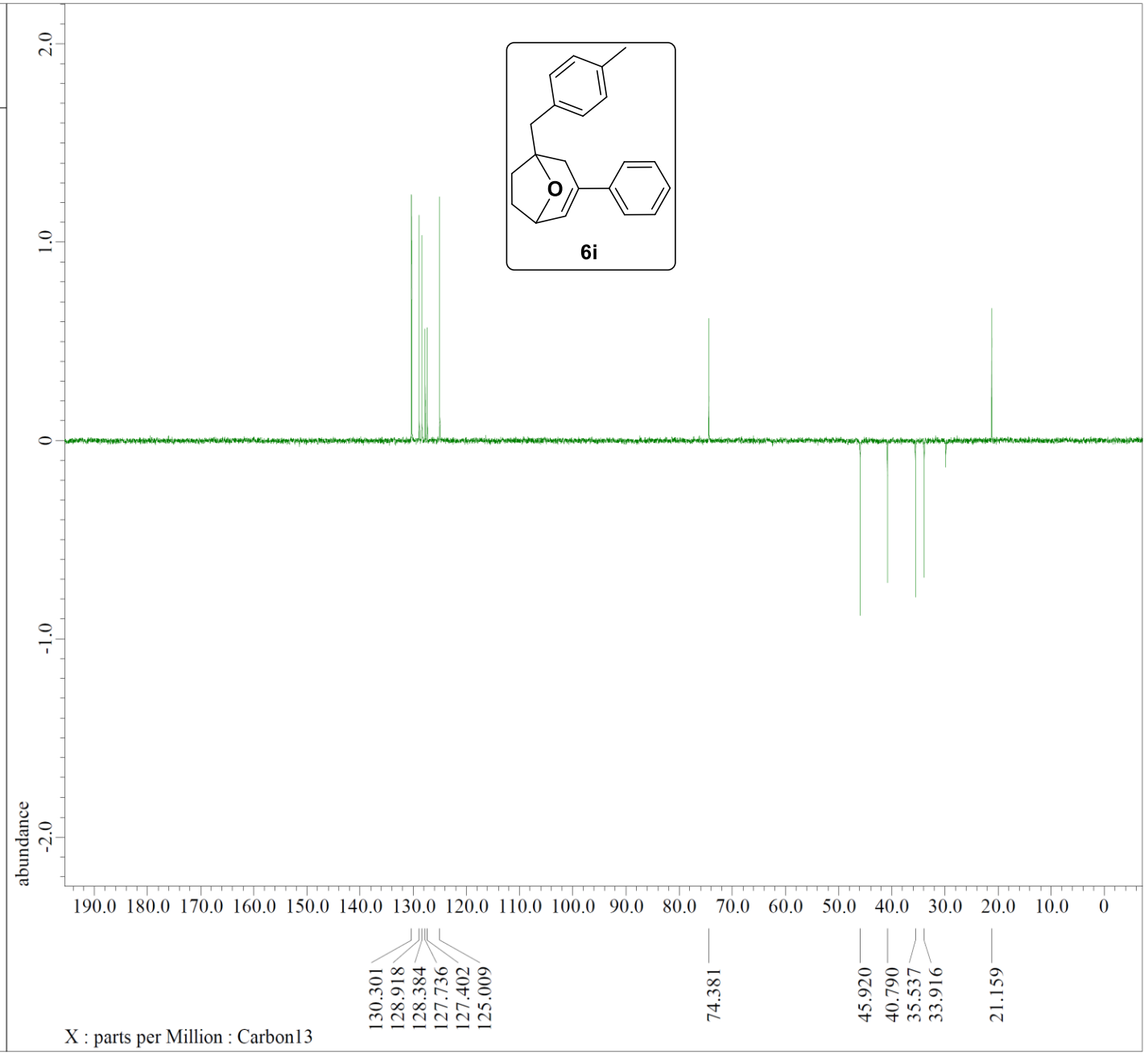
JEOL RESONANCE

```
Filename          = 104-108-108A_Carbon-1-4.jd
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 104-108-108A
Solvent           = CHLOROFORM-D
Creation_Time     =  1-JUN-2015 16:18:58
Revision_Time     =  1-JUN-2015 18:58:24
Current_Time      =  1-JUN-2015 18:59:21

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.8[dC]
X_90_Width        = 9.56[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 5.3[dB]
X_Pulse           = 3.18666667[us]
Irr_Atn_Dec       = 22.008[dB]
Irr_Atn_Noe       = 22.008[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



JEOL RESONANCE

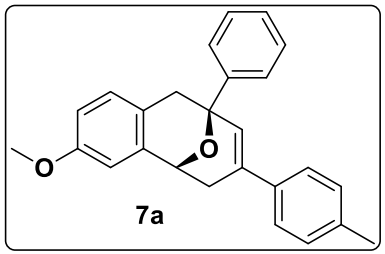
```
Filename           = 104-108-108A_DEPT135deg-1
Author             = delta
Experiment         = dept.jxp
Sample_Id          = 104-108-108A
Solvent            = CHLOROFORM-D
Creation_Time      =  1-JUN-2015 17:13:10
Revision_Time      =  1-JUN-2015 18:59:54
Current_Time       =  1-JUN-2015 19:00:03

Comment            = DEPT with decoupling
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 126
Total_Scans        = 126

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.6[dC]
X_Acq_Time         = 1.04333312[s]
X_Atn              = 5.3[dB]
X_Pulse            = 9.56[us]
Irr_Atn            = 2.4[dB]
Irr_Atn_Dec        = 22.008[dB]
Irr_Noise          = WALTZ
Irr_Pulse          = 12.031[us]
Irr_Pwidth         = 0.115[ms]
Base_Line_Correct  = TRUE
Decoupling         = TRUE
Initial_Wait       = 1[s]
J_Constant         = 140[Hz]
Selection_Angle    = 135[deg]
Selection_Factor   = 1
Selection_Pulse    = 18.0465[us]
```

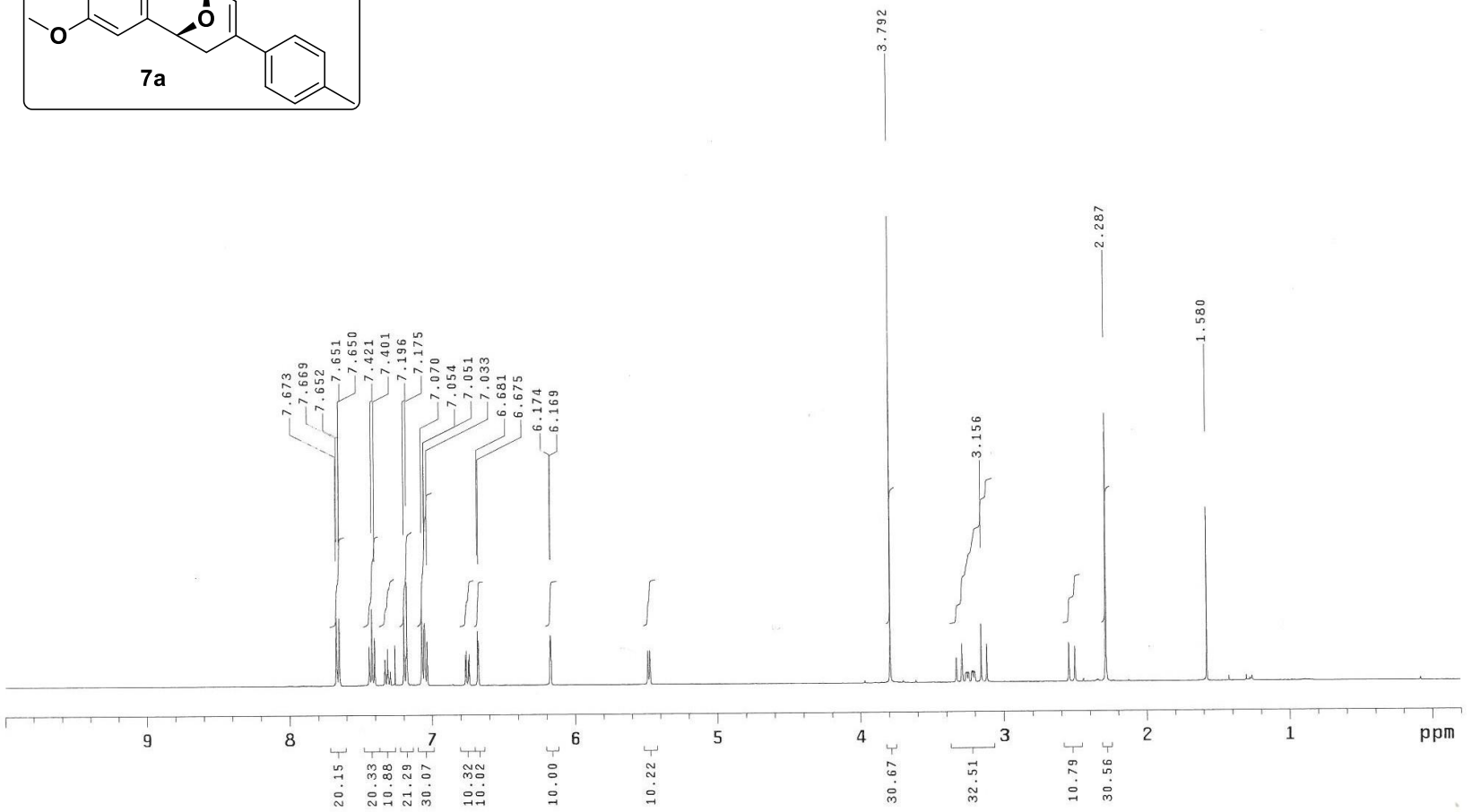


Jaya-20141225-2

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Dec 25 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

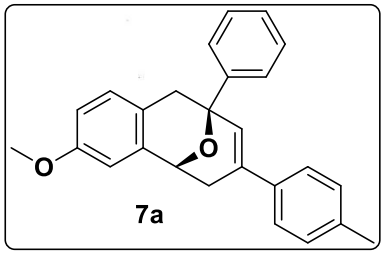




