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Orgno Photoredox Catalysis for Pschorr Reaction：A Metal-free and Mild Approach to 6*H*-benzo[*c*]chromenes

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Supporting Information

1. General information
2. Characterization for 6*H*-benzo[*c*]chromenes
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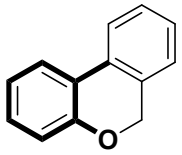
1. General information

^{1}H and ^{13}C NMR spectra were recorded on Brucker AVANCE 400MHz spectrameter (400 MHz for ^{1}H or 100 MHz for ^{13}C, respectively). NMR chemical shifts are expressed in δ values with reference to the residual solvents or TMS as internal standard. IR spectra were taken on an FTIR spectrophotometer. Mass spectra (MS) were measured by the +ESI method. Melting points were determined in open capillary tubes and are uncorrected. The reaction mixture was checked by thin-layer chromatography on silica gel plates (60 F-254) using UV light, or 7% ethanolic phosphomolybdic acid and heating as the visualizing methods. Flash column chromatography over silica gel was used for purification. Yields refer to chromatographically and spectroscopically (^{1}H-NMR) homogeneous materials. Reagents were obtained commercially and used as received.

2. Characterization for 6*H*-benzo[*c*]chromenes

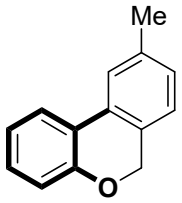
6*H*-benzo[c]chromene (2a)[1]



Obtained in 90% yield (32.9 mg and 0.2 mmol scale) as a colorless oil.

^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.79 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.75 (d, *J* = 7.2 Hz, 1H), 7.43 (ddd, *J* = 7.6, 7.6, 0.8 Hz, 1H), 7.33 (ddd, *J* = 7.6, 7.6, 0.8 Hz, 1H), 7.31-7.28 (m, 1H), 7.20 (d, *J* = 7.6 Hz, 1H), 7.12 (ddd, *J* = 7.2, 7.2, 0.8 Hz, 1H), 7.07 (dd, *J* = 8.0, 0.8 Hz, 1H, 5.18 (s, 2H).

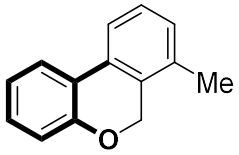
9-methyl-6*H*-benzo[*c*]chromene (2b)[2]



Obtained in 95% yield (37.2 mg and 0.2 mmol scale) as a white solid.

^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.73 (d, *J* = 7.6 Hz, 1H), 7.52 (s, 1H), 7.23 (ddd, *J* =8.0, 8.0, 0.8 Hz, 1H), 7.10 (d, *J* = 7.6 Hz, 1H), 7.05 (dd, *J* = 7.6, 7.6 Hz, 1H), 7.04 (d, *J* = 7.6 Hz, 1H), 6.98 (d, *J* = 8.0 Hz, 1H), 5.09 (s, 2H), 2.41 (s, 3H). ^{13}C NMR (100MHz, $CDCl_3$) ppm 154.8, 138.1, 129.9, 129.3, 128.6, 128.4, 124.6, 123.2, 123.0, 122.7, 122.1, 117.4, 68.4, 21.5.

7-methyl-6*H*-benzo[*c*]chromene (2c)



Obtained in 48% yield (18.9 mg and 0.2 mmol scale) as a white solid.

IR (film) 3028, 2923, 1598, 1456, 1251, 1040, 753 cm^{-1}.

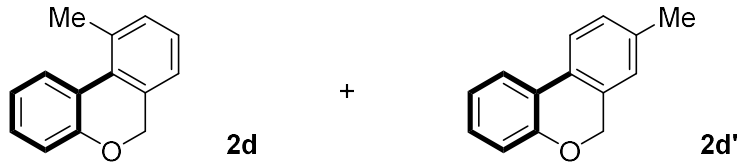
^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.04 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.59 (d, *J* = 7.6 Hz, 1H), 7.29 (dd, *J* = 7.6, 7.6 Hz, 1H), 7.26 (ddd, *J* = 7.6, 7.6, 1.2 Hz, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 7.07 (dd, *J* = 7.6, 7.6 Hz, 1H), 7.01 (d, *J* = 8.0 Hz, 1H), 5.19 (s, 2H), 2.33 (s, 3H).

^{13}C NMR (100MHz, $CDCl_3$) δ ppm 154.4, 133.1, 130.1, 129.9, 129.6, 129.3, 127.9, 123.5, 123.1, 122.1, 119.8, 117.2, 65.6, 18.5.

GC-MS: *m*/*z* (%) = 195 (100), 196 (66) $[M]^+$.

HRMS (CI) calcd for $C_{14}H_{12}O$ 196.0888 $[M]^+$, found 196.0899.

10-methyl-6*H*-benzo[*c*]chromene (2d) and 8-methyl-6*H*-benzo[*c*]chromene (2d')



Obtained in 98% yield (38.4 mg and 0.2 mmol scale) as a white solid.

An inseparable mixture, mole ratio of **2d**:**2d'** is 49:51 according to ^{1}H-NMR analysis.

IR (film, mixture) 2921, 1480, 1453, 1443, 1250, 1233, 1044, 1022, 152, 726 cm^{-1}.

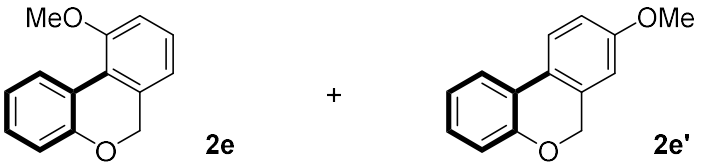
^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.83 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.74 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.63 (d, *J* = 8.0 Hz, 1H), 7.29-7.21 (m, 5H), 7.15-7.08 (m, 4H), 7.02 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.00 (s, 1H), 5.12 (s, 2H), 5.01 (s, 2H), 2.67 (s, 3H), 2.41 (s, 3H).

^{13}C NMR (100MHz, $CDCl_3$) δ ppm 156.5, 154.5, 137.7, 134.5, 134.2, 132.2, 131.4, 129.3, 129.2, 129.0, 128.7, 128.0, 127.3, 127.2, 125.3, 124.5, 123.1, 123.1, 122.7, 122.1, 122.0, 121.5, 117.4, 117.3, 69.8, 68.5, 23.1, 21.3.

GC-MS: *m*/*z* (%) = 195 (100), 196 (87) $[M]^+$.

GC-MS: *m*/*z* (%) = 195 (100), 196 (69) $[M]^+$.

10-methoxy-6*H*-benzo[*c*]chromene (2e) and 8-methoxy-6*H*-benzo[*c*]chromene (2e')[3]



Obtained in 73% yield (31.0 mg and 0.2 mmol scale) as a pale yellow solid.

An inseparable mixture, mole ratio of **2e**: **2e**' is 22:78 according to ^{1}H-NMR analysis.

IR (film, mixture) 2837, 1613, 1483, 1266, 786 cm^{-1}.

10-methoxy-6*H*-benzo[*c*]chromene (2e)

^{1}H NMR (400MHz, $CDCl_3$) δ ppm 8.42 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.30-7.22 (m, 2H), 7.10-6.97 (m, 3H), 6.83 (d, *J* = 7.2 Hz, 1H), 5.03 (s, 2H), 3.96 (s, 3H).

^{13}C NMR (100MHz, $CDCl_3$) δ ppm 156.5, 155.2, 134.5, 129.5, 128.7, 128.6, 123.0, 122.9, 121.7, 118.8, 117.3, 116.8, 111.4, 68.9, 55.6.

GC-MS: *m*/*z* (%) = 212 (100) $[M]^+$.

HRMS (CI) calcd for $C_{14}H_{12}O_2$ 212.0837 $[M]^+$, found 212.0835.

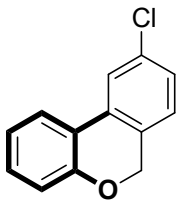
8-methoxy-6*H*-benzo[*c*]chromene (2e')[3]

^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.68 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.65 (d, *J* = 8.8 Hz, 1H), 7.21 (ddd, *J* = 8.0, 8.0, 1.6 Hz, 1H), 7.06 (ddd, *J* = 7.6, 7.6, 1.2 Hz, 1H), 7.00 (dd, *J* = 8.0, 1.2 Hz, 1H), 6.94 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.71 (d, *J* = 2.8 Hz, 1H), 5.11 (s, 2H), 3.86 (s, 3H).

^{13}C NMR (100MHz, $CDCl_3$) δ ppm 159.4, 154.0, 133.0, 128.4, 123.5, 122.9, 122.6, 122.2, 117.2, 114.0, 110.0, 68.5, 55.4.

GC-MS: *m*/*z* (%) = 212 (100) $[M]^+$.

9-chloro-6*H*-benzo[*c*]chromene (2f)[2]

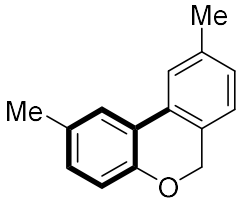


Obtained in 30% yield (13.0 mg and 0.2 mmol scale) as a colorless oil.

^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.69 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.69 (d, *J* = 1.6 Hz, 1H), 7.29 (ddd, *J* = 8.0, 8.0, 1.2 Hz, 1H), 7.27 (dd, *J* =8.0, 2.0 Hz, 1H), 7.10 (d, *J* = 8.4 Hz, 1H), 7.09 (ddd, *J* = 7.6, 7.6, 1.2 Hz, 1H), 7.03 (dd, *J* = 7.6, 1.2 Hz, 1H), 5.10 (s, 1H)

^{13}C NMR (100MHz, $CDCl_3$) δ ppm 154.8, 134.3, 132.0, 130.2, 129.7, 127.5, 126.0, 123.4, 122.4, 122.2, 121.9, 117.6, 67.9.

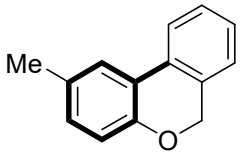
2,9-dimethyl-6*H*-benzo[*c*]chromene (2g)[2]



Obtained in 88% yield (37.0 mg and 0.2 mmol scale) as a pale yellow oil.

^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.53 (d, *J* = 1.6 Hz, 1H), 7.50 (s, 1H), 7.08 (dd, *J* = 7.6, 0.4 Hz, 1H), 7.02 (d, *J* = 7.6 Hz, 1H), 7.02 (d, *J* = 7.6 Hz, 1H), 6.88 (d, *J* = 8.0 Hz, 1H), 5.04 (s, 2H), 2.40 (s, 3H), 2.35 (s, 3H).

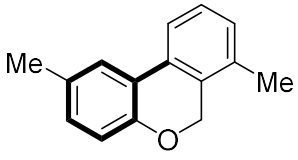
2-methyl-6*H*-benzo[*c*]chromene (2h)[1]



Obtained in 90% yield (35.3 mg and 0.2 mmol scale) as a colorless oil.

^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.73 (d, *J* = 7.6 Hz, 1H), 7.58 (d, *J* = 1.2 Hz, 1H), 7.41 (ddd, *J* = 7.6, 7.6, 0.8 Hz, 1H), 7.31 (ddd, *J* =7.6, 7.6, 0.8 Hz, 1H), 7.18 (d, *J* = 7.2 Hz, 1H), 7.09 (dd, *J* = 8.0, 2.0 Hz, 1H), 6.94 (d, *J* = 8.4 Hz, 1H), 5.13 (s, 2H), 2.41 (s, 3H).

2,7-dimethyl-6*H*-benzo[*c*]chromene (2i)



Obtained in 64% yield (26.9 mg and 0.2 mmol scale) as a white solid. mp 72-73 °C.

IR (film) 2923, 1501, 1459, 1250, 1028, 816, 736 cm^{-1}.

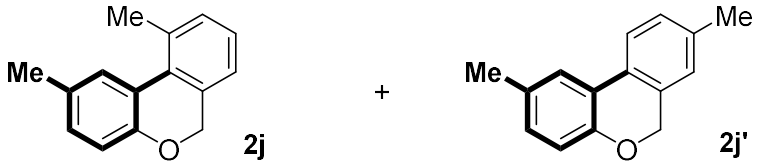
^1H NMR (400MHz, $CDCl_3$) δ ppm 7.58 (d, *J* = 8.0 Hz, 1H), 7.55 (d, *J* = 1.6 Hz, 1H), 7.29 (dd, *J* = 8.0, 7.6 Hz, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 7.06 (dd, *J* = 8.4, 1.6 Hz, 1H), 6.91 (d, *J* = 8.0 Hz, 1H), 5.16 (s, 2H), 2.39 (s, 3H), 2.33 (s, 3H).

^{13}C NMR (100MHz, $CDCl_3$) δ ppm 152.3, 133.1, 131.3, 130.2, 130.0, 129.9, 129.4, 127.8, 123.9, 122.8, 119.8, 116.8, 65.6, 20.9, 18.5.

GC-MS: *m*/*z* (%) = 209 (100) $[M-H]^+$, 210 (72) $[M]^+$

HRMS (CI) calcd for $C_{15}H_{14}O$ 210.1045 $[M]^+$, found 210.1048.

2,10-dimethyl-6*H*-benzo[*c*]chromene (2j)[4] and 2,8-dimethyl-6*H*-benzo[*c*]chromene (2j')[4]



Obtained in 77% yield (32.3 mg and 0.2 mmol scale) as a pale yellow oil.

An inseparable mixture, mole ratio of **2j**:**2j'** is 50:50 according to ^1H-NMR analysis.

IR (film, mixture) 2918, 1494, 1458, 1265, 820, 786 cm^{-1}.

2,10-dimethyl-6*H*-benzo[*c*]chromene (2j)

^1H NMR (400MHz, $CDCl_3$) δ ppm 7.62 (s, 1H), 7.25-7.20 (m, 2H), 7.08 (d, *J* = 7.6 Hz, 2H), 7.00 (d, *J* = 8.0 Hz, 1H), 4.96 (s, 2H), 2.69 (s, 3H), 2.41 (s, 3H).

^{13}C NMR (100MHz, $CDCl_3$) ppm 154.3, 134.6, 134.1, 132.1, 130.6, 129.4, 129.2, 128.4, 127.0, 124.3, 122.7, 117.0, 69.9, 23.1, 21.2.

GC-MS: *m*/*z* (%) = 209 (99), 210 (100) $[M]^+$.

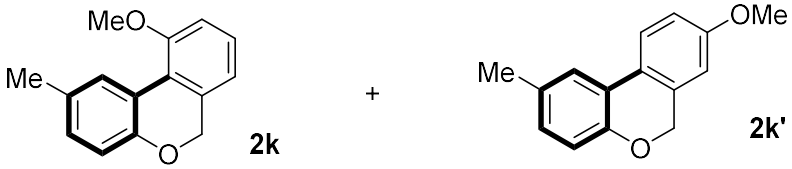
2,8-dimethyl-6*H*-benzo[*c*]chromene (2j')

^1H NMR (400MHz, $CDCl_3$) δ ppm 7.61 (s, 1H), 7.54 (d, *J* = 1.6 Hz, 1H), 7.21 (d, *J* = 7.6 Hz, 1H), 7.04 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.99 (s, 1H), 6.91 (d, *J* = 8.4 Hz, 1H), 5.08 (s, 2H), 2.40 (s, 3H), 2.39 (s, 3H). 2.39 (s, 3H).

^{13}C NMR (100MHz, $CDCl_3$) δ ppm 152.4, 137.5, 131.6, 131.3, 129.6, 129.1, 127.5, 125.3, 123.4, 122.8, 121.9, 117.0, 68.6, 21.3, 20.9.

GC-MS: *m*/*z* (%) = 209 (100), 210 (76) $[M]^+$.

10-methoxy-2-methyl-6*H*-benzo[*c*]chromene (2k) and 8-methoxy-2-methyl-6*H*-benzo[*c*]chromene (2k')



Obtained in 73% yield (33.0 mg and 0.2 mmol scale) as a pale yellow oil.

An inseparable mixture, mole ratio of **2k**:**2k'** is 24:76 according to ^{1}H-NMR analysis.

IR (film, mixture) 2923, 1611, 1495, 1463, 1278, 1026, 786 cm^{-1}.