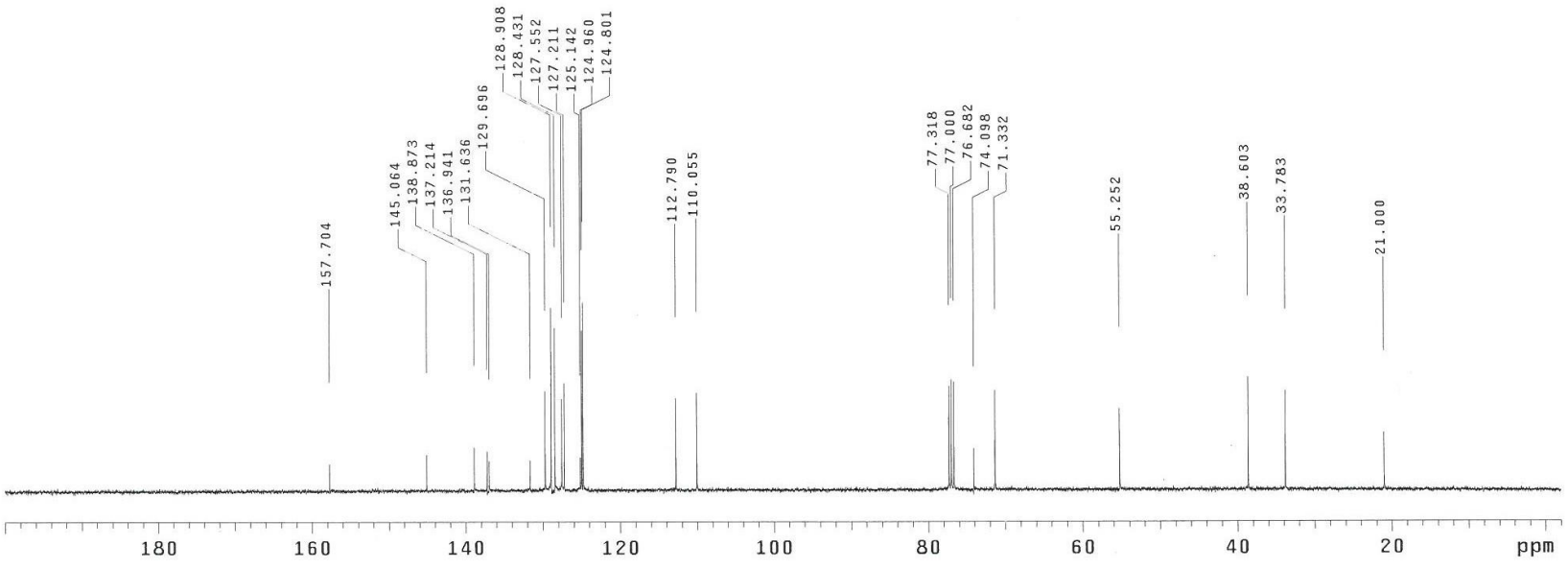
Jaya-20141225-2

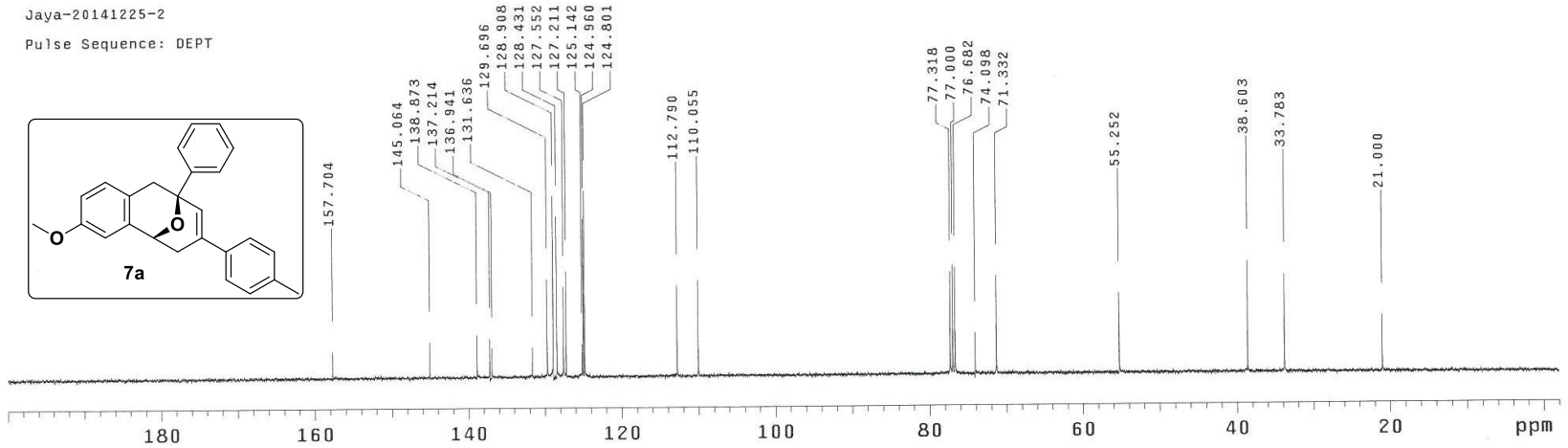
Pulse Sequence: s2pul

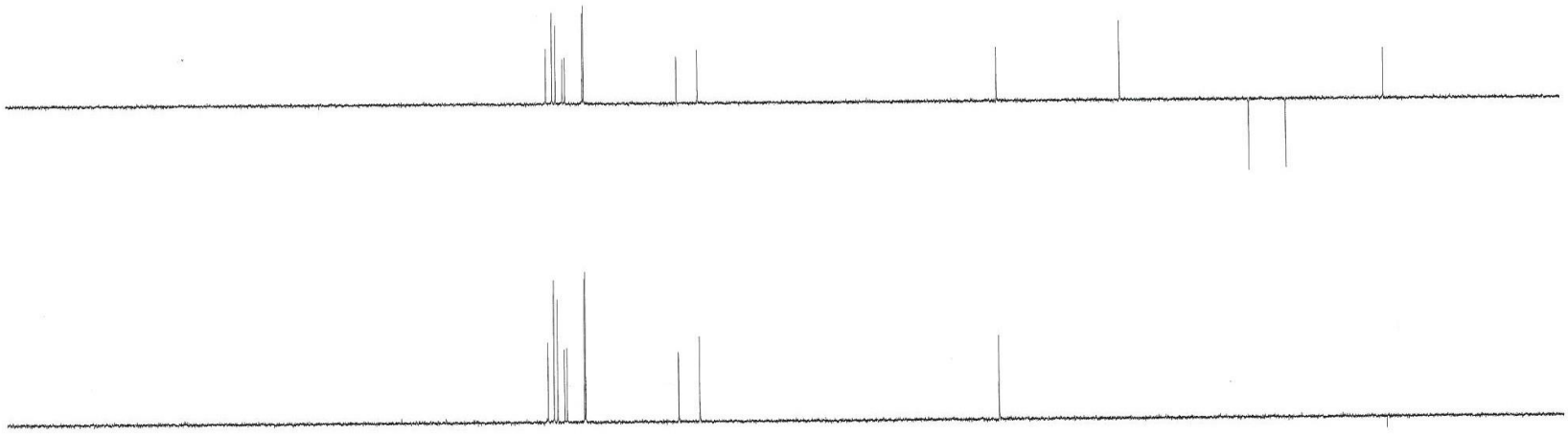
Mercury-400BB "MercuryPlus400"
Date: Dec 25 2014
Solvent: CDCl3
Ambient temperature
Total 1232 repetitions



7a



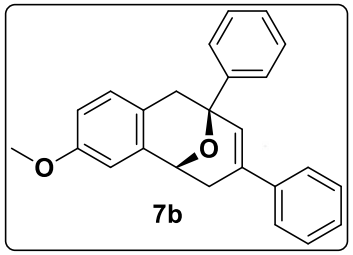

Jaya-20141225-2
Pulse Sequence: DEPT
7a
157.704
145.064
138.873
137.214
136.941
131.636
129.696
128.908
128.431
127.552
127.211
125.142
124.960
124.801
112.790
110.055
77.318
77.000
76.682
74.098
71.332
55.252
38.603
33.783
21.000
180
160
140
120
100
80
60
40
20
ppm



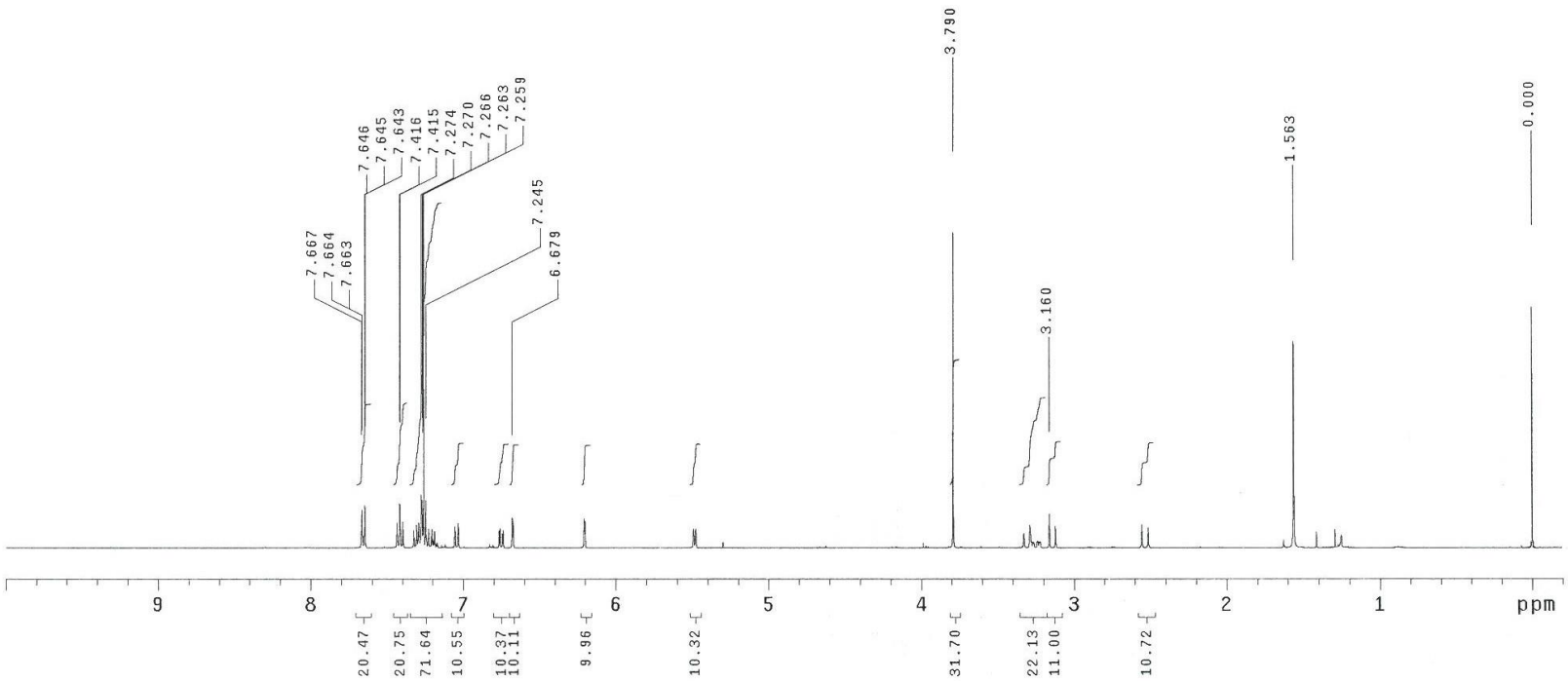
Milu-2014-P020

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Apr 7 2014
Solvent: CDCl3
Ambient temperature
Total 80 repetitions



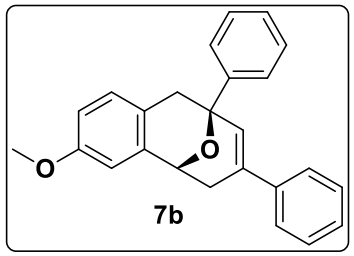
7b



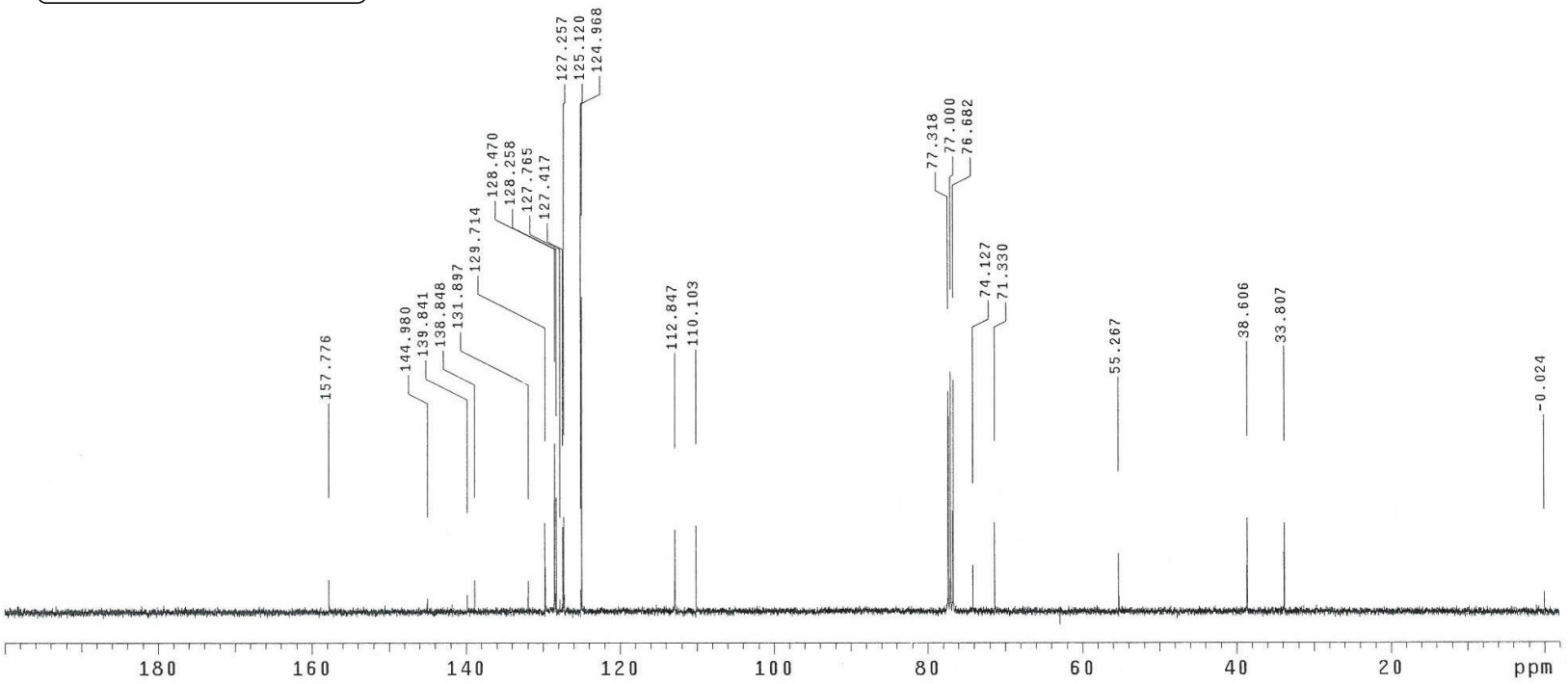
Milu-2014-P020

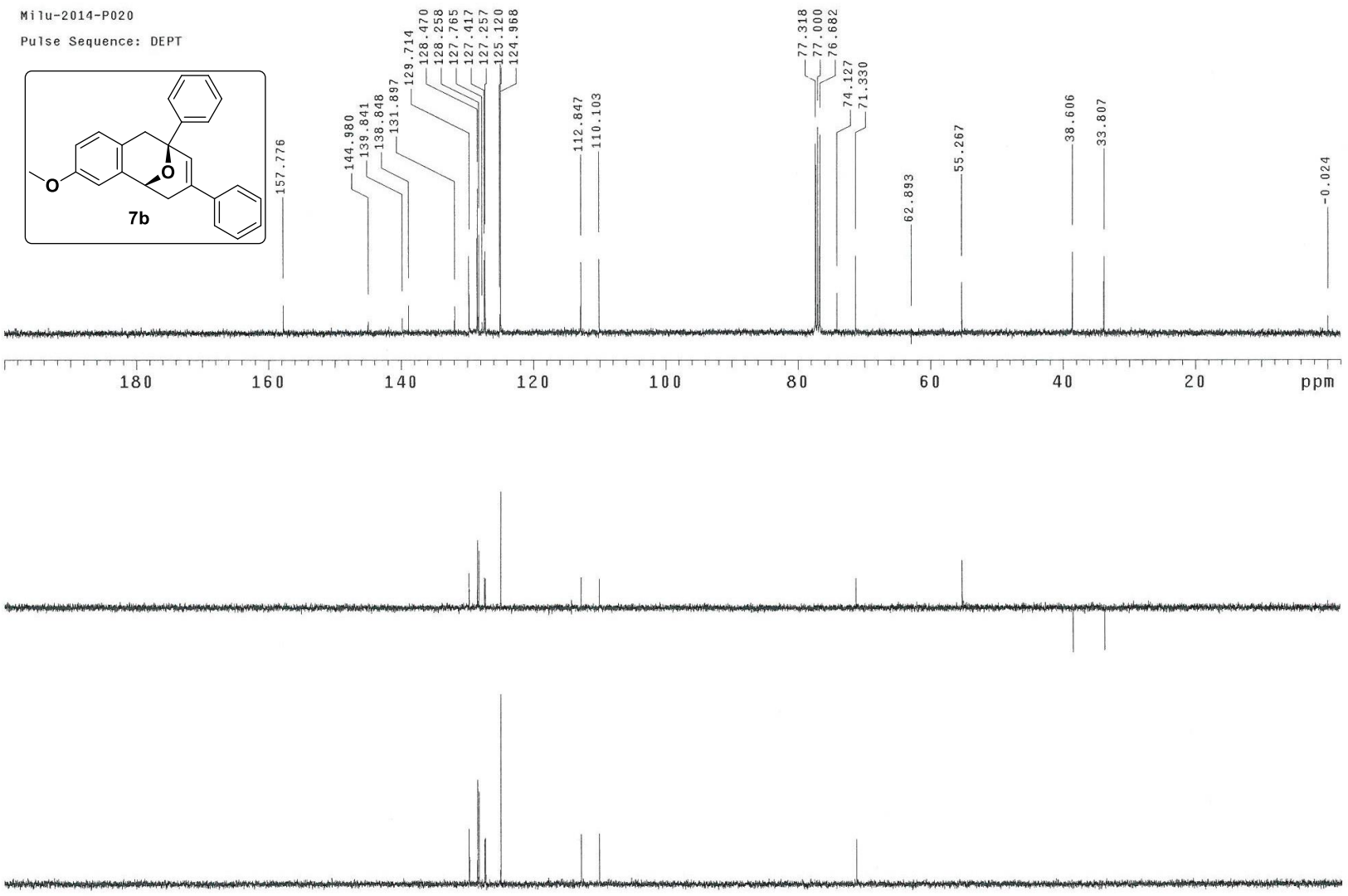
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Apr 7 2014
Solvent: CDCl3
Ambient temperature
Total 13280 repetitions



7b

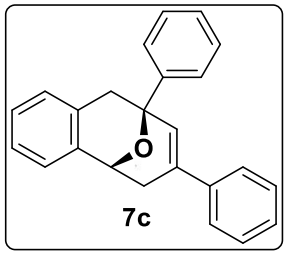



Milu-2014-P020
Pulse Sequence: DEPT
7b
157.776
144.980
139.841
138.848
131.897
129.714
128.470
128.258
127.765
127.417
127.257
125.120
124.968
112.847
110.103
77.318
77.000
76.682
74.127
71.330
62.893
55.267
38.606
33.807
-0.024
180
160
140
120
100
80
60
40
20
ppm

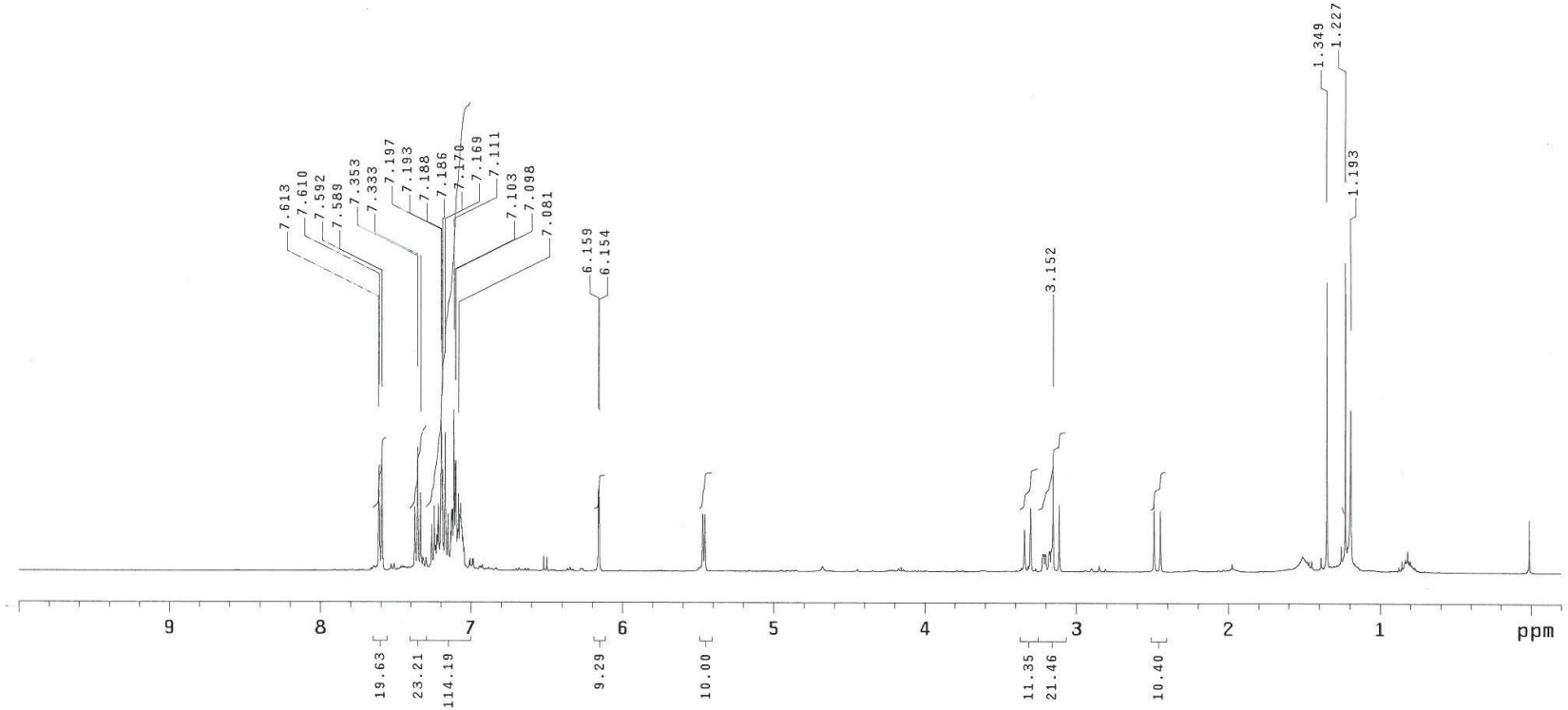
phph0005

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 15 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



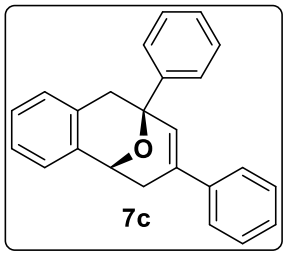
7c



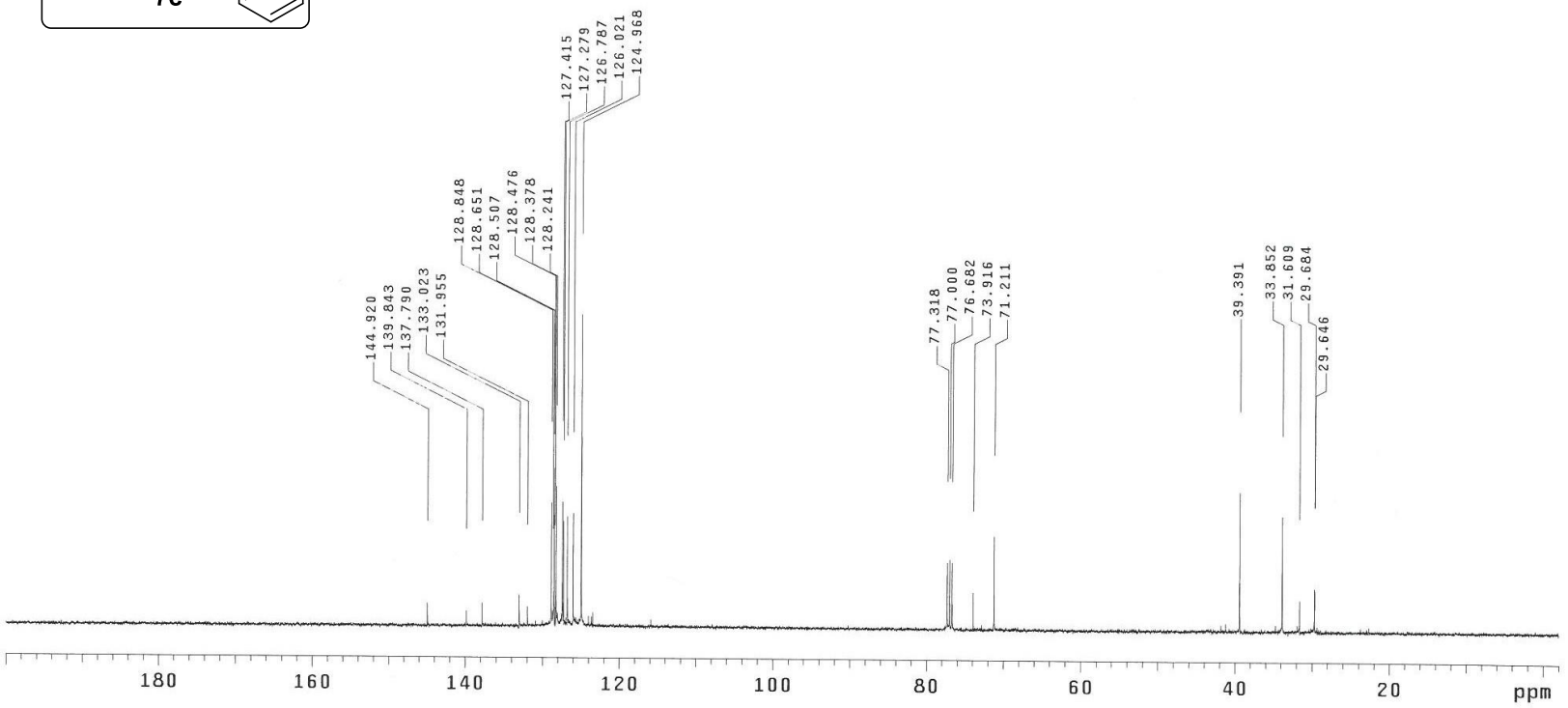
phph0005

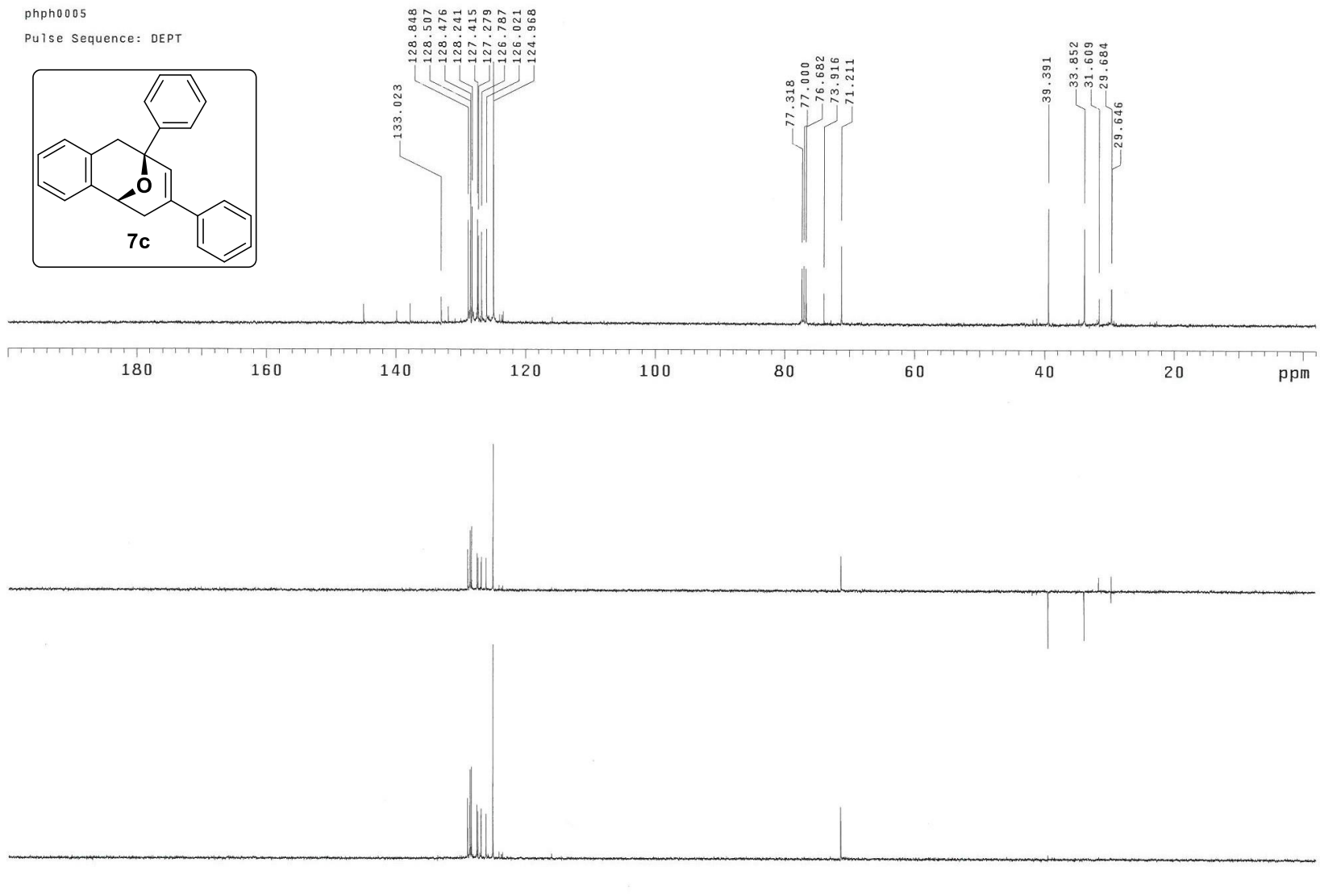
Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 15 2014
Solvent: CDCl3
Ambient temperature
Total 1392 repetitions



7c