10-methoxy-2-methyl-6*H*-benzo[*c*]chromene (2k)

^{1}H NMR (400MHz, $CDCl_3$) δ ppm 8.24 (s, 1H), 7.29-7.25 (m, 1H), 7.08-6.90 (m, 3H), 6.83 (d, J = 7.6 Hz, 1H), 5.00 (s, 2H), 3.97 (s, 3H), 2.41 (3, 3H).

^{13}C NMR (100MHz, $CDCl_3$) δ ppm 156.5, 153.1, 134.8, 130.7, 129.2, 128.3, 123.4, 121.9, 118.9, 117.4, 116.5, 111.4, 69.0, 55.6, 21.3.

GC-MS: *m*/*z* (%) = 226 (100) $[M]^+$

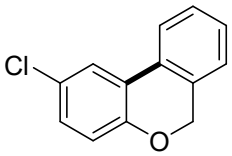
8-methoxy-2-methyl-6*H*-benzo[*c*]chromene (2k')

^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.65 (d, *J* = 8.4, 2.0 Hz, 1H), 7.50 (s, 1H), 7.03 (d, *J* = 8.4 Hz, 1H), 6.97-6.90 (m, 2H), 6.72 (d, *J* = 2.0 Hz, 1H), 5.08 (s, 2H), 3.86 (s, 3H), 2.39 (s, 3H).

^{13}C NMR (100MHz, $CDCl_3$) δ ppm 159.3, 151.8, 133.2, 131.4, 129.0, 123.1(×2), 122.7, 116.9, 113.9, 110.1, 68.6, 55.4, 20.9.

GC-MS: *m*/*z* (%) = 226 (100) $[M]^+$

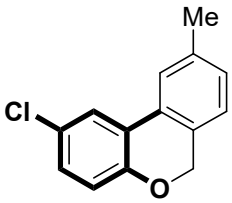
2-chloro-6*H*-benzo[*c*]chromene (2l)[1, 4]



Obtained in 72% yield (31.2 mg and 0.2 mmol scale) as a colorless oil.

^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.71(d, *J* = 2.4 Hz, 1H), 7.67 (d, *J* = 7.6 Hz, 1H), 7.41 (dd, *J* = 7,6, 7.6 Hz, 1H), 7.34 (ddd, *J* = 7.6, 7.6, 1.2 Hz, 1H), 7.20 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.18 (d, *J* = 6.8 Hz, 1H), 6.95 (d, *J* = 8.8 Hz, 1H), 5.14 (s, 2H).

2-chloro-9-methyl-6*H*-benzo[c]chromene (2m)



Obtained in 57% yield (26.2 mg and 0.2 mmol scale) as a white solid.

IR (film) 2923, 2853, 1612, 1488, 1200, 1019, 813 cm^{-1}

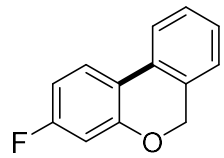
^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.70 (d, *J* = 2.4 Hz, 1H), 7.48 (s, 1H), 7.18 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.06 (d, *J* = 8.0 Hz, 1H), 6.93 (d, *J* = 8.8 Hz, 1H), 5.10 (s, 2H), 2.43 (s, 3H).

^{13}C NMR (100MHz, $CDCl_3$) δ ppm 153.3, 138.3, 129.1, 128.9, 128.9, 128.4, 127.1, 124.7, 124.5, 123.1, 122.8, 118.7, 68.4, 21.5.

GC-MS: *m/z* (%) = 229 (100), 230 (66) $[M]^+$, 232 (22) $[M+2]^+$.

HRMS (CI) calcd for $C_{14}H_{11}ClO$ 230.0498 $[M]^+$, found 230.0493.

3-fluoro-6*H*-benzo[*c*]chromene (2n)[4]



Obtained in 80% yield (32 mg and 0.2 mmol scale) as a white solid.

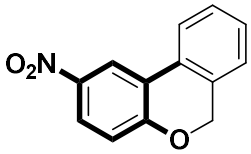
^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.70 (dd, *J* = 8.8, 6.4 Hz, 1H), 7.66 (d, *J* = 7.6 Hz, 1H), 7.40 (ddd, *J* = 7.6, 7.6, 1.2 Hz, 1H), 7.30 (ddd, *J* = 7.6, 7.6, 1.2 Hz, 1H), 7.17 (d, *J* = 7.6 Hz, 1H), 6.80 (ddd, *J* = 8.4, 8.4, 2.8 Hz, 1H), 6.74 (dd, *J* = 9.6, 2.8 Hz, 1H),

5.15 (s, 1H).

^{13}C NMR (100MHz, $CDCl_3$) δ ppm 163.3 (d, *J* = 245.9 Hz), 156.0 (d, *J* = 11.9 Hz), 130.5, 129.5, 128.6, 127.6, 124.7, 124.5 (d, *J* = 10.7 Hz), 121.7, 119.3 (d, *J* = 3.4 Hz, 1H), 109.3 (d, *J* = 21.3 Hz), 104.9 (d, *J* = 24 Hz), 68.7.

GC-MS: *m*/*z* (%) = 199 (38), 200 (21) $[M]^+$.

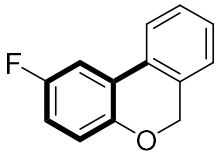
2-nitro-6*H*-benzo[*c*]chromene (2o)[4]



Obtained in 29% yield (13.2 mg and 0.2 mmol scale) as a pale yellow solid.

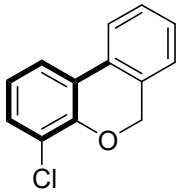
^{1}H NMR (400MHz, $CDCl_3$) δ ppm 8.63 (d, *J* = 2.8 Hz, 1H), 8.12 (dd, *J* = 8.8, 2.8 Hz, 1H), 7.78 (d, *J* = 7.6 Hz, 1H), 7.45 (dd, *J* = 7.6, 7.6 Hz, 1H), 7.38 (ddd, *J* = 7.6, 7.6, 1.2 Hz, 1H), 7.18 (d, *J* = 7.2 Hz, 1H), 7.05 (d, *J* = 8.8 Hz, 1H), 5.26 (s, 2H).

2-fluoro-6*H*-benzo[*c*]chromene (2p)[1]



^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.65 (d, *J* = 7.6 Hz, 1H), 7.44-7.39 (m, 3H), 7.19 (dd, *J* = 7.6, 0.8 Hz, 1H), 6.96-6.94 (m, 2H), 5.12 (s, 2H).

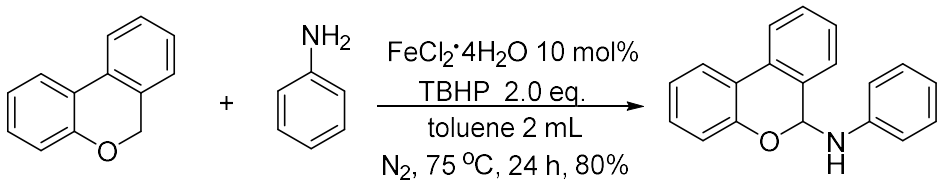
4-chloro-6*H*-benzo[*c*]chromene (2q)[6]



^{1}H NMR (400MHz, $CDCl_3$) δ ppm 7.70 (d, *J* = 7.6 Hz, 1H), 7.66 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.43-7.39 (m, 1H), 7.36-7.31 (m, 2H), 7.20 (d, *J* = 8.0 Hz, 1H), 7.01 (dd, *J* = 8.0, 8.0 Hz, 1H), 5.24 (s, 2H).

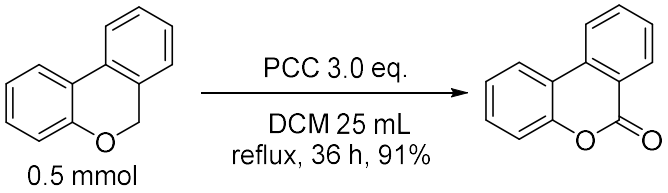
3. Post transformations of the obtained 6*H*-benzo[*c*]chromene

3.1 transformed into *N*-phenylamino-6*H*-benzo[*c*]chromene[7]



6*H*-benzo[*c*]chromene (0.5 mmol) and $FeCl_2 \cdot 4H_2O$ (0.005 mmol) were placed in a10 mL Schlenk tube. Toluene (2 mL) and *t*-BuOOH(1.0 mmol) were added. The tube was sealed and flushed with nitrogen, and the mixture was stirred at 75 °C. Phenylamine (0.5 mmol) was slowly added drop-wise into the system. The mixture was stirred for 24 h, then cooled to room temperature, poured into water (20 mL), and extracted with EtOAc (2×20 mL). Next the organic phase was and extracted with brine, dried with Na_2SO_4, evaporated under vacuum, and the crude product was purified by column chromatography (EA/PE=1:20).

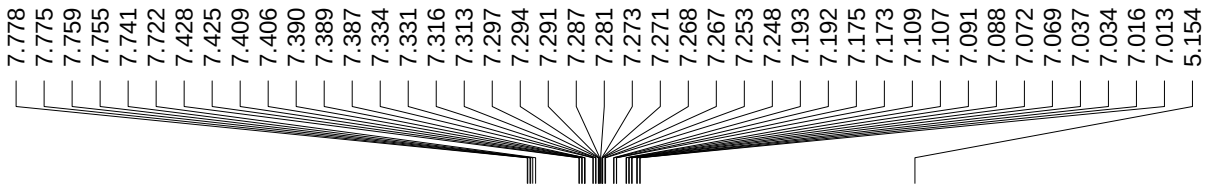
3.2 transformed into synthetic procedure for 2-oxo-6*H*-benzo[c]chromene[1]



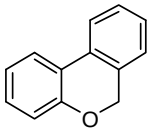
6*H*-benzo[*c*]chromene (0.5 mmol) was placed in a 100 mL round-bottom flask and dissolved with DCM (25 mL). PCC (0.5 mmol) was added and the mixture stirred under reflux for 12 h. Then PCC (1.0 mmol) was added into the mixture and continued to stir for a further 24 h. After cooling to room temperature, water (15 mL) and diethyl ether (30 mL) were added. The mixture was filtered to get the filtrate, and extracted with brine, dried with Na_2SO_4, evaporated under vacuum, then the crude product was purified by column chromatography (EA/PE=1:8).

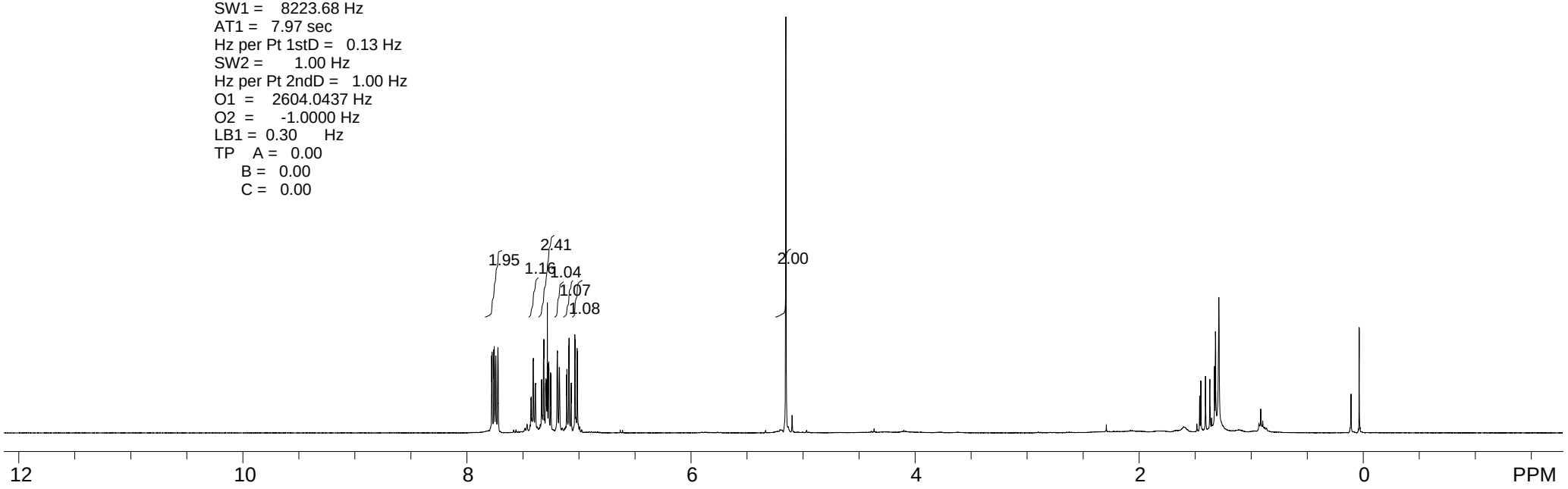
References:

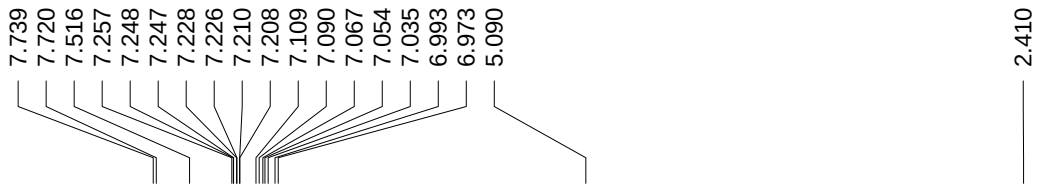
1. Sun, C.-L.; Gu, Y.-F.; Huang, W.-P.; Shi, Z.-J. *Chem. Commun*. **2011**, *47*, 9813.
2. Zhou, J.; Huang, L.; Li, Y.-Q.; Du, Z.-T. *Tetrahedron Lett.*, **2012**, 53, 7036.
3. Parisien, M.; Valette, D.; Fagnou, K. *J. Org. Chem*., **2005**, *70*, 7578.
4. He, Yan, Zhang, X.; Cui, L.; Wang, J. Fan, X. *Green Chem.*, **2012**, *14*, 3429.
5. Canoa, R.; Pérezb, J. M.; Ramónb, D. J.; MaGlackena, G. P. *Tetrahedron*, **2016**, 72, 1043.
6. Read, M. L.; Guandersen, L.-L. *J. Org. Chem*. **2013**, *78*, 1311.
7. Chen, D.; Pan, F.; Gao, J.; Yang, J. *Synlett*, **2013**, *24*, 2085.

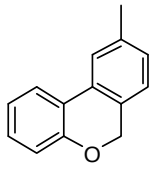


USER: BQF-28
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.150 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00









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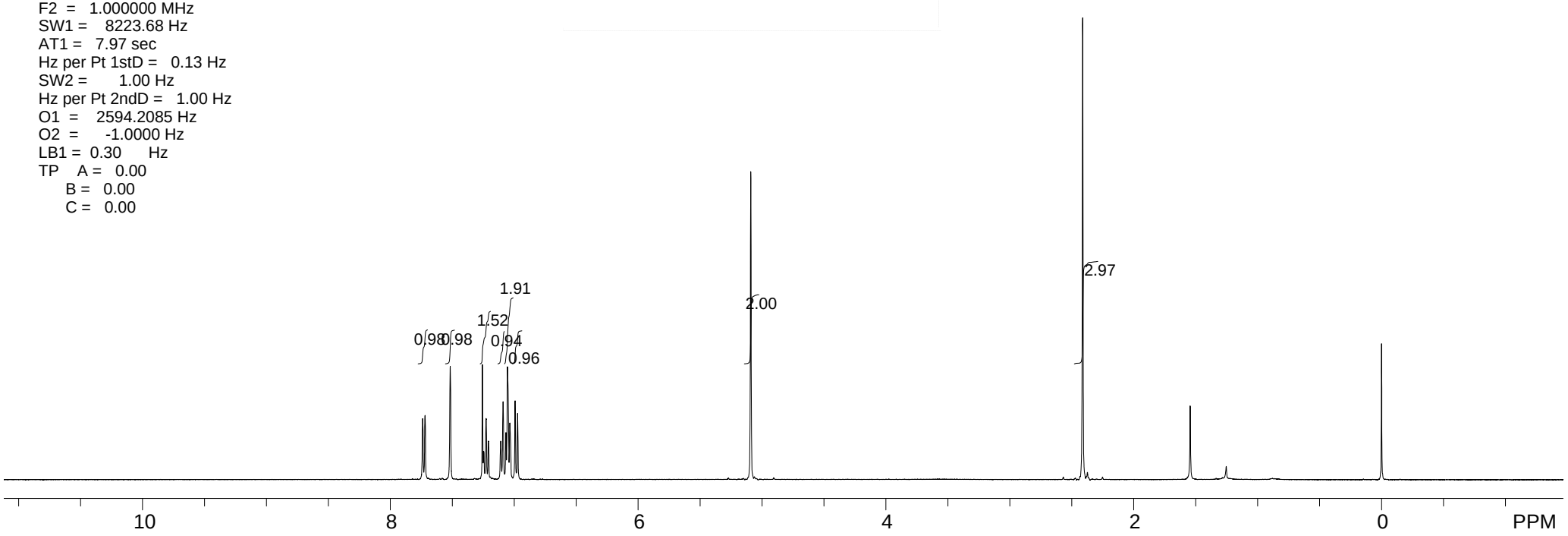
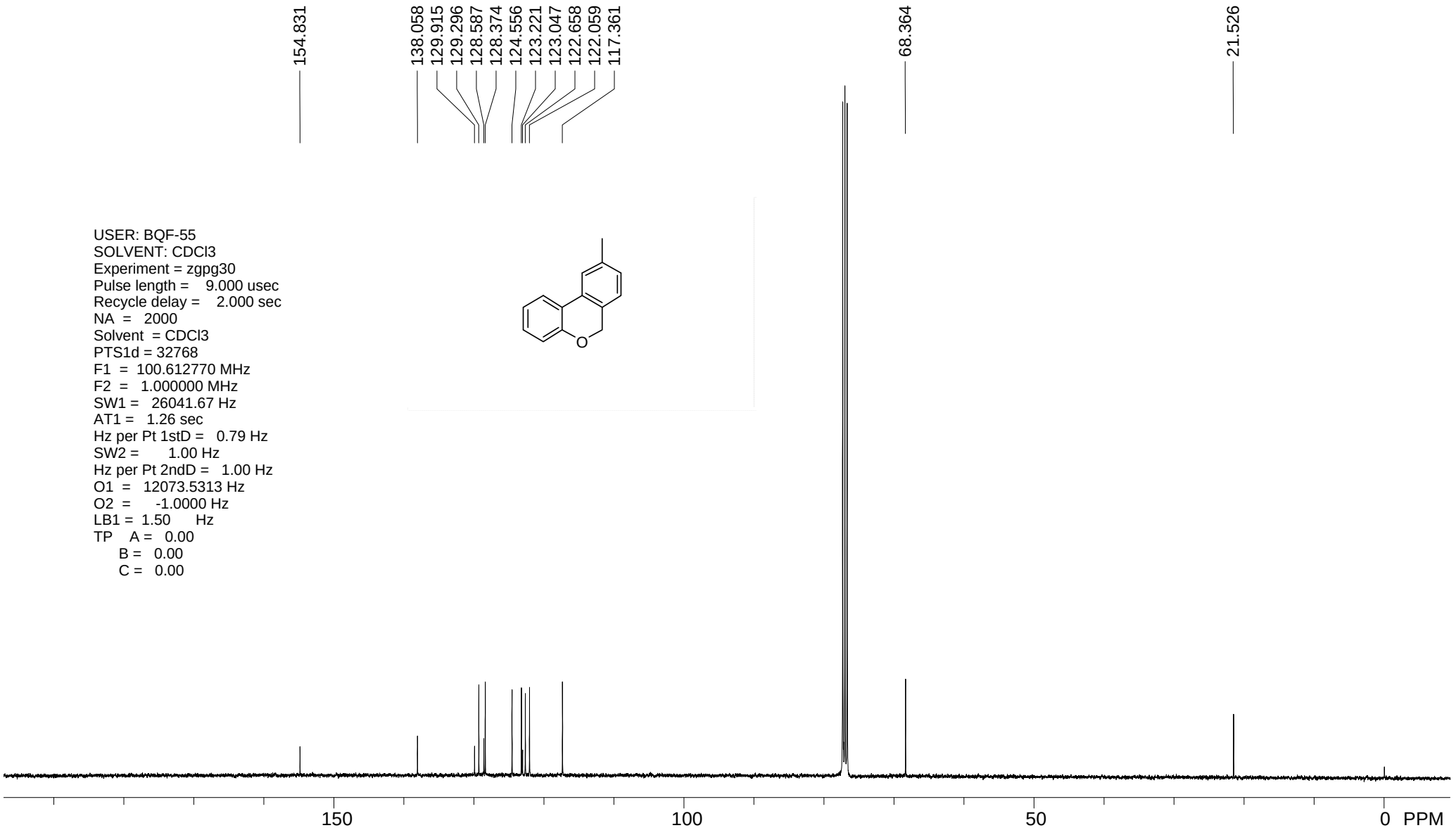
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2594.2085 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00




154.831
138.058
129.915
129.296
128.587
128.374
124.556
123.221
123.047
122.658
122.059
117.361
68.364
21.526
O
USER: BQF-55
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.000 usec
Recycle delay = 2.000 sec
NA = 2000
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 26041.67 Hz
AT1 = 1.26 sec
Hz per Pt 1stD = 0.79 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 12073.5313 Hz
O2 = -1.0000 Hz
LB1 = 1.50 Hz
TP A = 0.00
B = 0.00
C = 0.00
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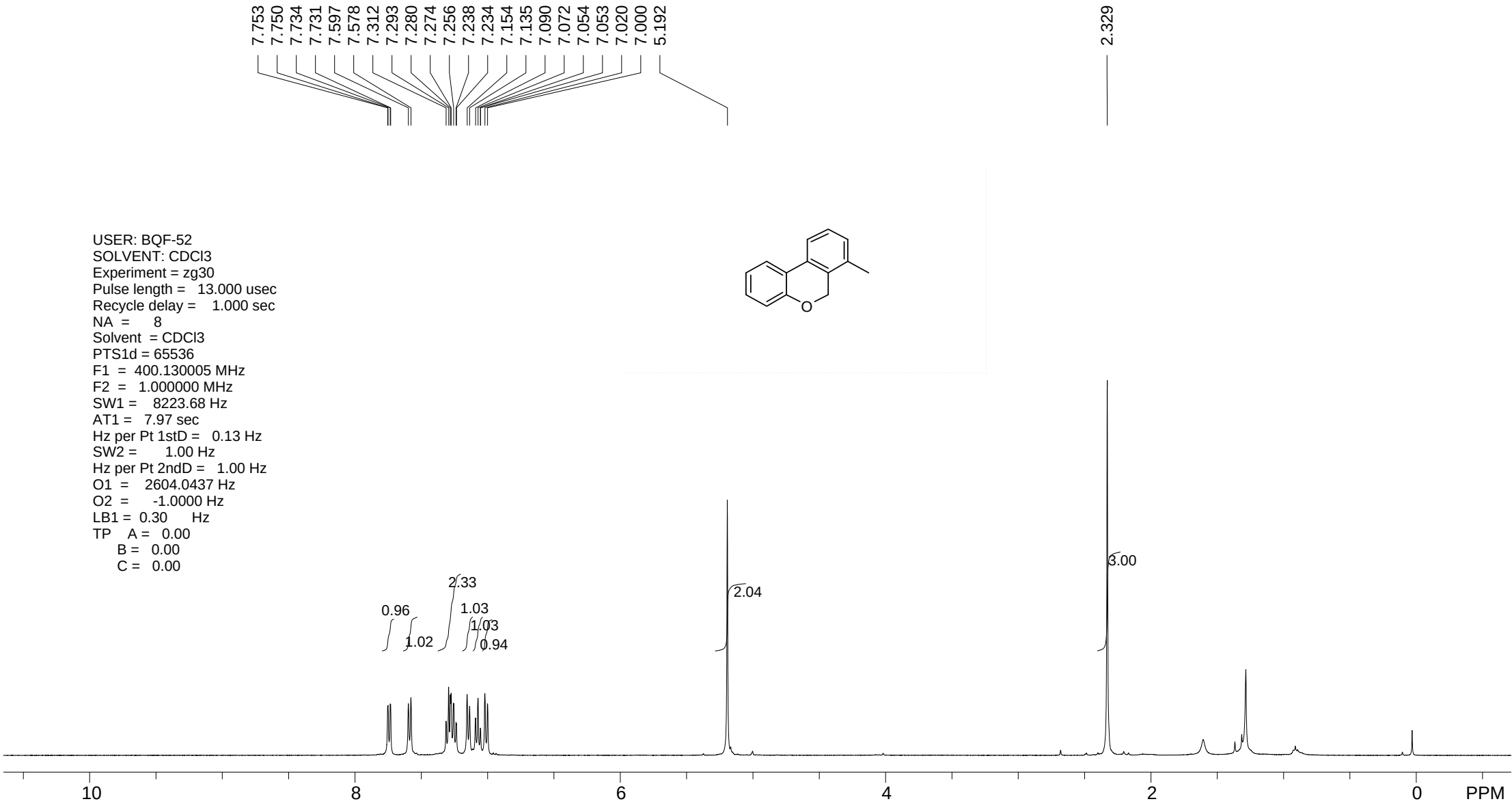
100
50
0 PPM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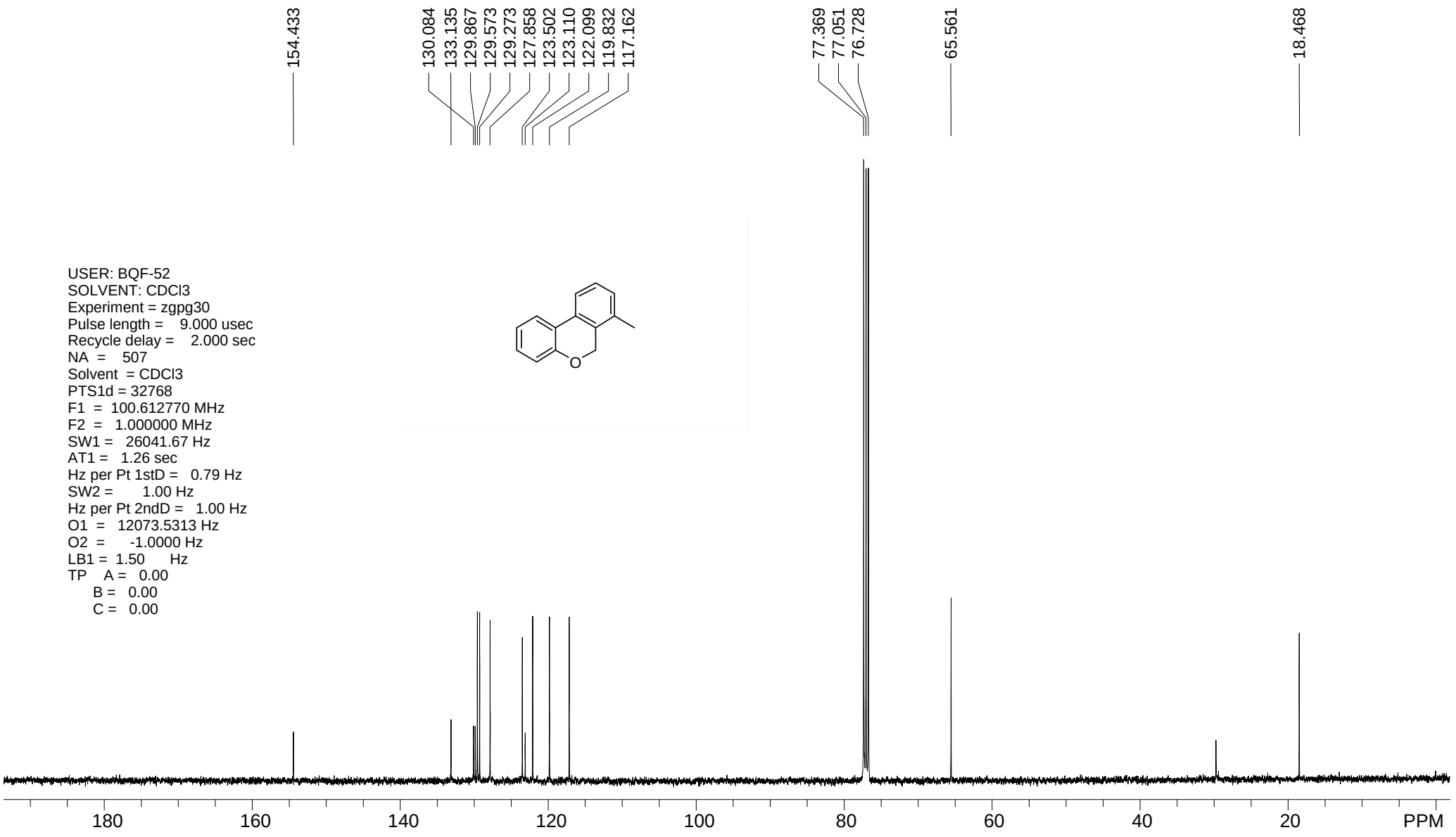
7.734
7.731
7.597
7.578
7.312
7.293
7.280
7.274
7.256
7.238
7.234
7.154
7.135
7.090
7.072
7.054
7.053
7.020
7.000
5.192
2.329
USER: BQF-52
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00
O
0.96
1.02
2.33
1.03
1.03
0.94
2.04
3.00
10
8
6
4
2
0
PPM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129.573
129.273
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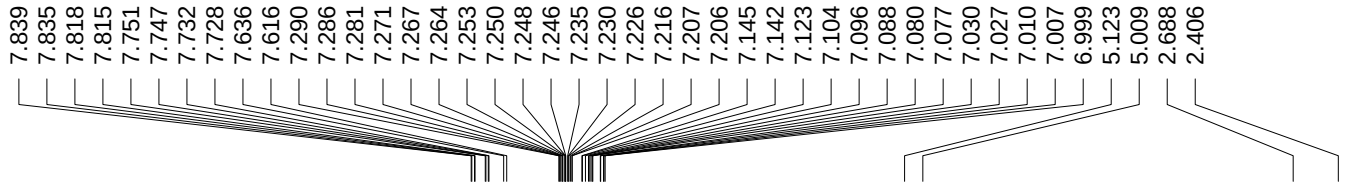
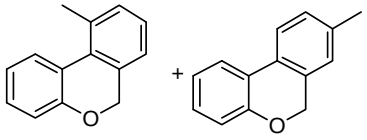
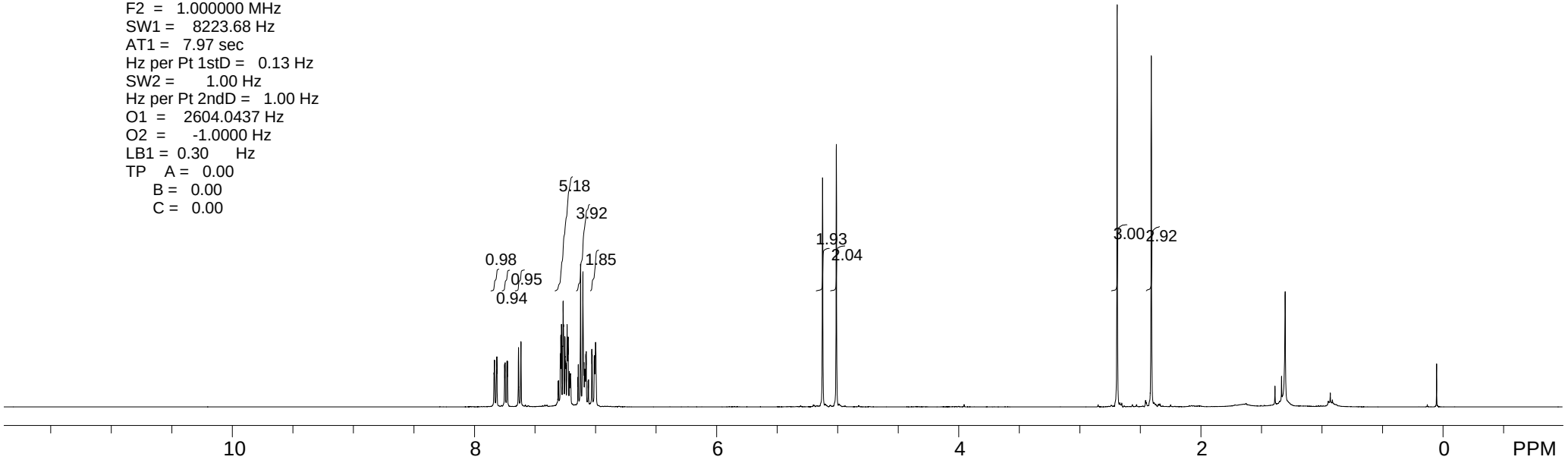
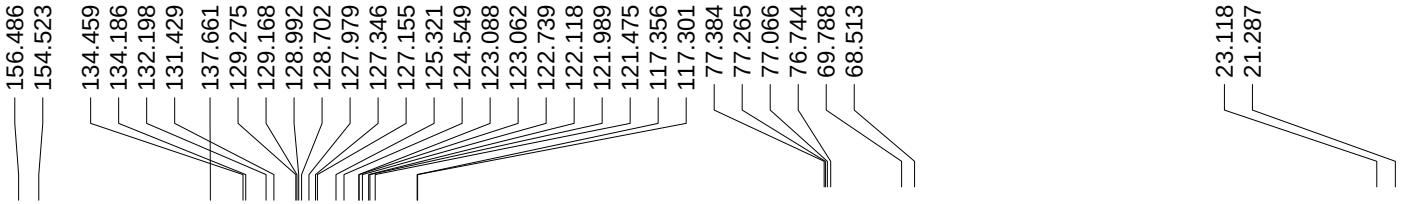
123.110
122.099
119.832
117.162
77.369
77.051
76.728
65.561
18.468
O
USER: BQF-52
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.000 usec
Recycle delay = 2.000 sec
NA = 507
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 26041.67 Hz
AT1 = 1.26 sec
Hz per Pt 1stD = 0.79 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 12073.5313 Hz
O2 = -1.0000 Hz
LB1 = 1.50 Hz
TP A = 0.00
B = 0.00
C = 0.00
180
160
140
120
100
80
60
40
20
PPM