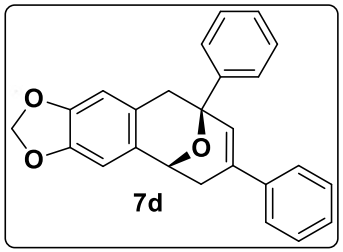



phph0005
Pulse Sequence: DEPT
7c
133.023
128.848
128.507
128.476
128.241
127.415
127.279
126.787
126.021
124.968
77.318
77.000
76.682
73.916
71.211
39.391
33.852
31.609
29.684
29.646
180
160
140
120
100
80
60
40
20
ppm

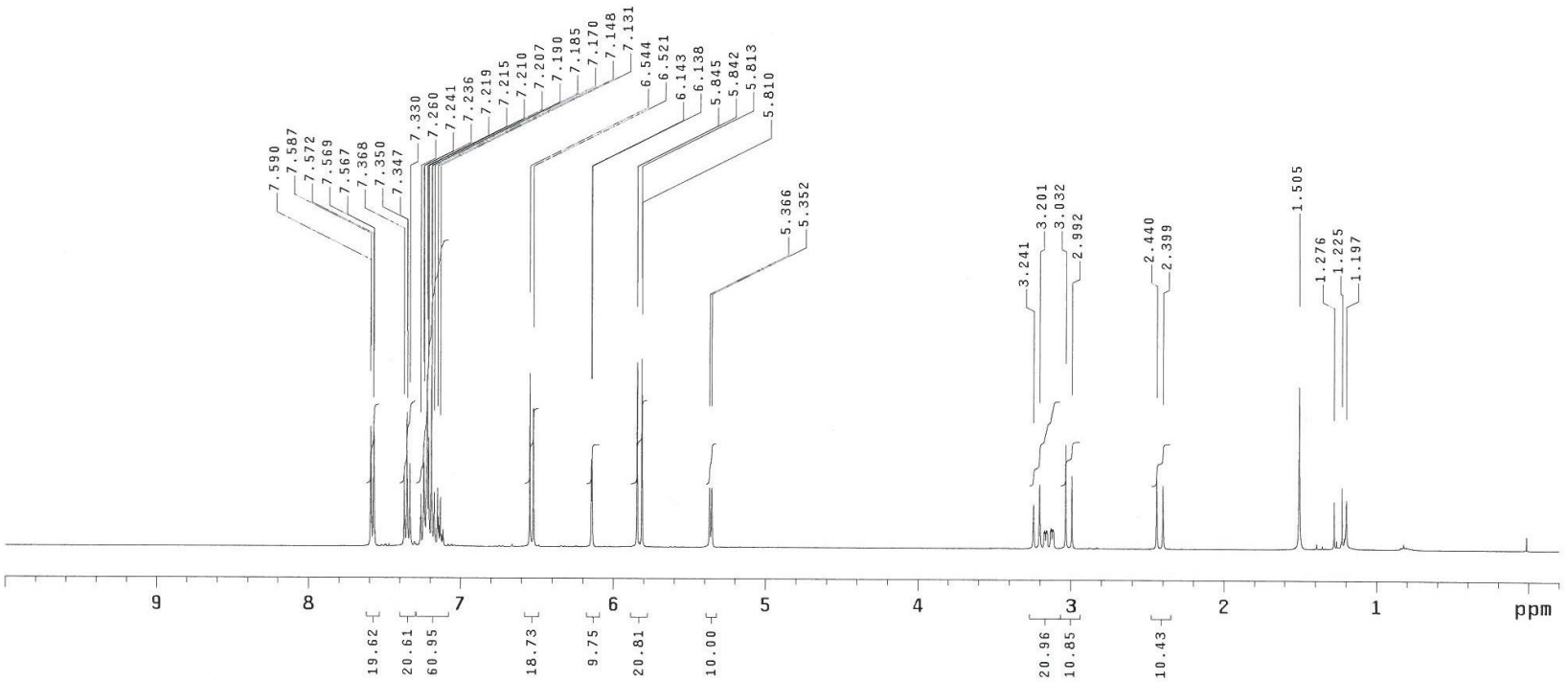
phph0003

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 15 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



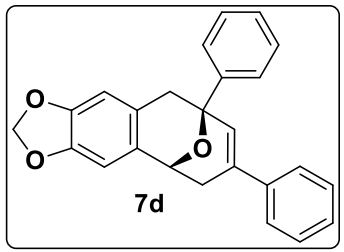
7d



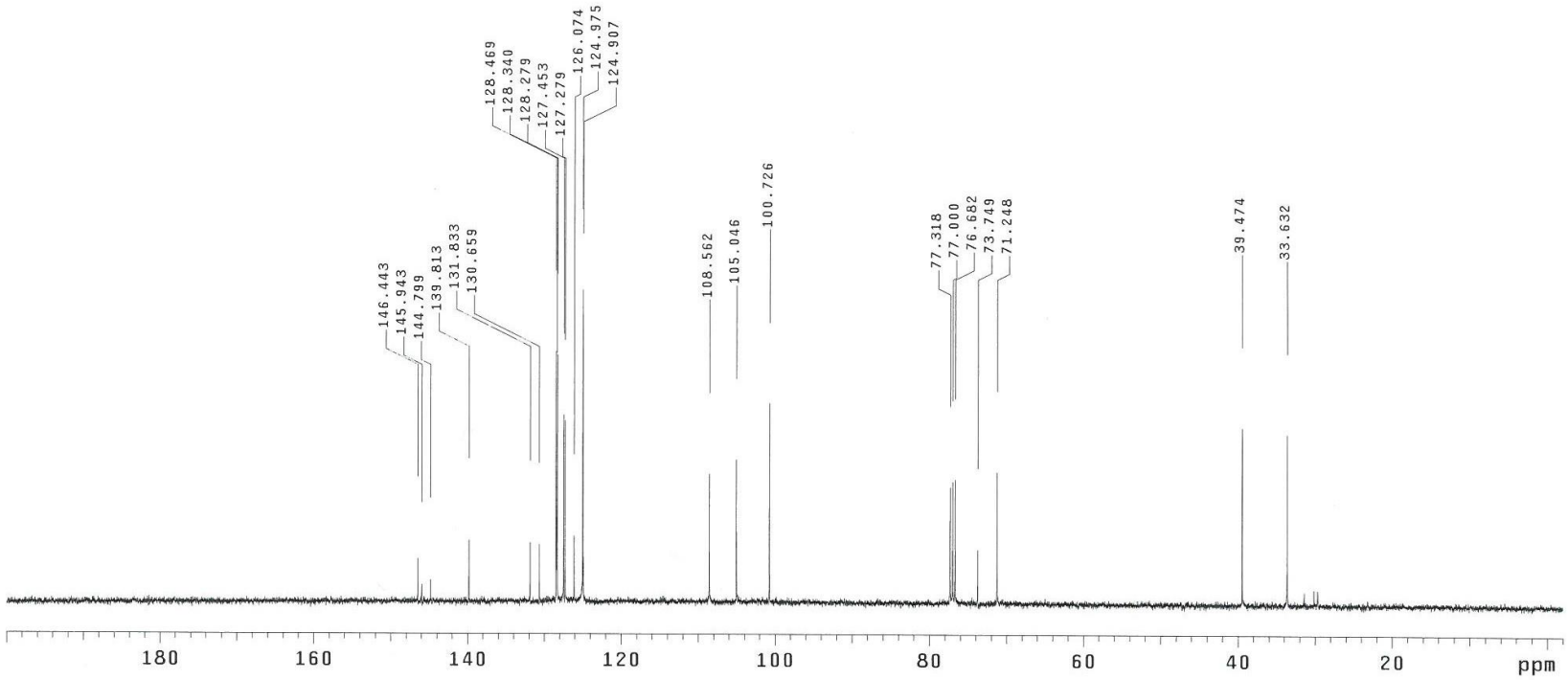
phph0003

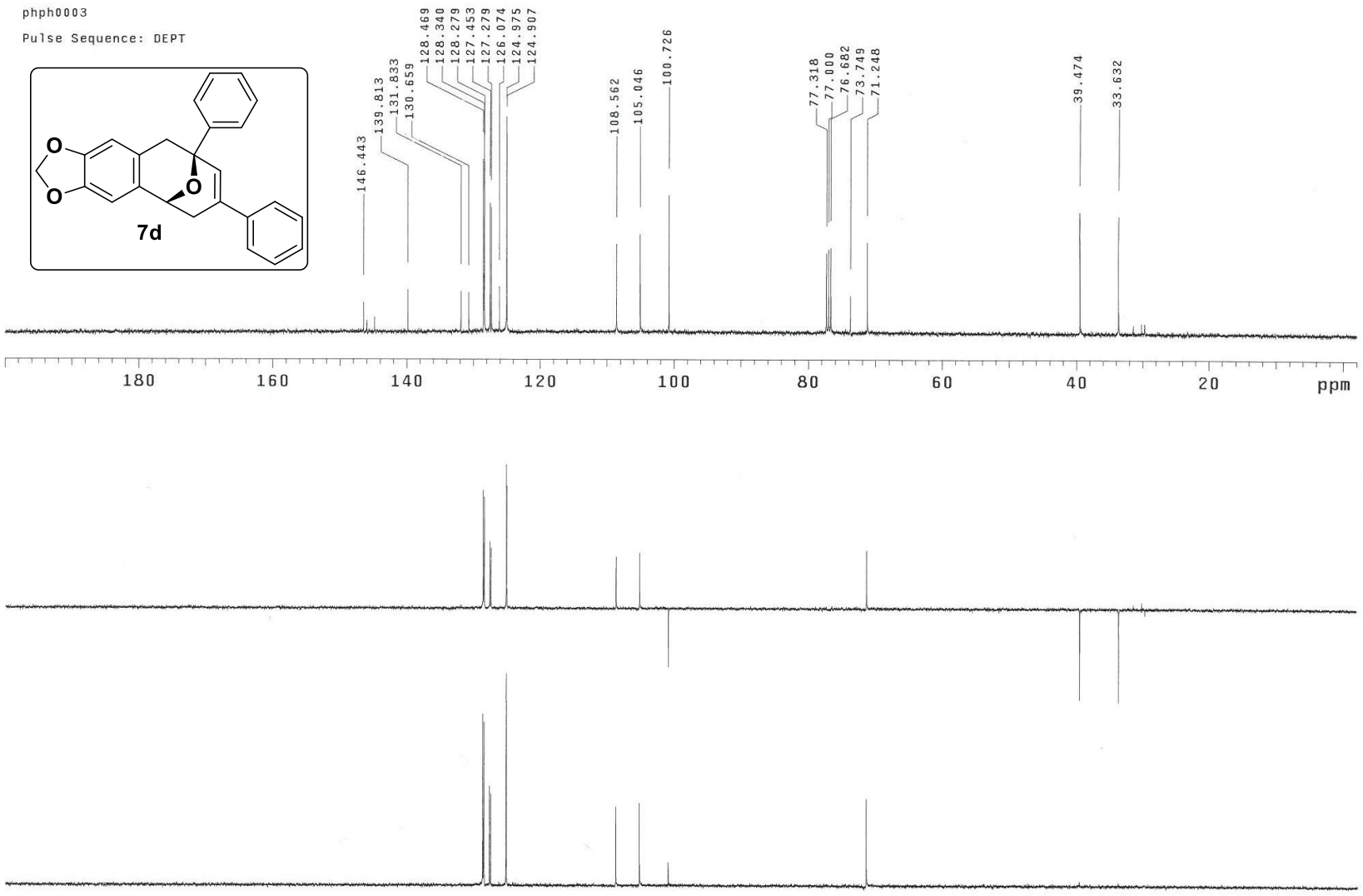
Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 15 2014
Solvent: CDCl3
Ambient temperature
Total 1120 repetitions



7d

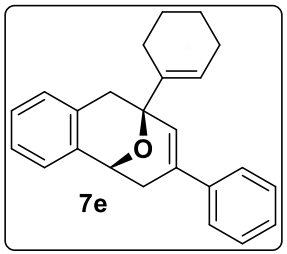



phph0003
Pulse Sequence: DEPT
7d
146.443
139.813
131.833
130.659
128.469
128.340
128.279
127.453
127.279
126.074
124.975
124.907
108.562
105.046
100.726
77.318
77.000
76.682
73.749
71.248
39.474
33.632
180
160
140
120
100
80
60
40
20
ppm

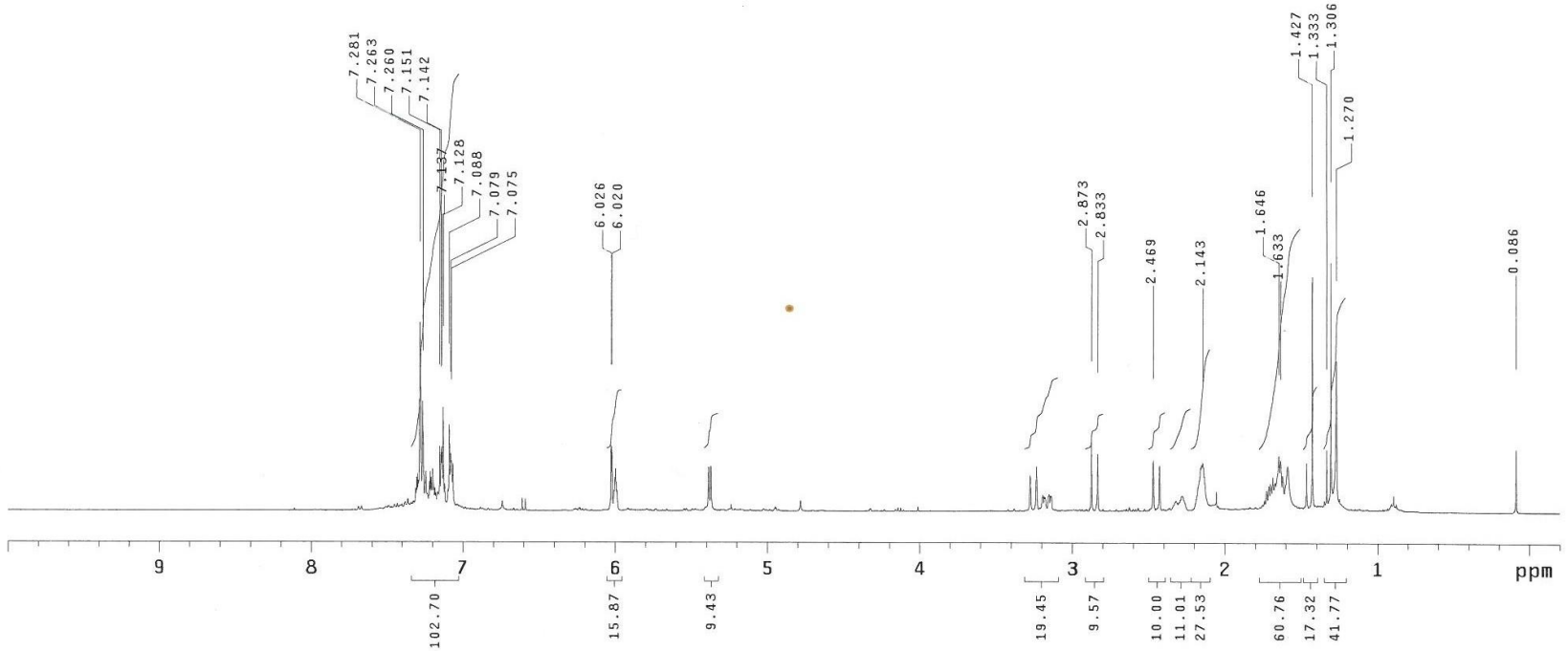
phph0001

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 15 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



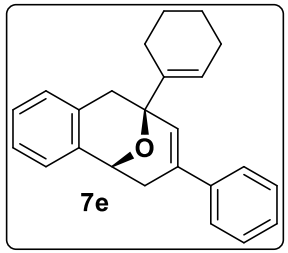
7e

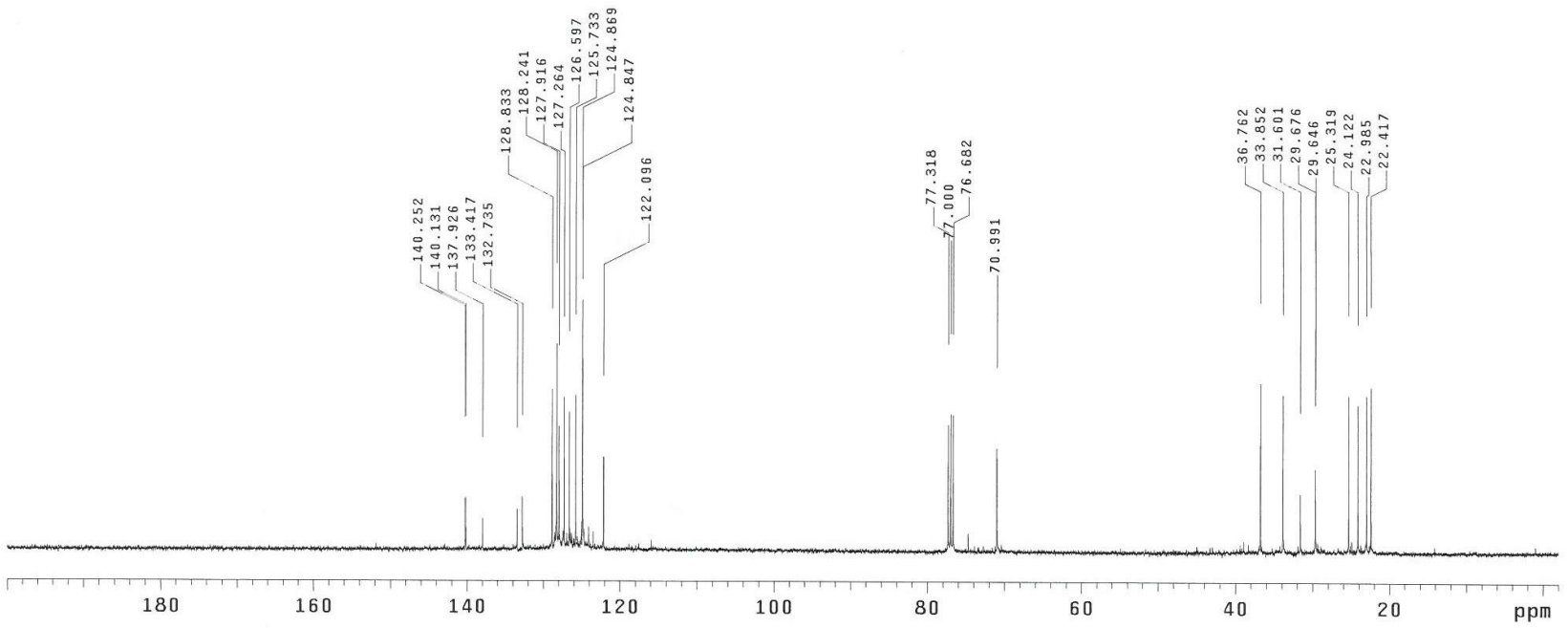


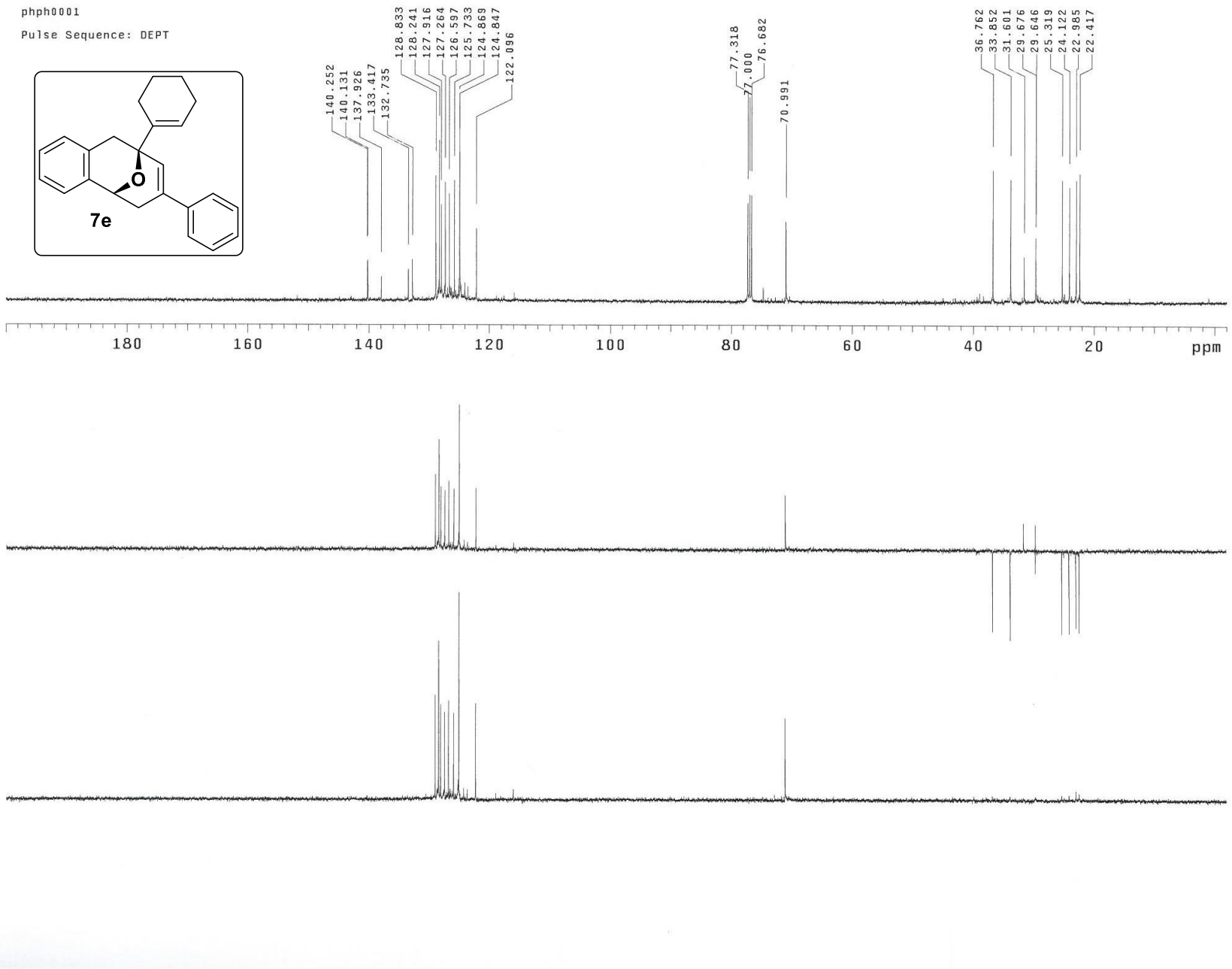
phph0001

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 15 2014
Solvent: CDCl3
Ambient temperature
Total 3152 repetitions



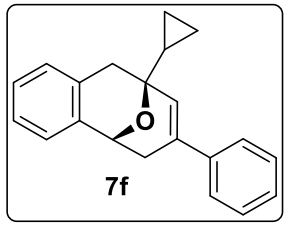



phph0001
Pulse Sequence: DEPT
7e
O
140.252
140.131
137.926
133.417
132.735
128.833
128.241
127.916
127.264
126.597
125.733
124.869
124.847
122.096
77.318
77.000
76.682
70.991
36.762
33.852
31.601
29.676
29.646
25.319
24.122
22.985
22.417
180
160
140
120
100
80
60
40
20
ppm

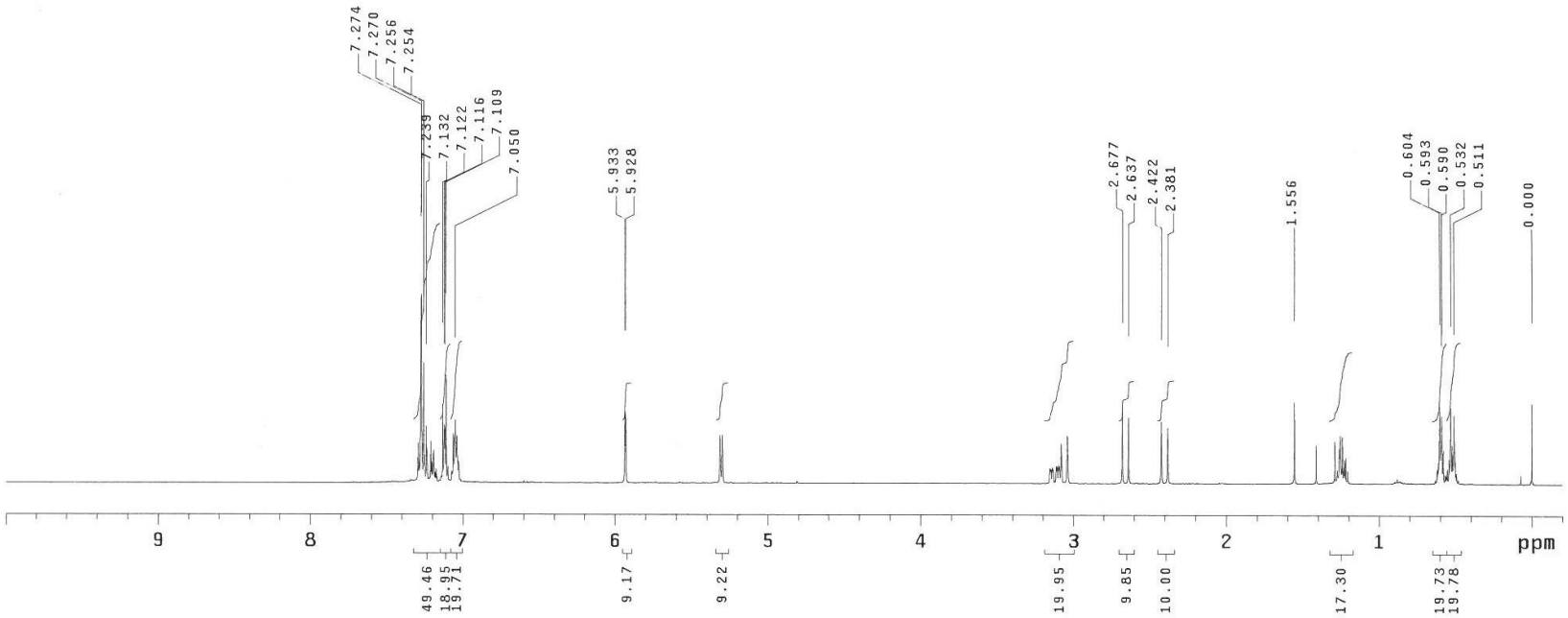
Milu-2014-P088B

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Jun 26 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



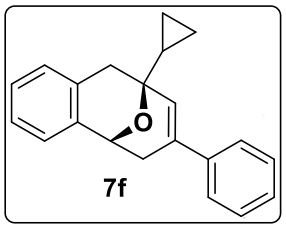
7f



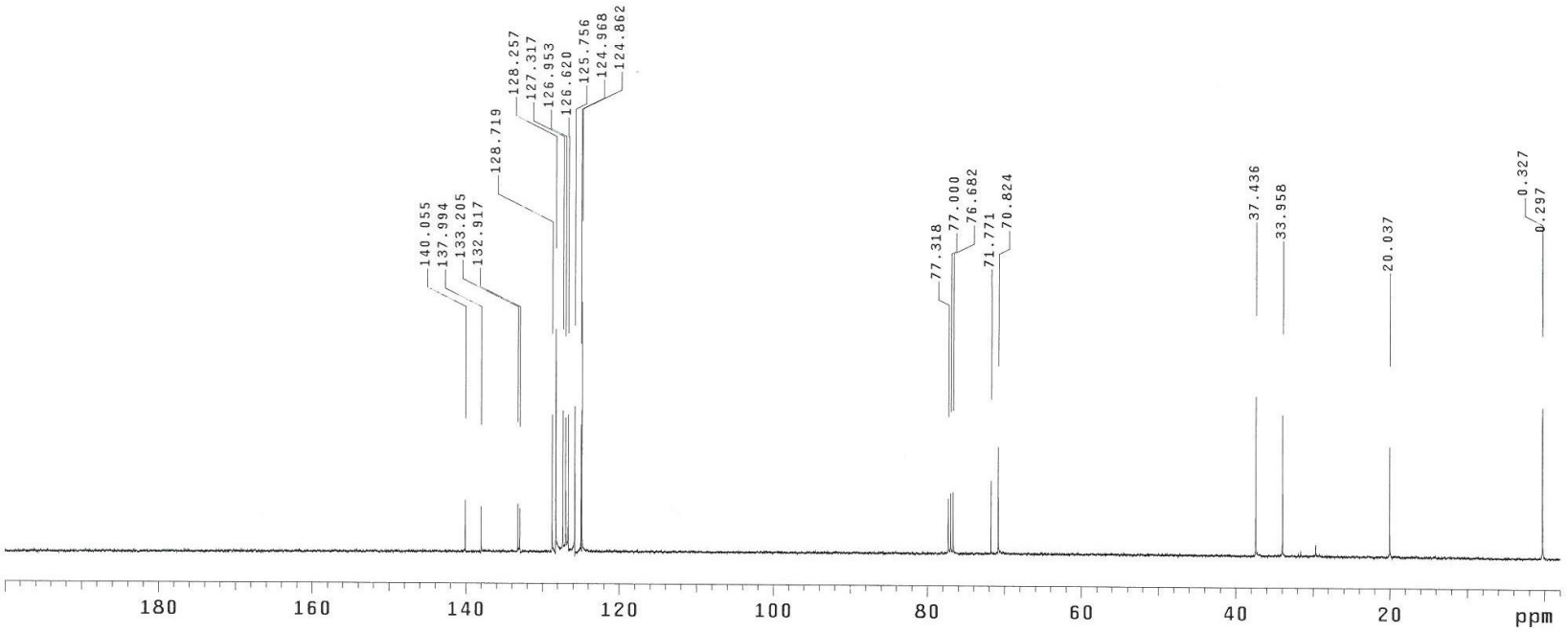
Milu-2014-P088B

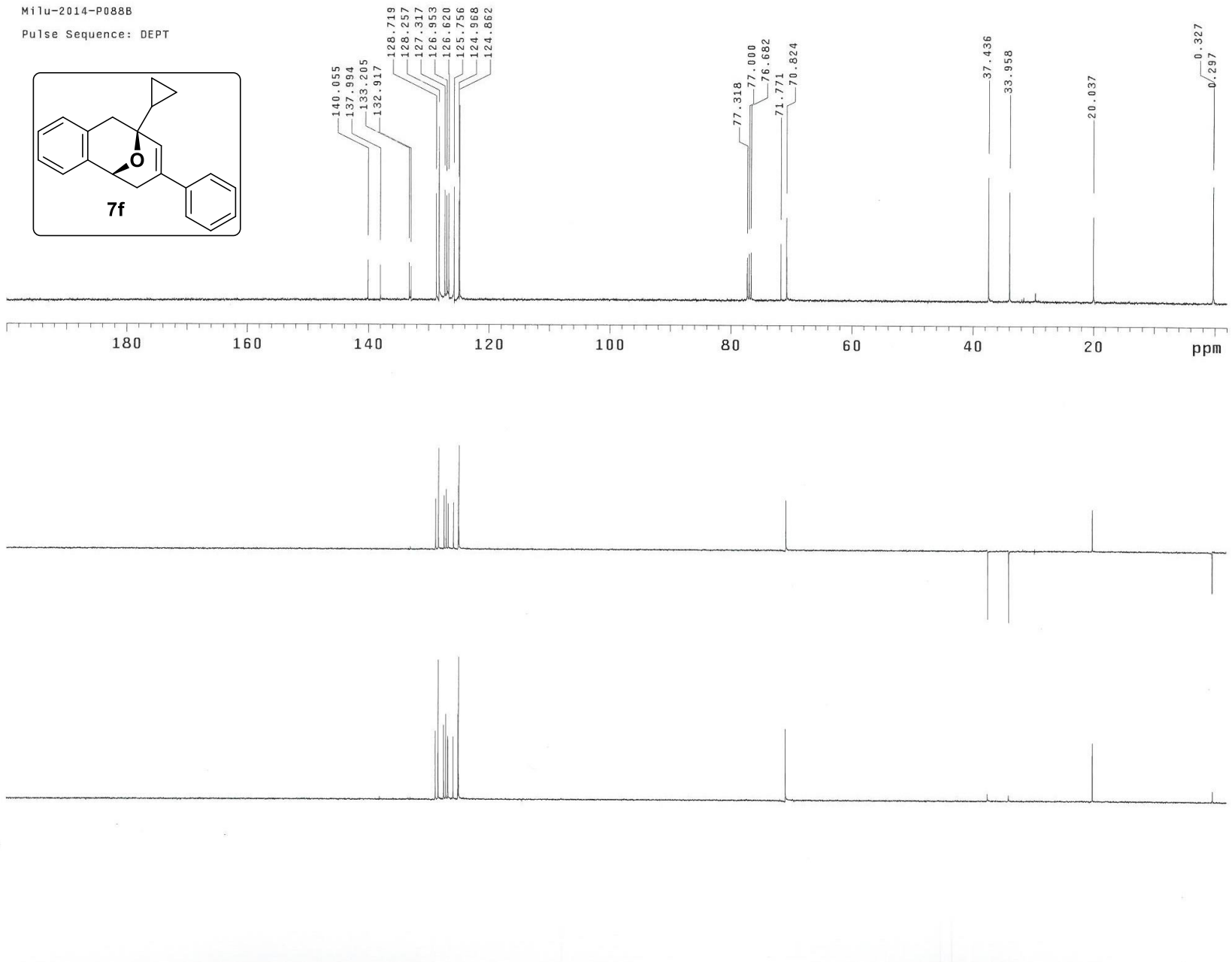
Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Jun 26 2014
Solvent: CDCl3
Ambient temperature
Total 64000 repetitions



7f



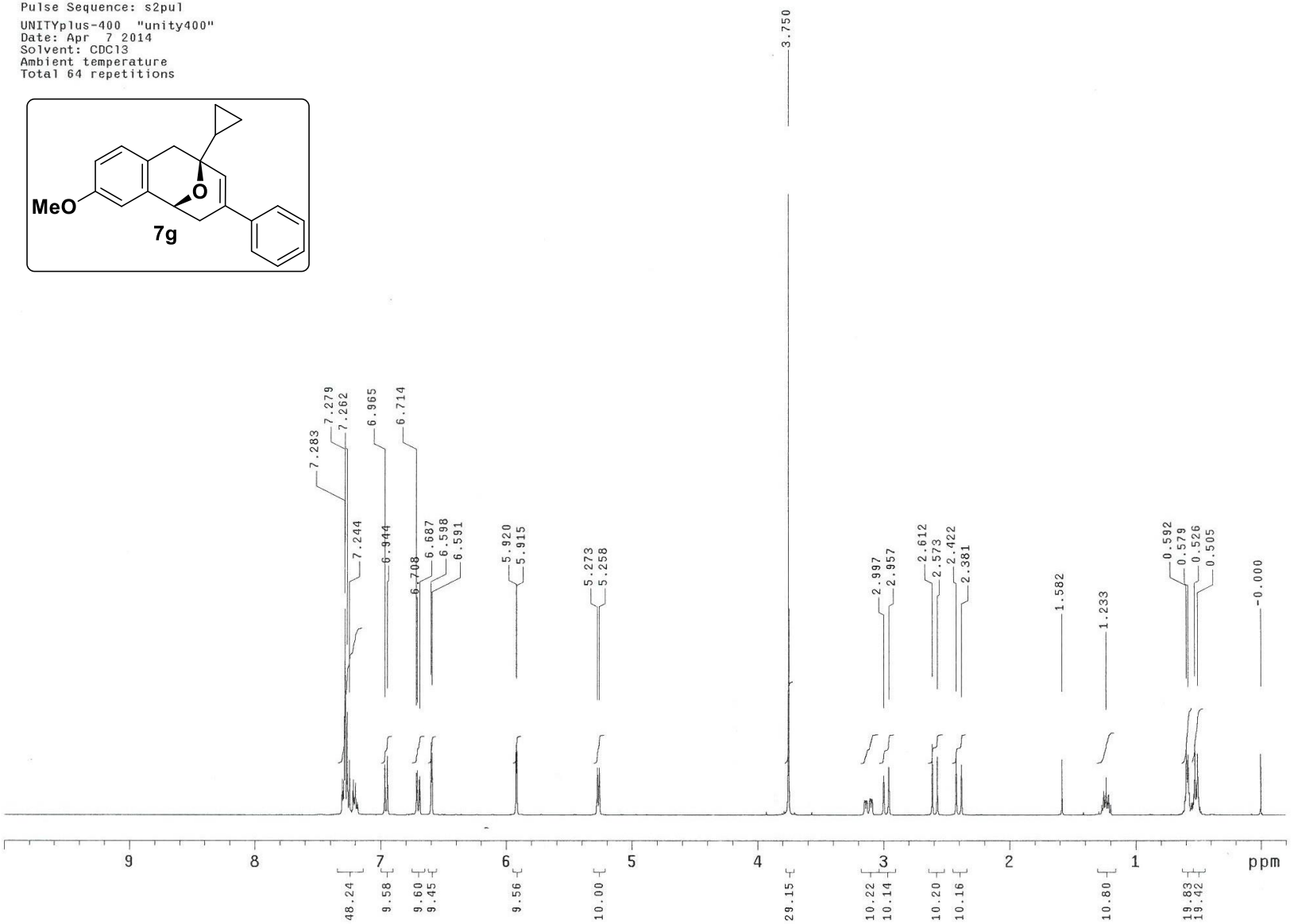

Milu-2014-P088B
Pulse Sequence: DEPT
7f
O
140.055
137.994
133.205
132.917
128.719
128.257
127.317
126.953
126.620
125.756
124.968
124.862
77.318
77.000
76.682
71.771
70.824
37.436
33.958
20.037
0.327
0.297
180
160
140
120
100
80
60
40
20
ppm

Milu-2014-P015

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Apr 7 2014
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

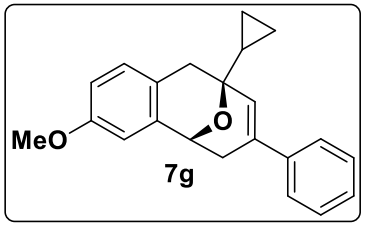
7g



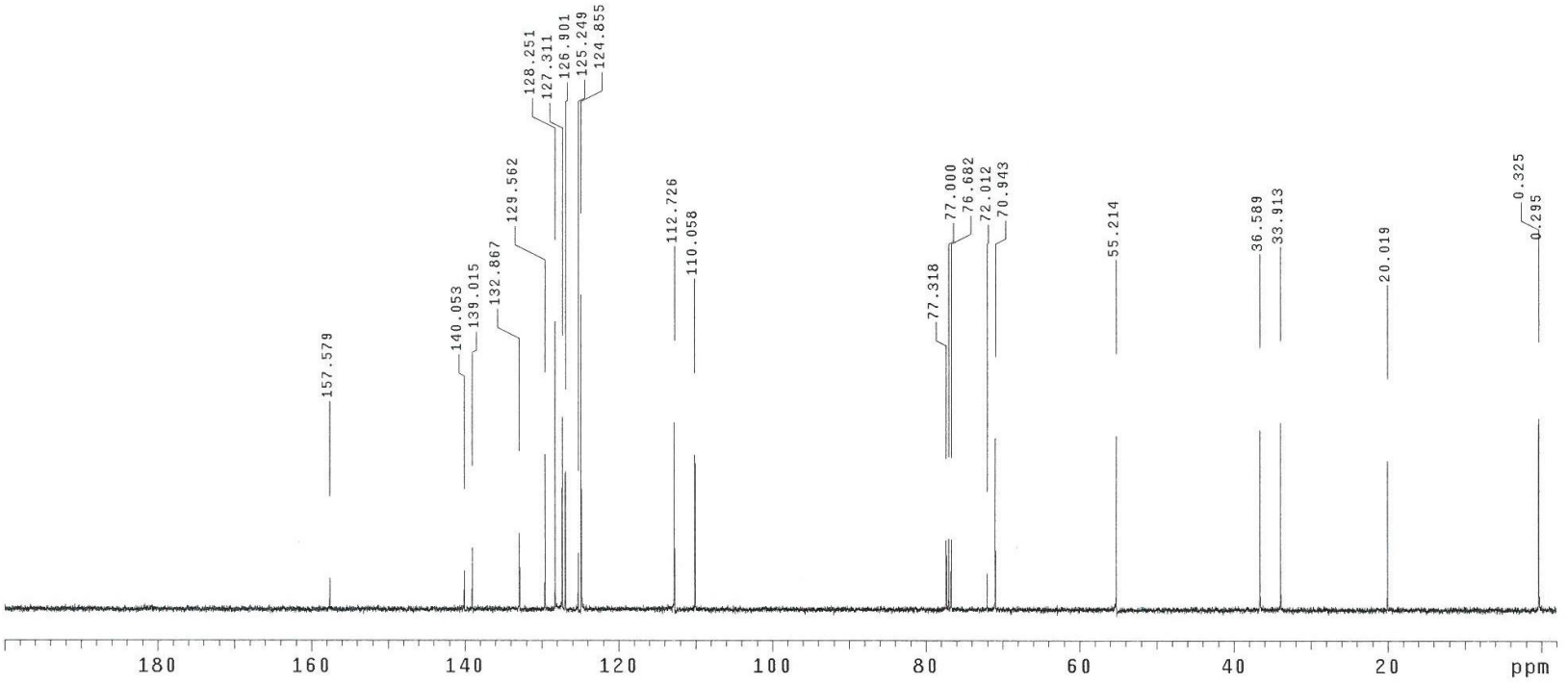
Milu-2014-P015

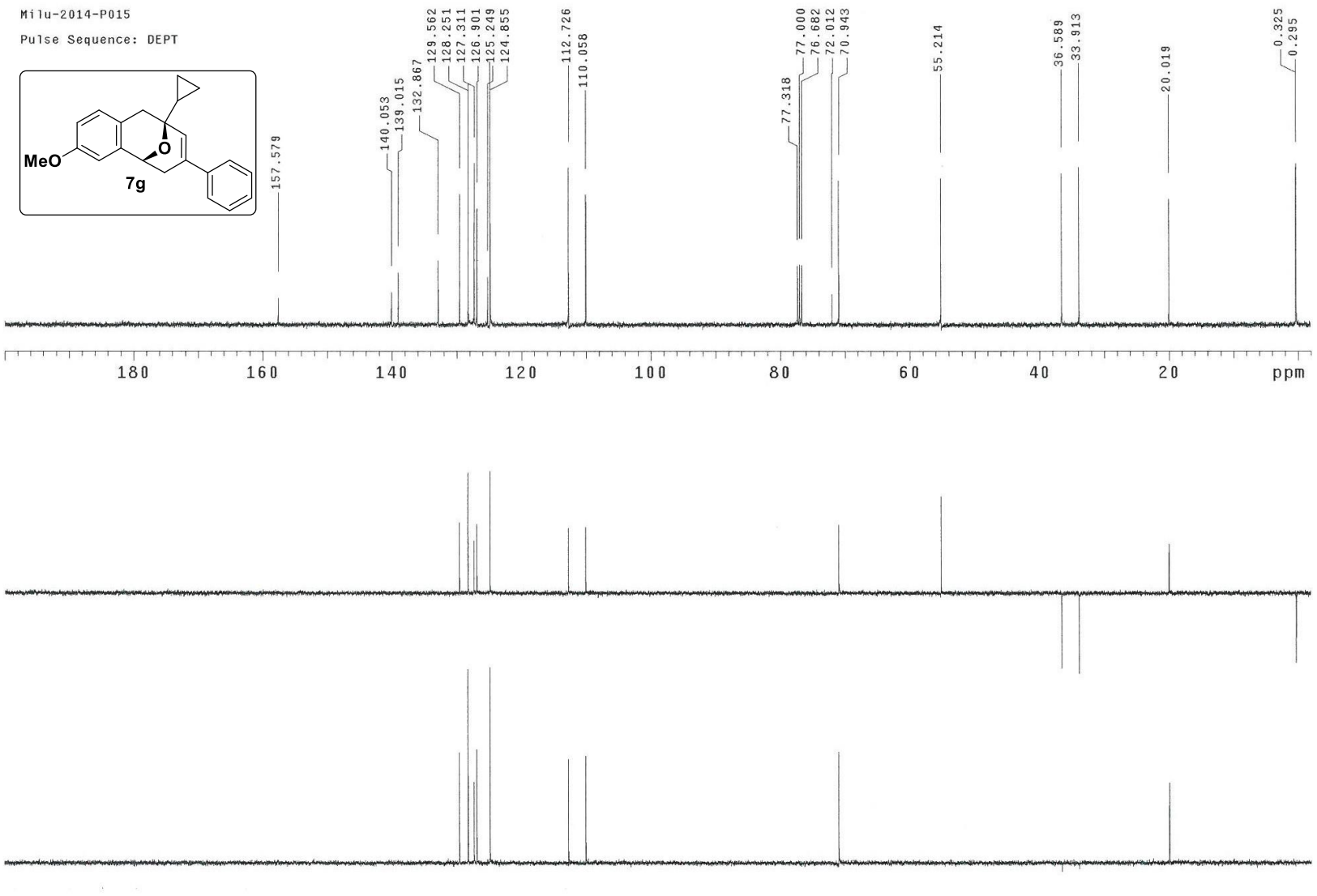
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Apr 7 2014
Solvent: CDCl3
Ambient temperature
Total 64000 repetitions



7g

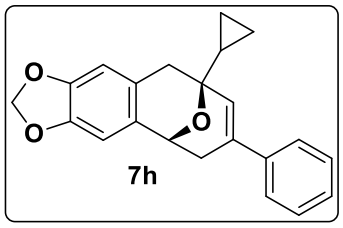


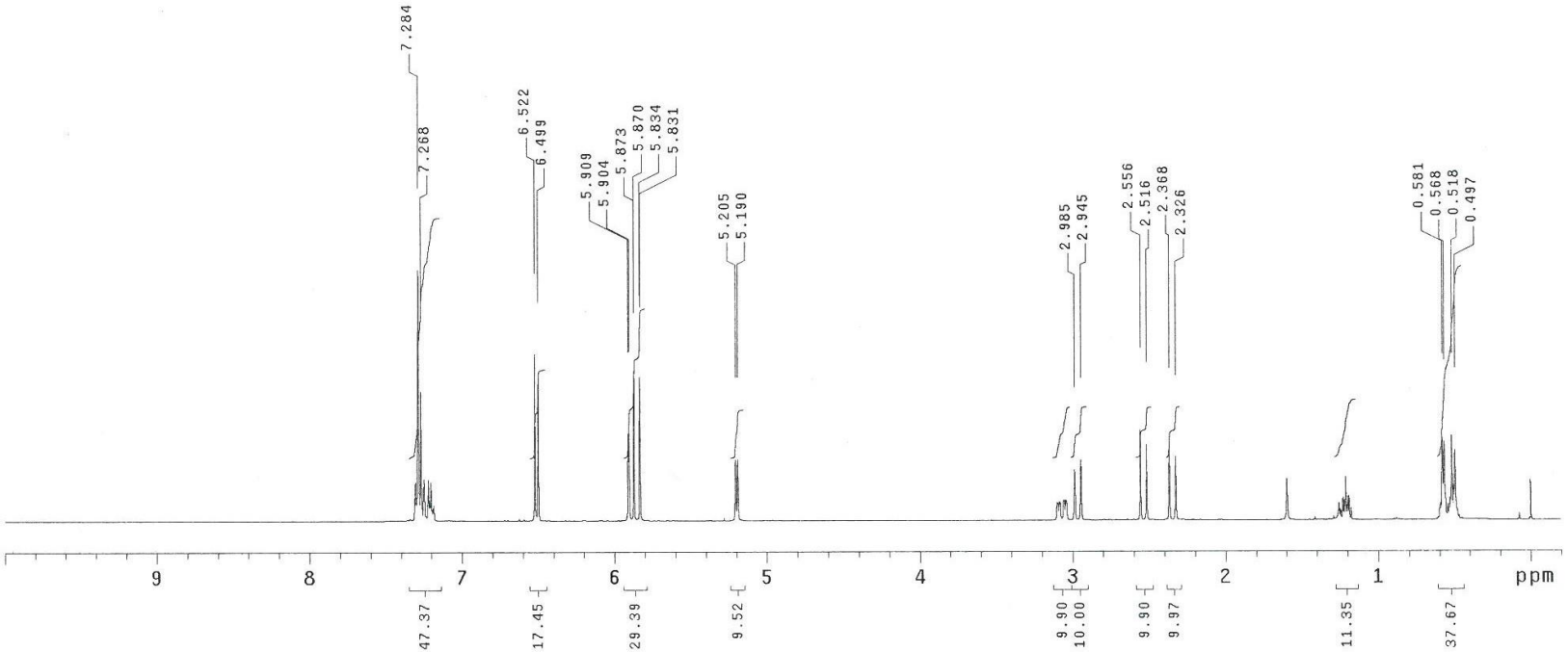

Milu-2014-P015
Pulse Sequence: DEPT
MeO
O
7g
157.579
140.053
139.015
132.867
129.562
128.251
127.311
126.901
125.249
124.855
112.726
110.058
77.318
77.000
76.682
72.012
70.943
55.214
36.589
33.913
20.019
0.325
0.295
180
160
140
120
100
80
60
40
20
ppm

Milu-2014-P031

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Apr 21 2014
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

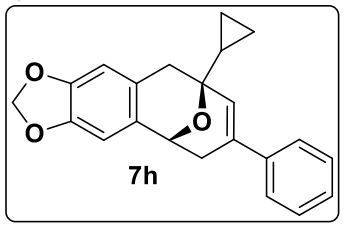


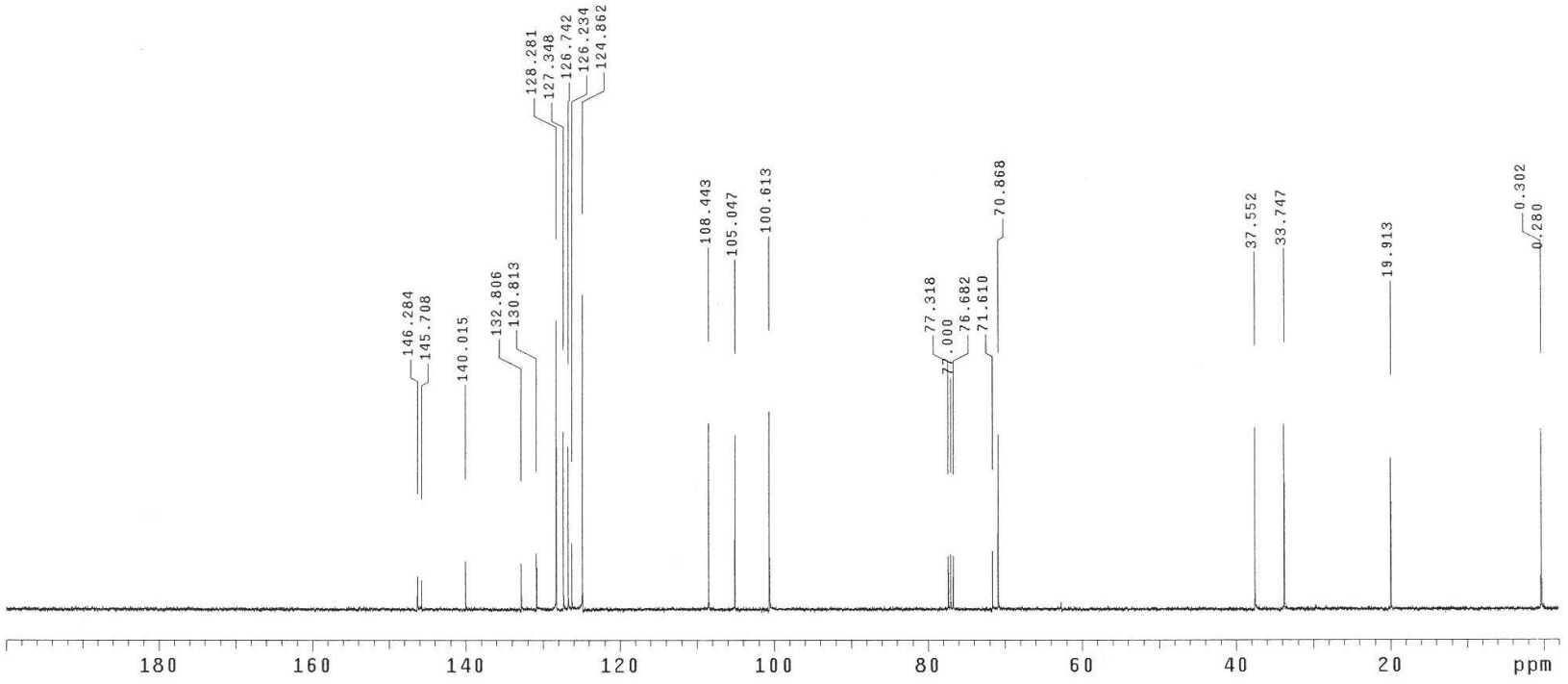


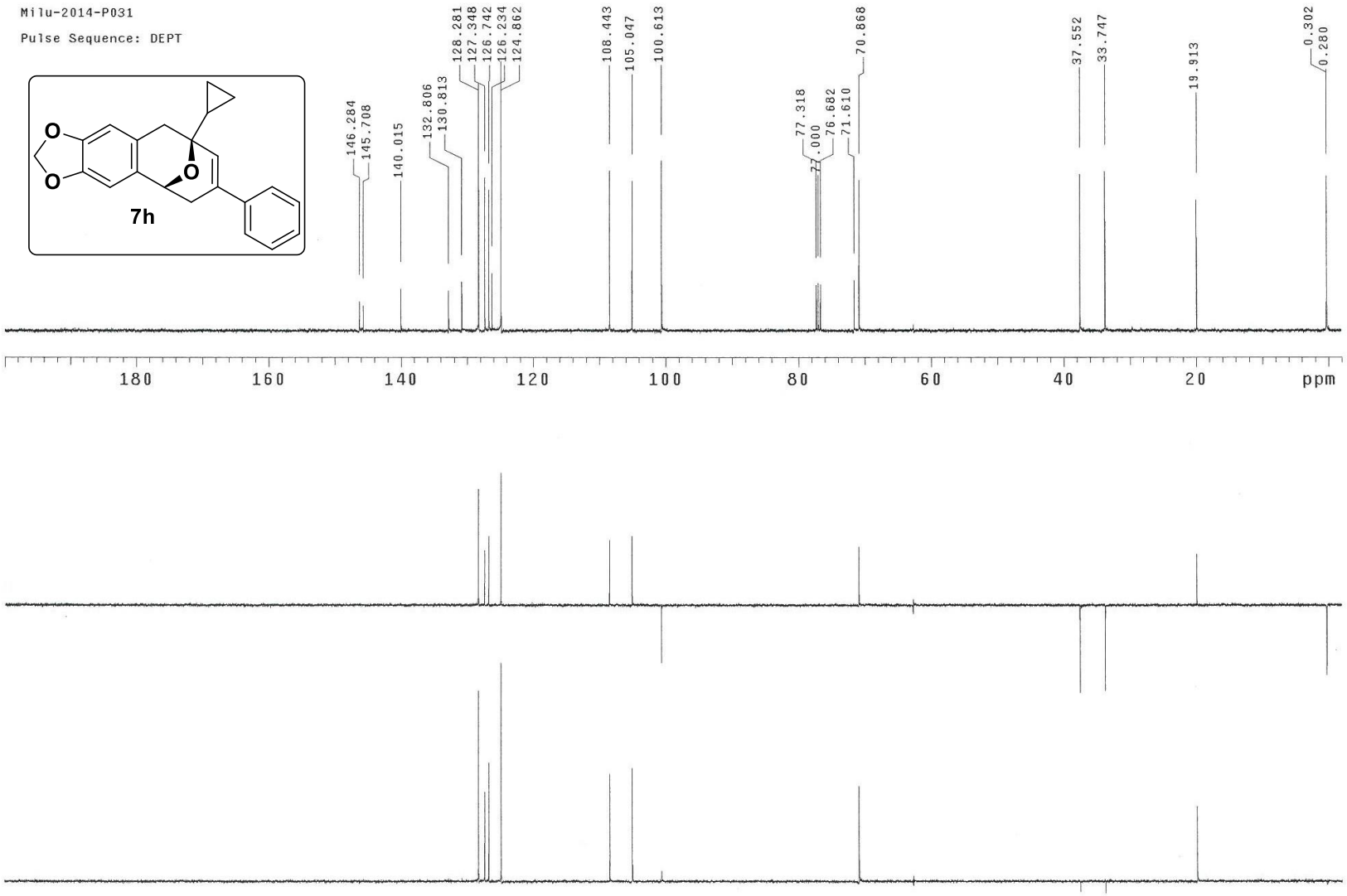
Milu-2014-P031

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Apr 21 2014
Solvent: CDCl3
Ambient temperature
Total 1744 repetitions



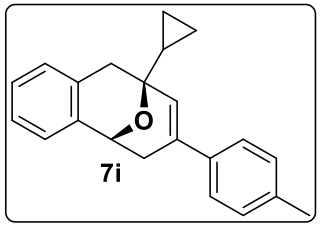