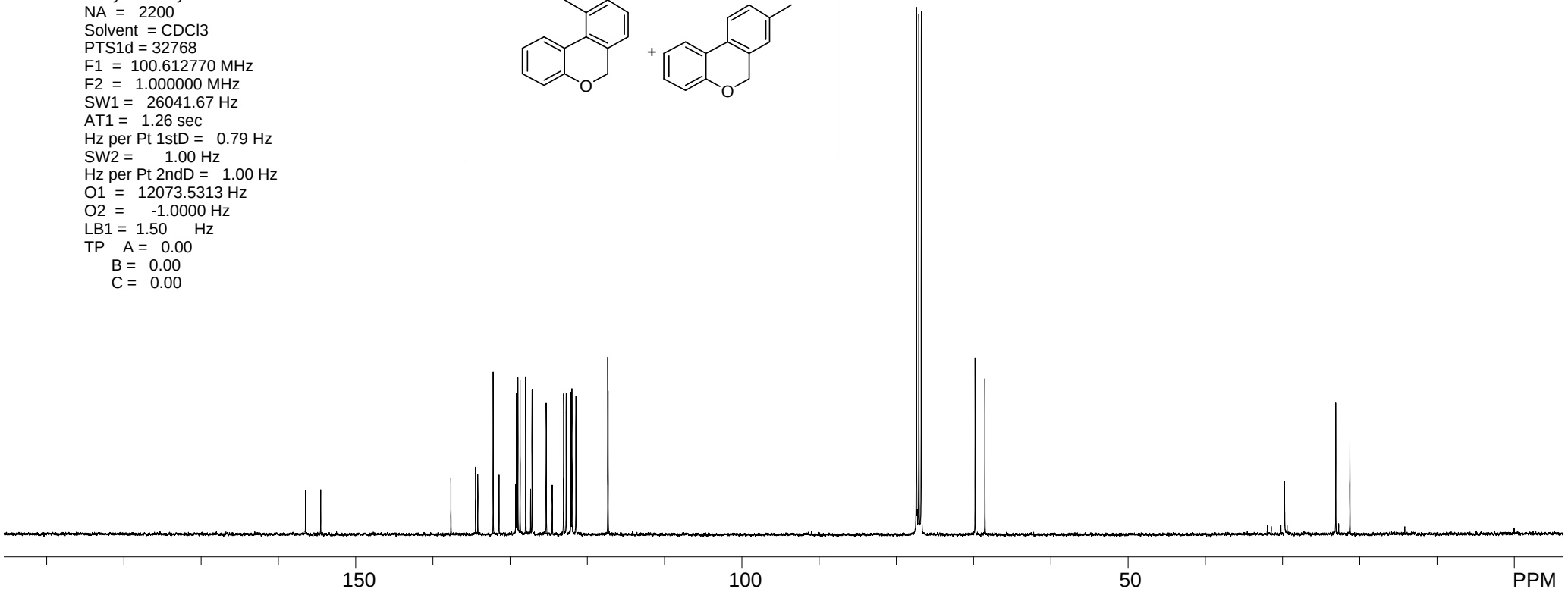


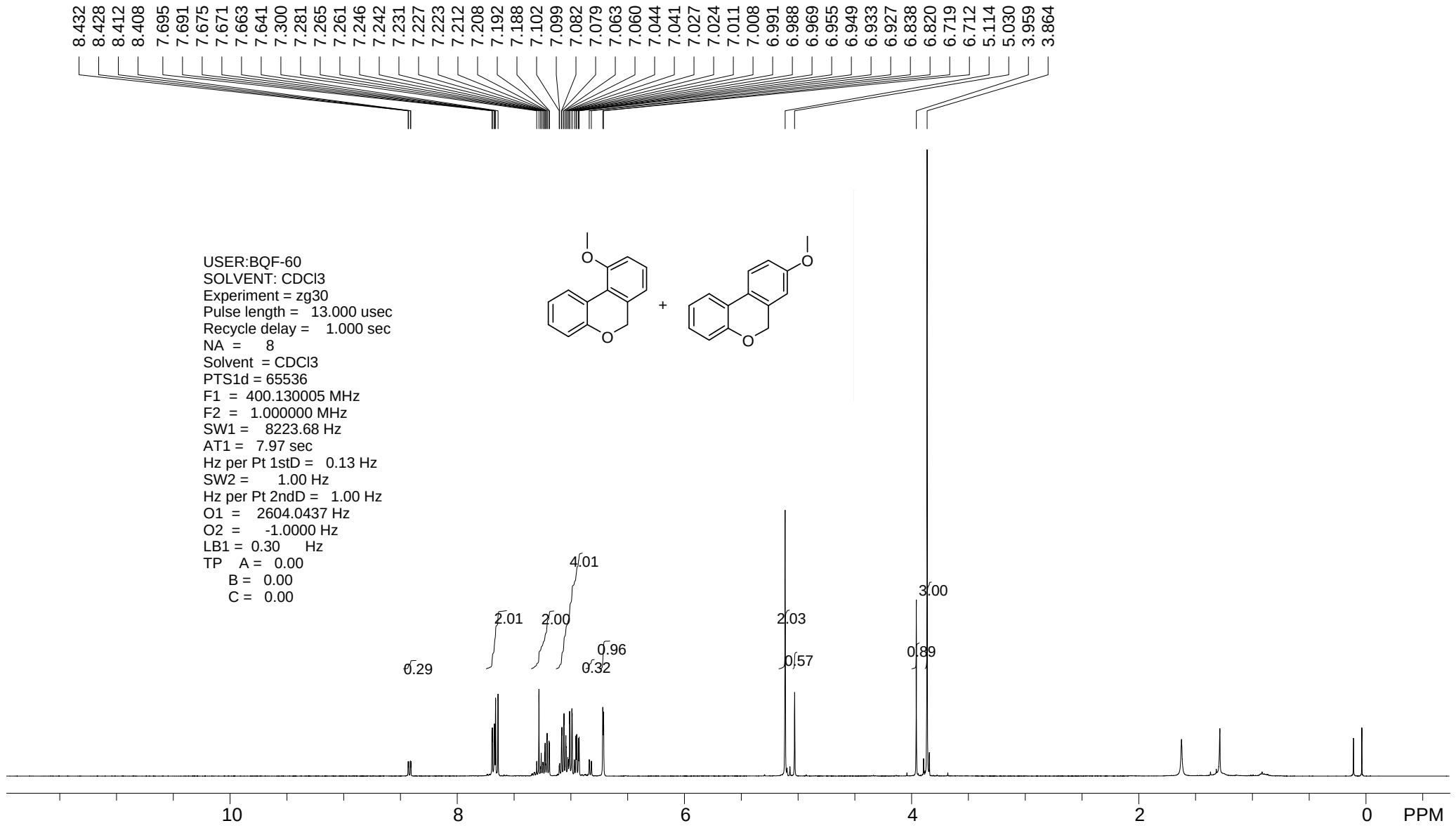
USER: BQF-51
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00





USER:BQF-51
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.000 usec
Recycle delay = 2.000 sec
NA = 2200
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 26041.67 Hz
AT1 = 1.26 sec
Hz per Pt 1stD = 0.79 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 12073.5313 Hz
O2 = -1.0000 Hz
LB1 = 1.50 Hz
TP A = 0.00
B = 0.00
C = 0.00




8.432
8.428
8.412
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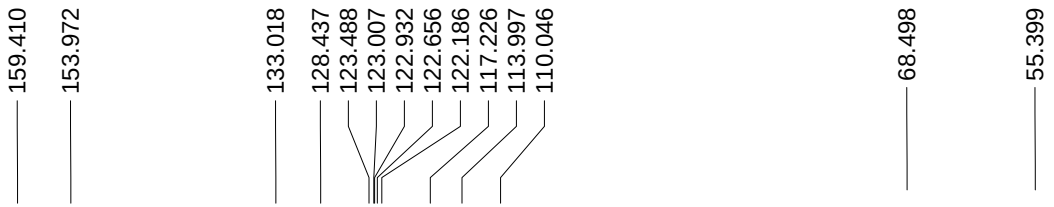
7.695
7.691
7.675
7.671
7.663
7.641
7.300
7.281
7.265
7.261
7.246
7.242
7.231
7.227
7.223
7.212
7.208
7.192
7.188
7.102
7.099
7.082
7.079
7.063
7.060
7.044
7.041
7.027
7.024
7.011
7.008
6.991
6.988
6.969
6.955
6.949
6.933
6.927
6.838
6.820
6.719
6.712
5.114
5.030
3.959
3.864
USER:BQF-60
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00
O
O
+
O
O
4.01
3.00
2.01
2.00
2.03
0.96
0.89
0.29
0.32
0.57
10
8
6
4
2
0
PPM



USER: BQF-60
SOLVENT: CDCl3
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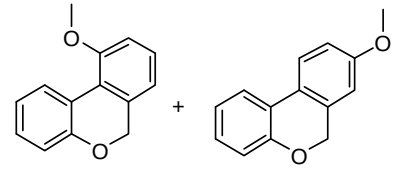
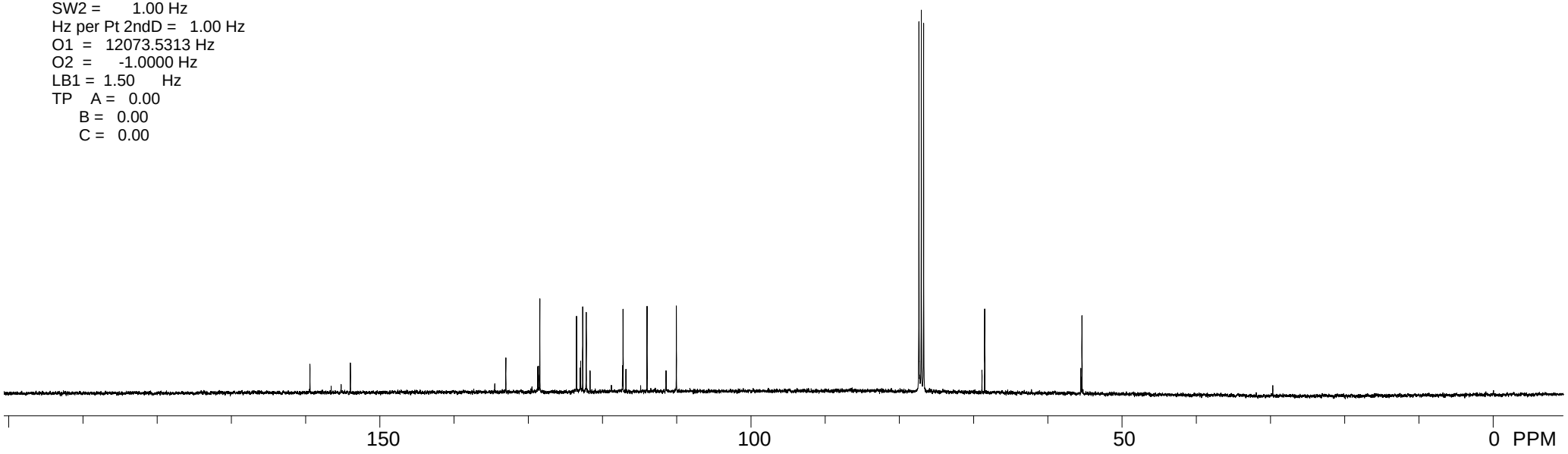
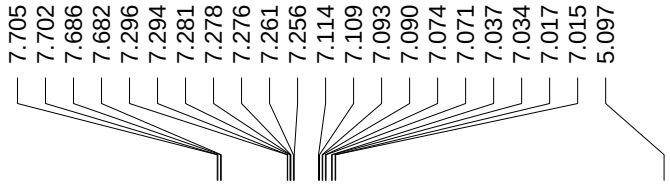
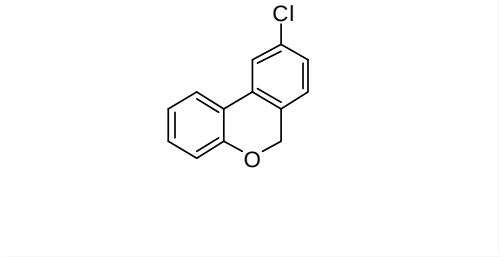
Recycle delay = 2.000 sec
NA = 603
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 26041.67 Hz
AT1 = 1.26 sec
Hz per Pt 1stD = 0.79 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 12073.5313 Hz
O2 = -1.0000 Hz
LB1 = 1.50 Hz
TP A = 0.00
B = 0.00
C = 0.00