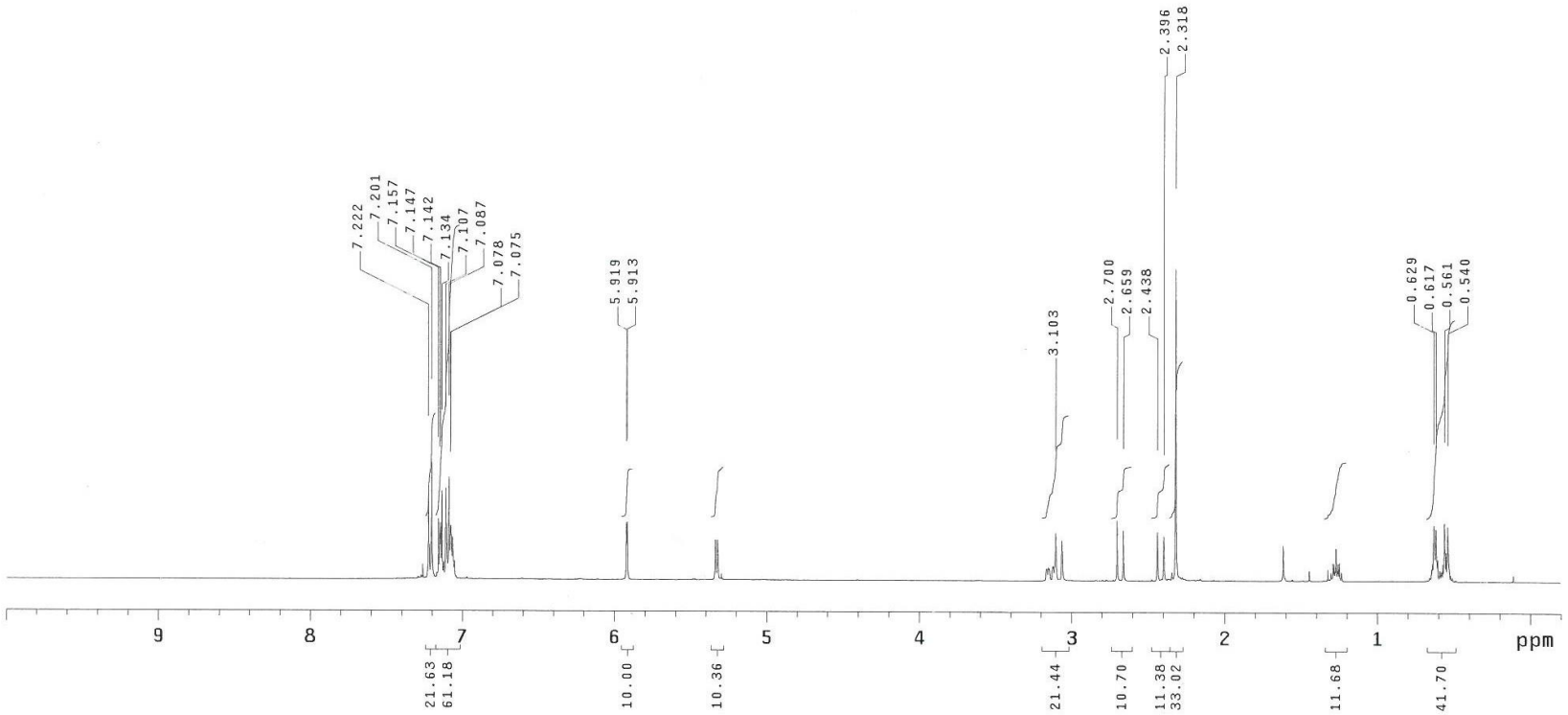

Milu-2014-P031
Pulse Sequence: DEPT
7h
146.284
145.708
140.015
132.806
130.813
128.281
127.348
126.742
126.234
124.862
108.443
105.047
100.613
77.318
77.000
76.682
71.610
70.868
37.552
33.747
19.913
0.302
0.280
180
160
140
120
100
80
60
40
20
ppm

Jaya-BC2014-73

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Dec 25 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

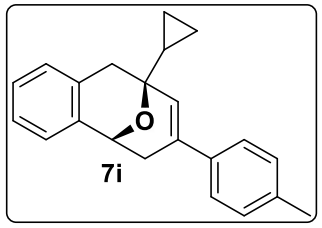




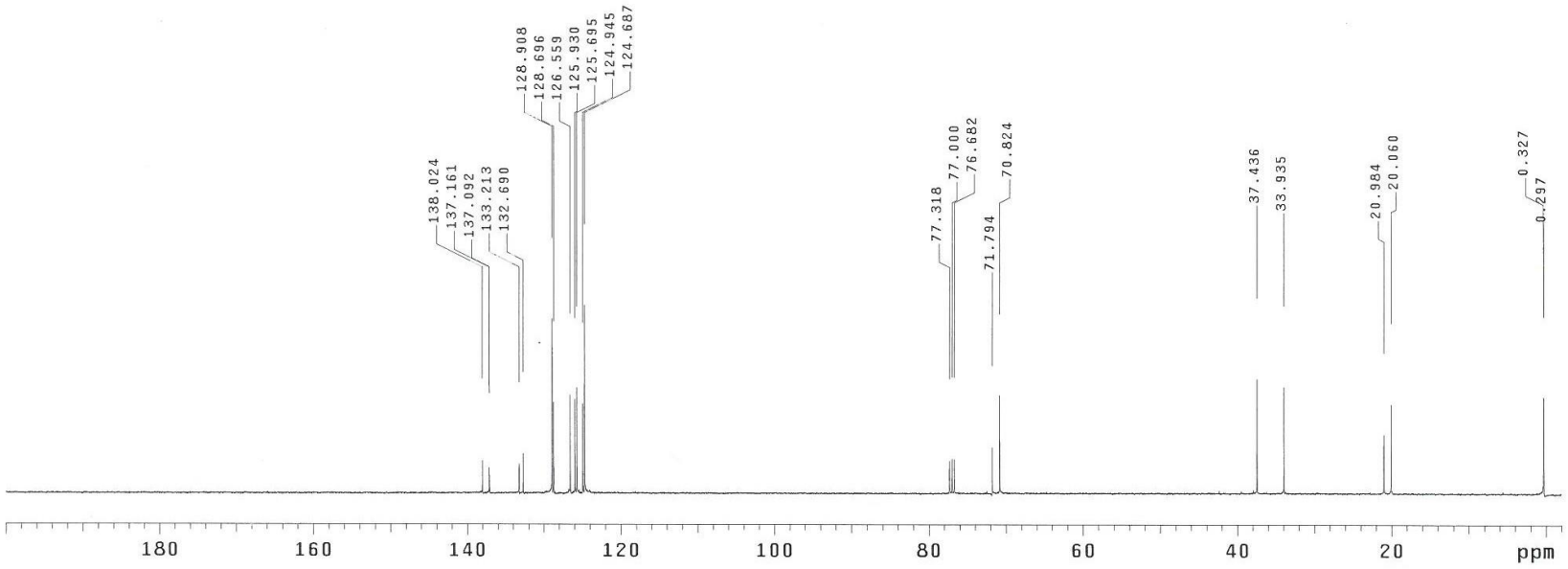
Jaya-BC2014-73

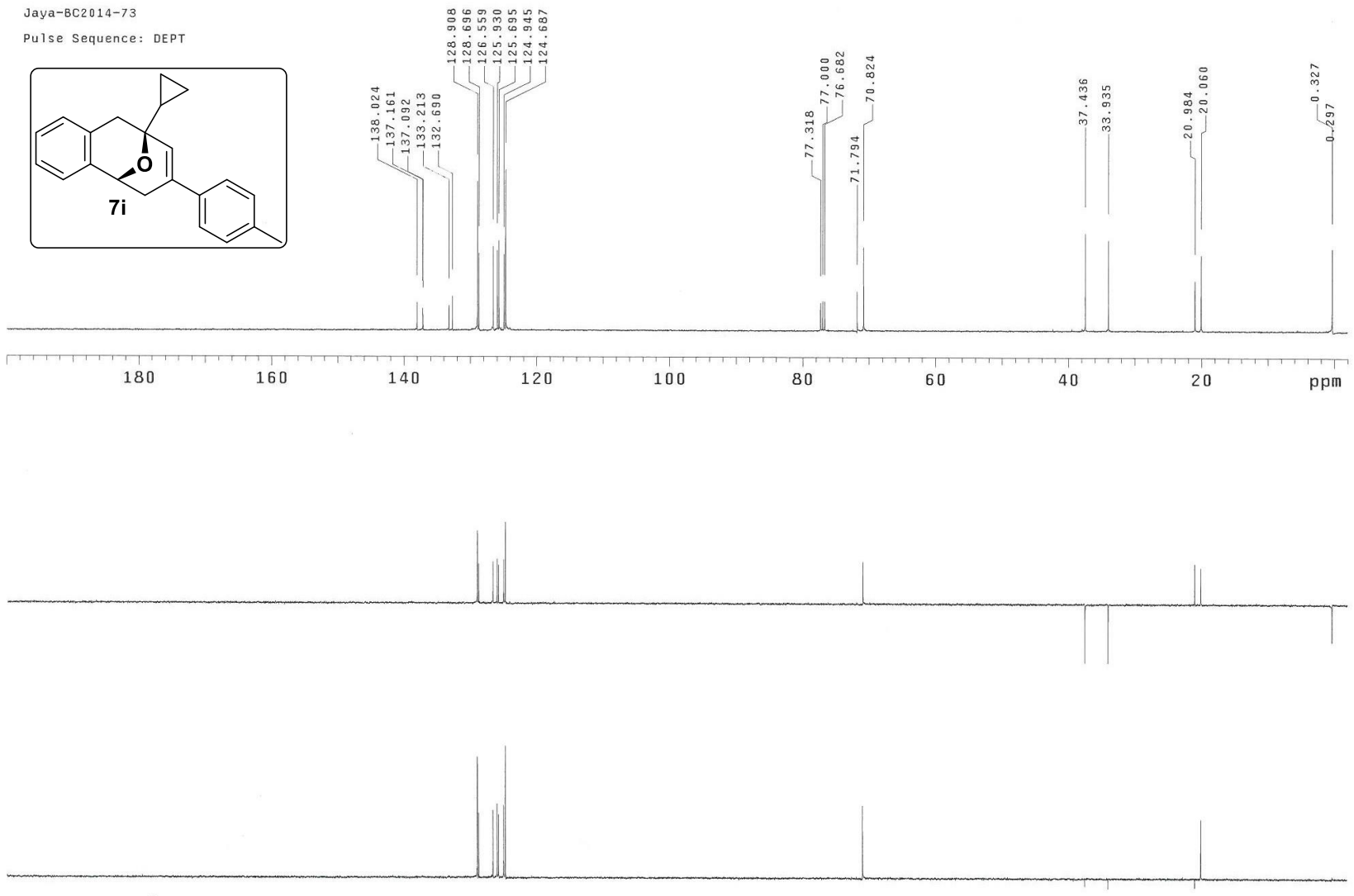
Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Dec 25 2014
Solvent: CDCl3
Ambient temperature
Total 656 repetitions



7i

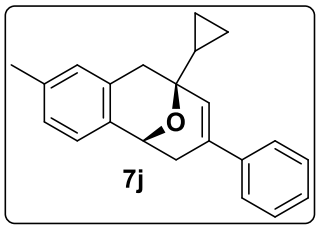



Jaya-BC2014-73
Pulse Sequence: DEPT
7i
O
138.024
137.161
137.092
133.213
132.690
128.908
128.696
126.559
125.930
125.695
124.945
124.687
77.318
77.000
76.682
71.794
70.824
37.436
33.935
20.984
20.060
0.327
0.297
180
160
140
120
100
80
60
40
20
ppm

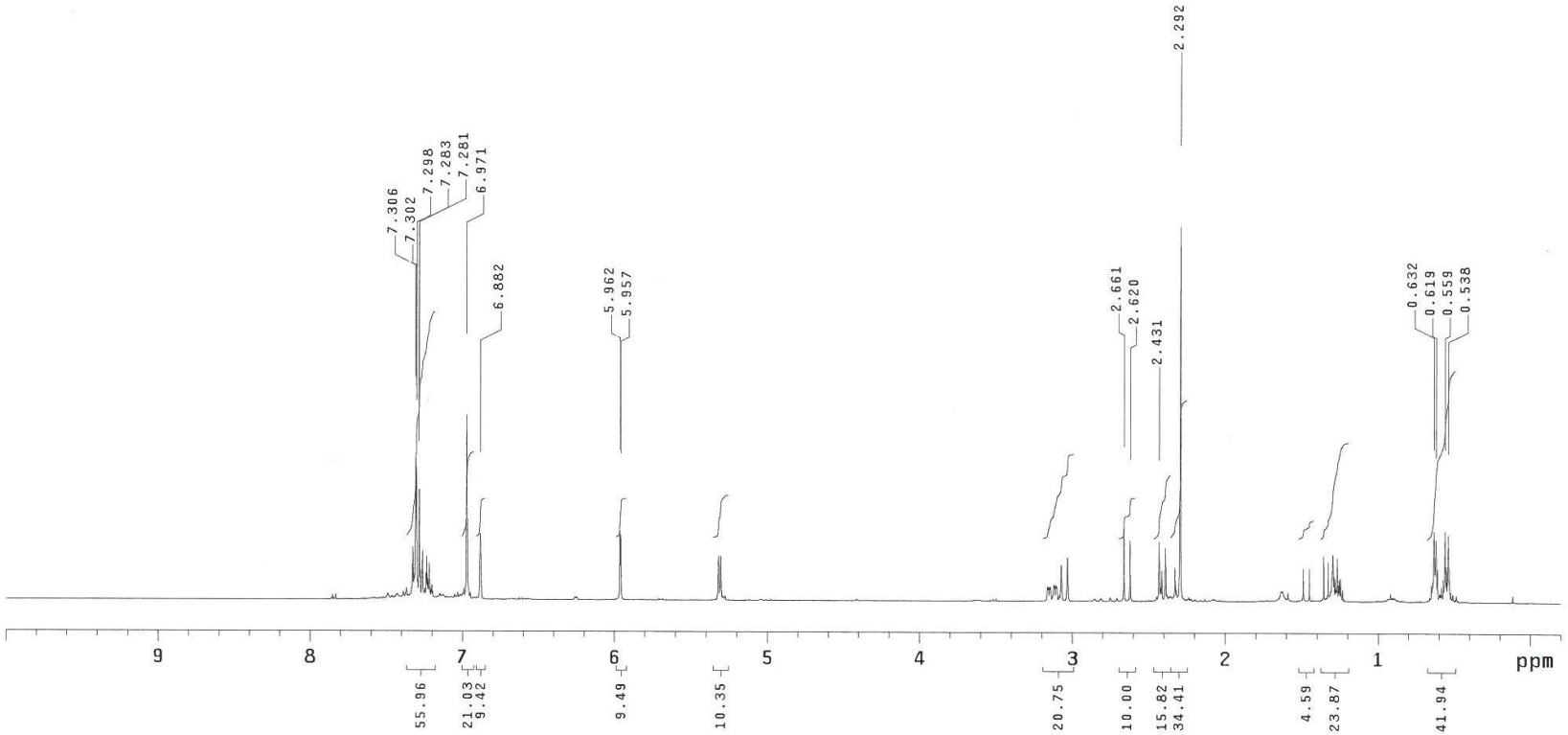
phph0002

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 15 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



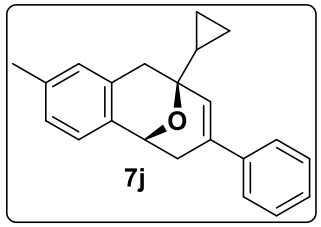
7j



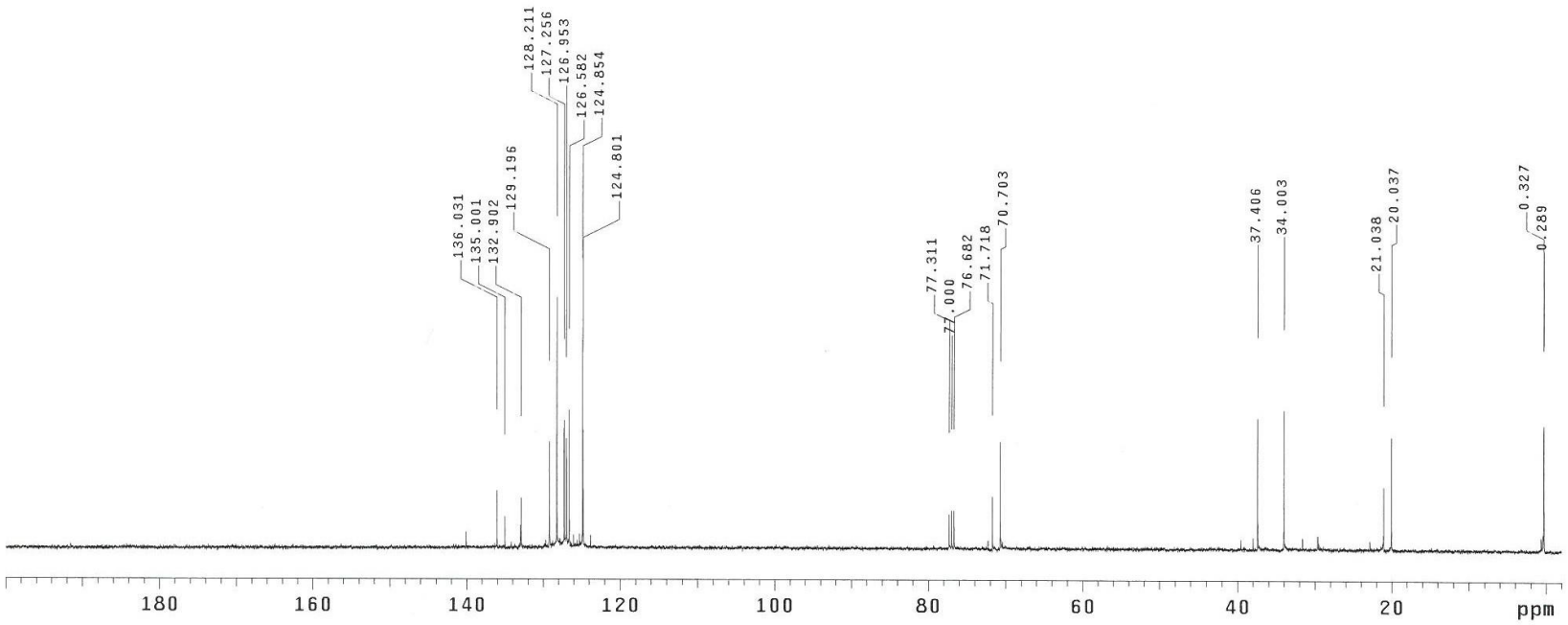
phph0002

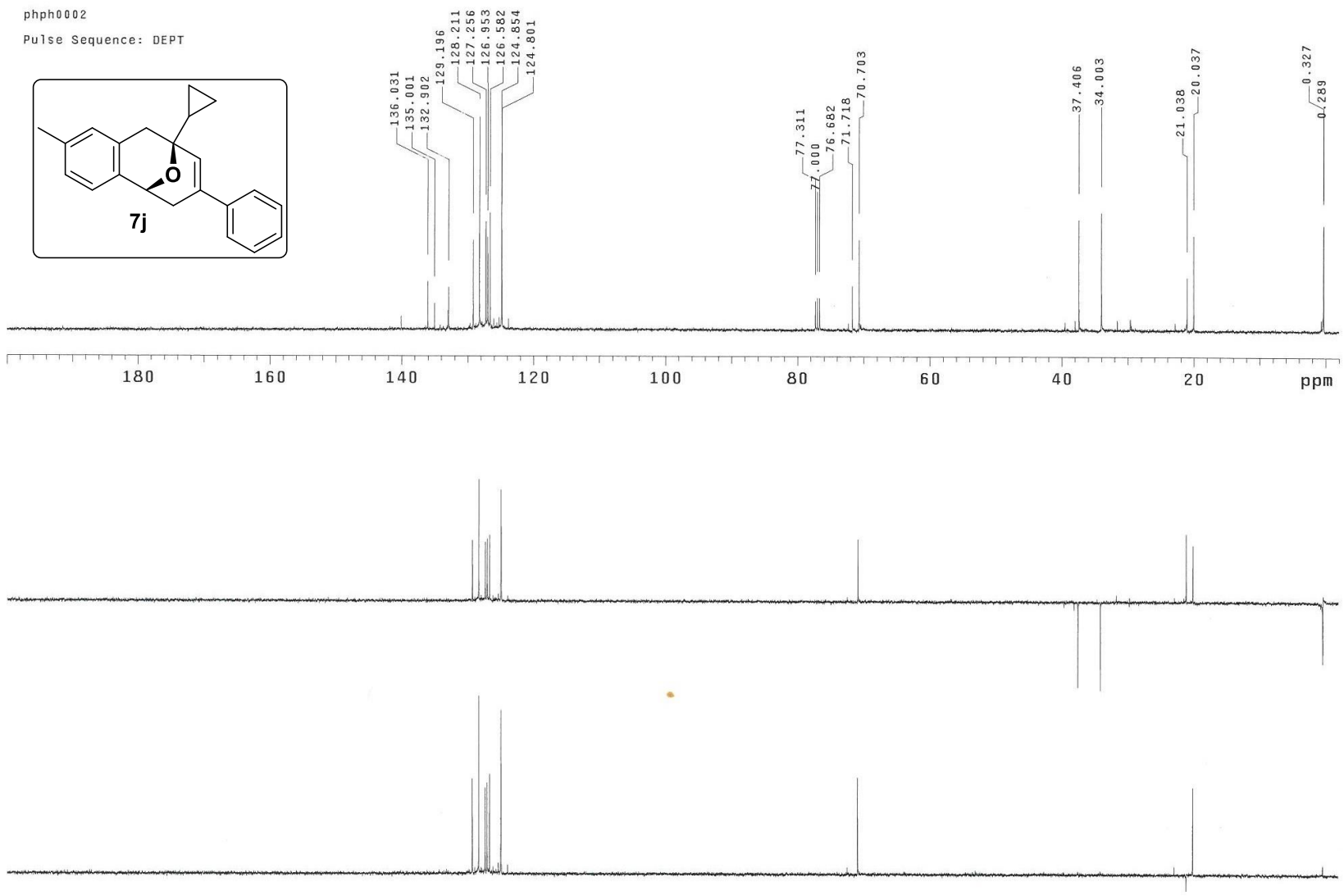
Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 15 2014
Solvent: CDCl3
Ambient temperature
Total 336 repetitions



7j

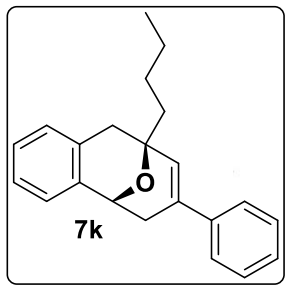


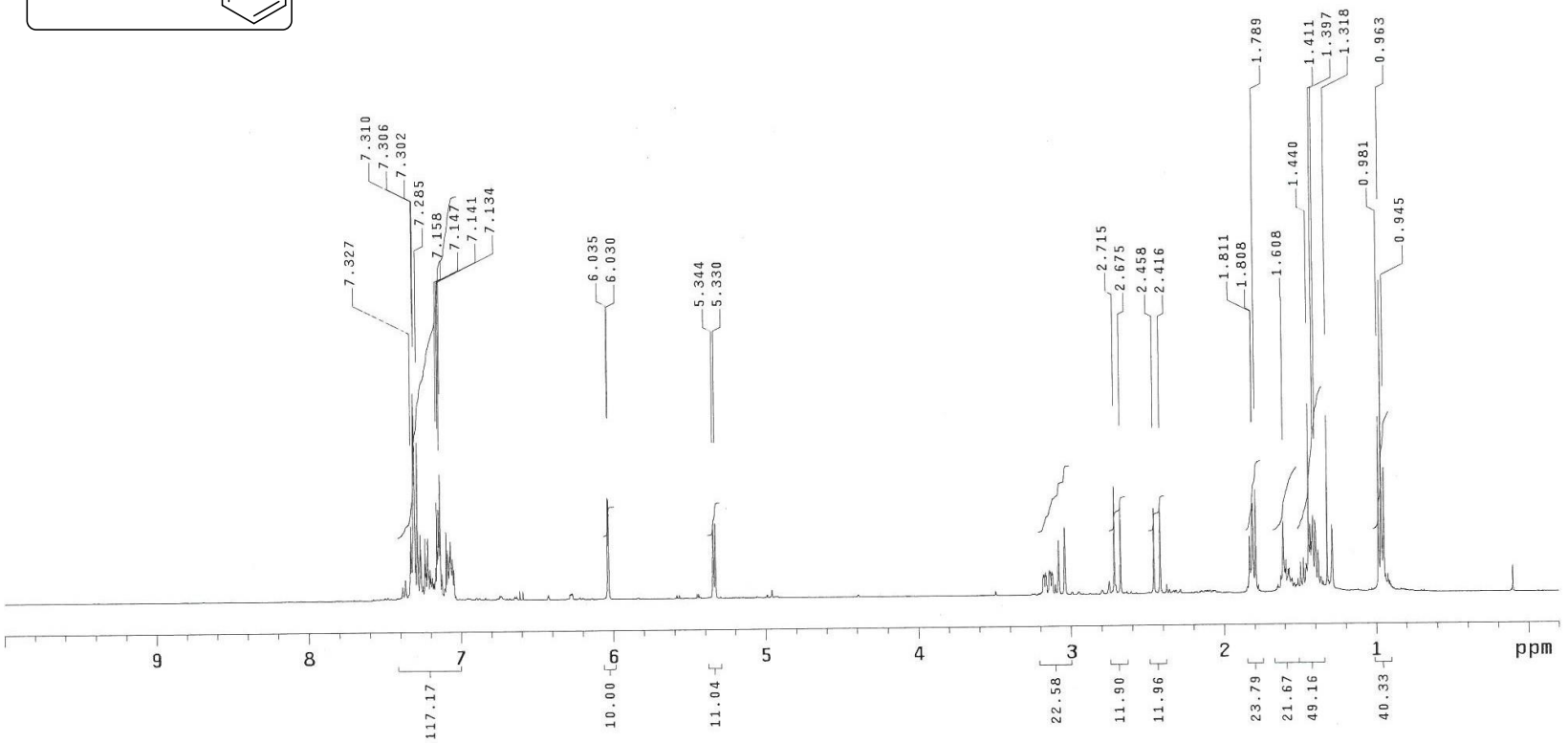

phph0002
Pulse Sequence: DEPT
O
7j
136.031
135.001
132.902
129.196
128.211
127.256
126.953
126.582
124.854
124.801
77.311
77.000
76.682
71.718
70.703
37.406
34.003
21.038
20.037
0.327
0.289
180
160
140
120
100
80
60
40
20
ppm

phph0010

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 17 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