USER:BQF-58
SOLVENT: CDCl3
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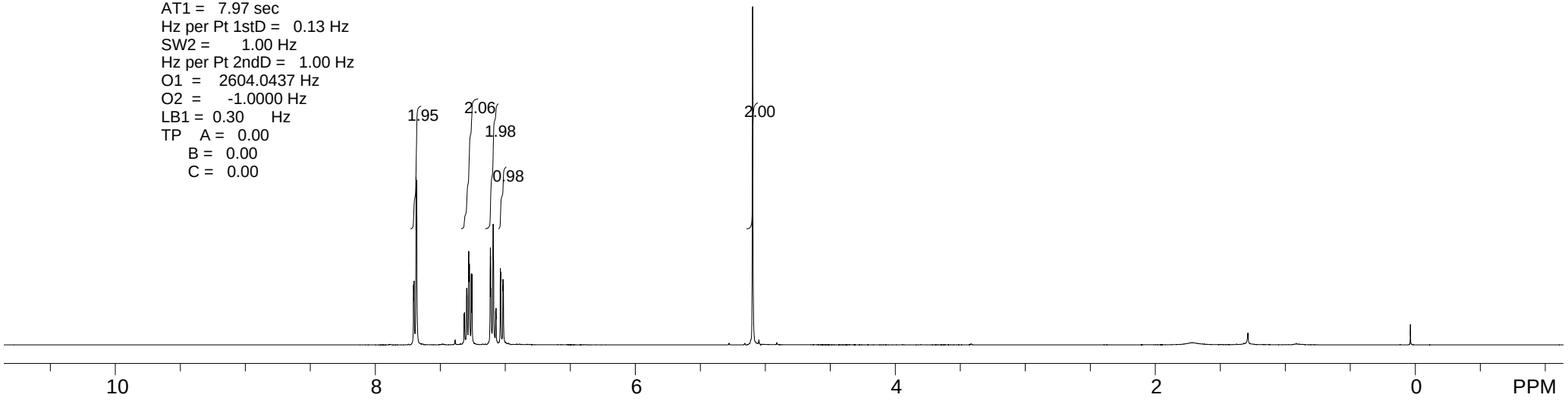
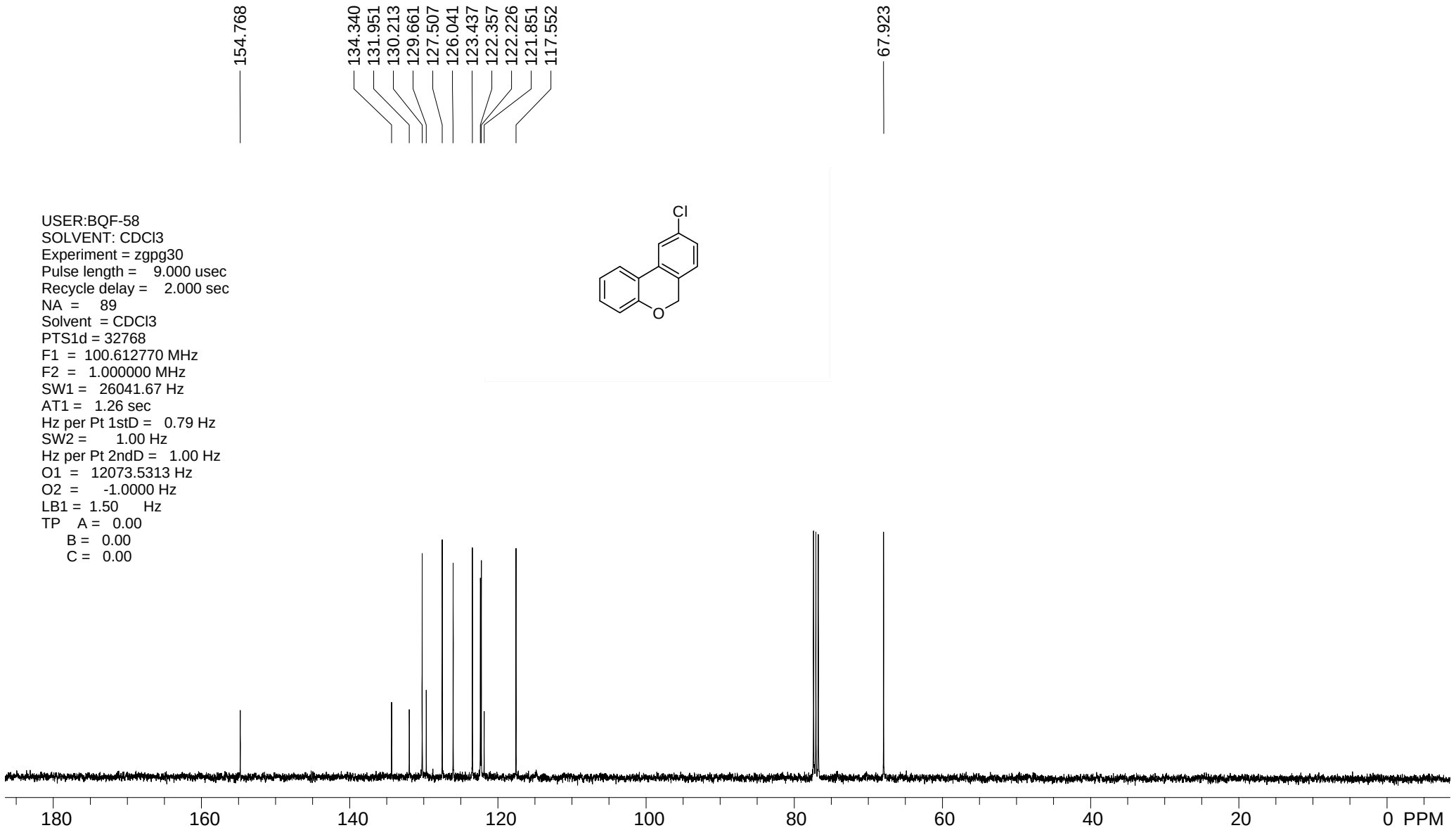
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00

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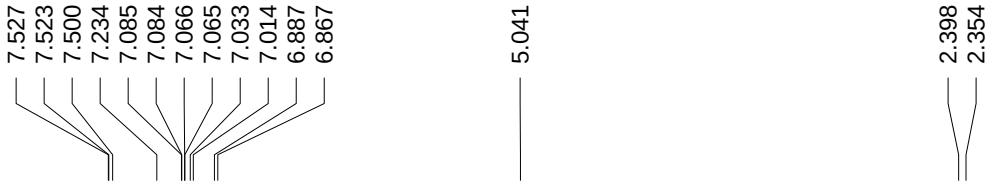
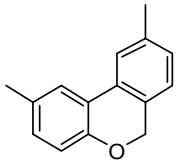
127.507
126.041
123.437
122.357
122.226
121.851
117.552
67.923
USER:BQF-58
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.000 usec
Recycle delay = 2.000 sec
NA = 89
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 26041.67 Hz
AT1 = 1.26 sec
Hz per Pt 1stD = 0.79 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 12073.5313 Hz
O2 = -1.0000 Hz
LB1 = 1.50 Hz
TP A = 0.00
B = 0.00
C = 0.00
Cl
O
180
160
140
120
100
80
60
40
20
0 PPM





USER: BQF-34
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.150 usec
Recycle delay = 1.000 sec
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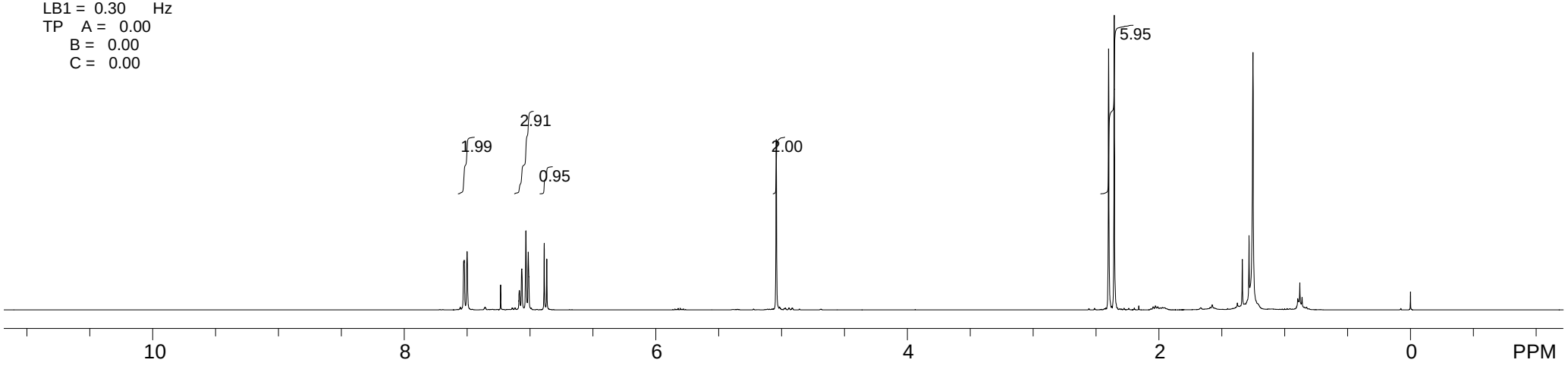
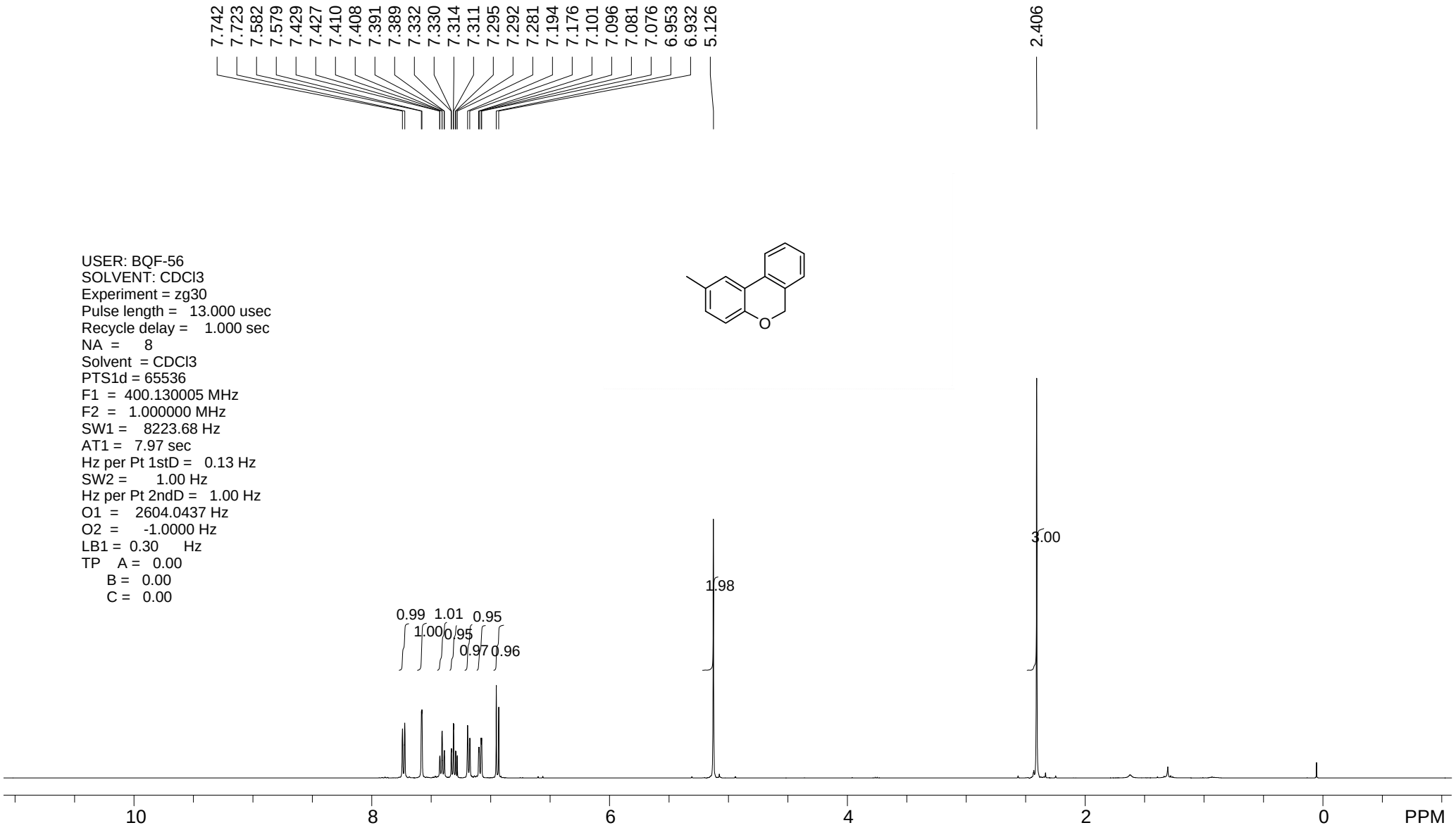
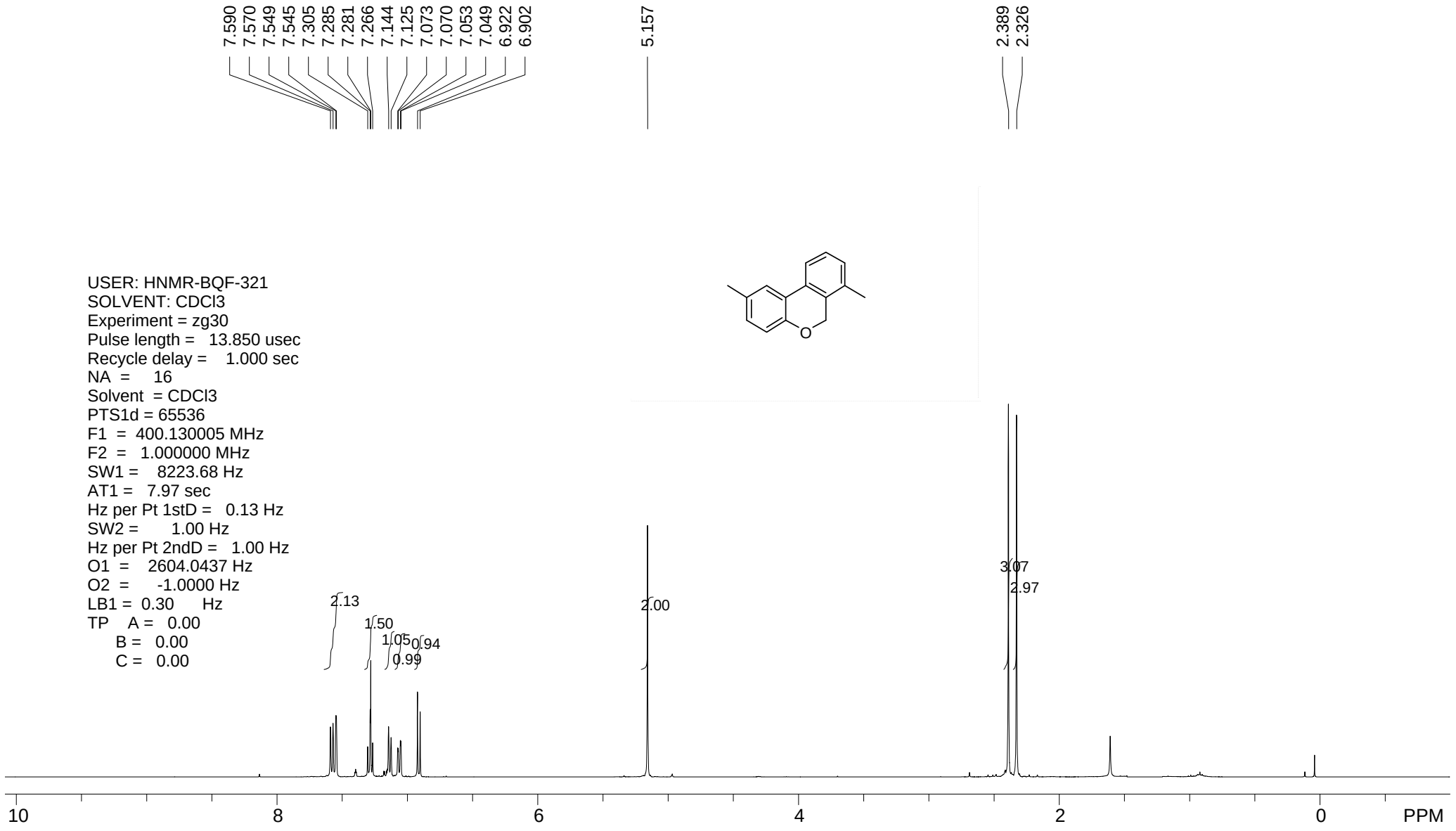
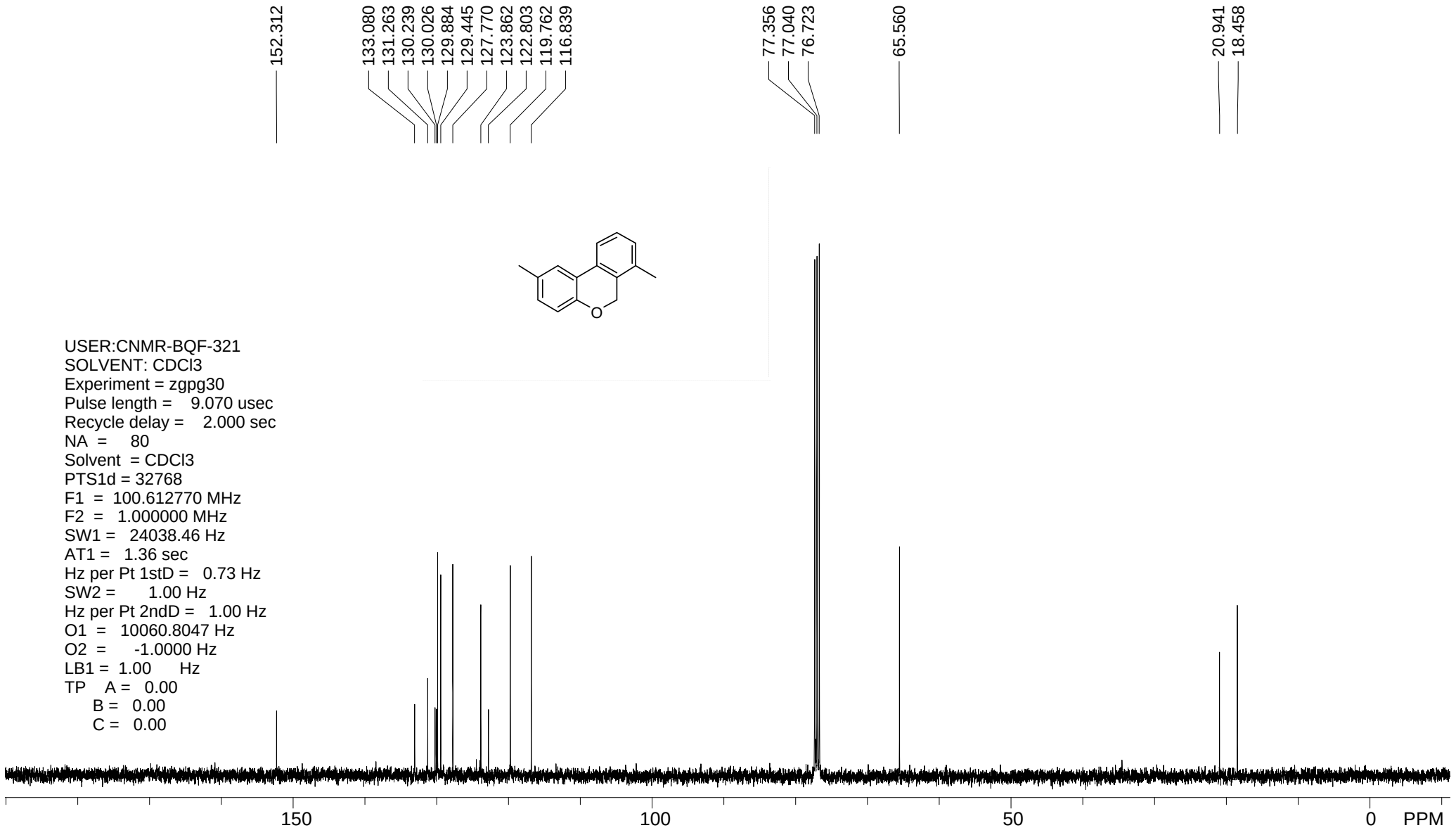
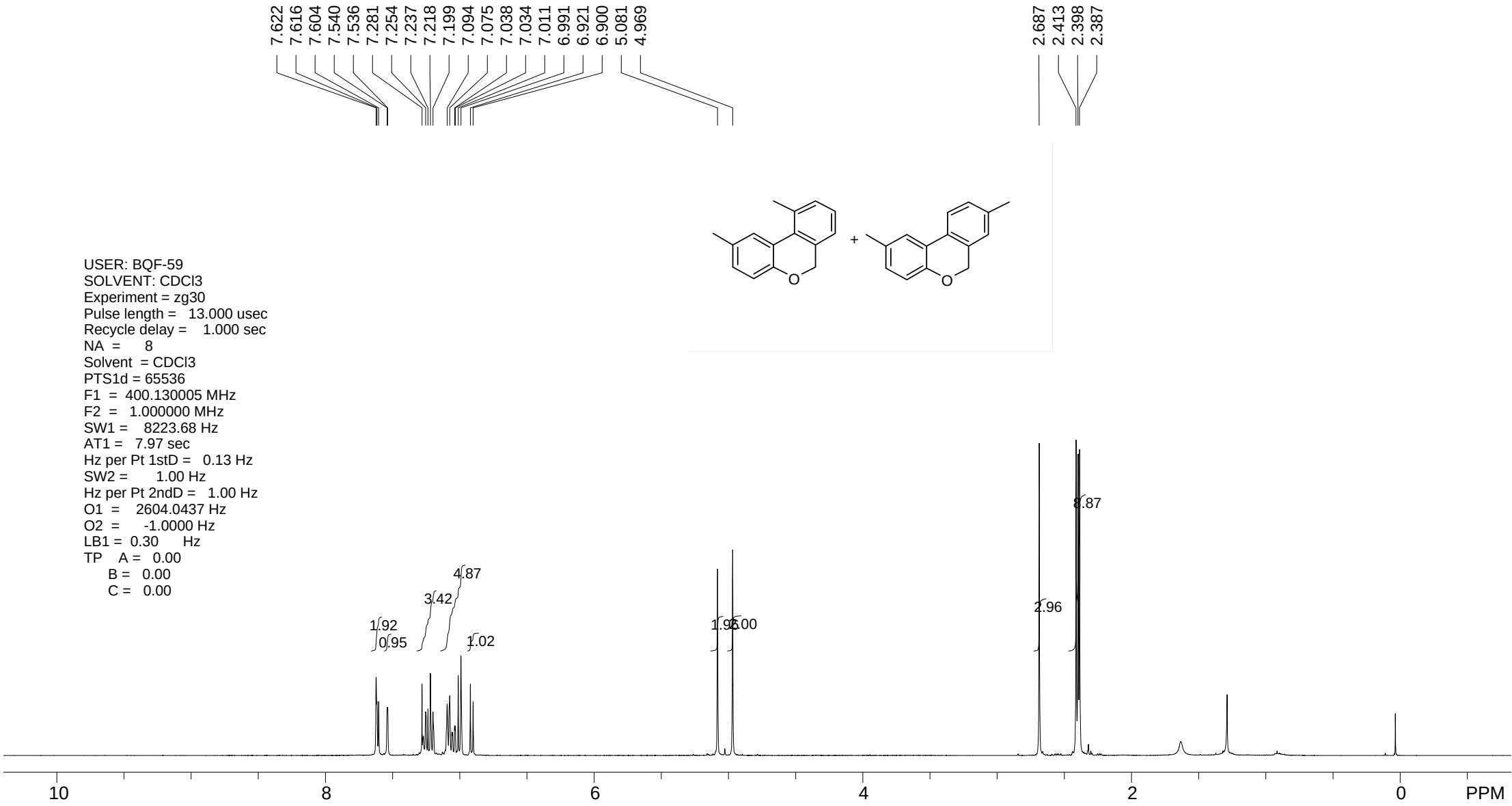
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2584.8296 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00

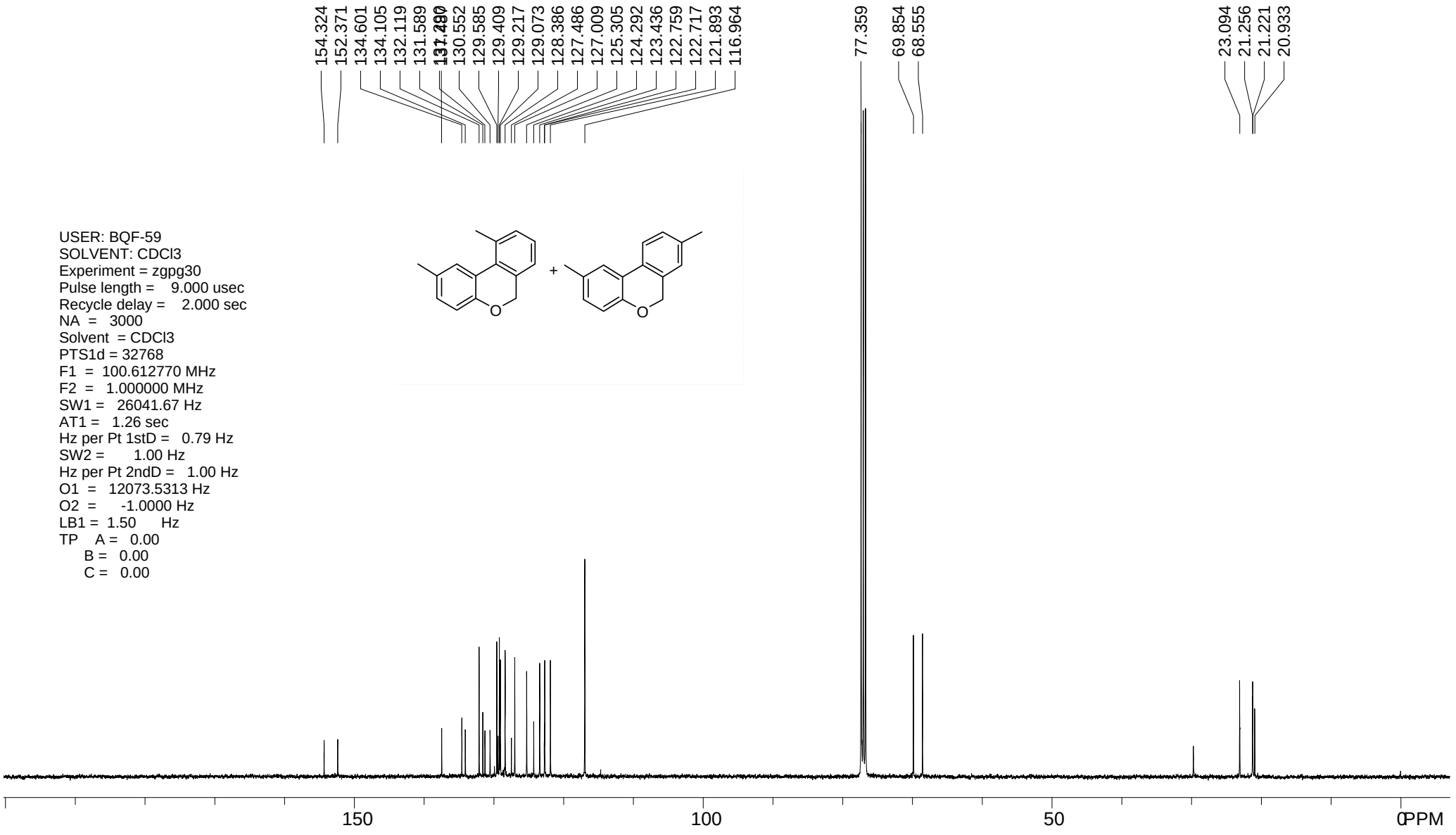


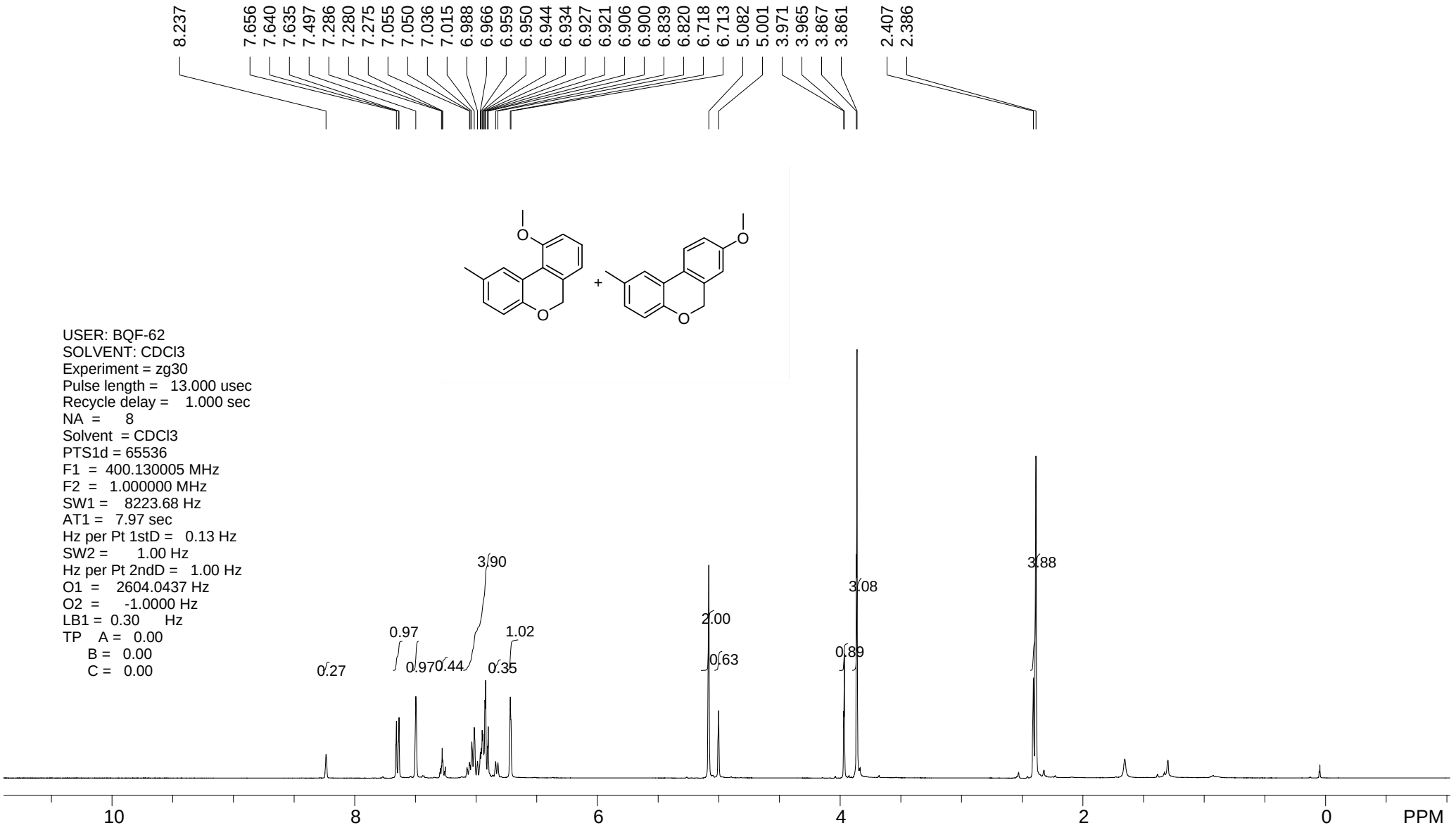

7.742
7.723
7.582
7.579
7.429
7.427
7.410
7.408
7.391
7.389
7.332
7.330
7.314
7.311
7.295
7.292
7.281
7.194
7.176
7.101
7.096
7.081
7.076
6.953
6.932
5.126
2.406
USER: BQF-56
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00
O
0.99
1.00
1.01
0.95
0.95
0.97
0.96
1.98
3.00
10
8
6
4
2
0
PPM

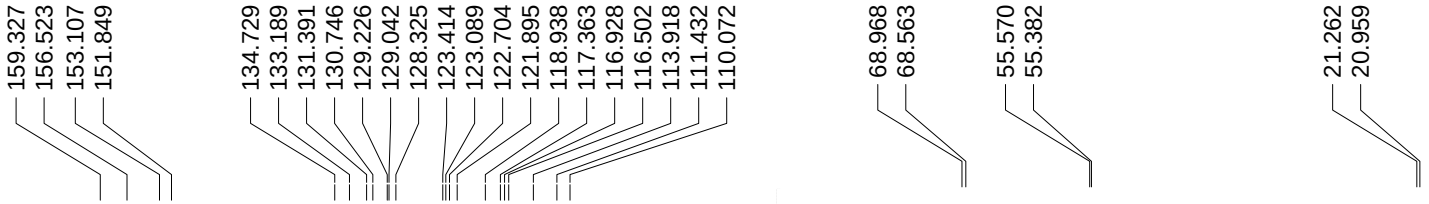

7.590
7.570
7.549
7.545
7.305
7.285
7.281
7.266
7.144
7.125
7.073
7.070
7.053
7.049
6.922
6.902
5.157
2.389
2.326
USER: HNMR-BQF-321
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.850 usec
Recycle delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00
O
2.13
1.50
1.05
0.94
0.99
2.00
3.07
2.97
10
8
6
4
2
0
PPM

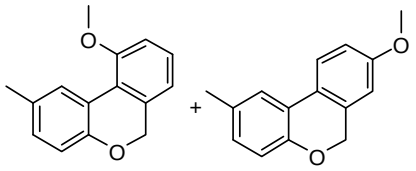

152.312
133.080
131.263
130.239
130.026
129.884
129.445
127.770
123.862
122.803
119.762
116.839
77.356
77.040
76.723
65.560
20.941
18.458
O
USER:CNMR-BQF-321
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.070 usec
Recycle delay = 2.000 sec
NA = 80
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 24038.46 Hz
AT1 = 1.36 sec
Hz per Pt 1stD = 0.73 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 10060.8047 Hz
O2 = -1.0000 Hz
LB1 = 1.00 Hz
TP A = 0.00
B = 0.00
C = 0.00
150
100
50
0
PPM