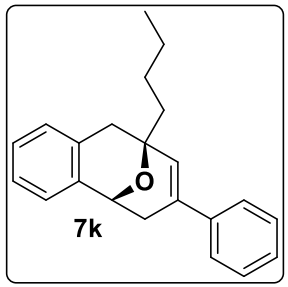


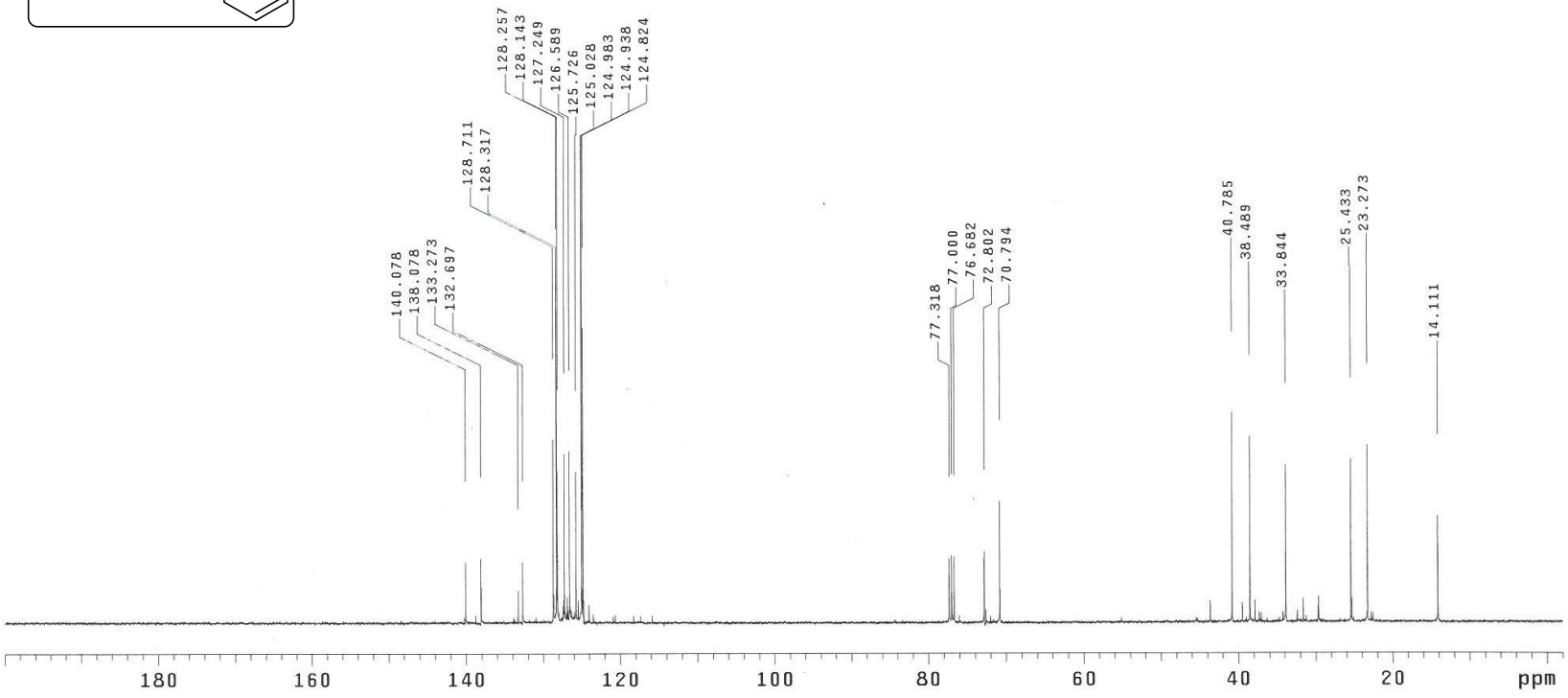


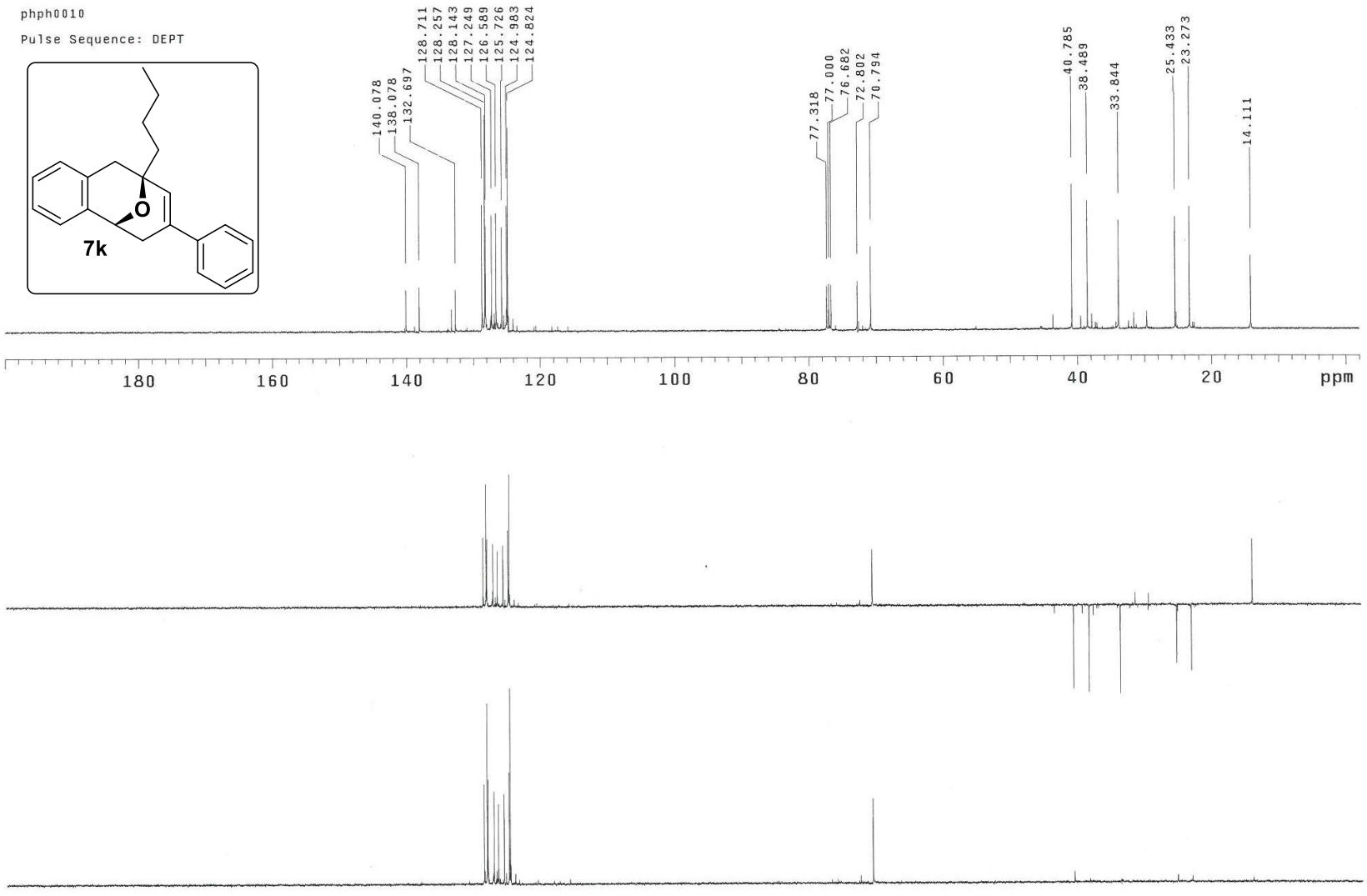
phph0010

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 17 2014
Solvent: CDCl3
Ambient temperature
Total 2016 repetitions



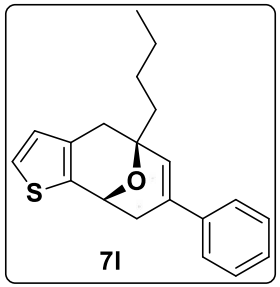



phph0010
Pulse Sequence: DEPT
7k
O
140.078
138.078
132.697
128.711
128.257
128.143
127.249
126.589
125.726
124.983
124.824
77.318
77.000
76.682
72.802
70.794
40.785
38.489
33.844
25.433
23.273
14.111
180
160
140
120
100
80
60
40
20
ppm

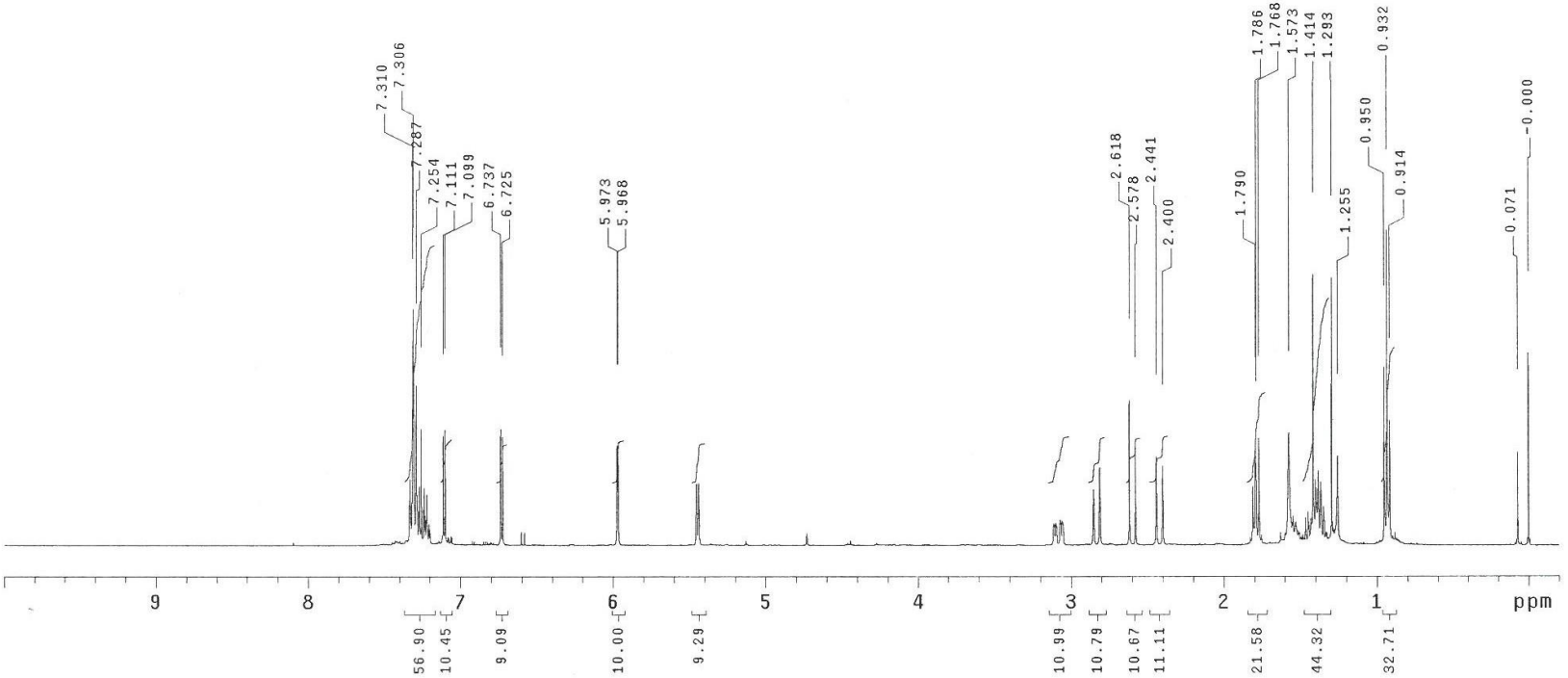
Milu-2014-P079B

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jun 19 2014
Solvent: CDCl3
Ambient temperature
File: U0119-4
Total 160 repetitions



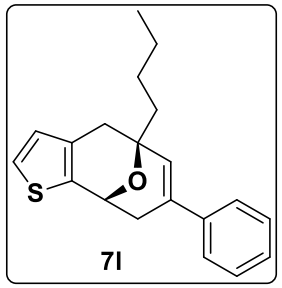
7l



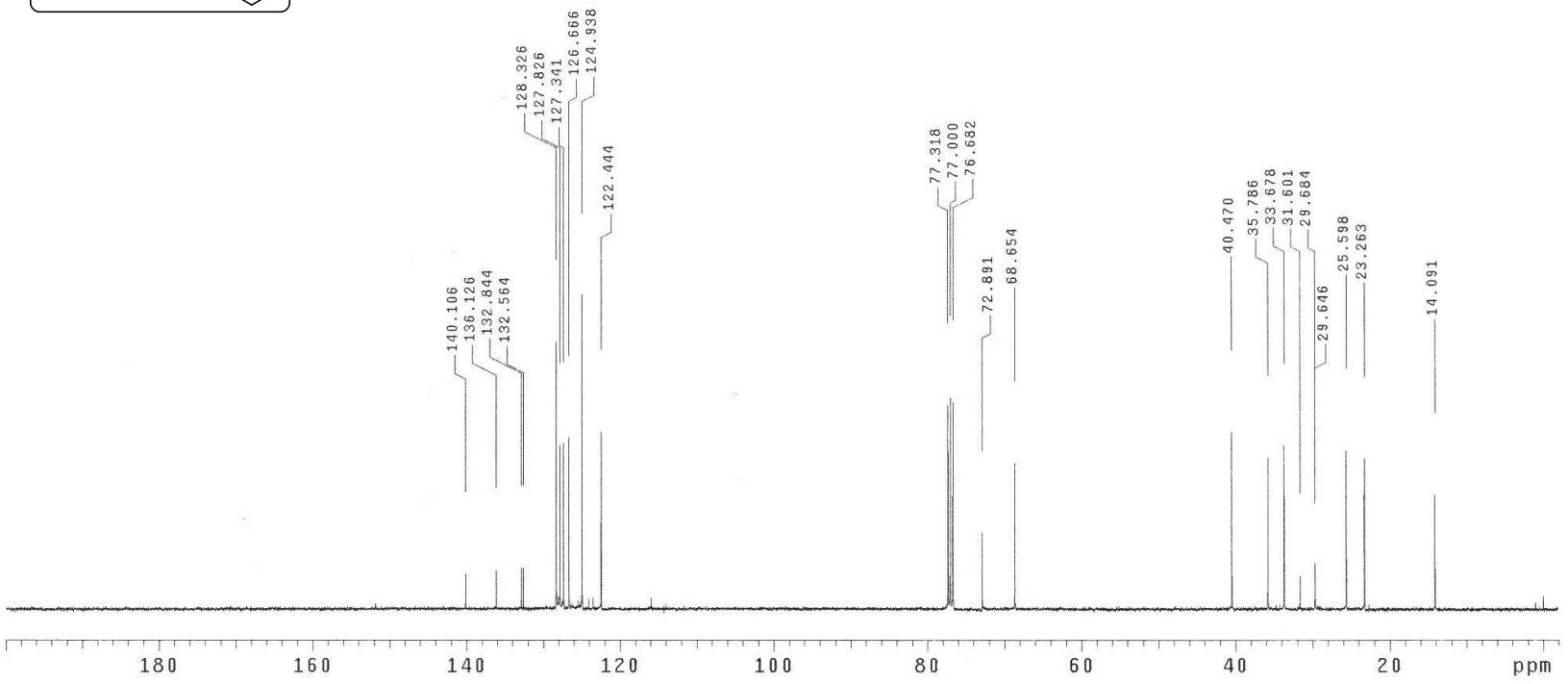
Milu-2014-P079B

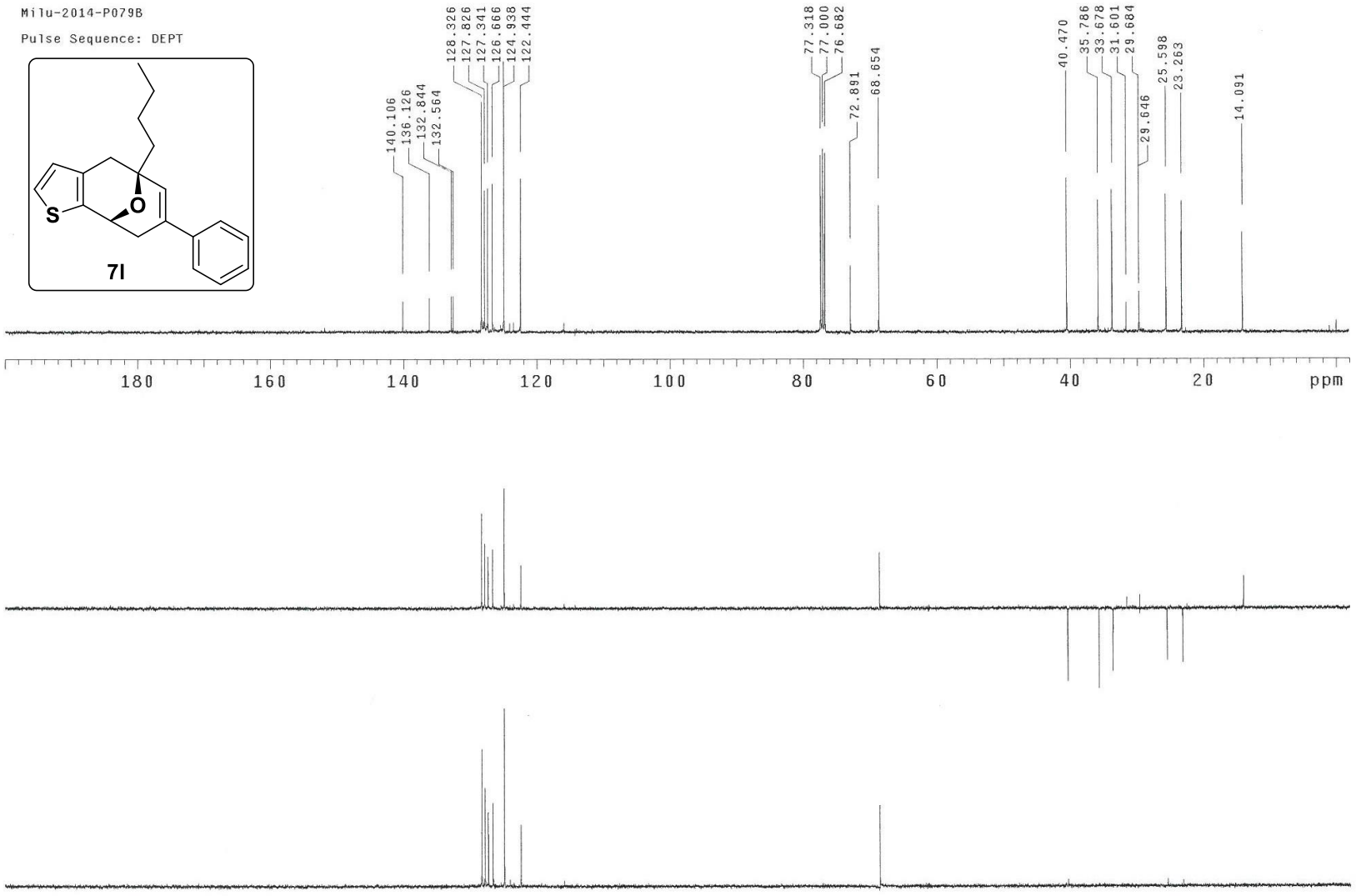
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jun 19 2014
Solvent: CDCl3
Ambient temperature
Total 16000 repetitions



7l



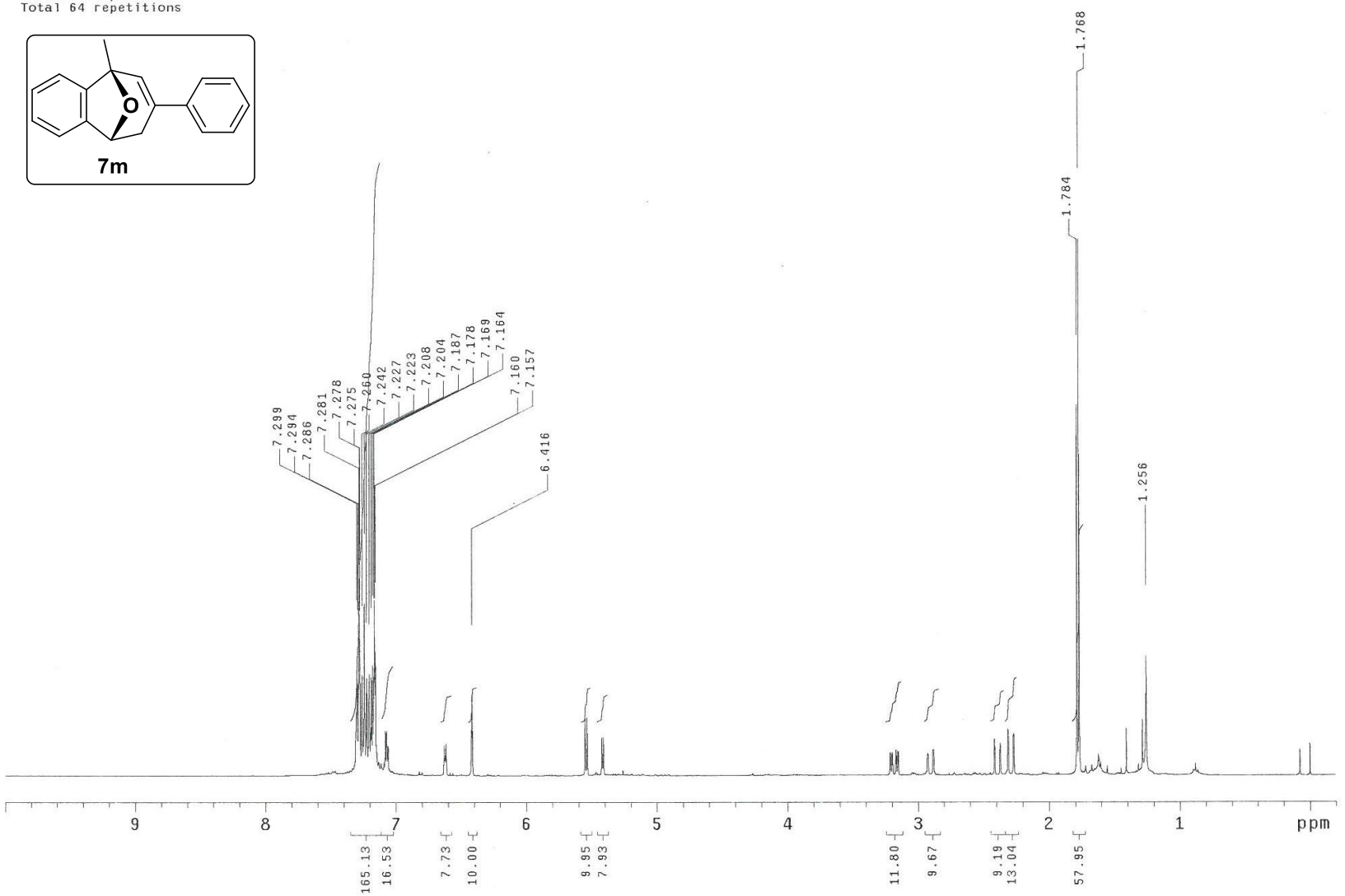

Milu-2014-P079B
Pulse Sequence: DEPT
S
O
7l
140.106
136.126
132.844
132.564
128.326
127.826
127.341
126.666
124.938
122.444
77.318
77.000
76.682
72.891
68.654
40.470
35.786
33.678
31.601
29.684
29.646
25.598
23.263
14.091
180
160
140
120
100
80
60
40
20
ppm

Milu-2014-P079A

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jun 20 2014
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

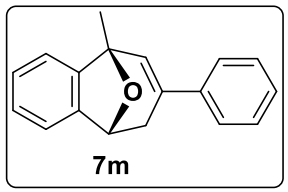
7m



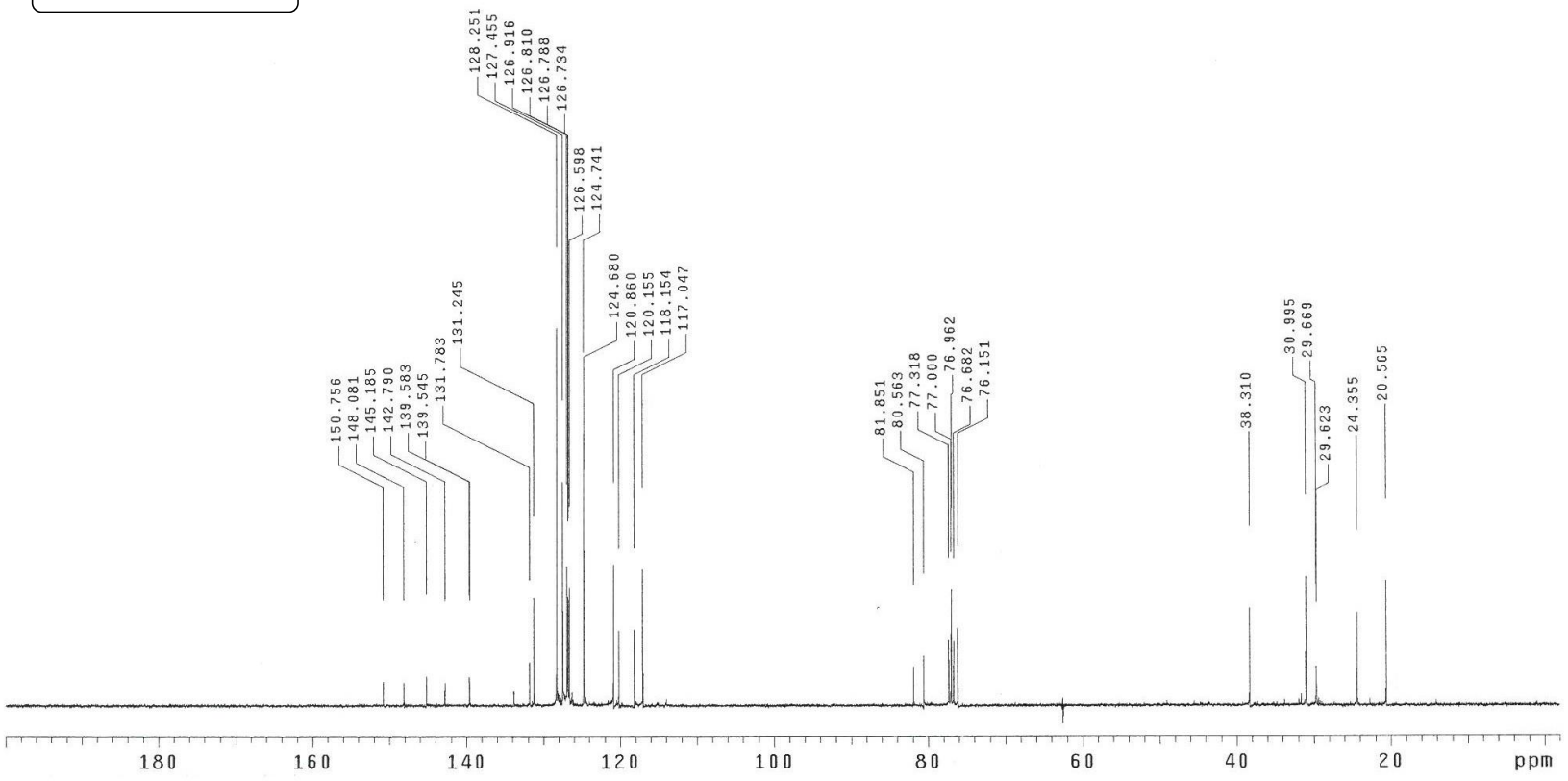
Milu-2014-P079A

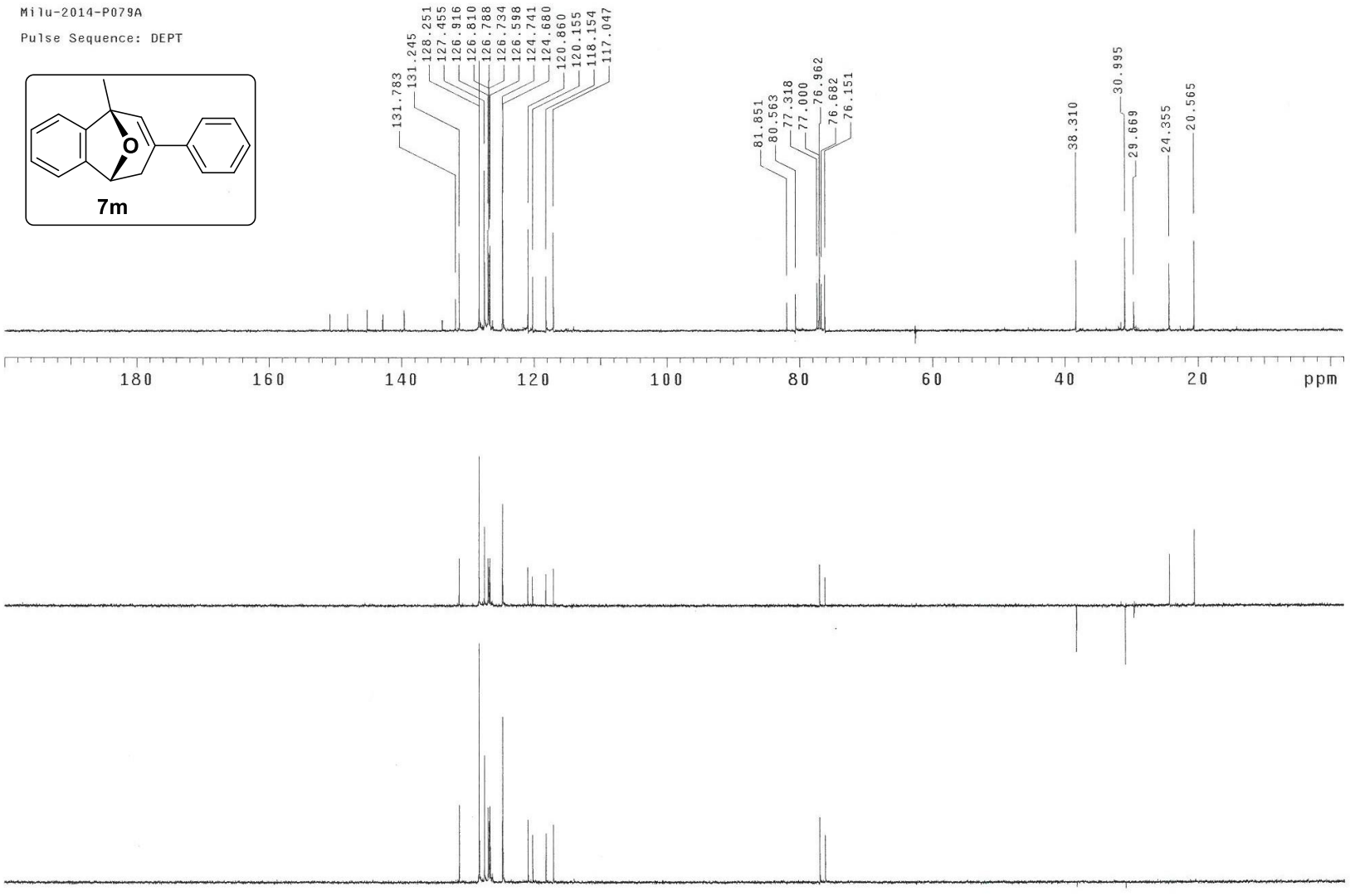
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jun 20 2014
Solvent: CDCl3
Ambient temperature
Total 4112 repetitions



7m



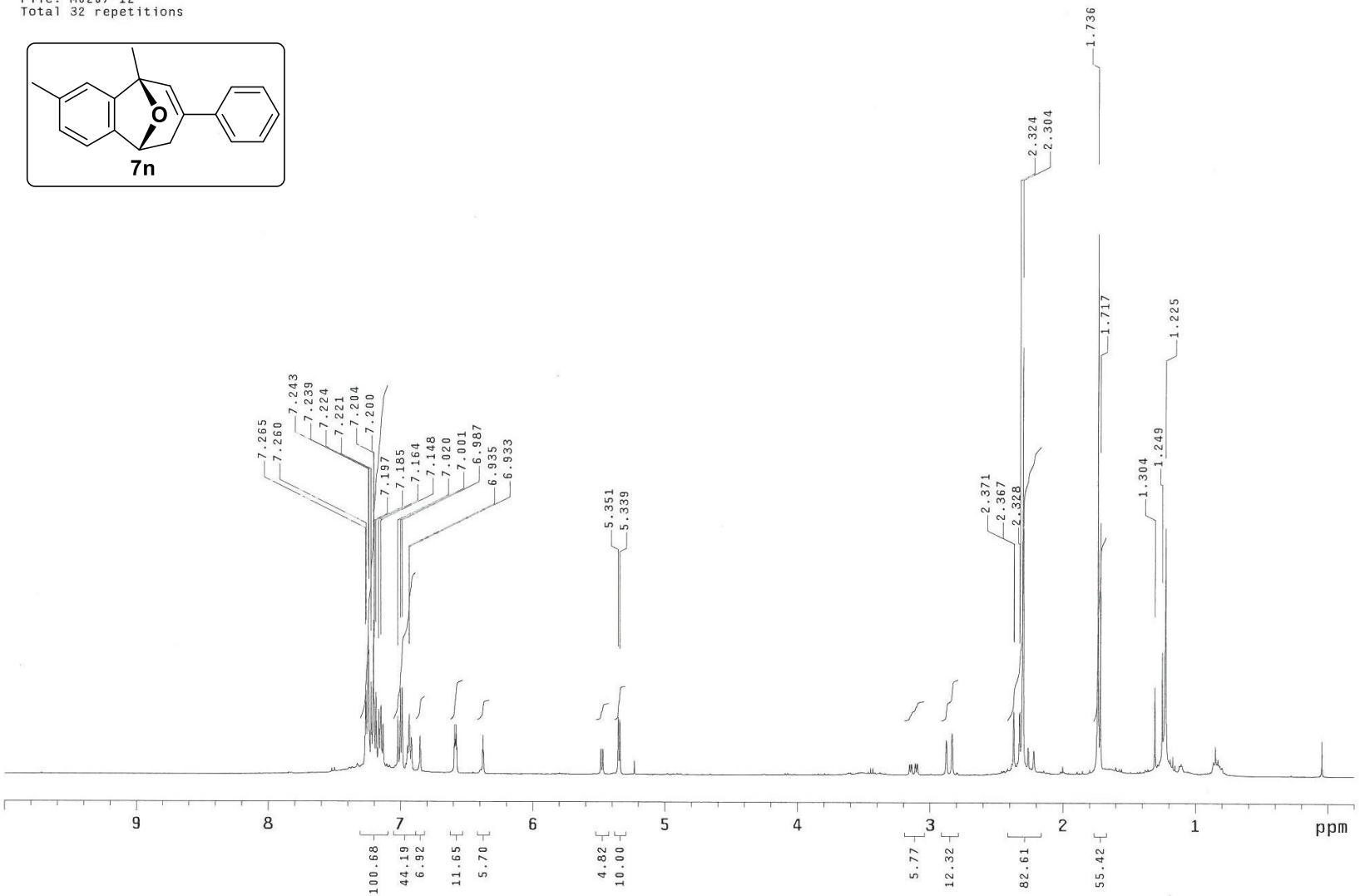

Milu-2014-P079A
Pulse Sequence: DEPT
7m
131.783
131.245
128.251
127.455
126.916
126.810
126.788
126.734
126.598
124.741
124.680
120.860
120.155
118.154
117.047
81.851
80.563
77.318
77.000
76.962
76.682
76.151
38.310
30.995
29.669
24.355
20.565
180
160
140
120
100
80
60
40
20
ppm

phph0004

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Sep 15 2014
Solvent: CDCl3
Ambient temperature
File: M0207-12
Total 32 repetitions

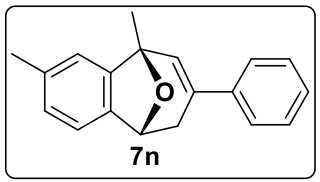
7n



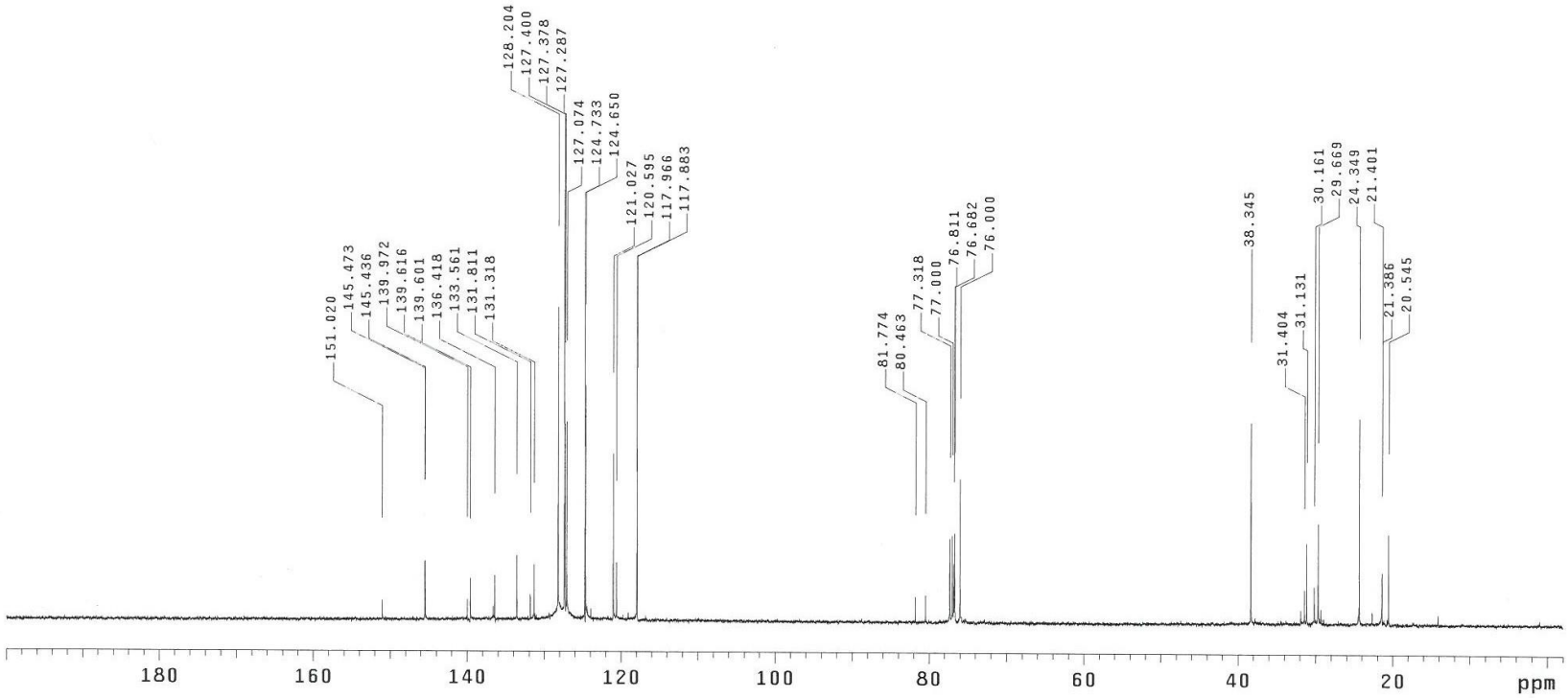
phph0004

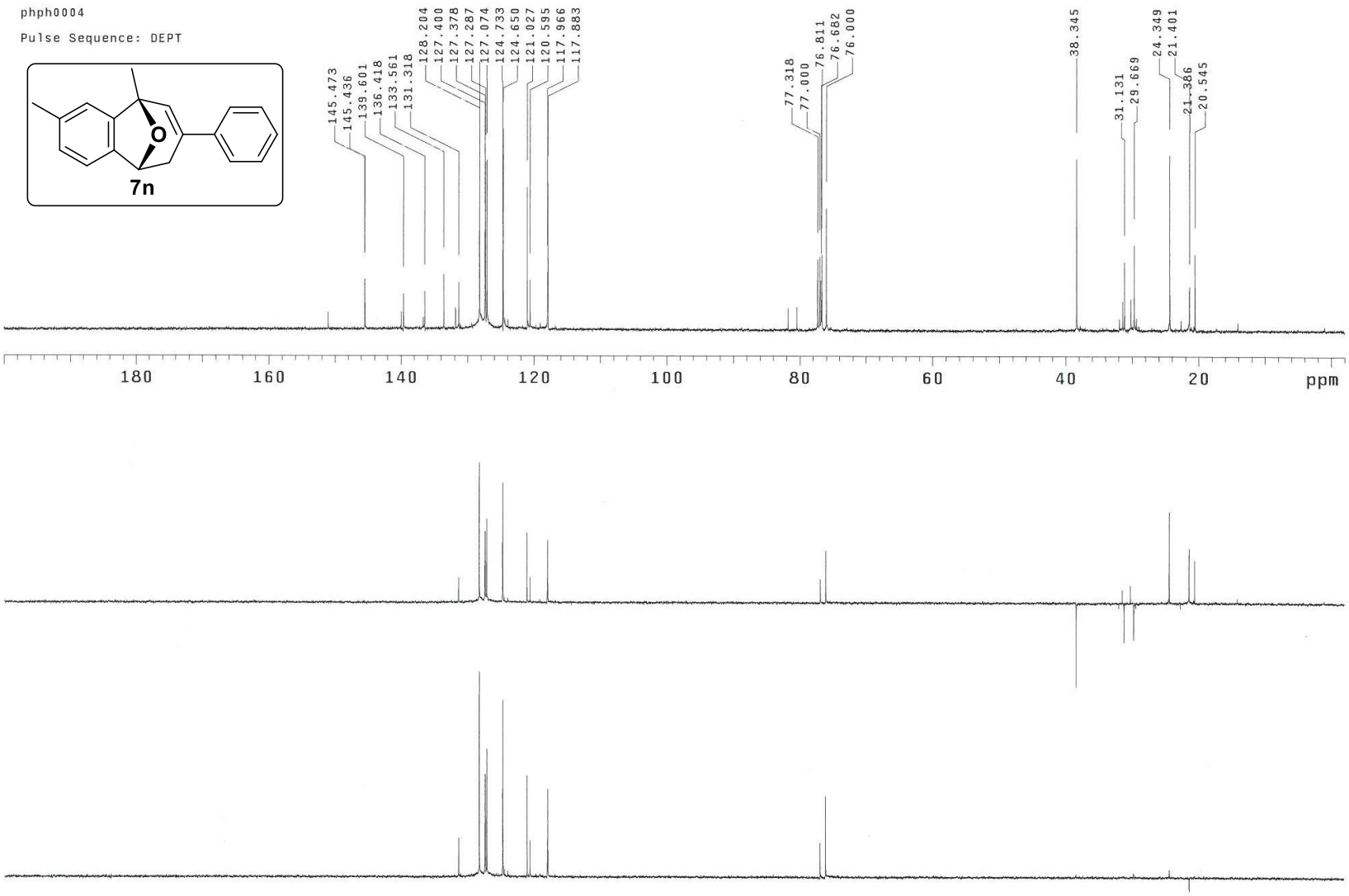
Pulse Sequence: s2pul

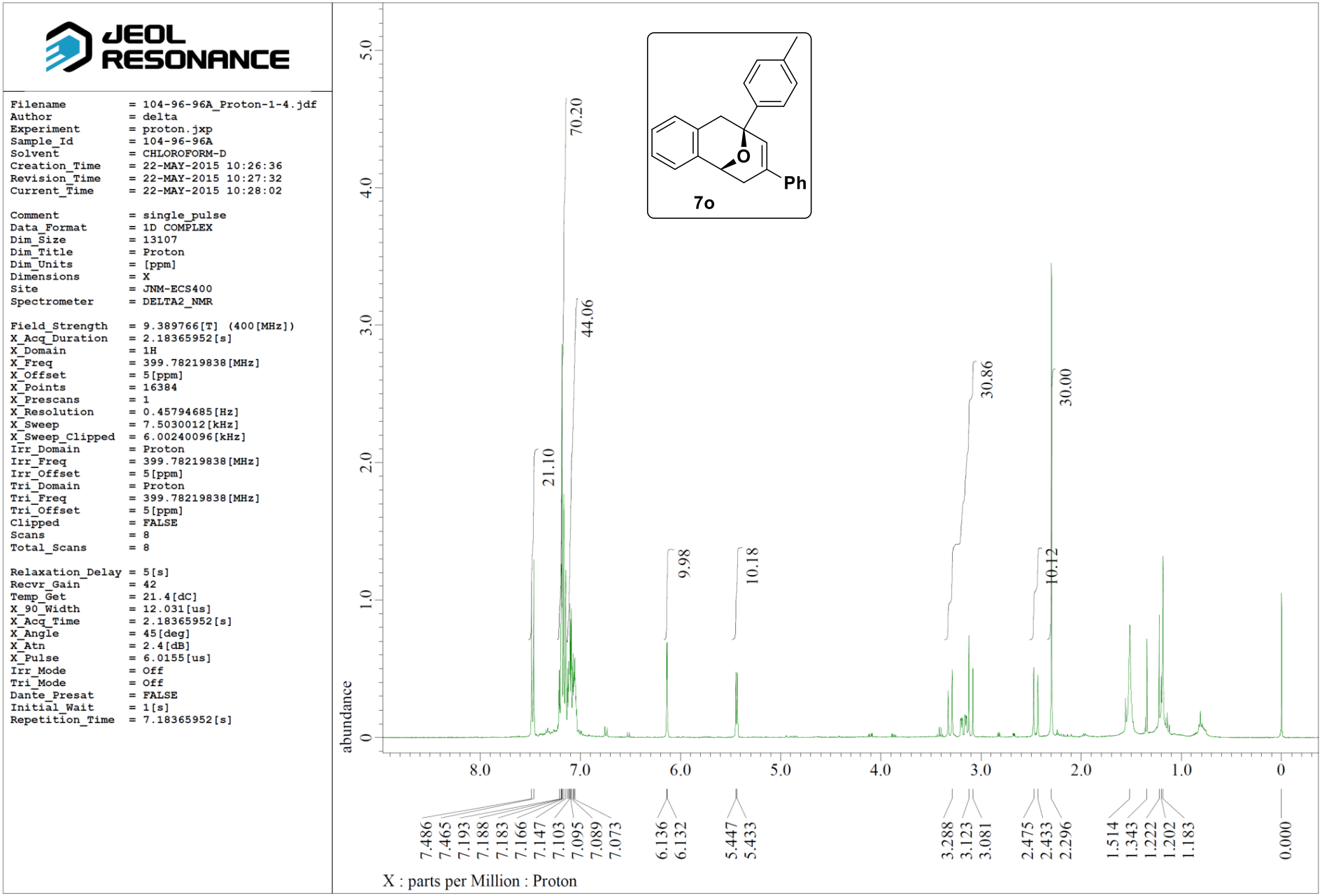
Mercury-400BB "MercuryPlus400"
Date: Sep 15 2014
Solvent: CDCl3
Ambient temperature
Total 32000 repetitions



7n

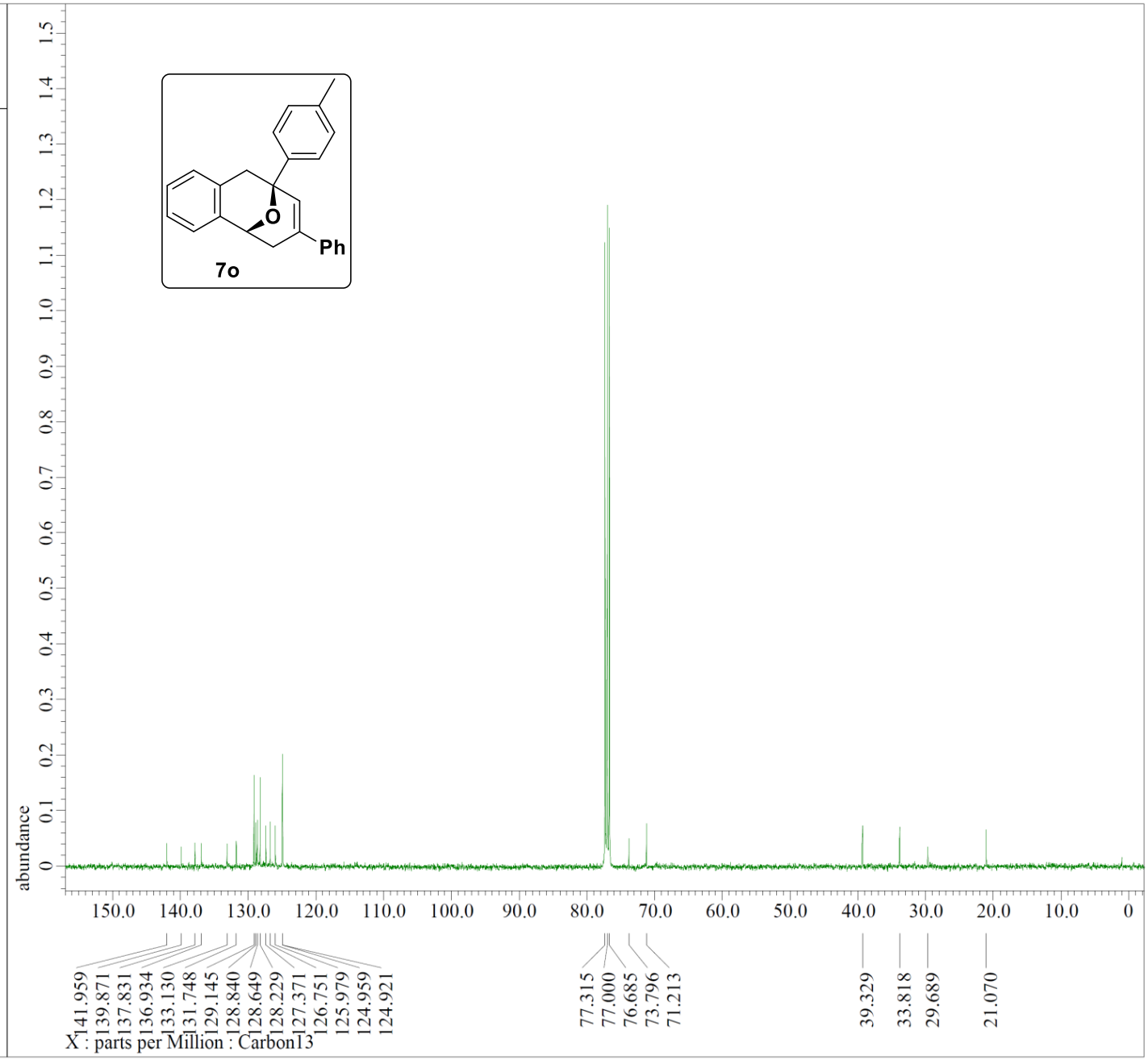



phph0004
Pulse Sequence: DEPT
7n
145.473
145.436
139.601
136.418
133.561
131.318
128.204
127.400
127.378
127.287
127.074
124.733
124.650
121.027
120.595
117.966
117.883
77.318
77.000
76.811
76.682
76.000
38.345
31.131
29.669
24.349
21.401
21.386
20.545
180
160
140
120
100
80
60
40
20
ppm


JEOL RESONANCE
Filename = 104-96-96A_Proton-1-4.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = 104-96-96A
Solvent = CHLOROFORM-D
Creation_Time = 22-MAY-2015 10:26:36
Revision_Time = 22-MAY-2015 10:27:32
Current_Time = 22-MAY-2015 10:28:02
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 42
Temp_Get = 21.4[dC]
X_90_Width = 12.031[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 6.0155[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
O
Ph
7o
abundance
70.20
44.06
21.10
9.98
10.18
30.86
10.12
30.00
7.486
7.465
7.193
7.188
7.183
7.166
7.147
7.103
7.095
7.089
7.073
6.136
6.132
5.447
5.433
3.288
3.123
3.081
2.475
2.433
2.296
1.514
1.343
1.222
1.202
1.183
0.000
X : parts per Million : Proton

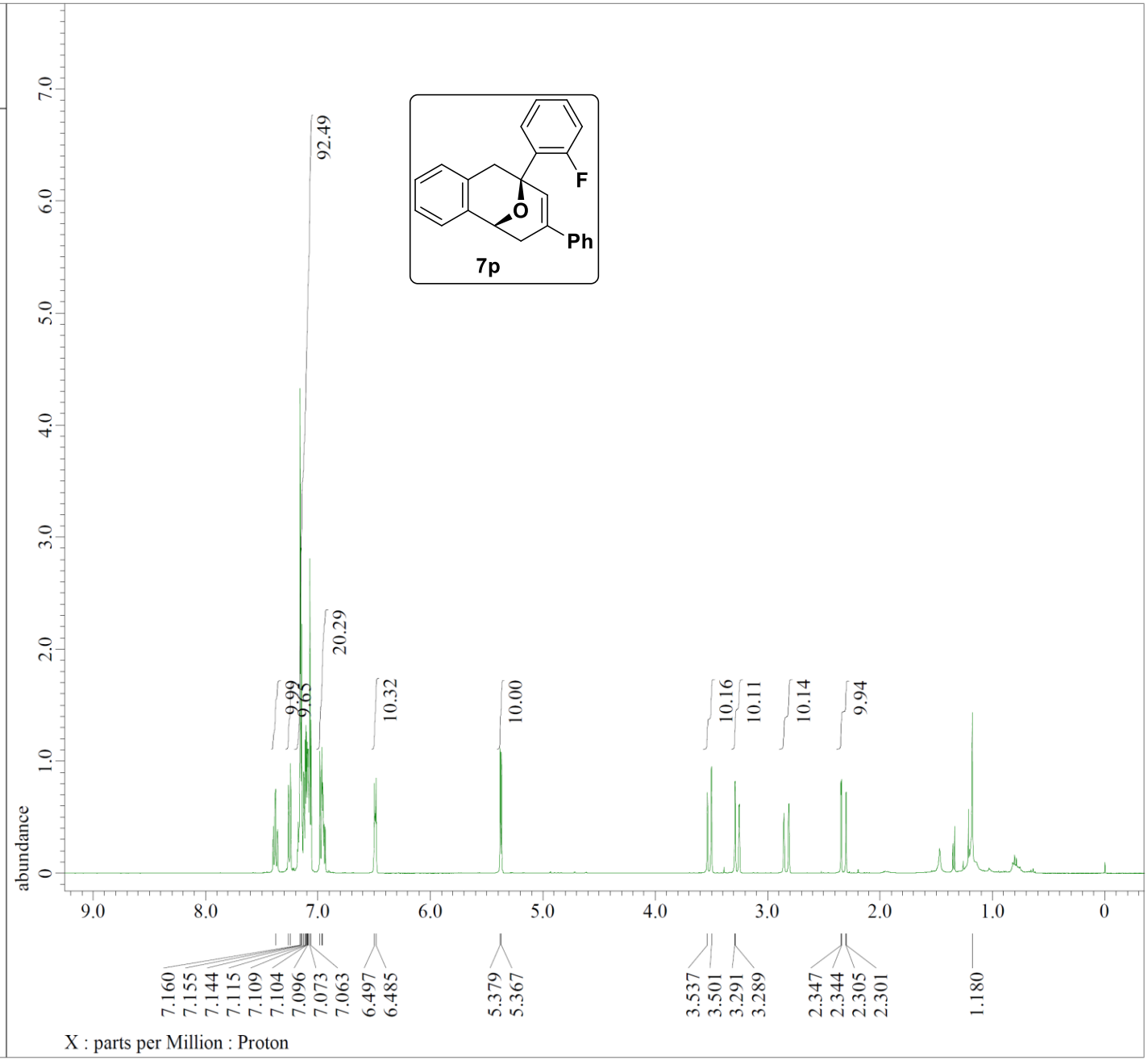
JEOL RESONANCE