7.622
7.616
7.604
7.540
7.536
7.281
7.254
7.237
7.218
7.199
7.094
7.075
7.038
7.034
7.011
6.991
6.921
6.900
5.081
4.969
2.687
2.413
2.398
2.387
O
+
O
USER: BQF-59
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00
1.92
0.95
3.42
4.87
1.02
1.95
3.00
2.96
8.87
10
8
6
4
2
0
PPM

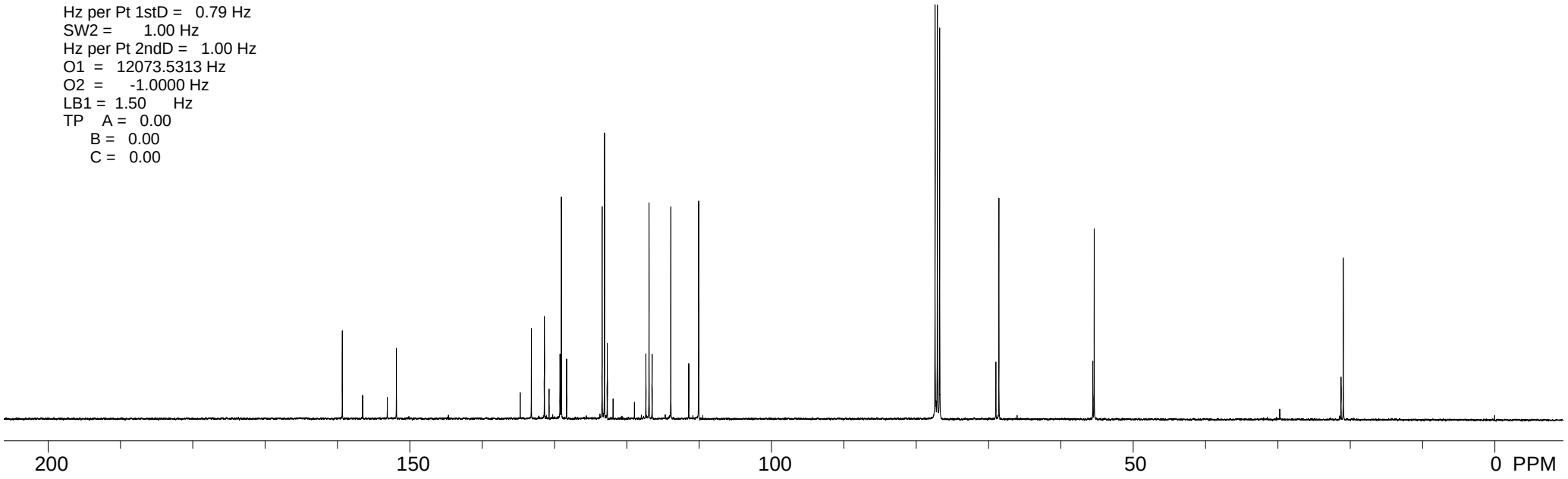

154.324
152.371
134.601
134.105
132.119
131.589
130.552
129.585
129.409
129.217
129.073
128.386
127.486
127.009
125.305
124.292
123.436
122.759
122.717
121.893
116.964
77.359
69.854
68.555
23.094
21.256
21.221
20.933
O
O
USER: BQF-59
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.000 usec
Recycle delay = 2.000 sec
NA = 3000
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 26041.67 Hz
AT1 = 1.26 sec
Hz per Pt 1stD = 0.79 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 12073.5313 Hz
O2 = -1.0000 Hz
LB1 = 1.50 Hz
TP A = 0.00
B = 0.00
C = 0.00
150
100
50
0
PPM

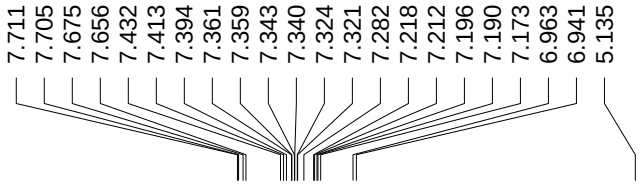

8.237
7.656
7.640
7.635
7.497
7.286
7.280
7.275
7.055
7.050
7.036
7.015
6.988
6.966
6.959
6.950
6.944
6.934
6.927
6.921
6.906
6.900
6.839
6.820
6.718
6.713
5.082
5.001
3.971
3.965
3.867
3.861
2.407
2.386
O
O
+
O
O
USER: BQF-62
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00
3.90
0.97
1.02
0.27
0.97
0.44
0.35
2.00
0.63
3.08
0.89
3.88
10
8
6
4
2
0
PPM



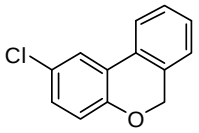


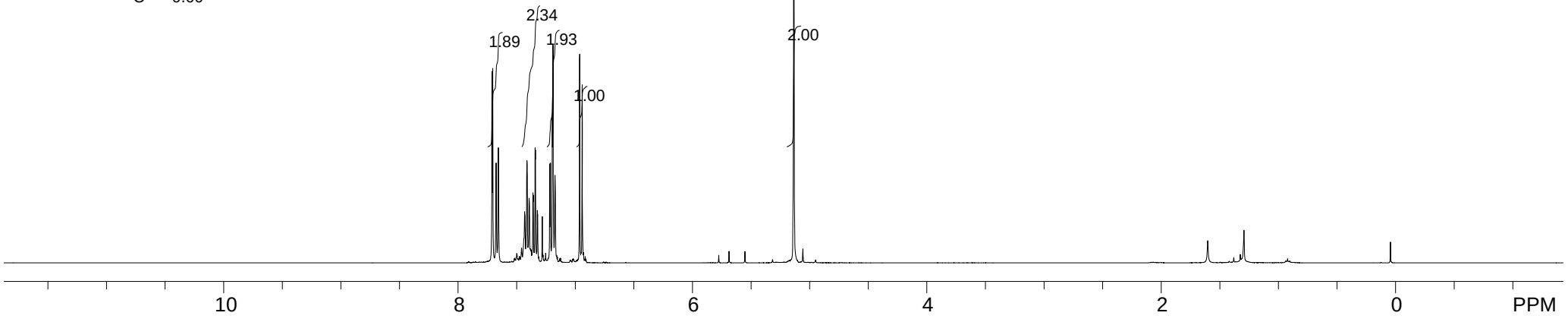
USER: BQF-62
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.000 usec
Recycle delay = 2.000 sec
NA = 3000
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 26041.67 Hz
AT1 = 1.26 sec
Hz per Pt 1stD = 0.79 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 12073.5313 Hz
O2 = -1.0000 Hz
LB1 = 1.50 Hz
TP A = 0.00
B = 0.00
C = 0.00

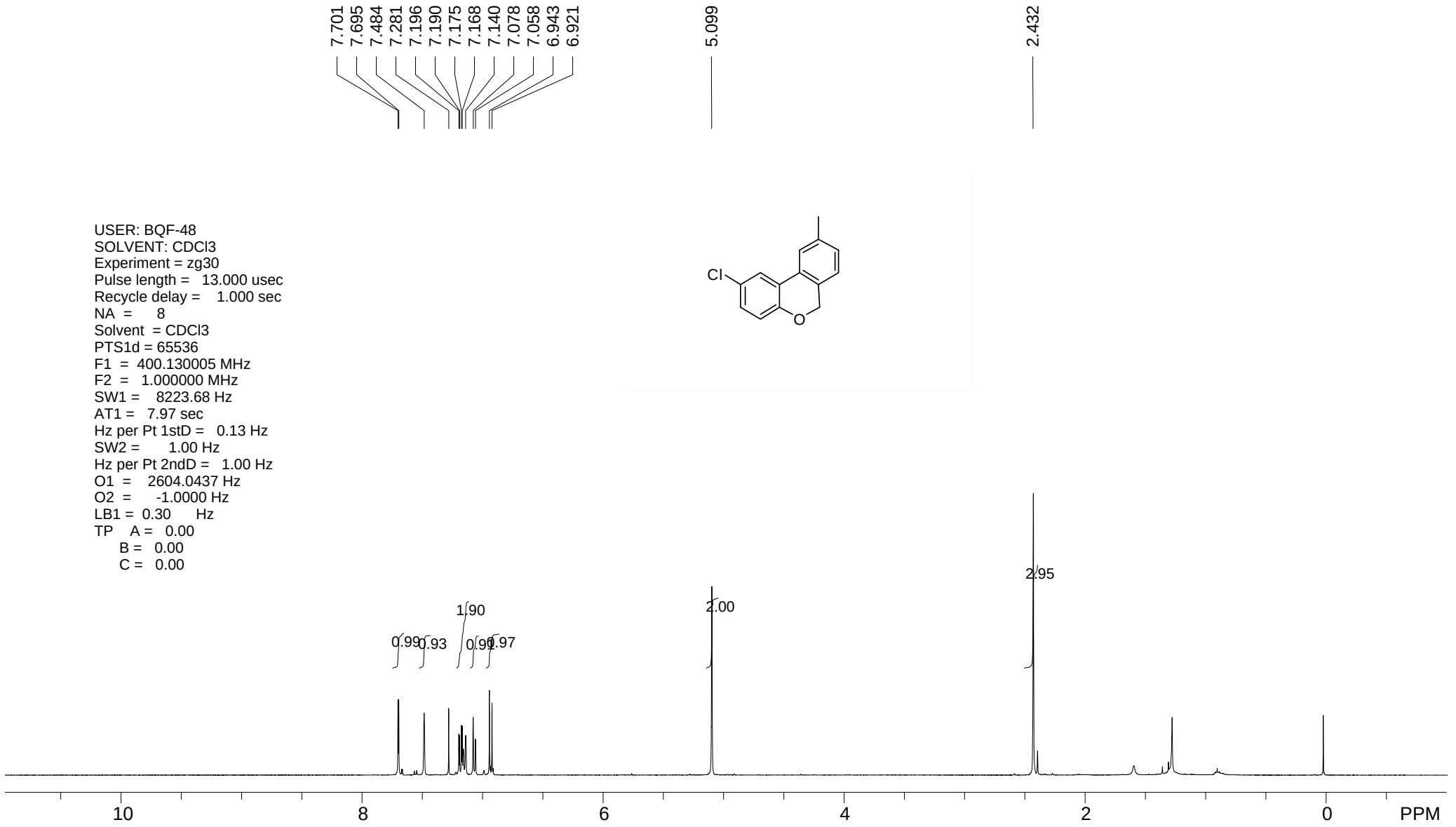


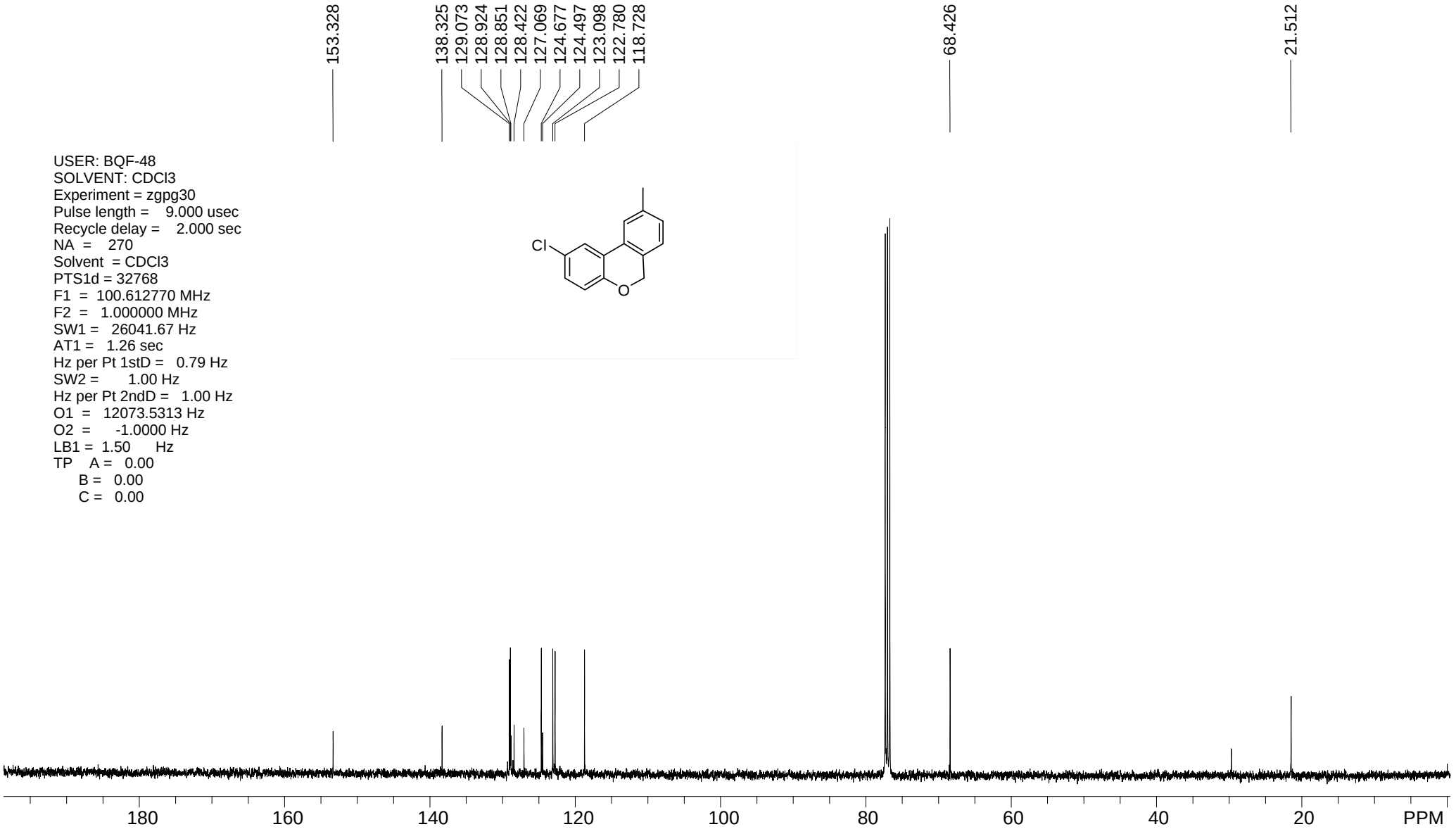


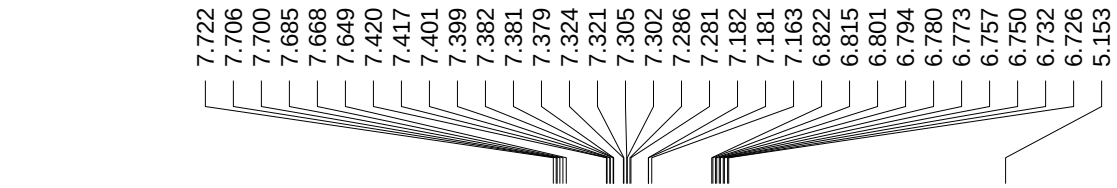
USER: BQF-32
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.150 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00