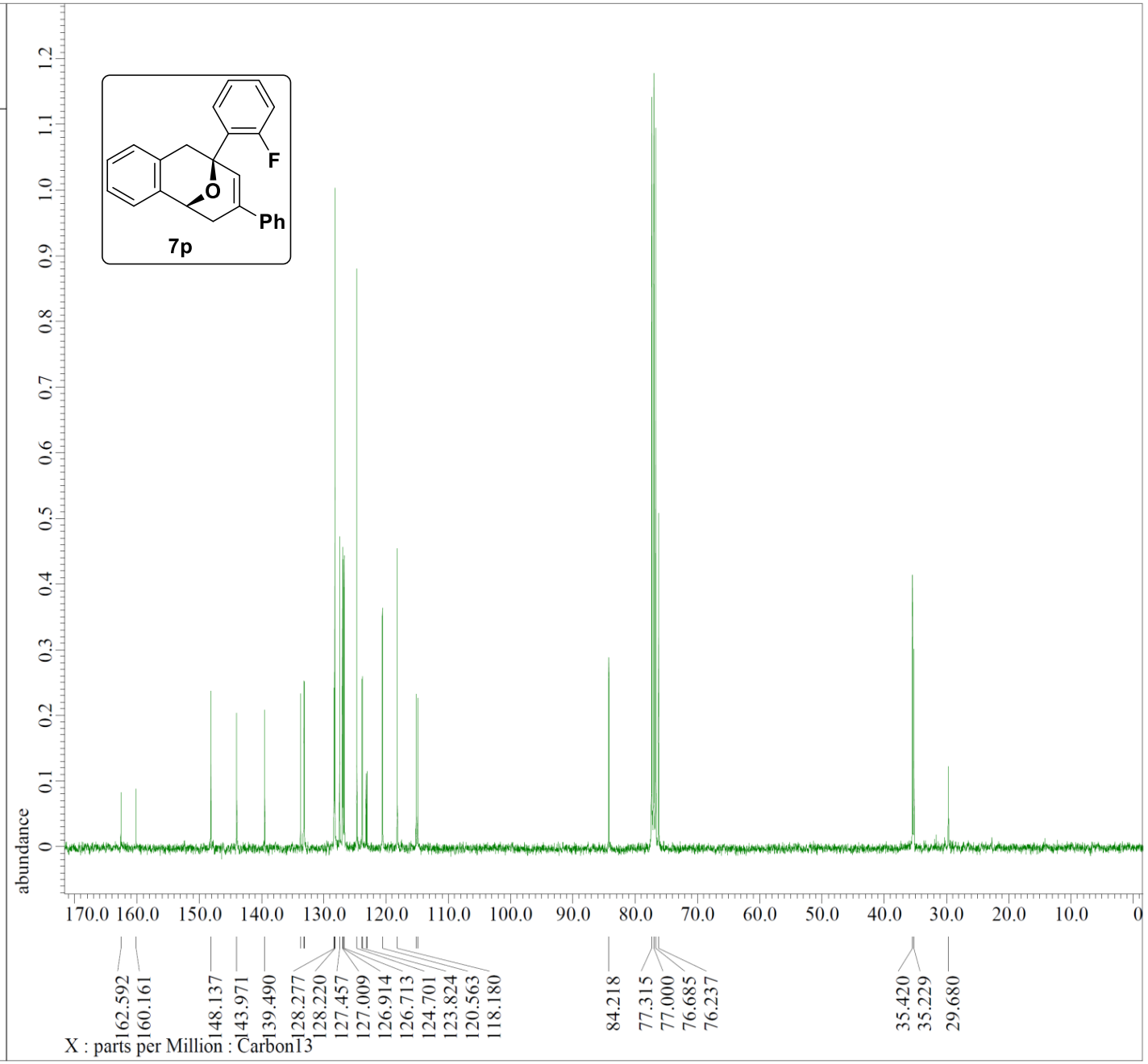
```
Filename          = 104-96-96A_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 104-96-96A
Solvent           = CHLOROFORM-D
Creation_Time     = 22-MAY-2015 10:29:39
Revision_Time     = 22-MAY-2015 11:27:49
Current_Time      = 22-MAY-2015 11:28:58

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22[dC]
X_90_Width        = 9.56[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 5.3[dB]
X_Pulse           = 3.18666667[us]
Irr_Atn_Dec       = 22.008[dB]
Irr_Atn_Noe       = 22.008[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



JEOL RESONANCE

```
Filename          = 104-101-101A_Proton-1-6.jd
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 104-101-101A
Solvent           = CHLOROFORM-D
Creation_Time     = 27-MAY-2015 09:14:39
Revision_Time     = 27-MAY-2015 09:20:52
Current_Time      = 27-MAY-2015 09:21:17

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 32
Temp_Get          = 21.3[dC]
X_90_Width        = 12.031[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2.4[dB]
X_Pulse           = 6.0155[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



JEOL RESONANCE

```
Filename          = 104-101-101A_Carbon-1-3.jd
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 104-101-101A
Solvent           = CHLOROFORM-D
Creation_Time     = 27-MAY-2015 09:17:42
Revision_Time     = 27-MAY-2015 09:37:48
Current_Time      = 27-MAY-2015 09:38:12

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 477.0
Total_Scans       = 477.0

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 21.7[dC]
X_90_Width        = 9.56[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 5.3[dB]
X_Pulse           = 3.18666667[us]
Irr_Atn_Dec       = 22.008[dB]
Irr_Atn_Noe       = 22.008[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```