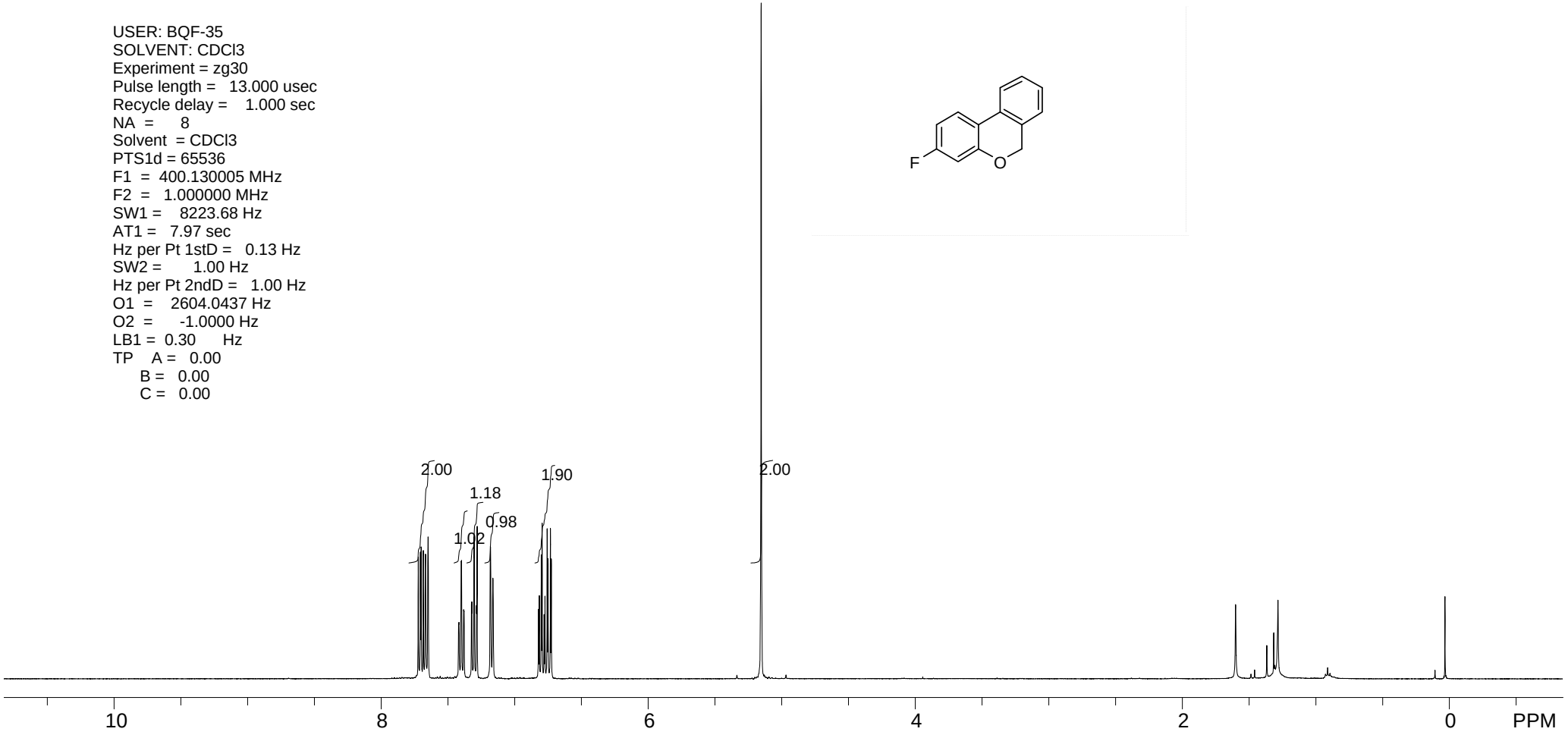


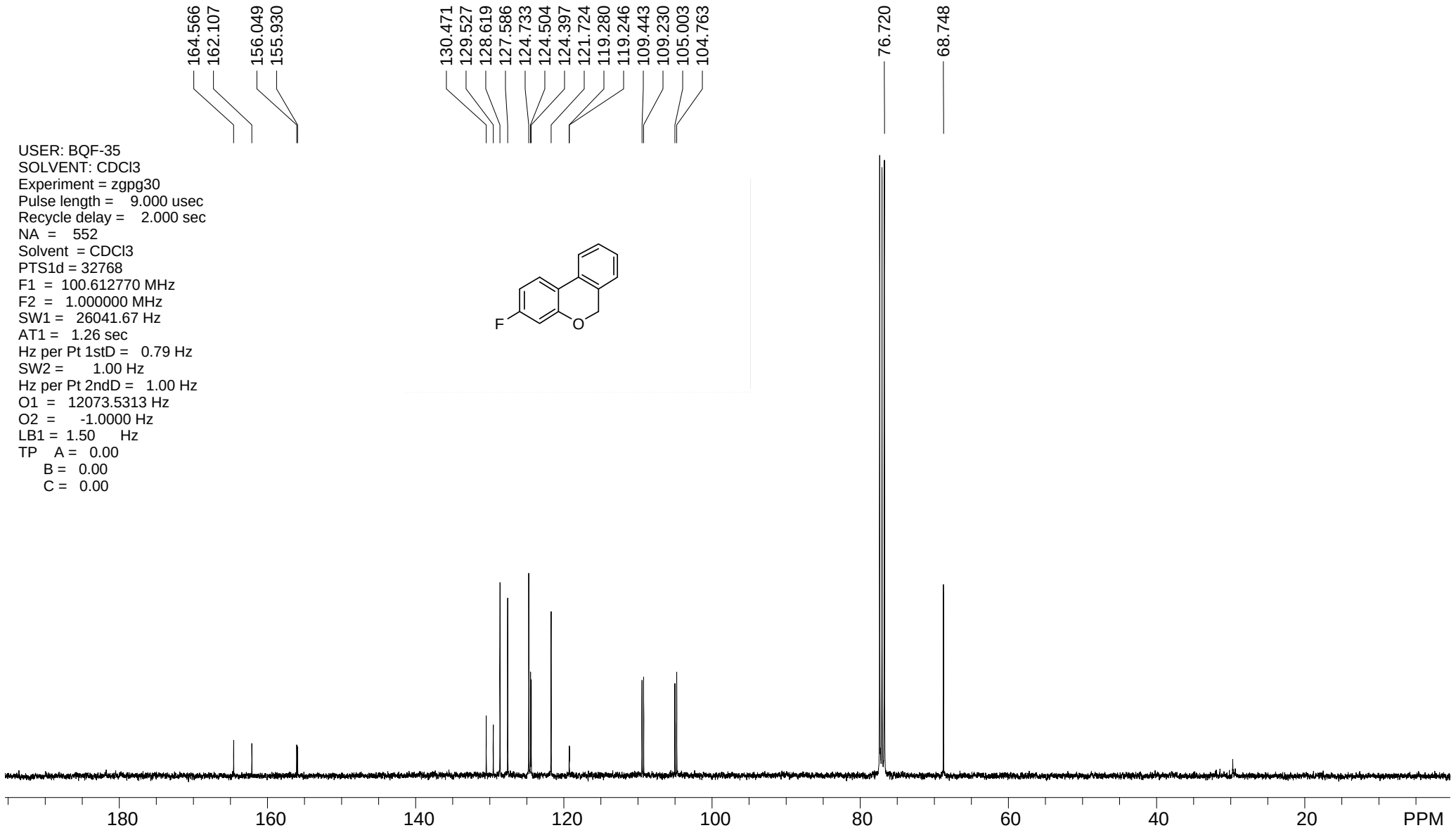

7.701
7.695
7.484
7.281
7.196
7.190
7.175
7.168
7.140
7.078
7.058
6.943
6.921
5.099
2.432
USER: BQF-48
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00
Cl
O
0.99
0.93
1.90
0.99
0.97
2.00
2.95
10
8
6
4
2
0
PPM

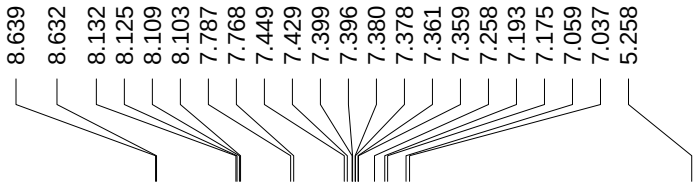

153.328
138.325
129.073
128.924
128.851
128.422
127.069
124.677
124.497
123.098
122.780
118.728
68.426
21.512
USER: BQF-48
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.000 usec
Recycle delay = 2.000 sec
NA = 270
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 26041.67 Hz
AT1 = 1.26 sec
Hz per Pt 1stD = 0.79 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 12073.5313 Hz
O2 = -1.0000 Hz
LB1 = 1.50 Hz
TP A = 0.00
B = 0.00
C = 0.00
Cl
O
180
160
140
120
100
80
60
40
20
PPM

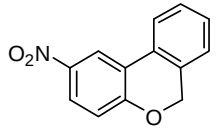


USER: BQF-35
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00

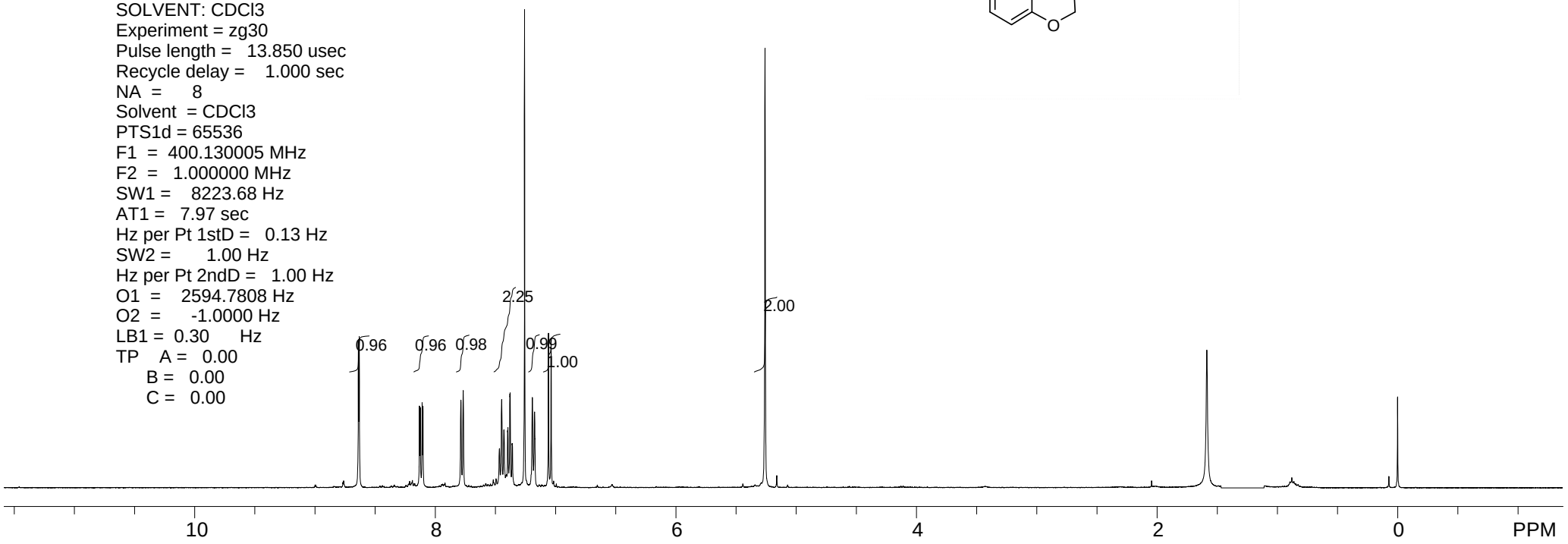


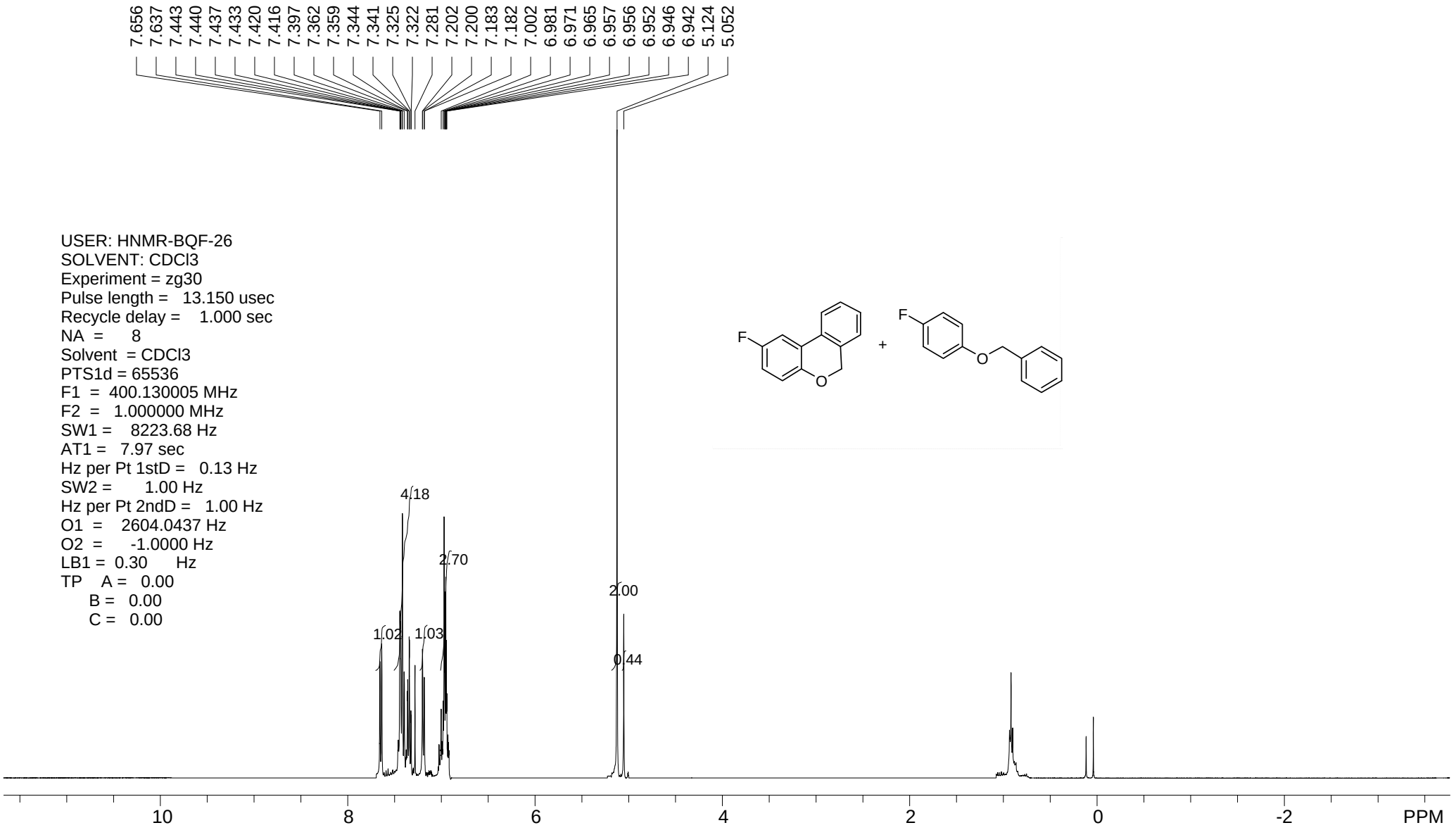

164.566
162.107
156.049
155.930
130.471
129.527
128.619
127.586
124.733
124.504
124.397
121.724
119.280
119.246
109.443
109.230
105.003
104.763
76.720
68.748
USER: BQF-35
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.000 usec
Recycle delay = 2.000 sec
NA = 552
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 26041.67 Hz
AT1 = 1.26 sec
Hz per Pt 1stD = 0.79 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 12073.5313 Hz
O2 = -1.0000 Hz
LB1 = 1.50 Hz
TP A = 0.00
B = 0.00
C = 0.00
F
O
180
160
140
120
100
80
60
40
20
PPM

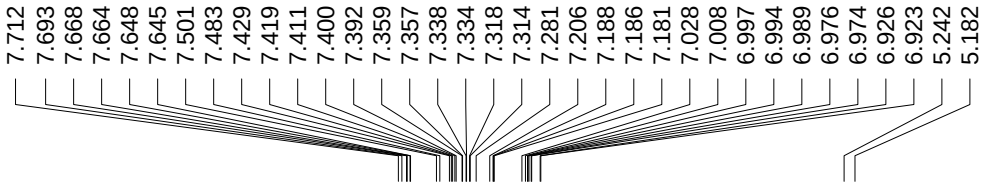




USER: HNMR-BQF-326
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.850 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2594.7808 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00




7.656
7.637
7.443
7.440
7.437
7.433
7.420
7.416
7.397
7.362
7.359
7.344
7.341
7.325
7.322
7.281
7.202
7.200
7.183
7.182
7.002
6.981
6.971
6.965
6.957
6.956
6.952
6.946
6.942
5.124
5.052
USER: HNMR-BQF-26
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.150 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00
4.18
2.70
2.00
1.02
1.03
0.44
F
O
+
F
O
10
8
6
4
2
0
-2
PPM



USER: HNMR-BQF-27
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 13.150 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8223.68 Hz
AT1 = 7.97 sec
Hz per Pt 1stD = 0.13 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2604.0437 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00